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ECN Workshop

THE EUROPEAN COMPOST NETWORK - TEN YEARS OF SUCCESSFUL ENGAGEMENT IN BIOWASTE MANAGEMENT

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EXECUTIVE SUMMARY

This presentation sets out the ethos and main activities of the European Compost Network since its inception ten years ago in 2002.

The European Compost Network (ECN) was established against the backdrop of the EU Landfill Directive (99/31/EC). Introduced in 1999, the Directive set, for the first time, targets for each member state to divert biodegradable municipal waste (BMW) from landfill. A significant proportion of this BMW would be suitable for biological treatment, such as composting and anaerobic digestion. Whilst this would be easily achieved by some member states, others from the then EU15 would face considerable challenges. Countries, such as Austria, Germany, Italy and the Netherlands, had in place established biological treatment infrastructure, with mature composting and anaerobic digestion sectors. These member states would be able to accommodate any additional biowaste diverted from landfill, whilst others with heavy reliance on landfill, such as the UK, Ireland and Greece, would need to plan, design and build new capability, thus facing many technical and practical challenges. In addition, there was speculation that the European Commission would develop a so-called “Biowaste Directive”. The European biological treatment sector thus needed an informed voice in Brussels to provide technical representation and argue its corner.

So why not learn from practitioners in these countries to share knowledge and technical know-how? This was the rationale for the establishment of ECN. Lead by Josef Barth and Prof Bidlingmaier in Germany, colleagues from Denmark, Italy and the UK came together to discuss options and formulate a plan of action. Following a series of meetings, ECN was formally launched in October 2002 in Budapest, Hungary. Initially, an initiative of the ORBIT Association, ECN set out to become the "Competent Reference Point for Biological Treatment in Europe". It would work through its Country Representatives, drawn from the membership, Operational Board and a series of Working Groups. The central ethos of ECN was to influence European policy, share information, develop quality criteria and work collaboratively with other organisations.

Shortly after its formation, the European Commission published a series of working documents on the management of biowaste, setting out its broad policy objectives. ECN was central to these discussions, representing its members in numerous meetings in Brussels. Communications on soils, the Animal By-Products Regulations, Waste Framework Directive, End-of-Waste criteria, and, latterly, the Fertiliser Regulation all followed suit. ECN has had an input into all of these, and continues to provide technical and strategic knowledge into the decision making process.

In addition to this policy work, over the past ten years ECN has disseminated vast amounts of information. It has organised a series of workshops across Europe on diverse topics, such as odour, compost marketing and anaerobic digestion. It continues to run the biennial ORBIT scientific conferences in conjunction with partners, such as IRSTEA. ECN has collaborated (and continues to collaborate) with other organisations representing the resource and energy sectors, being instrumental in forming a “Biowaste Alliance” in 2004. As if this wasn’t enough, ECN has led the way in develop a pan-European Compost Quality Assurance Scheme, aimed at harmonising compost quality assurance and defining a common standard.

These initiatives and work of ECN will be outlined in this presentation.

ECN-QAS - EUROPEAN APPROACH FOR QUALITY ASSURANCE OF COMPOST AND DIGESTATE

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EXECUTIVE SUMMARY

This presentation presents the concept of the European quality assurance scheme for organic resources in respect to quality criteria for compost and digestate.

Quality assurance schemes for compost and digestion products have been established themselves in the last 20 years successfully in various European Member States as a key element for the sustainable recycling of organic residues. The market and demand for quality composts and digestate increased on account of the beneficial properties of organic fertilisers and soil improver, the need for organic matter and continuously increasing prices for mineral fertilisers. This development and also the intention by the European Commission to define an End-of-Waste standard for compost and digestate led to a demand for European uniform quality standards for composting/digestion plants and composted/anaerobic treated products. The European Compost Network ECN met this challenge and developed a concept for a European quality assurance scheme. This concept includes quality standards for recycled organic resources, especially for compost and digestate, to facilitate the free cross-border movement of goods in the EU.

Based on the criteria laid down in the European standard “General criteria for certification bodies operating product certification” (EN 45011) and the experiences of existing quality assurance organisations in the field of biological waste management the ECN-QAS specifies requirements for quality assurance organisations in respect to aerobic and anaerobic treatment of bio-waste. Additional to these criteria specific requirements for the sustainable management of bio-waste with the aim to produce high quality products are given.

Quality assurance leads to improve the confidence of consumer in recycled organic materials and to increase the markets for organic goods. The pre-condition for a reliable European quality assurance is to harmonise the parameter of the treatment process and the requirements for product quality and to check them regularly by an independent control. Therefore ECN has set up a comprehensive quality assurance scheme including a positive list for input materials, requirements for the treatment process and quality criteria for the end products with declaration for their specific use.

1 INTRODUCTION

Quality assurance schemes for compost and digestion products have been established themselves in the last 20 years successfully in various European Member States as a key element for the sustainable recycling of organic residues. The market and demand for quality composts increased on account of the beneficial properties of organic fertilisers and soil improver, the need for organic matter and continuously increasing prices for mineral fertilisers. This development and also the intention by the European Commission to define an End-of-Waste standard for compost led to a demand for a European uniform quality standard for compost plants and composted products. The European Compost Network ECN met this challenge and developed a concept for a European quality assurance scheme within its working group “Standardisation and Quality Assurance”. This concept includes the characterisation of quality standards for recycled organic resources especially for compost and digestate with the aim to facilitate the free cross-border movement of goods in the EU.

1.1 Background

Accompanied by long-lasting discussions about an EU-wide legislation for biowaste which lately ended in a Communication of the EU Commission about biowaste [1] and the intention to regulate “the point when compost ceases to be waste” in the Waste Frame Directive [2] ECN started to work on Europe-wide quality assurance scheme for compost (ECN-QAS) based on the existing national quality assurance systems (QS) and experiences in the member organisations. The requirements for quality assurance organisations, for the process management and the quality of compost are laid down in the ECN-QAS Quality Manual, which was published in 2010. With the on-going discussion and the publication of the 2nd working document of the European Commission [3] to define End-of-waste criteria for biodegradable waste subject to biological treatment the European Compost Network started to enlarge the ECN-QAS Quality Manual with a European quality assurance scheme for digestate.

1.2 Targets of the European concept

The target of the set-up of an EU-wide quality assurance scheme for organic resources is to define Europe-wide standards for quality management and quality products from recycling processes. The pre-condition for a consistent quality is to harmonise the parameter of the treatment process and to check them regularly by an independent control. The target of ECN-QAS is to set a common basis for the existing quality schemes in Europe and to support Member States defining quality standards and developing a quality assurance scheme for composts and digestate.

2. Elements of the ECN-QAS

The ECN-QAS represents an independent quality assurance scheme and includes fundamental requirements for national quality assurance organisations (NQAO) for organic resources and basic requirements for a European compost standard in the first instance, followed by a European digestate standard. Besides the positive list for suitable input materials and requirements for process quality the scheme also includes quality criteria for the final product and analysing methods.
The European quality assurance includes:

- The conformity assessment of quality assurance schemes in European countries including the awarding of the ECN-QAS Conformity Label.
- Regular assessment of the production in the plants by the national quality assurance organisation (NQAO) by means of process requirements.
- Regular sample taking and analysis of the final product considering relevant quality parameters from independent, acknowledged labs and additionally the evaluation of the results by the national quality assurance organisation (NQAO).
- Documentation by the national quality assurance organisation (NQAO) with information about the quality properties of the product, legal requirements, the necessary compost declaration and information about use and application rates according to good practice.
- Awarding of the ECN-QAS Quality Label to composting or digestion plants by the national quality assurance organisations (NQAO).

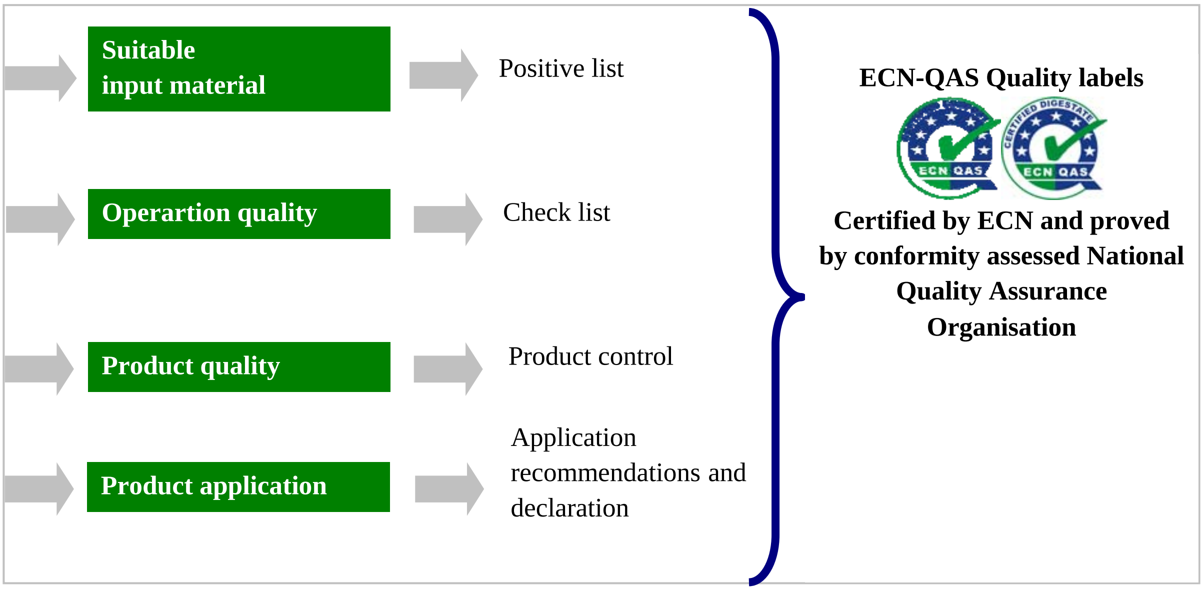

FIGURE 1: Concept of the European Quality Assurance (ECN-QAS) for compost and digestate

2.1 Process requirements

Process requirements for the production of composts are laid down in the ECN-QAS. These include the use of input materials defined in a positive list. The waste numbers and denominations of the European Waste Catalogue have been adopted and if necessary supplemented by explanations and specific requirements. As a basic principle only separately collected organic wastes are accepted.

Requirements on the process management and its documentation are defined too. This includes minimum guidelines about the adherence of process parameters to guarantee a sufficient sanitisation of the product. By means of a check list the operation quality of the plant is controlled and assessed through the NQAO at the plant inspection visit every two years.

2.2 Product quality

Quality products of organic resources have a widely homogenous composition based on defined input materials and are highly qualified humus products, which are placed on the market as soil improvers and organic fertilisers. They are used to preserve the content of organic matter in the soil and thus influence bio-diversity and soil fertility in a positive way. The fertilising efficiency of compost and digestate can be characterised via their nutrient content, whereby longer time periods, especially for compost, must be calculated (crop rotations) compared to mineral fertilisers. Further criteria, relevant for various applications, especially with the use as mixing components in growing media, are among others the plant compatibility and the salt content of the material.

TABLE 1: Quality criteria of the European Quality Assurance Scheme for the characterising of quality products

	Parameter	**Assessment**
Soil improvement	Organic matter	≥ 15 %, declaration
	Alcaline effective materials (CaO)	Declaration
Fertilisation	Nutrients (N; P, K, Mg)	Declaration
Material properties	Plant compatibility	Benchmark accord. to the test on germinable plants, declaration
	Water content	Benchmark for peak content, declaration
	Bulk density/volume weight	Declaration
	Grain size	Declaration
	pH-value	Declaration
	Electric conductivity	Declaration

2.3 Aspects the protection of the environment and consumers

Important parameters for the market of recycling products are the not desired ingredients which in quality assured organic resources can be reduced to a minimum through separate collection of bio-wastes together with an optimised process management which are harmless and environmentally safe for the individual application. Hereby to be named is the content of heavy metals and impurities like plastics, metals and glass, germinable seeds and plant parts

In order to achieve the quality label limit values must be kept for these parameters. They are based on a research [4] which has been contracted by the Commission's DG Environment in the framework of the development of the European Soil Protection Strategy. The formation of these limit values was the result on the comparing assessment of the present compost qualities in Europe with the pre-condition that separate collection of bio-waste is established in the countries. In order to comply with the precautionary requirements of the environment and consumer protection the long-term accumulation of harmful matter in the soil has been considered when identifying the limit value levels.

TABLE2: Precautionary requirements on the protection of environment and consumers

	Parameter	Assessment
Hygienic aspects	Salmonellae	0 in 25 g DM
Undesired ingredients	Impurities (glass, metals, plastics)	≤ 0.5 % DM
	Germinable seeds and sprouting plant parts	≤ 2 per litre
Harmful matter **Precautional limit values**[1]	**Heavy metals**	**mg / kg DM**
	Lead (Pb)	130
	Cadmium (Cd)	1.3
	Chromium (Cr)	60
	Copper (Cu)[2]	300[3]
	Nickel (Ni)	40
	Mercury (Hg)	0.45
	Zinc (Zn) [2]	600[3]

[1] Amlinger, F. et al. 2004: Heavy metals and organic compounds in waste used as organic fertilisers.

[2] Copper and zinc are classified as essential nutrients. Values over 110 mg Cu kg^{-1} DM and over 400 mg Zn kg^{-1} DM must be declared.

[3] These values are classified as benchmarks.

3 Quality monitoring

Quality monitoring of recycled products is realised through regular sample taking and analysing by independent labs which are recognised by the national quality assurance organisation. A pre-condition for such approval is a regular participation in ring tests.

The basis for the analysis of soil improvers and growing media are European Standards (EN). The analysing methods will be actualised correspondingly to the development in the European Standardisation of analysing methods. However, national analysing methods are also accepted in the ECN-QAS, so far as they are legally requested.

4 Certification

The national quality assurance organisation approved by the ECN-QAS is responsible for the monitoring of the bio-waste management plants and the product quality. The conformity check of the NQAO is executed in regular terms by the ECN Quality Manager. The approval is given by the Quality Committee of the ECN. In case of a successful participation in the ECN-QAS the national quality assurance organisation will award the ECN-QAS conformity label.

FIGURE 2: The ECN Conformity Label for national quality assurance organisations

Composting and digestion plants can be awarded with the ECN Quality Label by the national quality assurance organisation if they can proof the compliance with the ECN quality standards. The report of the analysis results will be sent to the Quality Committee of the ECN for monitoring and documentation.

This system doesn't replace the autonomy of national quality labels and certifications. However, it makes clear that a uniform product quality in the European context is given which simplifies com-post marketing over the country borders.

FIGURE 3: ECN Quality Label for compost and digestate

5 Forecast

The Commission started already to work on product standards for biodegradable waste subject to biological treatment under the Waste Framework Directive [1] [2]. The Member States are asked to actively participate in the process. In the report "End of Waste Criteria" [5] published by the European Commission in 2008 and as well in the current working document of the European Commission [3] a possible concept for end of waste criteria for composts and digestate - as proposed by ECN – was included and it was pointed out that the monitoring of a product standard for composts and digestate should be connected to a uniform, independent system of quality assurance. This would definitely contribute to legal security and deregulation of national control measures.

The current edition of the ECN-QAS Quality Manual includes the requirements for compost and can be ordered by ECN. After the revision in 2012 the ECN-QAS Quality Manual will be completed with the requirements for digestate.

ACKNOWLEDGEMENTS (IF ANY)

The European Quality Assurance concept ECN-QAS and the ECN-QAS Quality Manual was worked out by the members of the ECN working groups "Quality Assurance" and "Anaerobic Digestion". Thanks are given to all of them.

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UNITED KINGDOM - EXPERIENCE WITH IMPLEMENTING AND PROMOTING THE QUALITY PROTOCOLS FOR COMPOST AND DIGESTATE

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EXECUTIVE SUMMARY

The leading industry standard for composted source-segregated biowastes, BSI PAS 100, has become well known within the UK's waste management industry. It was developed in 2002 by Association for Organics Recycling's (AfOR) with the British Standards Institution, with support from the Waste and Resources Action Programme (WRAP). Its scope allows composting of controlled, source-segregated, biodegradable wastes, and it includes minimum compost quality criteria. Its sustained promotion by AfOR and WRAP has strengthened demand for quality composts in horticulture, landscape, agriculture and other markets. Building on experience with PAS 100, an equivalent specification for anaerobic digestates, BSI's PAS 110, was published in 2010.

Prior to 2007, compost derived from controlled biodegradable wastes was, in legal terms, still 'waste' even if compliant with BSI PAS 100. This meant that waste-derived compost could only be applied to land under waste regulatory controls, regardless of its quality. This placed significant administrative and legislative burdens on the composting industry and inhibited the recovery of controlled biodegradable wastes and their diversion from landfill for a number of years. In 2007 the Waste Protocols Project, a WRAP and the Environment Agency joint initiative funded by Defra, the Welsh Assembly Government and Northern Ireland Environment Agency Waste Protocols, delivered its first Quality Protocol for compost (CQP). This was followed in 2009 by the Quality Protocol for Anaerobic Digestate (ADQP). Independent certification of compliance with CQP / AD QP criteria is considered sufficient to ensure that the treated material can be classified as fully recovered and used without risk to the environment or harm to human health. The CQP and ADQP encourage businesses to transform controlled biodegradable wastes into valuable resources, rather than send them to landfill. Supply of compost and digestates as 'products' means that it is quicker and cheaper to apply them to soil and they are perceived to be of higher value as they do not carry the stigma associated with 'waste'. AfOR's Compost Certification Scheme and the Renewable Energy Association Limited's Biofertiliser Certification Scheme provide a framework for independent assessment and certification of compost and digestate compliance with, respectively, the CQP and the ADQP. WRAP's market development initiatives, compost supplier directory, freely available lists of composters who make CQP certified composts, and the general awareness raising carried out each year during Compost Awareness Week have contributed to continuing strong compost market growth and promotion of PAS 100 and the CQP. AfOR has also carried out a number of initiatives to promote its Compost Certification Scheme and the composts certified under this scheme, and to encourage market demand for and use of CQP composts. Promotion of PAS 100, the CQP and AfOR's Compost Certification Scheme has included supply of joint AfOR and WRAP press releases to media contacts. They communicated messages about the high quality of CQP composts, tonnes produced and locations where they are made, and highlighted the required independent assessment and certification under AfOR's Compost Certification Scheme. Other notable initiatives include training courses, that include highlight of drivers in policy and legislation and emphasise the market opportunities associated with high quality compost products; inclusion of PAS 100 and CQP in assessment criteria associated with compost producers' applications for capital support and equipment financing; and WRAP's extensive R&D programme and programmes aimed at building confidence in the use of composts and digestates, which have led to increased confidence in quality compost and digestates. Thanks to these initiatives and industry recognition of the benefits of producing and supplying composts and digestates with 'product' status, 1.43 Mt per annum of CQP compost is produced from about 160 certified composting processes in the UK each year from – around 50% of the total output in the UK. PAS 110 digestate is a less developed product, estimated to comprise 106 Kt of digestate output in the UK (from four sites as of May 2012). With 11 operators now having either achieved PAS 110 or registered for the Biofertiliser Certification Scheme (BCS) and working towards it, it is forecast that output could reach ~322 Kt digestate by the end of 2012/13.

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FRANCE - QUALITY EXPECTATIONS OF FARMERS AND CONSUMERS

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EXECUTIVE SUMMARY

This paper associates three complementary contributions respectively brought by Suez Environnement/Terralys and by 2 representatives of the agricultural sector, that will dress a comprehensive feedback regarding the current state of recycled fertilising products quality level and use in France and more particularly in west of France.

JL Martel will present the French context and recall the history of the two specific national end of waste standards giving the ability to various recycled compost to be put on the agricultural market and sold as organic soil improvers. Then he will give the specific return of experience of Suez Environnement concerning the production, the quality control procedures and the marketing of the three French main types of recycled compost : green waste compost, sludge compost and MSW compost ; this presentation will rely on the quick description of 3 typical composting plants respectively located in Rhone Alpes , Haute Normandie and Brittany regions and of the quality profile and use of each corresponding type of compost.

B Decoopman will give specific information on the regional quality insurance system called " CERAFEL" developed in Britanny for the use of organic fertilising products on vegetable crops and its specific application to MSW compost since 2005 developed in partnership with the municipality of Launay-Lantic where is located the first french MSW composting plant that has been revamped and that is able to produce a high quality MSW compost due to the introduction of the selective collection of domestic hazardous waste as batteries and chemicals and to the integration in the process line of an efficient refining equipment. The technical specifications of the "CERAFEL" quality standard and the obligations of the MSW compost producer will be described and commented.

E. Porcher is cultivating cereals crops on a 140 hectars farm located in the Vendée department and will bring his seven years return of experience about the use of sludge compost before oilseed rape crop. Compost origin and quality and compost use and fertilising effect integration in the fertilisation plan of the cultivation plots will be successively given and commented.

Session 1

(14) INCREASE OF BIO-WASTE RECOVERY WITH LOW - COST TECHNOLOGIES IN SAN CARLOS CITY, PHILIPPINES

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EXECUTIVE SUMMARY

Solid waste management (SWM) is a serious and growing problem in many developing countries. Although environmental awareness is increasing, the existing waste management systems cannot cope with accelerating waste generation, especially in fast growing urban areas. Hence, the Philippines released a new waste management law, the Republic Act 9003, in January 2001. Despite this law, waste disposal remains uncontrolled at many dumpsites that lack management, monitoring and environmental protection. Consequently, leachate and gas emissions are generated and pollute air and water resources and threaten public health and the environment.

San Carlos City is a medium-sized city with 133,000 residents located on the northeast site of Negros Island. To enhance the performance of its municipal waste management system the Local Government Unit (LGU) of San Carlos established a "*new way*" of SWM by segregating, collecting and managing all kind of municipal solid waste, especially by recycling organic wastes, the main generated waste fraction with > 50% of all collected waste. To provide the needed waste processing facilities the City launched a new Eco-Center in the year 2007. This technical measure was supported by designating local SWM officers, so called "C*ity Wards*", that are responsible for supervision and enforcement of SWM at community level and intense community information campaigns. The new Eco-Center integrates an information center, a sorting plant, a composting site and a clay-lined landfill with leachate treatment facility. All components were developed utilizing appropriate, low-cost technologies. The sorting facility, also called "*Gravity MRF*" was designed as in-house-unit whereby incoming waste can be segregated during down-passage along several slides by utilizing gravity force. All incoming wastes at the Eco-Center are segregated respectively processed and recorded on a daily basis. With the established technology, a waste diversion rate of around 60% can be performed since start of operation in 2007. From the collected 6,568 tons solid waste in 2010, around 3,395 tons of organic materials could be segregated and composted afterwards. Consequently, the organic waste content in the residual waste fraction for disposal at the landfill could be reduced significantly. Due to the enhanced waste segregation the useful lifespan of the landfill can be increased, likewise the potential for methane and leachate emissions lessened that would result if organic waste would be disposed at the landfill. Besides, the produced compost also provides useful soil enhancer for local farming. Considering that RA 9003 demands a waste diversion rate of 25 %, the performance of the Eco-Center San Carlos surpasses this target by far and hence was recognized as best practice with many regional and national awards. By applying a Climate Calculator for GHG assessment developed for the SWM sector, it was found that the increased material recovery at the Eco-Center results in reduction of GHG emissions of around 7,600 tons $CO_{2\text{-eq}}$/year (net) if compared with the baseline scenario 2005 when all collected wastes were disposed at a dumpsite and material recovery merely conducted by waste pickers.

San Carlos City is one of the first LGUs in the Philippines that did not only implement the new waste legislation but also showed that low-cost technologies coupled with community involvement are a very suited approach to enhance municipal SWM systems. The developed innovations proofed successful since available manpower and equipment could be more easily adjusted to the new project and local conditions. San Carlos City demonstrates that in the context of a developing country, the enhancement of municipal SWM is feasible and can be sustained, even with restricted budgets. In the meantime several other LGUs on Negros, Cebu and Guimaras Islands follow the unique Eco-Center approach and established similar SWM projects.

1 INTRODUCTION

1.1 Background

San Carlos City is one of the component cities of the Province of Negros Occidental in the Visayas region with a population of around 133,000 (Census 2010). The city lies at the western site of the Tanon Strait that separates Negros Island from Cebu Island and comprises a total land area of 45,150 hectares. San Carlos City is accessible by land through several highways but also provides access to sea transport with its national harbor. The city is the third largest urban area on Negros Island. Main economical activities are sugar cane, coconut, fruit, rice and fish farming as well as bio-ethanol production. The city provides various central services such as banking, schools, college education and hospitals.

Based on an average waste generation rate of 0.44 kg/day/resident as stated in the municipal SWM plan (San Carlos, 2006), a total of 58 tons of solid wastes are generated daily. However, the municipal waste collection serves only 41,250 residents living at the urban proper and adjacent barangays. Barangays are settlements that also represent the smallest administrative and political units in the Philippines. Presently, the city collects an average of 18 tons solid waste/day, mainly from the city center. According to the local waste ordinance, wastes have to be segregated at source into biodegradable, special and residual wastes. Materials that can be recycled at household level or directly sold to junkshops are not collected by the city but have to be taken care of by the households respectively delivered to the local Materials Recovery Facility (MRF). The Local Government Unit (LGU) collects solid waste at defined collection points along the roads, at central public locations such as the public market, city plaza, bus terminal, harbor, schools, hospitals, institutions and from commercial establishments.

Figure 1: Location of Study Area

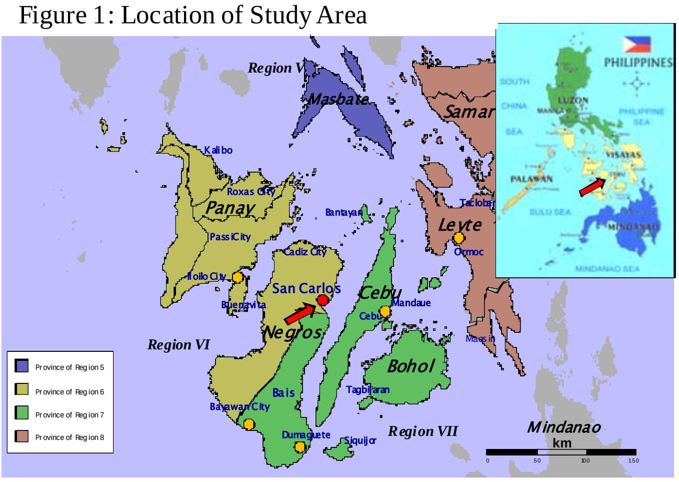
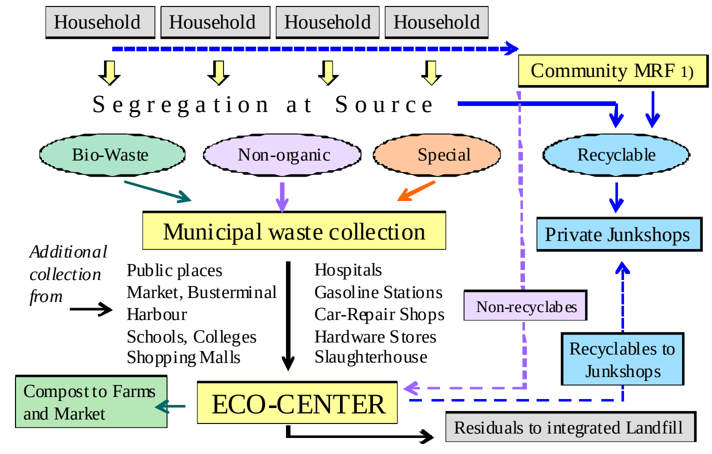


Figure 2 displays the main elements of the local SWM system in San Carlos and its interconnections. The households bring their segregated solid waste either to the local MRF or to the designated points for the municipal waste collection. The City transports the segregated wastes to the new City Ecological Center (Eco-Center), which is located around 7 km south from the urban proper. The Eco-Center is leased from a private owner but operated by the SWM Office of the LGU. This 5-hectare site has been operated since September 2007 and provides various facilities for waste management such as a training and information center, a sorting plant, a composting site, a Sanitary Landfill (SLF) and a leachate treatment facility. Prior to the launching of the Eco-Center solid waste was disposed at an open dumpsite without sorting, pretreatment, compaction and leachate and gas emission controls. During that time materials recovery was basically performed by the informal sector particularly waste pickers that recovered only sellable materials such as metals, hard plastics and paper/cardboards in a “*raisin-picking*” approach. At that time organic wastes were mainly disposed because composting was hardly practiced at household and municipal level.

Figure 2:Waste management flow San Carlos City



1) – local facility to be operated by every barangay, the name for smallest municipal unit in Philippines

1.2 Legal framework

The Republic Act 9003, also known as the "*Philippine Ecological Solid Waste Management Act*" that came into effect in the year 2001, promotes the paradigm that waste is a resource that can be recovered. It stipulates that LGUs are the primary institutions to implement this act, and promotes active collaboration with the private sector and associations working on SWM. It encourages reduction of waste at source, recovery, reuse and recycling of wastes, which lead to mandatory targets. The law mandates at least 25% waste diversion by the year 2006 with increasing values every three years thereafter. "*Waste diversion*" refers to activities that reduce the amount of solid wastes from disposal facilities. Diversion activities include the processing, composting, recovery and recycling of collected materials from the municipal waste stream (RA 9003). To achieve this goal, every community has to establish a Materials Recovery Facility (MRF), implement segregation at source and process recyclables and biodegradables. While Republic Act 9003 emphasizes the importance of reduction, reuse and recovery, it also prohibits waste picking in segregation areas or disposal facilities, unless the owner or operator allows it. However, the waste legislation also encourages the participation of cooperatives and associations providing them opportunities to integrate the private sector into SWM. Although Republic Act 9003 sets targets for waste diversion, composting, material recovery and dumpsite closures, regulations regarding waste-to-energy options or other alternative waste treatment technologies such as mechanical-biological waste treatment or biomass utilization for energy generation are not clarified. As a first regarding initiative, the Department of Environment and Natural Resources (DENR) formulated a Department Administrative Order (DAO), which regulates the recovery and utilization of Alternative Fuels and Raw materials (AFR) for co-processing in cement kilns (DENR, 2010). A further relevant legislation is the Climate Change Act of the Philippines (Republic Act 9729) that was released in October 2009. This law requires municipalities to formulate local Climate Action Plans. The relevance of SWM in climate mitigation was emphasized in the National Climate Change Framework, which states that the SWM sector is third largest GHG emitter in the Philippines. The SWM sector accounts to 11,6 Mio tons CO_2eq/year avoidable emissions, which represent 11% of all anthropogenic GHG emissions in the country (Climate Change Commission of the Philippines, 2010).

1.3 Research objectives

The research aims to analyze approaches, strategies and low-cost technologies of the new SWM system of San Carlos City as a case study. Although RA 9003 was already released in 2001 and LGUs were mandated to close their dumpsites at the latest in 2006, most municipalities fail to establish waste segregation and phase out of waste dumping. Hence the study also identifies main parameters and conditions that are relevant for LGUs to implement the legal prescriptions through an integrated and innovative approach within the context of a developing country. Further objective of this study is to assess the socio-economic and environmental benefits as well as the sustainability potential of the established new Eco-Center.

2 METHODOLOGY

Already prior to the launching of the Eco-Center in 2007, all SWM tasks of the LGU were planned, coordinated and supervised by the established SWM office. This office has the following tasks and functions:

- SWM planning and reporting to the local SWM Board,
- Community information, education and outreach program,
- Waste collection, segregation and street sweeping,
- Material recovery, composting and residual waste management,
- Project and environmental monitoring,
- Reporting for the LGU and involved supervising authorities.

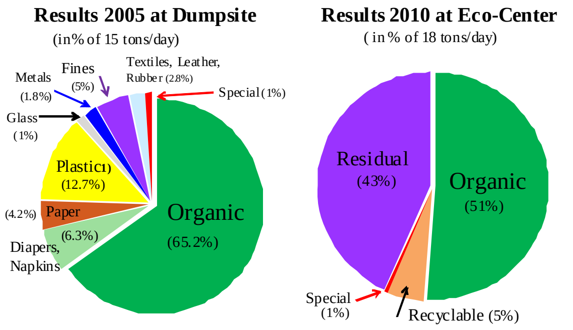
The author's have conducted several field visits and interviews with involved LGU employees. Documents regarding planning, operation and monitoring of the applied SWM systems were provided by the LGU and were used to obtain data from waste generation, input/output for waste collection and processing, financing and information about applied technologies and their efficiency. Additionally, a Climate Calculator developed by the German International Cooperation agency (GIZ) and the German Bank for Reconstruction (KfW) was utilized to assess the baseline scenario and benefits of the project related to the Greenhouse gases (GHG) emission situation (Giegrich & Vogt, 2009).

3 MATERIAL RECOVERY IN SAN CARLOS AND FUNCTIONS OF THE ECO-CENTER

3.1 Strategies for the implementation of the new SWM system in San Carlos City

Like many other LGUs, San Carlos City was confronted with the complex problem of increasing waste generation due to rapid urban development. To enhance the performance of its municipal SWM system the LGU established a "*new way*" of SWM implementation. The approach includes segregating, collecting and managing all kinds of municipal solid waste with emphasis on recycling organic wastes that are contained with >50% in the collected waste. To provide the needed processing facilities, the City launched a new Eco-Center in 2007. This measure was supported by designating local SWM officers, so called "*City Wards*", that are responsible for SWM enforcement at community level. Furthermore, the city established a SWM Board and involved a Non-Governmental Organization (NGO), called GENESYS Foundation to guide the involved stakeholders and development process. This NGO conducted school and community outreach programs and waste characterization studies to clarify the needed baseline data. Figure 3 summarizes the main findings of the waste characterization studies for the baseline situation in 2005 and compares them with the enhanced situation at the Eco-Center in 2010. In 2005, organic wastes formed the bulk of collected waste with around 65%. Plastic residuals (12.2%), used paper (4.2%) and Diapers/Napkins (6.3%) together accounted for further 23%. Based on the waste composition data it was decided that the segregation system should at least separate non-organic, organic and special wastes. This measure was designed to enhance organic waste processing and productivity at the Eco-Center to avoid potential environmental impacts from the mixing of bio-waste with special waste during the collection process. To guide the proposed development process the LGU formulated the following principles:

Figure 3: Waste collection & processing San Carlos City



Total SW collection 2005:	5,475 tons	Total SW collection 2010:	6,586 tons
Biowaste disposed in 2005:	3,570 tons	Biowaste recovered 2010:	3,395 tons
Non-organic recycled 2005:	345 tons (6.3%)	Non-organic recycled 2010:	329 tons (5%)

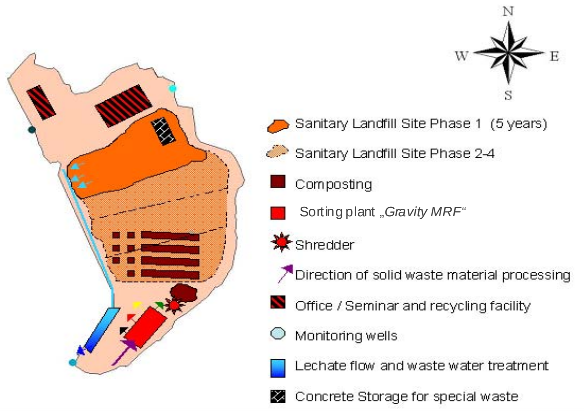
1) from the plastic waste fraction around 2.5% sellable as hardplastic, from the paper fraction around 2% in 2005

- Households have to segregate their wastes, whereas mixed wastes will not be collected,
- Waste will be collected in a segregated manner by the LGU,
- The new system will be supported with intense Information, Education and Communication Campaigns (IEC),
- The LGU implements a new Eco-Center that provides all needed facilities for final waste sorting, materials recovery, composting and waste disposal and establishes a new SWM office to supervise the new SWM system,
- Barangays that are located outside the collection area temporarily store their residual waste and coordinate later with the SWM office for final disposal at the Eco-Center, but conduct material recovery and composting on their own.

3.2 Main elements of construction and operation of the Eco-Center

The new Eco-Center started its operation in September 2007. The facility can be accessed through an unpaved, all-weather road. Main facilities are a sorting plant ("*Gravity MRF*") for final processing of waste, an administrative office, a motor pool, truck washing area, a mechanical-biological treatment and windrow composting area, vermiculture facility, storage places for recovered materials and special waste, perimeter fence and a 2 hectare clay-lined landfill for disposal of residual waste with leachate treatment plant. The MRF is a unique innovation that utilizes gravity force to slide down waste materials for segregation to increase material recovery as far as possible. Prior to the operation of the Eco-Center, materials recovery was mainly conducted by waste pickers at the dumpsite that had chosen to recover waste as their livelihood. Many of them were later on recruited by the LGU to conduct waste segregation works at the new sorting plant. Main objective of this measure was to formalize their employment status, enhance their working conditions and to utilize their skills for the newly established waste segregation process.

Figure 4: Site development plan Eco-Center San Carlos



The various elements of the Eco-Center were designed as appropriate technologies to make use of low-cost approaches whenever possible. Table 1 summarizes the main technical components of the Eco-Center and their corresponding investment costs.

TABLE 1 **Main technical elements and their investment costs at the Eco-Center San Carlos [1)]**

Component	**Total in PhP-Peso**	**Total in US-$**	**US-$/m^2 SLF**
Land Area: 10-year lease contract for 5 hectares	1,500,000	34,884	0.70
Investment cost to develop landfill cell 1 (0.66 hectare) [2)]			
1. Excavation works	599,979	13,953	2.11
2. Cost for base clay-liner of landfill	1,750,014	40,698	6.17
3. Cost for gravel liner and sand protection layer	1,299,976	30,232	4.58
Investment to establish supporting infrastructures at the Eco-Center (5 hectares) [3)]			
4. Drainage, leachate collection and treatment	1,000,008	23,256	0.46
5. Perimeter fence	799,972	18,604	0.37
6. Establishment of 3 groundwater monitoring wells	239,983	5,581	0.11
7. Energy supply	609,998	14,186	0.28
8. Composting facility	199,993	4,651	0.09
9. Office building	350,020	8,140	0.16
10. Central Material Recovery Facility (MRF)	1,000,008	23,256	0.46
11. Other developments (earthmoving, access road, etc.)	500,004	11,628	0.23
TOTAL	**9,849,955**	**229,069**	**15.72 [4)]**

1) – cost calculation based on an exchange rate of 43 Philippine Peso per 1 US-$
2) - costs calculated under positions 1-3 are divided by 6,600 m^2 landfill area to establish cost/m^2
3) – costs calculated under positions 4-9 are divided by 50,000 m^2 Eco-Center area to establish cost/m^2
4) – the average fill height of the landfill is projected with 8 m, whereas only 3 hectares of the Eco-Center area will be developed as landfill. Hence, the costs per m^3 disposed waste are: [(50,000 m^2/30,000 m^2)/8m] * 15.72 = 3.75 US-$/m^3 respectively 1.9 US-$/ton disposed waste

The Eco-Center was designed to substantially increase materials recovery. Prior to 2007, more than 5,000 tons of solid wastes were disposed at the former dumpsite. With the operation of waste processing technologies at the Eco-Center, the amount of residual waste disposed could be reduced to < 3,000 tons per annum. Table 2 summarizes the input/output data of the baseline situation 2005 and the Eco-Center operation since September 2007.

TABLE 2 **Waste collection, segregation and disposal data Eco-Center San Carlos (in tons)**

Year (in tons) [1)]	**Total collected waste (tons/year) [tons/day]**	**Segregated organic waste (tons) [%]**	**Recyclables (tons) [%]**	**Residual waste disposed at SLF**	**Total operation cost (US-$) [5)]**
2005 [2)]	*5,475 [15]*	-	*345 [6.3]*	*5,130 [93.7]*	*134,884*
2007	7,157 [19.6]	2,460 [3)] [34.4]	717 [10.0]	3,980 [4)] [55.6]	269,767
2008	7,375 [20.2]	2,837 [38.5]	1,195 [16.2]	3,006 [40.8]	286,046
2009	7,177 [19.6]	4,418 [61.6]	428 [5.9]	2,331 [32.5]	239,535
2010	6,586 [18.0]	4,316 [65.5]	329 [5.0]	1,941 [29.5]	355,814
Total 07-10	**28,295**	**14,031 [49.6]**	**4,095 [14.5]**	**11,258 [39.8]**	**1,151,162**
Average/ton	**-**	**-**	**-**	**-**	**40.68**

1) – calculation of tonnage derived from m^3 measurements of LGU waste collection with average density of 0.3 tons/ m^3
2) – 2005 was chosen as baseline with dumpsite operation and low material recovery, mainly performed by waste pickers (data excluded in total sum)
3) – start of Eco-Center operation in September 2007, the calculation includes 4 month compost production at the Eco-Center in 2007
4) – from that around 2,800 tons were disposed at the former dumpsite
5) – based on exchange rate 43 PhP Peso for 1 US-$; total costs include waste collection and Eco-Center operation with employment of 24 workers

As shown in Table 2, the rate of materials recovery at the Eco-Center always surpassed 60% of waste input since the year 2009. The total amount of delivered waste as well as the amount of non-organic recyclable materials has been decreasing lately. This indicates that the intense IEC program of the LGU resulted in enhanced waste segregation and materials recovery at source. According to the data displayed in Table 2, the average total cost for the management of 1 ton solid waste (SW) in San Carlos are estimated with 40.68 US-$/ton. Based on the average waste generation of 0.44 kg/capita, each of the 41,250 served residents would have to pay annual SWM fees in the amount of 6.53 US-$ for a 100% cost recovery. This fee does not include investment cost for constructing the Eco-Center as listed in Table 1.

4 RESULTS AND DISCUSSION

4.1 Performance evaluation of the Eco-Center San Carlos

Most LGUs in the Philippines report that collected municipal wastes contain more than 50% biogenic materials. However, the failure to segregate bio-waste at source results in collection and disposal of organic materials with all related negative consequences for the environment such as leachate and methane generation (IPCC, 2006; Visvanathan, 2009). It appears that the organizational and technical efforts to segregate bio-waste and to process respectively market compost are too complex to be easily conducted and sustained by municipal departments. In general, composting of municipal bio-waste needs to be subsidized and hence depends more on municipal budgets and political will rather than consumer behavior and the market situation (Hoornweg et al, 2000; Paul et al, 2008; Alamgir, 2011). Unfortunalely, some of the efforts made by housholds that segregate their bio-waste are made futile by waste collectors who mix the various segregated wastes when emtpying the bins into the collection truck, a practise that discourages waste segregation at community level (De Guzman et al, 2006). Further issues are the lack of knowledge and low commitment to implement the legal prescriptions by many stakeholders. Albeit, composting offers many benefits such as producing humic substances to feed soils, increase of soil fertility, soil structure and water retention capacity, substitution of chemical fertilizers and others (Rothenberger & Zurbrügg, 2006; Bidlingmaier, 2009).

The LGU San Carlos established a new SWM system that efficiently segregates and processes all collected municipal wastes with a material recovery efficiency > 60%. The new system reduces waste disposal to a significantly lower level if compared with the baseline scenario in 2005. At that time all collected wastes were still disposed at a local dumpsite and material recovery merely conducted by waste pickers. Since start of operating the Eco-Center in 2007, a total of around 14,000 tons of organic waste and 4,095 tons of recyclable materials were recovered. The value of the recovered materials alone is estimated with 1.5 Mio US-$, whereas the cost for disposing these materials would have been around 0.74 Mio US-$. This means substantially cost savings for the LGU. Based on conducted laboratory analyses of compost samples, the produced compost (ca. 5,600 tons) is a valuable organic fertilizer that contains in total around 45 tons of Nitrogen (N), 60 tons of Phosphates (P) and 75 tons of Potassium (K) and a bulk of humic substances and micro-elements that largely contribute to sustain healthy soil conditions. Besides, harmful impacts of bio-waste disposal such as leachate or methane emissions are by far reduced since the disposed residual waste at the Eco-Center hardly contains any organic substances. By establishing a new SWM system, the LGU was able to provide the needed facilities to ensure that the various waste generators are served and supported in their SWM needs. The established SWM system is considered as very cost-efficient since 1 ton of SW management can be conducted for only 40.68 US-$ including all municipal SWM activities. This would translate to a SWM fee of only 6.53 US-$/cap/year based on an average waste generation rate of 0.44 kg/cap/day, an amount that could easily be refinanced by users if compared with other public services such as energy or water supply. Furthermore, the LGU San Carlos established additional 24 permanent jobs to operate the Eco-Center whereby many of the former waste pickers could be employed and their skills to segregate wastes utilized.

4.2 Assessment of climate benefits

Climate change is a serious environmental concern and subject of increasing research and debate in recent years. Many national environmental programs inspire to mainstream assessments of "*carbon footprints*" at the early stages of project planning to mitigate GHG emissions. To assess the GHG emission contribution of SWM systems, a GHG Calculator that was developed by the German International Cooperation agency (GIZ) and the German Bank for Reconstruction (KfW) was applied as a new tool to evaluate and compare GHG emission projections for the baseline situation 2005 and the operation of the Eco-Center in 2010. The tool standardizes the calculation of GHG emissions for typical SWM technologies such as controlled dump, landfill with and without gas collection, mechanical-biological treatment of waste prior to deposition, mechanical-physical stabilization and co-processing in cement kilns, recycling, composting and digestion of organic components. The user can choose which options to be compared. The user may use primary data, however, a set of default values is provided which can be used if data base is inadequate. Detailed information on how to apply and to chose applicable values for calculation are given in a manual (Giegrich & Vogt, 2009).

To clarify the GHG emission situation for SWM in San Carlos the following two options were considered:

- Scenario 1: Baseline situation in 2005 with mixed waste collection and disposal at a dumpsite. Waste recovery was mainly performed by waste pickers and restricted to the recovery of around 6% sellable materials such as scrap metals, hard plastics, glass and paper/cardboards. It was assumed that from the delivered waste at the dumpsite around 90% of metals, 10 % of cardboards and some 25 % of hard-plastics were recovered by waste pickers,

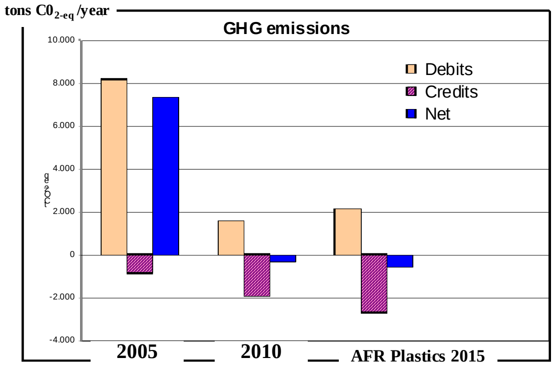
- Scenario 2: Established Eco-Center and recovery of 90% of all collected organic waste through composting as well as up to 95% recovery of recyclable materials such as plastic, metals and paper/cardboard.

The main input data for the GHG calculation are summarized as follows:

- Number of inhabitants: total of 133,000 whereas only 41,250 are served by the SWM,
- Waste mass collected: 6,570 tons/annum with specific waste mass: 0.44 kg/cap/day,
- Calorific value of total waste: 7,5 MJ/kg with total carbon content: 26,9%,
- wet waste with regenerative carbon of 17% of total C,
- Specific GHG production: 557 gCO_2 /kWh.

Figure 5 summarizes the results of the climate calculator application for the baseline scenario 2005 and the new Eco-Center in 2010. Debits of the displayed scenarios refer to GHG emissions resulting from deposition of waste in a landfill but also emissions caused by processing, e.g. to run machineries and provide energy needs etc. Credits are a result of recycling of components when recycled materials substitute new materials or when the treatment process itself avoids or lessens GHG emissions if compared with waste disposal. Net GHG effects are the sum of both debits and credits and are decisive for decision makers. Numbers are given as $CO_{2\text{-eq}}$ emissions in tons/year. All in all, three scenarios were elaborated applying this GHG Calculator for an annual waste collection rate of 6,570 tons. For the baseline situation 2005, GHG emission (net emission) of around 7,340 tons $CO_{2\text{-eq}}$/year are estimated. For the scenario 2010 with operation of the new sorting plant (*Gravity MRF*) and the composting facility at the Eco-Center, the GHG emissions were assessed with -330 tons $CO_{2\text{-eq}}$/year (net emissions). Hence the operation of the Eco-Center results in an emission reduction of > 7,600 tons $CO_{2\text{-eq}}$/year if compared with the baseline situation. To further reduce waste disposal the additional recovery of light-density plastic foils, bags, packages is proposed that are presently disposed at the landfill. These materials could well be utilized as Alternative Fuels and Raw materials (AFR) as already practiced by other LGUs, e.g. in Iloilo City. This would assist to further mitigate GHG emissions related to SWM respectively lengthen the useful lifespan of the landfill (DENR, 2010; Paul and Soyez, 2011). Although this measure would be a valuable contribution to further reduce waste disposal, the related GHG emission reduction would only be in the magnitude of 380 tons $CO_{2\text{-eq}}$/year (net emissions) if compared with the situation in 2010. Lastly, it should be mentioned, that GHG emissions may vary significantly for the various applied composting technologies (Chan et al, 2010; Ermolaev et al, 2012). Hence, the discussed findings of the Climate Calculator assessment need to be verified in a detailed manner, especially if carbon credits or other incentives of the carbon market are targeted.

Figure 5: GHG emission scenarios for the SWM system Sans Carlos for the years 2005, 2010 and 2015
(based on GIZ Climate Calculator; Giegrich & Vogt, 2009)



5 CONCLUSIONS

San Carlos City is one of the few Local Governments that has successfully implemented the provisions of the new waste management law. It further pioneered to design and test innovative approaches and low-cost technologies that can be sustained by a LGU in the context of a developing country. The developed innovations proved effective since available manpower and equipment could easily be adjusted to the local conditions operation requirements of the new Eco-Center. Based on the experiences of San Carlos City, a high degree of materials recovery (greater than 60% of all collected waste) can only be achieved by providing the needed mechanism for community information and participation, e.g. with a "*No Segregation-No Collection*" strategy coupled with intense community education and SWM organization at local level. The involvement of a local Non-Governmental Organization for community outreach and special school education programs for SWM greatly contributed to institutionalize waste segregation as an accepted habit among households and business establishments, e.g. with the message "*Throw Your Habits – Not Waste*". Likewise, the designation of SWM focal officers ("*City Wards*") greatly supported the enforcement of the SWM program at the local level. The various components established at the Eco-Center proved efficient and resulted in almost complete recovery of organic waste (50%) and non-organic, recyclable materials such as metals, hard plastics and paper/cardboard (around 10%). Consequently, residual waste disposed at the new landfill hardly contains organic components.

This results in substantial cost savings for the municipal government and enhanced landfill operation with extended lifespan and reduced emissions. Based on the results of a GHG emission assessment significant emission reductions were already achieved with the new SWM system. Whereas 7,340 tons CO_2-eq/year net emissions were calculated for the baseline scenario 2005, the new established SWM system with 60% material recovery reduces GHG emissions with 7,600 tons CO_2-eq/year resulting even in GHG benefits of 260 tons CO_2-eq/year. With that, San Carlos City demonstrates that in the context of a developing country, the enhancement of municipal SWM including climate mitigation aspects is feasible and can be sustained, even with restricted budgets. The fact that the municipality has managed to sustain the newly established SWM system over a time period of five years supports this statement.

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(93) ANAEROBIC CO-DIGESTION OF BROWN WATER WITH KITCHEN WASTE IN DECENTRALIZED, SOURCE-SEPARATION-BASED SANITATION CONCEPTS

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EXECUTIVE SUMMARY

Current centralized sanitation systems adopted by many developed and developing countries minimize the exposure of wastewater to citizens by flushing them away from households. However, the transportation process consumes large amounts of energy and water and gives rise to diluted sewage. In contrast, decentralized sanitation systems that treat source separated wastewaters would encourage the recycling of nutrients for agriculture, reduce household water consumption significantly and generate a source of clean energy. This study is initiated by an attempt to redefine urban communities as renewable resource recovery centres through the adaptation of “decentralized and source-separation-based sanitation concepts.

In this concept, the wastes are separated into various streams. A source separation toilet would be used to separate urine and feces from the souce. Other waste streams include grey water (from showers and cleaning) and kitchen waste. Among the various household wastes, brown water and kitchen waste which are high in organic matter are suitable for energy recovery by anaerobic digestion. This study presented the potential of using brown water and kitchen waste as feed sources for energy production. Mesophilic anaerobic co-digestion of brown water with kitchen waste in bench-scale (5 L) and lab-scale (30 L) reactors were carried out. The objective was to compare the performance between single-stage and two-phase systems, and between continuous stirred tank reactors (CSTR) and sequencing batch reactor (SeqBR) for the initial 110 days of operation. Monitoring parameters like reactor pH, COD, biogas volume and composition, VS, TS and VFA were analyzed twice a week. The kitchen waste/ brown water mixture was prepared daily and fed to the reactors in a semi continuous mode. All the reactors were operated in parallel and had an initial organic loading rate (OLR) of about 1 g COD/L.d. For the first 40 days of operation (i.e. OLR of 1 g COD/L.d), the effluent quality in terms of organic fraction was almost similar in all the configurations, with COD, TS and VS removal efficiencies of about 80, 55 and 65%, respectively. However, SeqBR was found to have higher organic removal efficiencies as compared to CSTR after the OLR increased to 2-3 g COD/L.d. Based on the bench-scale study, subsequent scaling up to 30 L lab-scale adopted the two-phase CSTR/ SeqBR digester configuration with similar operating conditions. During the initial 75 days of operation, the lab-scale two-phase CSTR/SeqBR digester showed better performance, as illustrated by the higher TS, VS, total COD and soluble COD removal efficiencies than the bench-scale reactors.

This study showed that mesophilic anaerobic co-digestion in two-phase CSTR/ SeqBR was a suitable technique for the treatment of brown water with kitchen waste. SeqBR systems were better at retaining more solids as compared to CSTRs, giving rise to improved waste stabilization and higher biogas production. Two-phase systems are still preferred over single stage systems as kitchen waste has the tendency to acidify rapidly, thus lowering the pH of the digester. Further evaluation of the performance of the two-phase CSTR/ SeqBR would be conducted before subsequent scaling up to pilot and full-scale demonstration plants in selected residential high-rise buildings in Singapore. The proposed decentralized and source-separation-based anaerobic digestion system is expected to provide a practical solution for managing large quantities of brown water and kitchen waste on-site. The well established infrastructure for sewer, waste collection and transportation are readily available for simple modifications to transfer the concentrated wastes directly to decentralized co-digestion systems. This, together with the high potential for producing a substantial amount of energy in the form of biogas, makes the proposed decentralized AD concept economically realistic.

1. INTRODUCTION

Sanitation issues may seem insignificant as compared to many other problems the society face today. However they are important because poor sanitation brings serious health risks and directly affects our quality of life. To minimize the exposure of wastewater to citizens, current sanitation systems flush away human excreta from households to centralized wastewater treatment plants. However, the transportation process consumes large amounts of energy and water and gives rise to diluted sewage. In contrast, decentralized sanitation systems that treat source separated wastewaters would encourage the recycling of nutrients for agriculture, reduce household water consumption significantly and generate a source of clean energy. Communities generate several different wastewater streams - yellow water refers to urine, brown water refers to faeces, black water refers to faeces mixed with urine, and grey water refers to wastewaters from showers and washing. Decentralized sanitation systems carry out the conversion of wastewater high in organic content (brown water/ black water) with kitchen and food waste to biogas using anaerobic digesters. Anaerobic digestion (AD) has been widely applied in the treatment of organic wastewaters due to its high degree of waste stabilization and methane generation. Methane is a potentially valuable by-product that can be used as a fuel in producing heat and electricity, and the significance of this advantage is increasingly being emphasized in our energy conscious society. Having a fuel value of approximately 5,850 kg-cal/m^3, biogas has been widely used in heating digesters and gas engines (L. K. Wang et al., 2007).

The separation of different wastewater streams and their treatments with the aim of energy production and nutrients reuse was demonstrated in the year 2000 within a housing estate for 350 to 400 inhabitants (Otterpohl et al., 1997) in the pilot project Flintenbreite in Luebeck, Germany. The concept comprises vacuum toilets with subsequent pasteurization and anaerobic digestion of black water together with kitchen waste in a semi-centralized biogas plant and finally recycling of the digested anaerobic effluent in agriculture. Previous research activities on the AD of black water in rural areas demonstrated successfully the feasibility of treating human waste in decentralised sanitation systems (Wendland et al., 2007; Kujawa-Roeleveld et al., 2005; Zeeman et al., 2008). However, source separation between feces and urine, and its subsequent resource recovery approach is limited. Moreover, such research in the urban context has been scarce. With increasing population and concerns over water and fuel shortage, an onsite decentralised and anaerobic treatment of domestic wastewaters in urban cities might be a more sustainable solution to the current centralized aerobic wastewater treatment plants.

An ongoing project with the objective of converting community wastes (brown water with kitchen and food waste) to energy is carried out by our research group ‘Residues and Resource Reclamation Centre (R3C)’ of Nanyang Technological University (NTU), Singapore. Other than significantly reducing daily household flush water consumption, the source separating toilet using 0.3 L of water per urine flush (yellow water) and 2.0 L water per feces flush (brown water) produces low diluted brown water that facilitates the subsequent energy recovery process. Yellow water is considered a good source of plant-available nitrogen and phosphorus that could be treated and used as fertilizer and soil amendments (Heinonen-Tanski et al., 2005). Other than improving the potential of energy recovery, the addition of kitchen waste as a co-substrate would also help to raise the current food waste recycling rate of 12% in Singapore.

This study carried out the mesophilic anaerobic co-digestion of brown water with kitchen waste in bench-scale (5 L) and lab-scale (30 L) reactors. The objective was to compare the performance between single-stage and two-phase 5-L CSTRs, and between CSTRs and SeqBR for the initial 110 days of operation. The reactor configuration with the better performance would then be adopted in the lab-scale (30 L) reactor study using the same feeding material.

2. METHOD AND MATERIALS

2.1 Feedstock and inoculum

The feed for the reactors in this study consisted of a mixture of kitchen waste and brown water in the ratio of 150 g/ 2L. Kitchen waste was collected once in a week from one of the canteens at NTU campus, where the majority of the waste came from Chinese, Indian, Indonesian and Malay food stalls. It was a mixture of meat, rice, noodles, vegetables and salad. After removing bones and non-food materials, the FW was then crushed by a kitchen blender to promote homogeneity of the substrate as well as disintegration of particulate organics. The blended FW was then mixed well, and stored in a refrigerator at 4°C. Brown water refers to fecal waste without urine. It was collected from a specially designed source-separation toilet located in our laboratory, where urine and feces was collected in separate tanks. Brown water used in this study was mixed with 2 L of flush water, collected once in a week and stored in a refrigerator at 4°C. Inoculation was carried out for CSTR and SeqBR systems using seed sludge collected from an anaerobic digester at the Ulu Pandan sewage treatment plant, Singapore. The pH, average total solids (TS), volatile solids (VS) concentrations and VS/TS of inoculum were in the range of 7.1, 24.9 g/L, 17.7 g/L and 0.71, respectively.

2.2 Experimental set-up

2.2.1. Bench-scale reactors

Anaerobic co-digestion of the kitchen waste/ brown water mixture was performed in the semi-continuously fed lab-scale reactors *viz.* two-phase continuous stirred tank reactors (CSTR), single stage CSTR and single stage sequencing-batch operational mode reactor (SeqBR). Two-phase CSTR consisted of acidogenesis reactor of 1.2 L working volume followed by methanogenesis reactor of 4.1 L (working volume). Both single stage CSTR and SeqBR had a working volume of 5.3 L each. All the reactors were operated inside a controlled-temperature room at 33^{o}C and fed once daily. The methanogenesis reactor of the two-phase CSTR was fed with the acidified effluent from the acidogenesis reactor. CSTR was operated with continuously mixing (mixing time: 5 min ON/5 min OFF) at 80 rpm using an overhead mechanical stirrer while SeqBR was operated in cycles, such that one cycle length consisted of 24 h, i.e. filling (1 h), reaction or mixing time (20 h); settling time (2 h); draw (30 min) and the ideal phase (30 min). The reactors were inoculated with anaerobic sludge (50% by volume) from an anaerobic digester treating municipal wastewater with a concentration of 17.8 g TS/L and 12.5 g VS/L. Gradually the reactor content was replaced by the kitchen waste/ brown water mixture.

2.2.2. Lab-scale reactors

A two-phase CSTR/ SeqBR configuration was used for the co-digestion of the kitchen waste/ brown water mixture for the up-scale to 30 L reactors. It consisted of an acidogenesis reactor (CSTR) of 7 L and methanogenesis reactor (SeqBR) of 23 L working volume. HRT of the acidogenesis reactor was maintained at 5 days while that of methanogenesis reactor was at 25 days throughout the initial 75 days of operation. The operating conditions are identical to that of the bench-scale CSTRs.

2.2.3. Waste input sampling and analytical procedures

The composite samples of the shredded feedstock and effluents (from the single-stage, two-phase CSTR's and SeqBR) were taken bi-weekly for characteristic analysis. pH value were measured using a compact titrator (Mettler Toledo) equipped with a pH probe (Mettler Toledo DGi 115-SC). Alkalinity, total (TS) and volatile (VS) solids were analysed according to the Standard Methods (APHA, 1998). Total and soluble chemical oxygen demand (COD) measurements were made using COD digestion vials (Hach Chemical) and a spectrophotometer (DR/2800, Hach). Soluble COD measurements were made using the supernatant of samples after centrifugation (KUBOTA 3700, Japan) at 10,000 rpm for 15 min. The determination of volatile fatty acids (VFAs) was carried out using a gas chromatograph (Agilent Technologies 7890A, USA), equipped with a flame ionization detector (FID) and a DB-FFAP (Agilent Technologies, USA) column (30 m x 0.32 mm x 0.50 µm) and the samples were filtered through Membrane Solutions 0.45µm cellulose acetate membrane filters. Total biogas production was monitored daily using a mass flow meter (McMillan Company, Model 50D-3E), while the biogas composition (methane, carbon dioxide and nitrogen contents) was analyzed by gas chromatograph (Agilent Technologies 7890 A, USA) equipped with a thermal conductivity detector (TCD). Carbon, hydrogen, nitrogen and sulfur were measured using an elemental analyzer (Vario EL Cube), and aqueous TOC and IC was measured using a TOC analyzer (TOC-V CSH, Shimadzu, Japan).

3. RESULTS AND DISCUSSION

3.1. Bench-scale digesters performance

The feed mixture was prepared daily and fed to the reactors in a semi continuous mode. All the reactors were operated in parallel and had an initial organic loading rate (OLR) of about 1 g COD/L.d. The acidogenesis and methanogenesis reactors were operated at a HRT of 4 and 16 days, respectively while both the single-stage CSTR and SeqBR were operated at 20 days HRT. For the first 40 days of operation, the effluent quality in terms of effluent organic fraction was almost similar in all the configurations, with COD removal efficiencies of more than 80% and TS and VS removal of up to 55 and 65%, respectively. However, when the HRT was decreased to 16 days by increasing the OLR, the performance of CSTR digesters (both single-stage and two-phase) dropped due to washout of active biomass. Total and soluble COD removal efficiencies dropped to 55 and 60%, respectively for the subsequent 50 days (i.e. about 3 times the HRT of 16 d). This was accompanied by a pH drop due to the accumulation of VFAs. In contrast, SeqBR was able to retain more solids in the reactor, which resulted in better removal of organics (93% for soluble COD and 77% for total COD). Thus, the HRT of CSTR digesters was later adjusted back to 20 days while that of SeqBR remained at 16 days. Table 1 presents the average organic matter removal efficiencies for all the reactors throughout the initial 110 days study period.

Table 1: Characteristics of influent, hydrolysate and effluent of bench-scale reactors

	HRT (days)	pH	TVFA (g-COD/L)	Soluble COD (g/L)	Removal Efficiencies (%)			
					TS	VS	Total COD	Soluble COD
Influent	-	6.2 ± 0.5	1.82 ± 0.50	12 ± 3	-	-	-	-
Hydrolysate	20	4.2 ± 0.1	8.40 ± 2.42	12 ± 4	-	-	-	-
Two-phase CSTR		6.9 ± 0.3	1.56 ± 1.53	-	61 ± 12	68 ± 12	61 ± 18	80 ± 20
Single-stage CSTR	20	6.7 ± 0.5	1.24 ± 1.32	-	51 ± 10	59 ± 10	52 ± 16	80 ± 17
Single-stage SeqBR	16	6.9 ± 0.1	0.39 ± 0.22	-	66 ± 8	71 ± 9	67 ± 22	92 ± 5

As shown in table 1, the TS, VS and total COD removal efficiencies of two-phase CSTR was 9-10% higher than that of single stage CSTR. Comparing the single stage reactors, SeqBR showed 15, 12, 15 and 12% higher TS, VS, total COD and soluble COD removal efficiencies, respectively, than the CSTR. The biogas yield of 0.5-0.6 m^3/kg VS added was recorded during this operation period. Methane content of 60-65% was observed in the total biogas. The first part of this study showed that two-phase CSTR performed better than single-stage CSTR while SeqBR reactor had the highest performance at a higher OLR (2-3 g COD/L.d) and shorter HRT (16-d) due to its ability to retain more solids. Thus, the scale-up for lab-scale reactor would adopt the two-phase CSTR/SeqBR configuration.

3.2. Lab-scale digesters performance

Similar to the bench-scale reactors, the feed mixture was prepared daily and fed to the reactors in a semi continuous mode with an initial OLR of about 1 g COD/L.d. The acidogenesis reactor (CSTR) was operated at a HRT of 5 days and OLR of about 1 g COD/L.d while the methanogenesis reactor (SeqBR) was operated at a HRT of 20 days and fed with the acidified effluent from the acidogenesis reactor. The performance of the reactors for the initial 75 days of operation is discussed in this study.

3.2.1. Hydrolysis and acidification efficiency

During hydrolysis and acidification, particulate organic matter in the feed mixture is broken down into simple and soluble organic matter such as VFAs. VFAs are then converted into acetic acid which would be consumed by methanogens to produce methane. The HRT of the acidogenesis reactor was maintained at 5 days for the initial 75 days of operation. No other pretreatment or control techniques were followed to prevent methanogenesis during the hydrolysis and acidification process. The kitchen waste/ brown water mixture had an average pH of 6.0 ± 0.5, TVFA of 0.96 ± 0.71 g-COD/L and soluble COD of 10.45 ± 7.73 g-COD/L. After hydrolysis and acidification in the acidogenic CSTR, the hydrolysate had a pH of 4.2 ± 0.6, TVFA of 5.21 ± 2.47 g-COD/L and soluble COD of 13.52 ± 8.21 g-COD/L (Figures 1a, 1b and 1c, respectively). It is interesting to note that significant soluble COD production did not take place during the studied HRT's. However, a significant conversion of non VFA-COD to VFA-COD occurred at an HRT of 5 days. There was a 5 fold increase in TVFA levels after hydrolysis and acidification, similar to the results for 5-L bench scale reactor (Table 1). In this study, short-chain fatty acids (SCFAs) i.e. acetic (C2), propionic (C3), butyric (C4), iso-butyric (iC4), valeric (C5), iso-valeric (iC5), caproic (C6) and heptanoic (C7) were analyzed. The main acidification products were H-Ac (33% of TVFAs), H-Bu (29% of TVFAs), H-Va (27% of TVFAs) and H-Pr (8% of TVFAs) comprising 97% of TVFAs. The higher molecular weight VFAs (caproic, heptanoic etc.) were produced in insignificant amounts. All the SCFAs produced from the acid reactor were consumed by the methogens present in the methane reactor at HRT of 20 days. In particular, butyrate removal was almost complete at this HRT, and no inhibitory effects of this acid were observed in the methane reactor.

3.2.2. Organic matter removal

The effluent from the methanogenesis phase reactor showed average values of 6.9 ± 0.2 and 0.19 ± 0.22 g-COD/L for pH and TVFA levels, respectively. Figure 1d shows the organic matter removal efficiencies observed for the lab-scale two-phase CSTR/SeqBR digester. The average removal efficiencies for TS, VS, total COD and soluble COD were 66 ± 24, 75 ± 20, 84 ± 17 and 93 ± 5%, respectively. These average values were exclusive of the first 25 d, which were considered the adaptation period for the reactors. As compared to the bench-scale two-phase CSTR and single-stage SeqBR, the lab-scale two-phase CSTR/SeqBR digester showed better performance, as illustrated by the higher TS, VS, total COD and soluble COD removal efficiencies. However, long-term performance evaluation is required to have a more consistent result even for lower HRT or higher organic loading conditions.

3.3. Overall comparison

The second part of this study successfully demonstrated the up-scaling from bench-scale to lab-scale reactor by adopting a two-phase CSTR/ SeqBR configuration, in terms of improved organic matter removal efficiencies. At HRT of 25 days, a stable and uninhibited process was achieved. Subsequently, the OLR for lab-scale reactor would be increased to 2-3 g COD/L.d by reducing the HRT from 25 to 22 days. This study showed that SeqBR systems were better at retaining more solids as compared to CSTRs, giving rise to improved waste stabilization and higher biogas production. Two-phase systems are still preferred over single stage systems as kitchen waste has the tendency to acidify rapidly, thus lowering the pH of the digester. Further evaluation of the performance of the two-phase CSTR/ SeqBR would be conducted.

3.4. Final discussion based on decentralized, source-separation-based sanitation concepts

With rising concerns over water shortage issues and the energy crisis today, the sustainable development of a society may depend, among other things, on how it handles water, sanitation and household residuals. The decentralized sanitation and reuse concept is a logical source separation-based approach that fits well in a sustainable development (Kujawa-Roeleveld et al., 2006). With rapid increase in population density in Singapore and the fact that more than 80% of the population live in high-rise buildings, the proposed decentralized and source-separation-based AD system is expected to provide a practical solution for managing large quantities of brown water and kitchen waste on-site. The well established infrastructure for sewer, waste collection and transportation are readily available for simple modifications to transfer the concentrated wastes directly to decentralized co-digestion systems. This, together with the high potential for producing energy in the form of biogas, makes the proposed decentralized AD concept economically realistic. Building AD systems near high-rise residential flats for on-site waste treatment and methane production would become an eco-friendly model for other countries, especially those experiencing rapid urbanization and water shortage. The challenge is in designing a space-friendly, odor- and pathogen-controlled AD system that can be incorporated into new and existing residential clusters. Based on our characteristics analysis of brown water and kitchen waste (data not

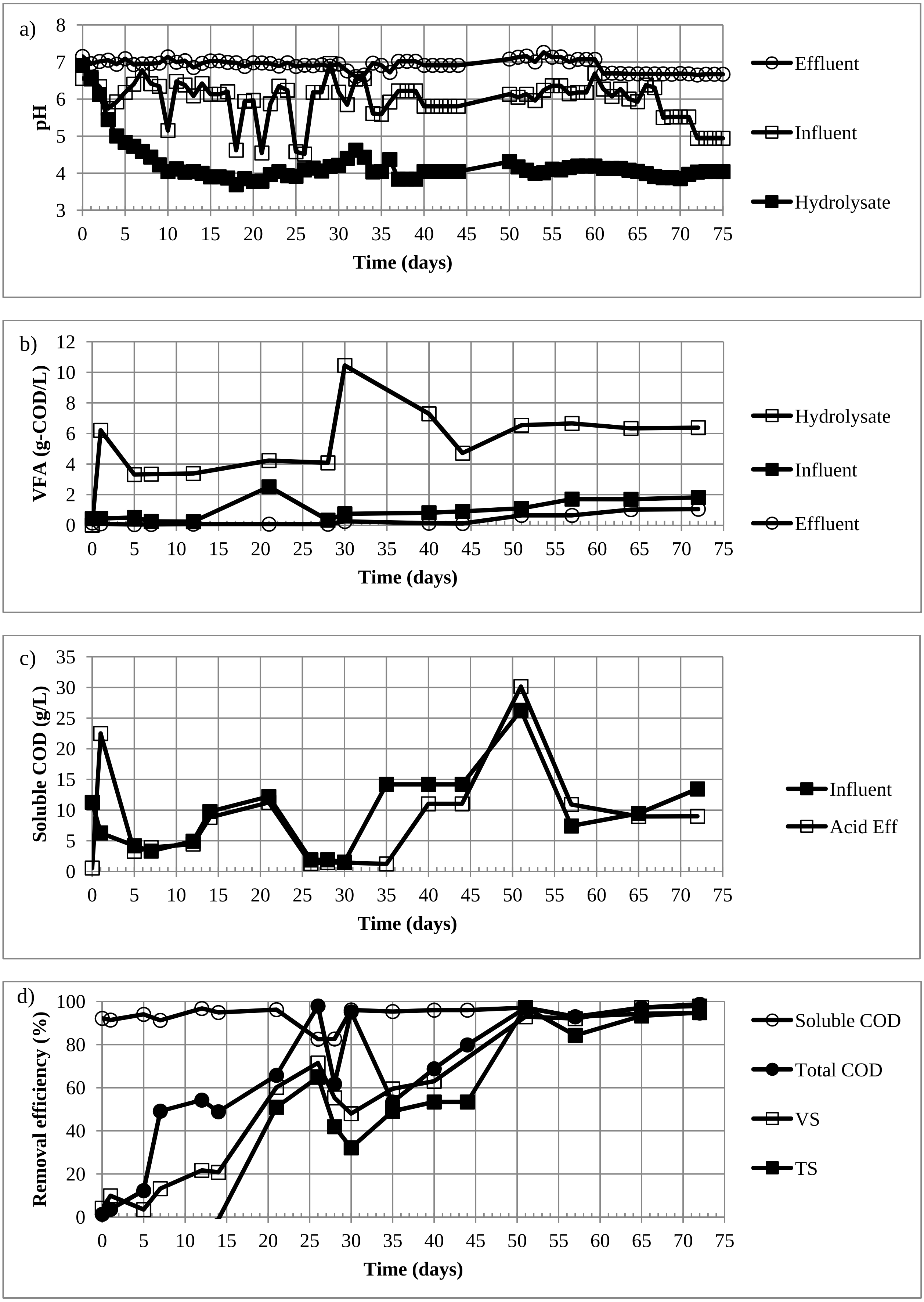

Figure 2: (a) pH, (b) VFA, (c) soluble COD levels, and (d) removal efficiencies.

shown), an estimated 21 kg VS generated through waste streams by 300 residents per building would yield approximately 10 m^3 methane considering 65% VS removal. This is equivalent to a daily electricity generation of 52 kWh assuming the generator efficiency of 50%. The energy recovered could be used for cooking or lighting purposes within the residential buildings while the digestate could be used as bio-fertilizer within the parks around.

4. CONCLUSION

Waste is not waste. Much of our disposed “waste” should not be considered waste, but misplaced “resource”. With the separate collection of yellow and brown water, the anaerobic treatment of brown water can be more stable without the high concentrations of ammonium. In this study, the anaerobic co-digestion of brown water with kitchen waste showed to be successful in waste stabilization and recovering energy from waste. The first part of this study discussed the operation of bench-scale reactors for the initial 110 days and found that SeqBR was better at retaining solids than CSTR and the two-phase system was less prone to VFA inhibition. The second part of this study discussed the initial 75 days of operation for lab-scale two-phase CSTR/ SeqBR digesters. It showed that mesophilic anaerobic co-digestion in two-phase CSTR/ SeqBR was a suitable technique for the treatment of brown water with kitchen waste. Further evaluation of the performance of the two-phase CSTR/ SeqBR would be conducted.

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(128) OPTIMIZATION OF ORGANIC WASTE HOME COMPOSTING

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EXECUTIVE SUMMARY

Within cities around the world, home composting can reduce the cost of disposing food waste (FW) and yard trimmings (YT). Besides reducing equipment and labour costs, home composting of organic waste eliminates its selective collection and mechanical processing, and reduces the fossil fuel required for these tasks as well as their resulting greenhouse gases. Nevertheless, the successful implementation of onsite composting depends on the active participation of households and the production of a safe soil amendment.

The research objectives of the project were therefore to define the operational parameters which optimize the composting process and quality. The research work was first initiated in the laboratory, at the IRSTEA (formerly Cemagref) Research Centre, of Rennes, France, using typical home composting systems (HC) loaded with an organic waste mixture consisting of equal volumes of wet FW and YT. During this experiment, the ground pile, the slatted wood bin, the plastic bin and the rotary drum were compared using different management practices (batch versus weekly feeding, weekly mixed versus not mixed, and with or without wood chips as bulking agent, BA); home composter performance was compared in terms of dry matter (DM), carbon and nitrogen mass balance, and pathogen/parasite counts. These laboratory results were validated during a second experiment conducted with the collaboration of 5 households of Montreal, Canada, where slatted and top/bottom perforated plastic bins were normally loaded and operated by the residents during the summer, while being monitored for temperature, mass loading and compost quality.

The laboratory results indicated that bin perforation had a significant impact on compost decomposition: concentrating the perforations at the top and bottom of the bin optimized convective aeration, but concentrated the decomposition at the bin bottom. Weekly mixing and good aeration also helped produce higher compost temperatures. Bulking agent addition retarded the composting process. Compost temperature regime and bin aeration or mixing had little effect on final compost quality and pathogen/parasite counts. For the experiment conducted with 5 households of Montreal, Canada, the best location for the home composter was found to be in a semi-shaded area. Once more, bins with perforations concentrated at their top and bottom produced the highest compost temperatures. Weekly loaded all bins did produce thermophilic temperatures, unless loaded with at least 10 kg of organic waste/week at over 15% DM. Despite compost temperatures seldom reaching thermophilic levels, pathogens were minimized in the final product with good bin aeration fostering a high level of organic waste degradation. To minimize the content of metals and toxic organics in the final product, the feed stock should be clean; the use of back yard herbicides and insecticides should be minimized.

1. INTRODUCTION

Being most active biologically, the organic fraction of the municipal solid waste stream is a growing concern for municipalities (Domingo and Nadal, 2009; Montejo et al., 2010). Accordingly, recycling this organic fraction through diversion and treatment has become a priority in Europe and North America (Wagner and Arnold, 2008; Burnley et al., 2007), with composting being recognized as the most practical and feasible method (Kim and Kim, 2010).

Home composting systems (HC) are recognized as a possible onsite treatment capable of reducing collection, transportation and processing costs for municipalities. Nevertheless, limited scientific studies pertain to the influence of their management practices on the quality and safety of the finished product (Köerner et al., 2008; Papadopoulos et al., 2009; Chiemchaisri et al., 2010). In West London, Smith and Jasmin (2009) studied the effectiveness of 290 L home composters in reaching thermophilic temperatures when fed kitchen waste, paper and yard trimmings (YT).

The organic waste in these HC remained mostly at psychrophilic (0-20^{0}C) and mesophilic (20-45 °C) temperatures except for a few cases reaching the thermophilic range during summer months as a result of a large input of waste. Nevertheless, the compost obtained was of good quality. In Thailand, Karnchanawong and Suriyanon (2011) evaluated the performance of six different polyethylene composting bins with a 200L capacity, batch fed with food waste (FW) and YT. The study concluded that improved temperature regimes where obtained with bins aerated through lateral perforations located mainly at their top and bottom, enhancing convective air displacement.

There is therefore a lack of knowledge for home composting, on the impact of management practices on compost quality. This project therefore investigated the effect of using bulking agent (wood chips), of regularly mixing the composter content, and of filling the composter either in batch or weekly. The home composters tested along with the management practices were: the Plastic (P) and Wood (W) bins, the Rotary Drum (RD) and the Ground Pile (GP). In the laboratory, the HC performance was compared to that of a laboratory reactor (LR). Following this laboratory investigation, the project monitored the quality of compost as produced by 5 households of the West Island region of the City of Montreal, Canada.

2. METHODOLOGY

2.1 The experimental home composters and organic waste

The laboratory portion of this study compared the performance of four commonly used home composting systems (HC) against a control, namely a laboratory reactor (LR). The 4 common HC were: a 400 L Plastic bin (P) measuring 0.70 m x 0.70 m by 0.80 m in height; a 400 L Wood bin (W) measuring 0.78 m x 0.65 m by 0.75 m in height; a 350 L metallic Rotary Drum (RD) with an internal diameter of 0.77 m and a length of 0.76 m, and; a Ground Pile (GP) measuring 0.65 m in height and 0.75 m in base diameter (Figure 1). The 300 L laboratory Reactor (LR) had an internal diameter of 0.70 m and a height of 0.80 m and had a manually controlled forced aeration system.

The source separated food waste (FW) was supplied by two restaurants of the city of Rennes, Bretagne, France. All FW was collected within 3 days of being produced and stored at 4 °C until used. The yard trimmings (YT) were obtained from the grounds of the Cemagref Research Institute of Rennes, France. While being collected, the FW and YT were sampled for physico-chemical characterization.

The field component of this study monitored the temperature regime, loading and final compost quality of 5 composters managed normally by indicial households of the Montreal West Island region. Two different types of polyethylene HC were used, namely the slatted bin and the top/bottom perforated bin. The participating households were selected randomly and coded A, B, C, D and E. The slatted composter of homeowners A, C and E offered a total capacity of 350 L while that with top/bottom perforations of homeowners B and D offered a capacity of 300 L. The organic waste (OW) fed to the composters consisted of FW and YT.

2.2 Laboratory experimental procedure

The laboratory component of this project consisted in testing the performance of, and final compost quality produced by an interior laboratory reactor (LR) and HC placed outside under a tent. This component was conducted at the Rennes IRSTEA (formerly Cemagref) Research Centre, France. All HC were randomly set-up outside under a tent to avoid rainfall and sunshine. The treatment combination applied to the HC is presented in Table 1. All HC were naturally aerated except those weekly mixed based on the assigned treatment. The compost mixture made of the same FW and YT was manually mixed and loaded without compaction into the HC. All batch-fed HC were loaded at once on the same day with a wet mixture mass of 83 to 85 kg, while the weekly fed HC were loaded weekly for 10 weeks at a rate of 8.3 to 8.5 kg/week. While filling each HC, Thermochron iButton temperature sensors (DS1921G-F5, Thermochron iButton, Dallas Semiconductor, USA) were installed at the center of the compost mass.

All composting systems were operated for 150 d. Every week during the first two months and monthly thereafter, all composts were sampled for characterization. The temperature sensors were retrieved after 70 d of composting, during one of the mixing operation. After 150 d, the HC and LR compost mass was weighed and sampled in triplicate for physico-chemical characterization.

The characterization at 0 and 150 d provided data to compute the loss in dry matter (DM), total carbon (TC), total Kjeldahl nitrogen (TKN), chemical oxygen demand (COD), and organic matter (OM) fractions, namely soluble OM, hemicellulose, cellulose and lignin. Furthermore, the final product at 150 d was analyzed for pathogens and parasites, trace elements and polycyclic aromatic hydrocarbon (PAH), to assess the compost quality and thus its suitability as soil amendment (Mato et al., 1994).

2.3 Field experimental procedure

For the field experiment, all five households were asked to normally manage their HC (Table 2). Waste log forms were supplied to each household to keep track of type, amount and date of waste loading. A 7.5 L kitchen collection bin (Norseman Plastic Ltd, Canada) and a 22 kg hook scale (Notlegalfortrade, China) were provided to each household to collect and measure the waste added to their HC. As normally done, each household manually loaded their individual HC without compaction. During the experimental 20 week period, the content of the HC was not mixed except for that of household E which was mixed occasionally.

The temperature of the HC content was measured weekly by the researcher on the project using a long-stem thermometer (PTC Instruments, Los Angeles, California, USA, model 8500D-II). This monitoring was conducted throughout the 20 week experimental period of June to October 2010. After 20 weeks, the content of all five HC was removed in 3 separate layers, namely top, middle and bottom, and triplicate samples were collected from each layer for characterization including dry matter (DM), pH, nutrients, trace elements and pathogen/pathogens. This characterization verified the compost quality and its suitability as soil amendment. The temperature regime and compost quality results were analyzed to recommend best management practices for home composting systems.

2.4 Analytical procedure

The bulk density of the fresh compost samples was determined by filling three 30 L pails without compaction, and weighing their content. The free air space (FAS) of the fresh compost samples was determined only for the laboratory tests, using an air pycnometer (Berthe et al., 2007) where an airtight cell is filled with compost and then pressurized while measuring the volume of injected air. At equilibrium pressure, the injected air provides an estimate of the free air space in the compost.

Before being analyzed, all triplicate compost samples were dried in an oven (SR 2000, Thermosi, France) at 80 °C until a constant weight was reached and then grinded to less than 0.5 mm (ZM model 1000 grinder, Retsch, Germany). The TC was determined by burning 10 mg samples at 900 °C (Thermo Scientific, FLASH 2000 Series, Organic Elemental Analyser, Courtaboeuf, France) according to AFNOR (2001a). According to AFNOR (1995), TKN was determined using an automatic distilling system (VAP 50c, Gehardt automatic distilator, Gehardt, Germany), after digesting 0.5 to 1.0 g samples with H_2SO_4 (automated Kjeldatherm TZ block digester, Gerhardt, Germany). The COD was determined by titration (Metrohm, Courtaboeuf, France) after digesting 60 mg samples with H_2SO_4 and $K_2Cr_2O_7$ (Kjeldatherm COD digestion block, CSB 20M, Gerhardt, Germany), according to AFNOR (2001b). The OM was determined by burning at 550 °C for 3 hours (Thermolyne 30400, Furnace, F30420 C-33, Essex, UK), according to AFNOR (1985). To correct all analytical results, residual moisture was determined by drying grinded compost samples at 105 ºC for 24 hours (SR 1000, Thermosi, France). The pH of wet samples was determined by soaking for 24 hours without shaking at 5ºC, in just enough distilled water to use a pH electrode (pH-Electode SenTix41, WTW, Weilhein, Germany) according to Adhikari et al. (2009). The soluble OM, hemicellulose, cellulose and lignin fractions were determined using a fibre extractor (VELP Scientific, FIWE 6 Extractor for raw fibre determination, Usmate, Italy) according to Van Soest (1963).

Escherichia Coli and Fecal Streptococci were quantified using microfiltration (AFNOR, 2001c). The presence of Salmonella in 25 g wet samples was verified using Petri plates with XLD agar (AFNOR, 2006). The presence of helminthe eggs in 1.5 g of wet sample was determined using the triple flotation technique (AFNOR, 2004). The trace elements were quantified by ICP-MS (Inductively Coupled Plasma Mass Spectroscopy) after digestion with nitric acid, and the PAH were analysed by chromatography and fluorimetric detection after extraction with hexane/acetone (AFNOR, 2000) respectively.

2.5 Statistical analyses

During the 150 d laboratory experiment, the triplicate compost samples were regularly collected for characterization. Thus, the physico-chemical characteristics were compared by the repeated measure ANOVA using the PROC GLM procedure at 95% confidence level (SAS Institute Inc., 2008).

For the field experiment and to test the effect of loading rate (kg of wet OW fed to the HC weekly) on compost temperature, the PROC ARIMA procedure was used to produced an autoregressive comparison using all five HC (SAS Institute Inc., 2008). To evaluate differences between HC, the physico-chemical characteristics of the triplicate samples collected from the top, middle and bottom layers of all five HC were also compared using ANOVA at a 95 % confidence level for within and between the HC using the PROC GLM procedure (SAS Institute Inc., 2008). Results were compared with those obtained from the laboratory experiment.

3. RESULTS AND DISCUSSION

3.1 Laboratory experiment

In terms of fed OW, the FW consisted generally of 49.9 % vegetables, 16.1 % fruit, 18.0 % root and tuber and 16.0 % cooked food residues with an average DM of 15 %, while the YT consisted of 90 % grass and 10 % tree leaves with a respective DM of 58 and 64 %. The composting formula consisted of 1:1 wet volumes of FW and YT, or 1:1:1 volume when BA was used. Loaded into the HC, the fresh mixture without BA offered a DM of 22.6 to 24.1%, a C/N ratio of 17 to 17.4 and a FAS of 66%, while that with BA offered a DM of 41 to 42 % (±1.6 – 2.5 %), a C/N of 65, and a FAS of 78 %. Adding BA dropped by half the soluble, hemicelluloses and cellulose fractions, while increasing the average lignin from 8 to 63 %. For an active microbial activity, initial compost mixtures should offer a C/N ratio between 15 and 35 and a pH between 6 and 8 (Stabnikova et al., 2005; Haug 1993; Zucconi and de Bertoldi, 1986), indicating that adding BA in this experiment may have a negative impact.

Figures 1 a, b, c and d illustrate respectively, the temperature profile obtained for the W, P, RD and GP composts exposed to the various management practices. For W, only the batch fed and weekly mixed composts produced temperatures exceeding 55 ºC, with W4 (without BA) reaching 64 ºC on day 6 compared to W1 (with BA) reaching 58 ºC one day later. The batch fed Plastic bin (P) compost reached thermophilic temperature above 60 ºC on days 3 and 6 for the treatments without BA and not mixed (P1) and without BA and mixed (P2), respectively. As for the weekly fed treatment (P3), the compost remained exposed to mesophilic temperatures. For the Rotary Drum (RD), both batch fed and mixed treatments, but without BA (RD1) and with BA (RD2) quickly reached peak temperature of 57 ºC after 3 days, but also quickly dropped to ambient on days 8 and 10, respectively. Nevertheless, RD produced thermophilic temperatures much faster than W, indicating its initial excellent capacity for aeration. The lack of sustained thermophilic temperatures resulted from the clogging of its aeration ports. All Ground Pile (GP) composts were mixed without BA. On day 4, both batch fed treatments reached thermophilic temperatures of 69 and 67 ºC, for the mixed (GP3) and not mixed (GP1) treatments, respectively, indicating a slight positive effect for mixing. The weekly fed compost never developed temperatures exceeding 30 ºC. All four HC type performed as well if not better than the LR, which, aerated at a rate of 0.05L/s, produce final compost with a high DM content.

Table 1: Treatments applied to the home composting systems during the laboratory experiment.

Treatment	**Home composting system**				
	Wood bin	**Plastic bin**	**Rotary drum**	**Ground pile**	**Laboratory reactor**
Batch loading					
With BA and mixed	X		X		
With BA and not mixed	X				
No BA but mixed	X	X	X	X	X*
No BA and no mixing		X		X	
Weekly loading					
With BA and mixed					
No BA but mixed	X	X		X	
No BA and no mixing					

*aerated continuously rather than mixed.

Table 2: Characterization of plastic home composters tested during the field experiment

Composter	**Plastic home composter**				
	A	**B**	**C**	**D**	**E**
Type	Slatted	Top/bottom perforations	Slatted	Top/bottom perforations	Slatted
Location	Tree shaded	Tree shaded	Tree shaded	Open sky	Semi shaded
Total capacity (L)	350	300	350	300	350
Family size	1	3	3	2	6
Compost mixing	No	No	No	No	Occasionally
Average OW input (kg/week)*	2.4	8.1	8.0	4.0	1.6

*as measured during the field monitoring program.

In summary, management practices had an effect on temperature regime. Batch feeding the HC produced thermophilic temperatures generally exceeding 55 ºC for all HC, while weekly feeding produced at best mesophilic temperatures. Adding BA to the compost mixture both retarded and reduced the duration of thermophilic temperatures, because WC dropped the pH from 6.1-7.7 to 5.7 and the moisture content from 78 to 58 %, while lowering the formula biodegradability. Furthermore, adding BA increased the FAS, which enhance aeration and heat removal. Mixing the HC compost had a variable effect, being positive for W, practically neutral for GP and positive for P. This effect reflected the capacity of the HC type in generating convective aeration forces, where W with perforations distributed over its height, lacked convective aeration, GP relied on both diffusive and convective aeration and P generated good convective aeration with perforations located at its top and bottom.

The bulking agent (BA) was the main element influencing the evolution and final compost characteristics. In general, adding BA had a significant impact ($p<0.01$) on DM, with the mixture starting at 40% and finishing at 80%, as opposed to treatments without BA starting at 20% and finishing at 65 to 80%. Mixing had no impact on final compost DM, whether BA was used or not. Finally, weekly feeding produced a lower DM of 65 % after 150 days compared to batch feeding at 80 %. In terms of TC, OM and COD for all composts, the management practice combinations had no significant effect ($p>0.05$). The addition of BA produced lower pH compost, through the experimental period, whereas weekly feeding produced a dip in compost pH between days 15 to 60, indicating poor decomposition. Among HC, only the RD produced a significantly wetter final product, because of the clogging of its aeration ports during the course of the experiment.

Parasite and pathogen levels for W with BA showed *E.* coli levels exceeding the French Standard of 1000 CFU/g, because of poor microbial activity during the maturation phase, especially for the unmixed treatment.

Besides the W compost, thermophilic temperatures had no impact on *E.* coli counts, with weekly fed treatments showing acceptable levels under 1000 CFU/g while remaining in the mesophilic temperature range. *Salmonella* and *helminthe* eggs were absent from all compost likely because the original organic waste was clean.

Not impacted by thermophilic temperature regimes, *Streptococcus* faecalis counts were especially high for the RD1 and RD2 (batch fed, mixed and without/with BA) and W3 (with BA, batch fed and not mixed), once more because of poorer microbial activity during the first 20 days of composting. Analyzed for W only because all HC were fed with the same sources of OW, the PAH and trace elements respected French and North American standards because of the clean and well sorted organic waste used to make the compost mixtures.

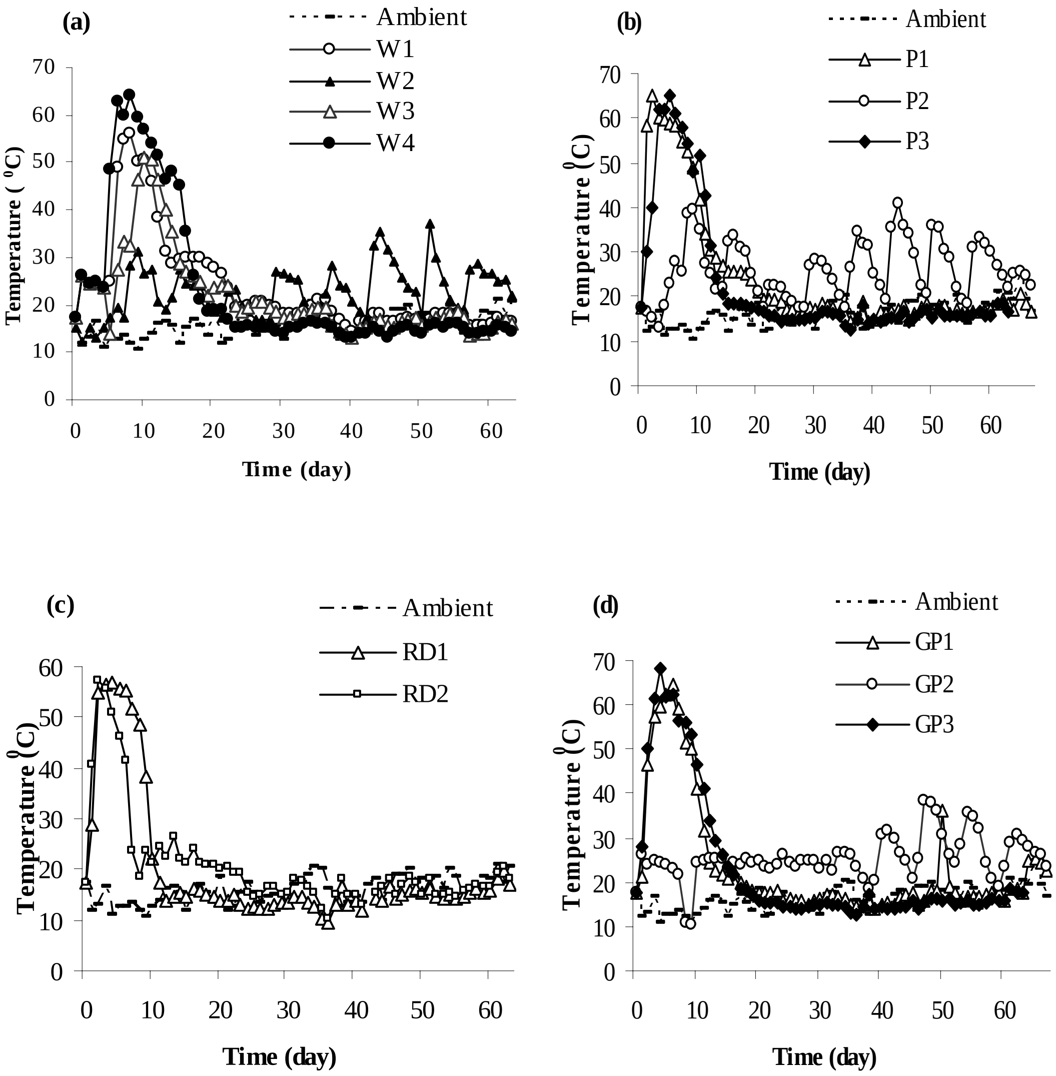

Figure 1: Temperature regime obtained with the laboratory home composting systems under different management practices. W1 – Wood bin with BA, batch fed and mixed; W2 – Wood bin without BA, weekly fed and mixed; W3 – Wood bin with BA, batch fed and not mixed; W4 – Wood bin without BA, batch fed and mixed; P1 – Plastic bin without BA, batch fed and not mixed; P2 – Plastic bin without BA, weekly fed and mixed; P3- Plastic bin without BA, batch fed and mixed; RD1 – Rotary Drum without BA, batch fed and mixed; RD2 – Rotary Drum with BA, batch fed and mixed; GP1 – Ground Pile without BA, batch fed and not mixed; GP2 – Ground Pile without BA, weekly fed and mixed; GP3 – Ground Pile without BA, batch fed and mixed; BA – bulking agent

3.2 Field experiment

For the filed monitored HC, most were loaded with 75 % FW and 25 % YT including grass clippings, garden residues and tree leaves. The food waste consisted mainly of fruit and vegetable residues with no meat and fish, except for a large amount of seafood fed to for home composter A in June. Also in July, home composter A received a substantial amount of ashes while home composter C regularly received flower stems and soil. Home composter D received generally a lower fraction of FW at 63 % and a higher fraction of YT at 37 %. The fed mixture offered an average DM of 12.5 to 15.9 %, except for home composter D fed a lower FW:YT ratio resulting in a higher DM of 41 %. In general, the C/N ratio of the fed OW ranged between 20 and 22, except for that of home composters A and D in the lower range of 13 to 14.5, resulting from their fed OW containing no tree leaves.

Tree leaves generally offer a C:N ratio in the range of 100-150. For all HC, the fed OW offered a COD level between 990 to 1155 mg/kg dm. All five HC were regularly loaded with OW but the amount varied among homeowners over the experimental period, depending on the number of persons in the household and their eating as well as gardening habits. Home composters B and C received the most OW at an average rate of 8.1 and 8.0 kg/week, as compared to home composters A, D and E which received OW at an average rate of 2.4, 4.0 and 1.6 kg/week (Figure 2 and Table 2), respectively. Home composters B and C received especially high amounts of OW during the first 12 weeks of experimentation.

Overall, home composter E was loaded with the lowest amount of OW at 1.6kg/week aty 15.9% DM, compared to all other HC but exhibited an average temperature above that of home composter A, B and C. The higher temperature profile despite a lower loading rate is explained by a monthly mixing and a partial exposure to sunshine. Home composter D exhibited the highest temperature profile ranging and averaging respectively above ambient, between 1 to 36 °C and 8 °C for an average OW loading rate of 4.0 kg/week. Peak temperatures were especially observed when the HC received large amounts of OW. The high temperatures observed for home composter D are associated with: its OW consisting of a FW:YT ratio of 2:1 but without leaves, thus offering a high biodegradability and dry matter; a low C:N ratio because of the absence of leaves; its location in full sunshine, and; the bottom/top position of its perforations enhancing convective aeration. The content of home composter A produced the lowest temperatures, ranging and averaging respectively above ambient, between 0 to 12 °C and 3 °C, despite receiving 2.4 kg/week at 12.5 % DM, because of the absence of tree leaves. The compost temperature profile of home composter B was generally higher than that of home composter C, although both their ranges were intermediate as opposed to that of home composters A and D. Whereas the temperature profile of home composter B ranged and averaged respectively above ambient, between 1 and 10°C and 6°C above ambient, that of home composter C ranged and averaged between 2 and 8°C and 5°C, respectively. Their loading rate was the highest at 8.1 and 8.0 kg of OW/week. Home composter B had bottom/top perforations as opposed to home composter C with opening created by slats. Furthermore, home composter B received a higher level of FW than home composter C, where FW has a higher biodegradability, as compared to YT and leaves. Both located in a shady backyard area, home composters B and C respectively received OW at a low DM of 12.5 and 14.5 % with a FW:YT ratio of 6.6:1 and 3.4:1. Nevertheless and despite their heavy OW loading rate, the intermediate temperature regime of both home composters B and C resulted from the low DM content of the fed OW and their shaded location. The auto-regression analysis of the temperature regime as compared to the loading rate also concluded that a large single input of OW exceeding 10 kg, resulted in a temperature increase, as long as the OW offered a DM of at least 15.9 %.

In terms of final compost quality, HC management had little influenced. Rather, the addition of ashes and soil tended to increase the level of heavy metals, without nevertheless exceeding Canadian and European regulations. The levels of pathogens found in the mixed compost of all HC produced variable Escherichia coli count, which nevertheless met Canadian and European regulations. Escherichia coli did not reflect the HC temperature regime likely because the top layer, never reaching thermophilic temperatures, contaminates the bottom layers. Less than 1000 Colony Forming Units (CFU)/g dm for Fecal Streptococci counts were observed for all HC. Even if YT can introduce Streptococcus faecalis from dog, bird and rodent droppings, Salmonella was not detected in any of the composts because of their absence in the original materials.

4. CONCLUSIONS

The objective of the project consisted in providing more information on the compost quality impact of various home composter management practices. The laboratory results were validated in the filed by monitoring 5 home composter normally operated by homeowners. The home composters tested along with the management practices were: the Plastic (P) and Wood (W) bins, the Rotary Drum (RD) and the Ground Pile (GP). In the laboratory, the HC performance was compared to that of a laboratory reactor (LR).

From the laboratory experiment, the following conclusions can be made:

- Top and bottom perforations in home composting bins enhance the aeration of the organic waste under treatment and tend to produce higher temperatures; such HC configuration eliminates the need for weekly compost mixing;
- Thermophilic temperatures are more easily achieved when the HC is batch fed, rather than weekly fed;

- Temperature regime and aeration rate seems to have limited impact on final compost characteristics unless the aeration ports of the HC become clogged during the process;
- Parasite and pathogen levels are generally low when clean waste is fed, but thermophilic temperatures have little impact on organism counts especially when weekly feeding, because of the contamination effect of freshly added waste;
- Adding a bulking agent such as wood chips is not necessary, when the mixture fed to the HC consists of equal wet volumes of FW and YT.

The field experiment of this project validated the observations made in the laboratory, despite their achievement in a different country. The field experiment provided the following conclusions:

- Home composters with bottom and top perforations enhance the convective aeration;
- A semi-shaded location, monthly mixing and weekly feeding of more than over 10 kg wet OW with equal volumes of FW and YT at a dry matter of at least 15 %, for 300-350L bins, maximizes the occurrence of thermophilic temperatures.

Over all, the best management practice for home composting is to use of 2 smaller bins (150-200L) with top and bottom perforations, filled in sequence. This can help eliminate fresh OW contamination and increase the relative mass of fed OW to reach higher temperature regimes.

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(131) IMPACT ASSESSMENT OF HOME-COMPOSTING PRACTICES ON THE QUANTITY OF COLLECTED WASTE

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EXECUTIVE SUMMARY

Society is reconsidering composting, this ancient technology, for the treatment and recycling of municipal organic wastes. Municipalities are now encouraging the use of home composting when very little is known about the environmental impact of this practice as compared to other more conventional means. Furthermore, there is a need to establish a standard method to evaluate the impact of such a practice, when the diversified tools presently used produce data which cannot be compared. Within the European LIFE project *Miniwaste*, a study was conducted to establish a standard method to evaluate the mass and composition of the treated biowaste. The experimented method used a combination of well recognized tools: conducting household surveys; getting households to measure the mass of biowaste (BW) fed into the composters, and; measuring household waste production and its residual organic fraction (ROF). The study of the results obtained thanks to these different tools enabled to propose a simplified protocol that will then be used by other municipalities to assess the impact of home composting in their own communities and compare their results with other European regions.

The study was conducted in the sector of Cesson Sévigné, a suburb of the City of Rennes, France. Within the 1000 households contacted, all living in individual homes, 300 were surveyed to characterize their organic waste (OW) management practices. People of 60 years and over were found to be most likely to practice composting. The telephone survey was also used as a simple tool to recruit households to further the study and monitor the mass and type of biowaste actually composted. Accordingly, 38 households volunteered (VH) to weight their fed biowaste during one year: the OW originated either from their kitchens or their yards.

4 parallel campains of household waste characterization (one per season) were conducted. The amounts of organic waste still present in the bins are recorded according to the bio-waste management reported by households. Groups were established to separate households that compost and weigh their composted waste (VH) from, those who compost but do not weigh their waste (NVH) and those who do not compost.

The statistical analysis of the results determined differences due to the seasons, to the fact of being weighing volunteers or to practice or not home-composting.

It appears that each individual assessment tool does not provide a complete response to the evaluation. Therefore the coupling of several tools is recommended. Finally, the method proposes to implement a survey and measure the output per capita for households 'composters' and 'non-composters.

1 INTRODUCTION

In September 2005, the Ministry of Ecology and Sustainable Development of France introduced a policy to reduce the mass of municipal solid wastes through recycling also to be applied to the organic fraction. Some municipalities have anticipated the creation of selective collection practices to recycle packaging, newspaper-magazines-fliers, as a first phase, and then as a second phase, the organic fraction of the solid waste stream. This program resulted in the wide use of home composting bins, within city suburbs and village. Between 2000 and 2007, the scientific division of the Ministry of Ecology and Sustainable Development of France, called ADEME, supported home composting through a large promotional campaign resulting in the installation of more than 900 000 units. As in other countries of Europe, 34% of the population in France is composting its organic waste to reduce the fraction of municipal solid waste (MSW) being landfilled (Indigo LH2, June 2008). In comparison, 25% of the Luxembourg population is practicing composting in 2001 (Administration of the Environment of Luxembourg, 2001), as opposed to 10% in Ireland in 2004 (EPA, 2009), and 35% in England in 2005 (DEFRA, 2007).

Representing about 30% of the municipal solid waste stream, kitchen and yard wastes can be recycled through home composting bins (ADEME, 2009). Several studies (Jasmin and Smith, 2003; ECCOVAL, 2012) report the annual composting of about 40 kg of kitchen waste and 80 to 100 kg of yard wastes. Nevertheless, it is difficult to correctly estimate the mass of composted biowaste (Rabeau, 2008), because the promotion of composting impacts production and quality of the recyclable collection (Mitaftsi and Smith, 2006; Resse and Langlois, 2008). Therefore, evaluating the complete diversion effect also requires the monitoring of the final compost usage (Read, Gregory and Philips, 2009), which can be accomplished through surveys among practitioners (Burnley, 2006).

Municipalities are increasingly interested in the true recycling of biowaste through home composting, considering that such practices aim at reducing waste collection and treatment, while still respecting health regulations. Several municipalities have monitored the composting activities with their region, but the mass balance complied was inconclusive or incomplete (Rabeau 2008, Resse and Langlois 2008). Furthermore, results cannot be compared because of the lack of standard evaluation method. To fill this gap and within the *Européen Life+ Miniwaste* project, the objective of the present study was to produce a standard evaluation method.

To finalize this protocol, an experimental phase was conducted within sectors of the city of Rennes, France, supporting the project, to evaluate two basic fluxes, kitchen and yard wastes, through two recycling method, in ground piles and in home composting bins. The project made use of the tools specified by ADEME, in its 2003 program, 'Selective collection and treatment of household organic wastes – performance indicators for quality'. Therefore, three tools were applied on one test sector. The final objective was to compare the field testing results and propose an evaluation method for the domestic organic waste stream.

2 METHODOLOGY

The method consisted in conducting a survey of households to evaluate the quantity of biowaste composted and to compare this quantity to that of the residual organic fraction (ROF) garbaged by the community. The evaluation method is based on the results obtained by these tools and applied to a sector of the City of Rennes.

2.1 Household survey

Some 1000 households were surveyed by telephone, within a community of 4303 households living within individual homes. This survey characterized the families, their living conditions and their recycling practices (example: composting or garbaging) for kitchen wastes (fruit and vegetable peels, coffee grindings and tea bags, table scraps, meat, fish) and yard trimmings (grass, leaves, weeds). The 299 completed surveys were classified based on their typology, considering their practice of composting or garbaging of OW. Four main categories were identified (Table 1):

- Households that recycled organic waste using a ground pile (Type 1- Pile).
- Households that recycled all organic waste using a composting bin (Type 2-CB+), (CB for composting bin).
- Households that recycled only fruit and vegetable peels using a composting bin (Type 3-CB-).
- Households that garbaged of organic waste (Type 4-NC) (NC for no composting).

During the telephone survey, household correspondents were invited to take part in a preliminary study on composting. These households were classified as « volunteer households » or VH.

Table 1: **Telephone survey household characterization**

Number of Households	Type 1 « Pile »	Type 2 «CB+ »	Type 3 « CB-»	Type 4 «NC»
Not volunteering (NVH)	35	26	43	161
Volunteering to weight their biowaste composted (VH)	9	13	16	

2.2 Quantity of composted biowaste

The 38 VH were equipped with a fish scale, a small pail to weight kitchen wastes, a large bag to weight yard trimmings and a chart to record monthly composted organic waste . Monitored during one year, all VH weight the kitchen waste and yard trimmings recycled through composting, and compiled results were regrouped under the three categories of Pile, CB+ and CB- (Table 2), in terms of total mass or mass per person.

Table 2: **Characteristics of the volunteer households conducting home composting**

Households (VH)	Type 1 « Pile »	Type 2 «CB+ »	Type 3 « CB-»
Number of households	9	13	16
Persons per household	3,1	3,5	3,1
Average Age	51	50	52
Average garden area (m^2)	353	324	489

2.3 Garbaged organic waste

This monitoring operation required the establishment of 7 groups of households. Since VH often exhibit better behaviour, their results were validated by measuring the garbaged ROF of NVH and VH households and comparing this against that of households not conducting composting. Overall, 7 groups of households were compared, as described in Table 3.

Table 3: **The 7 experimental household groups**

Households		Type 1 « Pile »	Type 2 « CB+ »	Type 3 « CB-»	Type 4 «NC»
NVH	Number of households	15	15	15	15
	Number of persons	45	42	46	43
VH	Number of households	9	13	16	
	Number of persons	28	45	49	

Garbage production and ROF content was measured for each season (June, October, January and April), for each 7 groups, using the standard procedure used in France (XP X 30-408). MSW composition was established using 12 categories (ROF, cardboard, complexes, textiles, sanitary textiles, plastics, combustibles, glass, metals, incombustibles, special wastes). The ROF category was broken down in kitchen and yard waste. The other sub-categories analyze the quantity in percentage of recyclable wastes (newspaper-magazines-fliers, packaging, and cardboard-ELA-plastic-metal-glass). Within the community monitored, the garbage containers were equipped with scales automatically measuring the mass of MSW produced per household, to compare MSW among the 7 groups of household, and based on the number of person per household.

3 RESULTS AND DISCUSSION

3.1 Composting practices of households living in individual homes

The survey indicated that 48 % of households were conducting composting, which is under the average reported for the City of Rennes, France, of 57 % or for the France, of 55 % (households living in individual houses), established by the LH2 Survey group in its report « National survey on the management of domestic organic waste » financed by ADEME within its National Plan to Support Domestic Composting. Also, these households were found to differ in the way they conducted composting, as presented in Table 4.

Generally, households using pile composting (Pile) were less inclined to recycle kitchen waste than those using composting bins (CB+), as also confirmed by the group LH2 finding that from 42 to 46 % of households used kitchen waste when composting in piles as compared to 57 to 70 % when composting in bins. Also, CB+ households compost more organic wastes than CB- households. As for yard trimmings, differences were not so obvious. The LH2 study reported a higher percentage of households composting grass with the compost pile, (56 %) as compared to the bin (44 %). In the present study, the percentage of households composting grass with the compost pile reached 60 % as compared to the bin at 76 %.

Accordingly, the telephone survey proved to be an effective (economical and fast) mean of obtaining information on composting practices from a large number of households. Composting was found to be well accepted by household with owners of over 35 years of age. Furthermore, the telephone survey verified other statistics such as the number of persons per household, but was not able to estimate the mass of organic waste composted.

Table 4: **Percentage of households composting their organic waste**

	Type 1 « Pile » %	Type 2 «CB+ » %	Type 3 « CB-» %
Composted kitchen wastes			
Fruit and vegetable peels	80	100	100
Food wastes	30	100	0
Coffee grindings, tea bags	80	93	82
Egg shells	80	79	82
Orange, citrus fruits	60	79	59
Meal /Fish	0	36	0
Bread	30	36	18
Seafood, shellfish	10	29	12
Yard trimmings			
Grass	60	86	76
Tree leaves	60	36	53
Cutting	60	57	24
Faded Plants/flowers	80	86	71
Weeds	60	50	35
Vegetable garden wastes	30	21	47

3.2 Composted organic waste as reported by volunteer households

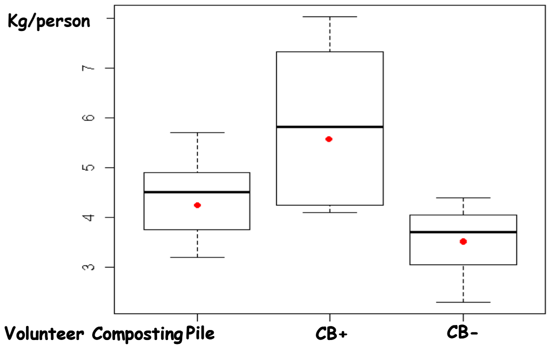
The one year study produced the following average yearly mass of composted organic waste per household: 253 kg for « Pile », 306 kg for « CB + » and 278 kg for « CB- ». Table 5 compares these quantities per person.

Table 5: **Mass balance of organic waste composted by season**

	Type 1 « Pile »		Type 2 « CB+ »		Type 3 « CB- »	
	Mass kg/person	Kitchen wastes %	Mass kg/person	Kitchen wastes %	Mass kg/person	Kitchen wastes %
Summer (June, July, Aug.)	19,5	57	29,2	55	16,2	61
Autumn (Sept., Oct., Nov.)	20,1	71	26,8	79	42,0	30
Winter (Dec., Jan., Feb.)	19,4	75	23,2	79	16,9	66
Spring (March, April, Mai)	24,6	50	19,0	74	22,3	39
Total	**83,6**	**62**	**98,2**	**71**	**97,7**	**43**

The statistical analysis of the date reveals that households « Pile » and « CB+ » conduct composting regularly throughout the year. But, in November one household « CB- » composted much more yard trimmings than other months. Over half of the households loaded their composting bin or group pile with 75 % kitchen waste and 25 % yard trimmings. Each household exhibited different management practices. The amount of biowaste composted was not proportional to the number of persons per household. Furthermore, fruit and vegetable peels, coffee grindings and tea bags and egg shells are the most frequently composted wastes. Yard trimmings are not so frequently composted in the bin as compared to the ground pile.
Figure 1 illustrates the mass of kitchen waste composted by households: the mass for group CB+ is most important, as compared to only 40 kg for group CB- composting only fruit and vegetable peels. When using a ground pile as opposed to composting bins, households tended to compost as much biowaste. Biowaste composting of 40 to 70 kg/person/year corresponded to the values reported by other studies, such as that of the ADEME report on the «Evaluation of domestic composting policies » by RDC Environment (2004), and « Local communities and garbage reduction » also produced by ADEME.

Figure 1: **Mass of kitchen waste composted monthly**



Grouping the households into 3 groups seems appropriate considering that each group composts a different mass of biowaste according to the practices declared in the survey. Also, results of MSW composition show that the garbaged ROF was lower for those VH as compared to the other households (NVH and NC).

3.3 Residual organic fraction found in the collected garbage

For each season and in terms of kg/person/year, the mass of MSW is presented in Table 8. Despite a wide range of values from 47 to 156 kg/person/year, resulting from the limited number of households studied, some interesting results can be observed:

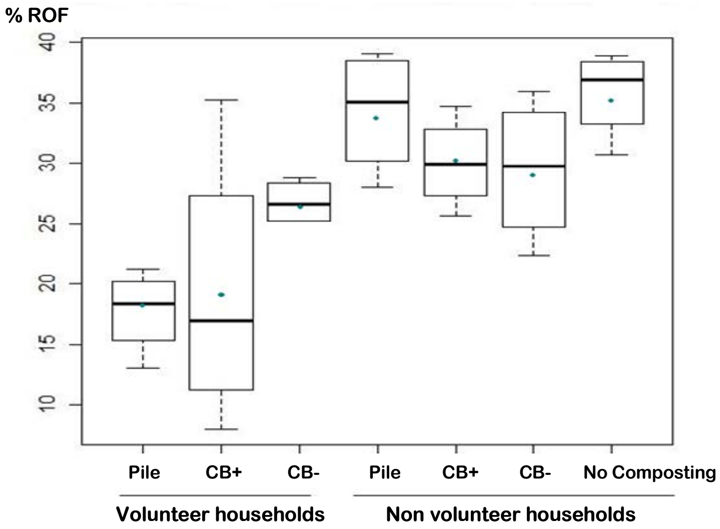
- Volunteer households (VH) composted more biowaste, and garbaged less residual organic fraction (ROF);
- There is a much higher amount of MSW when composting is not practiced;
- The production of MSW does not vary with seasons with the 7 groups of households.

Table 8: **Mass of MSW collected by the community (kg/person/year)**

Household			June	October	January	March
Volunteer (VH)	Composting	1-Pile	99	91	108	120
		2- CB+	82	65	63	57
		3- CB-	47	91	77	90
Non volunteer (NVH)		4- Pile	107	117	139	92
		5- CB+	119	123	110	99
		6-CB –	61	67	71	75
	Non composting	7- NC	148	138	154	156

Figure 2 presents the mass of garbaged ROF for each group, and each four seasons. The percentage of ROF is presented in terms of % wet mass.

Figure 2: **Percentage of residual organic fraction (ROF) in the garbage of households**.



For the VH (volunteer households), there are differences among groups :

- The percentage of organic waste found in the garbage is significantly lower for:
    - The « Pile» volunteer group versus the other households using a ground pile for composting;
    - The « CB+» volunteers versus the households using piles but not in the volunteer group,
    - The « Pile» and « CB+» volunteers versus those not practicing composting.
- The percentage of ROF does not vary significantly from once season to another within groups of households.

The garbage ROF made up of 85 % food waste because Rennes Métropole has a strong policy against accepting yard trimmings within its garbage. Table 9 illustrates that the garbage of VH had 8 to 22 kg of food wastes /person/year as compared to the other composting households (NVH) with 15 to 37, and the other non composting households with 40 kg /person/year. Furthermore, fruit and vegetable peels were found in the garbage of all households, contrary to the telephone survey.

Table 9: **Mass of food waste found in household garbage (kg/person/year)**

Household			June	October	January	March	Average
Volunteer (VH)	Composting	1-Pile	7,8	15,5	12,7	18,8	**13,7**
		2-CB+	9,2	11,6	21,5	4,5	**11,7**
		3-CB-	10,2	18,4	16,5	21,1	**16,5**
Non volunteer (NVH)		4-Pile	35,8	30,1	36,6	32,4	**33,7**
		5-CB+	28,1	31,1	30,3	26,4	**29,0**
		6-CB –	17,7	18,0	22,4	15,2	**18,3**
	Non composting	7-NC	42,8	45,8	37,4	38,9	**41,2**

Garbage composition and mass both show that food waste is more limited when composting is practiced,. If the MSW composition analysis has provided relevant information for this study , it is not necessary to conduct it systematically because the composition was not significantly different for different types of households. By cons, the different amounts measured between households 'composters' and 'non-composters' justify to accurately measure the quantities of MSW for each type of households.

5 CONCLUSIONS

Conducted among 300 households, the present project used three different tools to analyze the impact of home composting on the organic fraction of garbage. The method allowed for the sub grouping of households into three categories, based on their compost management practices: composting households which volunteered to weight their composted biowaste (VH); composting households which did not volunteered to weigh their composted biowaste (NVH), and non composting households (NC). Surveying the households by telephone initially, brings additional information pertaining to the number of person within the household. Nevertheless, this initial survey does not provide information as to the mass of biowaste composted.

During the telephone survey, volunteer households (VH) were found and these participated by weighing the biowaste composted. The biowaste weighing process demonstrated that both households using ground piles and composting bins recycled about the same amount of food waste. In the project, the experiment demonstrated that weighing and determining the fraction of garbaged residual organic fraction (ROF) produced the same results as weighing the biowaste fed to the composting systems. Therefore, this biowaste weighing activity is not necessary when conducting an evaluation if the garbage is well monitored. Furthermore, when asking households to weight their composted biowaste, the volunteers are generally more adept composters than those who do not volunteer. Finally, the mass of biowaste produced did not vary with seasons. In conclusion, a standard method for the determining of the impact of composting on biowaste recycling requires basically the periodic weighing of the garbage produced. Only 2 groups can be compared, those composting and those garbaging their biowaste.

The mass balance achieved through this *Miniwaste* project, compared to other studies, such as that conducted within the Greater City of Nancy, France, validates the standard method developed to evaluate the impact of composting. This method relies on both measures and averages. The measures are conducted on the garbage produced by both composting and garbaging households. The averages pertain to the amount of kitchen waste composted based on the type of home composting system used and the percentage of residual organics in the garbage (ROF). This simple and economical standard method will be further validated by the other partners of the *Miniwaste* project.

6 ACKNOWLEDGEMENTS

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(196) CITY TO SOIL : A COST EFECTIVE SYSTEM FOR COLLECTING AND COMPOSTING DOMESTIC ORGANICS

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EXECUTIVE SUMMARY

The diversion of organic waste to landfill is an increasingly important environmental and planning issue for municipal managers around the world. The impetus to implement such processes in local populations varies from country to country.

Directly involving the community in the recycling of organic material has proven to be very successful in achieving clean input material for composting into high-quality products and for their return to agriculture.

The basis of this paper is a New South Wales Government funded project called Groundswell, which used the City to Soil compost process and a new and innovative compost process using very little machinery.

In a number of Australian urban and rural communities, City to Soil, an integrated system of community engagement, waste management, diversion and application has been implemented that directly addresses the issue with a broad range of associated environmental, economic and social benefits.

Community engagement in this program provides a direct connection for the householder between their organic waste outputs in the city and the soil that produces their food.

'The City to Soil system provides one essential contribution– it potentially engages everyone to re-route their everyday kitchen and garden waste back into the food production system. At the same time it educates people about the value of nutrients in the soil. It supports farmers. It supports the environment. It gives people a sense of something that THEY can do.' (The Magic Pudding – Pamphlion/Chavalier 2010)

In terms of industry understanding, waste management and recycling have the same origins in that all discarded materials are seen as a very big problem, requiring very large and complex solutions. In this paper it is argued that City to Soil is very different in that it sees clean, source-separated organic waste as an opportunity and an asset that can be developed into a high-quality compost product for use in local soils.

This in turn can generate very large savings to both the local council and the end user of the compost in terms of reduced fertiliser application, increased yield, increased organic matter and soil carbon, increased water efficiency, increased land value and other benefits.

In this program where the participants are being asked to 'become part of the solution' this collection and compost process is as its very heart about food quality and food security through the protection of soils.

In the implementation of this project, the limited budget and the distances between sites meant that a conventional shred and turn compost process would not be cost effective. As a result, a new compost process was utilised where shredding and turning is not required, in conjunction with using a two-part inoculant. This resulted in a low-cost process, with little mechanical input and application in even the most remote community.

The City to Soil collection system and Groundswell Compost process are now used in councils beyond the trial area with the compost process being very successfully applied in numerous locations both in Australia and overseas including Newport in Wales, UK and Noumea, New Caledonia.

In all instances the collected organic materials have contained contamination levels of less than half of one percent, indicating that the Community Engagement Strategy was exceptionally successful. In addition all compost products produced have complied with all the necessary standards and regulations and have consistently produced a compost which received and 'unrestricted use' classification.

1. THE CITY TO SOIL COLLECTION PROCESS

To ensure the success of the collection system in providing a clean source-separated product, a community engagement strategy was designed to give people the right tools, information and motivation.

It was necessary to first be able to provide the right tools to enable people to universally do what was wanted. The collection system needed to be odour free and easy to manage at a kitchen level.

The most appropriate tools found were the Bio Bag compostable liner bags and the Max Air vented bin. These allow people to use similar practices in their kitchen to what they were previously doing and at the same time eliminate odours in both the kitchen and the wheeled bin or kart.

In the Groundswell project, by registering the number printed onto any roll of bags against the numbered street address the project staff were able to give away prizes for no contamination in randomly selected bags which were picked out at the compost site.

The successful owners of the prize-winning bags were given a $100 hamper of fresh fruit and vegetables, reinforcing the concept that City to Soil is about food and soil.

The information about the collection was provided in such a way as to overcome literacy and language issues. It was provided in graphic form in a simple two-colour brochure and was communicated using existing council information media.

The simple but powerful message was to “Become Part of the Solution” – ‘if you put your food and garden waste into this bin, we will compost it and get it back into agriculture’. The message was supported by media use of stories of farmers using the product.

This project has demonstrated that government and the waste industry not only consistently underestimate the community, but they provide systems that are built to fail and do so regularly.

Universal participation is achievable if your system contains the right motivators for source separation. Using existing government research we found that there were six reasons why people might participate and individuals may respond to only one or two of these but others may respond to more.

The six motivators are:

- Help address Climate Change
- Reduce waste to landfill
- Reduce waste costs
- Improve agricultural soils
- Support local farmers
- Win prizes

By consistently including all six in message the Engagement Program was able to reach the entire community. The prizes awarded for zero contamination selected on delivery of the mixed material to the compost site, generated positive messages and community conversation about the program and the fact that it was about food production and not waste management.

1.1. The Groundswell Compost Process

When the Groundswell Project commenced it was quickly realised that with the combination of up to a five-hour drive between sites and a limited budget, it would be impossible to use the standard compost process which involved shredding and then turning on a weekly basis.

The cost of moving heavy machinery from one site to another would have quickly consumed the entire available budget.

An innovative option was chosen and trialled which involved no shredding, the use of a two-part inoculant, a cover and minimal turning.

In the collection of the majority of domestic organic waste, most material is no longer than your arm and no thicker than your thumb. With the addition of food to this mix, the right level of moisture and a compost accelerant it has been possible to design a process without the use of heavy machinery.

The products chosen for the Groundswell compost process were the Photon range from VRM in Townsville, Australia.

Compost processes are generally perceived to be either aerobic or anaerobic. The Groundswell process produces no odour despite the fact that it is a two-phase static pile system. Regardless of the mixtures used in this process on numerous sites it has never caused any odour issues.

The Groundswell compost process uses microbiology which are both aerobic and anaerobic or both. The VRM range of products includes Effective Microorganisms (EM) that is used around the world to treat sewage, waste water, stagnant ponds, waste and agricultural soils.

However, the Photon range builds substantially on EM and includes purple non-sulphur bacteria and other organisms.

Once the material has been collected from households, it is brought to a compost site where it is laid out on the ground and the small amount of contaminant is manually removed.

The combination of yard waste and kitchen waste is then wet down with non-chlorinated or rested water to a moisture level of between 40% and 80% and the inoculant added at the rate of 1 litre of each part per 10 cubic metres of organic materials.

The material is then formed into a windrow with a dip in the centre, covered with a tarpaulin or silage wrap and left to sit for six weeks.

The covers are then removed and the pile is checked for contamination. Once any contamination missed in the first stage is removed, the covers are replaced for an additional 6 weeks.

When the process is used on a farm it is even easier because there is no contamination to remove and the entire process can be completed with standard on-farm equipment.

The process documents can be obtained from the Groundswell blogspot at: www.groundswellproject.blogspot.com

After eight to 12 weeks, depending on the particle size of the original feedstock, the temperatures should drop slowly and the pH should return to neutral. Piles can be left covered to mature and to prevent drying out – moisture levels should be kept at around 40%.

The material after this period can be put through a rotating drum trommel or a large industrial sieve. A product left to mature can reach a Cation Exchange Capacity of as high as 73.

2. DISCUSSION

As long as humanity exists, given varied social circumstance, it will produce a relatively consistent stream, per person, of organic wastes daily. The average individual will produce organic wastes every day in the preparation and consumption of food.

An urban population and its outputs are an organic function of life. To exist, certain levels of input are required. These inputs necessitate outputs, which in turn become part of the resource input for agricultural sustainability.

It should be kept in mind that household waste is only one small part of the potential organics waste stream. There is also substantial opportunity in commercial and agricultural sources.

The City to Soil model collects a high-quality, source-separated product with the specific objective of delivering the product back to agriculture.

In so doing it recreates the urban/rural link, and the structure of this collection process rewards the urban community for supporting the rural community.

The system can be used to link the collector, the composter and the farmer in a cooperative model.

Tenders could be designed by government, if need be, so that the Collector/Processor/ Farmer are a single contractual identity in terms of the household or commercial collection. In this instance, as is the current practice, the material collection and part of the processing would be paid for by the person disposing of the material.

The true ‘full value’ of the product in terms of yield, soil structure, water efficiency and carbon is only realised when the product is applied to the land. This is where the real values lies – in the ‘third’ affect.

Under the existing system, many city based compost firms are paid a price per tonne to take the raw product – while they do make a profit at this level, their true capital gain is only realised when they sell the product. Often they make their product with scant regard for the end market.

Little beyond the point of processing has ever occurred to them, other than charging farmers a price for the end product. When you add to this the cost of transport combined with the cost of spreading on the land the purchase price can be prohibitive.

Few compost manufacturers have truly recognised the real value of application to the land and made their products accordingly

Indeed farmers who do use these products are not set up so as to gain and/or register the real value because they see the product as simply another input.

2.1. The Benefits

Under the City to Soil collection system the ‘garbage dollar’ can be used to collect and transport the product to its point of optimum benefit and the resulting model is far cheaper than landfill.

Once collected and processed, the product is transported to the land where it is made into a range of solid or fluid products – modified to agronomic standards depending on soil and crop application.

Values accruing to the farmer include:

- Increased yield
- Improved crop quality
- Reduced water use
- Improved water efficiency
- Improved soil structure
- Increased microbial activity
- Reduced nutrient leakage
- Reduced fertilizer costs
- Reduced erosion
- Carbon sequestration
- Increased land value
- PR value (increasingly important in the Climate Change market)

The benefits accruing can be realised directly by the farming family. Research will identify new areas of value and benefit. Relationships with large corporations requiring carbon credits can be developed as the scheme progresses.

The end point of the City to Soil program and Groundswell compost process is that it enables the entire community to participate in real capacity building.

In the broad social picture it enables the establishment of a model that ensures links between the urban and rural community and links the economy back to the true producer of community wealth, the farmer.

The City to Soil engagement model, even with a different compost process, has the capacity to be used anywhere, building real soil quality, enriching communities and protecting the environment – simply by changing the way that organic waste is regarded.

3. CONCLUSION

I do appreciate that I am putting this paper before some of the more informed composters and soils scientists in the world and that I am asking you to consider that both the City to Soil collection system and the Groundswell compost process have proven to be dramatic innovations on what you have been doing, with enormous future potential, because they bring to the conversation on organics diversion something which I believe to be very new.

I do realise that the proposition I have put forward many of you will find challenging or even confronting. I ask you to consider that innovation and the exchange of ideas is not new to science – indeed it is the true heart of science.

When Candice Pert discovered receptors in the human brain for opiates some 40 years ago, the scientific ramifications were enormous because she proved that either the human body was designed to take opiates from external sources or that the human body was designed to make opiates internally.

Either way, in a social structure at the end of the drug-drenched 1960s and at the start of the legally constrained 1970s where opiates were seen to be habit forming, dangerous and associated with criminals, Dr Pert’s work was very challenging for both her colleagues and the public.

It came to be accepted for its truth.

In closing can I leave you with a quote from that same period by economist and President of the UK Soils Association, Ernst Friedrich "Fritz" Schumacher.

“The modern word tends to be sceptical about everything which demand’s mans higher faculties. But it is not at all sceptical about scepticism, which demands hardly anything.”

Communities around the world have lost sight of the relationship between healthy people, healthy food and healthy soil. Everywhere it has been used City to Soil has rebuilt this relationship.

Everything you are is everything you eat. All you are and ever will be depends on the quality of your food.

The soil is your mother – it warrants your care and protection.

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Session 2

(25) CARBON SEQUESTRATION MECHANISM BY USING CHARCOAL (BIOCHAR) AND COMPOST IN FARMLAND

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EXECUTIVE SUMMARY

For global warming prevention, sequestration mechanism of carbon in soil used with charcoal (biochar) was studied, and analysis method of charcoal carbon amount in the soil was established quantitatively. By using charcoal carbonized from biomass materials and food waste compost in the farmland, carbon sequestration for long period and increase of microorganisms in the soil are expected.

Food waste compost was made in composting factory. FIGURE 1 shows flow chart of preparing food waste compost with charcoal in the factory. Wood charcoal powder and compost made from food garbage were used to the red-clayey soil farm land located in the suburbs of Tokyo.

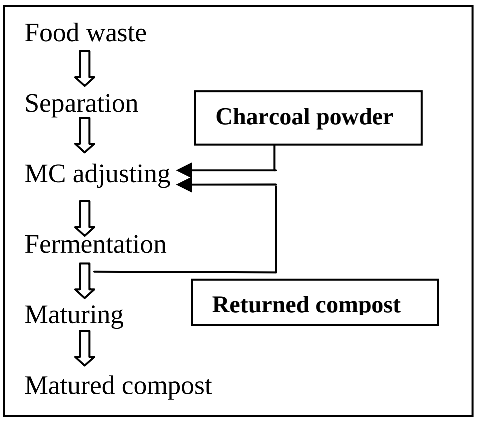


FIGURE 1. Flow chart of preparing food waste compost with charcoal in composting factory.

As aggregate of the soil can be developed with microorganisms in the soil, adenosine tri-phosphate (ATP) concentration in the soil was measured. The structure of the aggregate was estimated with the wet screen method. The mean weight diameter (MWD) of the soil was calculated with particle diameter (mm) and the integration number of the aggregate (%).

As shown in FIGURE 2, the ATP concentration increased in the soil used with the charcoal and the compost, and about the half value of which was observed in the soil with compost and without charcoal. There no change was observed in the soil without the addition.

The MWD value of the soil was about 30 before the test. FIGURE 3 shows that after ten days using the charcoal and the compost, the MWD value increases to 58. In the soil with the compost and without charcoal, the MWD increased to 48. It was found that the soil was aggregated with microorganisms in the soil.

The aggregation of the soil in farmland was developed by using the charcoal and the compost. It was suggested that the soil was aggregated with microorganisms proliferated on the surface of the aggregate in the soil.

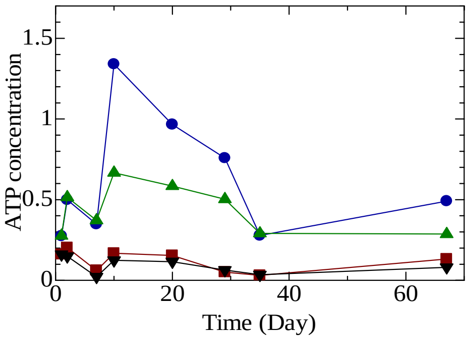


FIGURE 2. Time dependence of ATP concentration of the soil.

● : with charcoal and compost, ▲ : with compost, ▼ : with charcoal and ■ : no additives.

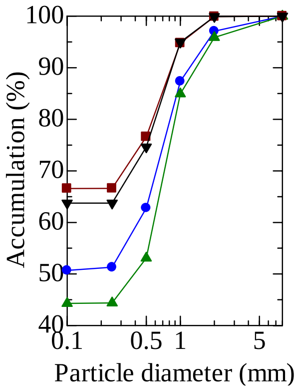


FIGURE 3. Particle diameter distribution of the soil.

● : with charcoal and compost, ▲ : with compost, ▼ : with charcoal and ■ : no additives.

1 INTRODUCTION

1.1 Background

By using charcoal (biochar) carbonized from biomass materials in the farmland, carbon sequestration for a long period and plant growth promotion due to soil property improvement are expected (Tanaka et al., 2011-a). The soil property improvement, such as water-holding ability and water- and air-permeability, partly comes from aggregation of the soil, because where there are a lot of micro voids and pores among the soil aggregates, which brings the soil softness and well-aerated environment. It is well known that the aggregates can be developed with microorganisms in the soil.

1.2 Research objectives

In this paper, in order to establish the sequestration mechanism of carbon in the soil used with charcoal in the farmland, analysis method of the undegradable carbon (UDC) amount based on carbon of the charcoal in the soil was developed for quantitative estimation of the carbon sequestration. The aggregation of the soil in the farm land where charcoal and/or compost were used is studied. The influence of the microorganisms concentration in the soil on the aggregation of the soil was also discussed.

2 EXPERIMENTAL

2.1 Charcoal preparation

Biochar was prepared from waste of wood pallet for transportation with folk lift as a raw material which was carbonized at 500^{o}C-600^{o}C in process of self-burning in the iron box furnace. Some characteristics of charcoal as follows; the fixed carbon amount of 75%, the specific surface area of 205 m^2/g, the bulk density of 0.2 g/ml, the pH of 8.1 and the grain size of under 3 mm.

2.2 Food waste compost

The compost was made in a factory in a suburban area of Tokyo dealing with 100 tons of food industry waste a day (Tanaka et al., 2011-b). FIGURE 1 shows flow chart of preparing food waste compost with charcoal in the factory. Undesired materials such as plastic, steel and aluminum materials are separated from food waste with a separator in FIGURE 2. Then, several percent of charcoal and returned compost are mixed to the food waste, and the mixture is thrown into the top of a fermentation tank (65 m^3) in FIGURE 3. The temperature of the mixture increases to 60-70^{o}C, because aerobic microorganisms proliferate largely on the surface of the charcoal. After one week, the first fermented compost is pulled out of the tank. Then, the compost is piled for two months with aeration, and finally the matured compost is obtained.

TABLE 1 shows the components of the matured compost with charcoal. N, P and K elements in the compost are well balanced, and C/N ratio of ca. 10 means that the compost is well matured.

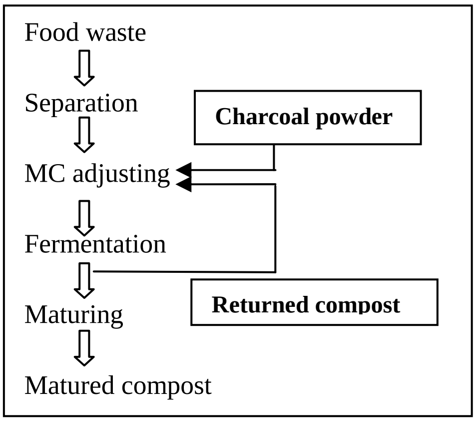


FIGURE 1. Flow chart of preparing food waste compost with charcoal in composting factory.

FIGURE 2. Two separators into food garbage from plastic bags.

FIGURE 3. Twelve fermentation tanks of 65 m^3.

TABLE 1. Component analysis of food compost with charcoal.

Analysis items	Sample A	Sample B
T-N （%)	3.2	3.8
NH_3-N(mg/100g)	151	102
NO_3-N （mg/100g)	46	35
P_2O_5 （%）	1.9	2.0
K_2O （%）	1.2	1.1
C/N ratio	10.2	9.9
MC(%)	20.5	16.4
charcoal (%)	10.1	14.7
pH	8.55	8.60

2.3 Farmland

The charcoal powder and the compost made from food garbage were used at the rate of 2 t/10 a and 1 t/10 a, respectively, to the red-clayey soil farmland located in the suburbs of Tokyo. TABLE 2 shows using condition of the compost ant the charcoal in the farmland.

TABLE 2. Using condition of compost and charcoal in farmland.

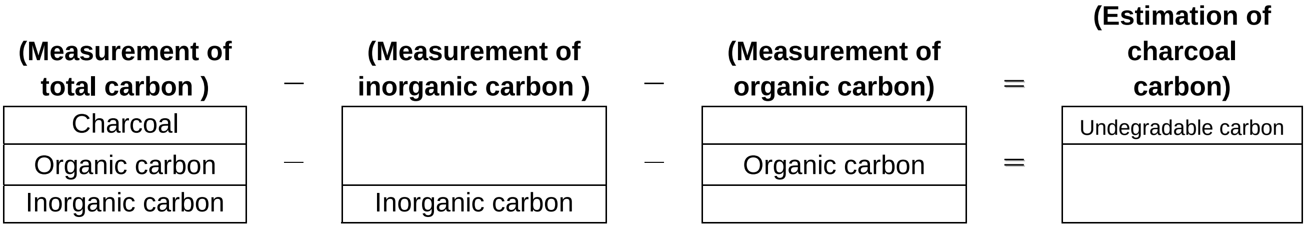
using condition	vegetable	charcoal (t/10 a)	compost (t/10 a)	Area (m^2)
charcoal +compost	vegetable	2	1	140
compost	vegetable	0	1	140
no additives	vegetable	0	0	140
charcoal	non	2	0	21

2.4 Microorganisms

The ATP concentration was measured with Luminometer UPD-4000 (Meidensha Corp.). When ATP, to which d-luciferin has been added, changes to adenosine monophosphate in the presence of luciferase and Mg^{2+}, light at a wavelength of 560 nm is emitted. Distilled water (20 ml) was added to 2 g of the sample and stirred with a tube mixer at 2500 rpm for 1 min. Then 250 µl of this suspension was withdrawn with a micropipette and an ATP measuring kit (Meidensha Corp., Lucifer AS) was added to it.

2.5 Undegradable carbon amount

FIGURE 4 shows the analytical method of estimation of UDC amount. The total carbon amount (T-C) and the inorganic carbon amount (TIC) derived from carbonate in the soil of the farmland were measured with the solid sample combustion method. The organic carbon amount (TOC) was measured with the Tyulin method (the titration method). The UDC amount was estimated by deducting the TIC amount and the TOC amount from the T-C amount.



(Measurement of total carbon) − (Measurement of inorganic carbon) − (Measurement of organic carbon) = (Estimation of charcoal carbon)

(Measurement of total carbon)		(Measurement of inorganic carbon)		(Measurement of organic carbon)		(Estimation of charcoal carbon)
Charcoal						Undegradable carbon
Organic carbon	−		−	Organic carbon	=	
Inorganic carbon		Inorganic carbon				

FIGURE 4. Estimation of undegradable carbon (UDC) amount in the soil.

2.6 Aggregate

The structure of the aggregate was estimated with the wet screen method. Several grams of the soil sample was put through the set of sieves 2 mm, 1 mm, 500 µm, 250 µm and 100 µm placed in a vessel filled with water, and then the vessel was slowly shaken up and down for 5 mins. After sample over the each sieve was dried for 24 hrs at 105°C, it was weighed. The mean weight diameter (MWD) of the soil was calculated with particle diameter (mm) and the integration number of the aggregate (%).

2.7 SEM photography

The surface of the aggregates was observed by scanning electron micrography. After the sample was freeze-dried in liquid nitrogen in a vacuum, it was fixed by osmic acid evaporation. The surface was then coated with a thin film of sputtered Pt–Pd alloy.

3 RESULTS AND DISCUSSION

3.1 Undegradable carbon

Carbon storage effect in the soil was estimated in the farmland used with the charcoal and the compost. Time dependence of various carbon amounts in the soil of the farmland was shown in FIGURE 5. The T-C, TOC and UDC amounts in the soil used with the charcoal and the compost remain larger than those in the soil with the compost.

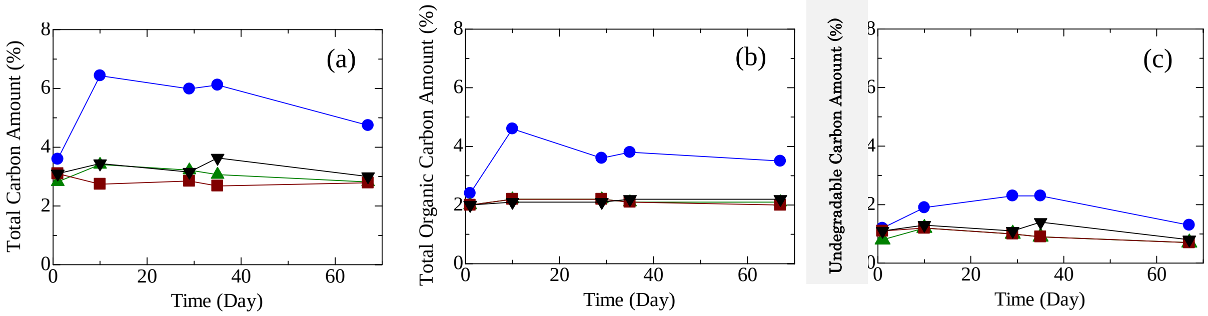


FIGURE 5. Time dependence of (a) T-C amount, (b) TOC amount and (c) UDC amount in the soil.
● : with charcoal and compost, ▲ : with compost,
▼ : with charcoal and ■ : no additives

3.2 Microorganisms

The concentration of microorganisms in the soil can be estimated by measuring the ATP concentration extracted from microorganisms. As ATP exists in mitochondria in the cytoplasm, the concentration of ATP can be used as an indication of microorganism activity.

FIGURE 6 shows time dependence of ATP concentration in the soil of the farmland. The ATP concentration increased drastically after using both of the charcoal and the compost, and about the half value of which was observed in the soil with compost and without charcoal. Then the ATP concentration in both of the soils gradually decreased. There no change of the ATP concentration was observed in the soil with charcoal and in the soil without additives.

It was suggested that this increase of the ATP concentration in the soil with the charcoal and the compost came from the accelerated proliferation of the microorganisms on the surface of the charcoal. Wood has pores that range from several to several tens of microns in diameter and originate from tracheae and charcoal prepared from carbonized wood has pores of almost the same size. Tanaka et al. (2005) and Yoshizawa et al. (2006-a, 2006-b) found that the proliferation of composting microorganisms was enhanced on and in bamboo charcoal as a medium to which rice bran had been added as a nutrient.

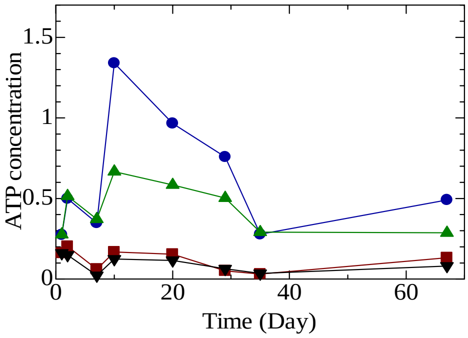


FIGURE 6. Time dependence of ATP concentration of the soil.
● : with charcoal and compost, ▲ : with compost,
▼ : with charcoal and ■ : no additives.

3.3 Aggregation

FIGURE 7 shows the accumulation curve of the particle size of the soils sampled after 10 days from using biochar and compost in the farmland. In the soils with non-additives and with the charcoal, fractions under 250 µm are 65%, which decrease 44 - 51% in the soils used with the compost and charcoal mixture and with the compost, accompanied with the increase of aggregates from 1 to 5 mm.

FIGURE 8 shows time dependence of the MWD value of the soil. The MWD value of the original soil in the farmland was about 30 at the beginning of the test. In the soil without additives, the aggregate was scarcely developed. In the initial period after using the charcoal and the compost mixture and compost, the MWD value increased to 50 - 60, and then gradually decreased.

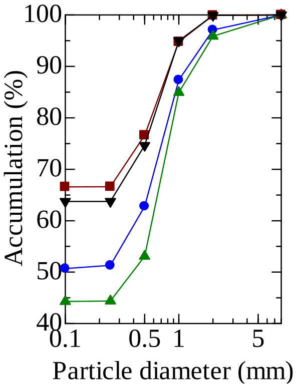


FIGURE 7. Particle diameter distribution of the soil.
● : with charcoal and compost, ▲ : with compost,
▼ : with charcoal and ■ : no additives.

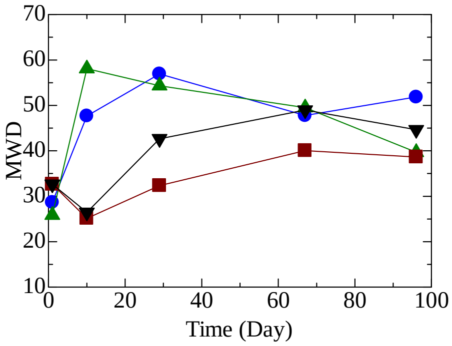


FIGURE 8. Time dependence of the MWD value of the soil.
● : with charcoal and compost, ▲ : with compost,
▼ : with charcoal and ■ : no additives.

FIGURE 9 shows the SEM photograph of the surface of the typical aggregate of the soil with the charcoal and the compost. The diameter of the aggregate, was almost 2 mm with smooth surface in FIGURE 9 (a). Magnified photographs on the surface of the aggregate are shown in FIGURE 9 (b), (c) and (d). Many kinds of microorganisms that proliferated on the surface were observed. It was suggested that the soil was aggregated with microorganisms proliferated on the surface of the aggregate in the soil.

FIGURE 10 shows the SEM photographs of the surface of the typical aggregate of the soil with the compost. Almost the same surface morphology was observed as the ones with charcoal and the compost.

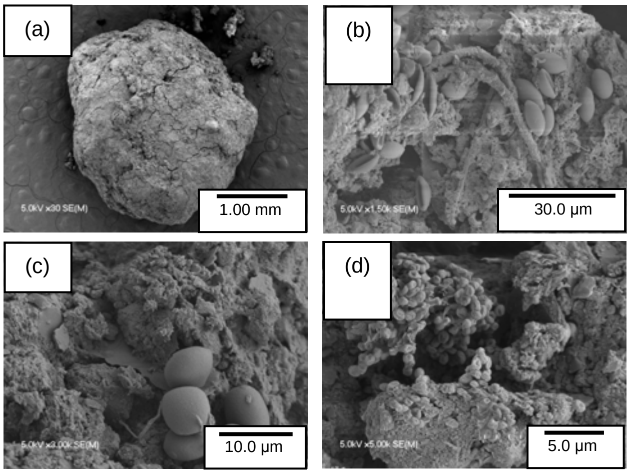


FIGURE 9. SEM photographs of the aggregate of the soil with charcoal and compost mixture.

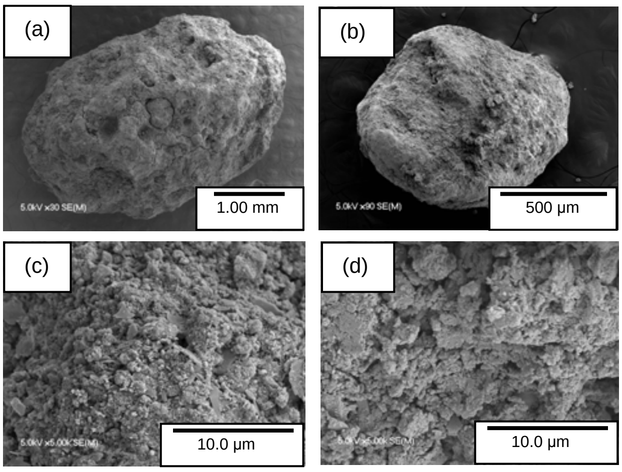


FIGURE 10. SEM photographs of the aggregate of the soil with compost.

4 CONCLUSIONS

Carbon storage effect in the soil was recognized in the farmland used with the charcoal and the compost by establishing estimation method of UDC content.

For the effect of the charcoal and the compost using in the farmland on the increase of the soil aerobic microorganisms, the time change of the ATP concentration was measured on the soils with the charcoal and the compost. The effect of the charcoal and the compost on aggregation of the soil was studied.

The ATP concentration increased drastically after using both of the charcoal and the compost, and about the half value of which was observed in the soil with compost and without charcoal.

The aggregation of the soil in farmland was developed by using the charcoal and the compost. It was suggested that the soil was aggregated with microorganisms proliferated on the surface of the aggregate in the soil.

5 ACKNOWLEDGEMENTS

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(103) COMPOSITIONAL SPACE CONSTRAINTS DISTORT C MINERALIZATION MODELS OF ORGANIC RESIDUES

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EXECUTIVE SUMMARY

Carbon mineralization of organic residues in soils is a complex process where labile C is mineralized to end products and stable C accumulates in the soil. The CO_2 evolving during C mineralization in soils contributes to global warming while microbial byproducts and stable C contribute to soil aggregation and C sequestration. The capacity of the soil to sequester C physically, chemically and biochemically depends not only on soil minerals but also on biochemical composition of added organic residues. In C models, biochemical and ash fractions of organic residues are often related to the mineralization of labile C using regression analysis. There are three misconceptions using raw proportions in C models that lead to numerical biases. First, because biochemical fractions are constrained to a close compositional space, their confidence intervals about means may extend to below 0 or above 100 and are thus conceptually meaningless. One fraction is also redundant due to closure: in a composition made of D parts, one component can be deduced from the difference between 100% and the sum of other components, hence leaving D-1 degrees of freedom. Finally, the C forms are often reported on different scales of measurement (fresh, dry or organic matter basis). As a result of redundancy and the various scales of measurements, raw concentration data are spoiled with spurious correlations. Although spurious correlations that were first identified by Karl Pearson in 1897, no solution to this problem has been proposed until recently with the development of compositional data analysis. Spurious correlation may distort linear models such as multiple regression and principal component analysis. Our objective was to show the nature of spurious correlations among raw concentration data of organic residues and to elaborate a compositional model that avoids numerical biases. Isometric log ratio (*ilr*) coordinates were computed from sequential binary partition of balances between two sub-compositions and were so arranged as to reflect the system under study, i.e. CO_2 = f(initial biochemical composition). Using a published dataset, we balanced C and N forms with ash, as well as more labile and more recalcitrant C and N forms. There were *D*-1 *ilr* coordinates (orthogonally arranged balances) as imposed by the principle of orthogonality. We showed that there were spurious correlations among components expressed either on dry or organic matter basis, because correlation coefficients differed in magnitude, sign and significance depending on scale of measurement. In contrast, the *ilr* coordinates were free from spurious correlations by definition because they are orthogonal to each other. The *ilr* coordinates are also free to range in the real space (±∞) because ratios can be very high or very low depending on numerator and denominator. The R^2 values for equations relating the labile C pool to raw proportions on dry or organic mass bases or to balances were high (0.95) and similar. However, the problem is one of interpretation. The dry and organic mass scales produced different independent variables except N_{SOL}. The variable C_{HEM} was assigned a negative coefficient on the dry mass scale and a positive one on the organic mass basis while the HEM fraction is part of the C labile pool. One must conclude that regression models on dry or organic mass scales are accurate but incoherent, even contradictory. On the other hand, using balances, the labile C pool was shown to increase with decreasing C/N ratio and increasing ratios between more labile and more recalcitrant C forms, as expected from theory. Compared to using raw concentration data, the relationships between the labile C pool and composition of organic products can be interpreted consistently after recognizing their intrinsic compositional nature. Multivariate analyses should be conducted preferably using the scale-invariant isometric log ratio transformations rather than raw proportions. Balances can be elaborated based on sound theory on C mineralization depending on chemical and biochemical composition of organic products. However, the balances proposed here could be re-arranged or changed upon amalgamation depending on the nature of the data and the hypotheses tested, as long as they remain orthogonal to each other. This paper warns carbon data analysts that the specific numerical properties of compositional data require special log ratio transformation before conducting univariate or multivariate analyses. The *ilr* approach could provide an unbiased carbon index for the contribution of organic products to greenhouse gases and C sequestration in soils.

1 INTRODUCTION

Organic amendments and fertilizers as well as crop residues are important sources of carbon and nitrogen contributing to soil quality and plant nutrition. A moment's reflection suggests that the composition of organic products, not only total C and N and ash content, determines their C and N mineralization in soils. The current approach to model C in soils is thus to relate biochemical C fractions such as C pools of organic materials to C mineralization parameters using multiple linear regression analysis (Gabrielle et al., 2004; Lashermes et al., 2009; Parent et al., 2011). The estimates of biochemical components are assumed to be random variables free to range in the real space ($\pm\infty$).

1.1 Background

The composition of organic products is represented by strictly positive data constrained between 0 and 100%. In such a space, at least one component is not independent as computed by difference between 100% and the sum of others. Similar to a mixture of components in a recipe, any one fraction in an organic product must increase at the expense of other fractions due to closure to the unit of measurement and combinations such as all components being equal to 0 or 100% are impossible. The trivial case is a 2-compositional system where the correlation coefficient between both components must be exactly -1: any increase in one component decreases the proportion of the other by exactly the same value. There are thus D-1 degrees of freedom in a composition of D-parts. Multivariate models using raw proportions are thus distorted by spurious correlations due to redundancy and change in scale of measurement (Aitchison, 1986).

Compositional data analysis (CDA) using log-ratio transformations avoid these difficulties (Egozcue and Pawlowski-Glahn, 2006). Aitchison (1986) proposed using additive and centered log ratio transformations. Later on, Egtozcue et al. (2003) proposed using isometric log-ratios (*ilr*). The *ilrs* are coordinates with orthonormal basis that generates D-1 balances of components arranged in a way to describe the system under study and facilitate interpreting the results (Egozcue et al., 2003). The *ilr* approach appeared to be the most appropriate in the multivariate analysis of compositional data (Filzmoser and Hron, 2011) and to describe balances in geochemical systems (Buccianti, 2011), nutrient diagnosis of plant tissues (Parent, 2011) and as unbiased index of soil aggregation (Parent et al., 2012).

1.2 Research objectives

Our objective was to compare raw proportions and *ilr* transformations of biochemical composition of organic products in multiple regression models of C and N dynamics in soils using a published dataset (Thuriès et al., 2002).

2 METHODOLOGY

2.1 Dataset

In the Thuriès et al. (2002) incubation study, model parameters were mineralizable C as dependent variable and nine fractions as follows: ash, soluble (SOL) C, hemicellulose (HEM) C, cellulose (CEL) C, lignin (LIG) C, SOL N, HEM N, CEL N, and LIG N, all expressed on dry matter basis.

2.2 Isometric log ratio (orthogonal balances)

Isometric log ratio transformations are based on balanced contrasts between groups of fractions. Balance coefficients for partitions refer to positive, negative, and null codes, s is the number of negative components in the contrast, r is the number of positive components, and (r+s) is the total number of components to be partitioned. Partitions were selected to represent the order of decomposability of biochemical components (Table. 1).

Table 1. Sequential binary partitions for the Thuriès et al. (2002) dataset

Ilr balance	C_{SOL}	C_{HEM}	C_{CEL}	C_{LIG}	N_{SOL}	N_{HEM}	N_{CEL}	N_{LIG}	Ash	r	s
1. $[C_{SOL},C_{HEM},C_{CEL},C_{LIG},N_{SOL},N_{HEM},N_{CEL},N_{LIG} \mid ash]$	1	1	1	1	1	1	1	1	-1	8	1
2. $[C_{SOL},C_{HEM},C_{CEL},C_{LIG} \mid N_{SOL},N_{HEM},N_{CEL},N_{LIG}]$	1	1	1	1	-1	-1	-1	-1	0	4	4
3. $[C_{SOL},C_{HEM} \mid C_{CEL},C_{LIG}]$	1	1	-1	-1	0	0	0	0	0	2	2
4. $[C_{SOL} \mid C_{HEM}]$	1	-1	0	0	0	0	0	0	0	1	1
5. $[C_{CEL} \mid C_{LIG}]$	0	0	1	-1	0	0	0	0	0	1	1
6. $[N_{SOL},N_{HEM} \mid N_{CEL},N_{LIG}]$	0	0	0	0	1	1	-1	-1	0	2	2
7. $[N_{SOL} \mid N_{HEM}]$	0	0	0	0	1	-1	0	0	0	1	1
8. $[N_{CEL} \mid N_{LIG}]$	0	0	0	0	0	0	1	-1	0	1	1

The i^{th} *ilr* coordinate was computed as a contrast between two groups of components as follows (Egozcue and Pawlowsky-Glahn, 2006):

$$ilr_{\mathrm{i}} = \sqrt{\frac{rs}{r+s}}\,ln\frac{g(x_+)}{g(x_-)} \qquad (1)$$

Where $g(x_+)$ and $g(x_-)$ are the geometric means of components in group x_+ and x_-. For example, the 3rd balance is computed as follows :

$$[C_{SOL}, C_{HEM}\,|C_{CEL}, C_{LIG}] = \sqrt{\frac{2\mathrm{x}2}{2+2}}\ln\left(\frac{\sqrt{C_{SOL}\mathrm{x}C_{HEM}}}{\sqrt{C_{CEL}\mathrm{x}C_{LIG}}}\right) \qquad (2)$$

3 RESULTS AND DISCUSSION

3.1 Thuriès et al. (2002) data reported on organic mass scale

Thuriès et al. (2002) reported data on a dry mass scale. The organic mass basis can be computed by dividing the biochemical fractions by (100-%ash). The biochemical fractions of organic products are presented in Annex 1. The isometric log ratios in Annex 2 were computed according to the sequential binary partition in Table 1. Correlation coefficients between components on dry and organic bases are presented in Tables 3 and 4, respectively.

Table 3. Correlation coefficients between components on dry mass basis; for 17 observations, r = 0.468 at P = 0.05 and r = 0.590 at P = 0.01.

Component	Total C	C_{SOL}	C_{HEM}	C_{CEL}	C_{LIG}	Total N	N_{SOL}	N_{HEM}	N_{CEL}	N_{LIG}	Ash
Labile C	-0.486	0.517	0.174	-0.704	-0.755	0.737	0.933	0.353	0.040	-0.375	0.216
Total C		0.036	0.367	0.678	0.553	-0.149	-0.557	0.259	0.307	0.478	-0.926
C_{SOL}			-0.047	-0.050	-0.570	0.099	0.404	-0.034	-0.273	-0.532	-0.279
C_{HEM}				-0.211	-0.253	0.561	0.085	0.849	0.667	0.329	-0.398
C_{CEL}					0.552	-0.602	-0.749	-0.285	-0.032	0.268	-0.536
C_{LIG}						-0.424	-0.664	-0.238	0.029	0.548	-0.342
Total N							0.752	0.712	0.578	0.279	-0.060
N_{SOL}								0.223	-0.047	-0.310	0.295
N_{HEM}									0.672	0.296	-0.367
N_{CEL}										0.783	-0.292
N_{LIG}											-0.379

Table 4. Correlation coefficients between components on organic mass basis; for 17 observations, r = 0.468 at P = 0.05 and r = 0.590 at P = 0.01.

Component	Total C	C_{SOL}	C_{HEM}	C_{CEL}	C_{LIG}	Total N	N_{SOL}	N_{HEM}	N_{CEL}	N_{LIG}
Labile C	-0.836	0.690	0.090	-0.738	-0.736	0.862	0.946	0.271	0.032	-0.418
Total C		-0.625	0.159	0.650	0.627	-0.718	-0.865	-0.087	0.163	0.392
C_{SOL}			-0.199	-0.382	-0.735	0.346	0.646	-0.189	-0.338	-0.693
C_{HEM}				-0.356	-0.415	0.307	-0.028	0.784	0.630	0.148
C_{CEL}					0.507	-0.790	-0.763	-0.397	-0.081	0.184
C_{LIG}						-0.576	-0.611	-0.392	-0.132	0.522
Total N							0.871	0.503	0.365	0.033
N_{SOL}								0.107	-0.092	-0.333
N_{HEM}									0.672	0.155
N_{CEL}										0.647

The correlation coefficients differed in terms of magnitude, significance and sign whether components were expressed on dry or organic mass basis. The differences in correlation coefficients between scales of measurement indicate spurious correlations. Such spurious correlations were due to scale dependency and data redundancy. As shown by correlation coefficients, the labile C pool increases as C_{SOL} and N_{SOL} increase and as C_{CEL} and C_{LIG} decrease. Changing the scale of measurement (either dry or organic mass basis) may lead to different interpretations of the results from multivariate models such as incoherence and contradictions. Because multiple regression analysis, principal component analysis and other multivariate analyses depend on the degree of correlation between variables, the number of candidate predictor variables and their coefficients may vary between models and lead to incoherent conclusions.

Correlation coefficients between labile C and balances are presented in Table 5. As expected, the labile C pool increased with the balance between organic and inorganic components, the balance between more easily decomposable and the balance between more recalcitrant C and N forms (*ilr1-2* and *ilr5-7*) increased and as the ratio between C forms and N forms decreases (*ilr2*).

Table 5. Correlation coefficients between balances; for 17 observations, r = 0.468 at P = 0.05 and r = 0.590 at P = 0.01.

Component	ilr1	ilr2	ilr3	ilr4	ilr5	ilr6	ilr7	ilr8
C labile	-0.194	-0.701	0.859	0.427	0.272	0.137	0.413	0.509
ilr1		-0.162	-0.002	-0.606	0.364	0.158	-0.802	0.100
ilr2			-0.675	0.137	-0.298	0.023	0.085	-0.601
ilr3				0.065	0.501	0.058	0.166	0.686
ilr4					-0.413	0.100	0.759	-0.330
ilr5						0.005	-0.368	0.780
ilr6							-0.044	-0.138
ilr7								-0.064

The three equations relating the labile C pool to components expressed on dry or organic mass basis or transformed into *ilr* balances explained most of the variation in the labile C pool (Fig. 1).

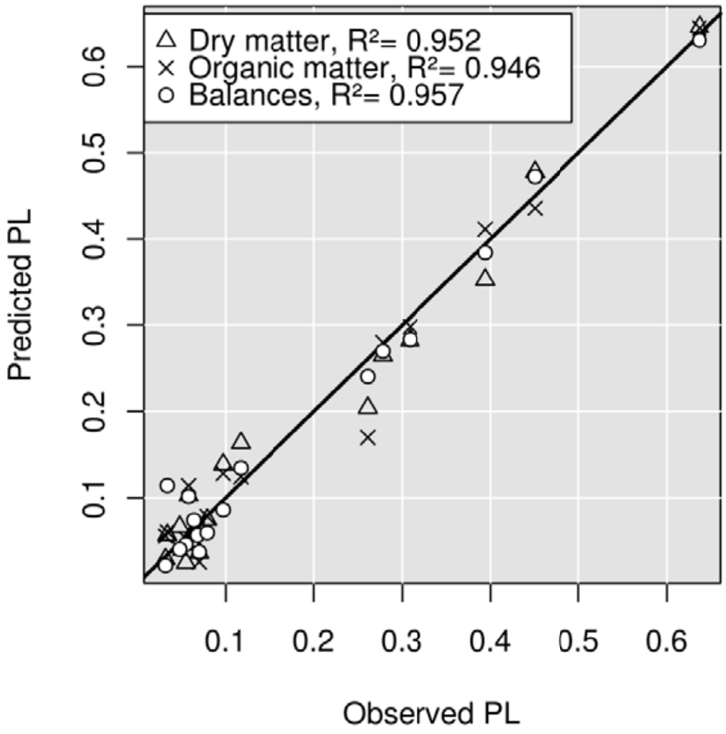

Figure 1. Relationships between observed labile C pool (PL) and the ones predicted by the three regression equations.

Retaining coefficients by the Akaike information criterion (AIC), the multiple stepwise regression equations were computed as follows:

Dry mass scale:

$$Labile\ C = 0.1248 - 0.0017ash - 0.0044C_{HEM} + 0.0388N_{SOL} + 0.1898N_{CEL} - 0.683N_{LIG} \quad (3)$$

Organic mass scale:

$$Labile\ C = 1.3344C_{SOL} + 1.6876N_{SOL} + 3.4107N_{HEM} \quad (4)$$

Scale-invariant balances (Table 1):

$$Labile\ C = 0.3180 + 0.0477ilr_1 - 0.0577ilr_2 + 0.0661ilr_5 + 0.1137ilr_6 + 0.0990ilr_7 \quad (5)$$

While the R^2 values were all in the order of 0.95 whatever the scale-dependency or invariance, the problem with scale dependency and data redundancy is one of interpretation. Equations using the dry and organic mass bases were spoiled with spurious correlations, hence leading to different interpretations. The assumption of normal distribution was also incorrect. The dry and organic mass scale models produced different independent variables except N_{SOL} although raw analytical results are exactly the same. The variable C_{HEM} showed a negative coefficient on the dry mass scale and a positive one on the organic mass basis while the HEM fraction is labile and should contribute positively to the labile C pool. The modeler can only conclude that these regression models are accurate but incoherent, even contradictory. On the other hand, using balances, the labile C pool increased with decreasing C/N ratio and increasing ratios between more labile and more recalcitrant C forms, as expected.

The SBP is arranged to facilitate interpreting results (hypothesis testing). The nature of biochemical data and of the theory to be tested may suggest other types of balances among the components of organic products. The balances proposed here could be re-arranged as long as they remain orthogonal to each other. T

CONCLUSIONS

This paper warns carbon data analysts that the specific numerical properties of compositional data require special transformation before conducting univariate or multivariate analysis. Compared to using raw concentration data, the relationships between the labile C pool and composition of organic products can be interpreted consistently after recognizing first their compositional nature. Multivariate analyses should thus be conducted preferably using the scale-invariant isometric log ratio transformations rather than raw proportions. The *ilr* approach could provide an unbiased carbon index for the contribution of organic products to greenhouse gases and C sequestration in soils.

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Annex1. Biochemical composition of plant residues in the Thuriès et al. (2002) experiment

Product	Labile C	C_{SOL}	C_{HEM}	C_{CEL}	C_{LIG}	N_{SOL}	N_{HEM}	N_{CEL}	N_{LIG}	Ash
	Fraction of added C	g g-1 dry mass basis (x100)								
coffk	0.055	16.8	7.4	18.4	11.1	0.29	0.49	0.09	1.1	3.1
wgrap	0.070	7.0	2.5	11.2	29.9	0.39	0.66	0.06	1.62	8.9
dgrap	0.058	16.3	3.6	13	16.4	0.7	0.48	0.19	0.88	7.1
olivp	0.048	12.5	6.1	13.1	15.2	0.63	0.07	0.19	1.08	8.8
kokoa	0.278	24.7	4.2	8.7	6.1	2.71	0.61	0.61	0.61	9.1
shepm	0.064	9.0	14.8	5.9	8.2	1.07	0.63	0.2	0.33	28.1
chicm	0.309	16.9	11.8	5.5	3.4	5.24	0.12	0.48	0.22	32.3
nfeat	0.068	3.3	20.2	10.6	20.4	1.08	4.0	2.51	6.97	3.8
featm	0.450	18.6	24.2	3.2	1.1	7.81	6.23	0.88	0.26	2.8
guano	0.637	17	0.1	0.1	0.4	15.5	0.01	0.01	0.06	43.3
gnofer	0.394	9.1	12.4	5.0	0.8	4.62	3.83	0.9	0.16	40.4
comfer	0.261	14.3	2.1	10.8	9.7	2.62	0.11	0.39	0.62	25.5
compo a	0.117	11.2	4.7	11.9	9.7	1.52	0.2	0.53	0.62	32.2
compo b	0.097	11.2	3.3	10.9	10.8	1.07	0.17	0.5	0.72	34.4
compo e	0.034	7.3	2.1	7.1	12.3	1.33	0.04	0.18	1.15	40.4
compo +	0.079	9.7	2.4	8.5	13.3	1.11	0.01	0.27	1.19	32.1
compo p	0.032	6.4	2.7	6.7	18.5	0.83	0.07	0.18	1.33	40.2

Annex 2. Data in Table 2 *ilr*-transformed according to the SBP model in Table 1

Product	C labile	ilr1	ilr2	ilr3	ilr4	ilr5	ilr6	ilr7	ilr8
coffk	0.055	-0.763	5.093	-0.248	0.580	0.357	5.534	-0.371	-1.770
wgrap	0.07	-1.838	4.371	-1.476	0.728	-0.694	2.054	-0.372	-2.331
dgrap	0.058	-1.476	4.353	-0.645	1.068	-0.164	2.866	0.267	-1.084
olivp	0.048	-1.874	5.067	-0.480	0.507	-0.105	-1.301	1.554	-1.229
kokoa	0.278	-1.380	3.217	0.335	1.253	0.251	1.341	1.054	0.000
shepm	0.064	-2.872	4.201	0.506	-0.352	-0.233	0.861	0.375	-0.354
chicm	0.309	-2.854	3.866	1.183	0.254	0.340	1.121	2.670	0.552
nfeat	0.068	-0.085	1.856	-0.588	-1.281	-0.463	-1.430	-0.926	-0.722
featm	0.45	-0.030	1.753	2.426	-0.186	0.755	0.373	0.160	0.862
guano	0.637	-4.909	2.332	1.875	3.632	-0.980	0.360	5.194	-1.267
gnofer	0.394	-2.879	1.830	1.670	-0.219	1.296	0.416	0.133	1.221
comfer	0.261	-2.659	3.789	-0.625	1.356	0.076	11.393	2.242	-0.328
compo a	0.117	-2.859	3.895	-0.393	0.614	0.145	-25.702	1.434	-0.111
compo b	0.097	-3.022	3.926	-0.579	0.864	0.007	-2.930	1.301	-0.258
compo e	0.034	-3.600	4.139	-0.870	0.881	-0.389	-1.472	2.478	-1.311
compo +	0.079	-3.339	4.777	-0.790	0.988	-0.317	-0.594	3.330	-1.049
compo p	0.032	-3.527	4.223	-0.985	0.610	-0.718	-1.412	1.749	-1.414

(136) CARBO-PRO: A SIMULATION MODEL TO MANAGE SOIL ORGANIC MATTER THROUGH COMPOST APPLICATIONS

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EXECUTIVE SUMMARY

The decline in soil organic C (SOC) content leads to reduced fertility and biological activity and reversely to greater susceptibility of soil to erosion. The loss of SOC has been identified as a major threat towards the soil resource. In addition, soils account for one of the largest terrestrial C reservoirs and small but consistent increases in SOC stocks could mitigate climate change effects by storing atmospheric CO_2-C in soil organic matter. Annual application of exogenous organic matter (EOM) to cultivated land may lead to long continued accumulation of SOC. In Europe, recycling of organic biodegradable wastes through composting or anaerobic digestion is encouraged and is expected to increase in the future. The produced composts and digestates applied on cultivated soils differ in their potential contribution to SOC, depending on their origin and degree of transformation before being added to soil. Since C stocks change slowly, repeated applications are needed to observe an increase in SOC.

Multi-compartment models of C turnover in soil accurately simulate SOC dynamics in the long-term under different climatic conditions and soil types. RothC is one of the most widely used model that simulates SOC dynamic based on relatively few parameters and input data. In RothC, total organic C in EOM is distributed into pools of labile (DPM), resistant (RPM) and humified (HUM) organic matter according to partition coefficients (f_{DPM}, f_{RPM} and f_{HUM}, respectively). Equations based on the standardized Indicator of residual organic carbon I_{ROC} have been developed to calculate the partition coefficients for any kind of EOM and then simulate the evolution of SOC after repeated applications of EOMs.

The objective of this study was to develop a web based simulation model allowing the estimation of the effect of repeated or single EOM land application on SOC storage and various associated soil properties.

The CARBO-PRO tool is based on the RothC model associated with databases of soil, EOM and climate characteristics. CARBO-PRO makes it possible to simulate the evolution of SOC for any chosen strategy of EOM application under all French soil and climate conditions. Many soil properties are related to SOC contents, including soil aggregate stability, soil water retention, and nutrient availability. In the CARBO PRO tool, available pedotransfer functions relating aggregate stability, cation exchange capacity and water retention to SOC content have been associated to the RothC model. Thus together with SOC increase, the evolution of related soil properties is simulated for the tested scenario of EOM applications.

1 INTRODUCTION

1.1 Background

Soil organic carbon (SOC) constitutes one of the largest C reservoirs on earth. The amount held in the first meter of soil is approximately 1500 Gt C, that is about twice the amount of C contained in the atmosphere as CO_2 (about 750 Gt C) and three times the amount of C contained in vegetation (about 500 Gt C) (IPCC, 2001). Between 1850 and 2000, the soil C pool has lost about 78 ± 12 Gt C due to land use change. In comparison, 270 Gt C were emitted to the atmosphere as CO_2 due to fossil fuel combustion (Lal, 2009). In addition to land use change, Bellamy et al (2005) showed that arable land in United Kingdom and Wales tended to loose SOC between 1978 and 2003, which may be attributed to a decrease of C inputs in cultivated soils due to intensive management (Ciais et al., 2010).

Soil organic carbon (SOC) is involved in most of the functions and ecosystem services provided by the soil (Van-Camp et al., 2004). It is a key determinant for the chemical, biological and physical fertility of soil and determines its ability to ensure sustained food production (FAO, 2005). The SOC provides nutrients for plants, increases the ability of soil to retain nutrients, increases soil porosity and water holding capacity, leading to improved soil tillage and workability (Van-Camp et al., 2004). The soil aggregate stability and thus the resistance of soil to erosion (Le Bissonnais et al., 2002) is also highly related with SOC content. For these reasons, the loss of SOC has been identified as a major threat to the quality of the soil resource in a proposal for a directive for soil protection (European Commission, 2006).

Land application of organic amendments derived from waste materials from urban, industrial or agricultural activities (referred to as exogenous organic matter: EOM) constitutes an important practice which can be used to increase or maintain SOC contents (Arrouays et al., 2002) and to improve soil properties related to SOC levels. In Europe, recycling of organic biodegradable wastes is expected to increase in the forthcoming years while decreasing waste landfilling and incineration without energy recovery (European Commission, 2010). Land application after some biological treatment (e.g. composting or anaerobic digestion with digestates production) is likely to be one of the most environmentally friendly waste management options (European Commission, 2010). However, detailed environmental assessments to allow a proper comparison with other waste management options are still lacking. Therefore it is also impossible to identify the best biological treatment in term of environmental performance. In this context, the production of quantitative applied tools allowing the effects of EOM land application to the soil quality to be estimated is urgently needed to identify the most suited waste management options.

1.2 Research objectives

The objective of this study was to develop a web based simulation model allowing estimation of the effect of repeated EOM land application on SOC storage and various associated soil properties. The developed model is based on the RothC SOC dynamics model, published pedotransfer functions and database for climatic, soil and EOM characteristics data.

2 METHODOLOGY

CARBO-PRO allows estimating of the potential C storage in soils after land application of EOM and changes in certain soil properties arising from: aggregates stability, cation exchange capacity and water-holding capacity. The C simulation model is based on the RothC carbon dynamics model (Coleman and Jenkinson, 1999; Jenkinson and Rayner, 1977) allowing to simulate the potential C storage in soil after EOM application. The simulated C storage output is then used to estimate changes in the different soil properties using pedotransfer functions (Fig. 1).

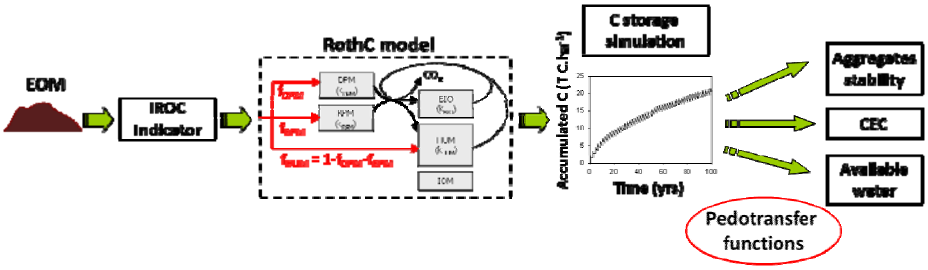

Figure 1 **General scheme of CARBO-PRO**

2.1 Potential carbon storage in soil after PRO applications

The RothC model describes the dynamics of soil C using three organic C pools: HUM (humified organic C, mean residence time in soil of 50 years), BIO (microbial biomass, mean residence time of 1.5 years) and IOM (inert organic C which does not degrade during the duration of simulation). When an EOM is applied to the soil, its organic C is partitioned into a pool of labile C: DPM (mean residence time in soil of 1.2 months), a more resistant C pool: RPM (mean residence time of 3.3 years), and possibly into the humified pool (Fig. 1). The necessary inputs for the model include weather and soil texture data as well as an indicator of EOM stability. The application includes databases providing inputs for the RothC model which are detailed bellow.

2.1.1 Weather data

The application includes monthly weather data for metropolitan France regions for the parameters: average air temperature, cumulative rainfall and potential evapotranspiration (PET) by the Penman method (Penman, 1948). The data are average values over a ten years period (2001-2010) from all weather stations within the regions. The number of weather stations ranged between 1 and 6, with a mean of 2.6. The user can also type in manually his weather data.

2.1.2 Soil texture data

Choices of soil textural classes are proposed to define the proportions of clay, silt and sand making up soil (% Sand = 100-Clay - Silt). A range of 17 soil textural classes is proposed, based on the GEPPA soil texture classification (Richer-de-Forges et al., 2008).

2.1.3 Bulk density of soil

The bulk density of soil is used to calculate soil C stocks from soil C contents and conversely, in order to apply pedotransfer functions to estimate changes in various soil properties based on C contents.

Bulk density is estimated from the clay and silt content and initial soil organic carbon using the pedotransfer function of Martin et al. (2009) implemented in the R software (R development core team, 2011). This pedotransfer function was developed from the French soil quality data network (RMQS) throughout France territory and is therefore especially well-suited to this area.

2.1.4 Organic matter quality of EOM

The C contained in the EOM is partitioned in the entry pools of the RothC model according to partition coefficients, namely f_{DPM}, f_{RPM} and f_{HUM} for the proportion of C in the DPM, RPM and HUM pools respectively (Fig. 1). These partition coefficients are determined from empirical functions based on the indicator of residual organic C (I_{ROC}, Lashermes et al., 2009) which is derived from standardized laboratory characterizations of EOM (AFNOR, 2009; Lashermes et al., 2009) by the following equations (Peltre et al., 2012):

$$f_{DPM} = -1.254\ I_{ROC} + 115.922$$
$$f_{RPM} = 0.979\ I_{ROC} - 8.928$$
$$f_{HUM} = 100 - f_{DPM} - f_{RPM}$$

Where $I_{ROC} = 44.5 + 0.5\ SOL - 0.2\ CEL + 0.7\ LIC - 2.3\ C3d$ with SOL, CEL and LIC the soluble, cellulose and lignin+cutin like fractions, respectively, of the Van Soest biochemical fractionation (AFNOR , 2009a) and C3d the proportion of EOM-C mineralized after 3 days of incubation with soil under controlled conditions at 28°C (AFNOR, 2009b). The I_{ROC} values for different types of EOM are issued from a database developed in a previous study gathering biochemical characteristics of more than 400 EOMs (Lashermes et al., 2009). Alternatively, the I_{ROC} can be estimated from the biological stability index (BSI) which is an older indicator of EOM stability that is still used in France (AFNOR, 2005b; Linères and Djakovitch, 1993). When typing a BSI value, the I_{ROC} value is automatically computed according to an empirical equation developed using all EOM present in the database ($R^2 = 0.43$):

$$I_{ROC} = 0.6418*BSI + 36.009$$

2.2 Additional nitrogen mineralization

Nitrogen in EOM is present in large part in organic form, bounded to organic matter more or less stabilized against biodegradation. Therefore, only a small fraction of N provided by the EOM is immediately available for plant uptake, whereas the remaining organic part is progressively mineralized during organic matter decomposition. The amount of additional mineral nitrogen ($\Delta N\ min$) released in relation with the increased soil organic matter (ΔC) is calculated by multiplying the amount of total soil nitrogen derived from EOM ($\Delta C / C:N$) by the annual coefficient of mineralization of soil organic matter (K2, second term in equation below) (Henin and Dupuis, 1945; Rémy and Marin-Laflèche, 1976) according to the equation (Comifer, 2011):

$$\Delta N \min = \frac{\Delta C}{C:N} * \frac{12 * (0.2 * \theta mean - 1) * Fsyst}{(CaCO_3 * 0.3 * 20)(Clay + 20)}$$

The total amount of soil nitrogen is derived from the C storage simulation output (ΔC), assuming a constant C:N ratio of 9 and using the ‘Fsyst’ factor accounting for the organic amendments application rate and level of biological stability of the EOM (Table 1). The assumption is made that crop residues are returned to the soil to half of the total amount.

Table 1 **Fsyst factor values for different frequency of EOM application and different type of EOM.**

Frequency of applications	Never	5-10 years		3-4 years		1-2 years	
Type of product		Type A	Type B	Type A	Type B	Type A	Type B
Fsyst factor	0.90	1.00	0.95	1.05	1.00	1.10	1.02

Type A : product with slow decomposition rate (I_{ROC} >65%) ; Type B : product with fast decomposition rate (I_{ROC} <65%)

2.3 Change in soil properties following EOM application

Increasing levels of SOC after EOM application results in many changes of soil properties such as aggregate stability, cation exchange capacity (CEC), water holding capacity etc. CARBO-PRO includes a module estimating these properties based on published pedotransfer functions.

2.3.1 Soil aggregates stability

The soil aggregates stability can be characterized by the method of Le Bissonnais (AFNOR, 2005a; Le Bissonnais, 1996) that combines slow wetting, fast wetting and mechanical breakdown tests on soil aggregates of size 3-5 mm. The size distribution of aggregates is measured after each test and the mean weighted diameter of aggregates (MWD) is calculated. The higher the MWD, the higher the soil aggregate stability is. In CARBO-PRO, the pedotransfer function of Darboux et al. (2009) is used to estimate the MWD after a fast wetting test by the method of Le Bissonnais reproducing the effect of a high intensity rainfall according to the equation:

$$\text{MWD (t)} = 0.34 \text{ orgC (t)} + 0.008 \text{ Clay} + 0.17$$

Where *orgC* and *Clay* are the organic carbon and clay content of soil (in %). This equation has been built from a database of 480 samples mainly from surface horizons of cultivated soils in France (Darboux et al., 2009).

2.3.2 Cation exchange capacity (CEC)

The soil cation exchange capacity is estimated in CARBO-PRO from the pedotransfer function of Manrique et al. (1991):

$$\text{CEC(t)} = 3.67\, orgC\text{(t)} + 0.196\, Clay + 3.238$$

With the CEC expressed in milliequivalents / 100 g soil (meq 100 g^{-1}), the organic C content and clay content expressed as %. This function has been developed from over 5000 soil samples from the United States. We used it assuming that the diversity of soils used for its calculation includes the diversity of soils of metropolitan France.

2.3.3 Water holding capacity

The available water capacity or available water content (AWC in mm) is the water that can be stored in soil available for plant uptakes. It is the difference between the moisture at field capacity (θ_{33} in mm) and at permanent wilting point (θ_{1500} in mm).

The soil water retention properties are related to both its texture and its organic carbon content (Hudson, 1994, Rawls et al., 2003).

The estimate of available water capacity in the topsoil horizon proposed in CARBO-PRO is based on pedotransfer functions of Rawls et al. (2003) developed from 12000 soil samples from the United States national database of soil characterization. These functions predict the water content at field capacity and at permanent wilting point (at -33 and -1500 kPa, and noted θ_{33} θ_{1500}, respectively). This relation provides lower confidence for fine-textured soils having clay contents > 50% for which results should be interpreted cautiously.

2 RESULTS AND DISCUSSION

3.1 General interface

The general interface is made up with three sub-windows: two windows for data inputs (soil and EOM data) and a main window with three tables of results for soil carbon storage, soil properties and scenario comparison (Fig. 2). The top bar contains a project management menu to create and save projects containing up to 5 scenarios, buttons to switch the displayed scenario and add scenarios to the comparison tool. Additional functions make it possible to change the language (French/English), to display information about the application and details on calculation, to e-mail the administrator or to logout.

3.2 Data input

The input data are defined using two windows: a climate and soil data window and an EOM data window (Fig. 2, left side). Climatic data are defined by choosing a French region in a dropdown list. Soil textural class is defined in a second dropdown list. After setting the initial soil C content, the bulk density of soil is computed using the pedotransfer function previously described. Soil parameters also include calcium carbonate content and depth of the ploughed layer. In the EOM data window, a dropdown menu contains a range of 25 predefined types of EOM. Each type of EOM is associated with an I_{ROC} indicator value (median values ± third and first quartiles) and organic matter content. All default parameters can also be modified manually. Other fields allow defining of the applied dose of EOM, the dry matter content, the application frequency and the month where the EOM is applied. The example in Fig. 1 presents the application of a sewage sludge compost in the Brittany region of France on a silt clay soil at the rate of 10 tons of compost applied every second year in September.

3.2 Evolution of soil carbon storage

The window presenting the potential soil C storage displays the result of the simulations drawing soil C accumulation over a period of 100 years (Fig. 2, right side). Several key values and indicators are displayed over a period which can be defined by the user (20 years in the example presented in Fig. 2: total C applied, amount of C stored, portion of applied C stored in soil and yearly rate of storage. The amount of additional N mineralized during the year for which the results are displayed (20th in the example in Fig. 2) is also presented. All results are calculated based on the median value of the I_{ROC} indicator associated with the type of EOM previously selected. Results corresponding to the first and third quartile of the I_{ROC} value are also displayed as upper and lower curves in the figures and values between parentheses in the boxes. Detailed results can be displayed, comprising evolution of the soil C accumulation in each pool of the RothC model. The graphics can be exported as a pdf file and the numeric values of the simulations can be exported as an Excel file.

In the example presented, the application of 10 T of composted sewage sludge every second year results in the accumulation of 5.7 T of soil C per ha after 20 years, which corresponds to 41.0% of the total amount of C applied (13.8 T C ha^{-1}). The 20th year after the first application, 10.7 kg ha^{-1} of additional N would be mineralized.

test CP : Scénario par défaut

Bretagne
Silty clay

Clay content (%)	33
Silt content (%)	60
Sand content (%)	7
Org C content (%)	2
Bulk density (g/cm³)	1.4

Inititial soil carbon content

CaCO₃ content (%)	0
Tilled depth (cm)	29

Sewage sludge compost

	Q1	Median	Q3
IROC (%)	69.3	76.3	81.3

ISB (%): 0

Dose applied (T RW ha^{-1})	10
Dry matter content (%RW)	50
Organic matter content (%DM)	55.06

Application every 2 year
In september

Over 20 year

Results after 20 of application under the terms of the choosen scenario

Total carbon applied (T C ha^{-1})	13.8
Amount of stored C (T C ha^{-1})	5.7 (5.2; 6))
Proportion of stored C (% of total C applied)	41 (37.9; 43.2)
Storage rate (T C ha^{-1} y^{-1})	0.28 (0.26; 0.3)
Additional nitrogen mineralized the 20st year (kg N ha^{-1})	10.7 (9.9; 11.3)

Figure 2 General interface of the Carbo-PRO application. Data input windows (left side) and result window (right side)

3.3 Evolution of soil properties

The window on soil properties displays results of the evolution of soil properties deriving from the soil C accumulation. Three graphics are displayed for aggregate stability, cation exchange capacity, and changes in available water content after EOM application, along with boxes displaying numerical values for a period defined by the user (after 20 years of application in the example in Fig. 3). The starting values for soil properties (initial value) is automatically computed but can be modified which results in a re-computation of the final value and % of increase based on the new starting value. As for the soil C accumulation results, soil properties results are provided with lower and upper values corresponding to lower and upper values of the I_{ROC} indicator.

For the example presented, repeated applications of composted sewage sludge would lead to a 6.1% increase in aggregate stability (mean weight diameter of aggregates after a fast re-wetting of soil), 3.0% increase of cation exchange capacity and 0.3% of available water content.

3.4 Scenario comparison tool

The scenario comparison tool makes it possible to easily compare the results of different simulated scenarios. This screen contains bar charts with numeric value displayed at the top of the bars (Fig. 4). Each bar corresponds to a scenario previously defined. Up to five scenarios can be compared simultaneously. The comparison tool includes charts for stored C, aggregate stability, cation exchange capacity and available water content. All results are expressed as percentage of evolution compared to the initial state at the start of the simulations.

	Initial	Final	% increase
Aggregates stability (mean weight diameter in mm)	0.8	0.8 (0.8; 0.8)	6.1 (5.7; 6.5)
Cation exchange capacity (meq.100g^{-1})	17	17.6 (17.5; 17.6)	3 (2.8; 3.2)
Available water (mm)	46.3	46.4 (46.4; 46.4)	0.3 (0.3; 0.3)

Figure 3 Soil properties result window

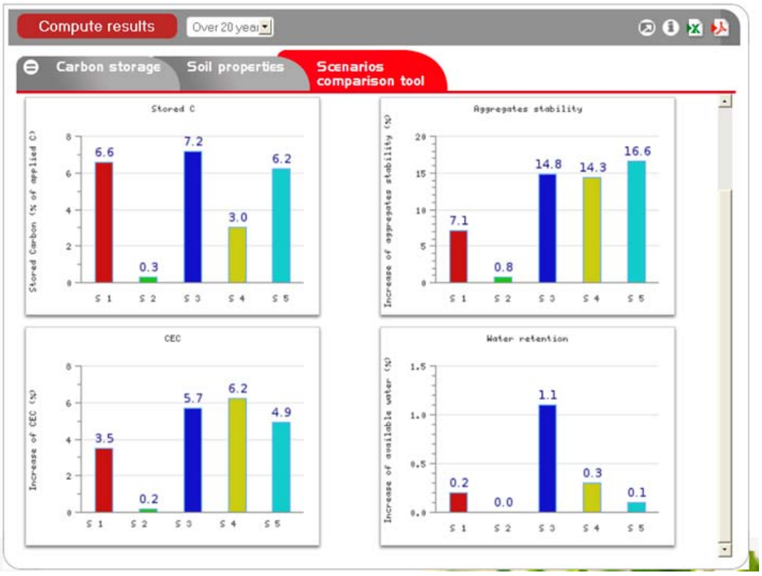

Figure 4 Window of scenarios comparison tool.

4 CONCLUSION

The simulation tool developed during this study allows an easy assessment of the amount of C that can be potentially stored in soil after the application of widely different types of organic amendments and the potential associated effects on aggregate stability, cation exchange capacity and water retention. The CARBO-PRO tool comprises databases for most of the parameters necessary to conduct the simulations so that very few data are required from the user. These databases comprise data for a broad range of EOM and pedoclimatic conditions. Besides it can also be used for other types of EOM or pedoclimatic conditions not contained in the proposed databases simply by replacing the proposed input values. The interface has been designed to be intuitive and self-explanatory and also includes a scenario comparison tool allowing an easy and interactive comparison of the combined effects of different types of EOM and pedoclimatic situations. Overall the CARBO-PRO tool is likely to be useful whenever researchers, professionals of waste recycling or farmers want to assess the potential effect of EOM land application on soil carbon storage and subsequent change in soil properties.

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(149) MODELLING THE DYNAMICS OF ORGANIC POLLUTANTS IN SOIL FOLLOWING THE SPREADING OF SLUDGE-GREEN WASTE COMPOSTS

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EXECUTIVE SUMMARY

The aim of this work was to evaluate our COP-Soil model derived from the COP-Compost model (Zhang et al., 2012; Lashermes et al., submitted), using experimental data that described the evolution of carbon from organic matter (OM) and organic pollutants (OPs) after the incorporation of composts into soil. We used the results of experiments where sludge-green waste composts, containing residues of ^{14}C-fluoranthene, ^{14}C-nonylphenol, ^{14}C-glyphosate and ^{14}C-linear alkylbenzene sulfonate from composting treatment (Lashermes et al., 2012a), were mixed to soil and incubated for 140 days under controlled aerated conditions (Haudin et al., in preparation). Sequential extractions had been performed on soil-compost mixtures at different days. The fraction recovered though $CaCl_2$ extraction was defined as “soluble fraction”, while the one obtained through methanol or NH_4OH extraction was defined as “adsorbed fraction”. After chemical extractions, soil had been combusted and ^{14}C-CO_2 released trapped before counting by Liquid Scintillation Counting; the fraction of non-extractable residues of ^{14}C-OPs (NER) could be directly deduced. These three fractions and ^{14}C-CO_2 trapped in the NaOH solution (mineralization of ^{14}C-OPs) were used to calibrate the OP module.

Simulations presented in this work were performed with Matlab® software and its optimization toolbox for solving nonlinear least square problems. The mineralization of OM from sludge-green waste composts during the incubation with soil was well simulated with the parameters estimated by Zhang et al. (2012) for the simulation of the OM evolution during green waste composting. However, a compartment of humified OM with no degradation was added into the COP-Soil model. Due to the adsorption of soluble organic OM on soil particles, included minerals, which was different from composting environment, the compartment of soluble OM, originally assumed as biodegradable inside compost OM, was reduced to half and the remaining part was included into a compartment of slow degradability. The parameters estimated for OP module were the rates of NER formation Fr, the rate of OP degradation (dr) and the sorption coefficient (Kd) that was treated separately in the different runs. Using some relationships established between the coefficients of adsorption, Kd, for OPs and the different organic carbon (OC) compartments and coupling the OP module with the OC one (Run D) produced the best simulation. The toxic effects of OPs and the possible related inhibition of microbial activity were not included in the COP-Soil model and could be considered in a future work.

1 INTRODUCTION

There has been a considerable Europe-wide increase in recent years of the treatment of organic waste by composting. However, composts may contain organic pollutants (OPs) like surfactants, polycyclic aromatic hydrocarbons (Brändli et al., 2005) and pesticides. The characterization of their content and their availability in the final composts are of paramount importance when estimating the environmental impacts of recycling practices on agricultural soils (Bright and Healey, 2003). The COP-Compost model was developed by Zhang et al. (2012) and Lashermes (submitted) to simulate the dynamics of organic matter (OM) and OPs during composting. The objective of this study was to extend our COP-Compost model to soil in order to simulate the evolution of OPs availability following composts application into soil.

The new model called COP-Soil was used to simulate the evolution of carbon from OM and OPs after the incorporation of composts into soil. The model comprises two modules: a first one describing the evolution of organic carbon (OC) and a second one describing the fate of OPs. The OP module was calibrated for four OPs:

a polycyclic aromatic hydrocarbon (fluoranthene), two surfactants (4-n-nonylphenol – NP, and a linear alkylbenzene sulfonate – LAS) and an herbicide (glyphosate). This calibration was performed using data from experiments where soil was incubated with composts containing residues of ^{14}C-labeled OPs (Lashermes et al., 2012a; Haudin et al., in preparation).

2 DESCRIPTION OF THE COP-SOIL MODEL

The structure of the COP-Soil model is presented in Figure 1. The OC and OP modules can be used separately or coupled. The model was programmed in Matlab® language (The Mathwork, USA).

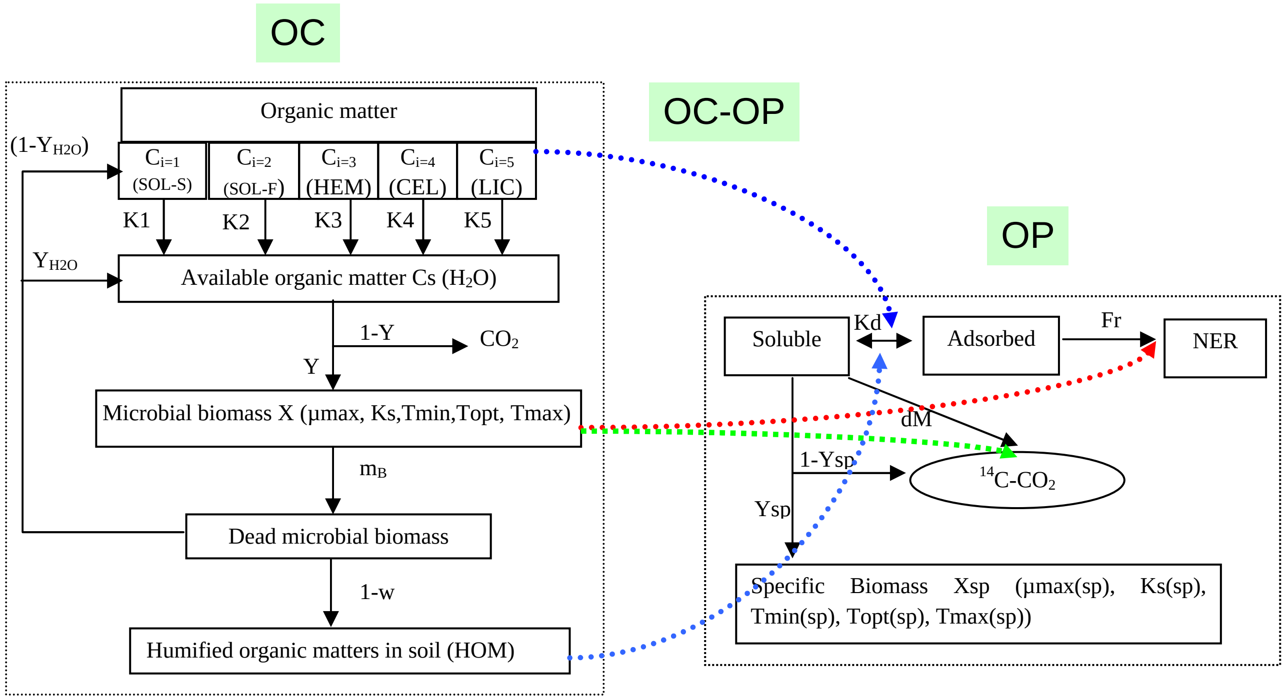

FIGURE 1 **General scheme of the COP-SOIL model.**

2.1 Organic carbon module

The schema of the OC module is taken from Zhang et al. (2012) and Lashermes et al. (submitted). However, the COP-Soil model dealt with soil environment, which was different from the COP-Compost model. Therefore, a humified OM, coming from dead microbial biomass, was added into the COP-Soil model.

2.2 Organic pollutant module

In the OP module (taken from Lashermes et al., submitted), a specific biomass was added. The growth of the specific microbial biomass and the assimilation of the soluble OPs were simulated using Monod kinetics, modulated by the temperature growth-limiting function proposed by Rosso et al. (1993).

2.3 Model coupling OC and OP modules

Three hypotheses were proposed for the coupling of OC and OP modules:

- **Hypothesis 1**: During soil incubation, the OP sorption coefficient *Kd* was assumed to be linked to the different OC compartments. This hypothesis was used successfully to evaluate the sorption capacity of plant residues with respect to herbicides, based on their biochemical fractions such as cellulose, hydrosoluble and lipid compounds (Gaillardon et al., 1983). Moreover, the biochemical nature of organic substrates strongly influenced the degree of OP sorption (Benoit et al., 1996) and the OP sorption capacities of lignin were found to be markedly higher than those of cellulose (Xing et al., 1994).
- **Hypothesis 2**: Most OPs were assumed to be degraded by microorganisms through co-metabolism during soil

incubation, the microorganisms using compost OM as a primary energy source.

- The level of OP degradation was therefore supposed to depend on the potential degradability of the chemicals and on the overall microbial activity driven by OM decomposition (Laine et al., 1997).
- **Hypothesis 3**: The formation of non-extractable residues (NER) from adsorbed OPs was assumed to depend on the chemical reactivity of the OPs and overall microbial activity. Indeed, the influence of microbial activity on NER formation was reported by Benoit & Barriuso (1997) and Kästner et al. (1999). The parent chemicals, and the metabolites formed from their degradation, may be incorporated into growing biomass (Barriuso et al., 2008) or linked to OM after oxidation reactions catalyzed by extracellular enzymes (Gevao et al., 2000).

3 DATA ACQUISITION FOR MODEL CALIBRATION AND EVALUATION

The soil-compost mixtures were incubated under controlled aerated conditions (Haudin et al., in preparation) and the experimental results were used for the calibration of the OC and OP modules. The composts used in these experiments had been obtained in a previous work (Lashermes et al., 2012a), where the fate of four ^{14}C-OPs ([methyl-^{14}C] N-(phosphonomethyl) glycine or GLY; [3C-ring-^{14}C] fluoranthene or FLT; [U-ring-^{14}C] 4-n-nonylphenol or NP; [U-ring-^{14}C] sodium linear dodecylbenzene sulfonate or LAS) initially brought to a same initial mixture of wastes (wastewater sludge and green wastes) had been studied during the composting. The mineralization of OM and the degradation of ^{14}C-residues of OPs were followed during the incubation of soil-compost mixtures over a period of 140 days.

The CO_2 released from the mineralization of compost-OC and the biochemical fractions of composts at day 0 (Lashermes et al., 2012b) were used to calibrate the OC module. Due to the adsorption of soluble OM on soil particles (included minerals), which was different from composting environment, the compartment of soluble OM in hot water (H_2O) at day 0, originally assumed as biodegradable inside compost OM, was reduced to half and the remaining part was included into a compartment of slow degradability (SOL-S). According to Zhang et al (2012), SOL-F fraction was near 0 at the end of composting, so SOL-F was assumed to be equal to 0 at the beginning of the soil incubation.

Sequential extractions had been performed on soil-compost mixtures at days 0, 14 and 140 (Haudin et al., in preparation). The fraction recovered though $CaCl_2$ extraction was defined as “soluble fraction”, while the one obtained through methanol extraction for FLT, LAS and NP or through NH_4OH extraction for GLY was defined as “adsorbed fraction”. After chemical extractions, soil had been combusted and ^{14}C-CO_2 released trapped before counting by Liquid Scintillation Counting; the fraction of non-extractable residues of ^{14}C-OPs (NER) could be directly deduced. These three fractions and ^{14}C-CO_2 trapped in the NaOH solution (mineralization of ^{14}C-OPs) were used to calibrate the OP module.

The sorption coefficients (*Kd*) were assessed for each OP from batch adsorption equilibrium tests with fresh composts and soil separately. They were calculated by weighting the individual sorption coefficients by the percentage of total OC attributed to soil and compost in the mixture. The *Kd* of each OP was also calculated from the ratio of the adsorbed fraction to the soluble fraction.

4 MODEL CALIBRATION AND EVALUATION

To run the OC module, 14 parameters were necessary. Twelve fixed parameters were obtained from Zhang et al (2012) and two (K_1 and w) were optimized by inverse modeling. The objective function was the same as in Zhang et al (2012).

The parameters of the OP module (see Table 1) were optimized for each experiment. The total root mean square error (*RMSE-OPtot*) was defined as the sum of all the *RMSE-OP (j)* calculated between the experimental and simulated values of the *j* variables (*j*=1-4 for the soluble and adsorbed fractions, NER and CO_2, respectively).

Runs A and B were performed with the OP module only, so that the evolution of OC would have no influence on the parameters; the OP and OC modules were coupled in runs C and D. During all runs, the rates of NER formation (*Fr* for the OP module alone and *Frc* and *Frb* for the coupled model) and the rate of OP degradation (*dr*) were optimized, while the sorption coefficient (*Kd*) was treated separately in the different runs:

- In Run A (without coupling) and Run C (with coupling), experimental *Kd* values were used as fixed values and *Kd* parameters were not calibrated.
- In Run B, *Kd* was optimized for each OP.
- In Run D, the real *Kd* during soil incubation was linked with OC pool values for each OP.

The sum of *RMSE-OPtot* obtained in a run was multiplied by the number of parameters required estimation in order to evaluate the overall performance of the simulations. The best run was the one that produced the lowest weighted sum of *RMSE-OPtot*. The objective was to limit the number of parameters required estimation.

5 RESULTS AND DISCUSSION

5.1 Calibration of the OC module

The mineralization of total OC from composts was well simulated when initial microbial biomass was 0.88 g C in every 100 g C brought into the soil by composts, with RMSE values of 8.50. Lower initial microbial biomass (for example 0.05% of initial total OC) under-estimated the CO_2 release at the beginning of soil incubation and over-estimated it in the middle of the incubation period.

5.2 Calibration of the OP module

5.2.1 Comparison of the different simulation hypotheses (Runs A to D)

The simulations generally improved from Run A to Run D, as shown by the decrease in the sum of *RMSE-OPtot* (Table 1). Simulation of runs D with the experimental results is presented in Figure 2.

Coupling with the OC module improved the simulation with marked decrease in the sum of *RMSE-OPtot* between Runs A (without coupling) and C (with coupling); both runs used experimental *Kd* as fixed values for the *Kd* parameter, while *Fr* and *dr* were optimized. For Runs A and C, the evolution of OP speciation was the best simulated for NP (average *RMSE-OPtot* of 78.8 and 49.4, respectively) and LAS gave the poorest fits (average *RMSE-OPtot* of 142.1 and 101.5, respectively), probably because the experimental *Kd* was too high.

When optimizing the *Kd* parameter (Run B), the optimized *Kd* values were five times as low as the experimental values for LAS and the simulated values of soluble LAS matched the experimental observations. This was also the case for GLY-1, while for NP, GLY-2 and GLY-3, a small difference was observed between the experimental *Kd* (used in Runs A and C) and the optimized *Kd* (Run B). For the surfactant LAS, the *Kd* calculated for the soil-compost mixture from experimental *Kd* measured in batch was too high when compared with the experimental results of the water extraction of OP from OP speciation. Lashermes et al. (2012) found similar results

The link of *Kd* parameters with organic carbon pools (Run D) further markedly improved the simulation when compared with Run C. The LAS was the best simulated OPs (average *RMSE-OPtot* of 8.4) and GLY gave the poorest fits (average *RMSE-OPtot* of 21.3).

Run	Model coupling		GLY			LAS			NP			Sum	Weighted sum
			GLY-1	GLY-2	GLY-3	LAS-1	LAS-2	LAS-3	NP-1	NP-2	NP-3		
A,B,C,D		NER0	33.01	50.32	52.46	75.92	61.06	68.77	90.27	87.99	87.56		
A Experimental Kd	no	Kd	18.70	18.70	18.70	13.46	13.46	13.46	7.760	7.760	7.760		
		Fr	0.0017	0.0008	0.0011	-0.0027	0.0326	0.0449	-0.0064	0.0025	-0.0021		
		dr	0.0890	0.0702	0.0746	1.4265	1.1791	0.7575	0.0679	0.0373	0.0469		
		RMSE-OP_{tot}	156.6	51.5	50.6	150.8	122.0	153.5	92.6	76.5	67.3	921.4	1842.8
B Optimization of Kd	no	Kd	2.26	19.73	17.84	1.802	2.438	2.548	5.479	6.882	6.301		
		Fr	0.0010	0.0008	0.0015	-0.0071	0.0341	0.0501	-0.0067	0.0043	-0.0023		
		dr	0.0136	0.0736	0.0730	0.2225	0.2476	0.1733	0.0503	0.0366	0.0388		
		RMSE-OP_{tot}	55.9	47.3	47.5	78.9	69.8	105.8	81.2	71.2	49.5	607.2	1821.7
C Experimental Kd	yes	Kd	18.70	18.70	18.70	13.46	13.46	13.46	7.760	7.760	7.760		
		Frc	-0.0056	-0.0055	-0.0065	-0.0327	-0.0290	-0.0412	-0.0057	-0.0114	-0.0077		
		Frb	0.0124	0.0110	0.0150	0.0266	0.0504	0.0742	0.0000	0.0220	0.0131		
		dr	0.1530	0.1198	0.1362	1.1506	0.9377	0.6294	0.1371	0.0656	0.0947		
		RMSE-OP_{tot}	141.9	31.8	36.3	111.6	100.1	92.7	53.5	55.8	38.9	662.6	1987.7
D Kd Link	yes	Frc	-0.0033	-0.0039	-0.0045	-0.0184	-0.0422	-0.0367	-0.0067	-0.0085	-0.0039		
		Frb	0.0092	0.0086	0.0118	0.0048	0.0725	0.0836	0.0000	0.0182	0.0059		
		dr	0.0241	0.1076	0.1043	0.1417	0.1195	0.0825	0.0895	0.0511	0.0570		
		RMSE-OP_{tot}	22.0	19.9	22.0	8.5	7.3	9.4	10.2	14.6	9.4	123.5	370.4

TABLE 1 Parameter values and evaluation statistics of OP simulations of Runs A, B, C, and D for three OPs

5.2.2 Selection of the optimum run to calibrate the model

Run D appeared to produce the most efficient simulations. Indeed, when requesting the optimization of only three parameters (*dr*, *Frc* and *Frb*), it produced the lowest sum and weighted sum of *RMSE-OPtot*. The simulated amounts of soluble and adsorbed fractions, NER and mineralized OPs with Run D accounted for more than 86% of the variability of the corresponding experimental values, thus testifying that the COP-Soil model was able to predict satisfactorily the experimental behavior of each OP studied. The best simulated pools were the soluble and adsorbed fractions, with average E_f values of 1.00 ± 0.00 and 0.99 ± 0.01, respectively. The simulations also agreed with the experimental evolution of mineralized ^{14}C-CO$_2$, with an average E_f of 0.96 ± 0.01. The simulations also agreed with the experimental evolution of NER, with an average E_f of 0.81 ± 0.04.

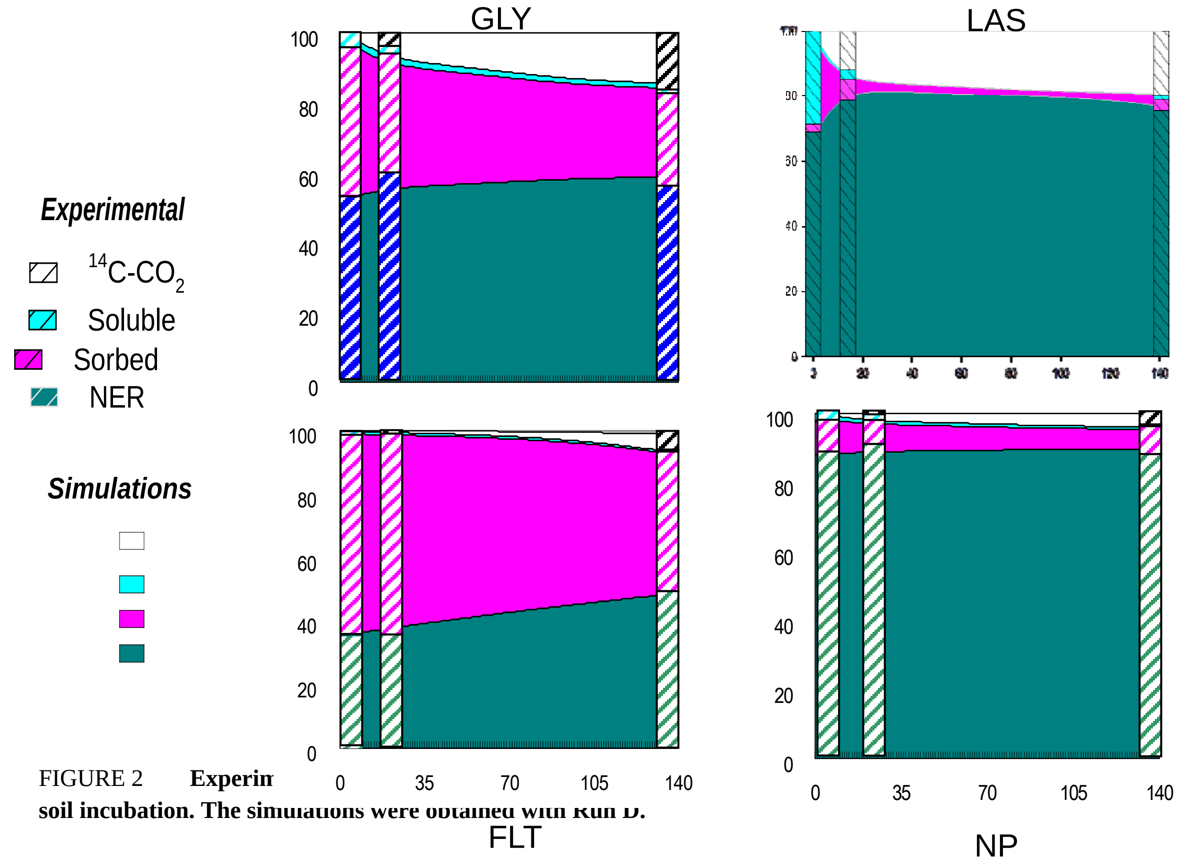

FIGURE 2 **Experin** **uring soil incubation. The simulations were obtained with Run D.**

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(169) MODELLING THE IMPACT OF URBAN COMPOST APPLICATION ON NITROGEN DYNAMICS IN A CULTIVATED SOIL

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EXECUTIVE SUMMARY

Urban compost use in agriculture has since long been seen as a mean to reverse the decline of soil organic matter and to improve soil physical conditions, leading to both agronomic and environmental benefits. However, the application of urban compost to soil affects nitrogen dynamics in soil through the release of mineral nitrogen that has to be considered in plant fertilization to avoid nitrate leaching and ground water contamination related to excessive or unbalanced supply of nutrients. The need of realistic site specific and local studies still exists for developing sustainable urban compost application practices, to provide adequate nitrogen for crop growth, and minimise leaching of nitrogen into groundwater. This research aimed at evaluating the effect of repeated application of three urban composts on the dynamics of nitrogen (N) in a cultivated loamy soil and to compare these treatments with farmyard manure application and with a control treatment without any amendment application. The dynamics of nitrogen was evaluated by sampling destructively the soil and by measuring its mineral nitrogen content. The deterministic model PASTIS, which accounts for and links transport processes in soil, crop nutrient uptake and carbon and nitrogen transformations in soil, was used to describe and predict nitrogen balance in a soil-plant system.
Experimental results showed that accumulation of mineral N were significantly higher in the amended treatments than in the control treatment due the mineralization of organic N brought by the amendments. PASTIS gave correct predictions of nitrogen dynamics in bare and cultivated plots. Furthermore, simulations showed that nitrogen availability of urban composts, from previous and last applications, was predominantly driven by: (1) the quality of organic matter of amendment (2) mineral-nitrogen content and (3) cropping conditions. Composts with high biodegradability presented higher nitrogen release the year following their application, while more stable composts with low biodegradability allowed availability of N after several years of compost application. Soil mineral content had increased in all treatments that received organic amendment which increased N plant uptake from 60 to 118 Kg N.ha^{-1} during two years compared to soil without amendment. Simulated N fluxes showed that application of organic amendments increased nitrogen leaching from 6 to 17 Kg N. ha^{-1} for two years compare to control treatments with 30% of the leaching that occurred during the first winter period following amendment application when the soil was kept bared and 40 % of the losses occurred during the dormancy period of winter wheat.

1. INTRODUCTION

1.1 Background

The application of urban composts affects nitrogen (N) dynamics in cultivated soil through the progressive release of mineralized N from organic forms of N. Nitrogen balance in the soil- plant-water system in case of application of urban composts is not easy to quantify for several reasons **(Amlinger et al., 2003)**. First, there is a lack of standard methods to assess N release from compost. Second, the N dynamics in soil after compost incorporation can be affected by environmental conditions (e.g. soil type, climatic conditions) and management practices (e.g. rate and frequency of compost application, crop rotation,...) There is still a need of long-term experiments to study the effect of repeated compost applications on N dynamics (**Hansen et al., 2006**) in order to better know the dynamics of compost N release. This will make possible a better management of compost application and increase the efficient use of N by crops and minimise N leaching into groundwater.

1.2 Research objectives

Our study investigated the performance of different types of urban composts based on their N availability for crops and potential groundwater contamination through mineral N leaching. The N dynamics after compost application was simulated with the soil-plant model PASTIS (**Garnier et al. 2003**) based on data from a long term field trial where different urban composts are compared. The objectives were (i) to test the accuracy of the PASTIS model at simulating the N balance at field scale after repeated compost application, considering various compost types, and using the simulated results and, (ii) to investigate processes and factors that control N cycling in soil, in order to understand how compost quality influences the N dynamic at short-term, immediately after compost application, and at longer term, after 10 years of repeated compost application.

2. METHODOLOGY

1.3 Field experiment

A long-term field experiment located in Feucherolles (Yvelines, France) has been carried out since 1998 (INRA-VERI collaboration). Three different composts are applied: (i) a biowaste compost (BIO) resulting from the co-composting of green wastes and source-separated organic fraction of municipal wastes; (ii) a municipal solid waste compost (MSW) made from mechanically separated organic fractions after selective collection of dry and clean packaging; and (iii) a co-compost of green wastes, wood chips and sewage sludges (GWS). The 3 composts are compared to a farmyard manure (FYM), as reference amendment and to a control treatment without organic application (CONT). The field experiment includes four replicate blocks of the 5 organic treatments randomly distributed within each block and is cultivated with a wheat (Triticum aestivum L.) - maize (Zea mays L.) succession. Composts and manure have been applied on wheat stubbles in early September 1998, 2000, 2002, 2004, 2006. In 2007, barley was sown instead of maize, due to the presence of maize rootworm (*Diabrotica virgifera*). Consecutively additional organic application was performed in September 2007 on barley stubbles. All organic matters are applied based on the same C rate, with an average of 4 tons organic C per hectare at each application. Wheat residues are exported while maize residues are incorporated into the soil after harvest.

1.4 Field and laboratory measurements

Our study was conducted during the 2 years after the 6th application of composts and manure, from September 2007 to September 2009. The kinetics of C and N mineralization were measured for soil organic matter and all organic amendments during incubation of soils sampled in the ploughed layer of the different treatments and control soil-organic amendment mixtures in laboratory conditions. The dynamics of mineral nitrogen was measured at the field scale over the 2 years following amendment application in 2007 (Sept. 2007 - Sept. 2009), by periodic soil sampling under bare or cultivated conditions. Soil temperature was recorded daily from 0 to 160 cm soil depths with temperature sensors. Tensiometers and time domain reflectometry (TDR) probes have been installed at 7 depths (20, 40, 60, 80, 100, 130, and 160 cm) within five plots, one per treatment, to monitor soil water pressure head and water content. Soil water potential and water content measurements have been carried out three times per week from November 2007 to October 2008.

1.5 Model description

The mechanistic PASTIS model includes three submodels describing: i) transport processes, ii) crop growth and nutrient uptake and iii) carbon and nitrogen transformations in soil (CANTIS). Hydraulic parameters of the van Genuchten's functions were estimated from field measurement of soil water content and pressure head data using an inverse procedure with the water flow part of PASTIS. Soil dispersivity was determined by fitting convection-dispersion equation (CDE) to resident bromide concentrations profiles measured during a tracer study carried out between October 2007 and June 2008. All heat flow parameters were taken from Findeling (2003). All maize and wheat related parameters were derived from the database of the crop model STICS **(Brisson et al., 2003).** Biological parameters were obtained by fitting the submodel CANTIS to the results of laboratory incubations; mineralization kinetics of C and N from incubations of soils and soil-organic amendment mixtures.

3. RESULTS AND DISCUSSION

Detailed results of physico-chemical properties of the BIO, GWS, MSW and FYM amendments applied in 2007 are presented in **Table 1**. The four amendments significantly differed for their organic carbon content, organic and mineral N content, C/ N ratio and pH. These differences could be attributed to their distinct input materials and levels of compost maturity.

The MSW compost was found to be the less stabilized amendment ($P< 0.05$), because it was the least mature compost (no maturation period during composting process, vs. 8 and 12 weeks for the other composts). Moreover, MSW had the largest proportion of easily biodegradable cellulose and the lowest proportion of more recalcitrant lignin compared to the other products **(Table 1)**. GWS, BIO and FYM amendments showed slow mineral N release during the entire incubation period. Net N mineralization corresponded to 3, 3 and 4 % of added organic N for GWS, FYM and BIO, respectively. The MSW compost showed a temporary net immobilization of mineral N, during the first 14 days of incubation, related with its high C/N (23.9), and high C/N of the cellulose fraction (53). After 14 days of incubation, net positive N mineralization occurred again for MSW compost. Net N mineralization, between the 14 and the end of incubation (91 days) was significantly higher in the soil-MSW compost mixture than the others amendments and corresponded to 26 % of added organic N for MSW compost.

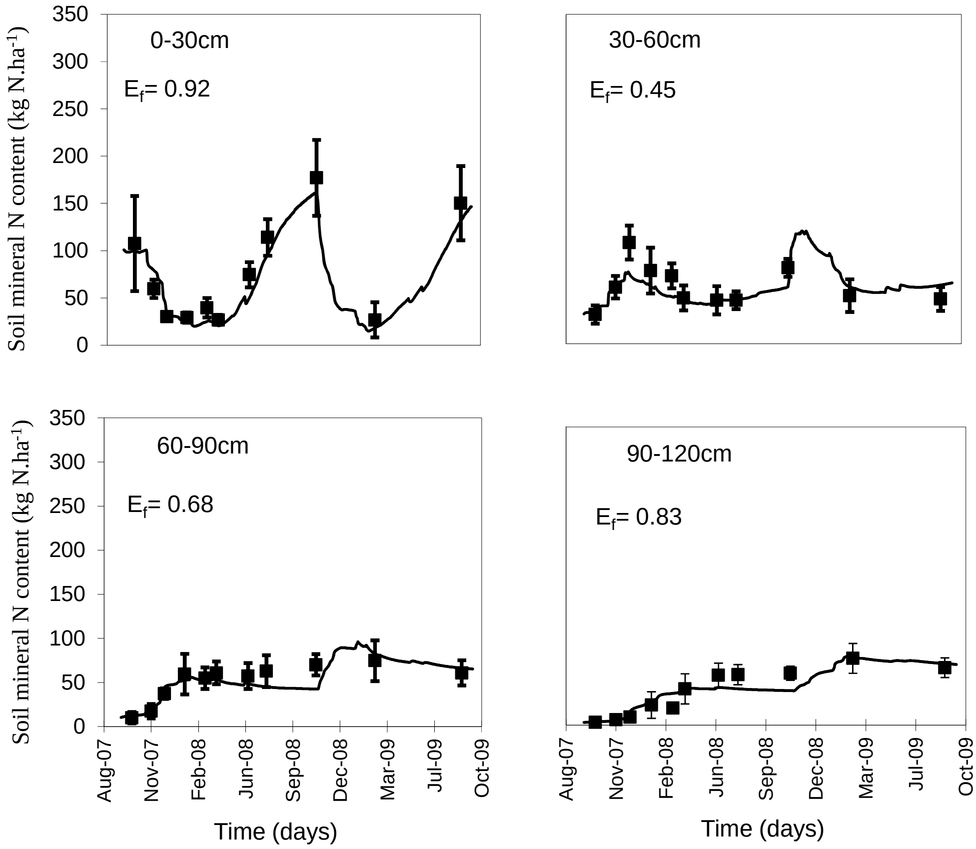
TABLE 1 Physico-chemical and biochemical characteristics of organic amendments applied in September 2007 (mean of 3 replicates)

		GWS	MSW	BIO	FYM
Amount applied	t dw.ha^{-1}	16.6 (±2.9)	10.5(±2.3)	16.2(±5.1)	13.3 (±2.8)
Corg†	g.kg^{-1}dw	238.3 (±3.8)c*	359.3 (±6.4)a	228.7 (±5.0)c	274.7 (±4.2)b
Norg	g.kg^{-1}dw	23.3 (±0.5)ab	15.07 (±0.7)c	22.5 (±0.2)a	20.8 (±0.7)b
Mineral N	g.kg^{-1}dw	4.0 (±0.2)b	7.0 (±0.1)a	3.3 (±0.1)c	1.4 (±0.0)d
C/N		10.2 (±0.4)c	23.9 (±1.4)a	10.2 (±0.3)c	13.2 (±0.3)b
pH, H_2O		7.4 (±0.0)bc	6.99 (±0.4)c	7.6 (±0.0)b	9.29 (±0.0)a
Mineralized Carbon††,	%Corg	16	56	10	15
Composting process:					
Fermentation phase,	days	42	30	35	
Maturation phase,	days	90	0	60	
Soluble fraction	%Corg	55	35	46	63
	C/N	7	13	7	11
Hemicellulose –like fraction	%Corg	4	7	8	0
	C/N	17	10	8	0
Cellulose –like fraction	%Corg	20	45	15	17
	C/N	28	53	17	48
Lignin–like fraction	%Corg	21	14	31	20
	C/N	18	27	18	14

* Different letters between composts for the same variable indicate significant differences at 5 %
† Corg, organic carbon content; Norg, organic N content ; C/N, organic ratio
††he excess mineralized carbon after 91 days in the soil-organic amendment mixtures was expressed as a percentage of the compost organic C applied during incubation (after subtracting mineralized C of the control treatment)

Experimental results showed that accumulation of mineral N was significantly higher in the amended treatments than in the control treatment. The differences between amended and control treatment could be explained by both the mineralization of organic N brought by the amendments and by the mineral N content of amendments. The mineral N evolution in soils was well simulated by PASTIS in the GSW, FYM, BIO and Control treatments as shown by the high model's efficiencies calculated in bare and cultivated soils. The examples of GWS treatment in bare conditions are presented in **Figure 1.**

FIGURE 1 **Simulated (lines) and observed (symbols, ± standard deviation) soil mineral nitrogen content (N) in the treatment amended with the green waste and sludge compost (GWS) and kept uncropped in the different layers, Ef is the model efficiency coefficient.**



Noticeable differences occurred during the year after the application of MSW compost and the model underestimated soil mineral N content of all soil layers**.** The overestimation by the model of N immobilization in the 0-30 cm soil layer during the initial stages of MSW decomposition in September 2007 was likely to be the main reason of the underestimate of N leaching into lower depths during winter**.** We assumed that the limited diffusion of mineral N in soil may have delayed the decomposition of MSW compost limited by necessary available mineral N due to its high C/N.

In a first step, the mineral Nitrogen (N) balance was calculated from September 2007 to September 2009 by taking into account mineral N input (from fertilizers and amendment) and N outputs, as obtained by PASTIS in the various treatments with and without the last amendment application in September 2007. The N-NH_4 losses through ammonia (NH3) volatilization within the first 24 h after amendment application were measured using field-based wind tunnel trials. It reached 4, 16, 18 and 53 % of N-NH4 applied for BIO, GWS, MSW and FYM, respectively, that were removed from the N balance. In a second step, the effect of the previous and last applications on N mineralization, N crop uptake and N leaching were analyzed (Tables 2 and 3) using mineral N balance calculated by PASTIS in step 1.

The increase of net mineralized N compared to control treatment varied from 35 to 95 kg N.ha^{-1} and corresponded to the lower value of 2.8 % of total N applied from previous applications of MSW compost while it was between 4.2 and 6.3 % for the other organic amendments (Table 2, for bared soil). On the contrary, the MSW compost presented the highest proportion of mineralized organic N during the 2 years following its application with 28 kg N.ha^{-1} in bare soil, corresponding to 18% of applied organic N while the mineralized N from less biodegradable amendments was between 4 and 13 kg N.ha^{-1} corresponding to 1.1 to 3.7% of total organic N applied. The organic amendments with low mineralization rate immediately after application (GSW, FYM, BIO) resulted in a larger mineralization of soil organic N after several applications because they contributed to a larger increase of the organic nitrogen pool of soil than the MSW compost characterized by a high biodegradability which contributed to a higher short term release of mineral N.

The net mineralized N was affected by the crop presence.

The crop presence reduced the mineralized N after the 6 previous applications (for example: from 85 to 69 kgN.ha^{-1} for GWS treatment in Table 2) and after the last amendment application (for example, from 28 to 16 kgN.ha^{-1} for MSW treatment). This was related to the modification of moisture condition in soil when plants were present.

Under cultivated conditions, soil water content decreased largely due to the plant uptake; therefore the N mineralization was reduced. Nitrogen immobilization during maize crop residue decomposition also contributed to the decrease of net N mineralisation.

TABLE 2 **Additional net N mineralization in soil due to the previous applications and due to the last applications during the 2 years (Sept. 2007- Sept. 2009) after the last application. N mineral release was assessed in the absence (bared soil) and presence of culture (cultivated soil).**

		GWS	MSW	BIO	FYM
		6 previous applications			
Total N applied	**kg N/ha**	2033	1236	1511	1728
Bared soil: Mineralized N	**kg N/ha**	85	35	95	81
Bared soil: Mineralized N	**% total N**	4.2	2.8	6.3	4.7
Cultivated soil: Mineralized N	**kg N/ha**	69	37	79	65
Cultivated soil: Mineralized N	**% total N**	3.4	3.0	5.2	3.8
		Last application (2007)			
Organic N applied	**kg N/ha**	352	155	352	266
Mineral N applied	**kg N/ha**	55	61	51	10
Bared soil: Mineralized N	**kg N/ha**	4	28	13	5
Bared soil: Mineralized N	**% organic N**	1.1	18.0	3.7	1.8
Bared soil: Available N	**% total N**	14.5	41.1	15.5	5.4
Cultivated soil: mineralized N	**kg N/ha**	0	16	8	1
Cultivated soil: mineralized N	**% organic N**	0	10.3	2.0	0.3
Cultivated soil: Available N	**% total N**	13.4	35.6	14.8	3.9

The simulations with PASTIS of N crop uptake (Table 3) showed that: i) the previous amendment applications on soil organic N made possible the increase of N crop uptake comparing to the control treatments from 13 to 80 kgN.ha^{-1} with the following order: MSW<BIO<GWS=FYM, and ii) the last amendment application additionally increased the N crop uptake compare to previously from 9 to 47 kgN.ha^{-1} with the following order FYM<GWS<BIO<MSW. The crop N recovery due to the residual effect of previous organic amendment applications (from 1998 to 2006) represented 1-5 % of total N applied through amendment application, the lowest proportion being calculated from the MSW compost. The response of N crop recovery due to the last amendment application ranged from 9 to 21 % of the N applied with the largest proportion found for the MSW compost.

The application of organic amendments increased N losses through leaching up to 12 Kg N ha^{-1} in comparison with the control treatment without considering the last organic amendment application (Table 3) with the following order MSW<BIO<GWS<FYM. Such losses represented less than 1 % of total input of N amendment applied from 1998 to 2006. The N losses by leaching related to the last application were low for FYM (1%) and higher for the other treatments especially for MSW (3% of N leaching). It was mainly related to the high amount of mineral N in this amendment and its ability to mineralize nitrogen rapidly. We calculated that 30% of the N losses by leaching occurred just after amendment application from September 2007 to April 2008 when the soil was kept bared and 40 % of the losses occurred from October 2008 to April 2009 when the field was cropped with winter wheat. The wheat crop used very little N during winter dormancy and active N uptake by crop only begun when the wheat dormancy was over in early march. Soil mineral nitrogen in excess during the dormancy period of winter wheat (October-late February) was subject to leaching losses.

From tables 2and 3, we can conclude that the amendments did not have the same effects just after their applications and after repeated application of several years. The last application increased the crop uptake and N leached also through the mineral N applied with the organic amendments because the mineralized N from the last application was not high enough to increase these two variables.

MSW, with its rapid degradability and mineral nitrogen content, has delivered rapidly nitrogen at short term just after its application that was used by plants and also submitted to leaching the two following years. At the opposite, FYM and GWS, as slower degradable, continuously delivered nitrogen and showed higher impact on plant uptake at long term.

TABLE 3 **Additional N plant uptake by maize and wheat and N losses (evaluated at 2 m depth) compared to the control treatment due to the last and the previous applications are calculated by the model over the 2 years (Sept. 2007 - Sept. 2009). During, this period the soil was cultivated with maize from April to October 2008 and wheat from October 2008 to August 2009.**

	GWS	MSW	BIO	FYM
	6 previous applications			
Total N applied (Kg N/ha)	**2033**	**1236**	**1511**	**1728**
Crop N uptake (Kg N/ha)	**80**	**13**	**68**	**80**
Crop N recovery (%)	**4**	**1**	**4**	**5**
N leached (Kg N/ha)	**10**	**0**	**2**	**12**
N leached (% of N applied)	**0.5**	**0**	**0.1**	**1**
	Last application (2007)			
Total N applied (Kg N/ha)	**407**	**216**	**403**	**276**
Crop N uptake (Kg N/ha)	**38**	**47**	**44**	**9**
Crop N recovery (%)	**9**	**21**	**11**	**3**
N leached (Kg N/ha)	**7**	**6**	**7**	**1**
N leached (% of N applied)	**2**	**3**	**2**	**0.3**

4. CONCLUSIONS

The objective of our work was first to evaluate the ability of the PASTIS model to reproduce the N dynamic in soil after the application of 4 types of organic amendments in a cultivated soil. The adaptation of the model was necessary to take into account the low availability of N for compost with high C/N such as the MSW compost. The second objective was to evaluate the effect of repeated previous applications of composts on N availability in soil compared to recently applied compost. The repeated application of organic amendments increased the total N mineralization, plant uptake and leaching compare to control treatment. The MSW treatment presented the largest N availability immediately after application because of its high degradability and high mineral N content. On the other hand, the repeated applications of the other more stabilized organic amendments led to larger N release due to increase soil organic N.

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Session 3

(54) THE SWEDISH VOLUNTARY AGREEMENT FOR CONTROL OF METHANE EMISSIONS FROM BIOGAS PLANTS

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EXECUTIVE SUMMARY

The Swedish Waste Management Association has set up a voluntary agreement for control of methane emissions from biogas and upgrading plants, starting in the year 2007. There were a number of reasons to start this system, mainly environmental (GHG emissions and odour), economical and safety issues, but as important was to show that the biogas industry acts responsibly and is pro-active regarding these issues, especially in relation to authorities. The voluntary agreement system has been reviewed once and is currently set up in two parts; internal routines for leak detection and emission measurements performed every 3 years by an external consultant. The results from the emission measurements are used to calculate annual mass emissions and with these results at hand the plant has to set specific targets for their mass emissions and show that they take measures to reach these set targets.

Leak detection results generally show that biogas plants have a few larger leaks and upgrading plants often have many, but smaller leaks in the gas equipment. When the system started it was found that many plants did not perform leak detection regularly and some did not even have access to leak detection equipment. It was also found that leaks contributed substantially to total mass emissions.

Contracted measurement consultants perform measurements and calculations of total mass emission within the system. Measurements are typically made in ventilation systems and CO_2 release. European and international measurement standard methods are being used for these measurements when applicable. Many measurement and calculation methods have had to be adapted to the varying circumstances found at plants, and these experiences have since been gathered in a Methane Measurements Handbook.

To date measurements and calculations have been performed at 18 individual biogas plants and 29 upgrading plants, many of these have also been measured more than once. The available results show that the majority of plants have total methane losses of less than 2 % (per plant) of the produced methane. A few outliers have been identified, especially during the first 3 years, with methane losses of up to some 10 %. In these cases measures have been taken already and these plants now have much lower emissions during the second round of measurements (2010 and onwards).

For upgrading plants it is clear that chemical scrubber plants have very low methane emissions compared to other techniques. Different end-of-pipe solutions are becoming common and they are generally found to be working very well taking emissions down close to zero, but at some plants they were also found to be out of order or not even in use. For water scrubber and PSA plants there is a negative relationship between the size of the plant and its relative emissions.

Current challenges for the system are how the results can be used to show sustainability criteria for biogas as a vehicle fuel and also how the system can be expanded to the waste water treatment sector. An on-going measurement challenge is how to perform measurements from open tanks and storages, where a separate study has been initiated. For this study a first phase with literature studies and interviews has already been finished and this will be followed shortly by measurements at a pilot facility (sludge storage).

1 INTRODUCTION

Biogas is formed during the anaerobic degradation of organic material. It can be produced from any material that is biodegradable and consists mainly of two gases, methane and carbon dioxide. Biogas today is primarily produced from sewage sludge, household waste and energy crops. Biogas production and use has gained increased interest over the last few decades, mainly due to its many environmental benefits. As a result the annual production is increasing. Biogas can be used as an important source of renewable energy. It can be used for heat production or heat and power production, as a raw material in industry and as a vehicle fuel. Biogas can also be used to replace natural gas in the gas distribution grid. In order to use biogas, it needs to be cleaned to a greater or lesser extent, depending on the use. Biogas to be used as a vehicle fuel, or to replace natural gas, needs a treatment whereby the carbon dioxide is separated from the methane. Removing carbon dioxide increases the energy content per volume. This is known as biogas upgrading, and several techniques are commercially available. The techniques that have been studied so far in the voluntary agreement are PSA (Pressure Swing Adsorption), water scrubbing and chemical scrubbing.

Biogas production and the use of its energy content create several environmental benefits. Replacing fossil energy leads to decreased CO_2 emissions. The environmental benefit is dependent on how the biogas is produced and used. Biogas production from manure leads to a “double” positive environmental effect. Not only does the biogas replace fossil energy, thus decreasing carbon dioxide emissions, but also the methane emission that usually occurs during manure storage is avoided. However, while biogas use does lead to decreased emissions of fossil carbon dioxide, methane is, in fact, a strong greenhouse gas and thus emission of methane has to be avoided or minimized during the process. The global warming potential for methane over a 100 year timeframe is 25 times that of carbon dioxide (IPCC, 2005).

Overall, the environmental benefits, and in particular the decrease in fossil carbon dioxide emissions, mean that a certain level of methane emissions can be tolerated while still maintaining a positive environmental effect. How high the emissions can be before the total effect becomes negative depends on the raw material used. A study has shown that for production and upgrading of biogas from manure, the methane emissions can be up to 22-26 %, while for organic waste the level is 12-17 %, and for grass, straw or beet tops 8-16 % (Börjesson and Berglund, 2003). In general though, the greatest environmental benefit is obtained when emissions are minimized. Apart from the environmental aspect, there are also other reasons to avoid methane emissions. There is, of course, an economic incentive not to lose the methane produced. A loss of methane is a loss of income. Safety is also an important consideration, as methane can form explosive mixtures with air. Plus, emissions can lead to odour problems in and around the plant. Thus, the Voluntary Agreement was initiated with the aim of quantifying and minimizing methane slip from biogas production plants and biogas upgrading plants.

1.1 Background

A Swedish study (Persson, 2003) concluded that in some cases methane emissions from biogas upgrading plant were higher than the 2% that was at that time the level generally guaranteed by the plant’s manufacturers. Another Swedish study (Gunnarsson et.al, 2005) showed small methane emissions at various points in the plant, with a total methane slip of between 0,5% and 4%. In 2007, the Voluntary Agreement was initiated (Persson et.al. 2007) in order to establish a systematic approach to quantification and minimization of methane emissions. The system for the Voluntary Agreement was then revised in 2009 (Holmgren, 2009). A more detailed description of how to measure and calculate the methane slip was later published (Holmgren, 2011). However, one remaining problem is that methane slip from surfaces such as uncovered digestate tanks can be hard to quantify and a separate study has therefore been initiated. For this study a first phase with literature studies and interviews has already been finished (Holmgren et.al, 2011) and this will be followed shortly by measurements at a pilot facility (sludge storage).

1.2 Research objectives

Initially the objectives were to gain knowledge of the size of the emissions that at the time were more or less unknown. The work has since been focused on developing a system using standardized measurement methods (published by international standardization organisations) to high extent. Knowledge from the work has been documented and used to develop standardized calculation methods. The more difficult sources of emission (such as open surfaces) are now being studied further with the aim to suggest measurement methods applicable in field measurements.

2 METHODOLOGY

For the voluntary agreement it has been decided to only include CH_4 (methane), although low concentrations of N_2O have been detected in emissions from biogas plants (Gunnarsson et.al, 2005).

There are three actions performed in the voluntary agreement, which are described in the following sections:

- Inventory of systematic emissions (section 3)
- Leak detection (section 4)
- Emission measurements and calculations (section 5)

The system boundaries for the different types of biogas plants are presented in Fig. 1-3. Only methane emissions within the following boundaries are quantified:

- Only parts of the plant of which the plant owner has control over are included
- Only parts connected to production or cleaning/upgrading of biogas are included. Thus methane emissions that arise from utilization of biogas or digestate are not included

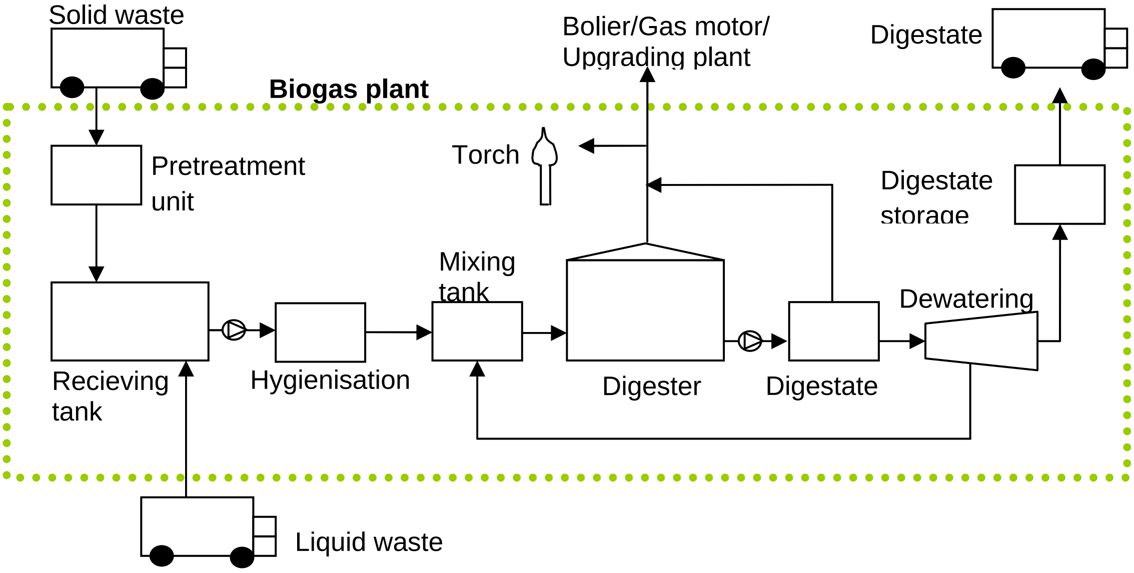

FIGURE 1 **System boundaries for a biogas plant.**

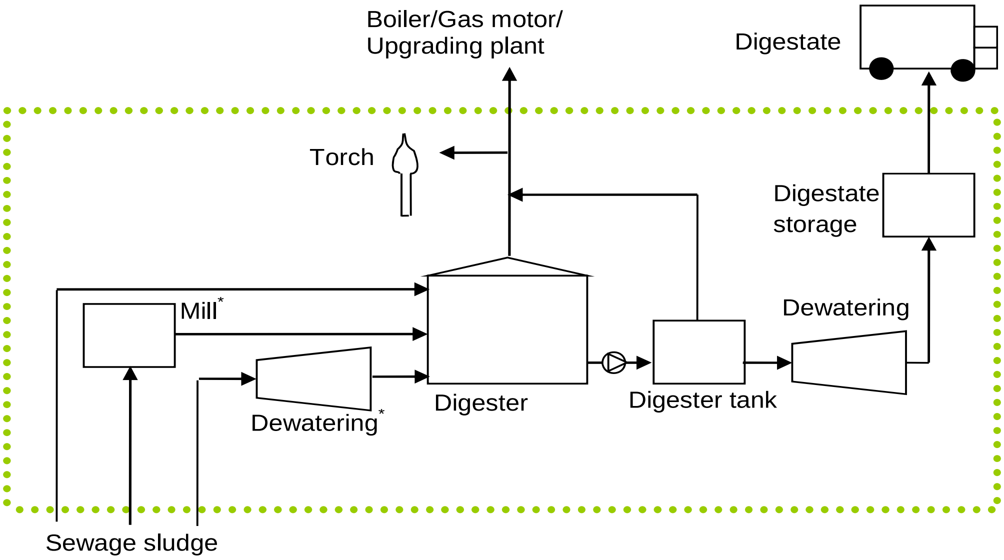

FIGURE 2 **System boundaries for a sludge digestion plant.**

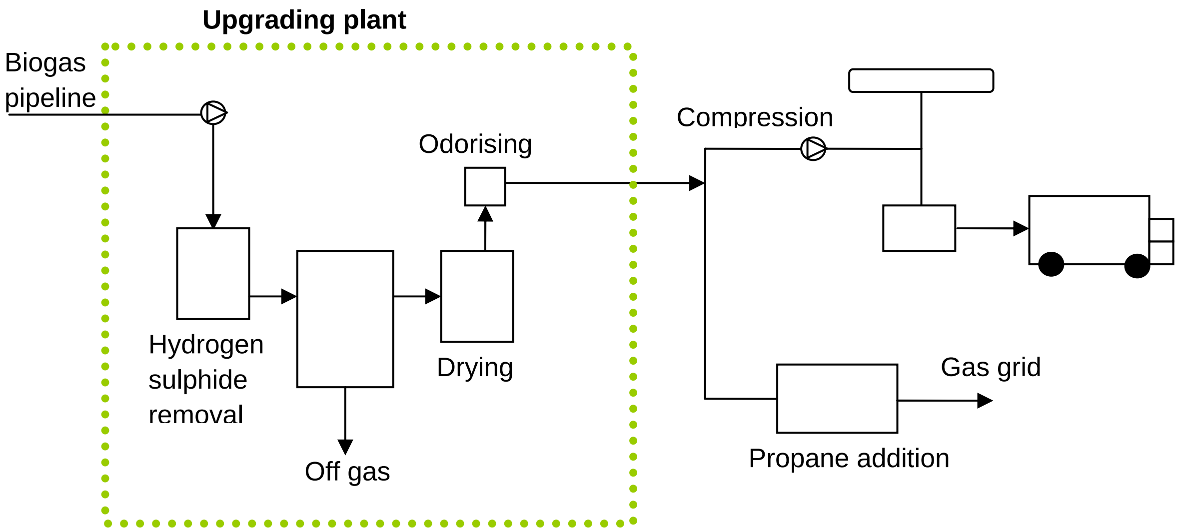

FIGURE 3 **System boundaries for a biogas upgrading plant.**

2.1 Biogas plant

The Voluntary Agreement system starts where the substrate is delivered to the plant (Fig. 1) (transport is thus not included). Even though plants can differ, all parts of the biogas production plant are included in the context of the agreement: from storage of substrate, pre-treatment processes, mixing, digestion, post-digestion and storage of digestate at the plant. The system covers stages up until the point at which the digestate is transported from the plant by truck or in a pipeline (thus emissions during transport, storage on farms or spreading are not included). For produced biogas, the system covers up until the point at which the gas is transferred to a boiler, engine, a turbine or to an upgrading plant.

2.2 Sewage sludge digestion plant

Biogas is often produced at waste water treatment plants. Treatment of waste water includes processes that are not related to biogas production and therefore only the parts of the waste water treatment plants that are directly related to biogas production are included in the Voluntary Agreement. Methane emission during the water cleaning process is thus not included in the system. These types of plants will be referred to as biogas plants below. The system of Voluntary Agreement begins when the sewage sludge enters the digester or at the point where the sewage sludge is treated before the digestion (in e.g. mills or centrifuges) and ends after the digestate storage (Fig. 2). For the digestate, the system ends when it is transported from the plant by truck or in a pipeline. The system ends when the produced biogas is used in a boiler, an engine, or a turbine, or is transferred to an upgrading plant.

2.3 Biogas upgrading plant

For the upgrading plants, the Voluntary Agreement starts when the gas enters the building containing the upgrading equipment and ends when the gas is cleaned, dried and odorised (Fig. 3). Methane emissions during transport of the upgraded biogas, compression, propane addition, gas storage or emissions at filling stations are not included.

3 INVENTORY OF SYSTEMATIC EMISSIONS

In the Voluntary Agreement, the plant's staff is required to make an inventory of systematic emissions. These shall be marked on a map or plan of the plant, which is then reviewed together with a measurement consultant. To be able to do this, staff needs a thorough knowledge of the plant. At least one of the staff members participating in the inventory shall have taken the course on the Voluntary Agreement (these are regularly organised by the Swedish Waste Association). A classification plan can be helpful in making the inventory of systematic emissions. A classification plan is mandatory for Swedish biogas plants and it describes where explosive gas mixtures can occur and thus where systematic emissions may be expected.

3.1 Results

From experience the following points of emission can be found at biogas plants and sewage sludge digesting plants:

- Central ventilation (whole or part of the plant, mechanical or natural ventilation)
- Ventilated tanks (mixing tanks, process water tanks, digestate tanks)
- Digester (systematic emissions at overflows and/or leaks collected in building ventilation on top of the tank)
- Dewatering systems (usually ventilated)
- Digestate storage (liquid/solid fraction stored on site, note system boundaries)
- Instruments for gas analysis (rotameter readings)

From experience the following points of emission can be found at biogas upgrading plants:

- Off-gas (CO_2 release)
- Central ventilation (whole or part of the plant, mechanical or natural ventilation)
- Instruments for gas analysis (rotameter readings)

4 LEAK DETECTION

Leak detection shall be performed at least once a year, but it is strongly recommended that it is performed on a monthly basis. It can be an advantage to use an external resource from time to time to avoid “domestic blindness”. A checklist shall be prepared that is used as a protocol for the leak detection and it needs to be archived for inspection. Recommended methods include traditional leak detection instruments, leak detection sprays and ocular and odour control. Every plant shall have at least one leak detection instrument available, as well as having access to leak detection spray. It is important to distinguish between leak detection instruments and gas alarms, where the former has a probe connected to be able to reach parts that are otherwise hard to access. The instruments shall be serviced and calibrated as recommended by the manufacturer.

Systematic leak detection shall be performed throughout the entire plant with a leak detection instrument and leak detection spray. The instrument is moved along potential leakage points, both at a distance from the equipment, and very close to it. Detected readings are noted in a protocol. If possible, the exact source of the leakage shall be determined and noted, and if possible the leakage shall be addressed and stopped immediately.

If careful leak detection is performed too often there is the risk that it is carried out less thoroughly with time. To overcome this, intermediate leak detection shall be done at more regular intervals, say weekly or monthly. The reading of the instrument is checked at a number of well-defined places, not too close to potentially leaking equipment, but at least 1 m away. The readings at each place is plotted on a diagram, which will enable detection of a trend. If a diagram indicates a possible leakage, careful leak detection can then be done in that area.

4.1 Results

From the results of the leak detections it could be concluded that the biogas plants in general had few, but larger leaks. Typical leakage points were relief valves and digestate storage tanks. The leak detection results showed that in general the upgrading plants had many, but smaller leaks. Typical leakage points were gas equipment parts and compressors.

5 EMISSION MEASUREMENTS

Every third year measurements are done by an external measurement consultant, during normal production conditions. The resulting methane emissions are extrapolated to a yearly basis from this measurement. To be able to make a careful quantification of emissions, measurements shall be done during a longer time and data shall be logged. The measurement shall continue until a stable signal is detected or a stable pattern is found. As a general rule, measurement shall be done during one hour at each point. Methane slip at each point is related to the total flow of methane in the plant by calculation. The slip is always related to the methane flow in the raw gas, but for upgrading plants the slip is also related to the measured total flow of upgraded methane. The emissions shall always be determined downstream of any end-of-pipe gas treatment processes, i.e. at the point of emission to air.

5.1 Methane concentration

Methane concentrations are measured by a Flame Ionization Detector (FID)-instrument equipped with a Non Methane Hydrocarbon Cutter (that filters out other hydrocarbons than methane). FID-instruments are able to measure hydrocarbons at levels from a few ppm's up to usually 100 000 ppm (10 vol.-%). The methods of measuring methane with this instrument are described in the international standard EN ISO 25140:2010. When using this method at biogas plants it is important to know if, and how, the methane measurement by the instrument is affected by carbon dioxide concentrations. For even higher concentrations of methane, gas samples are taken in bags for later analysis in a laboratory with Gas Chromatography (GC). This method is described in the international standard EN ISO 25139:2011. If the methane slip in a measurement point is lower than 0.1 % of the total amount of methane in the plant and less than 10 % of the total slip, the methane concentration can be measured with a leak detection instrument instead. The methane concentration at emission point x is denoted $C_{emis,x}$ (vol-%).

5.2 Gas flow

Measuring the flow is quite a challenge since the conditions vary between different measurement points and different plants, the measurement points are rarely prepared for flow measurements. The general method for measuring flow in a duct is Pitot tube measurements which are described in the international standard ISO 10780:1994. In this type of measurement the speed of gas is measured and the flow is then calculated by multiplying the speed by the cross section area of the duct. The flow from a ventilation opening can be determined by a sensitive hot wire instrument. For natural ventilation valves, an instrument with a low detection level is needed.

When it is technically or practically impossible to perform measurements, other methods have to be used, such as using fan data or default values. There are also methods available, published in a European standard (EN 15446:2008), which enables the user to approximate the mass flow out of leaking equipment (Holmgren, 2011). The gas flow in emission point x is denoted $Q_{emis,x}$ (Nm^3/h).

5.3 Calculations

Methane emissions are reported in relation to the total methane flow through the plant, i.e. the methane loss. In order to perform the calculations of methane loss, some data from the production and/or upgrading plant is needed (Table 1).

TABLE 1 **Plant data needed for calculations (P=Production plant, U= Upgrading plant)**

	Operand	Operation	Annual	Unit
Methane concentration, raw gas	C_{raw}	P/U	P/U	Vol-% (1h average)
Production/load, raw gas	Q_{raw}	P/U	P/U	Nm^3/h
Methane concentration, raw gas	CP_{raw}	P/U	P/U	Vol-% (1 year average)
Production/load, raw gas	P_{raw}	P/U	P/U	Nm^3/year
Methane concentration, clean gas	C_{clean}	U	U	Vol-% (1h average)
Production/load, clean gas	Q_{clean}	U	U	Nm^3/h
Methane concentration, clean gas	CP_{clean}	U	U	Vol-% (1 year average)
Production/load, clean gas	P_{clean}	U	U	Nm^3/year

All gas flows (Q) are given at standard conditions 0°C (273.15 K) and 1 atm (101.3 kPa). For a single emission point, the methane loss (%) is calculated by dividing the methane flow in the emission with the methane flow through the plant (Equation 1).

$$Loss_{raw,x} = \frac{C_{emis,x} \cdot Q_{emis,x}}{C_{raw} \cdot Q_{raw}} \cdot 100 \quad (1)$$

For upgrading plants, the methane loss (%) shall also be calculated in relation to the methane content in the cleaned gas. The reason for this is the lower measurement uncertainty for measuring the methane flow in the cleaned gas compared to the raw gas (Equation 2).

$$Loss_{clean,x} = \frac{c_{emis,x} \cdot Q_{emis,x}}{c_{clean} \cdot Q_{clean} + \sum_{i=0}^{n} c_{emis,i} \cdot Q_{emis,i}} \cdot 100 \quad (2)$$

The relative methane losses in different emission points are summed up to get the total relative loss for the plant. This calculated loss is then extrapolated to a yearly emission *Emis* (Nm^3 CH$_4$/year) by multiplication with the annual production. For an upgrading plant the total emission in also calculated based on the yearly production of cleaned methane gas, using the same equation (Equation 3).

$$Emis = CP \cdot P \cdot \frac{\sum_{i=0}^{n}(Loss_i)}{100} \quad (3)$$

In (Holmgren, 2011) a number of examples of calculations for different emission points are carefully described. Also a few special cases are studied, eg. mathematical modelling of methane loss from digestate (Hansen et.al. 2006).

5.4 Results

The first round of measurements were performed between 2007-2009 at a total of 18 production plants and 20 upgrading plants, and the results showed considerable differences in methane emissions from the plants. Since then, a further 6 upgrading plants have joined the voluntary agreement and most of all the plants have already been visited again for new measurements during the second round 2010-2012.

The results from the measurements and calculations are shown for the productions plants in Table 2 and for the upgrading plants in Table 3. The running average is the result from the measurements during the second round for most of the plants, and the result from the first round for those plants that have yet not been visited during the second round.

TABLE 2 **Results, methane losses from production plants**

	Loss$_{raw}$ (average)	**Loss$_{raw}$ (median)**	**Loss$_{raw}$ (upper quartile)**
Results, first round 2007-2009	1,6 %	0,7 %	
Running average 2007-2012	1,5 %	0,3 %	2,5 %

TABLE 3 **Results, methane losses from upgrading plants**

	Loss$_{clean}$ (average)	**Loss$_{clean}$ (median)**	**Loss$_{clean}$ (upper quartile)**
Results, first round 2007-2009	2,7 %	2,1 %	
Running average 2007-2012	1,6 %	1,1 %	2,1 %
PSA	*2,5 %*	*1,8 %*	
Water Scrubber	*2,1 %*	*2,0 %*	
End-of-pipe	*1,0 %*	*0,7 %*	
Chemical Scrubber	*0,2 %*	*0,2 %*	

6 SUSTAINABILITY CRITERA

The upper quartiles of the running averages (2,5 % and 2,1 % respectively, Tables 2 and 3) were used as default values for the sustainability criteria calculation tool developed for the Swedish gas industry. These values are meant to be used for any plant that have yet not performed measurements. Since methane emissions have a great influence on the calculated greenhouse gas emission saving from the use of biofuels and bioliquids, these relatively high default values are not favourable and will be a driver towards more measurements.

Calculations are only needed for processes and substrates that do not fall within the directive's default greenhouse gas emission savings, e.g. 73 % for biogas from municipal organic waste as compressed natural gas. Any industrial organic wastes are not included in this directive's default value so calculations will be needed then. For processes that fall within the directive's default values there is however no need to account for real methane emissions (or transport emissions for that matter).

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(71) SOIL N_2O EMISSIONS FROM RECOVERED ORGANIC WASTE APPLICATIONS IN VERSAILLES PLAIN (FRANCE)

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EXECUTIVE SUMMARY

Within the framework of the French Gessol3 programme (Prostock project) we sought to characterize the emissions of nitrous oxide (N_2O) from organic wastes used on the Versailles plain in relation with the main soil types of this area (221 km^2). Four different soils with various textures were chosen (WRB classification): - **(1)** a haplic luvisol **(2)** a calcaric cambisol - **(3)** a stagnic cambisol and - **(4)** a cambisol. Four organic waste products were also chosen: a composted green waste **(G)**, a composted green waste mixed with an urban sludge **(GS)**, a dehydrated sludge of a sewage treatment plant **(DS)**, and horse manure **(HM)**. These four products had various nitrogen and carbon contents. Their C_T/N_T ratios varied between 6 and 38. The four soil types were first sieved and sub-samples were prepared by homogeneously adding the various waste products and repacked in cylinders of 15 cm diameter and 10 cm height to provide an equivalent of 4 t C_{org} ha^{-1} of input. Two additional soil treatments were prepared in the same way for control: one consisted of the soil with no organic waste (only water addition) called **SC** and the other with an addition of an UAN solution (half urea- half ammo nitrate) to provide an equivalent of 117 kg N ha^{-1} called **NC.** By addition of water, the soils were maintained to their maximum water holding capacities. The soil cylinders were placed for 15 min in an incubator to measure the rate of gas production. N_2O and CO_2 emissions were measured for thirteen days. In parallel to the soil core flux measurements, soil samples were prepared under the same conditions to measure soil mineral nitrogen content variations. Using the membrane press of Richards after the flux experiment, intact soil cores (cylinder of 5 cm in diameter and 2.5 cm height) were taken from the flux cylinders to characterize hydraulic potentials in relation with soil water contents.

For all the soil types, 13 days after treatment, DS and HM treatments showed the largest CO_2 losses. These two wastes have a fast mineralization rate. However, their C_T/N_T ratios were very different (6 and 38, respectively) this leads to a non-significant production of NH_4 during the mineralization for the horse manure as opposed to the dehydrated sludge. Independently of the organic waste that was applied, the magnitude of CO_2 emissions followed the order: Soil(2)>(4)>(3)>(1). This rank was linked to the soil aeration and depended of the various soil textures.

The Nitrous oxide emissions followed the dynamics of nitrification of soil ammonium. Three days after treatment, N_2O fluxes were negligible for the G and GS treatments. Only the initial mineral nitrogen content of the wastes contributed to the emissions. Concerning the DS treatment and for the four soil types, there was a slowdown or complete stop of the nitrification rate leading to a slowdown of N_2O emissions. These slowdowns were even lower when the soil CEC (Cation Exchange Capacity) and pH were high. Nitrous oxide emissions were higher with higher soil clay contents (soils 2 and 4). Clay soils have a higher water holding capacity and due to the buffering effect of their CEC, they could maintain an active nitrification rate with high soil NH_4^+ contents. Magnitudes of N_2O emissions with the four soils broadly followed the same order as for CO_2 exchange. Nitrous oxide emissions were largest for the calcareous soil (Soil 2). For this soil the ratios between N-N_2O emissions (net of the soil control) and the total nitrogen amounts of the various treatments were 9.6% for the NC, 6% for the DS, 3.5% for the HM, 2% for the GS, and -0.5% for the G treatments. For the two gases (CO_2 and N_2O) effect of the soil properties were as important for the magnitudes of emissions as the effect of the wastes characteristics.

1 INTRODUCTION

1.1 Background

In France the main source of organic waste is farming and forestry activities with a production of 374 Mt yr^{-1}. The livestock manures (from pasture or from recoverable excrements) contribute largely to this amount (298 Mt yr^{-1}) and 100% of the livestock manures are recycled in agriculture and participate in soil fertilization and amendment.

In large suburban areas like in the Paris region, away from livestock areas, household wastes and municipal sewage sludge remain the only local source of organic matter to enrich agricultural soils. According to ADEME (2009), in France 38 Mt yr^{-1} of municipal and household wastes are collected every year, with 12 Mt as green waste and 22 Mt as household waste. The production of municipal sewage sludge is estimated to 0.9Mt of dry matter per year. Only small fractions of the urban wastes are applied to the soils: 1.5 Mt yr^{-1} for the household refuse, 4 Mt yr^{-1} for the green waste and 0.5 $Mt_{(dry\ matter)}$ yr^{-1} for the sewage sludge (ADEME, 2009). After collection these various wastes are spread directly or can undergo various treatments (mixing, composting, anaerobic digestion, liming, thermal drying, ..).

The use of organic waste products as fertilizer is an emergent sector in agriculture. In addition, to their fertilizing values (intake of mineral products for the plants) spreading of organic matter improves soil properties (aeration, water retention, stimulates biological fauna and flora, reduces crusting and erosion). However, it may lead to health risks (presence of pathogens) or damages to the environment: accumulation of trace metals and organic micropollutants, GHG emissions like carbon dioxide or nitrous oxide (N_2O).

For many soils N_2O gas is produced mainly by denitrification. The addition of organic matter stimulates the biological processes bringing mineral nitrogen but also organic carbon necessary for the development of denitrifying bacteria (Burford, 1975). At equal nitrogen inputs, organic matter is suspected to lead to higher N_2O fluxes than with only mineral fertilizers.

Within the framework of the French Gessol3 program (Prostock project) we sought to develop spatial methodologies to improve the management of organic wastes spreading at a suburban territorial scale and to evaluate their effects on soil carbon stocks, nitrate leaching and N_2O emissions.

This study is focused on the risk of greenhouse gas (N_2O) emissions after application of organic wastes on soils. The Versailles plain of the Paris suburban area was chosen as the study territory. Four different organic wastes produced and used as soil amendment in the Versailles plain were selected. Similarly four types of soils with contrasted texture from the plain were selected. N_2O and CO_2 flux measurements were estimated during 13 days by using a laboratory setup with soil columns prepared by combining both types of soils and organic waste treatments. The major characteristics of soil types and organic wastes compared to the N_2O emission potentials are highlighted in this study.

2 METHODOLOGY

2.1 Context of the study

The Versailles plain represents 221 km² of the Paris suburban area with an agricultural area of only 2200 ha, cultivated mainly with cereal crops. In this suburban plain livestock farming activities have become scarce and the main local sources of organic wastes are provided from the municipal wastes, sewage sludge, and green waste. The presence of numerous equestrian centers led also to a significant production of horse manure. In total nearly 8000 tons of dry matters are applied on the arable lands per year.

In this context four organic waste products were chosen: a composted green waste **(G)**, a composted green waste mixed with an urban sludge **(GS)**, a dehydrated sludge of a sewage treatment plant **(DS)**, and horse manure **(HM)**. These four products had various nitrogen and carbon contents. Their C_T/N_T ratios varied between 6 and 38 (Table 1). The amount of organic waste applied on soil was adjusted for an input of 4 tCorg ha^{-1}. Given the various C_T/N_T ratios of the products, applied nitrogen doses were variable, ranging from 109 to 694 kgNt ha^{-1}.

Fig. 1 shows the soil map of the Versailles plain and indicates the localizations of the four selected soils. According to de WRB classification they correspond to a : - **(1)** haplic luvisol: - **(2)** calcaric cambisol: - **(3)** stagnic cambisol: - **(4)** cambisol. Major characteristics of the soils are given in Table 2. Soils (1) and (4) have similar clay contents (~16%) which are lower than soils (3) and (2) (~31%). Soil (2) is a calcareous soil (18% of $CaCO_3$) with a basic pH of 8.2 when Soil (1) is acidic (pH=6.2) and largely silty (75%).

2.2 Laboratory experimental setup

Nitrous oxide and CO_2 emissions were measured during 13 days following application of the organic waste using a laboratory setup. In this setup (Fig. 2) the soil is placed for 15 min in a sealed incubator of 10L connected to the input and output of two air gas analyzers (IR spectrometers: LICOR 820 for the CO_2 and ThermoEnvironment Instrument: 46C for N_2O). For this closed circuit the kinetics of gas accumulation is linear and emissions are proportional to the slope of the concentration increases.

The four soil types were first sieved (1 cm) and sub-samples of 1.78 kg of dry soil were prepared by homogeneously adding the various waste products to provide an equivalent of 4 t C_{org} ha^{-1} of input.

The sub-samples were repacked in cylinders of 15 cm diameter and 10 cm height. Three replicates were made by groups (combination of soil types and category of organic wastes). Two additional groups were added for control: one consisted of the soil with no organic waste (only water addition) called **SC** and the other with an addition of an UAN solution (half urea- half ammo nitrate) to provide an equivalent of 117 kg N ha^{-1} called **NC**. Soil temperature was kept constant at 20°C throughout the experiment. The soils were also maintained by water infiltration to their maximal water holding capacities to keep the soils under optimal conditions for denitrification.

In parallel to the flux measurements, soil samples were prepared under the same conditions to measure soil mineral nitrogen content variations. Inorganic nitrogen contents were analysed according to the ISO-14256-2 standard.

After the flux experiment, intact soil cores (cylinder of 5 cm in diameter and 2.5 cm height) were taken from the flux soil-cylinders to characterize their soil hydraulic potentials in relation to water contents using the Richards method of the membrane press (Richards, 1948).

3 RESULTS AND DISCUSSION

The average maximum of water holding capacities for the four selected soils were estimated 2 days after addition of water to: 31%, 34 %, 28%, 27% (w w^{-1}) for the soils (1), (2), (3), (4), respectively. The bulk densities of the soil cylinders estimated as the ratio between the dry soil mass and the volume of the soil in the cylinders were 1.32; 1.22; 1.39 and 1.36 (kg dm^{-3}) and were used in turn to estimate the mean rate of aeration for the 4 soils thus, Water Filled Pore Spaces (WFPS) were estimated to 82%, 77%, 82% and 74% for soils (1), (2), (3) and (4) respectively (Table 2).

Fig. 3 shows the relationships between WFPS and soil water pressure heads in pF (pF =Log(h) with h in cm) for the 4 soils obtained with the Richards method of the membrane press. Our experimental soil conditions for the gas flux measurements corresponded to pF values between 1 and 1.5. These pF values are lower than the field capacities usually estimated for an additional pressure of 0.33 bar (330 cm or pF = 2.5). The corresponding high values of WFPS, higher than 62% allow us to guarantee a N_2O production through denitrification. Indeed, the 62% value for the most cases indicates the threshold at which denitrification becomes active (Grundmann, 1987). In Fig. 3 the variations of WFPS with pF are practically parallel for the soils (1) and (3) on one side and for the soil (2) and (4) on the other side. For high pF values, soils (2) and (4) retain more water than soils (1) and (3). This is related to the clay contents of each soil pair, around 31% for soils (2) and (4) and 16% for soils (1) and (3). Fine textures favor the presence of micro pores which retain more water and seem to promote denitrification rates.

Fig. 4 shows the cumulative CO_2 emissions for the various soils and treatments. The coefficients of variation for the 3 replicates defined as the ratio between the standard deviations and the averages were around 15%. They were larger for weak emissions like for soil controls (SC) and UAN treatments (NC), when CO_2 fluxes were weak and the sensitivity threshold in the measurement method was more important than the value.

Independently of the type of organic waste that was applied, thirteen days after treatment, the magnitudes of CO_2 fluxes followed the order: Soil(2)>(4)>(3)>(1), This rank could be partly linked to the soil aerations. According to Linn (1984) mineralization optima are often observed in intermediate soil moisture conditions around WFPS~60%. In this study, the soils (2) and (4) have lower WFPS compare to the soil (1) and (3).

With an identical carbon supply whatever the soils, DS and HM treatments showed largest carbon mineralization rates. For the four soils, 13 days after treatment CO_2 losses were in average 758 kg C ha^{-1} for DS and 450 kg C ha^{-1} for HM while for the soil controls they were in average 76 kg C ha^{-1}.

Figures 5 and 6 show the variations of ammonia contents and the cumulated N_2O emissions during 13 days after treatments. Initial ammonia contents were higher for GS, DS and NC treatments and corresponded approximately to the $N\text{-}NH_4^+$ doses calculated with $N\text{-}NH_4^+$ wastes contents and mixing factor with soil (Table 1). With the exception of DS treatment, NH_4^+ concentrations decreased with time due to the nitrification rates of the waste products. For the DS treatment there was an accumulation of NH_4^+ over time probably due to the strong mineralization of the sludge as already observed with CO_2 emissions and due to its low C_T/N_T ratio (around ~6). As shown in Figure 5, this accumulation leads to a slowdown in the nitrification rate. These slowdowns were largest when soil CEC and pH were low. For the calcareous and clay soil: soil (2), accumulation of ammonium is only transient between days 1 and 3 while for less clayed and acid soils accumulation of NH_4^+ persisted until days 7 or 13. The nitrification process produces acids, lowers the pH of the biological population and causes a reduction of the growth rate of nitrifying bacteria. The optimum pH for *Nitrosomonas* and *Nitrobacteris* is between 7.5 and 8.5 and nitrification stops at a pH below 6.0 (Grunditz, 2001). The alkalinity and the higher CEC for the soil (2) insured adequate buffering capacities for nitrification.

For the N_2O fluxes the coefficients of variations (CV's) on the 3 replicates were larger than for CO_2 emission ones with a mean value around 37%. These large CV's were linked to N_2O coming from the denitrification process in itself. Denitrification occurs generally in "hot spot" sites corresponding to anoxia and compacted zones with accumulation of organic matter and subtract. These microsites are therefore very heterogeneous and affect the spatial distribution of the N_2O sources. The Nitrous oxide emissions followed the dynamics of nitrification of soil ammonium. Three days after treatment, N_2O fluxes were negligible for the G and GS treatments. Only the initial mineral nitrogen content of the wastes contributed to the emissions. Concerning the DS treatment and for the four soil types, the slowdown or the stop of nitrification lead to a slowdown of N_2O emissions. Like for NH_4^+ dynamics these slowdowns were lower when the soil CEC and pH were high. Nitrous oxide emissions were higher with high soil clay content i.e. for soils (2) and (4). Clay soils have a higher water holding capacity and due to the buffering effect of their CEC, they could maintain an active nitrification rate. Magnitudes of N_2O emissions with the four soils broadly followed the same order as for CO_2 exchanges (Table 3). Nitrous oxide emissions were largest for the calcareous soil (Soil (2)). For this soil the ratios between N-N_2O emissions (net of the soil control) and the total nitrogen applied were 8.71% for the NC, 5.97% for the DS, 3.51% for the HM, 1.05% for the GS, and 0.47% for the G treatments (Table 4). The emission factor with the synthetic fertilizer (UAN) was higher than with the organic DS treatment but the N_2O dynamics between these two products are very different (Fig. 5). For the UAN treatment after 13 days N_2O cumulated emissions seem to be stabilizing and most of the N-NH_4^+ appears to be nitrified while for the sewage sludge treatment the N_2O flux continues to increase indicating an additional contribution with the nitrogen mineralization. In fact, for soil (2) with the DS treatment we continued to measure the emissions up to 47 days after treatment application and we totalized a cumulative loss of 69.65 kg N-N_2O i.e. approximately 10% of the total nitrogen applied (Table 2). For soil (1) the cumulative N_2O emissions for DS and HM treatments were less than for the soil control ones (SC) and negative fluxes were measured. These negative values corresponded to atmospheric N_2O uptakes by soils and were probably related to the N_2O respiration by microorganisms leading to the reducing of N_2O in N_2. Large N_2O respiration generally occurs when nitrate is not available in soils and under very limiting condition in oxygen (Chapuis-Lardy, 2007). For the HS and DS treatments we can assume, that soil nitrate contents were very low due to the nitrification stop and that the high rates of carbon mineralization consumed more oxygen and favored anoxic conditions compare to the soil control treatments. These two conditions could explain the net N_2O uptakes for the DS and HM treatments.

4 CONCLUSIONS

This study allowed a better assessing of the potential losses in greenhouse gas from soils after treatments with organic wastes. Biogas emissions were assessed for various types of soils and recovered organic wastes, having different physicochemical characteristics. The soils were selected on criteria of representativeness of the suburban area of the Versailles plain and for their contrasted texture. The recovered organic wastes are produced locally and are used for agricultural soil as amendment and fertilizer.

We have shown a very marked effect of soil types on the greenhouse gas production. Clay soils seem to be more favorable to the N_2O emissions. They have higher water holding capacities and higher cation exchange capacities which could maintain an active nitrification rate with high NH_4^+ content by buffering effect.

We have shown a marked effect of the waste types. The horse manure and sewage sludge mineralize very quickly but their C_T/N_T were very different, providing with the mineralization, weak quantities of N-NH_4 for horse manure and large quantities for sewage sludge. The composted green waste (G) and the composted green waste mixed with an urban sludge (GS) were poorly biodegradable. Only the initial mineral nitrogen contents of the products have an effect on the N_2O emission dynamics for the first three days.

Figures

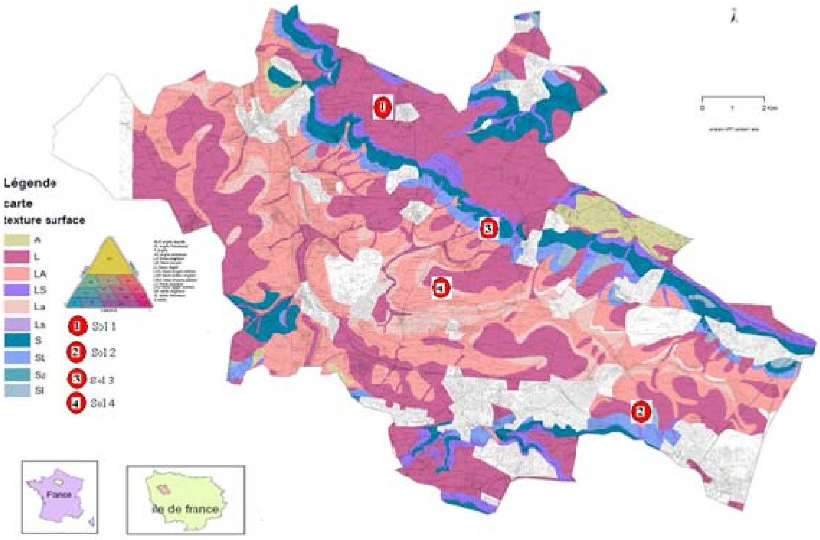

FIGURE 1 **Soil map of Versailles plain with the locations of the four selected soils**

FIGURE 2 **Illustrations of the experimental setup**

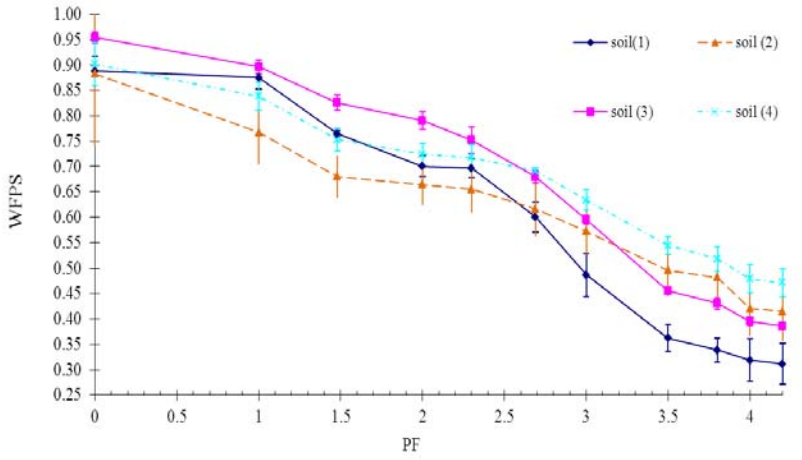

FIGURE 3 **Relationships between soil water pressure head in pF (pF=Log10 (h in cm) and water filled pore space (WFPS). The soil cylinders used during N_2O flux experiment were maintained between pF= 1 and pF= 1.5**

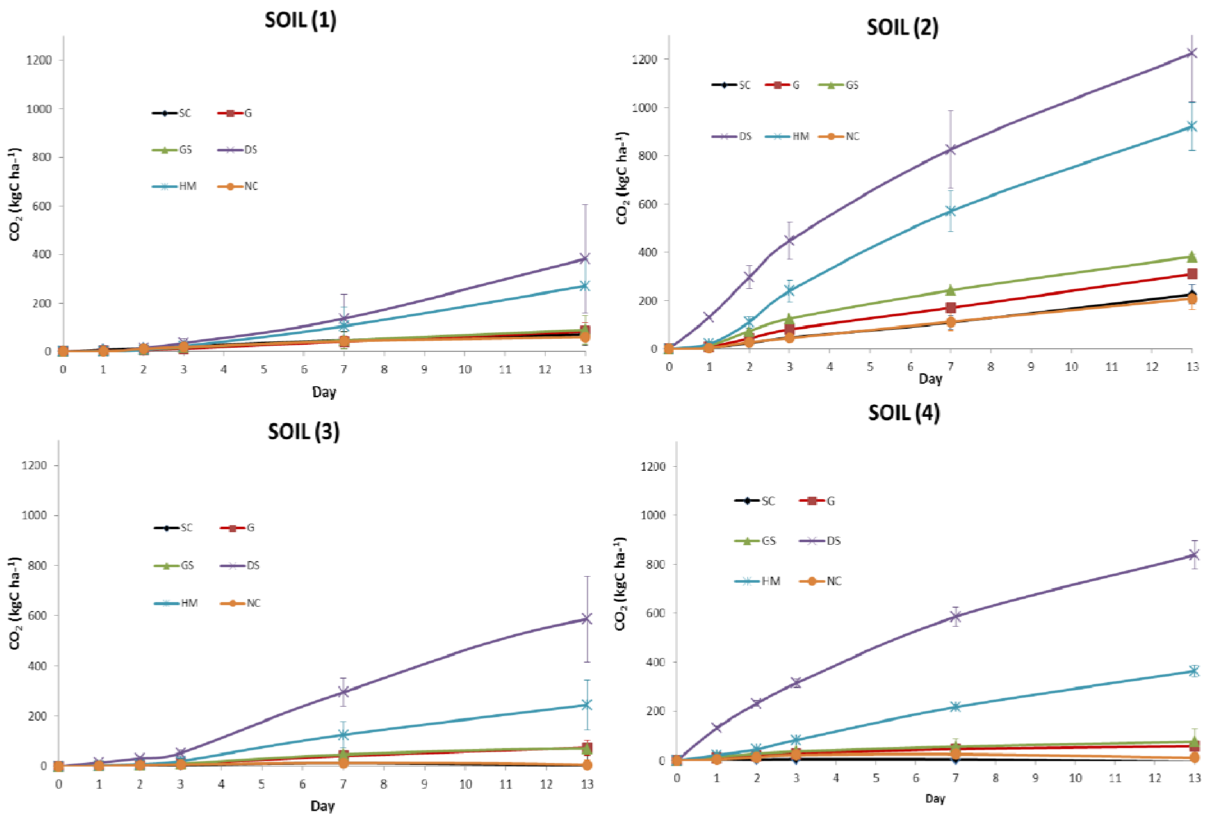

FIGURE 4 **Cumulated emissions of CO_2 for the 4 soils and the 6 treatments: SC Soil Control, G composted Green waste, GS composted green waste mixed with urban sludge, DS Dehydrated Sludge of a sewage treatment plant, HM Horse Manure, NC Nitrogen Control**

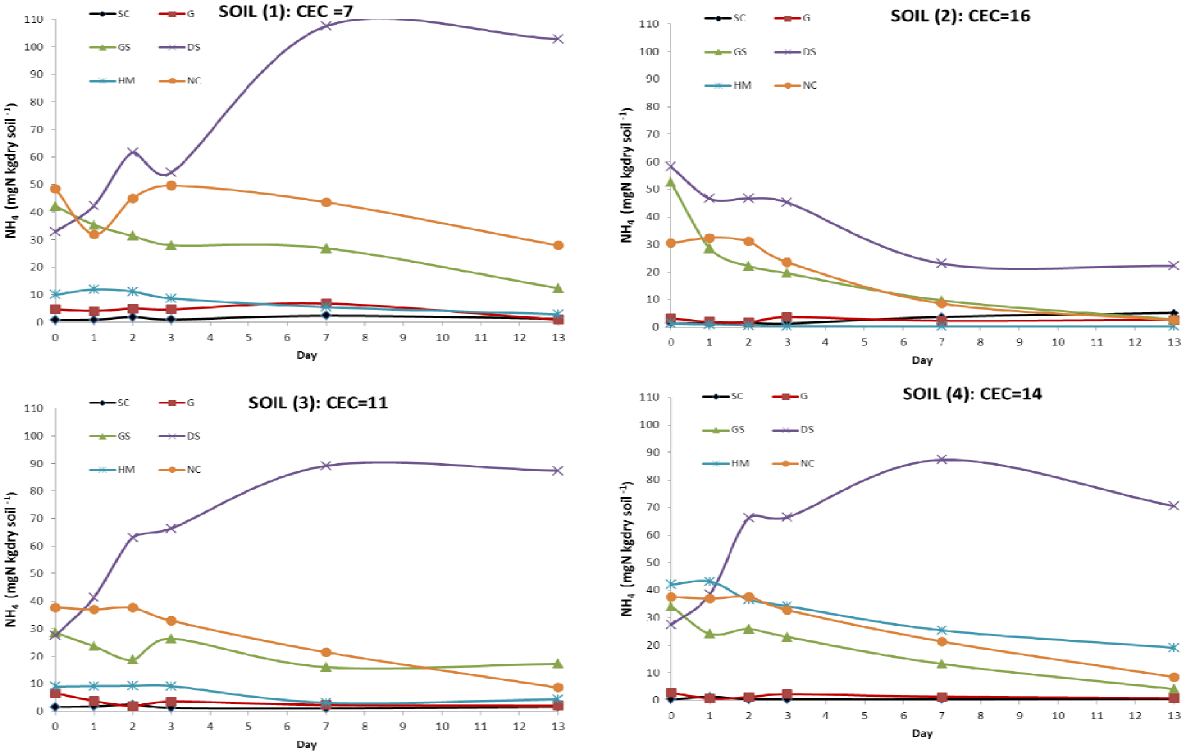

FIGURE 5 **Variations of N-NH_4 contents for the 4 soils and the 6 treatments: SC Soil Control, G composted Green waste, GS composted Green waste mixed with urban Sludge, DS Dehydrated Sludge of a sewage treatment plant, HM Horse Manure, NC Nitrogen Control**

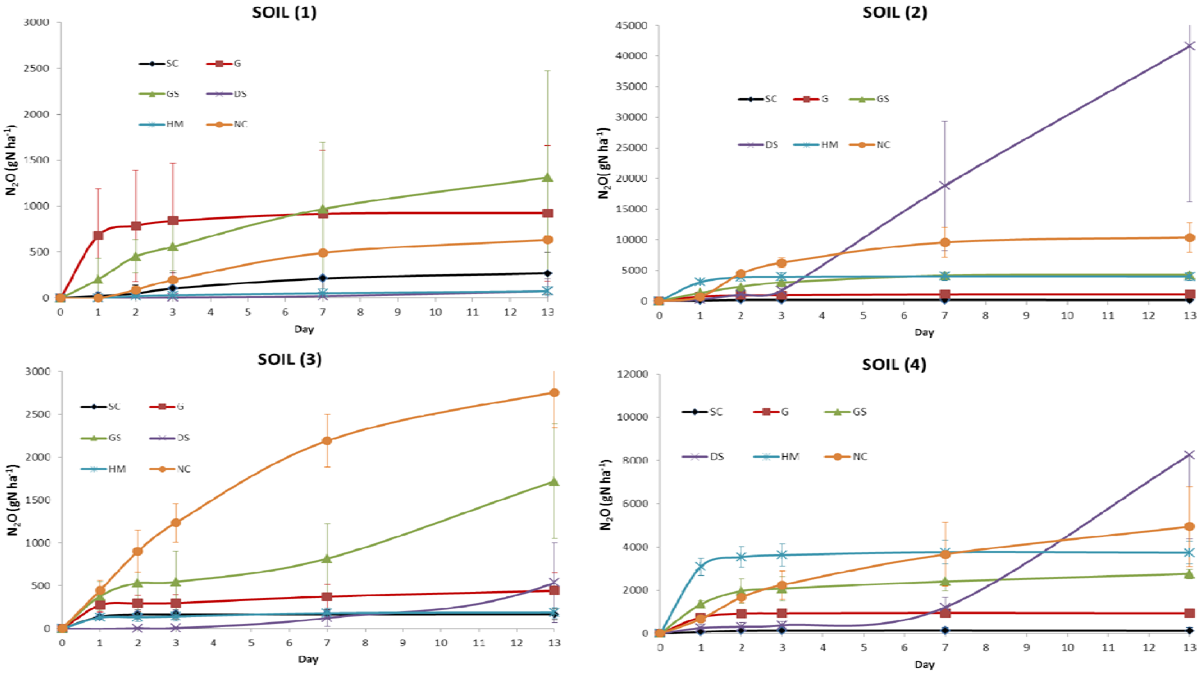

FIGURE 6 **Cumulated emissions of N_2O for the 4 soils and the 6 treatments: SC Soil Control, G composted Green waste, GS composted Green waste mixed with urban Sludge, DS Dehydrated Sludge of a sewage treatment plant, HM Horse Manure, NC Nitrogen Control**

Organic Waste	**N g kg dry matter^{-1}**					**Nitrogen Inputs kg N ha^{-1}**		
	N-NH_4	**N-NO_3**	**N_T**	**C_T**	**C_T/N_T**	**N_T**	**N-NH_4**	**N-NO_3**
G	0.33	0.02	12.86	231	18	199	2.2	0.4
GS	3.46	0.14	24.81	260	10.5	387	54	2.2
DS	2.19	0.01	67.19	407	6.1	694	22.8	0.1
HM	0.71	0.49	11.04	418	37.9	109	7	4.9

TABLE 1 **Composition of organic wastes and amounts of nitrogen inputs for soil cylinders. N_T and C_T correspond to the total nitrogen or carbon contents.**

Soil Type	**Clay(%)**	**Silt(%)**	**Sand(%)**	**CaCO3(gkg^{-1})**	**θ (%)**	**WFPS(%)**	**CEC(cmol+ kg^{-1})**	**pH**	**M_O(g kg^{-1})**	**C_O(g kg^{-1})**	**N_T(g/kg)**	**C_T/N_T**
Soil(1): haplic Luvisol	15.2	74.5	10.3		31	82	7.28	6.2	17.2	9.93	1	9.9
Soil (2): calcaric cambisol	35.3	45.5	19.2	180.9	34	77	16.15	8.2	34	19.7	1.78	11.1
Soil(3): stagnic cambisol	16.2	40.9	42.9		28	82	10.82	7.3	28.9	16.7	1.54	10.8
Soil (4): cambisol	26.8	65.6	7.6		27	74	14.04	6.8	23.6	13.7	1.28	10.7

TABLE 2 **Soil characteristics: texture, carbonate content, soil gravimetric moisture contents, water filled pore space, cation exchange capacity (CEC), pH, organic matter and carbon contents, total nitrogen contents.**

Treatment	**CO_2 (kg C ha^{-1})**				**N_2O (g N ha^{-1})**			
	Soil(1)	**Soil (2)**	**Soil(3)**	**Soil (4)**	**Soil(1)**	**Soil (2)**	**Soil(3)**	**Soil (4)**
SC	73	227	2	3	269	193	166	134
G	84	311	74	57	924	1125	442	933
GS	91	383	71	75	1312	4275	1720	2753
DS	383	1225	587	838	76	41613	536	8263
HM	270	922	245	364	75	4016	186	3735
NC	62	210	6	11	634	10382	2754	4938

TABLE 3 **Mass balance of gas emissions after 13 days of measurement**

Treatment	**R C-CO_2 (%)**				**R N-N_2O (%)**			
	Soil(1)	**Soil (2)**	**Soil(3)**	**Soil (4)**	**Soil(1)**	**Soil (2)**	**Soil(3)**	**Soil (4)**
G	0.29	2.09	1.79	1.36	0.33	0.47	0.14	0.40
GS	0.45	3.90	1.71	1.81	0.27	1.05	0.40	0.68
DS	7.75	24.96	14.62	20.88	-0.03	5.97	0.05	1.17
HM	4.95	17.38	6.06	9.04	-0.18	3.51	0.02	3.30
NC					0.31	8.71	2.21	4.11

TABLE 4 **Ratios between C-CO_2 and N-N_2O emissions (net of the soil control) and the total carbon (C_T= 4 t C ha^{-1}) or total nitrogen applied (N_T) after 13 days of measurement.**

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(141) $^{G}E_{S}$TABOUES, A DECISION TOOL TO ASSESS GREENHOUSE GASES OF SEWAGE SLUDGE TREATMENT AND DISPOSAL ROUTES

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EXECUTIVE SUMMARY

Sewage sludge production increases continuously reaching almost 20% (946 700 tons of dry matters in 2003 to 1 118 795 tons of dry matters in 2007) during the last decade. In 2007, 70% of the produced sewage sludge was spread (directly or after composting). The remaining 30% was incinerated (with or without household wastes) or landfilled.

Nowadays, sludge reduction is a major concern. This activity has to become more sustainable and stakeholders have to be careful to the environmental impacts of sludge treatment and disposal routes.

To help stakeholders in that way, we developed a decision tool called $^{G}E_{S}$TABoues. $^{G}E_{S}$TABoues is a tool based on the "Bilan Carbone®" method (ADEME 2009). It was developed for stakeholders dealing with wastewater treatment plants (plant manager, public administration…) to quantify greenhouse gases (GHG) for each type of emissions and each process of sludge treatment and disposal routes. The tool was developed with VisualBasic programming language.

This tool can be used in a four step procedure:

(i) The user built as many wastewater treatment plants (WWTP) he wants. He should specify the WWTP capacity, the type of sewage network and the water treatment system.

(ii) Then the user creates all the sewage sludge treatment processes used for each WWTP. He has to choose different parameters for each step of sludge treatment and disposal. The user has the opportunity to choose either its own data or data collected through a literature review and implemented as "default value" in the tool.

(iii) Once each WWTP and the treatment and disposal routes of each WWTP created, the user can choose different graphic presentations to assess the impact of greenhouse gases emissions (Figure 1). $^{G}E_{S}$TABoues calculate all emissions (direct and indirect) of carbon dioxide, methane and nitrous oxide for all steps of sewage sludge treatment (storage, thickening, anaerobic digestion, aerobic digestion, dewatering, alkaline stabilization, composting, drying) and sludge disposal route (land application, incineration, incineration with household wastes, landfilling). Emissions from infrastructures and transports are also considered. These graphs present emissions for each step (thickening, dewatering, land spreading…), each gas (dioxide carbon, methane and nitrous oxide) and each origin of greenhouse gases (combustible, electricity, direct emissions, avoided emissions, infrastructure, chemicals and transport). The graphs can be presented either as values or percentages.

iv) Finally, the user can compare different sewage sludge treatment processes and disposal routes options and create reports with Microsoft Word through an export button. Each report summarizes the mass and energy balance as well as the selected graphs to be exported.

In this study, this tool is used to compare 3 systems on a same WWTP to help stakeholders to identify which processes have the worst environmental impact all along the treatment and disposal route, which emission is overwhelming and to help them selected the most interesting system from an environmental point of view.

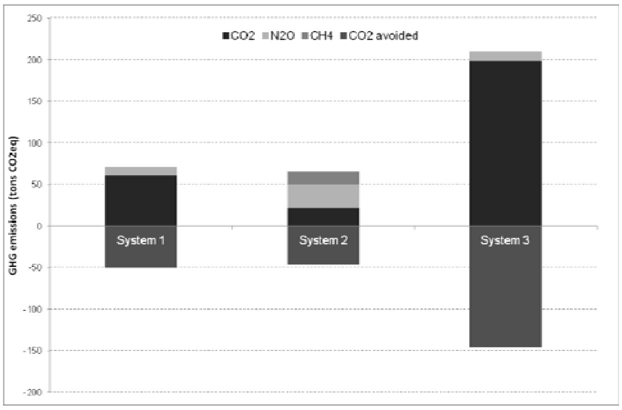

FIGURE 1 **Example of $^{G}E_{S}$TABoues results**

1 INTRODUCTION

1.1 Background

During the last 20 years, European directives were established to obtain a good quality of aquatic systems. Those regulations adapted in French laws lead to consequences on sewage treatment. In March 2010, there was 18 637 wastewater treatment plants (WWTP) in France treating the pollution of 75 million equivalent inhabitant (equivalent to the charge of 18699 towns) (Golla et al., 2010).

Sewage sludge treatment and disposal can constitute up to 40% of total emissions associated with wastewater treatment (Shaw et al., 2010). A range of different stabilization and end use technologies are widely available, each technology associating different costs and environmental impacts (Brown et al., 2010). Sewage sludge increases continuously and about 1 100 000 tons of sewage sludge were produced in 2008. Sewage sludges are treated (dewatering, stabilization and sanitization) with different technologies depending on WWTP capacity and final disposal route. Four disposal routes are currently possible: 70% of sludge is spread on agricultural soils (directly or after composting), 20% is incinerated and the last 10% is landfilled.

The technologies involved in both sludge treatment and disposal have different energetic costs and variable consequences on the environment and Global Warming. Understanding the greenhouse gases (GHG) emissions associated with different sewage sludge management practises is likely to influence public opinion and municipal decision-making (Brown et al., 2010).

1.2 Research objectives

In order to help stakeholder in their decision making, we develop a decision tool, called $^{G}E_{S}$TABoues, to assess the impact on Global Warming of different sewage sludge treatment and disposal routes.

This paper aims to present $^{G}E_{S}$TABoues tool which was developed to model and calculate GHG emissions from different sewage sludge treatment processes and disposal routes options. The tool was designed to compare the GHG impact of different sewage sludge management options.

A comparison of 3 sludge treatment and disposal route systems is presented in this paper. One system represent data coming from a real WWTP, the two others are optional systems that could be interesting for the WWTP managers.

The first part of this paper will present the framework of $^{G}E_{S}$TABoues. The second part presents the studied systems and the last part how the results impact on Global Warming.

2 METHODOLOGY

2.1 $^{G}E_{S}$TABoues tool

The $^{G}E_{S}$TABoues tool allows the calculation of the carbon footprint of sludge treatments and disposal routes. The method underlying this tool is explained in Pradel and Reverdy (2012). It is based on the "Bilan Carbone®" method (ADEME, 2009), a general method used to quantify GHG generated from all physical processes which are necessary for any activity or human organization. In our method, three GHG are recorded: carbon dioxide (CO_2), methane (CH_4) and nitrous oxide (N_2O). Each gas has his own Global Warming Potential (GWP): 1 for CO_2, 25 for CH_4 and 298 for N_2O.

In $^{G}E_{S}$TABoues, direct and indirect emissions are considered as well as avoided emissions. This tool doesn't take into account biogenic CO_2 emissions as they belong to the short cycle of carbon and as they are considered (by convention) as "carbon neutral".

In $^{G}E_{S}$TABoues, GHG emissions are quantified for x tons of sludge produced by a wastewater treatment plant of x per-captia equivalent (PCE) during one year.

Default values were defined for each process, including inputs, energy use, chemical consumption and GHG emissions. Data were collected from literature (Pradel and Reverdy, 2012). Emissions and credits for each process were classified as direct emissions, chemicals, electricity, fuels, transport, infrastructure and avoided emissions.

$^{G}E_{S}$TABoues tool was developed with VisualBasic programming language. It is made with 4 different windows (Figure 2) : WWTP creation, System creation/modification, System comparison, Results export.

FIGURE 2 $^{G}E_{S}$TABoues main window screen shot

2.1.1 WWTP creation

This window is used to create different WWTP with their characteristics. In this part, user can choose the WWTP capacity (in per-captia equivalent), the type of sewer network (combined sewer system, separated sewer system, partially separated sewer system) and the type of water treatment (activated sludge, biological treatment…).
During this step, dry matter quantity of sludge produced is accounted according to 3 methodologies:

- User knows the dry matter quantity and use it (in dry matter ton/year),
- User doesn't know the dry matter quantity but knows BOD_5 and suspended matter quantities (in kg/day),
- User doesn't have any data; dry matter quantity is calculated based on Guérin-Schneider (2001).

2.1.2 System creation/modification

During this step, users can create several systems for each WWTP. Each system is composed by different treatments and disposal routes that user can select (Table 1). For each treatment and disposal route selected, a window opens to be completed either with users' values or with default values coming from the literature.

TABLE 1 **Treatment and valorisation/disposal routes in GESTABoues**

Treatment	Valorisation/Disposal
Storage	Land application Incineration Incineration with household wastes Landfilling
Thickening *(gravity, flotation, thickening grid, thickening table, thickening drum, centrifugation)*	
Anaerobic digestion stabilisation	
Aerobic digestion stabilisation	
Dewatering *(classical centrifugation, high performance centrifugation, direct centrifugation, belt filter, press filter, sludge dewatering reed beds)*	
Liming stabilisation	
Composting	
Drying *(thermal, solar)*	
Transports, Infrastructures	

At the end of systems creation, $^{G}E_{S}$TABoues takes into account GHG emissions during transports (polymers, sludge, chemicals, ashes …). A new window opens to be filled by the user for that purpose. Then a window opens to show already calculated GHG emissions from infrastructure (civil engineering and electrical/mechanical equipments).

The next step provides results presented as bar chart graphs. The graphs present emissions for each step (thickening, dewatering, land application…), each GHG (carbon dioxide, methane and nitrous oxide) and each origin of GHG (combustible, electricity, direct emissions, avoided emissions, infrastructure, chemicals and transport). They can be presented either as values or percentages. In Gestaboues, 10 types of bar chart can be shown.

2.1.3 Systems comparison

$^{G}E_{S}$TABoues was also designed to compare different systems in order to analyse the best option either in sludge treatment or disposal route. By selected several systems for each WWTP, it is possible to compare them using the same bar charts graphs than for a single use (see "system creation/modification" section).

2.1.4 Results export

User can create reports with Microsoft Word trough an export button. Each report summarizes the mass and energy balance as well as selected graphs to be exported.

2.2 Studied systems choice

To validate $^{G}E_{S}$TABoues, we choose to test three different systems of a same WWTP. The WWTP has a capacity of 8500 PCE. Sewage is collected with a partially separated sewer network and treated with extended aeration. The WWTP produces around 71 tons of dry matter of sludge during one year.

The WWTP characteristics, the studied systems and the data collected for each system are shown in Table 2.

The first system is the current system of the studied WWTP. The sludge treatment and disposal route system is composed by gravitational thickening, belt filter dewatering, liming and land application. System 2 and system 3 are variants of this system.

We proposed in system 2 to change the stabilization process by replacing liming by composting and in system 3 to change the disposal route by incineration with household wastes so as stabilisation is not any more necessary.

TABLE 2 **Wastewater treatment plant and systems characteristics and inputs in each system**

	System 1	System 2	System 3
Capacity (PCE)	8500		
Network	Partially separated sewer system		
Water treatment	Extended aeration		
Sludge quantity (DM tons)	71.36		
BOD_5 (kg/j)	510		
Thickening	Gravitational thickening		
Dewatering	Belt filter		
Stabilisation	Liming	Composting	-
Disposal	Land application	Land application	Incineration with household wastes (co-incineration)
Polymers (kg)	Thickening = 0 Dewatering = 180	Thickening = 0 Dewatering = 180	Thickening = 0 Dewatering = 180
Electricity (kWh)	Thickening = 713 Dewatering = 1819	Thickening = 710 Dewatering = 1708	Thickening = 710 Dewatering = 1708
Lime (t)	Liming = 44	0	Co-incineration = 923
Co-substrate (t)	0	Composting = 200	0
Coal (kg)	0	0	Co-incineration = 99
Fuel	Land application = 32 L/h	Land application = 33 L/h	Co-incineration = 5

3 RESULTS AND DISCUSSION

3.1 System 1

System 1 generates about 71 tons CO_2eq (all gases merged) during one year. GHG emissions are shared between:

- CO_2 = 61 tons CO_2eq,
- N_2O = 10 tons CO_2eq,
- Avoided emissions = - 51 tons CO_2eq (-21.8 from mineral fertilisers and -28.9 from carbon sequestration)

TABLE 3 **GHG emissions of system 1**

Tons CO_2eq		**Treatment and disposal steps**				
		Thickening	**Dewatering**	**Liming**	**Land application**	**Total**
Emission origin	Direct emissions	0	0	0	10.2 (as N_2O)	10.2
	Chemicals	0	0.77	42.9	0	43.7
	Electricity	0.063	0.16	0.031	0	0.25
	Fuel	0	0	0	0.50	0.5
	Transports	0	9.4	1.1	5.8	16.3
	Infrastructure	0.17	0.20	0.24	0.003	0.61
	Avoided emissions	0	0	0	-50.7	-50.7
Total		0.23	10.5	44.3	16.5 / -50.7	**71.5 / -50.7**

We can conclude from Table 3 that:

- Thickening emissions are insignificant.
- 90% of dewatering GHG emissions is generated by polymers transport between suppliers and WWTP.
- Liming is responsible to the most important GHG emissions. During liming, more than 95% of GHG are generated by lime production.
- Land application emissions come from N_2O direct emissions and sludge transport between WWTP and fields.
- Avoided emissions are generated during land application (-50.7 tons CO_2eq). Avoided emissions are due to the non use of mineral fertilizers and to the carbon sequestration (-0.25 kg CO_2eq/kg dry matter based on SYLVIS, 2009).

3.2 System 2

65.2 tons CO_2eq are produced by system 2 and -46.8 tons CO_2eq are avoided. GHG emissions are shared between:

- CO_2 = 21.9 tons CO_2eq,
- CH_4 = 15.2 tons CO_2eq,
- N_2O = 28.1 tons CO_2eq.

TABLE 4 **GHG emissions of system 2**

Tons CO_2eq		**Treatment and disposal steps**				
		Thickening	**Dewatering**	**Composting**	**Land application**	**Total**
Emission origin	Direct emissions	0	0	15.2 (as CH_4) 25.0 (as N_2O)	3.1 (as N_2O)	43.3
	Chemicals	0	0.77	0	0	0.77
	Electricity	0.063	0.16	0.77	0	1.0
	Fuel	0	0	0.22	0.46	0.68
	Transports	0	9.4	5.7	2.8	17.9
	Infrastructure	0.17	0.19	1.2	0	1.6
	Avoided emissions	0	0	0	-46.8	-46.8
Total		0.23	10.5	48.1	6.4 / -46.8	**65.2 / -46.8**

We can conclude from Table 4 that:

- Thickening and dewatering emissions are similar between systems 1 and 2. During thickening, they are negligible and during dewatering, emissions are produced by polymers transport.
- More than 70% of the emissions are generated by sludge composting. The other 30% are mainly divided between dewatering (10.5 tons CO_2eq) and land application (6.4 tons CO_2eq).
- More than 80% of composting emissions are N_2O and CH_4 direct emissions. About 12% of the remaining emissions are generated by sludge transport between WWTP and composting site (composting site is supposed to be at a 50km distance).
- Land application emissions are 6.4 tons CO_2eq, an half is generated by direct emissions. About -47 tons CO_2eq are avoided. Avoided emissions are due to the non use of mineral fertilizers and to carbon sequestration (respectively -21.8 and -25 tons) as for system 1.

3.3 System 3

The third system is responsible of a production of 210 tons CO_2eq and -146.5 tons CO_2eq of avoided emissions:

- CO_2 = 198.6 tons CO_2eq,
- N_2O = 11.4 tons CO_2eq.

TABLE 5 **GHG emissions of system 3**

Tons CO_2eq		**Treatment and disposal steps**			
		Thickening	**Dewatering**	**Incineration with household wastes**	**Total**
Emission origin	Direct emissions	0	0	162.9 (as CO_2) 11.4 (as N_2O)	174.3
	Chemicals	0	0.77	1.6	2.4
	Electricity	0.063	0.15	0.73	0.94
	Fuel	0	0	16.0	16.0
	Transports	0	9.4	6.6	16.0
	Infrastructure	0.17	0.19	0.007	0.40
	Avoided emissions	0	0	-146.5	-146.5
Total		0.23	10.5	199.2 / -146.5	**210.0 / -146.5**

We can conclude from Table 5 that:

- Thickening and dewatering emissions are similar with systems 1 and 2 (same processes).
- 95% of GHG production is generated during sludge incineration with household wastes.
- About 85% of the emissions generated during incineration are direct emissions. During incineration, sludge and household wastes are mixed. This leads to the emission of fossil CO_2 as household wastes not only contain biogenic carbon in opposition to sludge. Fossil carbon is inevitably incinerated and generated even if it doesn't originate from sludge.
- Avoided emissions in system 3 (-146.5 tons CO_2eq) are due to the non use of fuel replaced by the heat generated by the incineration with household wastes.

3.4 Mass and energy balances

Mass and energy balance for each system are shown in Figure 3.

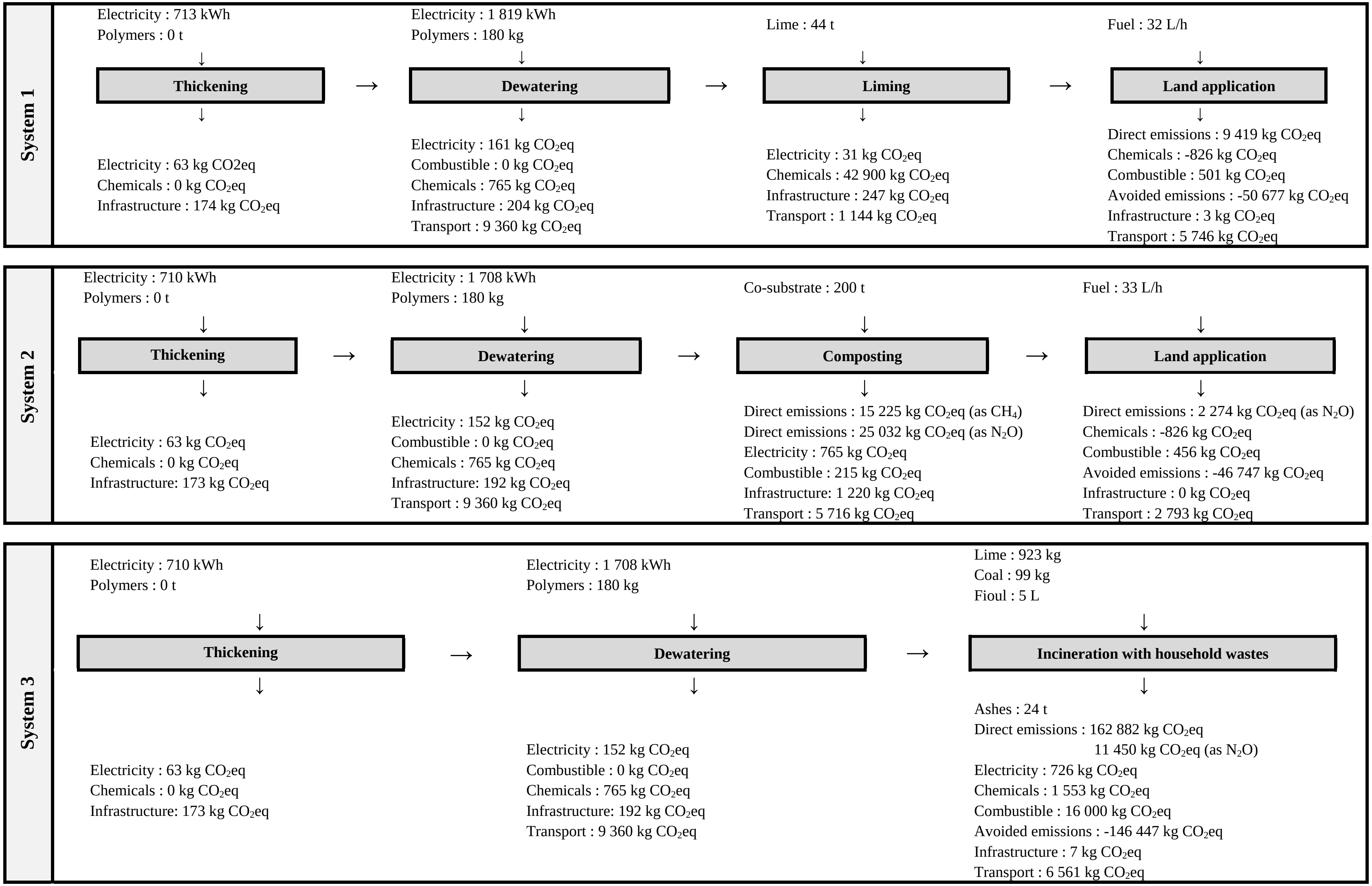

FIGURE 3 **Mass and energy balance for the 3 systems**

3.5 Systems comparison

The three systems were compared regarding the nature of the GHG emitted (Figure 4). Regarding global emissions, the second system is more effective. It produces 65.2 tons CO_2eq against 71.4 and 210 tons CO_2eq for respectively the system 1 and the system 3.

Regarding avoided emissions, the second system is the less interesting (-46.8 tons CO_2eq), but if we sum the global emissions with the avoided ones, it remains the best system. It generates 18.4 tons CO_2eq against 20.7 tons CO_2eq for the system 1 and 63.5 tons CO_2eq for the system 3.

In systems 1 and 3, emissions are mainly CO_2 whereas in system 2, emissions are allocated on a balance between CO_2, CH_4 and N_2O.

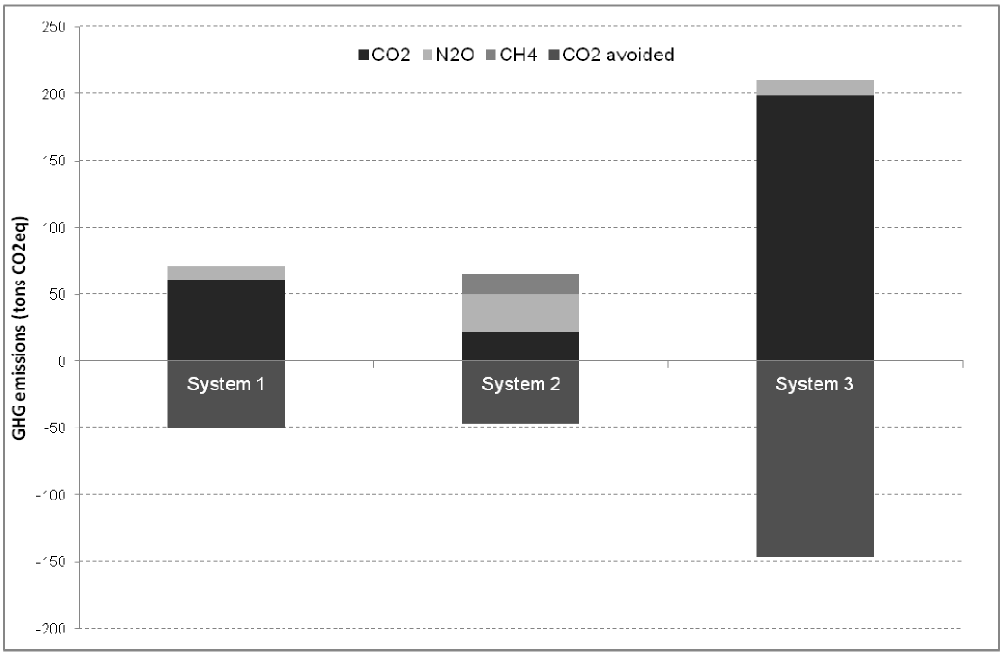

FIGURE 4 **GHG emissions comparison in terms of gas nature**

Figure 5 compares the three systems regarding each treatment and disposal steps and the origin of emissions.

Emissions during thickening and dewatering are identical because treatments are the same.

Land application GHG emissions vary between system 1 and 2 due to the nature of the sludge. Limed and composted sludges haven't the same agronomic composition and composting decrease dry matter sludge and so the spread quantities. N_2O emissions generated after spreading and avoided emissions (mineral fertilisers) calculated based on the N content of the sludge are different between the two types of sludge spread. During land application in the first system, 16.5 tons CO_2eq are generated and -50.7 tons CO_2eq are avoided. During land application in the second system, 6.4 tons CO_2eq are generated and -46.8 tons CO_2eq are avoided.

System 1 produces 2.6 more GHG emissions and only 1.1 more avoided emissions than system 2. The sum between general emissions and avoided emissions leads to -34.2 tons CO_2eq for the system 1 and -40.4 tons CO_2eq for the system 2. Sludge composting before land application generates less GHG emissions than sludge liming.

In comparison with sludge spreading in systems 1 and 2, incineration with household wastes appears to be the worst option from an environmental point of view.

The system 2 is the most interesting regarding the Global Warming impact. However, these results should be considered cautiously as data are order of magnitude (average values) and the uncertainty wasn't recorded.

Results are also representative from an environmental point of view and should be compared with economic or social dimensions to give a global approach in waste management. Indeed, a WWTP has to answer to environmental objectives, but it answers first to costs and local pressures.

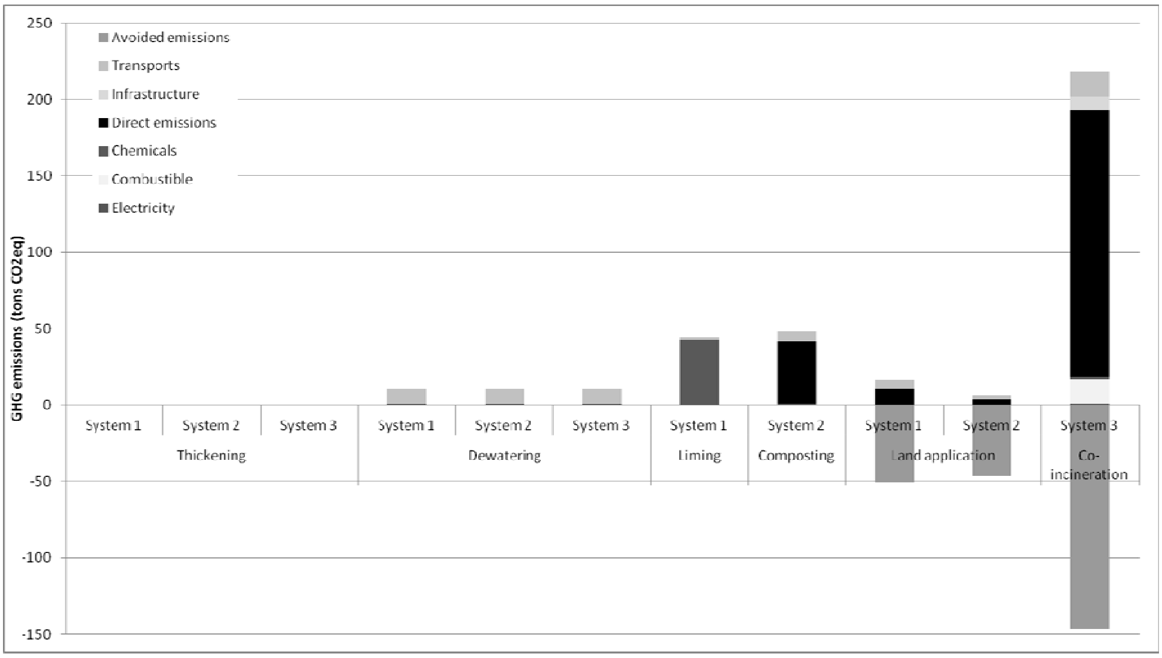

FIGURE 5 **GHG emissions comparison for each treatment step and emissions origins**

4 CONCLUSION

This study was conducted to validate $^{G}E_{S}$TABoues tool and to illustrate the type of results that can be obtained between several sludge treatment and disposal routes systems.

Regarding the first results, the system 2 is the most interesting as the 2 others. The results are order of magnitude to compare different systems and to identify the benefits and drawbacks of each system.

However this tool only takes into account greenhouse gases emissions and their impact on Global Warming potential. If we would like do have a complete analysis, we would have to do a Life Cycle Assessment (LCA) completed with economic and social study. We could identify the most respectable environment systems and optimise the worst ones.

5 ACKNOWLEDGEMENTS

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(173) GREENHOUSE GAS EMISSIONS FROM HOME COMPOSTING IN PRACTICE

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EXECUTIVE SUMMARY

Home composting is a wide-spread treatment option for biological household waste in Sweden. According to the Swedish Waste Management association (Avfall Sverige, 2011), 60 000 tonnes of compostable waste were home composted in 2010. This represents a rather large portion (10 %) of all the household waste treated biologically. Nevertheless, research on process efficiency and environmental effects of home composting is scarce. The objective of this study was to investigate the influence of different process parameters on greenhouse gas (GHG) emissions (methane, CH_4, nitrous oxide, N_2O and carbon dioxide, CO_2) from home composting under real household management.

Eighteen home composts in use by single households were randomly selected within the city of Uppsala, Sweden. The composts were studied for 13 months from June 2010 until June 2011. The households were requested to keep their normal composting practices. Among the 18 composts there were eight different bin types. Headspace gas, ambient gas and compost material samples were collected 13 times during the study period. At the sampling times, ambient and compost temperatures were measured. The gas samples were analysed by gas chromatography for CO_2, CH_4 and N_2O, and the material samples were analysed for pH, moisture and ash content. Information about compost management and feeding was continuously provided by the households via protocols. The material fed to the composters was mainly kitchen food waste. In some cases garden waste and other biological materials were added. The average amounts of material added to all composts varied from 0.21 to 0.51 kg/day with an extreme of 1.35 kg/day in one of the composts. The temperature in most composts followed the ambient levels. The overall average pH in the composts was 7.2. The moisture was generally high with an overall average of 73 % wet weight.

Average concentrations of CO_2, CH_4 and N_2O were 6430, 28 and 5 ppm above ambient levels, respectively. Both CH_4 and N_2O were represented as ratios to CO_2 in order to estimate and be able to compare emission rates. High moisture content was expected to give higher methane emissions, but such a relationship could not be discerned. Composts with higher temperatures had high ratios of CH_4:CO_2 at some sampling occasions. It was not possible to establish any single variable affecting the N_2O emissions. The average emission rates, given as ratios CH_4:CO_2 and N_2O:CO_2 were 0.38 and 0.15 %, respectively and were relatively low but within the same order of magnitude or lower than those reported in other studies on home composts (Andersen et al., 2010a, Amlinger et al., 2008, Chan et al., 2011). When compared with large-scale composting (Ermolaev et al., 2012, Amlinger et al., 2008, Hermann et al., 2011), home composting seemed to emit less CH_4.

In conclusion, in this study the emissions of CH_4 and N_2O were relatively low. Methane emissions from home composting seemed to be smaller than from large composts. Notably, composts with extremely large amounts of material added, with more frequent mixing or bad aeration, displayed the highest GHG emission rates. This suggests that home compost units should not be too large to avoid intensified process when mixing the compost.

Keywords: composting, greenhouse gas, kitchen waste, methane, nitrous oxide.

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1 INTRODUCTION

Home composting is a wide-spread treatment option for household kitchen waste. In Sweden about 60 000 tonnes of compostable waste or 10.2 % of all the household waste treated biologically were home composted in 2010 (Avfall Sverige, 2011).

During the composting process, aerobic microbial degradation of organic material is the predominating process which after sufficient time results in a stabilized, non-toxic, pathogen free and plant nutrient rich product (compost). Knowledge on process efficiency and environmental effects of home composting is scarce.

Two possible byproducts from composting are the potent greenhouse gases (GHG) methane (CH_4) and nitrous oxide (N_2O). These gases have high global warming potential (GWP 25 and 298, respectively over 100 years) (IPCC, 2007) and therefore are of interest for investigation in many biological ecosystems. The production of both CH_4 and N_2O is coupled to limited oxygen supply which can occur at different phases of composting. Methane production takes place under strictly anaerobic conditions and is conducted by methanogens (Jäckel et al., 2005). The production of N_2O is possible when oxygen is present but limited (Cabrera et al., 1994), and can occur during both nitrification and denitrification (He et al., 2001). While different phases exist sequentially in batch composting, which is common in large-scale applications, in home compost the material is added constantly, causing all the different composting phases to occur simultaneously. Considering that methanogenesis, nitrification and denitrification can occur in parallel but most likely in different areas of the material during home composting, the estimation of parameters affecting the emission sources is difficult.

Moisture affects the composting system in different ways. The liquid phase in the compost material constitutes a solvent for gases, nutrients and metabolic products and thus acts as an important exchange environment for microorganisms (Zhang et al., 2011) Higher moisture content provides better degradation conditions for aerobic microorganisms until a point is reached when too high moisture obstructs ventilation causing reduced oxygen supply which can directly increase CH_4 emission (Amlinger et al., 2008). Generally, material moisture levels of 50 - 60 % are advised for good composting (Gajalakshmi and Abbasi, 2008, Haug, 1993). Lower moisture can cause higher N_2O emissions, as shown by Hwang and Hanaki (2000) who also suggest that the denitrification pathway becomes more complete (resulting in N_2 as the end product) when the moisture content reaches 60 % and above.

The scale of composting is another important factor when trying to understand the influence of moisture and structure on the GHG emissions. For instance Beck-Friis et al. (2000) observed that the emissions from large extensively managed (turning interval two to four weeks) compost windrows were much higher (3 times more N_2O and 4 times more CH_4) than from small heaps managed more intensively (turning interval one to two weeks).

The temperature influences the activity of methanogens, nitrifiers and denitrifiers in the composting process (Hellmann et al., 1997), thus affecting the CH_4 and N_2O emissions. Amlinger et al. (2008) compared different composting systems and showed that higher CH_4 emissions were observed in systems where temperatures reach above 40 - 50 °C, while N_2O appeared at temperatures below 45 °C. Jiang et al. (2011) also found a correlation between temperature and CH_4 emissions. Higher temperatures (above 60-65 °C) can reduce N_2O and CH_4 by inhibiting the microorganisms, but this will also reduce the overall degradation rate and composting performance (Hellmann et al., 1997, Beck-Friis et al., 2001).

Considering that CH_4 is produced by strictly anaerobic microorganisms, composts with low oxygen supply have been shown to give the highest emissions (Zeman et al., 2002, Szanto et al., 2007, Jiang et al., 2011). Zeman et al. (2002) reported the normal range of methane emissions during the compost cycle to be between 1-4 % of initial C. However, Beck-Friis et al. (2000) measured CH_4 concentrations of up to 47 % in the pore gas, which means that the emission could be higher if the CH_4 produced inside the compost would not be oxidized by methanotrophs in other areas of the piles. Beck-Friis et al. (2003) and Jiang et al. (2011) showed that the CH_4 emissions decrease rapidly with increasing ventilation and oxygen supply.

Attempts to couple different factors like C:N ratio, temperature, pH, compost process stage and microbial development to fluxes of N_2O have been made in multiple studies (Jiang et al., 2011, de Guardia et al., 2010b, Andersen et al., 2010b), but in conclusion, they suggest that various multiple factors in combination seem to cause peaks at different times.

The objectives of this study were: (1) to investigate the emissions of CH_4 and N_2O from home composting of food waste in relation to the degradation of organic material (expressed as levels of CO_2), (2) to analyse possible influences on this relation by different process parameters and management factors and (3) to compare our results with others in the field of food waste composting.

2 METHODOLOGY

2.1 Selection and monitoring of composts

GHG emissions and material samples from 18 composts in southern Uppsala (Sweden) were analysed between June 2010 and June 2011. The composts were maintained by private households who agreed to join the study. Having chosen every third record from the municipality registry book of home composts in a few areas of Uppsala close to Ultuna, the participants were initially contacted by phone. In Sweden it is compulsory to register your home compost before treating kitchen waste.

The households were requested to continuously fill in the protocols to provide information about compost management and feeding. The management protocols were periodically distributed and collected. The requested information included details on type and amount of waste added (kitchen waste, garden waste or additional material), dates of additions, mixing and emptying. The households were requested to keep their normal composting practices.

2.2 Gas sampling and analysis

Compost gas and ambient air were sampled from each compost on 13 occasions. It took several days to sample all 18 composts. At samplings, compost gas was collected from under the unopened lids of the compost units and injected in 20 ml vials with gas tight rubber septa, using a 60 ml syringe connected to a 50 cm plastic tube (3 ml void volume). Samples of ambient air, around 5 meters from the compost bins, were taken using a similar procedure. At each sampling, four vials were filled with compost gas and two with ambient air.

Half of the vials were analysed for CH_4 and N_2O using a Perkin Elmer Clarus 500 (Perkin Elmer, Waltham, MA, USA) gas chromatograph (GC) equipped with capillary columns (Elite-Plot Q), and flame ionization (FID) and electron capture (ECD) detectors. The other half of the vials were analysed for CO_2 and CH_4 using another GC system Perkin Elmer Clarus 500 - Arnel (Perkin Elmer) equipped with two packed columns in series (Heysep N69/80 and Molecular sieve 13x 45/60), followed by thermal conductivity detector (TCD) and FID detectors. Headspace auto samplers TurboMatrix 110 (Perkin Elmer) fed the gas samples to the GC systems.

In all results shown, ambient concentrations have been subtracted from the compost gas concentrations. Since the objective was to analyse the GHG emission factors in relation to degradation, the GHG emissions are presented as ratios of CH_4:CO_2 and N_2O:CO_2, calculated subtracting the ambient gas concentrations.

2.3 Material sampling and analyses

At each sampling occasion the temperature of the ambient air and the compost material was measured using a k-type thermocouple (Probe type 80104, Thermo-Electra, Pijnacker, the Netherlands) attached to a Fluke 52 K/J thermometer (Fluke Co., Everett, WA, USA). The temperature inside the compost was measured at the depth of 13 cm in the centre of the bins. Material samples were collected after gas sampling and temperature measurements were taken. Material (~200 ml) was gently extracted from the centre of each bin at approximately 10 cm depth with minimal disturbance to the rest of the pile. Samples were collected into plastic bags, closed and analysed within the same day (in rare cases, frozen at -20 °C on arrival to the lab and analysed later) for moisture content, pH and subsequently for ash content. Part of the material was thoroughly mixed with deionised water in volume proportion 1:5 in 50 ml plastic tubes and after 30 minutes pH was measured using “inoLab Level 1” meter (WTW GmbH & Co. KG, Weilheim, Germany) with SenTix 81 electrode (WTW). The moisture content was determined by drying 3 x 100 g material in ceramic or aluminium cups at 105 °C for 14 hours (Eklind et al., 2007). Ash content was determined after heating the dried and weighed samples in an oven at 550 °C for four hours (Eklind et al., 2007).

3 RESULTS

3.1 Bin types

All bins in the study were closed and insulated. Among the 18 composts there were eight different bin types, and 9 of the bins were of the same type (Greenline Master, Table 1). Four bin models consisted of two separate and identical compartments. All compartments in use were sampled. More details on each bin type can be found in Johansson (2011).

TABLE 1 **Compost bin types, compost abbreviations and volumes. The bins were static, unless stated otherwise.**

Bin types	Abbreviations	Volume, dm^3	Note
Greenline Master	gm1-gm9	375	Most common type
Handy	hy1	300	
Mully	my1, my2	300	
Mullbänken	mb1, mb2	2×200	Two compartments
Jora	ja1	2×135	Rotating, 2 compartments
Greenline Master Junior	gr1	200	
Gröna Johanna	gj1	300	
Corrocomp	cc1l, cc1r	2×115	Rotating, 2 compartments

3.2 Protocol data

The protocols from the households contributed information with different levels of detail about amounts, composition and dates of wastes addition. Most households specified the volumes added, while some provided the weights instead. All volumes were converted to weights using appropriate densities (Ermolaev et al., 2011).

The material added to the composts was mainly food waste, but in some cases it also consisted of garden waste, structural amendment and other waste. Both the added amounts of food waste and the total added amounts, including garden waste and structural amendment, fluctuated greatly between composts and sampling occasions (Figure 1).

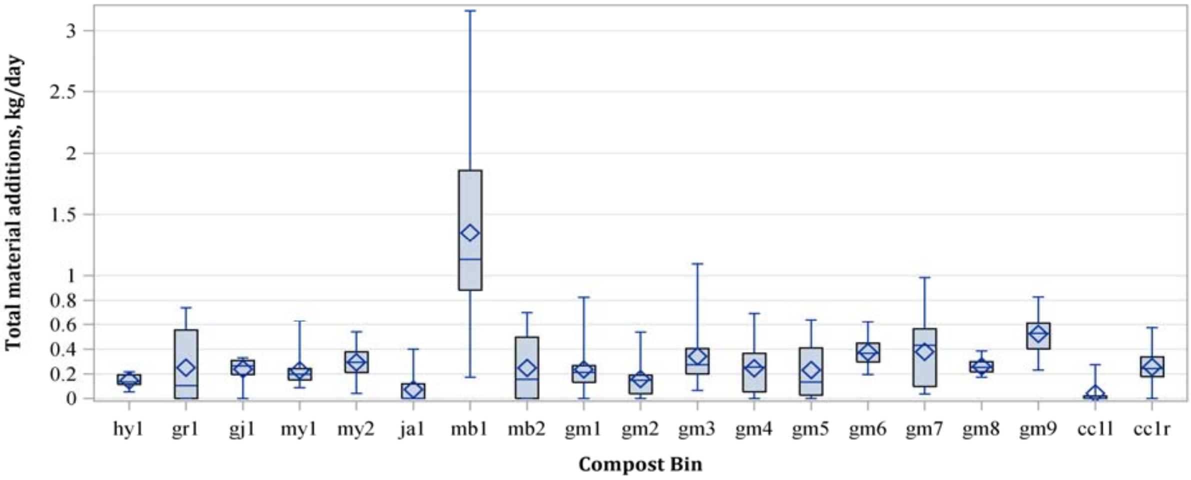

FIGURE 1 **Variation of total material additions to the composts between each sampling occasion. Legend: interquartile range (IRQ, ⊟) with median value (−) inside boxes, ◊ mean value, ⍰ whiskers include the range of all values above and below IRQ.**

The amounts of material additions varied over the year from 0.21 kg/day average for all composts (at sampling occasion 10 in March) to 0.51 kg/day (at sampling occasion 6 in October). Compost mb1 received substantially larger amounts of waste (on average 1.35 kg/day) and was mixed frequently (on the average once every 5 days).

3.3 Temperature and material properties

In 12 of the 18 composts, the temperature in the compost responded proportionally (data not shown) to the change in ambient temperatures, which varied from 29 °C in the summer to -15 °C in the winter. At least at one of the sampling occasions, the remaining 6 composts (mb1, mb2, gm1, gm6, gm7, gm8) had a higher temperature than what could be expected due to this proportionality (Figure 2). In the compost mb1, larger amounts of waste were added compared to the other composts (Figure 1) and this compost was mixed frequently. The compost gm7 had a higher temperature, possibly caused by mixing close upon the sampling 10 (37 °C inside compost and 2.5 °C outside), but the amount of waste added was similar to previous sampling occasions.

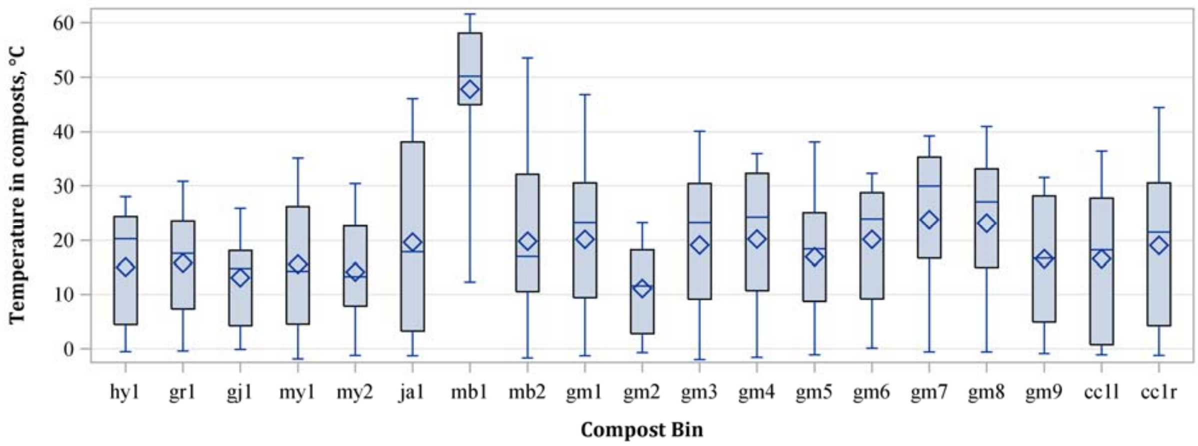

FIGURE 2 **Temperature variation (n = 13) in 18 compost bins within the one-year study period. See Figure 1 for explanation of symbols.**

The average overall pH in the compost materials during the study period was 7.2. The pH, however, varied between the sampling occasions and at sampling 9 (January) the average pH value for all composts was 5.7 while it was 7.7 at sampling 2 (July). The average pH in 10 out of the 13 sampling occasions was above 7 and in one compost, ja1, it was always above 7 (Figure 3). In 6 of the composts, the pH never dropped below 5.

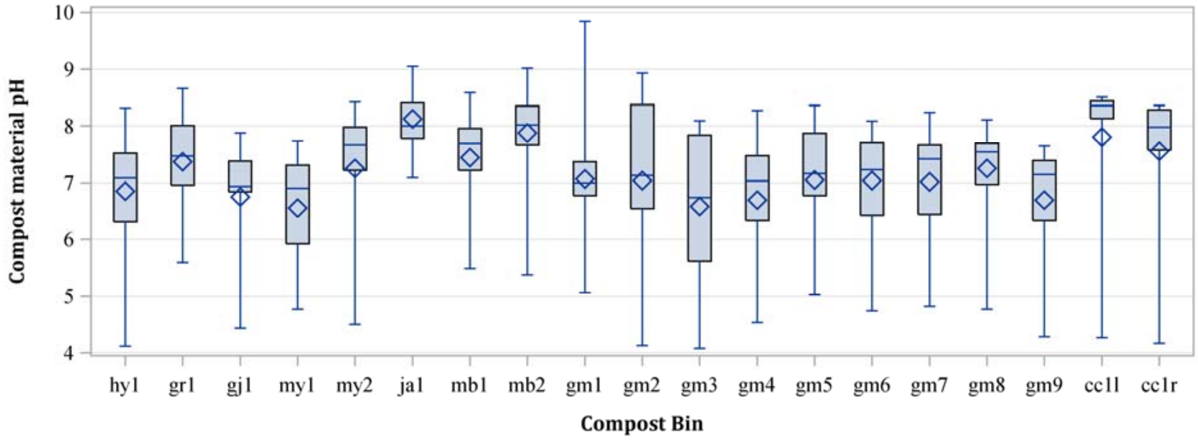

FIGURE 3 **Distribution of pH values (n = 13) in 18 compost bins within the one-year study period. See Figure 1 for explanation of symbols.**

The moisture was generally high with an overall average of 73 % of wet weight, but varied greatly. All measured values ranged between 28 % (sampling 2, compost gm6) and 92 % (sampling 5, compost gm2). In 88 % of all collected samples the moisture content was above 60 %, and in 68 % of the samples the moisture content was higher than 70 % (Table 2).

TABLE 2 **Average, minimum and maximum moisture content values (n = 18) at different sampling occasions for all compost bins in the one-year study. Date format: day/month.**

	Moisture content at each sampling occasion, % wet weight												
Date	*2/6*	*24/6*	*26/7*	*16/8*	*29/9*	*21/10*	*10/11*	*29/11*	*19/1*	*7/3*	*4/4*	*10/5*	*16/6*	--
Sampling	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*	*12*	*13*	**All**
Mean	69	70	69	70	76	76	74	78	74	75	75	71	71	**73**
Min	40	28	36	37	56	64	37	65	56	56	65	48	51	**28**
Max	87	85	85	82	92	87	91	86	86	86	87	81	85	**92**

3.4 Emissions

The average ambient CO_2, CH_4 and N_2O levels were 397, 1.7 and 0.3 ppm, respectively, for all sampling occasions. Average concentrations of CO_2, CH_4 and N_2O in compost gas were 6430, 28 and 5 ppm above ambient levels, respectively. The CO_2 concentrations varied, after adjustment for the ambient level, between 69 ppm (compost mb2, sampling 3) and 139000 ppm (i.e. 13.9 % in compost cc1r, sampling 6) (Figure 4). The absolute highest concentration of CH_4 was 730 ppm during sampling occasion 13 for the compost cc1r. Multiple instances when CH_4 concentration was near ambient level were also observed (Figure 4). For N_2O the highest concentration observed was 127 ppm in compost gm3 (sampling 12). The instances with the lowest N_2O concentrations (below 0.01 ppm) were observed in composts my2, gm2, gm3 and (sampling 9, 6 and 10, respectively).

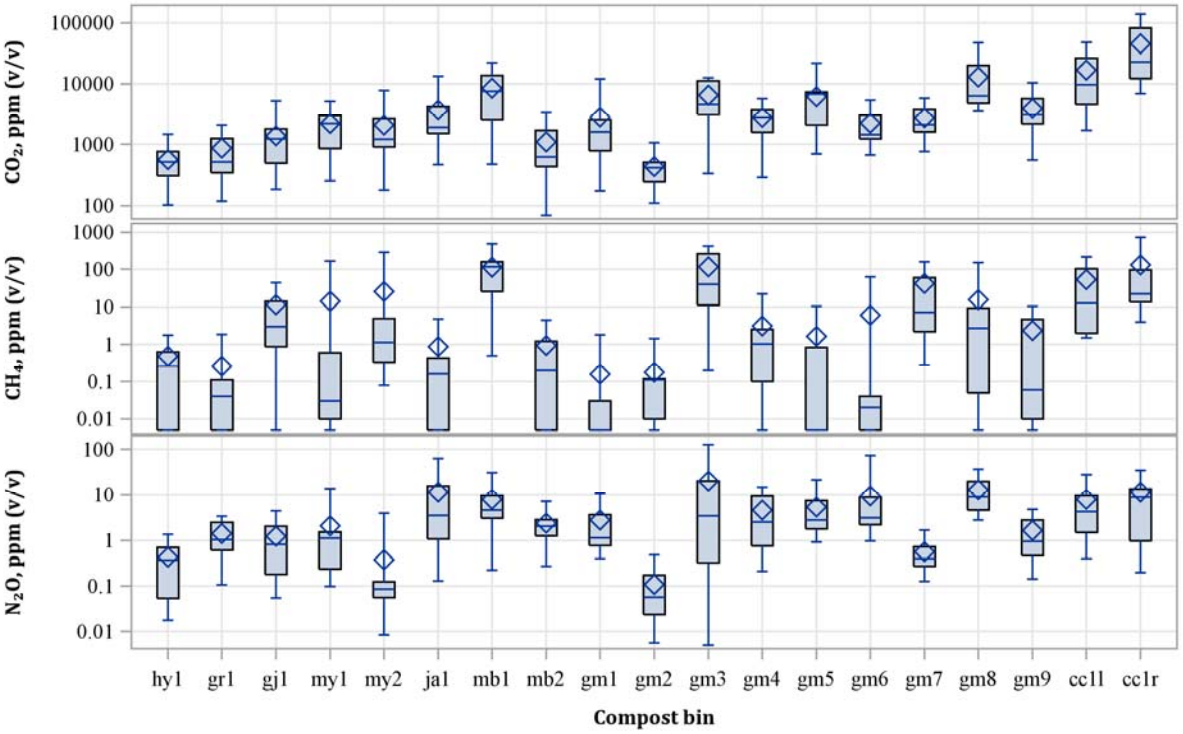

FIGURE 4 **Compost gas concentrations of CO_2, CH_4 and N_2O (n = 13) after adjustment for the ambient level in 18 compost bins within the one-year study period. Negative and zero values have been replaced with 0.005 ppm. Note that the Y axes have logarithmic scales. See Figure 1 for explanation of symbols.**

The CH_4:CO_2 ratio averaged at 0.38 % and the N_2O:CO_2 ratio at 0.15 % (Table 3). The averages were affected by a low number of measurements with extreme observations and therefore the median values were much lower (0.04 % and 0.07 %, respectively). Emissions ratios varied largely both among different composts and among the different sampling occasions. The CH_4:CO_2 ratio varied seasonally the pattern was similar to that of the ambient temperature. No such co-variation with temperature was noted for N_2O:CO_2 (data not shown).

TABLE 3 **Average, median and mean values (n = 234) for CH_4:CO_2 and N_2O:CO_2 ratios expressed as % of CO_2 concentration in the one-year home compost study.**

Ratio	Mean (%)	Median (%)	Min. (%)	Max. (%)
CH_4:CO_2	0.376	0.037	-0.024 (sampling 5, mb2)	5.078 (sampling 5, mb1)
N_2O:CO_2	0.147	0.067	-0.004 (sampling 10, gm3)	2.413 (sampling 12, gm6)

4 DISCUSSION

Using the ratios of CH_4:CO_2 and N_2O:CO_2 in this study allowed evaluation of emission factors which are related to degradation rates of the organic material. Such ratios provide a simple method to compare the emission factors of different composts (Table 4) and composting systems, even if scales and activity intensities are very different. For instance in cc1 rotating bin the lid was closed tightly and had very small ventilation holes comparing to other bins, which might indicate that the high levels of CO_2 observed in this bin (average 16800 ppm for cc1l and 45200 ppm for cc1r, Figure 4) were due to insufficient aeration. This assumption was also supported by the fact that similar temperatures and amounts of added material in other bins resulted in far lower CO_2 concentrations. Using the ratio method in this case, made it possible to establish that the CH_4 emission from cc1r (in relation to degradation rate, Figure 5) was among the highest compared to the other composts. In the case of compost mb1, the reasons for the high CO_2 observed were different and caused by higher addition rates (Figure 1) and overall higher temperatures (Figure 2). Nevertheless the ratios showed similar tendency of emissions being higher for CH_4 than in other composts (Figure 5). In both cc1r and mb1 the N_2O:CO_2 ratios were not different to the values noted in other composts, suggesting that high CO_2 concentrations had a lesser effect on N_2O emission.

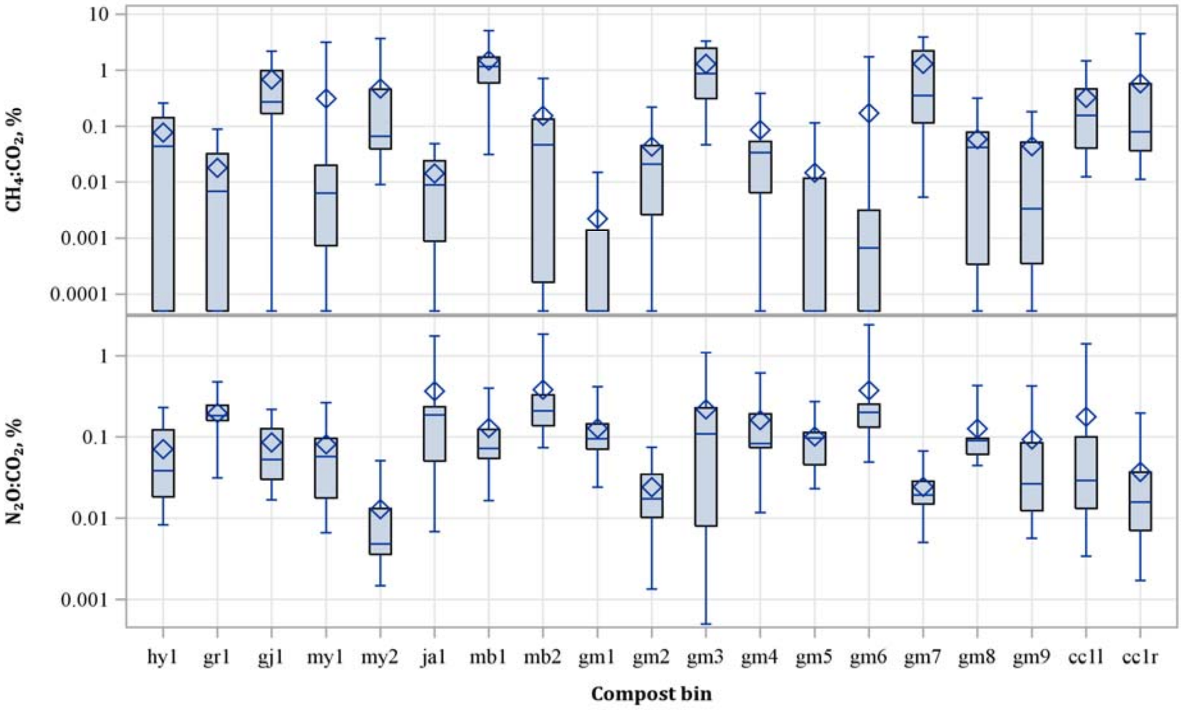

FIGURE 5 **Variation of CH_4:CO_2 and N_2O:CO_2 ratios (n = 13) for the 18 different compost bins within the one-year study period. Negative and zero values have been replaced with the lowest values estimated in each range. Note that the Y axes have logarithmic scales. See Figure 1 for explanation of symbols.**

The emissions (expressed as ratios) were relatively low for both CH_4:CO_2 and N_2O:CO_2 (mean 0.38 and 0.15 % and median 0.04 and 0.07 %, respectively) and within the same order of magnitude or lower than those reported in other studies on home composts (Table 4). A study by Andersen et al., (2010a) on home composting in Denmark reported emissions corresponding to ratios in the range 0.75 - 4.43 % for CH_4:CO_2 (depending on aeration rates) and 0.18 - 0.21 % for N_2O:CO_2. Amlinger et al. (2008) reported emissions equivalent to a CH_4:CO_2 ratio of 2.32 % and N_2O:CO_2 of 0.19 % for home composting in Austria. Chan et al. (2011) investigating home composting alternatives in Australia, measured emissions from aerobic and vermicomposting systems. Based on their results the calculated ratios were 0.68 and 0.18 % for CH_4:CO_2 and N_2O:CO_2, respectively, for the aerobic home composting system (Table 4). In the comparisons above the differences in emissions observed could have been due to differences in experimental set-ups, but comparing the overall average values of ratios should provide a good representation of emission factors and GHG performance of the respective composting systems.

The CH_4:CO_2 emission ratios measured in our study are similar or lower, compared with those reported from large-scale batch composting. Amlinger et al. (2008) reported emissions corresponding to a CH_4:CO_2 ratio range of 0.94 - 1.19 % for large-scale composting, while Ermolaev et al. (2012) observed ratios of 0.1 - 1.5 %, with a mean of 0.75 % for large-scale composting of food waste. In a life cycle assessment (LCA) study, Hermann et al. (2011) estimated that the methane emission can be relatively high depending on the type of composting, aeration and waste composition (Table 4). Chan et al. (2011) suggested, in agreement with our results, that CH_4 emission in their aerobic home composting experiments was smaller than in large-scale systems.

TABLE 4 **Gas emission ratios reported in the literature from studies of different compost systems (recalculated into gas volume ratio, averaged for different treatments). Figures are mean values with the median values given in parentheses.**

Source	**System and waste type**	**CH_4:CO_2, %**	**N_2O:CO_2, %**
Present study	Home composting (containers) of FW, GW and SA	0.38 (0.037)	0.150 (0.067)
Amlinger (2008)	Backyard composting (containers) of FW, GW, SA	2.32	0.193
Andersen (2010)	Home composting (hi mixing) of FW	4.43	0.180
	Home composting (low mixing) of FW	1.11	0.214
	Home composting (no mixing) of FW	0.75	0.179
Chan (2011)	Home composting (aerobic bins) of HW, GW and SA	0.68	0.177
	Home composting (vermicomposting) of HW, GW and SA	0.78	0.074
Ermolaev (2012)	Covered Windrows (low aeration) of BW	0.73	0.004
	Covered Windrows (hi aeration) of BW	0.76	0.001
De guardia (2010a, 2010b)	Reactor (different aeration rates) of HW	0.39 - 0.58	0.000
	Reactor (different aeration rates) of FW	0.00 - 0.58	0.00 - 0.009
Hermann (2011)	Industrial composting (various types, LCA study) of BW	0.52 - 14.9	0.11 - 0.181
Amlinger (2008)	Windrow composting of BW	0.94	0.096
	Mechanical biological treatment of BW	1.19	0.087

FW= food waste, GW= garden waste, SA= structural amendment, HW= household waste, BW= biological waste.

The average N_2O emission in our study was in the same range as in other home composting studies (Chan et al., 2011, Andersen et al., 2010a, Amlinger et al., 2008). The median value was lower than the averages in all of these studies, indicating that few incidents of high emissions had a high influence on the overall average N_2O emission. When compared with large-scale studies, essentially all N_2O emission ratios are lower compared with the ratios from home composting (Table 4).

The main reason suggested for increased GHG emission rates is insufficient aeration as discussed in several studies (Zeman et al., 2002, Szanto et al., 2007, Jiang et al., 2011). In our setup the oxygen (O_2) concentrations were not measured, but could be assumed to be low when CO_2 was high (due to air gas composition) and in our data high CO_2 (and assumingly the low O_2) was one of the parameters influencing the CH_4 concentration (Figure 4). This was demonstrated in the compost cc1r, which had one of the highest average concentrations of CO_2 and CH_4. Similar relationships were also observed for the bins gm3 and mb1, which also were among the bins with the highest CO_2 and CH_4 levels throughout the samplings (11-12 for gm3 and 4-5 for mb1).

High moisture content is in many cases discussed as one of the main reasons for bad aeration causing higher GHG emissions (Hao et al., 2001, Jiang et al., 2011, Amlinger et al., 2008). In our study the moisture content was generally high, (Table 2) with an average of 73 %, compared with the large-scale food waste composting which had a moisture content range of 40 - 65 % (Ermolaev et al., 2012). Furthermore, all home composts were naturally aerated, while the large-scale food waste compost was mechanically aerated. In spite of this, the median CH_4:CO_2 ratio in the present study was lower than for the large-scale compost. Possible reasons for this might be the small scale of the home composts, which means that the distance for oxygen to diffuse from the surface to the degradation zone is far shorter than in large-scale compost. If less anaerobic zones are formed inside home composts, this could explain the lower ranges of CH_4 emissions comparing to large-scale systems. Similar effect was observed by Beck-Friis et al. (2000) who noted less CH_4 produced in smaller compost heaps. Moreover in that study it was also shown that higher CH_4 concentrations were detected deeper in the compost heaps then at the edges.

This could be due to growth of methanotrophs on the surface layers of the composts as discussed by Jäckel et al. (2005) and could be one possible explanation for the low ratios seen in our study. In addition to the negative effect of moisture on aeration, the positive effect on denitrification has been discussed by Hwang and Hanaki (2000). Several studies have attempted to couple poor aeration to increased N_2O (Jiang et al., 2011, de Guardia et al., 2010b, Andersen et al., 2010b) and in conclusion, they suggest that multiple factors, varying throughout the process, in combination can cause peaks at different times. Moreover, Hao et al. (2001) suggested that higher aeration can lead to more N_2O being released.

Temperature is another influential variable and Amlinger et al. (2008) in their study suggest that compost temperatures below 45 °C could favour N_2O production, while temperatures above 40-50 °C facilitate CH_4 emissions. In our study the temperature in the composts was generally low (Figure 2). This could be another reason for higher N_2O:CO_2 compared to large-scale processes, which usually have higher temperatures. In just a few composts in our study the temperature was higher, which demonstrated a higher level of activity, especially during the winter months. Composts mb1, mb2, gm6, gm7 and gm8 had higher temperatures with high ratios of CH_4:CO_2 at some sampling occasions.

The temperature in the compost is directly affected by the rate of the overall degradation process, which is dependent on aeration and availability of energy in the material. For compost mb1 the reason for the higher temperature was most likely the large amounts of material added and frequent mixing. The mb1 bin size was similar to the other bins in our study, but its ventilation capacity was obviously not dimensioned for supplying sufficient amounts of oxygen for such an intense degradation process.

Mixing is often discussed as essential for good aeration (Beck-Friis et al., 2000, Szanto et al., 2007, Amlinger et al., 2008). Considering the high moisture content of the substrate in our study, we expected lower methane emission ratios for composts turned more frequently. We could however not find any such relationship. Furthermore, Andersen et al. (2010a) showed that more frequent mixing of home composts resulted in 8-12 % higher CH_4 emissions, partly attributed by the instant release of trapped CH_4 at the mixing. A similar effect was observed by Ahn et al. (2011) during dairy manure pile composting, when the GHG emission was 20 % higher in turned piles. Different mechanisms could be responsible for such effects of mixing including increasing decomposition rates consuming the oxygen and destruction of the methanotrophic layer on the surface.

5 CONCLUSIONS

In the current study the emission rates of CH_4:CO_2 were low and within the same range as compared to other home compost studies. Even though the home composts in our case had higher moisture content than the majority of large-scale composts, the CH_4:CO_2 ratios were lower in comparison to the large-scale composts. On the other hand, the N_2O:CO_2 ratio for home composts seem to be larger than for large-scale composts.

Composts with extremely large amounts of material added, being mixed very often and having bad aeration, showed the highest GHG emission rates. Thus, not mixing the home composts and avoiding overloading are the important recommendations for reducing the GHG emission.

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(226) CARBON FLOWS OF WASTE BIOMASSES APPLICABLE TO AGRICULTURAL SOILS IN FINLAND

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EXECUTIVE SUMMARY

Agriculture, industry and municipalities produce waste biomasses that are not adequately exploited. Carbon (C) and nutrients in the waste biomasses are usually linked and although nutrient flows have been assessed from the global scale to the farm level, C flows have been studied to a lesser degree. Recycling C from waste biomasses to crop production has the potential not only to improve soil productivity, but most importantly also to mitigate climate change. The C sequestration (CS) potential depends not only on the quantity of C but also on the decomposition rate of the C compounds in soil.

Agrifood waste biomasses have substantial potential for recycling C to crop production based on their volumes, location and safety. In addition, a large proportion of waste biomass from the paper and pulp industry is exploitable in agricultural soils as organic amendments. This is of interest because the paper and pulp industry is the biggest exploiter of biomass resources in Finland and therefore the volume of waste biomasses produced is high. The CS capacity of these biomasses is presumably high also due to the slow decomposition of wood fibre in soil. The examined biomasses are almost all exploited in some way and only little is used as landfill. However exploitation in terms of CS is inadequate.

The aim of this study was to assess current C flows (volumes and routes) in waste biomasses that could be applied to agricultural soils in Finland. The results of the study contribute to determining the associated CS potential in agricultural soils. The effects of organic amendments on emissions, soil productivity and replacing fossil inputs in crop production are not considered.

Data for the C flows were collected from administrative databases and supplemented with additional statistical and primary data. Data on C quality derives from national and international research literature.

This study shows that the C cycle in agrifood waste biomasses is open in Finland. C in organic material is not fully returned to crop production and it could be better exploited. The most substantial C flow in the agrifood system is represented by manure, which though exploited is done so inadequately in terms of CS when used continuously on animal farms. Municipal biowastes and sewage sludges are currently mostly used in landscaping. Although CS is possible, the CS benefits of organic amendments in crop production are not realized. In the paper and pulp industry fibre sludges are mostly disposed of by incineration or landscaping in landfills. Incineration of high moisture biomasses seldom produces any energy, but it could consume energy.

Only a small proportion of the examined waste biomasses applicable to agricultural soils (2%) is exploited in agriculture. The annual unexploited C potential is 0.26 Mt in addition to 0.66 Mt of C in manure that is also inadequately exploited. The C included in inadequately exploited waste biomasses applicable to agricultural soils is the equivalent of 57% of annual greenhouse gas emissions from the Finnish agriculture sector. Apart from the agrifood waste biomasses, fibre sludges from the paper and pulp industry have substantial potential for CS, especially when C quality is taken into account.

Despite the positive effects of C on crop production and the environment, it´s recycling in the agrifood system has not been pursued until recent years during which there has been increasing awareness of climate change. This is demonstrated by the open unexploited flows of waste biomass C. The waste biomasses applicable to agricultural soils that originate from outside the agrifood system are overlooked to an even greater extent. Furthermore C quality seems to be a key parameter when evaluating the potential of CS in soil. Consequently, there is a need for increased understanding of C dynamics when adding various waste biomasses to agricultural soil.

1. INTRODUCTION

Agriculture, industry and municipalities produce waste biomasses that are not adequately exploited. Carbon (C) and nutrients in the waste biomasses are usually linked and nutrient flows have been assessed at spatial scales from global to farm level, but C flows have been studied less (Antikainen et al. 2005, Smil 1999, Smil 2000). Recycling C from waste biomasses of the agrifood system to crop production is particularly important for soil productivity. Restoring C in the soil also sequesters it and mitigates climate change (Freibauer et al. 2004, Lal 2004). Waste biomasses have C sequestration (CS) potential, which depends on the decomposition rate of the C compounds. Global annual CS in agriculture is estimated to be 400-1200 Mt, which annually accounts for 5-15% of fossil-fuel-derived C emissions on a global basis (Lal 2004). In Finland, the paper and pulp industry is the biggest exploiter of biomass resources and therefore the volume of waste biomass from the sector is high. A substantial share of the waste biomasses, so-called fibre sludges, can be applied to soil as organic amendments, and the CS capacity of the biomasses is presumably high due to slow decomposition of wood fibre in soil (Phillips et al. 1997, Fierro et al. 1997). The waste biomasses exploited in agriculture have to meet the biological, chemical and physical quality standards stipulated by legislation or handling requirements. To meet the requirements, some of the waste biomasses have to be treated by composting, hygienisation or dewatering. In addition, in some cases the usability and agricultural and economical value of waste can be increased by the treatment. Although all the studied biomasses are currently exploited and only a minority is landfilled, exploitation is inadequate from the standpoint of CS. The CS potential of waste biomass in agricultural soils is determined by the volumes and quality of the C to be recycled, and by the C dynamics in soil. The aim of this study was to assess the current C flows (volumes and routes) in waste biomasses applicable to agricultural soils in Finland.

2. MATERIALS AND METHODS

The quantity of manure was calculated based on statistical data for animal numbers and manure production coefficients of animal types in Finland (Finnish Ministry of Agriculture and Forestry 2002, MMM Tike 2011). Manure properties and the assumptions regarding shares of slurry and solid manure were based on reports and statistics (Grönroos et al. 2009, Viljavuuspalvelu 2012). Only manure produced by confined animals was included, while manure produced during the pasture period, and not available for enhanced recycling, was excluded. The data for the municipal waste biomasses were based on biowaste and sewage sludge production statistics (Statistics Finland 2011a, Ministry of the Environment 2011). Although these biomasses are exploited as treated products, such as composts, statistical data for the end products are not available. The quantity of fibre sludges from the paper and pulp industry was derived from the statistics of the pulp and paper industry, which summarize all fibre sludge quantities (Finnish Forest Industries Federation 2012). However, their properties vary. The C content of paper and pulp industry waste biomasses was based on primary data from the industry. The exploitation pathways of the waste biomasses were based on statistical data (Rantanen et al. 2008, Ministry of the Environment 2011, Statistics Finland 2011, Finnish Forest Industries Federation 2012). Data on biowaste exploitation in agriculture are rare but were assumed to be equivalent to the exploitation rate of sewage sludge because the masses are usually processed together.

3. RESULTS AND DISCUSSION

Animal manures in Finland are mostly used as fertilizer on the animal production farms (Table 1). Manure as an organic amendment is found to be more effective in storing C in the soil when added to cereal-based rotations compared with grass-based rotations and to soils with low soil organic C content. Grassland in rotation also tends to increase soil C content in comparison with cereal cultivation (Holeplass et al. 2004). Therefore, manures can be regarded as being inadequately exploited on animal farms where continuous manure application and grasslands have already affected soil C content. Municipal biowastes and sewage sludges are currently exploited after anaerobic digestion (AD), composting, or both, mostly in landscaping. Although CS is possible, the benefits of CS of organic amendments for crop production are not realized. Municipal wastes are considered as untreated, although their exploitation in agriculture usually requires treatment, which can cause C losses. According to a study on composting household biowaste with various litter amendments, C losses ranged from 11 to 61% of initial C, depending on the amendment and C/N ratio of the composted mixture (Eklind & Kirchmann 2000).

C losses from composting sewage sludge are supposed to be negligible due to a low and easily degradable C content after anaerobic digestion, which is the common pretreatment for sewage sludges in Finland.

Waste biomasses from the food industry were excluded from the assessment because they are already effectively exploited as feed and fertilizers or treated together with municipal biowastes (Huhtinen et al. 2007). Crop residues were regarded as being exploited in this assessment because they are left on the field or are used for animal bedding, the main uses of crop residues in Finland. The pressure for using crop residues for incineration is low because of their low energy density and high moisture content when harvested late in the season. Fibre sludges are mostly disposed of in the paper and pulp industry by incineration or landscaping in landfills (Finnish Forest Industries Federation 2012). Incineration of such high moisture biomasses seldom produces any energy, although it could consume it. In addition, it increases the load of difficult-to-exploit ashes. Minor amounts of fibre sludges are already used in agriculture. With reference to the data presented in Table 1, municipal waste biomasses and fibre sludges exploited in agriculture are the equivalent of 2% of all examined waste biomasses (0.94 Mt). Consequently, the annual unexploited C potential is 0.26 Mt, plus the inadequately exploited 0.66 Mt of C in manure. Some change in the quantity of waste biomasses in the future is expected to take place. Manure and sewage sludge volumes might remain constant, but the quantity of collected household biowaste is increasing. In 2007 there was 0.28 FM Mt of biowaste and it was assumed that it would increase by 0.22 FM Mt by 2016 due to more effective source separation and restrictions on using biowaste for landfilling (Huhtinen et al. 2004). The amount of paper and pulp industry waste is highly dependent on the production of paper products and therefore difficult to predict. In the last few years, several paper and pulp production units were closed in Finland (Finnish Forest Industries Federation 2012).

TABLE 1 **Potential (Mt a^{-1}), total organic carbon (TOC) (%), carbon (C) (Mt a^{-1}) and current exploitation (%) of the waste biomasses applicable to agricultural soils in Finland. DM: dry matter.**

BIOMASS	Mt DM	TOC, %	C, Mt	Material use, %	Energy use, %	Landfil-ling, %	Use in agricul-ture, %
Manures	1.77	37.2[1]	0.66	100.0[2]	0.0[2]	0.0[2]	100.0[2]
Biowaste	0.11[3]	43.4[1]	0.05	98.2[3]	0.1[3]	1.7[3]	12.0
Sewage sludge	0.15[2]	35.5	0.05	80.0[4]	0.0[4]	6.0[4]	12.0[4]
Fibre sludges	0.42[5]	44.3	0.18	23.7[5]	73.0[5]	3.2[5]	3.6

[1]Sonesson & Jönssön 1996, [2]Ministry of the Environment 2011, [3]Statistics Finland 2011a, [4]Rantanen et al. 2008, [5]Finnish Forest Industries Federation 2012

The C included in inadequately exploited waste biomasses applicable to agricultural soils is the equivalent of 57% of the annual 1.6 Mt C equivalent greenhouse gas emissions from the Finnish agriculture sector (Statistics Finland 2011b). Based on the literature, there are differences in the C quality of the studied waste biomasses. The rate of decomposition is dependent on the C structure and C/N ratio of the incorporated material. Adding unstable raw organic material, like manure or AD digestates, can even have a negative effect on soil C, whereas a fully composted mature amendment with humic substances can supplement the soil C content (Bernal et al. 1998, Garcia et al. 1991). Lignin-based C in fibre sludges is resistant to decomposition for at least a few decades (Fierro et al. 1999). This study shows that the C cycle of waste biomasses from the agrifood system in Finland is open. C is either not fully returned to crop production or its exploitation is inadequate. The most marked C flow in the agrifood system is manure, which is not adequately exploited with respect to CS. Treated municipal wastes are small in volume, but their role may increase when the quality of C is considered. Apart from the agrifood waste biomasses, fibre sludges from the paper and pulp industry proved to have substantial potential for CS, especially when the quality of C is taken into account.

4. CONCLUSIONS

Despite the positive effects of C on crop production and the environment, recycling of C in the agrifood system has not been targeted until relatively recently times, during which there has been increasing awareness of climate change. This is demonstrated by the open unexploited flows of waste biomass C. Waste biomasses originating from outside the agrifood system that can be applied to agricultural soils have been even more overlooked.

Furthermore, C quality appears to be a key parameter in evaluation of the potential CS in soil. Consequently, there is a need for increased understanding of C dynamics when adding various waste biomasses to agricultural soil.

On-going research of the authors will result in a better understanding of the CS potential of various waste biomasses applicable to agricultural soils through analyses and dynamic modelling of soil C changes through organic amendments.

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Session 4

(11) DIGESTATE AND COMPOST QUALITY – CASE STUDIES AT ANAEROBIC DIGESTION PLANTS WITH THE BTA PROCESS

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EXECUTIVE SUMMARY

The key for the successful anaerobic digestion of biowaste and/or municipal solid waste is an efficient removal of the impurities before the digestion step. This is not only indispensable for the reliability of the process and therefore the plant availability, but furthermore for the quality of the produced digestate or final compost. With the existing developments within the EU, the quality of the digestate / compost has become a focus of attention.

The most efficient way to remove these impurities is in the wet medium. This leads to a better quality of the "washed" rejects and to a higher recovery of the digestible organics to the anaerobic digestion. The BTA Process offers a corresponding technical solution which has proven its flexibility, robustness and reliability in over 40 plants installed worldwide since 1990 for different types of waste (e.g. food waste, commercial waste, biowaste or municipal solid waste).

The BTA® Process consists of two steps: an hydromechanical pre-treatment in which non-biodegradable contaminants bigger than 10 mm are separated as a "heavy" and a "light" fraction in the BTA Waste Pulper and as a "grit" fraction smaller than 10 mm in the BTA Grit Removal System. The clean organic suspension is pumped to the methanization step for its energetic valorization. Generally the digestate is dewatered in a solid-liquid separation step. The liquid phase is widely recirculated to the hydromechanical pre-treatment while the solid phase is aerobically stabilized by composting.

In the present paper, the further processing and destination of the digestate for the following reference projects is presented:

- In Castelleone (Italy), where biowaste and commercial waste are digested as co-substrates together with agricultural residues, the produced digestate is spread on the fields of nearby farms
- In the Biowaste Methanization Plant Kirchstockach (Germany) the dewatered digestate is mixed with green waste to produce quality compost
- In the Biowaste Methanization Plant Ieper (Belgium) the dewatered digestate is further processed to produce quality compost
- In the MBT Plant Valorlis (Portugal) the dewatered digestate is further processed to produce final (stabilized) compost

Finally, analysis carried out during the start-up and the operation tests for the MBT Plant Valorlis (Portugal), which treats Municipal Solid Waste, showed that the parameters for the content of physical impurities (content of plastics and of stones, glass splitters) are met.

These are examples which prove that it is possible to obtain a quality product out of the digestate without an extensive refining step but a simple screening for the partial recovery of structure material, demonstrating the advantage of an efficient removal of impurities in the upfront of the anaerobic digestion.

1 INTRODUCTION

1.1 Background

The key for the successful anaerobic digestion of biowaste and/or municipal solid waste is an efficient removal of the impurities before the digestion step. This is not only indispensable for the reliability of the process and therefore the plant availability, but it also has a direct impact on the quality of the produced digestate, the post-treatment and the products obtained from the digestate. With the existing developments within the EU, the quality of the digestate / compost has become a focus of attention.

1.2 Research objectives

Physical impurities in the digestate or derivative products can be reduced either prior or after the anaerobic digestion step. The advantage of an concept with an up-front removal of the contaminants is that also process risks for the anaerobic digestion process like scum formation, sedimentation, clogging, etc., inherent both to wet and dry digestion plants, can be avoided.

The best way to efficiently remove these impurities from the organic fraction with a minimal loss of digestible organics is in the wet medium. The impurities are removed from the digestible organics in the wet medium: The BTA Process offers a corresponding technical solution which has proven its flexibility, robustness and reliability in over 40 plants installed worldwide since 1990 for different types of waste (e.g. food waste, commercial waste, biowaste or municipal solid waste). Chapter 2 gives a short description of the BTA Process.

Chapter 3 discusses case studies for different BTA Plants are presented in which different types of waste are treated and in which different uses of the digestate and derivative products are given. All these plants have in common that due to the hydromechanical pre-treatment simple mechanical refining steps are sufficient to meet the legal requirements concerning the physical impurities in the material.

2 BTA PROCESS

The BTA® process is the original of hydromechanical waste treatment. It comprises two central steps:

- the hydromechanical pre-treatment
- the subsequent biological step towards anaerobic digestion

2.1 Hydromechanical Pre-Treatment

The hydromechanical pre-treatment facilitates efficient removal of impurities as well as complete transferring of digestible organic components into an organic suspension.

Within the BTA Waste Pulper the feedstock is added to pre-filled process water in order to separate the waste mixture into fractions by taking advantage of natural buoyancy and sedimentation forces. Thus, heavy materials are fed aside and light materials are skimmed off. Moreover, non-soluble digestible organic components are reduced to fibres by shearing forces and brought into suspension. In total, three fractions are effectively separated.

- Digestible organic fraction
- Light fraction: plastics, foil, textile, wood, etc.
- Heavy fraction: glass, metal, stones, bones, batteries, etc.

Having passed the BTA Waste Pulper, the organic suspension still contains sand and fine impurities < 10 mm, which are safely removed by the BTA Grit Removal System. This reliably ensures protection of downstream plant components from wear, silting up, sediments, and obstruction.

2.2 Anaerobic digestion

In this way, a cleaned and homogeneous organic suspension is pumped to the anaerobic digestion. The suspension is temporarily stored in a suspension tank. This way, the processing stage is decoupled from the digestion itself, keeping the latter independent from the working cycle of the waste reception unit.

The organic fraction is digested within the fermenter, generally as a one step, wet digestion under mesophilic conditions between 35 – 38°C. The digesters are fully mixed with compressed biogas injected with gas lances. In this way, a high homogeneity in the digester is ensured, avoiding the formation of clusters in which the anaerobic processes are not fully implemented and therefore allowing for the production of a clean and homogeneous digestate.

2.3 Further treatment of the digestate

The further treatment of the digested substrate can be adjusted according to the respective project. Generally, a solid-liquid separation is foreseen. The solid phase (ca. 30 % dry matter) is perfectly suitable for the further composting process and the production of stabilized quality compost. The liquid phase is widely utilised as process water and is retained within the cycle. This results in a decreased freshwater consumption as well as a reduced effluent amount.

3 CASE STUDIES FOR THE FURTHER USE OF THE DIGESTATE

3.1 Castelleone, Italy

A part of the digestate in the output of the digesters is pumped to a thickening unit consisting in a rotating drum grid. The thickened phase and the rest of the digestate are pumped to four storage tanks, the obtained centrate is used for the hydromechanical pre-treatment. The first tank is covered and connected to the biogas collection system to collect the residual biogas and to reduce the spread of odours. Finally, the liquid digestate is pumped from the storage tanks to the farms nearby, where it is used as fertilizer in the farm spreading it on the fields.

While the physical composition of the liquid digestate is ensured by the hydromechanical pre-treatment foreseen for the co-substrates (biowaste and commercial waste), the sanitary conditions are met by an sanitation step (70°C, 1 hour) of the suspension previous to the anaerobic digestion. The third requirement, the particle size < 12 m, is met by the fact that the organic suspension produced in the pulper is pumped through an integrated sieve with 12 mm mesh size.

3.2 Kirchstockach, Germany

The anaerobic digestion in Kirchstockach is a two step process preceded by a solid-liquid separation. While the liquid stream is pumped to the methane reactor, the solid phase is pre-treated in a hydrolysis step. The hydrolysed suspension is again dewatered. The liquid phase is pumped again to the methane reactor, while the solid hydrolysis residue, contaminant free and low in salt, is partly used for the production of quality soils and the remaining part is aerobically composted together with yard trimmings.

The quality of the hydrolysis residues is periodically monitored (see also Table 2). Tests confirm that the requirements from the fertilizer ordinance (Düngemittelverordnung) are met and that the material is considered free of impurities and especially suitable for fertilizing and soil improvement. The Fachvereinigung Bayrischer Komposthersteller e.V. has just confirmed in January 2012 the quality mark for compost to Ganser Entsorgung GmbH& Co. KG for the Kirchstockach plant.

3.3 Leper, Belgium

In Belgium, the biowaste is characterized by a high amount of green waste. For this reason, a trommel sieve with a mesh size of approx. 150 mm was installed in Ieper. Cutted green waste is mixed with the digestate cake obtained in the solid-liquid separation step (dewatering with screw presses). The filtrate goes back to the hydromechanical pretreatment where it is used as process water. The mixed material is composted in seven tunnels equipped with a forced aeration. After two weeks, during which the sanitation of the digestate is achieved, the material is moved to the curing building, where the material is further stabilized in six windrows. With a topturner, the windrows are frequently turned in order to stabilise the product. After approx. 6 additional weeks the matured compost is screened with a mobile trommel screen with a mesh of 10 mm. The final compost is brought to theovered storage building to await sale to its end-user.

Table 1 shows the average values from analysis carried out periodically from January 2009 till January 2012 on the matured compost. The standard from VLACO were met all along the years, allowing to sell the obtained product as quality compost in Flanders. Furthermore, the standard NF44-051 is met, requirement for the marketeability of the compost in France.

3.4 Valorlis, Portugal

The dewatered digestate obtained in Valorlis is mixed with structure material and then subjected during three to four weeks to a first intensive composting step in a closed composting hall with eight composting boxes with forced aeration. During this time the sanitation of the digestate is achieved at temperatures over 60°C. The stabilization of the material is executed in eight aerated windrows in a separate hall, which is covered, but not closed. Finally, in the refining step the raw compost is screened in a mobile trommel screen to partially recover the structure material.

Despite of the higher content of impurities in the input material (in VALORLIS 50.000 ton/year of Municipal Solid Waste are treated), analysis carried out in the frame of verification tests during the test operation showed that the following guaranteed levels for the physical impurities where observed (see also Table 3):

- Content of impurities bigger than 2 mm: less than 1% in weight of dry matter
- Content of stones bigger than 5 mm: less than 1% in weight of dry matter

Nevertheless, it can be observed that the values for heavy metals partially are above thresholds. These levels can only slightly be influenced during the process and are widely a function of the content of these elements in the waste.

For this reason in some European Countries aerobically post-treated digestate from Municipal Solid Waste per se cannot be used as quality compost.

4 CONCLUSIONS

The quality of the produced digestate or of the products obtained from the digestate is very important for the success of a methanization project. The above case studies, which reflect different types of anaerobic digestion plants for different types of waste, prove that it is possible to achieve the parameters concerning physical impurities necessary to obtain a quality product out of the digestate without an extensive mechanical post-treatment.
Thus, the efficient removal of impurities in the upfront of the anaerobic digestion by the BTA Hydromechanical Pre-treatment not only ensures a reliable anaerobic process, but at the same time allows for a successful valorization of the digestate or subsequent products as quality products.

Figure

FIGURE 1 **Simplified scheme of the BTA Process**

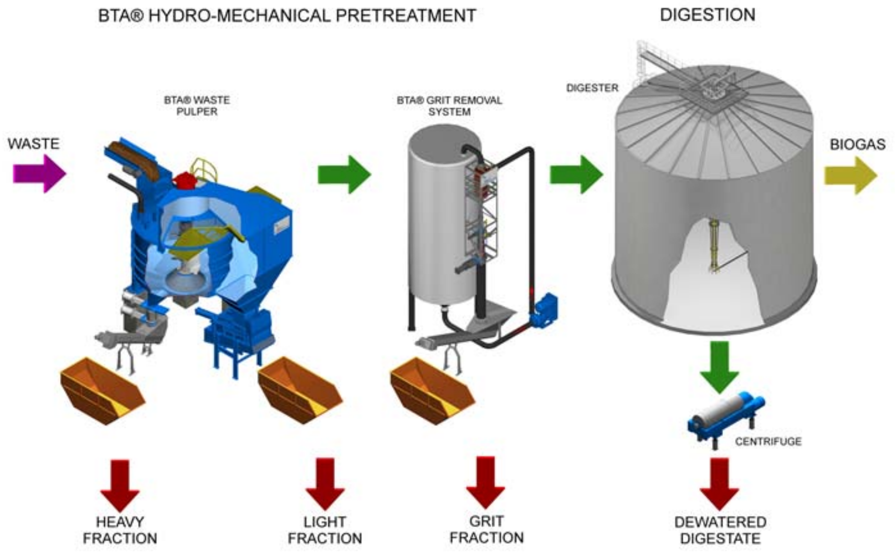

FIGURE 2 **Input material to Kirchstockach (biowaste with high content of green waste), Castelleone (FORSA and commercial waste) and Valorlis (Municipal Solid Waste) (from left to right)**

FIGURE 3 **Dewatered digestate from AD Plant Ieper**

FIGURE 4 **Maturation hall in AD Plant Ieper**

TABLE 1 **Analysis results from matured compost in AD Plant Ieper**

Parameters	**Unit**	**Average**	**Threshold by VLACO**
Dry matter	%	56,87	Min 50%
Organic dry matter	% / VM	25,19	Min 16%
N total (%/VM)	% / VM	0,83	
E.C. (1/5)	µS / cm	1.657,87	
pH (H2O)	-	8,51	6.5-9.5
Chlorides	mg / L	1.142,00	
NH4-N	mg / L	179,97	
NO3-N	mg / L	63,78	
Impurities > 2mm	%	0,20	Max 0.5%
Stones > 5mm	%	0,62	Max 2%
Germinable seeds	# / L	0,00	Max 1
Maturity degree	°C	27,92	
Class	-	IV-V	IV or V
Heavy metals			
Cadmium (Cd)	mg/ kg DS	0,50	Max 2
Chrome (Cr)	mg / kg DS	23,99	< 70
Copper (Cu)	mg / kg DS	53,33	< 90
Lead (Pb)	mg / kg DS	50,32	< 90
Nickel (Ni)	mg / kg DS	11,53	< 120
Zink (Zn)	mg / kg DS	270,60	< 300
Arsen (As)	mg / kg DS	4,36	< 15
Mercury(Hg)	mg / kg DS	0,13	< 1

TABLE 2 **Characteristic analysis results from hydrolysis residue in AD Plant Kirchstockach**

Parameters	Unit	Value	Threshold for compost
Dry matter	%	33	Min 45%
Organic dry matter	% / VM	73	Min 20%
pH (H2O)	-	7,8	
Impurities > 2mm	%	0,0	Max 0.5%
Stones > 5mm	%	0,0	Max 5%
Germinable seeds	# / L	0	
Maturity degree	°C	26	
Class	-	V	
Heavy metals			
Cadmium (Cd)	mg/ kg DS	0,3	< 0,75
Chrome (Cr)	mg / kg DS	36,3	< 70
Copper (Cu)	mg / kg DS	58,1	< 70
Lead (Pb)	mg / kg DS	21,7	< 75
Nickel (Ni)	mg / kg DS	12,3	< 25
Zink (Zn)	mg / kg DS	150	< 200
Mercury(Hg)	mg / kg DS	0,2	< 0,7

TABLE 3 **Analysis results from matured compost in MBT Valorlis**

Results	**Valorlis 1: matured compost**	**Valorlis 2: matured compost**
Moisture content (%fm)	32,9%	36,4%
Volatile Solids (% dm)	49%	37%
Impurities >2mm total (% dm)	0,8%	0,72%
pH	7,7	
Salt content (g KCl/l)	5,86	
Thereof glasses (% dm)	0,18%	0,09%
Thereof plastics (% dm)	0,58%	0,58%
Thereof metal (% dm)	0,04%	0,05%
Stone content >5mm (% dm)	0,21%	0,07%
Stability degree		V
Oxygen consumption –AT4 (mgO2/g dm)		2,49
Plant tolerance (% fm)		98
Rate of germinating seeds (plant/l)		0
Salmonella		negative
E. coli (cfu/g)		4
Facel streptococcus (cfu/g)		< 3
Pb (mg/kg)	163	
Cd (mg/kg)	1,15	
Cr (mg/kg)	61,2	
Ni (mg/kg)	40,5	
Hg (mg/kg)	0,4	
Cu (mg/kg)	139	
Zn (mg/kg)	312	

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(56) MODELLING OF SPROUT SUPPRESSION OF *FUSARIUM SP.* AND DON TOXIN IN ANAEROBIC HYDROLYSIS

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EXECUTIVE SUMMARY

In recent years the rainfall and moisture conditions, the long term surface water cover and low temperature are resulted together favourable conditions for microbial fungi in Hungary. The species of *Fusarium* strain are hibernated on the soil and vegetation residues and next year their conidia attack different parts of the plants. *Fusarium graminearum* is the most important agricultural pathogens in Hungary. This fungi decreases cereal quality (for example maize), furthermore may cause yield loss. Due to its toxin production, the fungi could have dangerous animal and human pathogen effect. Mainly trichothecene type mycotoxins can be found in Hungary; especially the DON (deoxynivalenol) toxin has detected and appeared on cereals (Szécsi and Bartók, 1995).

Fusarium sp. infected maize can be used as alternative biogas base material may be an efficient and environmentally friendly way of utilization of agricultural wastes. In the case of preliminary fermentation fungi wasn't detected in the bioreactor after 30 days, although exponentially increasing growth of the DON toxin was measured during the fermentation period, in correspondence with. The mycotoxins are mainly produced if some kind of stress affects the fungi physiology. Therefore, not only the reproduction of the fungi, but the amount of mycotoxin increase exponentially parallel to the decrease of O_2 level during the hydrolysis process in the case of the first phase of biogas fermentation.

According to studies, several factors influence the toxin production of the fungi for example the presence of water activity, pH, carbon and nitrogen sources in the log and lag phase of fungal growth (Gardiner et al., 2010; Zhang and Wolf-Hall, 2010). The reliability of the obtained data was tested by HPLC/UV-VIS method, but the toxin content of the fermented material has not been able to measure precisely by this technique. Therefore model experiments were set to evaluate the applicability of HPLC/UV-VIS technique for toxin detection in a biogas fermentation system.

The experiments were set in mesophilic (38 ° C) and thermophilic (55 ° C) circumstances, furthermore both in aerobic and anaerobic conditions. The amount of DON toxin is analyzed in base material and in hydrolised product by ELISA and HPLC/UV-VIS. However, the microbes could cause also eco-toxicological hazard, so seed germination assays were also investigated in pot experiments by maize seeds to examine the effect of the fungi and the toxin found in the fermented biogas material.

The seed germination assay was carried out at 22±2 ° C, and soil moisture was kept on minimal water capacity, and the water was supplied with boiled deionized water. Germinations of each treatment were compared to controls (%). The root length of germs (mm) were measured and variance analyse of Tukey was applied to detect the significant differences between treatments in order to detect the ecotoxicological effect of mycotoxins.

1 INTRODUCTION

Fusarium graminearum Schwabe (teleomorph: *Giberella zeae*/Schwein/Petch) is the most important pathogenic filamentous fungi (Kövics, 2000). The presence of fungi degrade the quality of the maize, they produce mycotoxins which has harmful effects on both livestock and human health (Eifler, 2011; Placinta et al., 1999). In our experiments *F. gramniearum* is selected, because this is the most important pathogen in Hungarian climate. The in vivo experiments have shown that one of the most common trichothecenes is the deoxynivalenol (DON, vomitoxin) (Szécsi, 1994). Alternatively, *Fusarium* and mycotoxin contaminated maize can act as a base material for biogas utilization. However, the toxin content should be examined in the fermentation end product. The advantage of pot germination is that the toxic effect is more visible in the seed germination and development of young plants, because pathogens cause developmental abnormalities or changes in the revenue of the crop (Radulescu and Negru, 1971).

2 METHODOLOGY

2.1 Anaerobic hydrolyses

Because of the physical properties of the end product of biogas fermentation, DON toxin content can not be analyzed by analytical methods. Therefore we carried out anaerobic hydrolysis model experiments for the DON toxin determination. The anaerobic hydrolysis was made at mesophilic temperature (38 °C), for 4 days, in a half litre glass jar with gas bag in laboratory incubator shaker (Heidolph Unimax 1010). We used only mesophilic temperature, because this fungus is not viable under termophilic conditions (*Figure 1*).
The experimental settings were the followings:

- control: 250 ml sterile deionised water and 12.5 g of maize grits,
- the test: 240 ml sterile deionised water, 12.5 g of maize grits and 10 ml *Fusarium graminearum* inoculum.

2.2 Fungus strain and media

The medium was inoculated with 6.4 x 10^7 conidia of *F. graminearum*. Inoculum was carried out on Papavizas selective medium (pH 5.2) (Szécsi és Mesterházy, 1998) and we used *Fusarium graminearum* strain (NCAIM F.00970). The medium consisted of 15 g peptone (Biolab), 1 g KH_2PO_4 (VWR), 0.5 g $MgSO_4.5H_2O$ (VWR), 0.5 g bile salts (Biolab) 20 g agar (VWR), 0.5 g pentachloronitrobenzene (PCNB, Sigma), 50 mg chlortetracycline hydrochloride (AppliChem), 100 mg streptomycin sulfate (AppliChem) per liter. 250 ml of the medium was dispensed in a glass tube, inoculated with macroconidia of *Fusarium* and incubated with shaking (190 r.p.m.) at 25° C for 7 days under aerobic conditions.

2.3 Deoxynivalenol toxin determination

The deoxynivalenol concentration was determined from the maize, the liquid phase of the hydrolyzed products and the maize germs at the Regional Biogas Plant Central Laboratory of Nyírbátor with enzyme-linked immunosorbent assay (ELISA) method. DON concentration was determined using the Ridascreen Fast R5902 DON ELISA kit (R-Biopharm Hungary Kft.) according to the manufacturer’s instruction.

2.4 Pot germination assay

For each pot experiments, 1 kg quartz sands (Scharlab), LG 33.95 maize hybrids’ seeds and the hydrolysed products were used. The infected and *Fusarium* free hydrolysed products were separated into two phases (suspension and liquid phase) with centrifuge (1500 g for 15 min). In this way 4 treatments were used in pot experiment. In the control case deionised water was used. We determined the optimal water quantities and every day it was corrected with sterile deionised water. The seeds were planted 3-4 cm deep (20 seeds per pot). The germination was at room temperature for 22 ± 2 ° C, in all cases 6 days. After the germination time we measured the length of developed roots. According to the European Commission 2006/576/EK specifies in the animal nutrition products the DON toxin allowable limit is 8 μg/kg. We repeated the pot germination assay with standard 10 μg/kg DON toxin. The germination assay performed similarly, but we sprayed the toxin on the surfaces of the maize.

3 RESULTS AND DISCUSSION

Before the anaerobic hydrolysis we determined the cell number on the Papavizas medium. *Figure 2* shows the macroconidia of *F. graminearum*. After the 4 days incubation period, we could not detect the presence of the mould. The macroconidia are not visible in the hydrolysed end-product, due to the enzymatic degradation of maize (*Figure 3*). In the treatments, concentration of DON did not changed in the control case (input maize: 1.08 µg/kg, control liquid phase: 1.05 µg/kg, *Fusarium* liquid phase: 1.18 µg/kg). Because the fungus destroyed in the anaerobic model experiment, the toxin concentration did not increased. Concerning the DON concentration there were no differences between treatments in the model experiments.

Regarding germination ability, more maize germinated in the control liquid phase, than in the control. In the case of the control suspension and the *Fusarium* liquid phase treatments the germination rate was the same as in the control. In case of *Fusarium* suspension 6% less seedlings was observed compared to the control. Based on the classification of the average root lengths (Németh, 1998) the control liquid phase and the suspension stimulated the germination of maize. The *Fusarium* suspension's effect was less significant. Due to the effect of *Fusarium* liquid phase, the root lengths were reduced, although this treatment is non-toxic based on the toxicological assessment category. General statistic test and the variance analysis of Tukey were also carried out between treatments (*Table 1*). Significant difference was found between the control, and the control liquid phase, the control suspension. The reason for this is probably that the presence of nutrients, coming from maize grits. Significant differences were not determined among the infected and *Fusarium* free treatments. The 10 µg/kg concentration DON toxin was not toxic effect on the maize germination. In the maize germs was the toxin content under detection limits (0.222 µg/kg). So the toxin in the soil can not penetration to the maize seeds and can not get to the livestock and humans. In summary, the results showed neutral effect of DON toxin on the maize germination.

4 CONCLUSIONS

Although the *Fusarium graminearum* destroyed in the biogas production and in the anaerobic hydrolyses, the DON toxin remained in the end-product and after utilization on lands the toxin can affect the germination of maize. The hydrolyzed product was observed to possess small inhibitory effect on germination in the case of *Fusarium* and DON-containing liquid phase. Further study was needed to identify the effect of DON toxin on the germination with germination tests using different concentrations of standard DON toxin. Under the pot germination assay the DON toxin can not penetration to the maize seeds. According to the ELISA test the toxin was not present in the maize germs. In the soil there are a lot of bacteria for example *Agrobacterium-Rhizobium* group. These bacteria can degrade the DON toxin in the soil. Further study is needed to identify the effect of toxin-degrading of these soil bacteria.

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Figure

FIGURE 1 **Anaerobic hydrolyses model experiment**

FIGURE 2 **Microscopic view of *Fusarium graminearum* (40x)**

FIGURE 3 **Microscopic view of enzymatic degradation of maize (40x)**

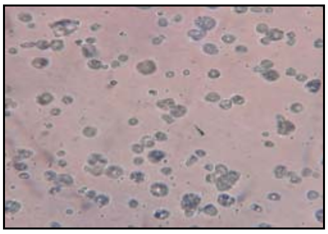

TABLE 1 **General statistics and the qualification the average root length in the percentage of the control (%)**

Sample name	**The average root length in the percentage of the control (%)**	**Qualification**	**Median**	**Modus**	**Mean**	**Standard deviation**
Control	100.0	non-toxic	12.35	13.60	12.09^{a}	3.99
Control suspension	142.1	stimulating	18.80	18.00	17.18^{b}	4.76
Fusarium suspension	122.0	stimulating	14.00	14.70	14.75^{ab}	4.86
Control liquid phase	135.0	stimulating	17.00	15.20	16.33^{b}	5.29
Fusarium liquid phase	116.6	non-toxic	13.45	15.00	14.10^{ab}	3.26
Control	100.0	non-toxic	10.90	10.90	10.12^{c}	2.86
DON toxin	112.0	non-toxic	11.45	11.50	11.33^{c}	2.46

* the same alphabetical index signs no significant difference ($P < 0.05$)

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(138) EFFECT OF LIGNOCELLULOSICWASTE DRY GRINDING ON METHANIZATION AND MICROBIAL POPULATIONS

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EXECUTIVE SUMMARY

Municipal solid waste (MSW) methanization is promising as it enables mass reduction of waste and methane recovery. Methanization has been implemented at the industrial level for decades, in particular on liquid effluents. However, solid waste substrates raise specific issues; process and performance optimization is still required. In the present work, we focus on the MSW lignocellulosic fraction, representing in France ~50% of the MSW mass (papers, cardboards, sanitary textiles, green waste). It is a renewable and high energy potential fraction. It is however recalcitrant to degradation due to its insolubility and a highly complex and heterogeneous structure. Current MSW methanization processes probably do not yet fully exploit this energy potential.

The work aims at assessing and understanding the effects of specific dry mechanical treatments applied to model lignocellulosic substrates (flat cardboard and organic wheat straw) on their subsequent methanization. Such treatments may induce contrasted effects by impacting the particle size and their other properties, including structural characteristics. The total accessible surface area may for instance be modified, as well as the release and accessibility of growth substrates and inhibitors. Model systems were established and degradation and methane production were assessed over time.

To test the effect of particle size and grinding method, the cardboard and the straw were finely grinded using distinct techniques (cutting -, ball -, impact -, and jet mills) with various settings and durations. Some techniques are expected to alter only the particle size, while others must in addition affect its crystallinity, such as ball milling. For the cardboard, a total of 3 distinct fine particle batches were obtained, with median particle sizes ranging from ~52 µm to ~170 µm. For the straw, 5 batches were obtained, with median particle sizes ranging from ~61 µm to ~170 µm. To study the methanization, samples of each particle batch were incubated in synthetic medium in replicated mesophilic anaerobic batch microcosms, inoculated with a rich biomass originating from a solid-waste mesophilic methanizer. Substrate degradation and methane production were measured over time by analyzing the biogaz production and composition and by characterizing the liquid phase (dissolved organic/inorganic carbon, volatile fatty acids). Bacterial and archaeal populations responsible for the bioconversion were analyzed by Automated Ribosomal Intergenic Spacer Analysis (ARISA), a commonly used fingerprinting technique.

Results showed that grinding methods tested had no influence on cardboard methanization performances. By contrast, a positive effect of fine grinding was observed for wheat straw methanization. In particular the ball milled fraction associated with the higher particle size reduction led to the highest methane yield. Besides, the structure of the bacterial and archaeal population was affected by the presence of the different pretreated fractions. This suggests that lignocellulose structure modifications lead to the selection of specific microbial communities able to degrade it. This work gives new insights into the solid waste methanization processes by studying the effects of substrates' structural modifications on the microbial populations, increasing the core knowledge required to establish more robust and efficient bioprocesses.

1 INTRODUCTION

Municipal solid waste (MSW) methanization is a promising bioprocess as it permits waste-to-energy conversion as well as waste mass reduction. These two features have lead MSW methanization to the crossroad between energy demand and ecological concerns.

The MSW biodegradable fraction is mainly composed of lignocellulosic materials, representing more than 50% of the MSW mass in developed countries. These materials include papers, cardboards, sanitary textiles and green waste. Similarly to natural lignocellulose, lignocellulosic waste contains cellulose, hemicelluloses and lignin. The precise composition depends on the materials, from the pure cellulose materials such as cotton to the mixed materials such as green waste (Foyle et al., 2007). These natural compounds have a high energy potential but they are not readily degradable under anaerobic conditions. This recalcitrance is due to their highly complex and heterogeneous structure and to the cellulose crystallinity (Mansfield et al., 1999). The presence of lignin especially acts as a barrier to biological attack (Chang and Holtzapple, 2000).

In order to become more easily biodegradable, lignocellulose needs to be pretreated. A lot of strategies have been developed, including physical/mechanical, chemical and biological pretreatments used alone or in combination (Hendriks and Zeeman, 2009; Taherzadeh and Karimi, 2008). All these processes aim to reduce cellulose crystallinity and lignin content and to increase the surface area accessible to enzymatic hydrolysis (Arantes and Saddler, 2011; Chang and Holtzapple, 2000).

Among mechanical means to enhance lignocellulose digestibility, dry grinding processes have many advantages. They do not produce effluents and they are not accompanied with toxic compound release, which can inhibit biological degradation (Palmqvist and Hahn-Hägerdal, 2000). Obviously, the main effect of grinding is the particle size reduction but depending on the technology used, it can often be associated to changes in fine structures such as cellulose crystallinity (Silva et al., 2012). Therefore, grinding processes can largely improve lignocellulose hydrolysis and many studies have been focused on the effects of lignocellulose grindings on its biodegradation. Most of them established a positive correlation between particle size reduction and biodegradability (Sharma et al., 1988; Silva et al., 2012; Yuan et al., 2011), while other studies concluded that there were no significant effect (Pommier et al., 2010; Rivers and Emert, 1988). This contrast mainly depends on the starting material but also on the grinding methods used (Vidal et al., 2011).

In the context of methanization, the microbial compartment plays a crucial role, since total conversion of lignocellulose to methane involves complex microbial communities (Li et al., 2009). Almost all the previously cited studies primarily focused on the total bioconversion potential, generally using pure enzyme cocktails, without examination of microbial populations. Consequently, there is a lack of knowledge on the microbial response to physical alterations of substrates, and as lignocellulose hydrolysis mostly occurs at the substrate surface in anaerobic environments (Lynd et al., 2002; McAllister et al., 1994), a lot of information can be obtained from microbiological studies.

The objective of the present work is to assess the effects of wheat straw - a commonly studied material - and cardboard grinding on their methanization and associated microbial populations.

2 METHODOLOGY

2.1 Substrates and grinding

The wheat straw (WS) (*Triticum aestivum* cv. Apache) provided by Fernand Meaux (St. Jean du Salés, Aveyron, France) and harvested in 2007 had a 8.8% moisture content and the flat cardboard (C) had a 7.2% moisture content. Prior to fine grinding, the substrates were comminuted by cutting milling with a 4 mm screen (WS-CM). For the wheat straw, an intermediate step of sieve-based grinding was added. Fine powders were obtained by ball milling (WS-BM1, WS-BM2, and C-BM), impact grinding (WS-IG and C-IG) and air-jet milling (WS-JM). Different operational conditions were applied to obtain various particle sizes. All grinding methods were performed as described in Silva et al. (2012). A total of 5 batches were tested for anaerobic degradation for wheat straw: WS-CM, WS-BM1, WS-BM2, WS-IG and WS-JM, and 3 batches for cardboard: C-CM, C-BM and C-IG. Analyses of particle size distributions were carried out by laser diffraction granulometry as described in Silva et al. (2012) and the results are given in Table 1.

2.2 Anaerobic incubations

For testing anaerobic digestion on fractions, batch reactors were carried out. These lab-scale reactors consisted of 330 mL glass bottles closed with a screw cap and a septum. Reactors were filled with 0.6 g dry matter (DM) of substrate mixed with a water solution, with a carbonate buffer (0.25 M) and an inoculum.

The inoculum originated from an anaerobic solid-waste digester operated in mesophilic conditions, and was added to each reactor to a final inoculum-to-substrate ratio of 2 volatile solids bases (VS). The water solution was added to reach a final 220 mL volume. Reactors were flushed with N_2 gas to obtain anaerobic conditions and then incubated at 35±2°C. Each reactor was operated in triplicate, except for the reactor CM1 that was operated in duplicate. A blank test without substrate was also conducted.

2.3 Analytical methods

Biogas production was monitored over time by pressure measurements and biogas composition was analyzed with a gas chromatograph as described in Qu et al. (2009). Liquid and solid phase analyses were performed on 1.5 mL samples removed through the septum with a syringe equipped with a needle. The fresh samples were centrifuged at 10 000g for 10 min. Both the pellets (for microbiological analyses) and the supernatants (for chemical analyses) were stored at -80°C. Dissolved organic carbon (DOC) and dissolved inorganic carbon (DIC) concentrations were measured in the supernatant with a BIORITECH 700 analyzer. Volatil fatty acids (VFA) concentrations were measured using a DX 120 Ion Chromatograph (Dionex) with an IonPAc ICE-AS1 column. A degradation yield was calculated at day 31 from the measures of DOC, DIC, CH_4 and CO_2 to estimate the amount of carbon released from the substrate. Cumulative CH_4 and CO_2 production were used for the calculation and DOC and DIC concentrations obtained at day 31 were subtracted from concentrations obtained at day 0. An average carbon content of 44% and 40% were used for wheat straw and cardboard respectively (Julien Grossin-Debattista, personal communication).

2.4 Microbial community analyses

Total DNA was extracted from the pellets sampled at day 0 and day 31 using the PowerSoil™ DNA isolation kit (MoBio Laboratories) according to the manufacturer's instructions. Before amplification, DNA was quantified with the Qubit® dsDNA HS assay kit (Invitrogen). For bacterial and archaeal fingerprinting analyses by Automated Ribosomal Intergenic Spacer Analysis (ARISA), the 16S-23S rRNA intergenic transcribed spacers (ITS) were amplified by polymerase chain reaction (PCR) using the ITSF/ITSReub bacterial primer set (Cardinale et al., 2004) and the 1389F (Loy et al., 2002)/71R (Casamayor et al., 2002) archaeal primer set. PCR conditions were as follows : 1X PCR buffer, 0.75U of Taq DNA polymerase (ThermoScientific), 0.2mM (each) deoxynucleoside triphosphate, 1.5mM $MgCl_2$, 0.2 µM(each) primer and around 2 ng of DNA template in a final volume of 25 µL. The mixture was held at 95°C for 15 min, followed by 35 cycles of 95°C for 30 s, 49°C for 30 s (54.8°C for the archaeal primer set), 72°C for 1 min and a final extension step at 72°C for 10 min. Amplified fragment separation by capillary electrophoresis and profile analyses were performed as described in Fechner et al. (2010), using the R package StatFingerprints (Michelland et al., 2009).

TABLE 1 **Batch reactors and particles' characteristics**

Substrate	**Treatment**	**Reactors**	**Median particle size (µm)**	**Particle size span**[a]	**Specific surface (m²/g)**
Wheat straw	Cutting milling	WS-CM	nd	nd	nd
	Ball milling	WS-BM1	170	3.57	0.105
		WS-BM2	65	5.64	0.323
	Impact grinding	WS-IG	163	2.90	0.1
	Air-jet milling	WS-JM	61	2.91	0.317
Cardboard	Cutting milling	C-CM	171	4.79	0.156
	Ball milling	C-BM	54	5.14	0.339
	Impact grinding	C-IG	61	4.83	0.331

nd: not determined

[a] the particle size span characterizing the particle size distribution was calculated according to Silva et al., 2012, as follows: (d90–d10)/d50, where the d10, d50 or d90 values represent the 10th, 50th and 90th percentile of total volume.

3 RESULTS AND DISCUSSION

3.1 Effects of different grinding methods on methane production and substrate biodegradability

Cumulative methane productions obtained for the different fractions of wheat straw and cardboard are shown in Fig. 1. For all the fractions, a good reproducibility was observed among replicates, and no methane production was detected in the blank test (data not shown). The observed methane production kinetics were similar to those commonly described for lignocellulosic substrate anaerobic degradation (Pommier et al., 2010; Qu et al., 2009). The curves had 3 noticeable phases, a first progressive increase followed by a rapid production and finally a stabilization as degradation neared completion. Similar trends were observed for total biogas production and a plateau was reached after 50 and 41 days for wheat straw and cardboard incubations respectively (data not shown). Methane and carbon dioxide were the major products detected in the gas phase and hydrogen was detected only at very low concentrations (less than 1%).

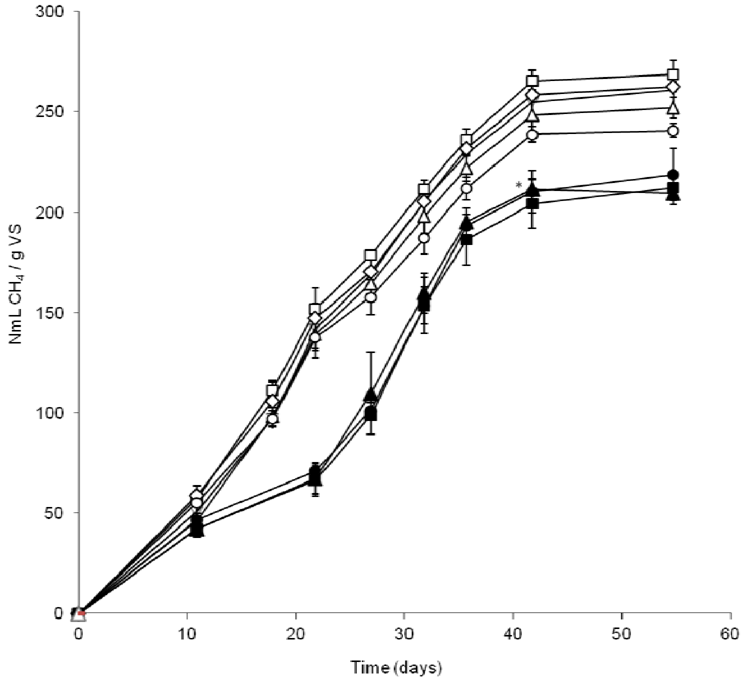

FIGURE 1 **Cumulative methane production during anaerobic batch experiments with wheat straw fractions (empty symbols) and cardboard fractions (filled symbols): WS-BM2 (☐), WS-BM1 (△), WS-IG (▽), WS-JM (◇), WS-CM (○), C-IG (■), C-CM (▲) and C-BM (●). Error bars are the standard deviations calculated for the triplicate samples analyzed at each time point. *Biogas composition was not analyzed at this time point, values were estimated based on the cumulative biogas production at the same time point and the biogas composition at the previous time point.**

The methane production kinetics obtained for all wheat straw fractions were similar (Fig. 1, empty symbols). Average maximum methane yields reached during incubation for each treatment are reported in Table 2. The WS-BM2 reactor showed the highest methane yield with 268.7 Nml CH4/g VS. This value corresponded to a 11.6% increase compared to the lowest methane yield, which was obtained for the 4 mm cut fraction (WS-CM). Values obtained for WS-IG and WS-JM reactors were close to the one obtained for WS-BM2 reactor, 260.9 and 262.6 Nml CH4/g VS, respectively. By contrast, WS-BM1 methane production was slightly higher than the value obtained for the WS-CM reactor. Chemical analyses in the supernatant revealed that acetate and propionate were produced. Unfortunately, sampling intervals did not permit to measure the maximal value for VFA and DOC concentrations, which were probably reached during the first week of incubation. Besides, degradation yield values obtained after 31 days of incubation showed that more substrate was utilized in WS-BM2 reactor than in the WS-CM reactor attaining a maximum of around 50% (Table 2). Hence, it appears that ball milling with reduced particle size had the strongest effect on wheat straw methanization.

This result is in good agreement with those obtained by Silva et al., where ball milled wheat straw showed the higher release of reducing sugars after hydrolysis with pure enzymes (Silva et al., 2012). As ball milling is known to decrease cellulose crystallinity (Chang and Holtzapple, 2000; Silva et al., 2012), it appears that this parameter, in addition to a surface area increase, is determinant in anaerobic degradation, since the performances obtained with similar average particle size with air-jet milling, which did not affect cellulose crystallinity, were slightly lesser. Moreover, the impact grinding treated fraction, with the lowest specific surface, showed similar performances as those obtained with air-jet milling. Since the impact grinding method causes a decrease in cellulose crystallinity which is not accompanied by a particle size decrease (Silva et al., 2012), this latter observation also suggests that decreasing lignocellulose crystallinity is as important as increasing surface accessible area to enhance lignocellulosic substrate anaerobic degradation.

For all the cardboard pretreated fractions, methane production kinetics were also similar (Fig. 1b). However, a different behavior appeared since no differences were observed between the 3 fractions tested in terms of maximum methane yields (Table 2). These values are similar to those obtained in Pommier et al. (2010). However, slightly more substrate was degraded after 31 days in the C-BM reactor than in the C-IG reactor (Table 2). These results also showed that the cardboard is more recalcitrant to degradation than the wheat straw in these conditions. The absence of grinding effects on cardboard methanization may be due to the nature of this material since it is homogeneous. Therefore it appears that the surface increase was not sufficient in the conditions tested to be a determinant parameter for cardboard methanization.

TABLE 2 **Maximum methane yield and degradation yield after 31 days of incubation obtained in batch experiments with the grinded fractions**

Reactors	**Average maximum methane yield (Nml CH_4/g VS)**	**Average degradation yield after 31 days (%)**
WS-CM	240.8 ± 3.3	41.4 ± 1.4
WS-BM1	252.0 ± 5.2	50.4 ± 11.0
WS-BM2	268.7 ± 6.9	52.3 ± 4.5
WS-IG	260.9 ± 6.5	53.0 ± 13.0
WS-JM	262.6 ± 2.4	44.1 ± 4.3
C-CM	209.1 ± 5.5	28.6 ± 5.0
C-BM	218.3 ± 12.8	32.0 ± 1.4
C-IG	212.2 ± 2.6	25.3 ± 1.1

3.2 Effects of different grinding methods on microbial community structure

Microbial community structure was assessed by ARISA for samples taken at day 0 and day 31during the phase of methane production. A first observation showed that profiles were similar at day 0 for all the reactors, for bacteria and archaea respectively (data not shown). The profiles were analyzed by principal component analysis (PCA) and representative biplots from triplicates are shown in Fig. 2a and Fig.2b, for bacterial and archaeal communities respectively. These two analyses primarily show that the cardboard degrading community profiles were well separated from the wheat straw degrading community profiles. Fig. 2a reveals that the bacterial community profile from the WS-CM reactor was very distant from the other wheat straw degrading community profiles. In addition, a more important profile shift occurred in wheat straw containing reactors compared to cardboard containing reactors. Besides, cardboard community profiles were less complex than wheat straw community profiles (data not shown). This difference is possibly due to the presence of a cardboard adaptated community but it cannot be excluded that it is due to a more advanced degradation in wheat straw reactors compared to cardboard reactors. Within archaeal communities the profile shift between day 0 and day 31 is more strongly marked than for bacteria (Fig. 2b). Similar to the bacterial WS-CM reactor community profile, the archaeal WS-CM reactor community profiles were quite distant from community profiles found in the other wheat straw reactors. Taken together, these results suggest that the substrate grindings led to microbial population changes. This is consistent with the general knowledge on lignocellulose degradation, as lignocellulose hydrolysis mostly occurs at the substrate surface in anaerobic environments (Lynd et al., 2002; McAllister et al., 1994)., It seems that not only the lignocellulolytic microorganisms are impacted by the treatments, as shown by the differences within archaeal community profiles, since archaea in anaerobic digesters are generally related to the methanogenesis function and not to the lignocellulolysis function. It is noteworthy that the cutting milling on straw seems to have an impact on the substrate sufficient to lead to the selection of specific microbial communities.

Further studies are needed to determine more precisely which microorganisms are influenced by these grinding processes and what are the colonization dynamics associated that could highlight the preferential degradation of specific particle types.

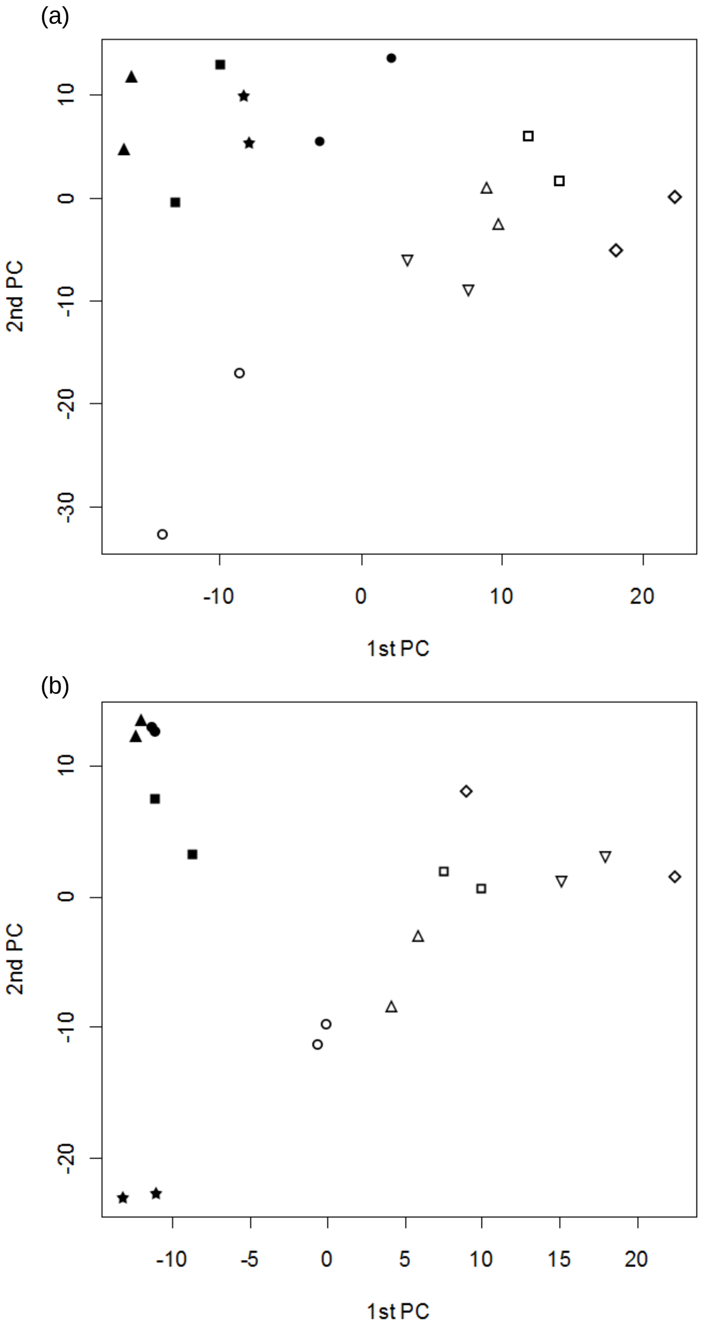

FIGURE 2 **PCA biplots of bacterial (a) and archaeal (b) ARISA profiles. Representative plots obtained for samples, at day 0 (★) and day 31 for WS-BM2 (□), WS-BM1 (△), WS-IG (▽), WS-JM (◇), WS-CM (○), C-IG (■), C-CM (▲) and C-BM (●). The explained variances for, respectively, principal components 1 and 2 are 40,50% and 23,38% (a), 40,12% and 30,01% (b).**

4 CONCLUSIONS

The effects of wheat straw and cardboard dry grinding on their methanization and associated microbial populations were assessed in this study. The experiments showed that grinding methods tested have no influence on cardboard methanization performances. By contrast, a positive effect of fine grinding is observed on wheat straw methanization. Ball milling associated with a high particle size reduction especially lead to the highest methane yield.

On the other hand, microbial communities responsible for the lignocellulose bioconversion to methane undergo structural changes in the presence of the different pretreated fractions. This suggests that lignocellulose structure modifications lead to the selection of specific microbial communities able to degrade it.

5 ACKNOWLEDGEMENTS

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(221) OVERVIEW AND PREDICTION OF ERADICATION OF SALMONELLA SENFTENBERG W775 DURING ANAEROBIC DIGESTION AND PASTEURIZATION PROCESSES

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EXECUTIVE SUMMARY

There is a great demand from public authorities at regional, federal and European level for more research on the biosafety of digestates originating from anaerobic digestion. Literature study shows that the current knowledge on this subject is fragmentary and that biosafety seems to be dependent on the input materials. We aimed to provide policy supporting advice concerning the biosafety of anaerobic digestion and the pre- and post-treatment processes which are used for the processing of a continuously growing variety of agricultural and organic biological waste products.

A first objective was to map out the international literature concerning the hygiene of biological processing in regard to human and animal pathogens. The second objective was the mathematical modelling of the evolution of selected and quantifiable indicatororganisms in function of the processing time. This mathematical modelling formed the basis for the development of a predictive model to evaluate different digestion processes concerning their hygienisation capacity.

At first, an extensive literature study was conducted, from which it seems that spore-foming bacteria can survive mesophilic and thermophilic temperatures and a pasteurization step of 1 h at 70°C. Other pathogens will not cause any problems on the condition that the retention time is long enough. The retention time and the temperature seem to be the most important parameters concerning the inactivation of these pathogens during biological digestion processes. The influence of other parameters like pH and dry matter content are less investigated.

In the next part of our study, the inactivation of pathogens was quantified by mathematical modelling. In this way, it can be predicted when, for example, a 5 $\log_{10}$ reduction of *Salmonella* Senftenberg W775 is reached during thermophilic digestion. This is important as according to the European Regulation (EC) No 1069/2009 other processes might be used as an alternative for the mandatory pasteurization step to digest category 3 material and/or manure, if they are able to reduce 5 $\log_{10}$ of *S*. Senftenberg W775 or *E. faecalis* dependent on the type of waste being digested.

It is not easy to characterize the diversity of waste streams on the presence of different pathogens. Moreover, the constitution of the waste streams, which are digested in a digestion plant, can vary much. Therefore, the emphasis was laid on the risk analysis of the digestion processes itself. The tool BioSafety@Digestion was developed for use within Microsoft® Office Excel®. The tool can be used to determine if a certain digestion process is safe to inactivate certain pathogens. The required data for this, are the temperature of the digester itself and the minimal retention time. The tool is based on the log-lineair microbial inactivation model.

As a conclusion, the temperature and the retention time seem to be the most important parameters concerning hygienisation during biological digestion processes. Moreover, mathematical modelling can be used for the prediction of pathogen inactivation and formed the basis for the development of the tool BioSafety@Digestion that indicates which processes are safe to inactivate certain pathogens.

1. INTRODUCTION

1.1 Litterature overview

Anaerobic digestion is gradually becoming more part of the biological waste processing. In addition to the reduction of volume and weight of wastes, anaerobic digestion provides many attractive features including decreased waste handling and disposal costs and reductions in the number of pathogens (Dumontet *et al*., 1999; Gerardi, 2003; Wright *et al*., 2003; Godfree and Farrell, 2005). The relatively high temperatures and long retention times of anaerobic digesters significantly reduce the presence of viruses, pathogenic bacteria and fungi, and parasitic worms (Gerardi, 2003).

This reduction of pathogens is an extremely attractive feature considering the increased attention given by regulatory agencies and the general public with respect to health risks, represented by the use of digested wastes for agricultural and land reclamation purposes (Gerardi, 2003). Therefore, more scientific research concerning the biosafety of digestates from anaerobic digestion is increasingly being requested (Ryckeboer, 2003; EPPO, 2006,2008).
At first, an extensive literature study was conducted to summarize the international literature concerning the hygiene of biological processing in regard to human and animal pathogens. To search for scientific data, next terms were inserted in Web of Science: 1) anaerobic digestion and pathogen, 2) drying and pathogen, 3) heat or temperature and pathogen and manure, 3) heat or temperature and pathogen and manure and digestion, 4) heat or temperature and pathogen and waste, 5) heat or temperature and pathogen and waste and digestion. In this paper only the inactivation of *Salmonella* Senftenberg W775 will be discussed during mesophilic and thermophilic anaerobic digestion and pasteurization processes. The log reduction required by Regulation (EC) Nr. 1069/2009 (formerly Regulation (EC) No 1774/2002) for pathogenic bacteria is 5 $\log_{10}$. For the inactivation of other human and animal pathogens, weed seeds and plant pathogens, we would like to refer to the PhD work of A. Ceustermans which will published in 2012.

1.2 Mesophilic digestion

The inactivation curves of *Salmonella* Senftenberg W775 are shown in figure 1. At lower mesophilic temperatures (33.3-35°C), *Salmonella* Senftenberg W775 is not reduced or only by 1 $\log_{10}$ after 24 h of anaerobic digestion (Knie *et al*., 2001; Ade-Kappelmann, 2008). In addition, after 7 d there was only a 3 $\log_{10}$ reduction (Knie *et al*., 2001). 21 days were needed for a 6 $\log_{10}$ reduction of this pathogen at a digestion temperature of 33.3°C (Knie *et al*., 2001) while *Salmonella* Senftenberg W775 was reduced by max 1.5 $\log_{10}$ after 20 days of incubation at 35°C (Smith *et al*., 2005). Less than 9 days were required for a 5 $\log_{10}$ reduction when the temperature was 36°C (OVAM, 2010), whereas this could be achieved within 7 days at higher temperatures (38.5-40°C) (Drča, 2007). In the study of Ade-Kappelmann (2008), however, this 5 $\log_{10}$ reduction was already obtained within 1 day of anaerobic digestion at a temperature of 39°C or 47°C, although 1 day was not enough in the work of Drča (2007).

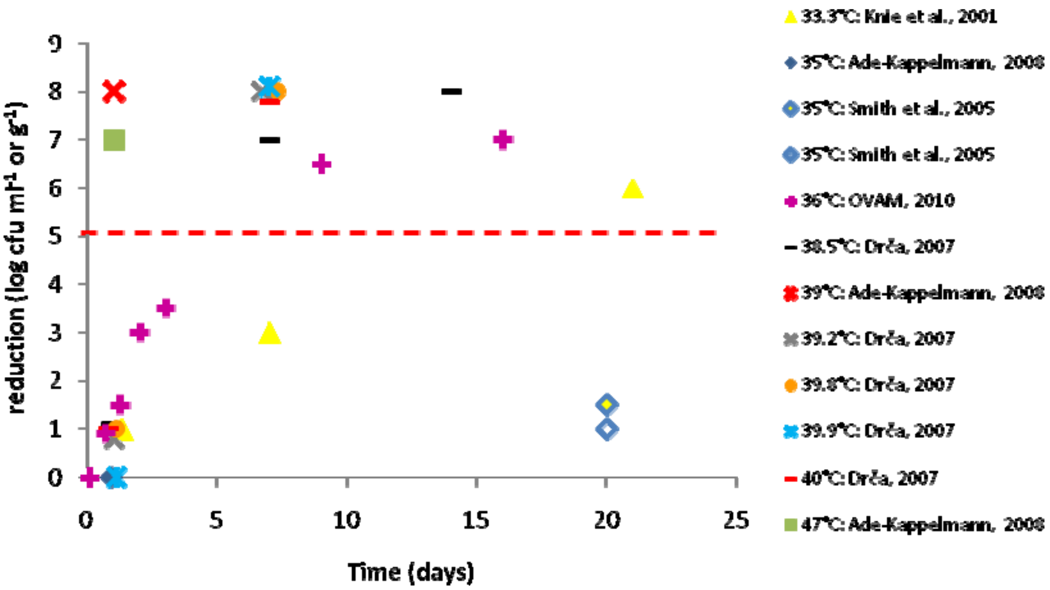


FIGURE 1 **Inactivation curves of *Salmonella* Senftenberg W775 during mesophilic digestion or incubation at different temperatures.**

Open symbol with yellow filling: incubation in a growth medium, open symbol: incubation in supernatant of centrifuged liquid raw sludge, symbol in the same colour: digestion. Red dashed line: log reduction required by Regulation (EC) Nr. 1069/2009

1.3 Thermophilic digestion

The different inactivation curves of *Salmonella* Senftenberg W775 during thermophilic digestion at different temperatures are shown in figure 2.

In German research, the inactivation of *S*. Senftenberg was determined at different thermophilic digestion temperatures (53, 53.5, 54, 54.5 and 55°C) (Knie *et al*., 2001; Ade-Kappelmann, 2008). However, the first sampling point in their research was only after 20 h or 24 h. The 5 $\log_{10}$ reduction was already reached at these points and consequently, this could have occurred earlier. Elving (2009) reported a 5 $\log_{10}$ reduction of *S*. Senftenberg after 5.6 h of incubation at 55°C. In the study carried out by Paluszak (1998), the digestion temperature was lower, namely 50.5 and 51°C, however, 2.5 h seemed to be enough to reach a satisfying inactivation of *S*. Senftenberg. Furthermore, this 5 $\log_{10}$ reduction seemed to be reached within an hour when this pathogen was submitted to an incubation temperature of 55°C (Smith *et al*., 2005). These authors also tested other strains of *Salmonella*, namely *S*. Typhimurium and *S*. Oranienburg and they observed that these strains were inactivated faster than *S*. Senftenberg during incubation at 55°C (Smith *et al*., 2005).

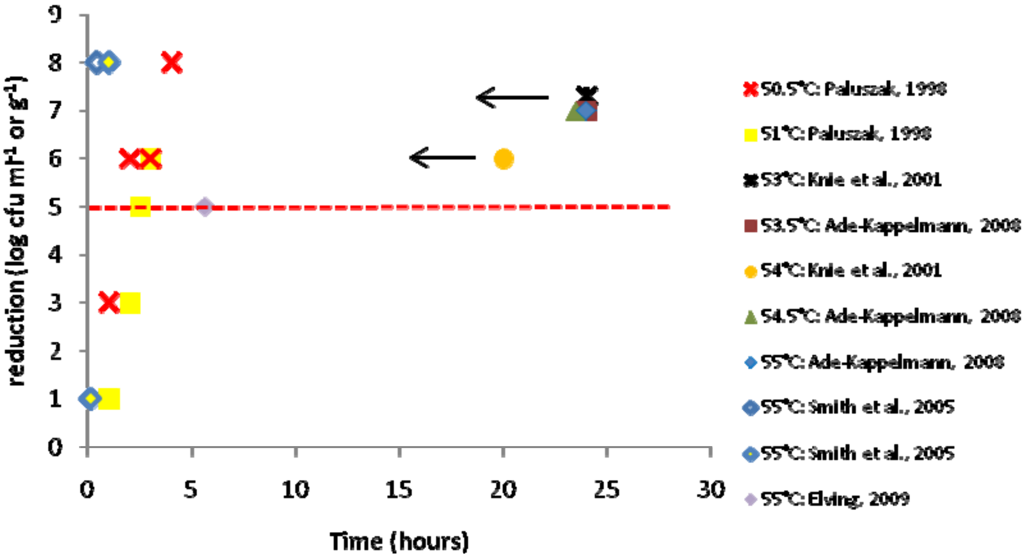


FIGURE 2 **Inactivation curves of *Salmonella* Senftenberg W775 during thermophilic digestion or incubation at different temperatures. Open symbol with yellow filling: incubation in a growth medium, open symbol: incubation in supernatant of centrifuged liquid raw sludge, symbol in the same colour: digestion, arrow: first sampling point. Red dashed line: log reduction required by Regulation (EC) Nr. 1069/2009.**

1.4 Pasteurization

The inactivation curves of *Salmonella* Senftenberg W775 during pasteurization are represented in figure 3. *S*. Senftenberg cannot survive for a long time during pasteurization at 70°C. In the study of Knie *et al*. (2001), a 7 $\log_{10}$ reduction was observed within 20 min pasteurization at 70°C. In our own experiment we could observe that *S*. Senftenberg was already eliminated within 5 min at 70°C (OVAM, 2010). Also Elving (2009) reported a 5.5 $\log_{10}$ reduction after 5 min of incubation in saline at 70°C. Smith *et al*. (2005) incubated three different *Salmonella* strains (*S*. Typhimurium, *S*. Senftenberg and *S*. Oranienburg) in two heating media (supernatant of centrifuged liquid raw sludge and a laboratory culture broth) at 70°C. All strains were inactivated within 10 s at 70°C (not shown in the figure). In some studies, the inactivation of *S*. Senftenberg was also monitored during the heating-up phase to reach a temperature of 70°C. A 4 $\log_{10}$ reduction of *S*. Senftenberg already took place before the temperature had reached 70°C (61°C after 44 min) in our own study (OVAM, 2010). The same was observed by Drča (2007). *S*. Senftenberg was already completely inactivated when a temperature of 70°C was reached after 67.4 min. This was also tested by Knie *et al*. (2001), who reported a 3 and 6 $\log_{10}$ reduction of *S*. Senftenberg when the actual pasteurization still had to begin (not shown in the figure).

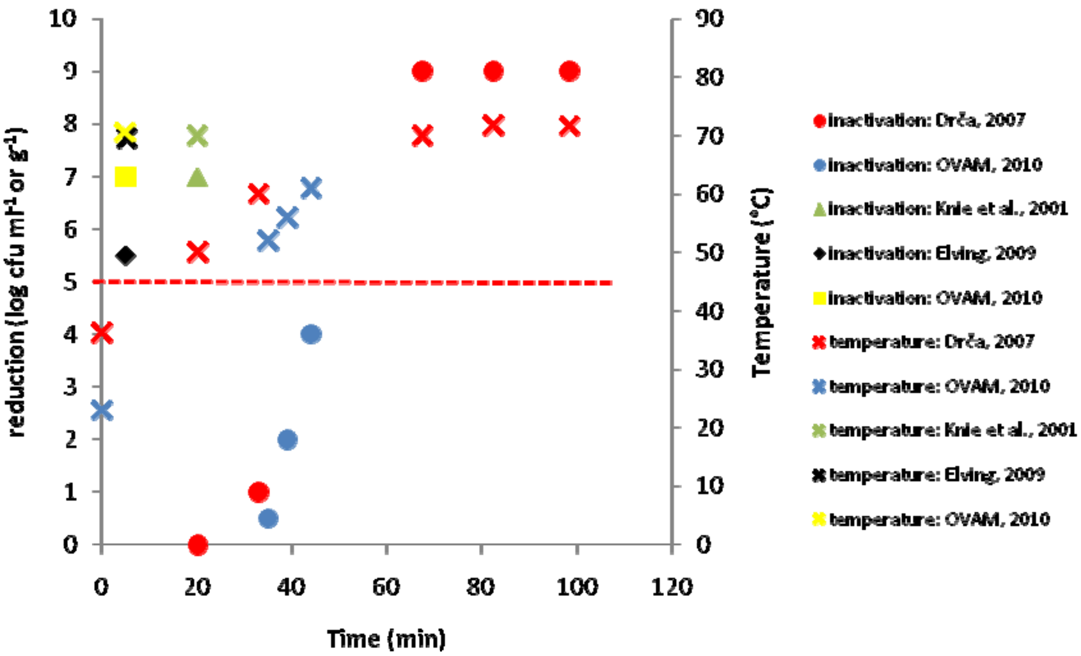


FIGURE 3 **Inactivation curves of *Salmonella* Senftenberg W775 during pasteurization. Small cross: temperature (2nd Y-axis), other symbols: inactivation (1st Y-axis). Red dashed line: log reduction required by Regulation (EC) Nr. 1069/2009.**

2. DEVELOPMENT OF THE TOOL BIOSAFETY@DIGESTION

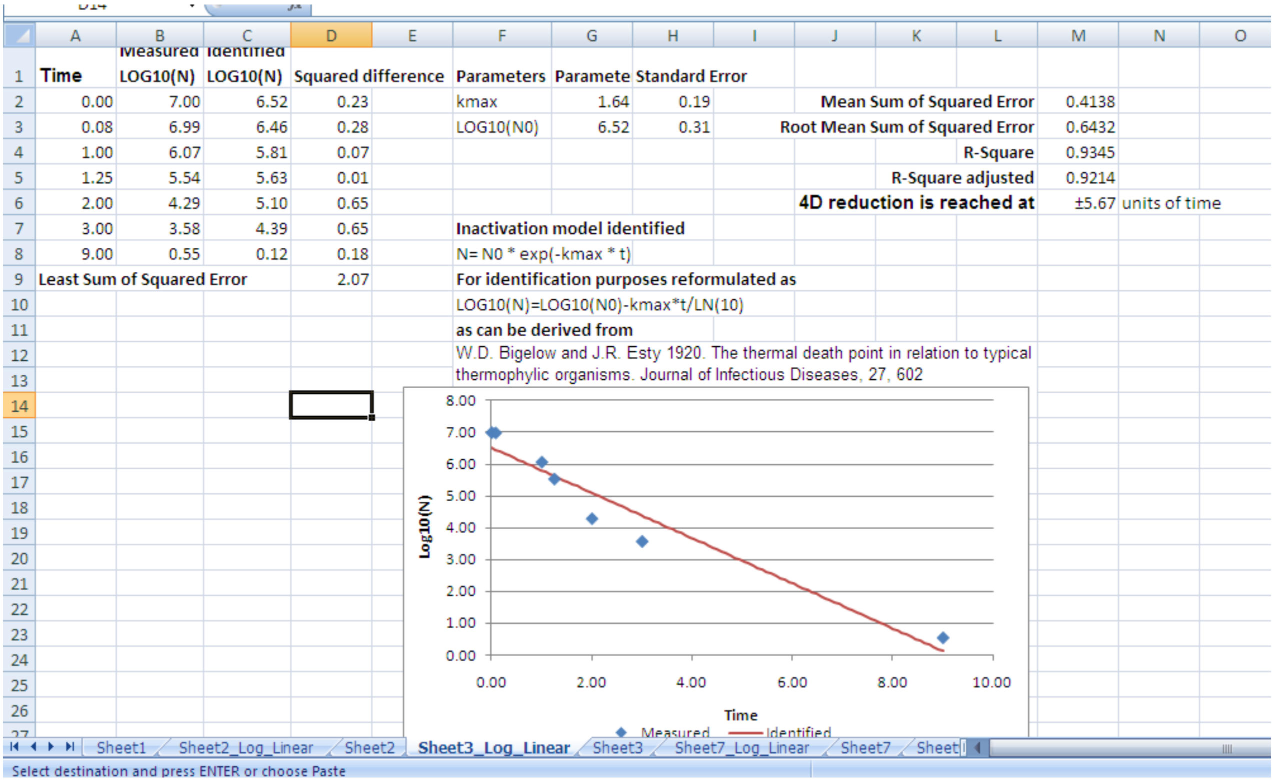
Several parameters including pH (Kunte *et al.*, 1998, 2000a,b; Fukushi *et al.*, 2003; Sahlström, 2003), volatile fatty acids (Kearney *et al.*, 1993b, Kunte *et al.*, 1998, 2000a,b; Sahlström, 2003), batch-wise or continuous digestion (Kearney *et al.*, 1993a) and solid contents (Jones, 1976; Kumar *et al.*, 1999; Termorshuizen *et al.*, 2003) have been shown to affect survival of pathogens and weed seeds during anaerobic digestion. However, temperature is indicated as the most important factor concerning the survival of pathogens during anaerobic digestion (Dumontet *et al.*, 1999). Moreover, inactivation by temperature depends on the retention time (Olsen and Larsen, 1987). It would be of great interest for all concerned parties, including policy makers, to predict the elimination of pathogens given the process temperature and retention time.

It is hypothesized that survival data performed under isothermal conditions can be used for predicting the elimination of pathogens under new circumstances. After collecting and modelling isothermal survival data, these models will be used to propose a model for predicting the time needed to reach a certain reduction in pathogen concentration at other fixed temperatures. This model can then be made available in a user-friendly Microsoft® Office Excel®-file in order to evaluate different digestion processes concerning their hygienisation capacity. The approach will be explained for *Salmonella* Senftenberg W775 – which is a model organism for human and animal pathogens – during mesophilic digestion.

Isothermal survival data of *S*. Senftenberg W775 during mesophilic digestion obtained from literature and an own experiment are presented in Table 1. The log-linear model was fitted to these data for each temperature separately with the application of GinaFiT (Geeraerd *et al.*, 2005). The first order inactivation rate constant (k_{max}) is represented in this tool (Figure 4). In this way, an inactivation rate is obtained for each temperature.

TABLE 1 **Isothermal survival data of *S.* Senftenberg W775 during mesophilic digestion.**

Time (days)	Population level ($\log_{10}$)	Temperature (°C)	Reference
0	6.97	33	Knie *et al.*, 2001
1	5.38		
7	3.32		
0	7.00	36	OVAM, 2010
0.08	6.99		
1	6.07		
1.25	5.54		
2	4.29		
3	3.58		
9	0.55		
0	8.36	38	Drča, 2007
1	7.97		
7	1.56		



Time	Measured LOG10(N)	Identified LOG10(N)	Squared difference	Parameters	Paramete	Standard Error
0.00	7.00	6.52	0.23	kmax	1.64	0.19
0.08	6.99	6.46	0.28	LOG10(N0)	6.52	0.31
1.00	6.07	5.81	0.07			
1.25	5.54	5.63	0.01			
2.00	4.29	5.10	0.65			
3.00	3.58	4.39	0.65			
9.00	0.55	0.12	0.18			
Least Sum of Squared Error			2.07			

Mean Sum of Squared Error	0.4138
Root Mean Sum of Squared Error	0.6432
R-Square	0.9345
R-Square adjusted	0.9214
4D reduction is reached at	±5.67 units of time

Inactivation model identified

N= N0 * exp(-kmax * t)

For identification purposes reformulated as

LOG10(N)=LOG10(N0)-kmax*t/LN(10)

as can be derived from

W.D. Bigelow and J.R. Esty 1920. The thermal death point in relation to typical thermophylic organisms. Journal of Infectious Diseases, 27, 602

Select destination and press ENTER or choose Paste

FIGURE 4 **Application of the log-linear model on the survival data at 36°C with the aid of GInaFiT.**

Afterwards, the *D*-value belonging to each temperature was determined based on these inactivation rates:

$$D = {\ln(10)}\Big/{k_{\max}}$$

Log_{10} *D*-values in function of the corresponding temperature delivers the *z*-value (specific heat resistance) (Figure 5).

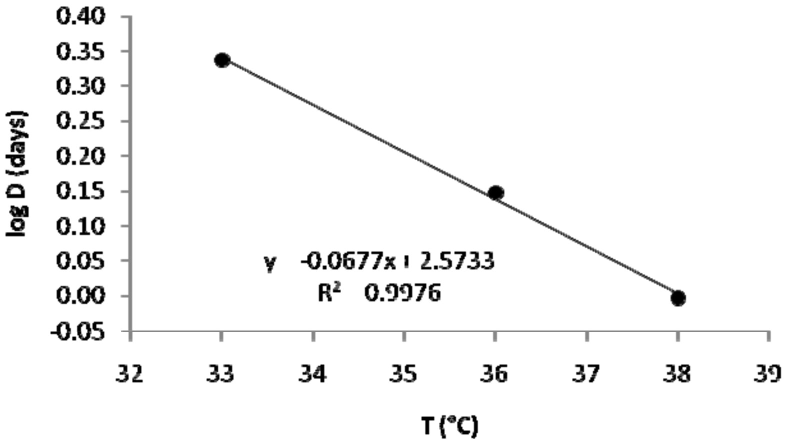
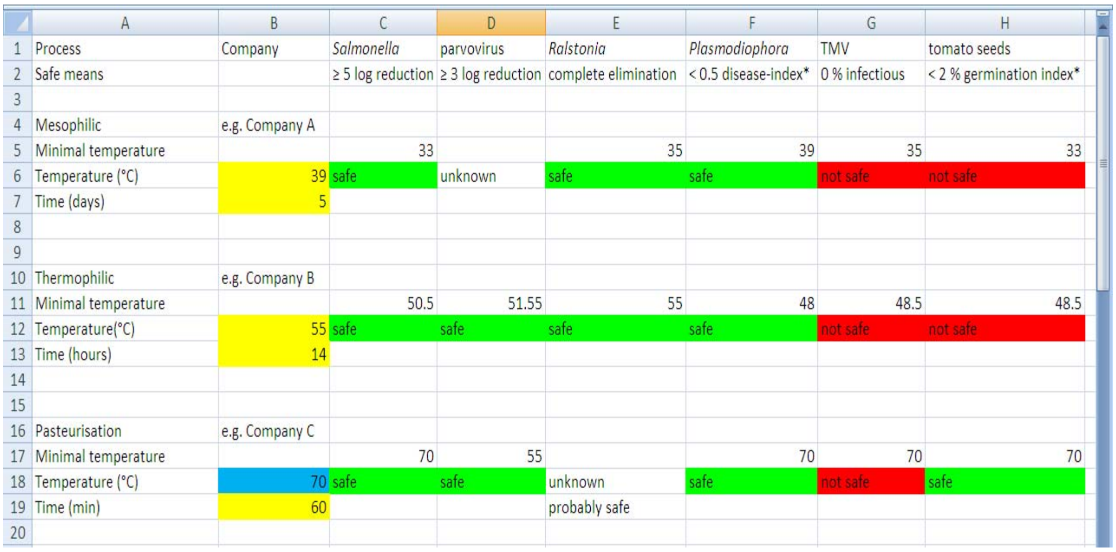


FIGURE 5 **Decimal reduction times in function of temperature.**

The *z*-value is needed to calculate the *D*-value at a certain temperature. The *z*-value is the temperature increase needed to reduce *D* with 90%; in this case z=14.77°C, which is outside the studied temperature range.

$$\log(D_T) = \log(D_{ref}) - \frac{T - T_{ref}}{z}$$

In this way, it can be calculated how much time is needed to obtain a certain $\log_{10}$ reduction of pathogens at a fixed temperature. According to Regulation (EC) No 1069/2009, for instance, a 5 $\log_{10}$ reduction of *S.* Senftenberg W775 needs to be demonstrated during thermophilic digestion when this process is used as an alternative for the mandatory pasteurization step. Afterwards, it can be determined if the retention time of a certain full-scale digester is at least as long as this 5*D*-value. If this is the case, the process will be highlighted as safe in the BioSafety@Digestion tool (Figure 6). Thus, for a particular process, temperature and retention time can be entered in the tool (in the yellow cells), after which the cells appear to indicate if this process is safe or not concerning the inactivation of *S.* Senftenberg W775. On the other hand, when a waste stream contains certain pathogens and an actual process would not be considered safe, one can determine with the aid of this tool which rise in temperature and/or prolongation of the retention time is necessary to obtain a safe end-product.



	A	B	C	D	E	F	G	H
1	Process	Company	*Salmonella*	parvovirus	*Ralstonia*	*Plasmodiophora*	TMV	tomato seeds
2	Safe means		≥ 5 log reduction	≥ 3 log reduction	complete elimination	< 0.5 disease-index*	0 % infectious	< 2 % germination index*
3								
4	Mesophilic	e.g. Company A						
5	Minimal temperature		33		35	39	35	33
6	Temperature (°C)	39	safe	unknown	safe	safe	not safe	not safe
7	Time (days)	5						
8								
9								
10	Thermophilic	e.g. Company B						
11	Minimal temperature		50.5	51.55	55	48	48.5	48.5
12	Temperature(°C)	55	safe	safe	safe	safe	not safe	not safe
13	Time (hours)	14						
14								
15								
16	Pasteurisation	e.g. Company C						
17	Minimal temperature		70	55		70	70	70
18	Temperature (°C)	70	safe	safe	unknown	safe	not safe	safe
19	Time (min)	60			probably safe			
20								

FIGURE 6 **Print screen of the BioSafety@Digestion tool for some illustrative processes (entered in the yellow boxes).**

Only the example of the inactivation of *S.* Senftenberg W775 during mesophilic digestion was explained above. The same method was applied to parvovirus and other processes. An inactivation model was not developed for all organisms visible in Figure 1.

Tobacco mosaic virus (TMV) is not inactivated during the typical retention times associated with these processes. *Ralstonia solanacearum* is a quarantine organism of which a complete elimination is required (EPPO, 2008; EPPO Standards, 2009), and, therefore, no model was developed. Also for *Plasmodiophora brassicae* and tomato seeds no model was set up, since the survival is expressed as categorical variables such as disease and germination index, respectively, and which need further investigation in order to select the best models which properly describe these data, respecting their specific statistical properties. The BioSafety@Digestion tool can be elaborated if additional data are gathered. The more informative data are incorporated, the higher the predictive power of the tool will become. The tool also contains a few protection mechanisms. For instance, when a too low temperature is entered, no result will appear (as it would be model extrapolation, which is not recommended) and the tool will indicate “unknown” (Figure 6) or “unknown, probably unsafe” or “unknown, probably safe”, whatever applicable given the current state-of-the-art knowledge included in the tool.

3. CONCLUSIONS

Accurate inactivation curves are available for *Salmonella* Senftenberg and this especially during mesophilic digestion. From these curves it becomes clear that the mandatory 5 $\log_{10}$ reduction of *S.* Senftenberg can be reached between 7 and 21 d and between 1 and 7 d when the temperature is 33.3°C and 38.5°C, respectively. Anaerobic digestion at higher temperatures demands shorter retention times concerning the inactivation of pathogens. As a result, they will be inactivated faster during thermophilic than during mesophilic digestion. Anaerobic digestion in the field, with its actual mesophilic and thermophilic temperature and retention time, is an accurate bio-control mechanism of utmost importance. Most pathogens will no longer be a risk after a pasteurization step of 1 h at a temperature of 70°C. *Salmonella* Senftenberg will be inactivated much faster.

The tool BioSafety@Digetion was developed to determine whether a particular digestion process is safe concerning the elimination of certain pathogens. The data required for this prediction are temperature inside the digester and the minimal guaranteed retention time. To make this tool more accurate, however, more isothermal survival data and models are needed concerning the different pathogens and seeds included.

4. ACKNOWLEDGEMENTS

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(233) EXPERIENCES WITH THE SUBSTRATE PROCESSING AND BIOGAS PRODUCTION FROM PACKAGED FOOD ON SUCH AN INDUSTRIAL SCALE PLANT IN ENGLAND

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EXECUTIVE SUMMARY

Huge amounts of industrial organic waste that for example arise from food processing, as expired products in supermarkets or as food leftovers in restaurants have to be disposed in an environmental friendly way. Because of the high amount of biodegradable organic components and the high humidity of this type of organic waste anaerobic digestion with biogas production is the most suitable technology to process such type of organic waste. Beside the production of renewable energy the organic waste is also a very suitable feedstock to produce a valuable fertilizer. To treat such type of organic waste it has to be considered that the amount of non-biodegradable contaminants for example plastic, glass, metals in the organic waste is usually very high. The contaminants have to be separated very early in the process before the biodegradable part of the organic fraction is charged into the digester. The separation of contaminants is important to avoid problems in the process and to assure that the digestate after anaerobic digestion of the organic material is of highest quality. For hygienic reasons this front end processing of the organic waste should be completely automated and in a completely closed system after waste delivery to the reception area of the plant. The front end processing must separate the contaminants efficiently and loss of biodegradable organics must be reduced to the lowest possible level. The quality of separated contaminants should make possible either thermal utilization or material recycling.

Further important process aspects have to be considered by treatment of such type of organic waste as sanitation and production of high quality fertilizer that can be utilized directly after anaerobic digestion without further treatment steps. For economic reasons it is very important to assure the process works very efficient with low energy consumption and with highest possible biogas yield. Industrial organic waste is a very suitable material for biogas production because of its high specific biogas yield. The front end processing of organic waste is the most important part of a biogas plant and must assure that the biogas plant can treat very heterogeneous organic waste fractions also with high a level of contamination of non-biodegradable material.

This article describes a new development of a front end processing technology that is realized in an industrial scale biogas plant in UK. The main targets of the development was a continuous operation, reduction of electricity consumption, increase of biogas production and more efficient separation of biodegradable from foreign non-biodegradable material. The new front end processing technology is meanwhile since several months in operation in a new biogas plant installed at Cannock, UK. This plant receives different types of organic waste mainly packaged food and has a processing capacity of 120.000 t/y and was already started up in spring 2011. The experiences from the first year of operation confirms that the front end processing is capable to treat organic waste with high amounts of contaminants especially plastics. Contaminants are removed efficiently and there is no need for further digestate treatment after the dewatering process and the digestate can be used directly as fertilizer in agriculture. This should be the preferred and is the simplest way of digestate utilization. As the impact reactor works very selective plastic bags are opened but not crushed to small pieces while the biodegradable waste fraction is disintegrated very efficient. That resulted in a higher biogas production. Continuous operation of the front end processing system and efficient processing of organic waste at very short retention time in the impact reactor results in very low electricity consumption compared to other front end processing technologies. The separated light fraction has very low amounts of biodegradable organic material and that reduces costs for disposal but also opens further opportunities like plastic recycling to improve the plant economics.

Session 5

(41) END-OF-WASTE CRITERIA FOR COMPOST: A SAFEGUARD FOR QUALITY?

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EXECUTIVE SUMMARY

With the revision of the Waste Framework Directive (WFD), the re-use and sustainable management of materials and resources, has now been introduced at EU level, by the instrument of "end of waste criteria " (EoW criteria). The Joint Research Centre of the EU Commission is working on the final draft of the technical document for the development of EoW criteria for biodegradable waste subject to biological treatment.

Bio-waste is a good example of a waste stream that offers a lot of opportunities for sustainable materials management and closing the material cycle. Across Europe, biowaste accounts for approximately 30 to 40% of municipal solid waste and is the largest household fraction. With good reason the EU Landfill Directive 1999/31/EC demands the gradual EU-wide diversion of biodegradable waste from landfills. In Europe, landfills, are major sources of methane emissions. With the extension of the separate collection of biowaste, an effective contribution to the reduction of methane emissions from landfills can therefore be achieved, thus supporting climate protection. Through the separate collection of biowaste and its application as compost in agriculture, mineral fertilisers can be substituted by 8% to 10%. Thus not only non-renewable resources for plant nutrients (for example phosphorous) are saved, but also peat from upland moors. Moreover, the separate collection of biowaste and its composting or fermentation serves as applied climate protection. Apart from that, there is a considerable reduction of CO_2-emissions due to the replacement of mineral fertilisers that need to be produced in energy intensive processes based on fossil fuels.

But a separate collection and treatment of bio-waste represents only a part of the life-cycle approach.

The Flemish Region has a long experience in optimal bio-waste treatment, providing the opportunity for a fixed list of allowable waste streams, to re-use them as a “quasi-product”, so called secondary raw materials. These secondary raw materials should not only comply with conditions related to the composition, the nutrient value, the maximum concentrations of well defined pollutants, but also with specific conditions relating to the use as fertiliser or soil-improver (determining the maximum possible quantity that can be applied on a specific kind of soil). A waste material loses the status of waste and becomes a secondary raw material from the time it meets this set of conditions.

Belgium is convinced that only the combination of separate collection of biowaste, together with a quality-assurance-system (QAS) and well-defined standards, will lead to compost or fermentation residues of excellent quality.

In the technical approach for the establishment of EoW criteria for biodegradable waste, Belgium emphasises not only to focus on standards for the resulting output streams, but also to implement an integrated chain approach, in order to ensure the excellent quality of the end product. With regard to EoW criteria for biodegradable waste, traceability is a big concern for Belgium. Will there be sufficient guarantee that the End-of-waste products will be properly used on the environmental level? Which legislation will be used to secure conditions for application? Belgium wants to emphasise the complexity when considering/establishing EoW criteria for biodegradable waste in comparison with EoW criteria for iron, steel and aluminium scrap.

As announced in the Communication of 18 May 2010 on future steps of the bio-waste management in the European Union, the Commission has abandoned the idea of a specific bio-waste directive. In order to improve the management of bio-waste in the EU, Member States have to implement the existing regulatory framework as good as possible, such as the properly enforcement of the Landfill Directive, the implementation of the life cycle thinking approach, the use of the end of waste process for certain waste streams. Only if compost or digestate, regarded as products, are obtained from continued processing installation and controlled by a QAS, a good marketing of the product is ensured and trade within the European Community is permitted.

1 INTRODUCTION

Biodegradable-waste is a topic that has been discussed for a long time now in Europe. The European Commission took several initiatives in order to improve the management of bio-waste. There was the discussion on the green paper, followed by the impact assessment in 2009. Finally the Commission took in 2010 the decision to abandon the idea of introduction of a new directive on bio-waste, and to focus on a better implementation of existing legislation. Member States have to enforce properly the Landfill Directive, to implement a life cycle thinking approach and to establish EoW criteria for biodegradable waste.

Belgium is convinced that the EU needs a specific legislation on biodegradable waste, if we want to maximise the positive advantages of the management of this material resource. If the Commission will only focus on the better implementation of the existing legislation and the establishment of End-of-waste criteria for only certain products of the biological treatment of biodegradable waste, a huge potential will be lost.

1.1 Background

Biodegradable waste isn’t just a waste; it has a lot of value if managed in a sound and sustainable way. If treated properly, it serves many purposes: we can recycle it to a compost or digestate that can be safely used on the soil, helping improving soil quality, adding to soil protection by means of balancing organic matter an nutrients in the soil.

On the other hand we are confronted with the EU-Directive on Renewable Energy, trying to give answers to the challenges of climate change and also looking to biodegradable-waste as a part of the solution. It shows us that biodegradable waste is a hot item and highly desired.

Belgium is convinced that we have to collect and treat biodegradable waste as a material resource on a sustainable way, in the first place. If we collect and recycle biodegradable waste to a product of outstanding quality, in such a way that it can be safely used on the soil, we are not only helping improving the soil quality and adding to soil protection, but also helping to fight climate change indirectly by means of carbon sequestration, substitution of mineral fertilisers and directly by reducing the negative effect of methane emissions of landfilling.

2 CURRENT SITUATION: WHAT DO WE HAVE? – WHAT IS HAPPENING?

2.1 Ambiguous legislation

In a lot of Member States, biodegradable waste isn’t managed sustainable and most of them are still struggling with the goals of the Landfill directive that demands to divert biodegradable waste from landfills, aiming to reduce the negative effect of the methane emissions.

What is still missing at the European level to make it work? The current situation is a clear proof of the fact that existing legislation, linked in one way or another to biodegradable waste, isn’t sufficient enough or is too confusing.

For example, both biowaste and biodegradable waste terminology are being used without a proper meaning. Biowaste is defined in the Waste Framework Directive and only consists of a part of biodegradable waste, as defined in the Landfill Directive. Besides, compost is sometimes being understood as a compost from biowaste only, while compost can be made of any biodegradable waste. Therefore, in order to be able to conduct an integrated approach to reduce landfill emissions, protect our soils and produce energy, all biodegradable waste should be considered within one legislation and the term compost should only be reflecting the biological process of aerobic degradation of the organic matter regardless the type of inputs composted (later on different qualities of compost can be defined, with different levels of traceability).

2.2 Landfill directive and diverting biodegradable waste from landfills

In practice, it seems a huge task for a lot of Member States. During the last TAC-meeting (Technical Adaptation Committee) for the Landfill Directive on February 10th, 2012, the first results of the 2007-2009 reporting exercise of Member States were presented by the European Commission, with special focus on the landfilling of biodegradable waste. It was clearly stated that the implementation of the Landfill directive is still poor in a lot of Member States. As for the attainment of the diversion targets of biodegradable waste, all Member States, which were to meet the 2009 target (50% diversion) had done so. However, when analysing results for 2009, there seems to be a significant risk that some of the Member States that postponed their target to 2010, will not meet that target. The final report of the European Commission is expected to be published in the coming months of 2012.

Today, the European Commission has expressed its concerns over the risk of missing the 2013 target of the Landfill Directive (for those Member States that decided to postpone their target by four years) if corrective actions are not applied very soon. The Commission is now planning to review the landfill diversion targets by 2014. But, will this solve the biodegradable waste issue?

3 WHAT DOES THE EUROPEAN COMMISSION PROPOSE?

3.1 Better implementation

The issue of better regulation is used by the European Commission to argue that we don't need a specific legislation for biodegradable waste. A better regulation is certainly linked with good implementation of existing legislation. But what if it seems that the existing legislation is not effective? We are convinced that the actual regulation needs a legislative support. In the past we have had already good examples: directives were created to support existing directives on the road to achieve their targets. For example the legislation on vehicles is supporting the air quality directive.

A specific directive on biodegradable waste will bring a strongly support to achieve different goals within the European union, such as the actual implementation of the Landfill Directive and not at least also the targets of the Directive on Renewable Energy.

3.2 Review the existing reuse and recycle targets to be raised in to their maximum feasible level by 2014 (art. 11.2 of WFD)

The WFD, in particular the procedure for the creation and implementation of EoW criteria is often mentioned as a sufficient measure to tackle the biowaste issue. We do not believe that this will be enough. On the contrary, the Waste Framework Directive indicates itself that further work on biowaste is needed.

This does not mean that the answer lies in amending the Waste Framework Directive, as proposed by the Commission, suggesting re-opening article 11 of the WFD. Specific, binding targets on the separate collection and recycling of biodegradable waste could be introduced there. But the discussion on the recycling targets will always be a big hurdle.

3.3 Establishing EoW criteria for Biodegradable waste

Apart from the fact that a lot of work is delivered by the Joint Research Centre of the European Commission and still has to be done for the finalisation of the report, Belgium together with several other Member States, is certain that EoW criteria for biodegradable waste can never be seen as a stand-alone solution for the sustainable management of biowaste. Furthermore, specifically for the issues concerning the EoW criteria, several items in the document are not yet clarified and a lot of questions remain not answered.

Requirements on input materials and on treatment processes and techniques

In the draft technical report, the establishment of EoW criteria is limited to biodegradable waste subject to biological treatment. What about the EoW status for other biodegradable wastes that can perfectly be applied as an organic fertiliser or soil improver without any pre-treatment?

The technical document proposes a positive list of separately collected input streams for the production of compost or digestate. The separate collection is a prior condition for a good quality of the end product. Having a positive list is useful as indicator and will narrow down the risks of contamination of the compost/digestate with potential pollutants and undesired material. But a positive list will not guarantee a prevention of dilution.

In the Flemish Region, there is no legally defined imitative list of input materials for biological treatment. We consider that the acceptance protocol of the input material should be the essential part in the Quality Assurance System of the treatment plant. The treatment plant itself is responsible for the input control on arrival on the treatment plant. The End-of-waste certification system should be based upon the principle of auto-control: the treatment plant implements an internal quality system, taking into account input and acceptation of the biowaste and monitoring of the quality of the treatment process, quality assurance of the end product and the reasoned application of the end product as a fertiliser or

soil improver. The accepted biodegradable waste should be subject to strict requirements: control on physical, chemical or biological contamination, a visual inspection and registration of type of biowaste before being treated. The incoming waste streams should comply with the requirements (minimum criteria) laid down in the national/regional waste regulation, being the same standards for the outcoming streams. This is the only way to prevent unacceptable diffusion of unwanted or polluting substances.
Moreover, the list must be flexible, in function of time and content, in order to allow new input streams, or to delete streams being proved not to be compliant with the acceptance standards.

Requirements on Product Quality
The European Commission wants only one set of EoW criteria for compost and digestate. Why only one set of standards for totally different products?
In the Flemish Region, in some cases, the liquid fraction of digestate is further treated, through biological treatment (nitrification-denitrification), effluent treatment (ultrafiltration, reverse osmosis) to several other end products. Since the organic matter fraction is further degraded, some of these will not comply with the EoW criteria, such as the minimum organic matter content. There is a serious interest of digestate producers to produce a mineral concentrate containing useful nutrients, but with a low value for organic matter, in order to facilitate the export. In these cases, the organic matter content is neither wanted nor interesting, while the intrinsic quality or value of particularly compost, is meanly the organic matter content. Within the vision of material recovery (nutrients), this is a positive evolution in the sector of digestion. Could treated digestate ever get the status of EoW and receive a formal recognition of a mineral fertiliser?

Application of end-of-waste criteria
Once compost/digestate has received the End-of-waste status, the sale of the product on the market must always be linked with the specific terms of use and application conditions as fertiliser or soil-improving substance: the maximum possible quantity that can be applied on a specific kind of soil must be defined and communicated. At this moment, these terms are not provided to be included in the statement of conformity, accompanying the EoW product?
Moreover, if an endproduct, complying with the End-of-waste criteria, is not used as a fertiliser or a soil improver, these endproducts are considered to be waste. How will this crucial aspect be guaranteed and controlled in practice? Here a problem of legal competency will show up.

4 NEED FOR AN INTEGRATED APPROACH

As mentioned in paragraph 3.3, a lot of work has already been delivered by Joint Research Centre of the Commission and a positive evolution of the technical report for the establishment of EoW criteria for biodegradable waste is clearly defined. But still, Belgium together with several other Member States wants to emphasise that EoW criteria for biodegradable waste can never be seen as a stand-alone solution for the sustainable management of biowaste. All of the proposed measurements related in one way or another to biodegradable waste, are valuable steps of the European Commission, but dispersed over different legislations. We do need an integrated approach.
Experience in Member States that have already established a system, shows that you need a mix of linked instruments reinforcing and supplementing each other. Such an integrated approach built on total chain approach, turns out good results.

4.1 Why a legislative proposal/ a directive? What should be in it?

A specific legislation can offer legal certainty, a long-term assurance, and the necessary level playing field, which guarantees a safe investment climate, indispensable to convince private and public operators to make the investments required. It is creating confidence necessary for setting up a policy concerning biodegradable waste.

Within an integrated approach, different specific elements can be detected, besides of course prevention of biodegradable waste:
An integrated approach consists of a combination of
-Awareness raising and information
-A high level of qualitative separate collection targets
-Control of the input streams linked with definitions and requirements
-A highly qualitative and controlled treatment process

-The use of a sound quality assurance system
-Control of the output streams (linked with standards) and the use of those streams on the soil.

Such an approach guarantees traceability up to the final receptor and results in higher quality products, which are safe to be used on the soil.

4.2 Prevention

Within that integrated approach, prevention deserves a privileged position. Waste that isn't produced doesn't have to be treated and asks for lesser investments. Supporting prevention needs a strategy supported by specific prevention targets, working on raising awareness, giving information and support.
Current practices which are successful are home composting, smart gardening and the prevention of food waste.

4.3 Need for separate collection

A legislative measure should certainly stress the need for separately collected biodegradable waste going into the composting and digestion facilities. Because the output streams coming out of those installations, need to be of an excellent quality. This helps creating confidence in their value. Above that, separate collection contributes in traceability throughout the system.

5 UNITED BUT DIVERSE

At the same time, while working towards harmonisation and a level playing field, we should not forget that local conditions can vary substantially. Consequently priorities are different between regions within the European Union, but even so between regions within one Member States. In one region the demand for organic carbon can be very high, in another there is more need for renewable energy. Certainly, those differences are to be taking into account. But there is no difficulty to include this into a legislative measure.

So the diversity between Member States is important, not only regarding local conditions but even so on the approach for biodegradable waste.
A legislative measure on biodegradable waste should be flexible enough: it should not only providing a firm framework for Member States still struggling with the issue but also providing the possibility to go further then what the legislation prescribes for Member States with a lot of experience.

6 CONCLUSIONS : THE FUTURE = A BIODEGRADABLE WASTE FRAMEWORK DIRECTIVE

In several of her strategies the EU Commission commits its self to work towards a sustainable, resource efficient recycling society. How to deal with biodegradable waste, will be a major litmus test to demonstrate whether as the European Union will carry into practice what it proclaims.

Streamlining the management of biodegradable waste on EU level is a challenge. We have an opportunity of a lifetime.
Will we be able to achieve a sustainable management of biodegradable waste reflecting the comprehensive closed loop system that nature has offered us and that we have to preserve? We should face the challenge and finally work together on a specific legislation. It will be one of the cornerstones for the EU to become that society showing courage in making sustainability a reality and not just a concept.

Finally, proper biodegradable waste treatment will undoubtedly create jobs and pave the way to new business opportunities. It is a win win opportunity. This is also the strong message of the EU Resource Efficiency Roadmap of 20 September 2011 (COM (2011) 571 Final): waste must be managed as a resource, a source of economic activities and jobs.

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(70) SEPARATE COLLECTION OF BIOWASTE ITSELF IN FRANCE AROUND THE COMPOST+ NETWORK

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EXECUTIVE SUMMARY

In France, only 2.2 million inhabitants are concerned by door to door organic waste collection (ref 1). French policy has never sought to strongly encourage the recovery of organic waste. Unlike some countries, there is no bond of means in France but only bond of results as far as compliance with the quality of the produced compost is concerned. This generates questions from French policymakers about treatment options and the evolution of the mechanical biological treatment sector over time. The current work of the Joint Research Centre (JRC) on « End of Waste » emphasizes a little more this uncertainty at the French level.

Given this situation, in 2006, a group of French local authorities involved in the separate collection of biowaste formed a network called the Compost+ Network, which became recently an association. The main objective of the Compost+ Network is to ensure the promotion and sustainability of a fully evolving sector in France, despite a not so incentive regulatory context. The network currently has 9 members representing more than 1 000 000 inhabitants. All are local authorities that implement a separate collection of organic waste on their territory or are about to do so.

Compost+ provides support to local authorities wishing to develop a separate collection of biowaste project. The network is also developing tools to sustain the quality of the compost produced and to share experiences. To achieve these objectives, communities at the initiative of the network and their partners share their experiences and pool their results. The network is also studying the quality of the compost produced (for instance with: the European Ecolabel or the “suitable for organic farming” label), outlets based on the territorial context, “community” composting, some treatment processes such as "extensive" composting, "industrial" composting or anaerobic digestion. The results obtained in the different territories, such as participation rates, analysis of the costs of collection and treatment, disparity in the sale price of compost, etc... are compared. To compare costs, Compost+ is in favour of calculating waste management global costs per inhabitant in a way to evaluate the economical impacts on taxpayers. Moreover, the costs analysis needs to take into account the performance system, the quality level of services and finally the territorial context as well. Otherwise, costs can't be understood themselves. The network has become as well a privileged interlocutor of the ADEME (French Environmental and Energy Management Agency) and of the ministries of agriculture and environment. The association takes part in working groups about compost standards and in the European consultation "End of Waste." It also organizes meetings or seminars on these topics.

As a conclusion, a very important room for improvement exists in France so that the biowaste collection finds the place that is hers, as it is the case in other European countries. Creating a Network like Compost+ was essential to give a new impulse to biowaste collection in France. Compost+ is on the way to provide the lack of technical support, information and studies required by the sector to go forward and get more developed. Today, the association is considered as an important stakeholder. But it has only one year and one permanent engineer since February 2012. Therefore, except its present involvement in many studies and working groups, many things remain to be done and results and conclusions of its work should be rich in 2012.

1. INTRODUCTION

Only French local authorities involved in biowaste collection and treatment compose Compost+ Network. Created in 2006, the network became an association one year ago in March 2011 in order to realize several projects required for developing and promoting biowaste collection in France.

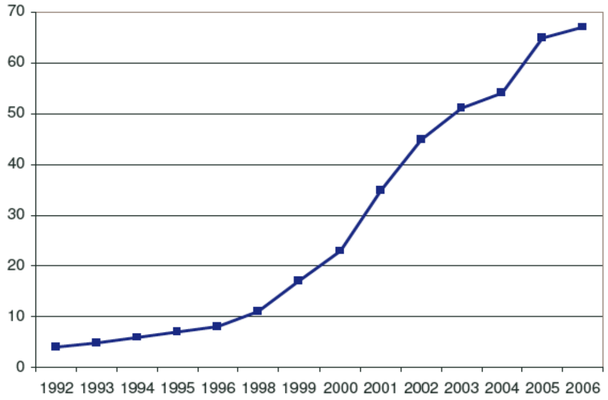
Compost+' objective is to promote and develop a sustainable biowaste collection sector in France.

Since the beginning, its main work is to compile objective data on collecting and processing techniques, recycling performance and systems cost in order to make it available to the Government and any local authorities wishing to develop a biowaste collection system. Compost+ shares its knowledge by participating in many studies, belonging to the main working groups and organising itself meetings, seminars or learning sessions. In 2012, Compost+ decided to launch three essential projects for the sector. First of all, writing a methodological guideline designed to help politic choices in biowaste management is its priority. Secondly, Compost+ wants to build a quality assurance system that could satisfy compost users and bring back confidence between them and producers. Ultimately, Compost+ aims to set up an observatory of biowaste collection system, following performances, costs and quality indicators.

The next articles will present more precisely the French context in which Compost+ has been created. Then are presented all the objectives followed by the association. In the methodology part is explained who are the members and which tools and means of action are chose to achieve those objectives. Compost+ developed as well a methodological approach on the understanding of waste management costs.

Recently, Compost+ recruited its first engineer in February 2012 in charge of project management, animation, and studies… First part of his work will be to update the French list of local authorities working on biowaste collection before starting the projects. The first results are expected by the end of the year.

1.1 Background



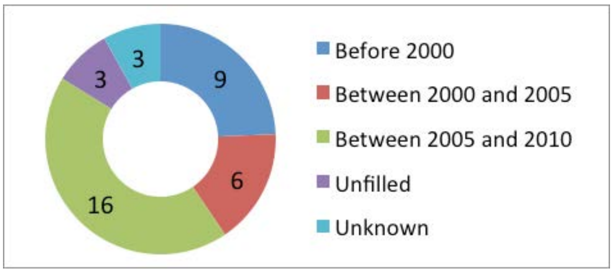
Graph 1: Number of local authorities engaged in biowaste collection increasing overtime (Extract from an ADEME study, 2006)

Biowaste recycling started in France during the nineties' by composting mixed household waste. But the mechanical biological process implemented those days was not enough efficient to warranty good quality composts. Consequently, use for these composts as fertilizer has dramatically polluted many fields with important amount of plastic and glass.

After this experience, the sector had to find new methods for recycling biowaste and above all to regain the confidence of farmers by performing the compost quality. Thereby, numbers of French local authorities started around 14 years ago to implement a door-to-door biowaste collection system as shown in the **Graph 1**. In 2006, there were 69 local authorities involved in biowaste collection all over France

In order to boost the sector, the ADEME created in 2006 a quality assurance system called Qualorg. Three years latter, the French Government made compulsory for all composts to respect quality standards. By setting bond of results, this framework aimed at bringing security and assurance. But the standards applied were not enough strict for farmers still afraid about using mixed waste compost. Instead of giving advantage to biowaste separate collection, that produces high quality composts, this situation led to a strong debate opposing biowaste collection to mechanical biological process. The following main issues were discussed in a subjective debate: system performances, collection costs and compost quality, contributing to reduce significantly the development of biowaste collection in France.

After 2005, the trend of local authorities involved in biowaste collection kept increasing (**Graph 2**). Although, the last survey realized by Compost+ counts around 100 local authorities involved, which means clearly that the trend has been slower than before.
At the same time, the number of mechanical biological plants decreases strongly from 78 in 2001 to 36 in 2006. But the ADEME counts around 50 projects for the next few years.



Graph 2: Distribution of local authorities per period for starting biowaste collection (37 localities investigated by Compost+)

Compost+ Network was created in 2006 during this period of debate in order to bring objective data compared to biowaste collection system. The network was also a mean to support a sustainable development of biowaste collection by allowing the sector to take profit of its 10 years members' experiences. From the beginning, the Network represents many local authorities from everywhere in France. It was also a way for them not to be isolated anymore.

1.2 Research objectives

Compost+'s objective is to develop and promote sustainable biowaste collection system by:

- Realizing and supporting any projects in line with this objective: even if biowaste collection has been practised for more than 10 years, its development is still reduced by lack of tools. The projects that Compost+ wants to support will make sustainable biowaste collection sector.
- Sharing good practises among members: local authorities are permanently looking for information in order to perform their system or even to compare themselves on practises and performances. Any members looking for information can use Compost+ as a technical platform.
- Communicating objective information: as a local authorities network, Compost+ is a privileged stakeholder for public organisms and the Government.
- Gathering knowledge: Compost+ aims to compile as much information as possible on biowaste collection: studies, experimentations, new solutions... Therefore, Compost+ works with international partners as the European Compost Network. In the same way, the association provides a technical and regulatory watch for members.
- Contributing to regulatory evolutions: Compost+ works with the other stakeholders, the French Government and Europe to create a regulatory framework adapted to the sector. For instance, the association took part in the "End Of Waste" discussion in charge of producing European biowaste recycling rules.

2 METHODOLOGY

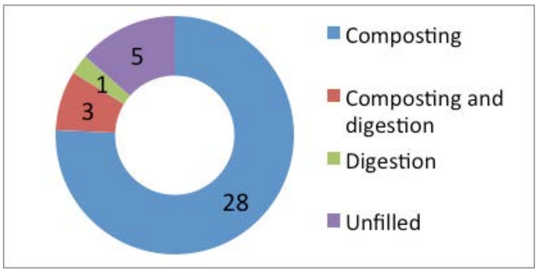
2.1 Members

Compost+ is composed exclusively of local authorities in charge of waste collection or treatment. The founder members decided to restrict the admission in order to better represent localities and their issues. Above all, Compost+ wants to be a network of local councillors helping other local councillors. While 6 French local authorities created the association in 2011, the network is 6 years old and most members have 10 years of experience in biowaste collection. The founder members are:

- Syndicat Centre Hérault
- Syndicat Mixte pout le Traitement des Déchets Ménagers du Bassin Est
- SMICVAL du Libournais Haute Gironde
- Cap l'Orient Agglomération
- Syndicat Intercommunal d'Electricité et d'Equipement de la Nièvre
- Syndicat Mixte de Puisaye
- Three more local authorities joined Compost+ by now:
- Symideme
- Syndicat Mixte du Secteur 4
- SMICTOM du Nord de l'Arrondissement de Redon

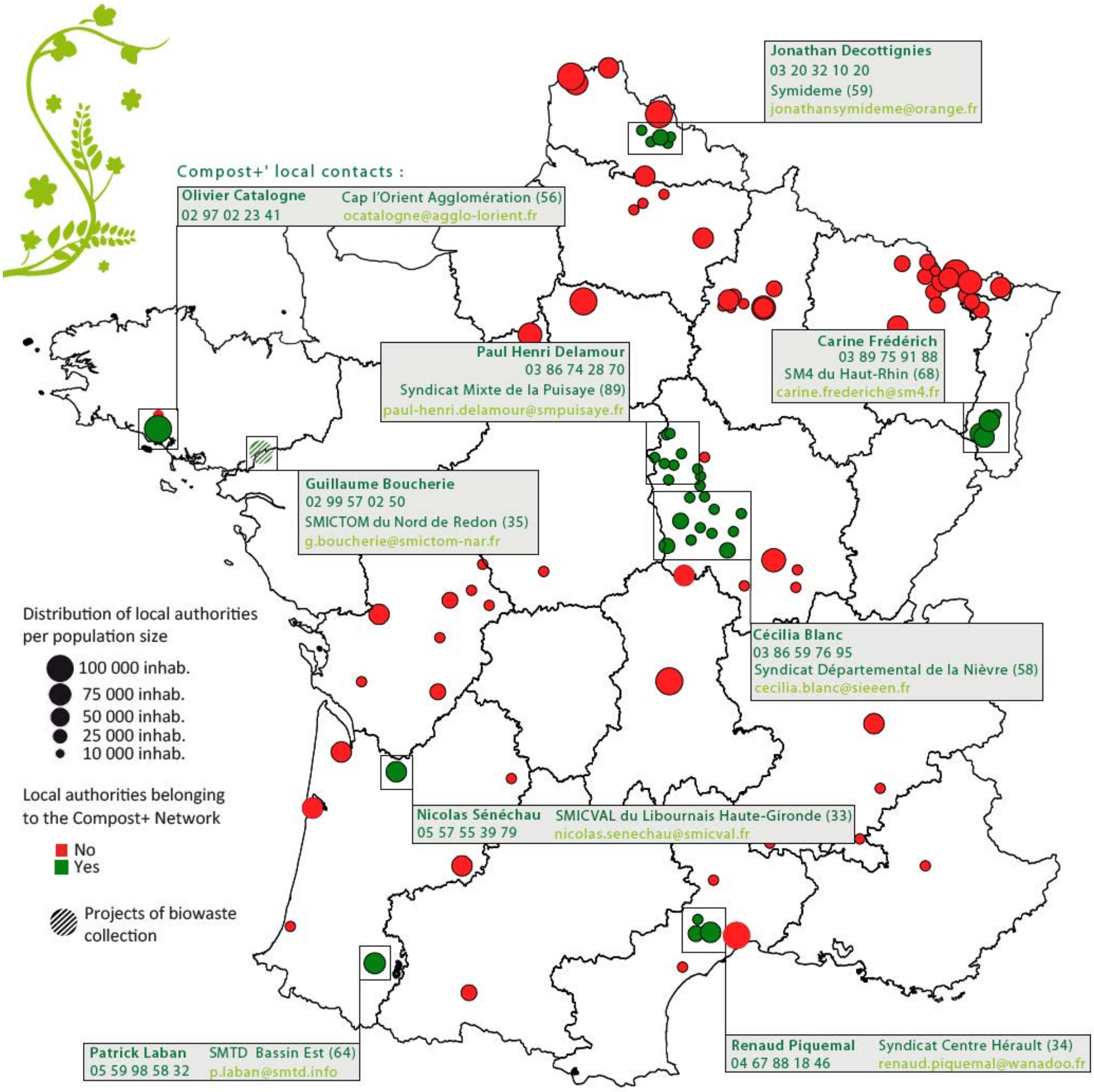
Although most of Compost+' members are composting, the network supports as well digesting process when biowaste are separately collected. As shown by **Graph 3**, extract from a Compost+ survey, unlike other European countries, biowaste digestion is not so spread in France.

More precisely, only 9 local authorities' digestion plants are running in 2011 according to a survey realized by AMORCE. Among them, only 3 are designed for biowaste separately collected. 8 more plants should come soon whose only one for biowaste.



Graph 3: Distribution of local authorities per type of biowaste treatment

Today, the Compost+ Network represents more than 1 million inhabitants. The following **map 1** presents the distribution of local authorities engaged in biowaste collection in France and the Compost+' local contacts for each member.



Map 1: Location of local authorities involved in biowaste collection in France

2.2 Tools and meanings of action

Compost+ plans to achieve these goals by:

- Realizing three main projects essential to make durable the development of biowaste collection. Precisely, Compost+ works on:
    - A methodological guideline for politics: this guideline is expected by local authorities who are thinking about biowaste recycling and needs information to better understand the main sector issues and to better control the advantage and inconvenience of biowaste collection. The final document will illustrate all the arguments used by real experiences.
    - A quality assurance system: considering that biowaste collection manages to produce a high quality compost, Compost+ wants to create a quality assurance system that could emphasis this high quality and by the way bring back confidence between compost users and producers. Moreover, the rules expected from Europe will make obligatory to strengthen the existing control system, today realized by the state in France.
    - An observatory of biowaste collection indicators: creating this observatory is a way to compile updated information on many topics as performance, technical methods and costs of biowaste collection. The main goal is to understand how bioawaste collection can be integrated in a global waste management system and which are the real impacts on efficiency and cost for this system and especially compared to the inhabitants' tax.
- Creating a website (www.compostplus.org) as a communication means and a technical platform for local authorities: the website will be the perfect tool to compile, share and then communicate information.
- Writing a trimestral Newsletter dealing about biowaste collection: the Newsletter is divided in the following three main parts: News, Manifestations, and Local Authorities Presentations. Biowaste regulation moves permanently and this newsletter highlights the impacts for local authorities.
- Organizing seminars: Compost+ organizes every 18 months meetings for members and other local authorities interested in biowaste collection. Each meeting takes place in a member local authority. Thus, while one part is dedicated to technical and regulatory news, it is also a good opportunity to visit composting platforms and to give details on the entire biowaste collection and treatment system.
- Participating in studies and working groups: as often as possible, Compost+ takes part in studies or working groups dealing on biowaste recycling. That work allows the association to voice the point of view of local authorities involved in biowaste collection. It makes Compost+ one of the privileged stakeholders working with the ADEME or the Government when biowaste is concerned.
- Organizing learning session: Compost+ holds learning session opened to local authorities agents whishing information about biowaste collection: impacts on global waste management, technical methods, collection organization, type of treatment…

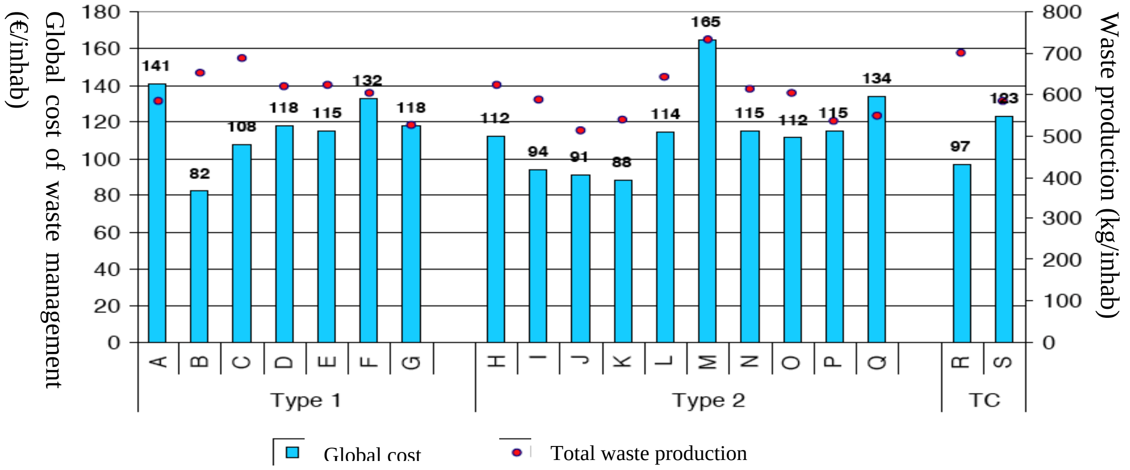
2.3 Compost+'s approach on waste management costs

Waste management cost is one the most important indicators watched by politics to make their choice. Different approaches can be used to calculate it. The industrial approach calculates collection and treatment costs per ton, which is actually interesting only when you need to compare the efficiency of two similar waste management systems. But the differences between them are often too important to make the comparison relevant (by instance biowaste collection can't be compare with mechanical biological treatment like this).

The second approach aims to calculate collection and treatment costs per inhabitant, which indicates directly the economical impact on taxpayers that really concerns local authorities.

In 2008, the ADEME realized a study on biowaste collection costs impact that shown that waste management systems with biowaste collection were globally only 5 to 10% more expensive than systems with mechanical biological treatment (**Graph 4**). Actually, this study concluded that better sorting waste reduces significantly treatment cost. Therefore, Compost+ defends a global cost approach instead of distinguished approach per type of waste.

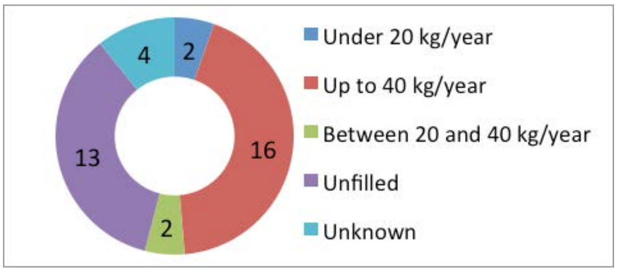
Finally, it is important as well to understand that local authorities choices are influenced not only by cost but also by many other singular parameters belonging to the territorial context: politic background, geographical position, type of climate, population size, competitive market…



Graph 4: Comparison of waste management system global cost per inhabitant (A to Q: with biowaste collection, R and S: with mechanical biological treatment)

Nevertheless, comparing global cost per inhabitant is not sufficient to reflect what is really done by the local authorities. Indeed, this indicator could be really down for localities with low quality of services. Therefore, it is essential to link it with other indicators like performance system and quality level of services.

As shown by the **Graph 5**, different performances can be reach dealing with the quantity of biowaste collected per inhabitant per year.



Graph 5: Distribution of local authorities per quantity of biowaste collected

Finally, it is important as well to understand that local authorities choices are influenced not only by cost but also by many other singular parameters belonging to the territorial context: politic background, geographical position, type of climate, population size, competitive market…

As a conclusion, there is as many waste management system as local authorities. And to compare them requires to calculate several indicators and besides to study many parameters in order to make a relevant analysis.

3 RESULTS AND DISCUSSION

Today, Compost+ is considered as an important stakeholder in biowaste collection sector. But the association has only one year and one permanent engineer since February 2012. Therefore, except its involvement in many studies and working groups, many things remain to be done.

Compared to its three main projects, first results should be available by the end of the year. First of all, Compost+ is updating the list of local authorities involved in biowaste collection in France. Indeed, last survey has been done in 2006 so that the data presented here are not exhaustive.

Dealing to the rest of its action, the website is already available but changes are expected this summer to make it as a real communication tool. The trimestral newsletter is send to Compost+' contacts and can be read right on the website by everyone. Compost+ co-organized the last Organic Meetings in Lorient in March with Cap Lorient local authority, and is planning the next Network Meeting that should take place in Pau.

4 CONCLUSIONS

Creating a network like Compost+ was essential to give a new impulse to biowaste collection and to give a help to the local authorities. As a conclusion, Compost+ is on the way to provide the lack of technical support, information and studies required by the sector to go forward and get more developed in France.

Moreover, with the new European regulatory framework in favour of biowaste collection, Compost+' objectives and actions will take a larger dimension. Results and conclusions of its work should be rich in 2012.

5 ACKNOWLEDGEMENTS

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(191) COMPOST QUALITY ASSESMENT AND EOW PROCESSUS APPLICATION TO 4 TYPES OF COMPOST

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EXECUTIVE SUMMARY

Analysis of compost is of major importance because it directly impacts on its final destination. According to the level of contamination and the national standards in each country, compost can be used on all crops including edible vegetables, on extended agriculture, on restoration lands, as landfill cover or simply landfilled if too contaminated. The End of Waste process introduced by the Waste Framework Directive 2008/98 and currently managed by the Joint Research Centre in Seville is moving a step forward. The compost that will acquire the European product status will no longer fall under the waste legislation, but will be considered as an ordinary good, freely tradable and exportable in all European countries.

Methods of analysis in European laboratories therefore need to be harmonised in order to :

1. Establish a sound set of European End of Waste standards, based on experience from various laboratories and on various types of composts, but all based on same methods;
2. Perform analysis on composts that will be valid and trusted across all Europe.

Three ring tests were performed in 2009, 2011 and 2012 on several compost types in order to evaluate the possible variations between laboratories results and methodologies. Green waste compost, biowaste compost, municipal solid waste (MSW) compost and sludge compost have been sampled and sent to specialised labs in Germany, the UK, France, Belgium and Spain. Parameters analysed were heavy metals, physical contaminants, PCB, HAP and agronomic parameters.

Discrepancies have been identified between labs for some pollutants and some compost types. In some cases, the heavy metals contents were 100% higher in one lab comparing to another one. The Chromium content is particularly subject to important variations between labs. The differences for the other metals are important, but analysis of variances (ANOVA) did not identify significant differences between labs. Physical contaminants are difficult to compare because each country has its own parameters.

Significant differences have not been identified between biowaste and green waste compost : except for dry matter content, which is more linked to composting methods than to the types of incoming waste, there is no significant difference between those two compost types made from source separated organic waste. However, Municipal Solid Waste composts significantly differ from the two other compost types on 2 pollutants : Zn and physical impurities. They also significantly differ from biowaste compost on Cd and Hg, and from green waste compost on Cr. Fluoranthen also significantly differs, but the concentration in MSW compost is much lower than in biowaste and in green compost. Based on average results, those three compost types do comply with the proposed End of Waste standards, while sludge compost does not comply because of higher content in Cu and Zn.

The Authors do not see a clear added value of the End of Waste for biodegradable waste because of the following reasons : a) transboundary movements of compost is not frequent, b) easier access to land will not be obtained through EoW because National legislation will still be able to limit the use of compost; c) harmonisation of compost standards throughout Europe does not have scientifically based grounds; d) some National standards systems will be strongly disturbed depending on the content of the positive input list of waste.

1 INTRODUCTION

Analysis of compost is of major importance because it directly impacts on its final destination. According to the level of contamination and the national standards in each country, compost can be used on all crops including edible vegetables, on extended agriculture, on restoration lands, as landfill cover or simply landfilled if too contaminated. The End of Waste process introduced by the Waste Framework Directive 2008/98 and currently managed by the Joint Research Centre in Seville, is moving a step forward. The compost that will acquire the European product status will no longer fall under the waste legislation, but will be considered as an ordinary good, freely tradable and exportable in all European countries.

Methods of analysis in European laboratories therefore need to be harmonised in order to establish a sound set of European End of Waste standards, based on experience from various laboratories and on various types of composts, but all based on same methods and perform analysis on composts that will be valid and trusted across all Europe.

The authors decided to carry out several compost analysis in different European laboratories to compare the results and methods currently used.
Additional questions were raised, related to the National standards and to the types of compost : “Is there any correlation between the level of impurities allowed in compost standards and the results obtained by National laboratories ?” and “Are there any significant differences between different types of composts, such as green waste, biowaste, municipal solid waste or sludge compost ?”

Three different analysis campaigns were carried out, in 2009, 2011 and in 2012.

2 RING TEST 2009 – Heavy metals

2.1 Test description

Six samples of the same compost were taken by the research centre of CIRSEE in spring 2009 from a single French MBT plant with AD stage, according to the European sampling standard EN12579. The compost originated from unsorted municipal solid waste, according to the following processes : pre-fermentation tube, trommel to keep the organic fraction < 20 mm, dry digestion, composting, screen 8 mm and ballistic separator.

The samples were sent to six well known and accredited laboratories in The Netherlands (NL), Germany (D), United Kingdom (UK), France (F), Belgium (B) and Spain (E).

The main parameters included :
- Dry matter, organic matter, pH, total N
- Heavy metals (Cd, Cu, Ni, Pb, total Cr, Zn, Hg)
- Physical impurities.

2.2 Results and discussion

2.2.1. Heavy Metals

The results acquired from the laboratories in NL, UK, B and F were consistent. Nevertheless, results in D were the lowest and those in E were the highest; with almost a 100% difference between both laboratories values (see table 1).

Table 1 : heavy metals concentration measured by the 6 laboratories

mg/kg DM	B	NL	D	E	UK	F	Mean	Std Dev
Cd	1,0	1,0	0,7	1,3	0,9	1,0	1,0	0,2
Cu	144	158	100	191	123	148	144	31,0
Ni	24	28	23	37	26	31	28	5,2
Pb	88	105	79	149	81	100	100	26,0
Cr total	36	41	30	59	32	44	40	10,6
Zn	338	403	320	671	323	413	411	133,4
Hg	0,4	0,4	0,4	0,4	0,3	0,5	0,4	0,1

The extraction method used for E, UK and F was the EN 13650, compared to the other labs that used their own extraction methods. It is important to highlight that in most cases, the methods are neither mentioned nor described in the results document provided by the labs.

Since no replicates were performed on the samples, it is difficult to conclude on any statistical interpretation. However, the differences within the results cannot be missed, illustrated by a relative standard deviation between 18 and 32%. The z scores calculated according to ISO 13528 show values around +1.9 for the Spanish lab compared to the German lab that indicates z scores of about -1.5, expressing the large variability.

The comparison between the results obtained from the labs and National Standards for heavy metals in compost confirmed that the MSW compost fully comply with the standards in D, UK, F, B and E. However the results were not compliant with the standards in NL, known as the more severe in Europe (see table 2).

Table 2 : National Standards for compost (* = values not respected by the compost tested)
Belgium : Federal standard, 2009 - Netherlands : Amended Fertilizer Act, 2008; Germany : Quality Assurance RAL GZ - Spain : Royal Decree on fertilizers cl C; UK : PAS 100 - France NFU 44051.

mg/kg DM	B	NL	D	E	UK	F
Cd	**2**	**1**	**1,5**	**3**	**1,5**	**3**
Cu	**150**	***90 ****	**100**	**400**	**200**	**300**
Ni	**50**	***20 ****	**50**	**100**	**50**	**60**
Pb	**150**	***100 ****	**150**	**200**	**200**	**180**
Cr total	**100**	**50**	**100**	**300**	**100**	**120**
Zn	**400**	***290 ****	**400**	**1000**	**400**	**600**
Se						**12**
As	**20**	**15**				**18**
Hg	**1**	***0,3 ****	**1**	**2,5**	**1**	**2**

The correlation coefficient calculated between the values set in the National Standards and the results given by the National labs is positive, indicating a possible relationship. Some of the coefficients were quite high, such as Zn (r = 0.91) or Cr (r = 0.82). However, these positive correlations are mainly explained by the Spanish situation that comprises high standards and high lab results. The other situations are more homogeneously distributed in the scatter diagram (see figure 1).

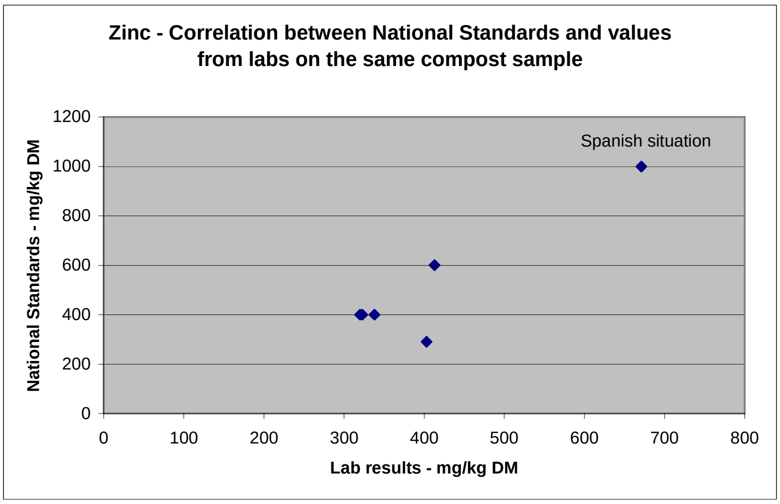

Figure 1 : Correlation between National Standards and values from laboratories on the same compost sample

2.2.2. Physical impurities

The National Standards set by the 6 countries vary largely, thus the interpretation of the results is quite difficult (see table 3). The parameter “stones>5mm” is the only identical parameter used by three Member States (D, B, NL).

Table 3 : National Standards for physical impurities (note that the French method for plastics analysis using bleach shows higher results, leading to a higher standard value)

% on DM	B	NL	D	E	UK	F
Glass + plastics + metals >2mm	<0,5	<0,5	< 0,5	< 3		
Glass + metals >2mm						< 2
Glass >2mm		<0,2				
Plastics >2mm					<0,25	
Other particules >2mm		<0,2				
Total contaminants >2mm					<0,5	
Glass >20mm		0				
Stones >5mm	<2	<2	< 5			
Stones >4mm					<8	
Films + PSE >5mm						< 0,3
Other plastics >5mm						<0,8
Total contaminants		<0,5				

If the total contaminants from the compost samples are summed up, then the standard deviation of 84% indicate a strong variation within the labs (see table 4).

Table 4 : physical contaminants in the MBT compost

% on DM	B	NL	D	E	UK	F	Average	RSD
Total contaminants	0,22	0,29	0,48	-	1,43	1,69	0,82	84%

2.2.3. Other parameters

The other parameters, dry matter, organic matter, pH and total N, indicate homogeneous results among the different laboratories.

3 RING TEST 2011

3.1 Test description

The compost from 8 plants operated by Suez Environnement in D, F, B and UK, made of green waste, biowaste or municipal solid waste, were sampled according to the European method EN 12579. The samples were sent to 5 laboratories in Germany, France, Belgium, Spain and the UK. In addition to the parameters already tested in 2009 (heavy metals, physical impurities and agronomic parameters), PAH and PCB were also tested in these samples.
With 8 different compost samples tested in 5 labs, the 2011 ring test enabled comparisons between laboratories, and between compost types. The results from the labs expressed as a value below a certain threshold (< x ppm) could not be handled for statistical analysis and have therefore been rejected.

3.2 Results and discussion

3.2.1. Comparisons between laboratories

3.2.1.1. Heavy metals

Z scores calculated with the ISO 13528 method show overall results below 2, which is interpreted as an acceptable result. However, variability among the labs shows fluctuations according to the types of compost. Spain shows the highest results for Zn and Cr (100% higher than the other labs) on compost 1, 5, 7 and 8; and for Pb on compost 5 (500% higher). Higher values for Ni and Cr (100% higher) on compost 2 and 4 were noted for the French lab. On the other hand, all other values were in accordance with the labs results. Relative standard deviations reflect this variability between laboratory results on heavy metals concentrations (see table 5).
None of the lab methods changed since the last ring test in 2009. Some of the compost results analysis showed similarities within the 2 ring tests concluded in 2009 and 2011 (i.e. higher concentrations for the Spanish lab); though there are still some exceptions.

Table 5 : Relative Standard Deviation on results on heavy metals from the 5 labs

	Relative Standard Deviation on results from the 5 laboratories							
	Compost 1	Compost 2	Compost 3	Compost 4	Compost 5	Compost 6	Compost 7	Compost 8
Cd	9,1%	18,9%	29,6%	21,3%	25,6%	11,7%	23,9%	10,6%
Cu	13,2%	7,8%	17,4%	6,7%	11,8%	5,9%	13,8%	12,4%
Ni	21,4%	76,7%	21,1%	41,9%	18,7%	21,6%	22,9%	57,1%
Pb	12,7%	11,7%	19,0%	18,2%	107,7%	9,3%	23,8%	14,7%
Cr total	45,6%	64,4%	39,5%	52,9%	29,0%	23,7%	56,3%	69,0%
Zn	38,1%	6,7%	14,4%	12,5%	7,8%	8,7%	19,2%	5,6%
As	0,0%	0,0%	3,3%	9,9%	0,0%	0,0%	0,0%	0,0%
Hg	61,6%	4,8%	13,6%	6,3%	30,2%	0,0%	39,1%	84,4%

The differences between lab results can become problematic when some results on the same compost are compliant with the Standards, while others are not. This can be illustrated by Mercury and Copper concentrations in compost n°8 (the proposed End of Waste threshold is 100 mg/kg DM for Cu and 1 mg/kg DM for Hg).

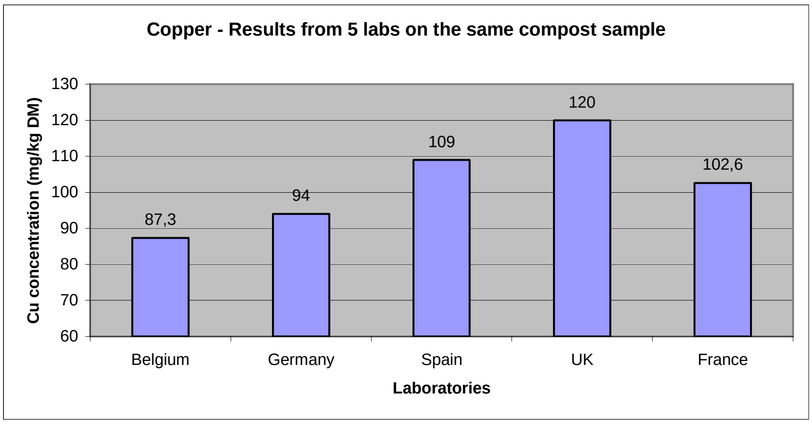

Figure 2 : Copper concentrations in the same compost sample, according to 5 different laboratories.

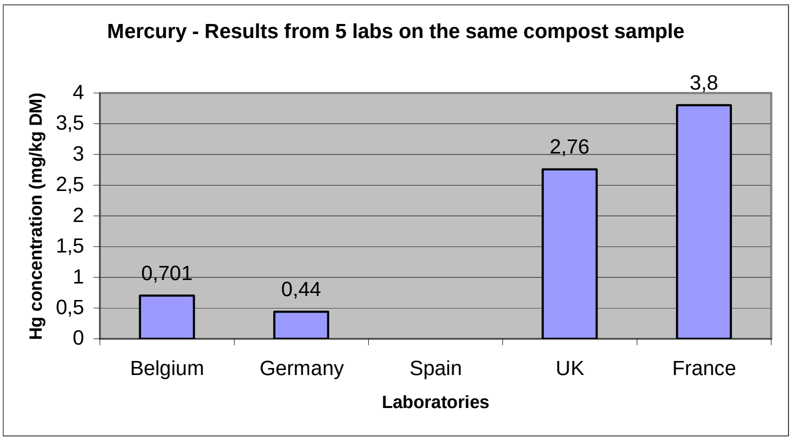

Figure 3 : Mercury concentrations in the same compost sample, according to 5 different laboratories

3.2.1.2. Other parameters

Values of the Relative Standard Deviation are low for most parameters : dry matter (RSD<2%), organic matter (RSD<20%), pH (RSD<7%) and minerals such as organic N, K_2O, P_2O_5, MgO and CaO (RSD<15%). However, N-NH_4 values are much more scattered between the various labs, with a RSD ranging from 65% to 132%.

For PCB and PAH, many results are expressed as a value below a given threshold, which prevent any statistical interpretation. Fluoranthen is the only organic pollutant where real concentrations are determined by all the labs, but the values are varying quite a lot (RSD from 16% to 74%).

3.2.1.3. ANOVA

Analysis of variance (ANOVA) was carried out on parameters showing high differences (i.e. heavy metals, fluoranthen, physical contaminants) and on more stable parameters (i.e. DM and OM). Except for Chromium which enabled to identify a significant effect of the labs on the results ($P<0.025$), no significant lab effect have been established for the other parameters. This is surprising when taking into account the high Relative Standard Deviations on some parameters (see table 5).

3.2.2. Comparisons between compost types

Three groups of compost were compared :

- Biowaste (4 plants in Germany, Belgium and France)
- Green waste (2 plants in France and UK)
- Municipal solid waste (2 plants in France).

Analysis of variance was carried out for the parameters heavy metals, fluoranthen, physical contaminants, DM and OM. Pairwise multiple comparisons procedures (Dunn's Method or Holms-Sidak Method) were performed in order to identify the couples that showed significant differences for each parameter (see table 6).

Table 6 : Statistical differences between compost types (* two surprising very high results on MSW compost were removed for the Hg comparison)

	Significant difference between compost types				
	Biowaste vs MSW	**Biowaste vs Green**	**Green vs MSW**	**Method**	**P**
DM	**yes**	**yes**	no	Dunn's	0,05
OM	no	no	no	-	0,153
Cd	**yes**	no	no	Holm-Sidak	0,05
Cu	no	no	no	-	0,107
Ni	no	no	no	-	0,801
Pb	no	no	no	-	0,606
Cr total	no	no	**yes**	Holm-Sidak	0,05
Zn	**yes**	no	**yes**	Dunn's	0,05
Hg	**yes**	no	no *	Dunn's	0,05
Fluoranthen	**yes**	no	**yes**	Dunn's	0,05
Physical contaminants	**yes**	no	**yes**	Dunn's	0,05

- Biowaste and green waste compost : except for dry matter content, which is more linked to composting methods (IVC) than to the types of incoming waste, there is no significant difference between those two compost types made from source separated organic waste.
- Municipal Solid Waste composts significantly differ from the two other compost types on 2 pollutants : Zn and physical impurities. They also significantly differ from biowaste compost on Cd and Hg, and from green waste compost on Cr. Fluoranthen also significantly differs, but the concentration in MSW compost is much lower than in biowaste and in green compost.

4 RING TEST 2012 - SLUDGE COMPOST

4.1 Test description

The compost from 2 French plants treating sludge from wastewater treatment plants were sampled according to the European method EN 12579. The samples were sent to 5 laboratories in Germany, France, Belgium, Spain and the UK. The same parameters were analysed (heavy metals, physical impurities, agronomic parameters, PAH and PCB).

4.2 Results and discussion

4.2.1. Comparisons between laboratories

4.2.1.1. Heavy metals

The results are given in table 7, and the Relative Standard Deviations are given in table 8. With the exception of Chromium for which the RSD is rather high (83%), the other RSD are much lower than the ones observed in 2011 for MSW and source segregated composts (see table 5). A more homogeneous mix obtained in sludge compost could be the reason. During the previous ring test in 2011, Chromium was also identified as a parameter showing large discrepancies between laboratories.

Table 7 : Heavy metals content in 2 sludge composts measured in 5 different labs

mg/kg DM	Compost n° 1					Compost n°2				
	Belgium	Germany	Spain	UK	France	Belgium	Germany	Spain	UK	France
Cd	0,83	0,64	0,7	0,78	0,87	0,97	1	0,7	0,9	1,19
Cu	155	140	125	165	159	208	220	172	234	219
Ni	19	13	12	14	27	18	17	14	15	31
Pb	54	55	50	63	63	40	44	41	39	54
Cr total	86	16	19	17	42	53	29	29	25	54
Zn	418	360	361	479	448	550	520	490	564	530
As	2,31	<5	<10	3,68	<4,1	3,13	<5	<10	5,32	<4,1
Hg	0,38	0,38	0,39	0,49	0,64	0,49	0,66	0,56	0,58	0,75

Table 8 : Relative Standard Deviations

	Compost 1	Compost 2
Cd	12%	19%
Cu	11%	11%
Ni	37%	36%
Pb	10%	14%
Cr total	83%	38%
Zn	13%	5%
As	32%	37%
Hg	25%	16%

4.2.1.2. Other parameters

Dry matter and organic matter contents and pH showed consistent results among labs. PCB levels were very low (0.002 to 0.003 mg/kg DM) and were often below the identification threshold, which prevented statistical analysis. PAH results were more scattered (total PAH from 0.7 to 2.6 mg/kg DM), with Residual Standard Deviation ranging from 40 to 60%.

As with previous campaigns, physical contaminants could not be easily compared due to the variety of parameters used by the different countries.

5 COMPARISON BETWEEN COMPOST TYPES

If we consider the average values, the composts made out of green waste, biowaste and MSW comply with the threshold proposed by the JRC for the End of Waste status (see table 9). But individual values for specific composts do not always comply (heavy metals or physical contaminants). However, the sludge compost is not complying with the proposed EoW standard because of the high content in Copper and Zinc (some Member States call for a revision of the values for Cu and Zn in the proposed European standards for EoW).

Table 9 : Average values for some pollutants on 4 compost types (* two surprising very high results were removed for Hg in MSW compost , ** Standard proposed by JRC Seville for future End of Waste status)

		Average values				
		Green	**Biowaste**	**MSW**	**Sludges**	EoW JRC
DM	%	0,59	0,70	0,56	0,55	
OM (on DM)	% on DM	0,45	0,44	0,49	0,44	
Cd	mg/kg DM	0,53	0,37	0,70	0,9	**1,5**
Cu	mg/kg DM	60,46	61,14	82,78	**180**	**100**
Ni	mg/kg DM	13,00	18,09	17,75	18	**50**
Pb	mg/kg DM	72,42	53,09	44,66	50	**120**
Cr total	mg/kg DM	18,55	27,79	38,48	37	**100**
Zn	mg/kg DM	194,19	197,21	279,16	**472**	**400**
As	mg/kg DM	5,03	3,11	2,85	3,6	
Hg	mg/kg DM	0,12	0,09	0,40 *	0,53	**1**
Fluoranthen	mg/kg DM	0,6150	0,3901	0,1894	0,4316	
Physical Impurities	% on DM	0,09%	0,26%	0,66%	0,94%	

6 END OF WASTE FOR BIODEGRADABLE WASTE

In 2011, the Joint Research Center in Seville carried out a screening exercise on a large number of composts from 14 Member States. The 162 samples came from composts or digestates made out of biowaste, greenwaste, sludge, MSW, manure or energy crops.

Very limited amount of results (on only 5 plants) were published in the Second Working Document in October 2011. At the time of writing this article, no further data were available yet. The results will certainly be very interesting to know. However, it seems that the analysis were all performed at a limited number of laboratories, making difficult a comparison between European laboratories and the consistency of the testing methods.

The Authors do not see a clear added value of the End of Waste for biodegradable waste because of the following reasons :

1. The first goal of the product status is the free transportation around Europe. If this aspect is quite important for streams like recycled metals or plastics, compost value is however very low and does not allow long transportation distances between Member States (except for small countries).
2. Easier access to land is not obtained through End of Waste. The waste legislation will no longer be applicable to compost, preventing waste Authorities to regulate/limit its use. But agricultural and environmental protection legislations will still be applicable. Member States and Regional Authorities will be able to set agricultural standards that suit their own country.
3. Harmonisation of compost standards in Europe is not scientifically justified. Soils characteristics, climate, agriculture, social habits, environmental concerns are very different throughout Europe. National Standards are the best environmental solution
4. European Product Status is a burden to National Product Status. There are strong differences between Member States. Some MS want to include MSW compost, or sludge compost, or digestates, while other countries will never accept them. Inclusion or exclusion of some streams in European End of Waste will be detrimental to one or the other.

7 CONCLUSIONS

The ring tests performed in 2009, 2011 and 2012 lead to the following conclusions :

1. There is a large variability both in terms of laboratory results and in terms of compost qualities. It is then of an upmost importance to consider a significant amount of samples and replicated analysis before drawing any conclusion.

2. Even if the differences identified among labs are not declared statistically significant, surprising variations are observed for the same compost. National Laboratories use different analytical methods, and a European harmonisation of analytical methods must be achieved before setting European End of Waste criteria for compost. This is particularly true if one considers the low added value of a European End of Waste status for compost.

3. The analysis of variance established significant differences between MSW composts and the 2 types of source separated compost for Zn, Fluoranthen and physical impurities. MSW composts contain more Zn and physical impurities but less fluoranthen. Composts made out of green waste, biowaste and MSW show an average quality level that complies with all the threshold values proposed for the EOW status, except MSW compost for physical impurities. Sludge compost show Cu and Zn contents that are higher than the proposed European threshold.

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(214) EXPERIENCE OF DEVELOPING A COMPOST QUALITY ASSURANCE SCHEME IN IRELAND

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EXECUTIVE SUMMARY

rx3, www.rx3.ie, is a Department of the Environment, Community & Local Government (DECLG) Programme established to help develop markets for recycled material in Ireland to form new products, including compost. Barriers to market development for compost products in Ireland identified in the past included the lack of a compost quality assurance scheme (CQAS) and a national compost quality standard (CQS) to prove that the process has been independently verified as being bona-fide and the compost is high quality and suitable for the intended purpose. rx3 has an objective to address this issue by working with stakeholders and using existing work.

Cré, the Composting and Anaerobic Digestion Association of Ireland, conducted research that was funded by the Environmental Protection Agency (EPA). This research analysed Irish compost quality databases and proposed appropriate limit values for certain parameters. Cré then developed an industry quality standard for source separated biodegradable waste derived compost, tailored to Irish conditions. This standard proposed limit values for parameters including heavy metals, pathogens, impurities, stability and organic matter. This report "*Development of an Industry-Led Quality Standard for Source-Separated Biodegradable Material Derived Compost*" was published in 2009.

In 2009, rx3 requested the National Standards Authority of Ireland (NSAI) to develop a CQS for source separated biodegradable derived compost. The NSAI used published research that compared Irish compost quality databases with databases and standards from other European countries. Irish Standard 441:2011 was published June 2011.

rx3 appointed Certification Europe to develop a CQAS using I.S. 441 specifications. Work started February 2011 and comprises three phases.

- Phase one: initial drafting of the relevant CQAS documentation.
- Phase two: trialling of the draft CQAS at suitable compost facilities. During this phase a competent auditor audits the facility using the Draft CQAS as criteria and identifies areas for improvement and recommendations. A site-specific training package and an action programme with defined responsibilities and timelines is developed and presented to the operator. Certification Europe assists the organisation implement recommendations and prepare for pilot certification. The key challenge is balancing CQAS requirements with existing regulatory requirements. Not adding regulatory burden is important for successful uptake of the CQAS, by the composting industry.
- Phase three: operating the CQAS on a pilot basis.

Twenty composting facilities have been recruited, audited and trained by March 2012, representing 69% (288,500 tonnes) of the operational capacity of Irish composting facilities. Results from the trialling phase have highlighted that the majority of producers are well prepared and have documented procedures and processes that support a seamless progression to certification. Furthermore, the feedback from operators has been very positive.

Certification Europe has developed a quality mark, to assist in marketing and identifying high quality compost, increasing overall awareness and understanding of quality issues in compost.

CQAS I.S. 441 will enter phase three in July 2012, when it will be implemented on a pilot basis, with facilities being certified and certified compost coming onto the market. An awareness programme is being implemented to develop awareness of the scheme in compost users. Following development, the CQAS will be self-funding.

1 INTRODUCTION

This paper details the steps that were taken in developing a compost quality assurance scheme in Ireland, from project inception to the current stage of development and future steps.

1.1 Background

Irelands composting sector has grown rapidly to 58 composting facilities in 2012, with estimated operational capacity of 415,100 tonnes. Capacity must grow further due to projected increases in waste generation and segregation rates due to regulatory drivers including Landfill Directive and Waste Framework Directive targets. Figure 1 shows estimated growth in capacity and composting facility numbers, 2000 to 2011, EPA, (2002), EPA, (2005), Intertrade Ireland, (2011), Forfás (2008), rx3 (2012).

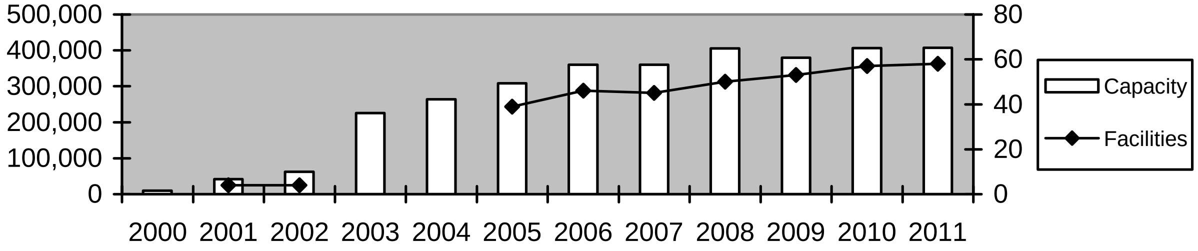

Figure 1: Number of composting facilities and approximate operational capacity 2000-2011

As composting capacity provision increases, commercial imperatives drive composting facilities to diversify revenue generation models from “gate-fee only” to “gate-fee plus compost sales”. This move, in turn, requires production of quality compost to guarantee human, environmental, animal, plant and soil, safety of compost applications in domestic, agricultural, horticultural and landscaping industries by maintaining consumer confidence.

Production of quality assured compost demonstrates that the composting process has been independently certified as being *bona-fide* and that the compost is high quality and suitable for the intended purpose. A CQAS also provides a quality mark, which assists in marketing and identifying high quality compost products.

Accordingly, a number of Irish organisations collaborated to deliver and to fund a sequence of three projects, which have delivered sequentially an industry compost quality standard, a national compost quality standard and a national compost quality assurance scheme. These three projects are described in this paper.

1.2 Objectives

The objective of the three-phase project is to develop a system to allow compost producers to assure compost users that compost purchased meets certain minimum quality criteria. This development in turn will support compost markets development in Ireland, leading to increased recycling and reduced landfilling of waste.

This objective is being delivered in three sub-projects. The identified objectives of these sub-projects are;

- To collate the compost quality databases from Irish compost samples, and compare them with databases and standards from other European countries in order to develop an Irish industry compost standard for source separated biodegradable materials.
- To develop an Irish Standard that specifies quality requirements for compost produced from source segregated, separately collected, biodegradable materials including biodegradable municipal waste.
- Develop a national compost quality assurance scheme.

1.3 Project overview – phases, timelines, participants, funders

This project has been implemented since 2008 with a number of participants leading and funding work. The table below shows project timelines and main actors in the project.

Phase	**Date**	**Work led by**	**Work funded by**
Industry compost quality standard	2007- 2009	Cré, www.cre.ie	EPA www.epa.ie
National compost quality standard I.S.441:2011	2010-2011	NSAI, www.nsai.ie	DECLG www.environ.ie through rx3 www.rx3.ie/Organics
CQAS 441:2011, development	2011	Certification Europe www.cqas.ie	
CQAS 441:2011, trialling	2011-2012		
CQAS 441:2011, pilot operation	2012-2013		
CQAS 441:2011, implementation	2013-	To be decided	Participating facilities
Communications activity	2008-2013	rx3	DECLG www.environ.ie

Table 1: phases, timelines, participants, funders

2 INDUSTRY COMPOST QUALITY STANDARD

Cré, the Composting and Anaerobic Digestion Association of Ireland, conducted and industry standard research project, funded by the Environmental Protection Agency (EPA). The purpose of this project was to offer a set of quality standards for compost derived from source-separated biodegradable materials, to promote the development of Irish markets for compost-based products, and to protect human, plant, soil and animal health.

This project collated the compost quality databases from Irish compost samples, and compared them with databases and standards from other European countries in order to develop an Irish industry compost standard for source separated biodegradable materials, tailored to Irish conditions.

2.1 Methodology - developing an industry compost quality standard

The following points outline the methodology used for developing the industry compost quality standard:

1. Results of laboratory analysis of 256 compost samples, made from source-separated biodegradable materials, at Irish composting facilities were collated. Statistical analysis was conducted on the collated database.
2. Upper limit values were determined for heavy metals and pathogens. Limit values for heavy metals were developed using the recommendations of Amlinger et al. (2004), which advocates that countries starting source-separated collection use the 90th percentile of the country's compost testing database plus 50%.
3. Pathogen limit values were determined by reference to limit values for *E. coli* and *salmonella sp.* applied in animal by-products regulations.
4. The impurities limit value was determined by comparing levels in Irish samples with limit values in European standards and by considering the limit values proposed in 2nd draft of the Technical Working Document for the Treatment of Biowaste and in End of Waste Criteria reports.
5. Stability parameter data was examined and Irish compost samples were tested using the oxygen uptake rate test (OUR). The OUR test was selected as it is relatively low cost and delivers a result fast. The OUR limit value was agreed using test results generated during the project.
6. The criteria for a compost standard outlined in End of Waste Criteria reports were examined and the standard amended to take cognisance of criteria such as a minimum requirement for organic matter content in compost.
7. A draft standard was presented to stakeholders in two separate consultative workshops. Based on comments from the two workshops and feedback from the project's steering committee, a final report was produced for acceptance by the steering committee.

2.2 Results

The industry compost quality standard that was proposed for Ireland is as shown in the table below.

Parameters	**Criterion**	**Limit Value**	**Units**
Heavy metals	Cadmium	1.3	mg/kg DM
	Chromium	92	mg/kg DM
	Copper	149	mg/kg DM
	Lead	149	mg/kg DM
	Mercury	0.4	mg/kg DM
	Nickel	56	mg/kg DM
	Zinc	397	mg/kg DM
Pathogens	*Escherichia coli* in fresh mass	1,000	cfu per g
	Salmonella sp.	0	per 25g
Organic matter	Organic matter minimum	20%	% dry weight
Impurities	Total glass, metal and plastic >2 mm Ø	0.5%	% by weight
Stability	Oxygen uptake rate	13*	mmol O2/kg organic solids/h
*NOTE: By 2014 there is an objective of a limit value of 10 mmol O2/kg organic solids/h.			

Table 2: Criterion limit values proposed by the industry compost quality standard

2.3 Industry Compost Quality Standard - Conclusions and Recommendations

The industry compost quality standard report "*Development of an Industry-Led Quality Standard for Source-Separated Biodegradable Material Derived Compost*" was published by the EPA in 2009. It recommended that the industry standard be considered "preliminary" and that it be updated as required while source-separated collection schemes and associated treatment facilities are being established in Ireland.

The report recommended that the industry standard be developed into a national standard, and that a voluntary, independently audited compost quality assurance scheme be developed around the standard. It also recommended that the CQAS develop a list of acceptable source separated feedstocks and that it determine and standardise sample frequency, methods and any allowed deviation from the Standard.

3 IRISH NATIONAL STANDARD

In 2009, rx3 requested the National Standards Authority of Ireland (NSAI) to develop a national standard for source separated biodegradable derived compost. A national standard is national specification based on the consensus of an expert panel and subject to public consultation. Accordingly, the NSAI established, and acted as secretariat to, a Working Group that represented composting sector stakeholders including producers, consumers, regulators, researchers, Government and composting experts.

This Working Group agreed definitions, feedstocks, sampling, analysis procedures, packaging requirements, and agreed limit values aligned to those suggested by the industry quality standard.

It specified that feedstock materials must comply with applicable legislation and specified the following criteria;

- It must have been segregated from non-biodegradable materials and collected separately, and must not be mixed, combined or contaminated with other potentially polluting wastes, products or materials.
- Plastics and packaging must have been independently certified as conforming to 'compostable' criteria.
- Wood feedstocks must not be impregnated with preservatives, paint, or non-biodegradable layers.
- Feedstock must not contain sewage sludge or any sewage derivative or mixed municipal solid waste material.
- Additives used must confer a benefit or reduce emissions, and not to dilute of characteristics that are limited.

The frequency of sampling is required to be in accordance with the facility's regulatory requirements, thus minimising additional regulatory burden of compliance with the compost quality standard. Methods for sample preparation for laboratory analysis are specified, as are test methods for each criterion.

The product quality limit values are as listed in Table 2, with addition of a limit value of 2 viable weed seeds per litre. The OUR method, which is now an accepted *CEN* method, was selected to determine stability. It is important to note that the stability limit values proposed are strict, particularly for the future.

This Irish Standard does not specify maturity tests at present as these tests are still being finalised by CEN TC 223 (Soil improvers and growing media).

The standard requires that parameters relating to beneficial qualities of compost be measured, including pH, plant nutrient content (total nitrogen, phosphorus and potassium, ammonium and nitrate nitrogen) and dry matter content. Following a formal, national, public consultation exercise, and subsequent Working Group review the standard was published by NSAI as Irish Standard I.S. 441, Quality requirements for a compost manufactured from source segregated, separately collected, biodegradable materials in June 2011. This standard is copyrighted and available to purchase through the NSAI. See www.rx3.ie/National-compost-quality-standard.

3.1 Discussion

The I.S. 441 limit values are compared with those defined in selected standards in figure 2 below where I.S. 441 is denoted by the 100% line. Note: the *Salmonella sp*. limit value is 0/25g for I.S. 441 and 0/50g for the end-of-waste proposal respectively, so the end-of-waste limit value is denoted as 50%. All other comparisons are in real numbers. Figure 2 indicates that I.S. 441 limit values are intermediate between other compost quality standards, except for the mercury limit, where the Irish limit is notably lower. I.S. 441 requires equivalent or lower limits than PAS 100 (except weeds and impurities), and Class 2 of 2nd draft of Biowaste EU working document, as used by Irish EPA. Class 1 limits are stricter or approximately equivalent to I.S. 441. Differential with criteria proposed in 2011 End-of-waste document is varied. These differences reflect Ireland’s stage in developing a brown bin source segregation system.

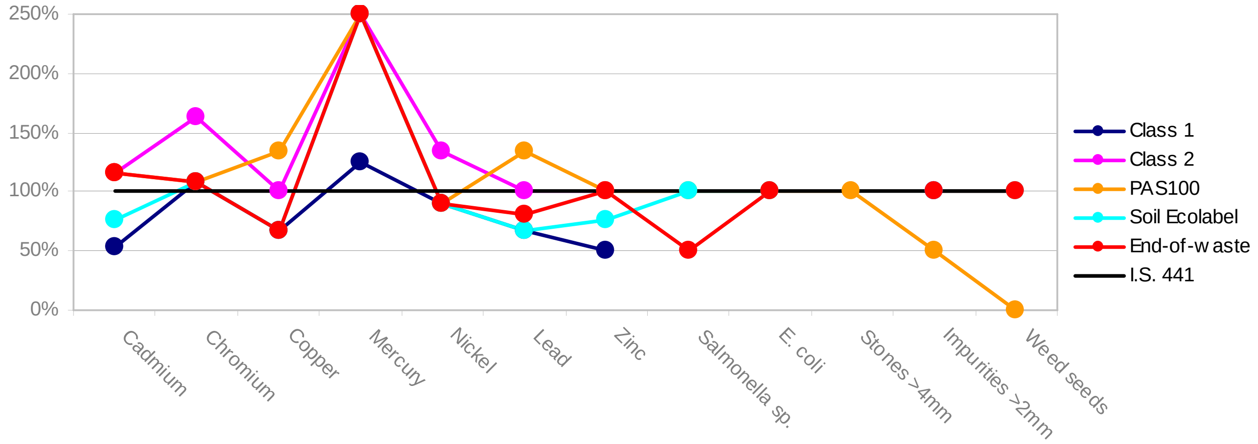

Figure 2: Comparison of I.S. 441 limit values (100%) to limit values of selected standards

This product standard is used as part of the compost quality assurance scheme described in the following section. Other applications of the standard are being pursued, including influencing the development of a digestate quality standard, underway in April 2012.

4 COMPOST QUALITY ASSURANCE SCHEME

The Market Development Programme 2006 tasked rx3 with developing a compost quality assurance scheme. Therefore, following a procurement process, rx3 appointed Certification Europe Ltd. to develop a National CQAS. Work on the compost quality assurance scheme project started February 2011 and comprises three consecutive stages.

1. Initial drafting of the relevant CQAS documentation – 6 months.
2. Trialling of the draft CQAS at suitable compost facilities – 12 months.
3. Operating the CQAS on a pilot basis – 12 months.

Running concurrently in all stages, and increasing significantly during stages 2 and 3 are awareness raising activities.

4.1 CQAS - drafting of documentation - February 2011 - August 2011

The objective of this phase was to gather and develop necessary knowledge. This was done by conducting a literature review of the relevant documentation including a review of existing compost quality assurance schemes to gather relevant information on the issues involved in developing a CQAS for Irish composting facilities. This phase saw the development of a quality assurance scheme for the composting industry along with the associated documentation and guidance and training materials necessary. A Certification Body inspection protocol was also developed.

4.2 CQAS - trialling the draft scheme - August 2011 - June 2012

The objective of this 2 step phase was to trial the CQAS and associated documentation on fifteen composting facilities. Twenty one composting facilities applied to participate, and allocations were made to accommodate all applicants. Trialling was used to evaluate the CQAS, to highlight any issues and to make improvements. During this phase a competent auditor audits the facility using the Draft CQAS as criteria and identifies areas for improvement and recommendations. A site-specific training package and an action programme with defined responsibilities and timelines is developed and presented to the operator. Certification Europe helps the organisation implement recommendations and prepare for pilot certification. A key challenge to the success and uptake by the composting industry of the CQAS is balancing CQAS requirements with existing regulatory requirements.

An in-depth audit of each facility against the requirements of the CQAS followed. This audit addressed all elements of the CQAS from the initial application process through to the sampling, quality management and operating conditions. The composting operator was left a ‘gap audit’ report which identified deficiencies (if any) in their system against the CQAS, and presented an action plan. Each participating site was then given ½ day overview training of the CQAS. This gave the operator an opportunity to understand the purpose of the trialling process, the workings of the CQAS and to promote discussion about the composting operations.

Next was an evaluation of the effectiveness of the operator in implementing the gap audit findings. This gave an indication of effectiveness of the CQAS and difficulties or issues that operators faced in implementing the CQAS. Twenty one composting facilities have been recruited, audited and trained by March 2012, representing 69% (288,500 tonnes) of the operational capacity of operational Irish facilities. Results from the trialling phase indicate that the majority of producers are well prepared and have documented procedures and processes that support a seamless progression to certification. Furthermore, and very importantly, the feedback from operators has been very positive.

4.3 CQAS - pilot operation - July 2012 - June 2013

CQAS I.S. 441 will enter stage three during July 2012, when it will be implemented on a pilot basis. The objective of this stage is to pilot the CQAS on fifteen composting facilities.

Certification Europe will commence the certification process with the facilities, following their application. Facilities will be audited against the requirements of the CQAS and if appropriate be awarded certification. Composting facilities that apply for the pilot stage will have started the certification process and it is expected that at the end of this phase a number of composting facilities will have been successful in getting certification to the CQAS.

During this first year of formal certification of composting facilities, financial support will be made available for composting facilities, so costs to operators are subsidised. The scheme will be self-financing from year 2.

5 AWARENESS RAISING

An awareness programme has been implemented concurrently with the development of I.S. 441 and the compost quality assurance scheme. This awareness programme has been conducted by rx3 to develop awareness of quality compost in compost users.

Examples of support activities conducted include; articles in newsletters and press and conferences; online directory listing quality composting facilities and quality compost products www.rx3.ie/Directories; engagement with stakeholders to raise awareness of quality compost; crop trials and pot trials demonstrating use of quality compost; quality compost characterisation study. See www.rx3.ie/Organics for details.

Certification Europe has developed a quality mark. This mark will assist in marketing and identifying quality assured compost, increasing overall awareness and understanding of quality issues in compost, by providing a unique visual identifier that purchasers can look for. The mark will be a central theme of a marketing and awareness campaign during the awareness raising stage. This mark is shown in figure 4.


NSAI
Standards
Irish Standard
I.S. 441:2011
Quality requirements for a compost manufactured from source segregated, separately collected, biodegradable materials

Figure 3: Irish national compost quality standard I.S. 441, published June 2011

Figure 4: Quality mark of the national compost quality assurance scheme CQAS 441:2011

6 CONCLUSIONS

The overall aim of this project was to develop an Irish compost quality assurance scheme for source separated biodegradable materials, in order to promote the development of markets for compost-based products on the island of Ireland as well as to protect human, plant, soil and animal health

A high degree of interest has been shown by composting facilities - Phase 2 was oversubscribed with 21 composting facilities participating, representing over 69% of Irish operational composting capacity.

This signals that composting facilities, having seen the benefits of producer participation in quality assurance schemes in food and other sectors, have confidence that their markets will be improved through participation.

The compost quality assurance scheme for compost will be in operation from July 2012. When the scheme has operated for some time, the benefits of improved quality and improved markets as a result of the quality compost project will become more apparent.

The success of the scheme is dependent upon awareness activities delivering the message about the scheme to the compost use sector. Industry, and rx3, activity will be delivering these awareness activities, as outlined in Section 5. In addition, other activities to support market development for quality compost are being considered and may be rolled out accordingly. These activities being considered include encouraging use of the compost quality assurance scheme in green public procurement, and encouraging recognition of the compost quality assurance scheme by regulators. The compost quality assurance scheme will inform Ireland’s engagement with EU end-of-waste processes for biowaste.

The compost quality assurance scheme gives the consumer an option to purchase quality, certified compost in which they have confidence that it is safe and meets high standards, and next actions must bear this consumer in mind.

Irelands experience to date with developing a compost quality assurance scheme has been illustrative of successful collaboration by a number of organisations in delivering a shared goal.

7 ACKNOWLEDGEMENTS

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Thanks are extended to Louise Connolly for helpful remarks in presentation of the manuscript.

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(216) THE MYTH OF WASTE AVOIDANCE-WASTE DOES NOT CAUSE WASTE

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EXECUTIVE SUMMARY

In the discussion about sustainability many people consider *waste avoidance* as a key measure how material flows of humans could be omitted. This is based on the conviction that waste avoidance protects the *environment* and permits sustainable acting. However, waste management data show that the gross domestic waste amounts, i.e. the total of recycled, treated and disposed household waste, bulky waste, street sweeping waste, green waste and market waste in Germany remains unchanged at a level of 500 to 600 kilograms per capita and year (kg/cap*a) during the last decades (similar also in other industrialized countries). This means no waste has been avoided. Why does waste avoidance not work?

The present paper is based on the thesis that waste avoidance does not work because waste avoidance does not exist. Waste cannot be avoided, only products. So far the fact is repressed: not the waste but the products are the reason for waste. Consequently only products can be avoided and not waste which has already emerged and accumulated as a result of human production and consumption (see the same misunderstanding in the sustainability debate related to the term “dematerialisation”). However, the absolute necessity that the high material throughput of people has to be reduced by product avoidance with its consequences of life style change is still a taboo for the majority of people in industrialised countries. The present paper wants to be conducive to eliminating the link of waste management to an alleged waste management domain called *waste avoidance*. The challenge consists in combining waste management/economy in the future with product economy in a higher-level domain of *sustainability*. This “new economy” of global matter management could be labelled as *adduct management or adduct economy* referring to the idea that matter is in permanent change or transformation. A*dduct* is both product and waste. In that way product avoidance - as disagreeable as this idea might be for many people - can be considered as one of the biggest challenges in the 21st century. This is the only way to find the path to sustainability as meant in the Brundtland-Report in 1987 contributing to the long-term conservation of the earth as a whole (not an “undefined” environment) by reducing the material throughput of mankind.

1. INTRODUCTION

Over centuries the handling of waste developed stepwise from a simple dropping of “chunk” in handcraft [*Kuchenbuch*, 1987] (see German *abfallen* = English *to fall off*), the disposal of excrements on the street and sweeping of “fine-grained” garbage in the household to a differentiated economic sector not dissimilar to production [*Hösel*, 1987, Grooterhorst, 2010]. Waste management in many countries of Europe is functioning and appears to be clearly structured. “Waste management” recycles and disposes waste. Also the future way of waste management in Europe seems to be clearly sketched out: less and less (avoidance), more and more separated (reutilization and recycling), cleaner and cleaner (treatment and disposal). Therefore, it becomes to a stepwise more rational – literally “calculable and reasonable” - waste management. Will it really develop in this linear way?

Derived from forestry and biology the idea of *sustainability* with its ecological load was developed in the 1980s and compiled in the Brundtland-Report [United Nations, 1987]. At that time it became clear that the use of matter in the industrialized countries would end in a depletion of earth resources, even in a severe damage of the *earth as a whole* (EARTH) [OPEN, 2011]. The basic concept of sustainability is in short: *Consume only the amount of matter that both the present and future generations can satisfy their (basic) needs*. An additional aspect of this principle is that the amount of matter available for human use is limited. The consumption of the individual and the mankind as a whole has to be coupled to this limit not putting the EARTH at risk. This refers to the sources of matter as well as the sinks of matter and at present it can be stated that especially the EARTH as sink becomes restricted.

In this regard the “rule” *sustainability* has to be extended: *Consume only the amount of matter that both the present and future generations can satisfy their (basic) needs and the earth as a whole will be maintained for all living beings*.

This means a double back coupling is needed from each human being; into the matter use of all other human beings and into the amount of matter which the earth can provide for human puposes without getting damages or destroyed.

Main addressees of this principle are the industrialized nations which are since the beginning of the debate about sustainability under criticism that their citizens use too much of the “natural resources” (This is the EARTH!) to keep their standard of living. For instance, in Germany the consumption of an individual is more than 70 tons of matter to keep the living standard [Schütz und Bringezu, 2008]. This figure results from the amount of biomass, ore, mineral construction material, and fossil energy carrier to generate all products in the country and abroad. In many countries of the earth this amount is clearly below 10 tons per individual and year.

But to what extent should that flow of materials caused by taking of biomass, ore, mineral construction material, and fossil energy carriers from EARTH be reduced? To date no one wants to reduce the existing number of products, no one wants to avoid what constitutes the prosperity with its comfort. At the end of consumption process the product is transformed into waste, so to speak, the “anti-product”. Through waste, the disagreeable concomitant phenomenon of the man made material flow is detectable directly with all senses. So, it was obvious that attention was given to the avoidance of waste not being used anymore and not to the products. The idea of waste avoidance coevolved with the idea of sustainability. As the term is put together from *waste* and *avoidance* it appeared “logic” that waste avoidance is a responsibility of the waste sector and waste management. Waste should be avoided “out of” the waste sector. As a result of these conclusions *waste avoidance* was written into the legal framework for waste management.

Since then waste management has to deal with that cuckoo´s egg called *waste avoidance*. And, everyone dealing with waste management is always facing the question: How to avoid waste? The answer probably is surprising: *Waste cannot be avoided!*

2. METHODOLOGY

2.1 Management of matter and material throughput

The waste sector is an integral part of the overall economy. In the worldwide management of material starting from extraction of raw materials, to production, trade and the consumption waste management is the final step (see also sewage water, exhaust air and other emissions). If the flow of materials changes in the front part of this sequence (chain) - basically it is a complex network of economical relationships in time-space – the character and amount of waste and other emissions will consequently change.

According to the SRU Report [SRU, 1990] **figure 1** shows a simple two layer model of this product-waste sequence which is driven by the motifs of humans whereas these motifs can be more comprehensive than just existential requirements [Grooterhorst, 2010a]. The motifs of a human being and his ability for target oriented awareness and remembrance result in the fact that from an “undefined”, very often nameless or not yet labelled matter in its “natural environment” (This is the EARTH!) becomes a “defined” matter in the so-called “civilized environment” (This is also the EARTH!) like raw materials, products, trading goods, consumer goods and finally waste whereat it is always the same matter. The undefined matter is water, soil, air and energy carriers. Both the natural environment and civilised environment are considered by humans as “our” environment or our sphere of living which is nothing else than the EARTH (an even more reduced view at the earth is “production factors and soil” (Adam Smith, 1723 - 1790; [Smith, 1999]).

An individual human being requires a certain amount of matter that can be measured in tons per year and consists of biomass, ore, mineral construction material, and fossil energy carriers. This amount consists of material flows inside and outside the country and is moved by or for each individual and is utilized to keep the standard of living. This annual demand per inhabitant which has a typical mean value for every national economy and that can be calculated using data of the national physical statistics; something which is already done in many European countries [European Commission, 2012] will be termed in the following ***material throughput***. The associated indicator is the *Total Material Requirement* (*TMR*) expressed either in tons per individual and year (t/cap/*a) or tons per nation and year (t/a) [Bringezu et al., 2002; Bringezu und Schütz, 2001; Bringezu, 2004].

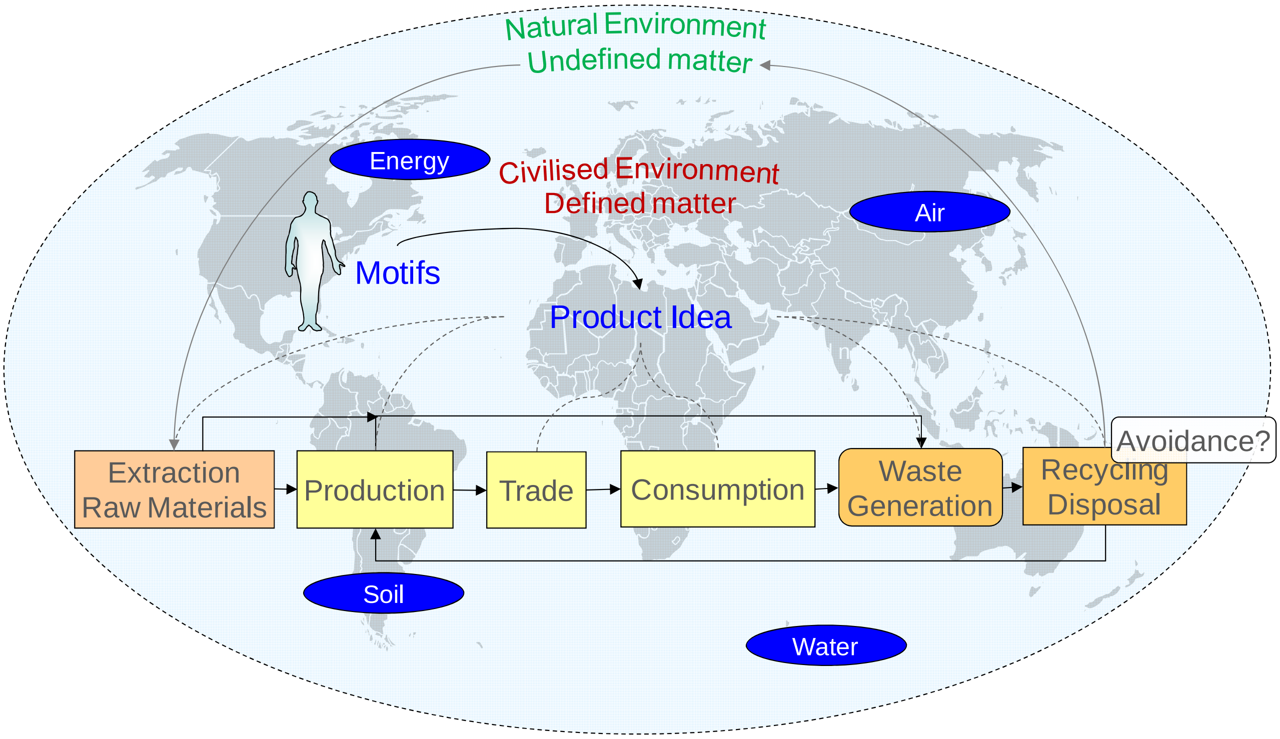


Fig. 1: Sequence of product and waste generation driven by human motifs in a simple and traditional two layer-model

Significant and sometimes fast alterations material throughput can happen in three ways:

1. Collapse of economic activities due to wars or economic crisis. The competition for finite resources is one of the causes of wars.
2. Mankind is forced to reduce the material throughput as it has negative consequences for the EARTH and therefore for all individuals at billions of their living locations. The sinks are either full or overused.
3. Mankind identifies all these risks and is capable to of taking the responsibility for his material throughput in terms of amount (sufficiency end efficiency) and properties (resilience and consistency) at an early stage (now!). Mankind recognizes the necessity to balance source and sink [OPEN, 2011].

Something will *happen* to “the concerned” or they *act* according the identified shortcomings. On the basis of the material balances of all countries of the planet– with usual inaccuracy of estimation – the *used* and *unused* quantities of matter extracted from the EARTH can be determined. The last available balance of 2008 provided by SERI in Vienna is given in **figure 2** (1980 - 2008). The *Global Material Extraction* (used + unused), taken from all countries of the world, is approximately 111 billion tons per year [SERI, 2012]. Also referring to the last available balance year of 2008 (used + unused) and the related world population of 6.8 billion the mean specific *Global Material Extraction* for one individual on earth is about 16.4 t/cap*a [SERI, 2012]. Assuming that the consumption patterns will not change in average and, thus, the mean specific *Global Material Extraction* for one individual will not change the development of the *Global Material Extraction* until 2100 can easily be estimated and is also given in figure 2. Referring to the optimistic world population forecast of IIASA [Lutz et al., 2008] the *Global Material Extraction* will increase until 2065 up to approx. 147 Billion tons per year slightly decreasing until 2100 to around 140 Billion tons per year due to the assumption that the fertility of the world population will decrease. If more humans come to the decision to increase their material throughput the curve would tremendously move upwards.

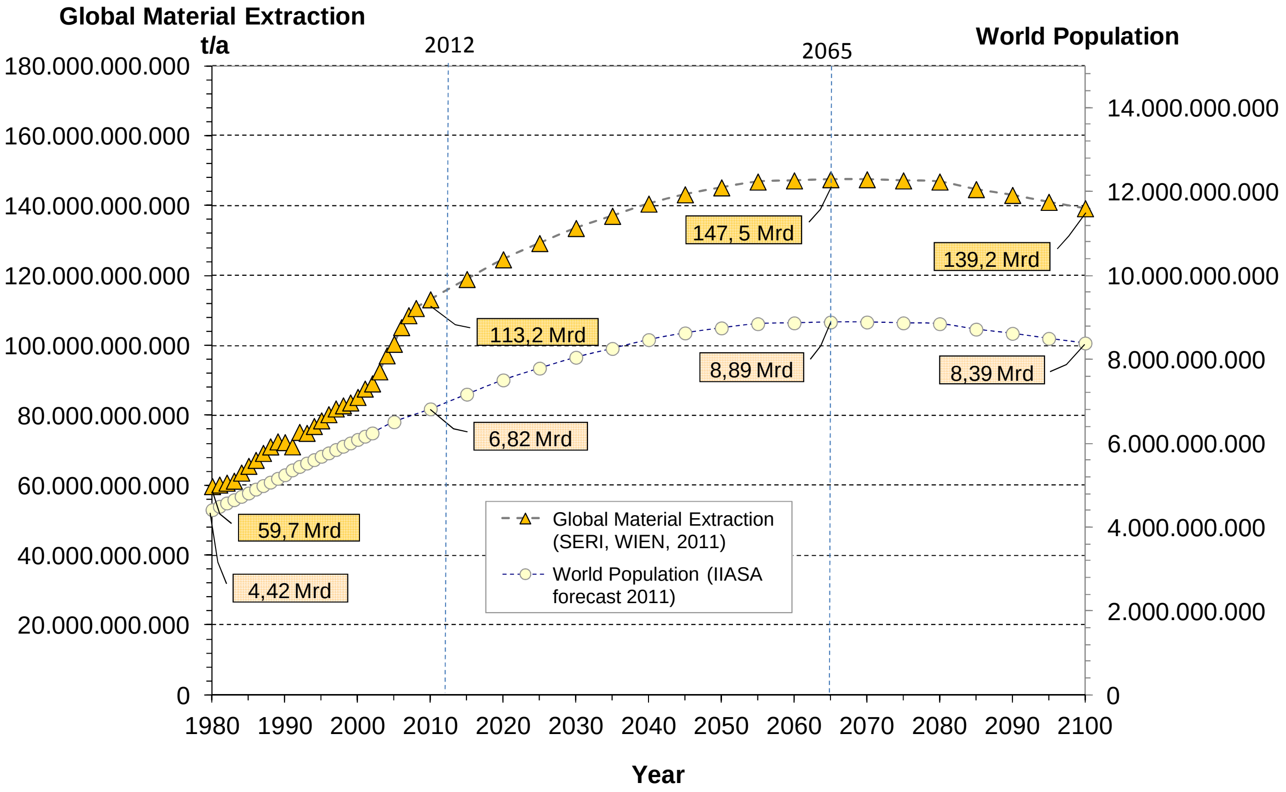
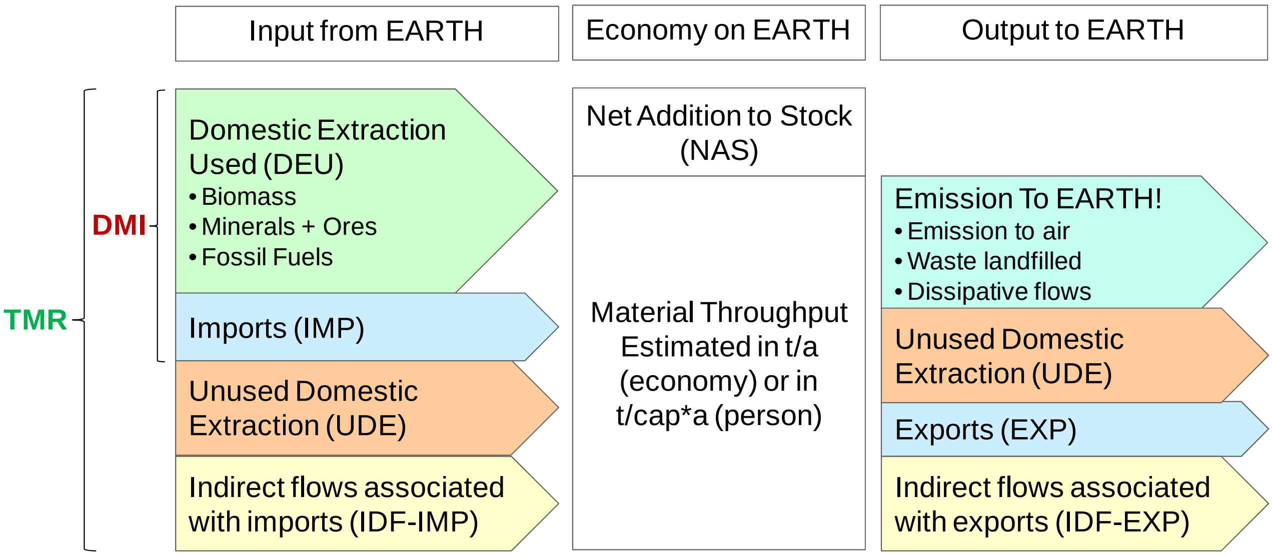


Fig. 2: **Past, recent and future Global Material Extraction. Figures based on data from of SERI [SERI, 2012} and IIASA [Lutz et al., 2008]**

To characterize the material balance for a certain country it is not sufficient to take into account only the *Domestic Material Extraction* of this country but, additionally, also material flows connected to imports and, thus, caused in other countries (elsewhere). **Figure 3** shows the principle diagram of such a material throughput balance [Bringezu, 2004; ETC/SCP, 2011). The part of material flow connected to imports and originated abroad is termed “ecological rucksacks”. The data for this are taken from national economy statistics and are already available or still under preparation for many European countries [ETC/SCP, 2011]. The human being with all his multiple motifs creates this material throughput in any moment of his existence. Part of these materials ends up e.g. in buildings, streets and rail road (NAS – Net Addition to Stock). The other part is used and partially transformed resulting in different categories of emissions (waste, exhaust air and dissipative losses). The sum of all this is termed *output*. It should be clear that the material flows originated by humans are always related to definable limits of the EARTH and not to limits of an undefined “nature”, “world” or “environment” [Grooterhorst, 2012].

A country’s material throughput supplied by the sources of the EARTH (excl. water; sometimes also excl. erosion) can excellently be summed up with the indicator **TMR - Total Material Requirement**. It is expressed either in tons per year (country) or in tons per individual and year (t/cap*a) considering the population of the respective country. The TMR accounts for domestic used material extraction, imports, unused domestic extraction as well as indirect material flows abroad connected to imports. With this indicator, difficult to determine [Eurostat, 2001 and 2008] but easy to understand for everyone (as expressed in units of weight), the total material throughput of a country and a citizen of this country can directly be compared with the material throughput of another country or another citizen of another country provided that the same accounting methodology is used. According to [ETC/SCP, 2011] the TMR of the EU-27, data base 2007, is approximately. 47 t/cap*year (the population of EU 27 is about 502 Mio.). The differences between the countries are considerable. The TMR of Germany is approx. 6.1 billion t/a or around 74 t/cap*a (reference year 2004), [Bringezu and Schütz, 2008]. More examples: For the Unites States the TMR was 86 t/cap*a in 2000 [WRI, 2012], Finland around 100 t/cap*a in 2010 [Finland Statistics, 2010], Switzerland around 47 t/cap*a in 2006 [Bundesamt für Statistik der Schweiz, 2008] and China 25 t/cap*a and 37.5 t/cap*a respectively [Xu M. and T. Zhang; Bringezu et al.; 2003] and Italy approx. 38 t/cap*a [Balzamo et al., 2004].

An overview on the progress of processing TMR values in the European Union are given in Bringezu and Schütz (2010). Additional TMR values are available in Bringezu et al. (2003) and Bringezu et al. (2004) and European Commission (2001).



Direct Material Input = DMI = DEU + IMP
Total Material Requirement = TMR = DMI + UDE + IDF-IMP

Fig. 3: Principle to characterize the material throughput of a human being or a economy with the indicator Total Material Requirement modified after Bringezu (2004) and ETP/SCP (2011}.

Meanwhile figures of the *Domestic Material Extraction* are generated for all countries of the world [SERI, 2012] which can be added up to the *Global Material Extraction* (EARTH). Assuming that the material throughput in a lot of developing countries is generated by themselves and is not burdened by “ecological rucksacks” from imports like in industrial countries, the figures for the *Domestic Material Extraction* of such countries (as far as not influenced by great amounts of raw material extraction) can give a first hint of their material throughput which can then be compared to the material throughput expressed as TMR of the industrial countries. Only some examples: SERI (2012) has estimated the *Domestic Material Extraction* (total of biomass, ores, mineral construction material, and fossil energy carriers) for India = 6.4 t/cap*a, Bangladesh = 1,8 t/cap*a, Kenya = 3,6 t/cap*a, in Pakistan = 3.4 t/cap*a; Ethiopia = 5.0 t/cap*a and Nigeria 4.3 t/cap*a.

Despite the methodological differences between TMR (industrial countries) and *Domestic Material Extraction* (listed developing countries) it becomes clear from magnitude that a citizen from an industrial country utilizes at least ten times more matter than citizens from developing countries where their matter use is even far below the average value of the *Global Material Extraction* (see above, 16.4 t/cap*a). In all these balances water is not considered. In the general international discussion about the use of the planet’s resources (maybe we better say: use of the planet) there will be the question for industrialized nations if these high amounts will be further available for them to the past and present degree. People in other countries of the world will demand access to the EARTH or only like to maintain the EARTH and therefore they like to have another kind of matter use regarding quantity and quality. Or in cases like China they gain access by acquisition of licences for raw material extraction all over the world. The future will show if this will develop in an ordered manner by international treaties or chaotically by wars.

Basically in the next decades there will be also in Europe a change in material throughput per capita, simply because there will be some billions more humans on earth. As the material consumption of the citizens in industrialized nations is evidently constantly above the mean of the EARTH since the 1970s - despite the debate about sustainability - these countries will have to reduce their material throughput [Bringezu, 2006, Linz, 2004; OPEN, 2011]. This can be viewed in the context of a global material management as *sustainability* at all [Daly and Farley, 2004]. It is not about to lower *relative* indicators like material use per monetary unit (t/Euro gross domestic production; resource efficiency) but the *absolute* material throughput per nation respectively per individual [Grooterhorst, 2011, Grooterhorst 2012].

It is more about the sinks and less the availability of resources e.g. metals, minerals, organic matter and energy carriers. Certainly the shortage of single resources will also cause a problem of the source called EARTH but the planet from a certain point is not able anymore to take more waste matter, and the therein contained agents as emissions or to cope with erosion, reduction in species, acidification of soil, marine pollution and climate changes by carbon dioxide emissions without doing harm to present and future mankind. Insofar the discussion about climate protection highlights the general problem of "earth management" as a balance between source and sink in terms of sustainability only fragmented as the material throughput caused by the combustion of fossil energy carriers is only a partial material throughput [Grooterhorst, 2011].

2.2 Myth and dogma of waste avoidance

Despite these facts regarding the material throughput it is claimed for 30 years now in *politics* (i.e. there where decisions are made) *waste avoidance* should be a method to fulfil the concept of sustainability. Following citation of 1978 by *Dr. Heinrich Freiherr von Lersner* [cited in Möcker, 1981], at that time president of the German "*Umweltbundesamt*" in Germany shows both the logic and confusion behind. In an era that still was influenced by the "oil crisis" from 1973 and "The limits of growth" [Meadows, 1972] he stated in an interview that he has "*to admit that in Germany we addressed the waste problem in certain way wrong. At first in 1972 we have enacted the Waste Management Law* [Note: German "Abfallbeseitigungsgesetz"], *and then we elaborated the waste management strategy with the focus on recycling i.e. recovery of waste. And now our attention turns from recovery to avoidance. The other way round would have been more logical*."

The crucial sentence is "*And now our attention turns from recovery to avoidance. The other way round would have been more logical.*" The necessity for shifting the attention towards *avoidance* of waste sounds "good" and matching, but still it is wrong. *Von Lersner* also made the false conclusion like the rest (including the author) who categorize the fields of activity of waste management: Apart from *recycling* and *disposal* of waste which can doubtlessly be allocated to waste management this line of argument implies that this is valid also for *avoidance*. *The false conclusion lies in the fact that waste cannot be avoided but only products.* Avoidance of products however is not a realm of waste management but has to be assigned to the politically hierarchical higher principle of sustainability and then coupled back into the product economy. Despite this situation politics and legislation have created an association with *waste* and *avoidance* and have fixed the priority of waste *avoidance before* waste recycling and waste disposal in the waste management laws almost in all countries of the European Union.

That the cart was put before the horse was also suspected by the former minister of environment *Klaus Töpfer* who commented in the 1980s about avoidance: "*Such a priority* [of avoidance] *would have extensive impact in areas that are basically reserved for energy and product politics and the economy* [cited in Looß and Katz, 1995]. At that time it could be formulated not better that it is basically about products that are to be avoided. Therefore it is surprising that the way towards sustainability – which requires on the material level a reduction of the material throughput by reducing products– should be broken by waste *avoidance* in the waste economy and in waste management.

This can only be explained by the fact that in nearly all areas of society *avoidance* of products is a taboo. The portion of recycled municipal waste could be increased significantly in the last 30 years, depending on the type of recyclables (metal, glass, paper, plastics, and organic matter). But the gross municipal waste quantity, which is the total of recycled and disposed waste, is constant for three decades with a quantity of 500 – 600 kg/cap*a in Germany (also in other industrialized countries) [Grooterhorst, 2009]. The reason for this is that citizens in industrialized countries were not successful in average to avoid *products* and, thus, to step into the reduction of material throughput as it would have been necessary on a global scale in order to meet the principles of sustainability.

Contributing to the discussion about waste avoidance that is repeatedly used as a proof for sustainable attitude and action I would like to develop the following thesis based on the previous illustration: *There is no waste avoidance but only product avoidance*. The waste sector does not have the power to avoid products. At certain stage of use all products become *emissions* and waste, respectively, but in the field of action and the system boundary of product economy and not of waste economy. Waste economy only exists because there is a product economy and disagreeable matter is disposed as waste. Deriving waste avoidance from the realm of waste economy would mean*: Waste is the cause of waste*. This would be a "magic" reversal of cause and effect [Jean Gebser (1905 - 1973), 1986); Grooterhorst; 2010a]. Similar phenomena can be observed in the financial sector where there have been attempts to fool people by explaining that money is the cause of money and not the basically underlying values of matter.

With combination of *waste* and *avoidance* a term has been coined and an alleged field of action was created that allows it to blank out the "sustainability gap" or blind spot of sustainability. By constantly repeating waste avoidance and their putative feasibility as a myth and as a dogma the imperative of sustainability, namely to avoid products and services as material throughput to maintain the EARTH for the future generations, can be suppressed and placed into the scope of waste management. Today waste management is *rational* and strongly segmented (Grooterhorst, 2010a) but it bears the heritage of the omitted actions in the *integral* [Gebser, 1986] realm of sustainability since about three decades [Grooterhorst, 2010b and Grooterhorst 2010c]. *The contradiction associated with waste avoidance that material throughput of an individual can be reduced by waste avoidance and not by product avoidance has to be dissolved.* It is about time to disengage the waste management as economical sector and as applied science from the narrow corset of the dualism; product economy on the one side and waste economy on the other side. Ideas have to be developed how waste economy - in terms of sustainable use of "defined matter" provided by the EARTH - can be integrated in a superior concept of matter management which is the basis for of the material throughput as measured in tons per individual and year. Then, *product avoidance* can have a clear effect on future municipal waste as the following considerations show.

The average material throughput of an individual on EARTH is approx. 16 t/cap*a. In an industrial country for example like Germany it is more than 70t/cap*a. Assuming that in this century also the German citizen can only have a material throughput like the planetary average then this would require a reduction of the material throughput by around 80 % in average [Bringezu, 2004 und Bringezu 2006]. For other industrial countries it will have the same magnitude. Municipal waste constitute with 500 to 600 kg/cap*a on the output side about 1 % of the total material flow in Germany. In **Fig 4** the assumed reduction by 80 to 70% starting from today up to a projection into the year 2100 corresponding to scenario 9 from *Meadows* (2006) is shown. Adapting the material throughput in Germany to the mean level of the earth by product avoidance a municipal waste quantity would be reached of about 1970. That this waste will qualitatively be "different" from the waste of the middle of the last century should be clear.

3. CONCLUSIONS

If in terms of sustainability principles *products* are avoided the less *waste* will be accumulated in future. This effect would also occur when wars or political crises reduce the matter input of a society. These events are not predictable on a serious basis (so called "wild cards" in prognosis). Assuming a "civilized" and therefore less erratic course of events then there will be raised more and more the question in the political debate why consumers in the industrialized nations use between 50 and 100 t/cap*a and citizens in the less developed countries less than 10 t/cap*a. As the EARTH cannot cope with a plus of material flows due to additional individual throughput and increasing world population (2 to 4 billion more in the next 5 decades) the quantity of material extraction placed at the disposal of mankind will be limited. The only question is how much is "enough" (see **fig. 5**). With respect to the figures provided by SERI (2012) regarding the *Global Material Extraction* (and thus the material throughput of one human of the earth) during the last decades and the related damages to EARTH which can be observed the limit value will be around or even less than 100 billion tons per year (with increasing standards of "environmental" protection!).

In this development the industrialized nations have to play the main role. The amount of material being at the disposal to everyone - one could also speak about a material quota or account – will be definitely below the present material in absolute terms. Depending on development of the world population and the level of the "limit value" the average matter quantity per human will be then less than the today around 16 t/cap *a referring to the EARTH. This will be not without an effect on the waste volume (see fig. 4). By product avoidance it will also turn into "real" waste avoidance. As there is no waste avoidance this error has to be removed from the European and national waste legislation. The avoidance of products in the sense of the principles of sustainability is a superior political realm in the international distribution of matter from EARTH. The laws of waste management are not suitable to reach these aims. An independent completely new realm of legislation is necessary called "**sustainability law**". The changes for the societies of industrialized nations will be dramatic because it is not only about to become more efficient in relative terms (e.g. material throughput in t/a*€ gross domestic product) or more "resilient" or "consistent" to protect a "not-defined" environment but also more "effective" in absolute terms reducing the material throughput at all (measured as TMR in t/a and t/cap*a, respectively) to maintain the earth as whole "forever".

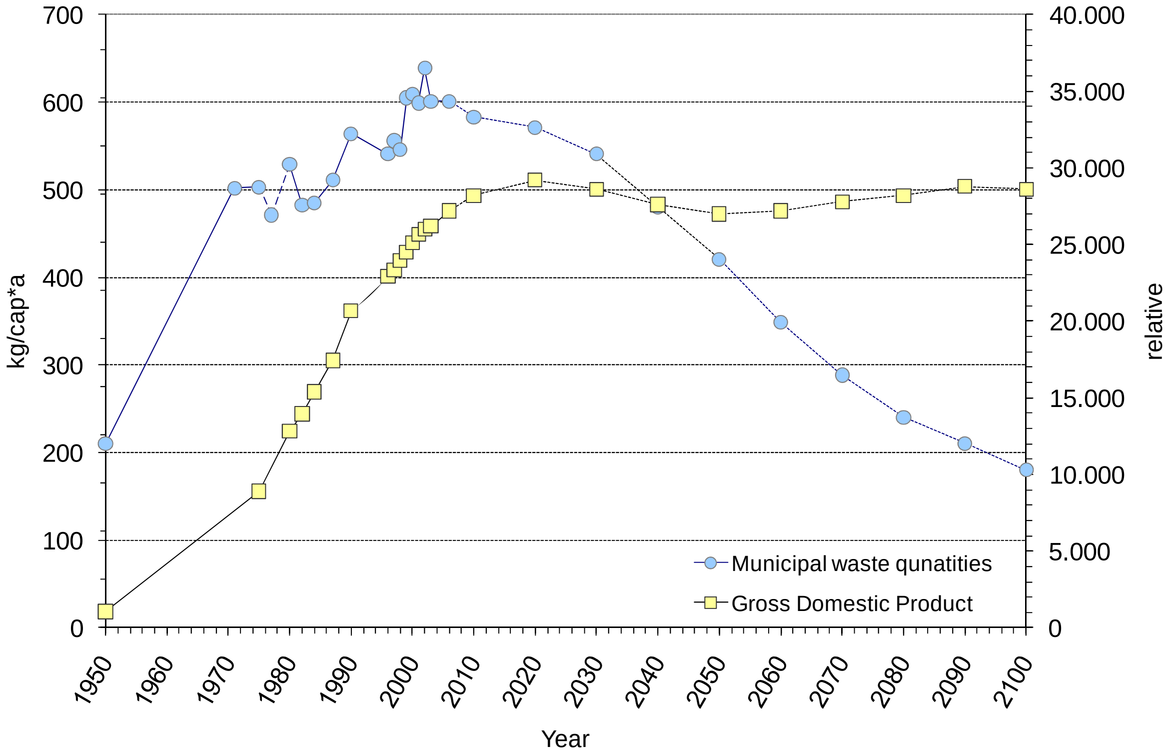


Fig. 4: **Forecast of Municipal Waste Quantities in kg per person and year for Germany until 2100 considering a overall reduction of material throughput**

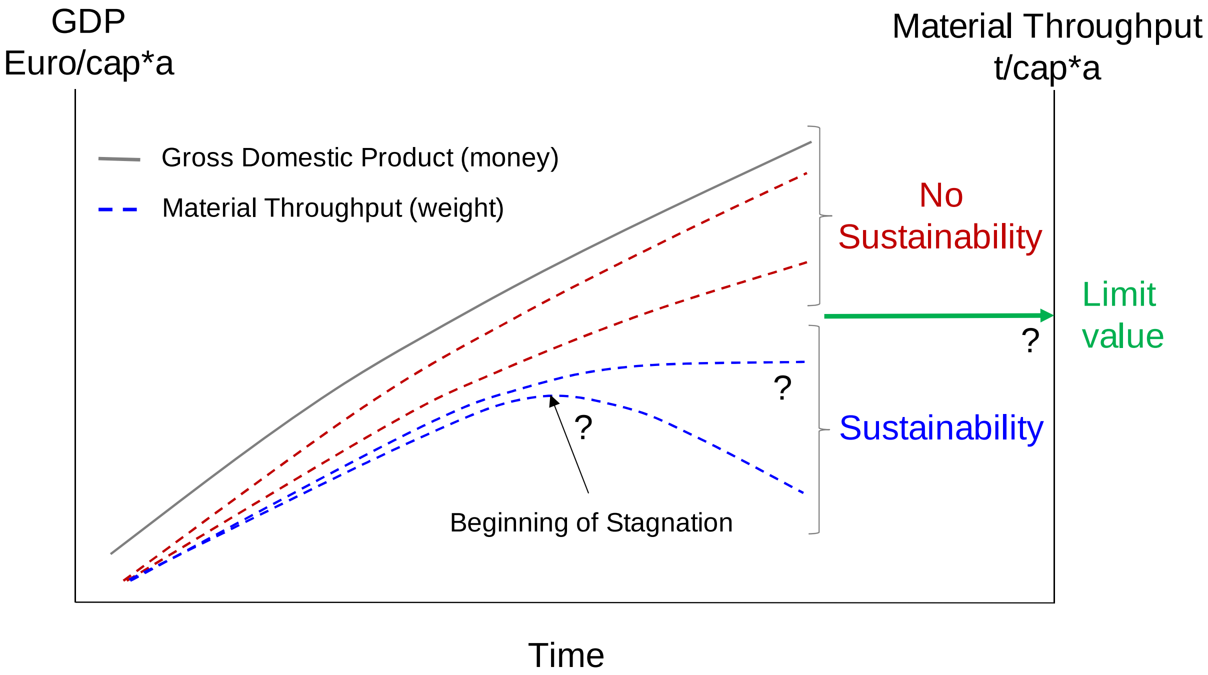


Fig. 5: **Only one sustainability derived from a clear limit value for the Global Material Extraction in t/a and the specific Total Material Requirement (TMR) in t/cap*a, respectively**

This means that every country has to operate in the interaction of all nations with less matter from EARTH. The basic principle of sustainability states nothing else than: *“We must treat others as we wish others to treat us”* (Golden rule!) [Bauschke, 2010]. May be intellectually different and rationally reduced but in its core the same *Immanuel Kant* (1724 - 1804) formulated his *Categorical Imperative*:

"*Act in such a way that you treat humanity, whether in your own person or in the person of any other, never merely as a means to an end, but always at the same time as an end.*" [cited in Pfister, 2007]. The concept of sustainability related to the whole earth in modern times fit to the ancient insight of humans: The basic principle of all animate and inanimate matter is permanent change. In Plato's *Phaedo* Socrates says to Cebes: "*Now the compound or composite may be supposed to be naturally capable, as of being compounded, so also of being dissolved*; ..." [Plato, 428 - 348; cited in (B. Jowett, 1892)]. The "undefined" matter passes product economy (extraction of raw materials, production, trade and consumption) and waste economy and is converted to "undefined" matter again (see fig. 1).

The key aspect is: *It is always the same matter*! In sustainability it is about this eternal change of matter and its interaction with life in general and humans in particular. As matter is constantly changed and transformed from raw material, primary product, product, trading object, consumer good and back to waste these materials could be considered as "***adduct***" (see concept of MIPS = Material Input per Unit Service) [Schmidt-Bleek, 1994]. In future we have to consider our material flow as *adduct economy* (see **fig. 6**). The waste economy would be assimilated by this. *Friege* and *Bilitewski*, two German waste researchers, give a future prospective (cited in BMU, 2004): *Waste economy must be considered of a comprehensive and holistic system of material and product flows.* Product and waste are perceived and managed separately according to the "mantra" of the specialisation of economy. But they can be described from a superior "whole" as a process of matter management by humans. Matter is extracted from EARTH and transferred as "input" into production, utilized and deposited as "output" back to EARTH. The concept of sustainability refers to this process as a whole. Therefore the separate consideration of product and waste is contradictory to the superior principle of sustainability and causes non- sustainability. The term *adduct* could make clear that product and waste economy form a new realm of consideration and management where these two are mutually conditioned and intermingled.

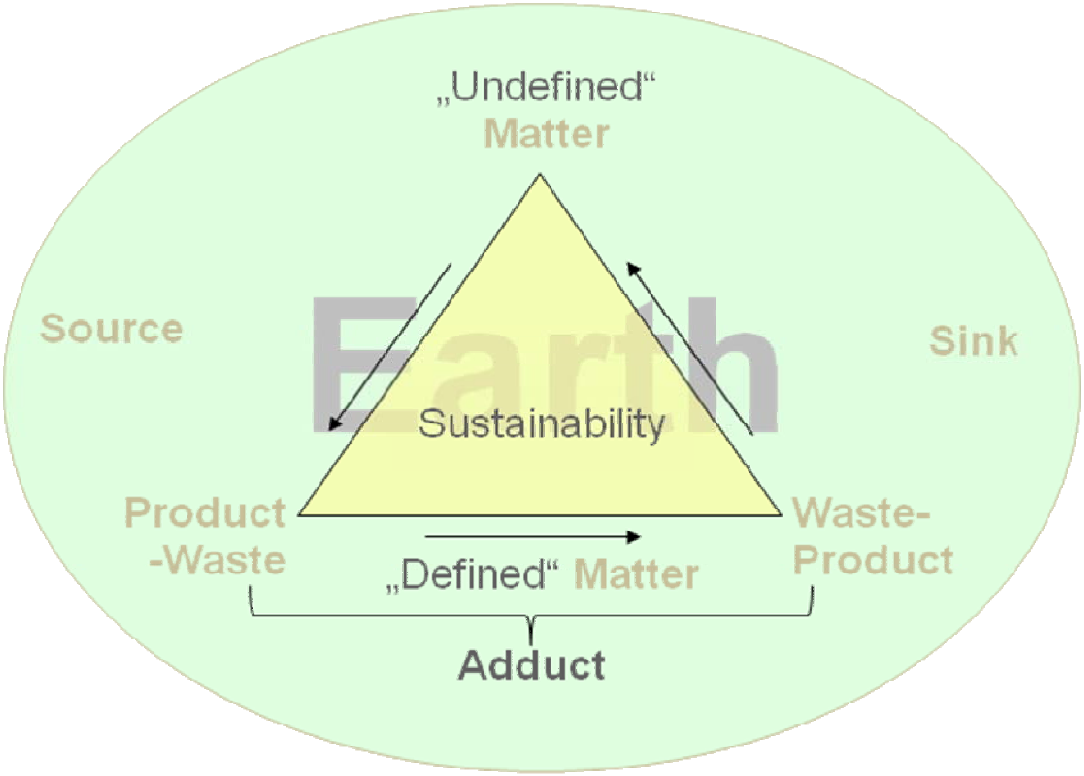


Fig. 6: **Forecast of Municipal Waste Quantities in kg per person and year for Germany until 2100 considering a overall reduction of material throughput**

Product and waste are complementary. Unlike products *adducts* are created in the context of the EARTH's capability to provide matter for human purposes (limit value of material throughput). *Adducts* have to be seen in relationship to the total amount of worldwide quantity of adducts (contingent of adducts).

Adducts are not substances, but items and services defined by human motifs that are referenced to the total consumption of a human which has to be extracted from EARTH. And that consumption is limited as the EARTH is only able to provide a limited *adduct* quantity for each human.

Regardless of whether we speak of a "sustainable economy of material flow", "adduct economy" or another term, waste management will not exist anymore in such an economy in the traditional sense. The main aspect of such an economy beside generation of *adducts* from "undefined matter" will be the generation of adducts **from the available "stock" into the "stock"**. That is not manageable from traditional recycling but only from a network of adducts where already all aspects of the idea of the *adduct* are realized in the material existence of the *adduct*. Ideas of an *adduct* that endanger the earth as a whole with respect to quantity and hazardousness will not be generated anymore. Regarding the material throughput of a human to be reached less than approx. 16 t/cap*a there would be a little range for unused potentials. This is also valid for the today waste. The supply of *adduct parts* or *primary adducts* in the *adduct* economy would remind at best to the "good old" waste management.

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Session 6

(50) BENEFIT OF A GOOD AERATION MANAGEMENT IN COMPOSTING

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EXECUTIVE SUMMARY

The sustainability and the development of composting activity depend on the capacity to produce, by efficient processes, compost with high agronomic and environmental quality and minimum health and environmental impacts. As composting is an aerobic process, one of the main ways to improve its management consists in using an appropriate aeration strategy. Indeed, this parameter influences not only the oxygen availability, but also other essential parameters such as moisture content and temperature. Therefore, the aims of this work were (i) to identify an optimised aeration strategy and then (ii) prove and quantify, by experimental results, the benefit of a good-aeration according to process indicators (temperature, gaseous emissions and matter biodegradation).

To define the optimised aeration strategy, an experimental design methodology was used at composting pilot scale. It allowed identifying the aeration effects on composting key parameters (temperature and oxygen concentration). Thus, it was possible to choose the parameters values that led to good temperature and oxygen concentration profiles for 27d of composting. To evaluate the benefit of this aeration, the process indicators, obtained with an unfavourable aeration (BIO-), were compared to those obtained with the favourable one (BIO+) for a composting test of 83d. The composting experiments conducted, to define the aeration strategy and BIO+/BIO- comparison, were performed on a mixture of biowaste and bulking agents, in the 160L composting reactors set up at Veolia Environnement Research Centre (Limay, 78). In this system, the controlled aeration was conducted with a proper and dry air blowing system with continuous flow monitoring during the tests. The compost temperature and oxygen emissions (O_2) were continuously monitored. The other gaseous effluents (carbon dioxide (CO_2), ammonia (NH_3), methane (CH_4), non-methane hydrocarbons (NMHC)) were collected and quantified at least every 180 minutes. Initial mixtures and composts at the end of the experiments were sampled for biochemical and chemical analysis (dry matter, organic matter, total organic carbon, nitrogen content) and for the estimation of organic matter stabilization (Carbon mineralization in soil-compost mixtures). For BIO+ and BIO-, additional samples were collected after 27d and 42d. As expected, the comparison of temperature and oxygen profiles for BIO+ and BIO- confirmed the necessity of a good aeration to reach and maintain high temperatures and to ensure aerobic conditions. Even if the total amount of matter losses is similar in both cases, the benefit of the aeration is proved by a faster organic matter biodegradation and stabilisation for BIO+ than for BIO-. O_2 consumption and CO_2 production confirmed this point. The study of NH_3 emissions reveals higher production for BIO+ than BIO- which results from the combination of several mechanisms: the degradation of nitrogenous organic matter and the effect of stripping related to temperature and aeration. This study also shows that emissions of CH_4 and NMHC are higher for unfavourable aeration mainly due to anaerobic periods.

To conclude, the aeration strategy is confirmed as an essential key to ensure well-composting process. The comparison of unfavourable and favourable strategy allowed quantifying the gain of this improvement which is essential to manage the process and design composting sites.

1. INTRODUCTION

The sustainability and the development of composting activity depend on the capacity to produce, by efficient processes, compost with high agronomic and environmental quality and minimum health and environmental impacts. As composting is an aerobic process, one of the main ways to improve its management consists in using an appropriate aeration strategy. Indeed, this parameter influences not only the oxygen availability but also other essential parameters such as moisture content and temperature. Therefore, the aim of this work was to prove and quantify by experimental results, the benefit of a good-aeration according to process indicators (temperature, gaseous emissions and matter biodegradation). To do this, the composting process indicators obtained with an unfavourable aeration (BIO-) were compared to those obtained with a favourable one (BIO+). The favourable aeration strategy results from an experimental design protocol that involved identifying the aeration effects on composting.

2. MATERIALS AND METHODS

2.1. Materials

The composting experiments were performed in the 160L, closed and insulated composting reactors set up at Veolia Environnement Research Centre (Bacheley (2008)). A controlled aeration, with a proper and dry air blowing system, was applied to the bottom of the reactor with continuous flow monitoring. The temperature was also continuously monitored in the core of the compost. The gaseous effluent was collected at the top of each reactor. Oxygen concentrations (O_2) were continuously monitored whereas the other gaseous effluents (carbon dioxide (CO_2), ammonia (NH_3), methane (CH_4), non-methane hydrocarbons (NMHC)) were collected and quantified at least every 180 minutes.

2.2. Methods

2.2.1. Definition of an optimised aeration strategy

The effects of the aeration on the composting of a biowaste mixture were studied in composting pilot using an experimental design methodology. It consists in doing an organized set of experiments, in order to acquire new knowledge by controlling one or more input parameters (environmental factors, experimental conditions, etc..) and to obtain an accurate statistical model of the results behaviour (Goupy (2006)). This methodology appeared more interesting than a "traditional" one (variations of the input factors, one after the other without any real advance planning) as it might allow to (i) enhance the technical knowledge, (ii) structure the experimental approach, (iii) maximize the ratio of information gathered by the number of tests performed and (iv) identify among all the factors considered, those whose influence is significant on composting and how they act. However, the use of experimental design also has several limitations that may limit its application. Indeed, the results must be measurable and the factors must be controlled. It also required good system stability (eg no uncontrollable influential factor). In the case of the work to optimize aeration strategy, the limits of experimental designs were considered acceptable due to the well control and measurement of composting parameters during pilot experiments.

To answer our objectives, trials were carried out following a full factorial design. It enabled the study two-level per factor. This simple configuration set-up, for m factors, the trials at the corners of a hypercube $[-1;1]^m$. The model calculated will thus be of second order (effects and interactions). It will eventually allow to classify their effects and to quantify them. For these experimental designs, the characteristic and controllable parameters (input factors) of the aeration were: the instantaneous air flow value (Q), the time during which the aeration rate is applied (T_{ON}), the time during which the aeration rate is null (T_{OFF}). The responses considered representative of the process were: temperature profile (T°C), profile of oxygen concentration (O_2), gaseous emissions and quality of compost. Nevertheless, it is not possible to analyze simultaneously the influence of these variables on all responses, thus their effects are studied separately. The impact of aeration was first studied on the parameters T°C and O_2 for three schematic stages in the composting process (increase, maintain and decrease of the temperature). An optimised aeration strategy was sequentially searched for each one of them (FIGURE 1). From T°C aspect, the intended profile is a quick increase of the temperature, followed by maintain at high temperature for at least 4 days and finally a slow cooling of the matter (4 weeks of composting fermentation). From O_2 aspect, the objective is to maintain aerobic conditions (O_2> 5-10%) (Mustin (1987), Haug (1993)).

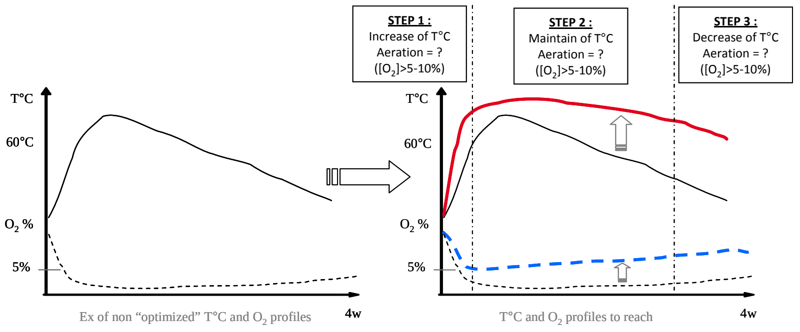

FIGURE 1 Descriptive of the intended profiles and the 3 schematics stages of composting

According to this methodology, more than 100 tests were performed in the composting pilot (described in part 2.1) to identify the optimised aeration strategy.

The aeration factors were modified and adapted to progressively identify the optimised combination for T°C and O_2 profiles. These tests were carried out on a specific and "constant" mixture of biowaste and green waste. These wastes were collected separately on 2 industrial plants, about 1 or 2 days before the tests. The mixture was handmade the first day of the experiment. The matter, at the beginning and the end of the tests, was characterised according to biochemical and chemical analysis (part 2.3). Once the optimised aeration was identified for this mixture, complementary composting experiments were performed to extend the validity area of this aeration. They consisted in composting experiments on other biowaste mixture at both pilot and industrial scales.

In this paper, all these experiments and their operating conditions are not presented, only main results are mentioned. Due to confidential and competitive aspects, the complete aeration strategy is not presented, but the methodology to reach it is detailed.

2.2.2. Study of the benefit of a favourable aeration: BIO+ and BIO- tests

To evaluate the benefit of a good aeration, the process indicators (temperature, gaseous emissions and matter biodegradation) obtained with an unfavourable aeration (BIO-) were compared to those obtained with a favourable one (BIO+) during pilot composting tests. For each aeration modality (BIO+ and BIO-), 3 replicates composting trials were performed. The BIO+ aeration modality results from the experimental design protocol (described before in part 2.2.1). The BIO- aeration modality represents limiting oxygen conditions. The average ratio of air supplied between non-limiting aeration / limiting aeration is about 2,5:1. This difference is highest during the first 27d of fermentation (5:1), whereas during curing phase the ratio is 1,3: 1. These tests were performed on 83d on an industrial mixture of biowaste and screening refusal. It was collected on the industrial plant about one day before the experiments and transported in big bag to VERI-Limay. Upon arrival, the matrix was manually mixed and then was placed in composting reactors after sampling for analysis. A turning was realised after 27d and 42d of composting. After 27d, 42d and 83d the mixture was weighed and sampled to biochemical and chemical analysis (part 2.3).

2.3. Analysis

For collected samples, the dry matter (DM) was determined after drying at 105°C during 48h (NF EN 13040). The organic matter (OM) was measured by loss on ignition during 6 hours at 480°C (NF EN 13039). Total nitrogen (TN) was determined by elementary analysis (NF EN 13654-2). The total organic carbon (TOC) was measured by oxidation of the organic carbon into CO_2 in presence of O_2 and elementary analysis (NF ISO 10694). The biochemical composition of composed organic matter was determined using the Van Soest fractionation method (XPU 44-162, AFNOR 2005): separation of soluble organic matters (SOLwater and SOL NDF), hemicellulose (HEM), cellulose (CEL) and lignin fraction (LIGN). The stabilisation of the matter was also studied thanks to mineralised carbon test on 28d (MinC28d) (XPU 44-163). Except for MinC28d and DM content also determined by VERI laboratories, all these analysis were realised by the SADEF, COFRAC laboratory specialised in environmental and agronomic analysis.

3. RESULTS

3.1. Definition of an optimised aeration strategy

In this section the results obtained with the specific methodology described in part 2.2.1 are presented

3.1.1. Increase of temperature

The O_2 and T°C profiles obtained for composting tests, under various aeration conditions (chosen according to an experimental design protocol), are presented in FIGURE 2a. The comparison of these results and their statistic treatment allow to define the aeration factor effects on composting responses and thus to choose the combination of Q, T_{ON} and T_{OFF} which induce a rapid increase of T°C with no O_2 limitation. The use of this aeration strategy on 48 tests confirmed its efficiency to provide a rapid increase of temperature without O_2 limitation (FIGURE 2b) with good reproducibility and repeatability (taking into account waste variability and experimental uncertainties).

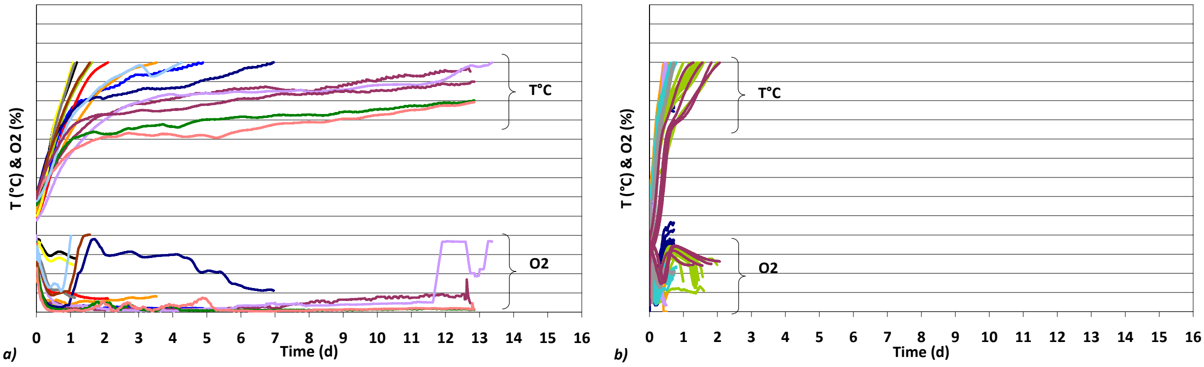

FIGURE 2 T°C and O_2 profiles obtained before (a) and after (b) the aeration optimisation for the rise of T°C

3.1.2. Maintain of the temperature

Once the aeration for the rise of the T°C was identified, it was looked for the one to maintain high temperature values with no O_2 limitation. Therefore, a new aeration experimental design was used to determine the aeration factors effects on composting responses during this period of the process. The O_2 and T°C profiles of the composting tests carried out are presented in FIGURE 3a. The aeration strategy chosen according to these results was then applied on 19 tests that confirmed its efficiency (satisfying T°C and O_2 profiles as illustrated FIGURE 3b). The reproducibility and repeatability is quite good according to experimental variability.

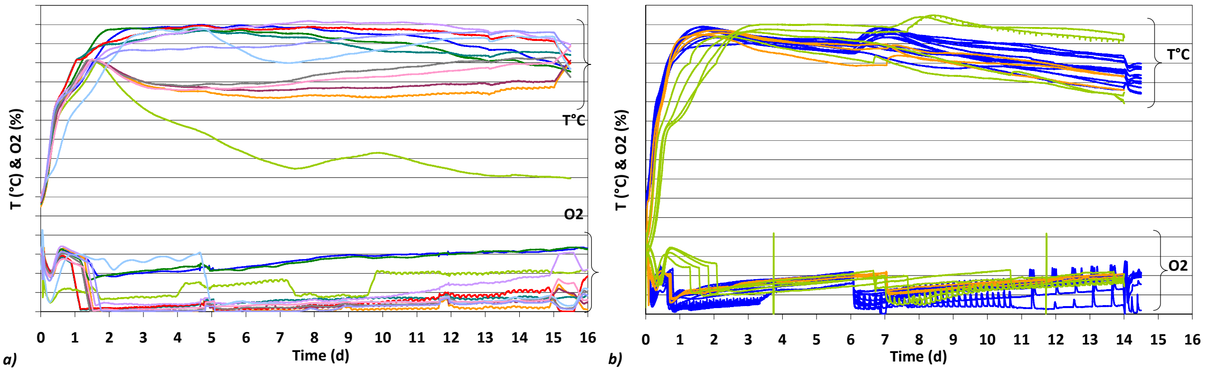

FIGURE 3 T°C and O_2 profiles obtained 2 before (a) and after (b) the aeration optimisation for the T°C maintain

3.1.3. Decrease of temperature

Even if the decrease of the matter temperature is inevitable due to the slowing down of the biological activity, the objective is to adjust the aeration to limit the cooling caused by the ventilation (exportation of the heat and insufflations of cooler air). Contrary to previous steps, the experimental design methodology was not used because the aeration factors have a small range of variation and few effects on composting responses. In such a case the traditional method is more relevant. The comparison of T°C and O_2 profiles related to tests performed with various aeration for this period of the process allowed to choose adapted Q, T_{ON} and T_{OFF}.

3.1.4. Global results

The methodology used (including experimental design protocol) was efficient to determine an aeration strategy, which ensure a rapid increase of temperature, its maintain at high value several days and then a slow cooling of the matter without O_2 limitation. The T°C and O_2 profiles obtained, with such aeration, are presented in FIGURE 4.

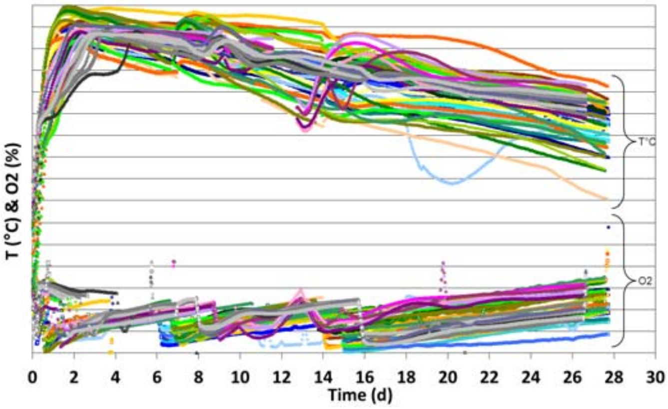

FIGURE 4 T°C and O_2 profiles obtained with the global aeration strategy

3.2. Study of the benefit of a favourable aeration: BIO+ and BIO- tests

In the previous part an optimised aeration was defined thus it was interesting to quantify its benefits compared to a non favourable one. Therefore, as presented in part 2.2.2, composting tests BIO+ and BIO- (favourable / unfavourable aeration) were compared.

3.2.1. T°C and O_2 profiles

The T°C and O_2 concentration profiles for BIO+ and BIO- are presented in FIGURE 5. For each aeration conditions, the 3 tests results are similar, that indicates a good experimental repeatability. For BIO +, the temperature rise rapidly: on average 3d are necessary to reach a maximum around 74°C. Then, the temperature is maintained above 60°C for about 26d. During this period, the residual O_2 content remains between 8% and 15%, demonstrating a non limiting aeration for the biological activity. The mixing after 27d induces a new increase of temperature (maximum during this period close to 65°C) while the O_2 contents remains mainly upper than 5%-10% (only 1d lower than 5%). For BIO-, the temperature increase is slower : 5d are necessary to reach 45°C and at least 30d are required to reach maximum temperature (maximum lower than 55°C). During this period, the residual O_2 content is close to 0% confirming a limiting aeration. After the second mixing at 42d, the temperature increases to a maximum around 50°C. During this period of the residual O_2 concentration starts to increase slowly. It observes a slowdown of the aerobic biological activity coupled with reduction in the oxygen consumption. The differences between BIO+ and BIO- T°C and O_2 profiles confirm (i) the use of a non-limiting aeration and a limiting one and (ii) differences on organic matter biodegradation.

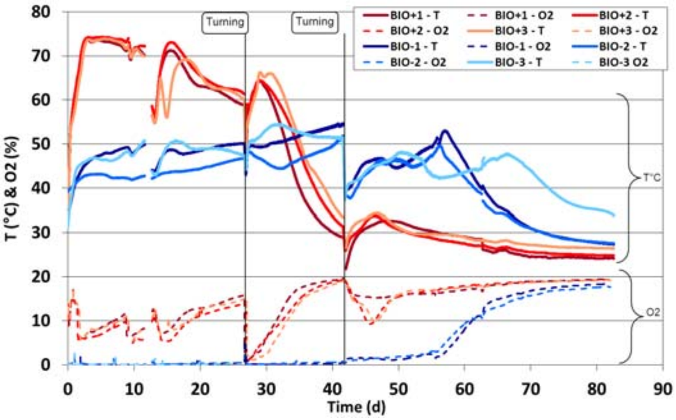

FIGURE 5 T°C and O_2 profiles for BIO+ and BIO-

3.2.2. Study of the biodegradation activity: comparison between BIO+ and BIO-

Both O_2 consumption and CO_2 production traduce the biodegradation activity. According to the good correlation between these parameters (r^2=0,99), only one of these is presented in this document, same conclusions are done for the other.

The CO_2 production, as illustrated in FIGURE 6, highlights differences in kinetic and level of production between aeration modality. For BIO+, CO_2 emission rate is maximal during the first 30d of composting (average of 17,5 $kgCO_2.tDM_0^{-1}.d^{-1}$). The mixing after 27d allows homogenising the matter and accelerating the biodegradation. The 5th week, there is a slowdown in CO_2 production (1,1 $kgCO_2.tDM_0^{-1}.d^{-1}$); it is the beginning of the curing phase. For BIO- (limiting aeration modality), the CO_2 production rate is constant during the first 60d (8,1 $kgCO_2.tDM_0^{-1}.d^{-1}$) then the degradation rate decreases rapidly (1.6 $kgCO_2.tDM_0^{-1}.d^{-1}$). The differences of kinetic are confirmed by the evolution of the CO_2 ratio between BIO+ and BIO- which is about 2,4 at 27d, 1,7 at 42d and 1,1 at the end. Therefore, it seems that for BIO+ the biodegradation occurs mainly during the first 30d and then slows down (at this point there is maybe more stabilisation than biodegradation of the matter) whereas for BIO- the biodegradation occurs during at least 60d.

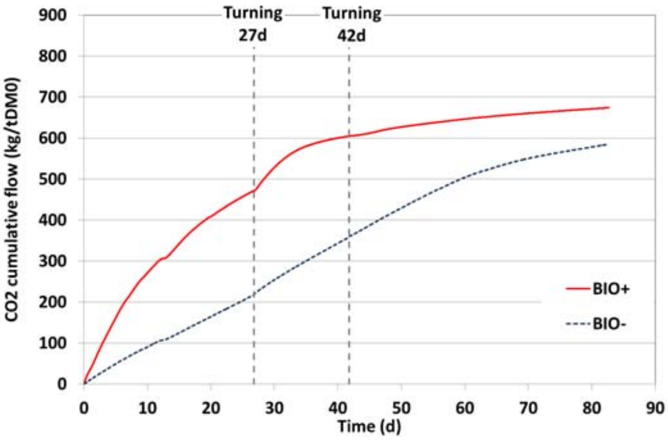

FIGURE 6 CO_2 cumulative flow for BIO+ and BIO-

As CO_2 production, the cumulative losses in organic matter are similar at the end of the tests for both aeration modalities (44% of the initial content for BIO+ and 41% for BIO-). But, once again, the degradation rates are different, since after 27d, the OM losses are 2,5 times higher for BIO+. These conclusions are also confirmed by the evolution of biochemical fractions (FIGURE 7a) and the carbon mineralisation on 28d (FIGURE 7b). Thus, the aeration strategy has more impact on the kinetic, than on the amount of OM losses. At the end of the process, the matter seems more stable for BIO+ than for BIO- according biochemical fractionation and mineralised carbon on 28d.

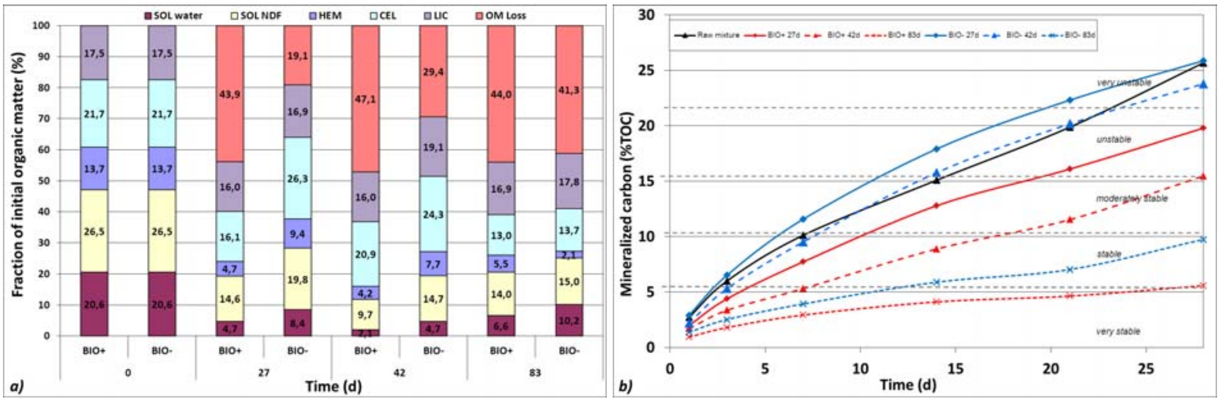

FIGURE 7 Biochemical fractionation (a) and mineralised carbon (b) results for BIO+ and BIO-

3.2.3. Study of CH_4 and NMHC emissions: comparison between BIO+ and BIO-

For CH_4, during the first 4 weeks, the cumulative flow (FIGURE 8a) is similar for both aeration modalities. The flow are very low and in the same order of magnitude as those found in literature: 184 to 525 gC/tDM_0 found by Bacheley

(2009) and about 1,67 kC/kgOM$_0$ presented by de Guardia (2010). 24 hours after the mixing at 27d, cumulative flux of CH_4 is higher for BIO+ than BIO-, but then there is no significant increase in the cumulative flux emitted for BIO+. On the contrary for BIO-, the cumulative flow of CH_4 still increases until the temperature reaches its maximum and then the CH_4 production slows down. After 12 weeks, cumulative CH_4 production is about 5 times higher for unfavourable aeration experiments (BIO-) than for favourable aeration experiments (BIO+).

For NMHC, the cumulative flow (FIGURE 8b) is superior for BIO+ than BIO- during the first 3d mainly due to a higher stripping of the volatile organic compounds contained in the initial substrate (higher air flow rate). Then, the emission seems more linked to the presence of local anaerobic areas and the kinetic of emissions are similar for both aeration conditions. After 42d, the NMHC cumulative flow is almost the same for BIO+ and BIO- but the last days seems to have a strong impact on this parameter. Indeed, at the end the NMHC cumulative flow is about 2,5 times higher for BIO- than BIO+.

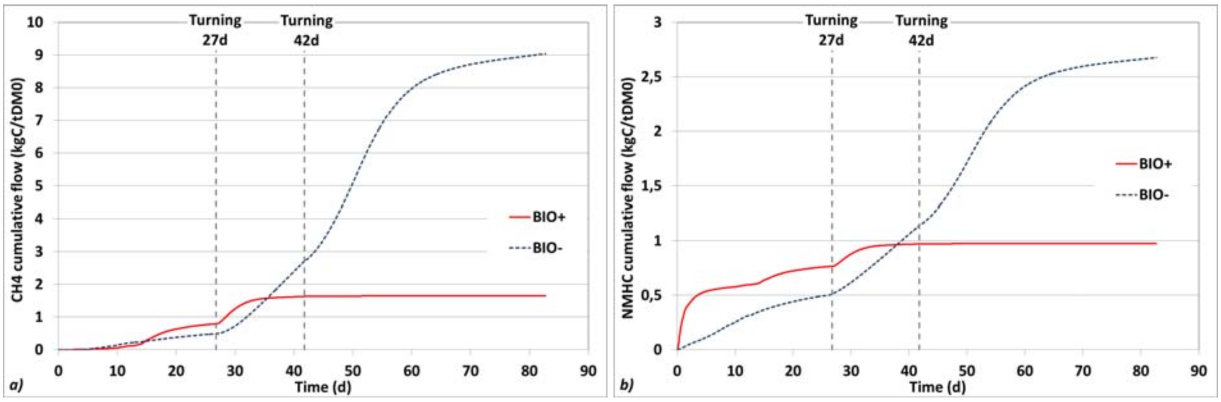

FIGURE 8 **Cumulative flow per mass of initial dry matter for CH_4 (a) and NMHC (b) obtained for BIO+ and BIO-**

3.2.4. Study of NH_3 emissions: comparison between BIO+ and BIO-

During the first 6 weeks of composting, the NH_3 cumulative flow is superior for BIO+ than BIO- resulting of higher temperature, aeration and biodegradation or biological activity (FIGURE 9). The emissions in literature are similar to those observed for BIO+, but superior to ones measured for BIO- (Bacheley (2008), Bacheley (2009), de Guardia (2010a-b), Pagans (2006)). After this period, NH_3 emissions are stable for BIO+ and slightly increasing for BIO-. The stabilisation observed for BIO+ is related to a lower biodegradation and matter temperature (close to 30°C), whereas the rise for BIO- is consistent with the increase of biodegradation and matter temperature (close to 45°C and then 30°C). At the end of the composting tests, the total amount of produced NH_3 is about 4 times higher for BIO+ than for BIO-.

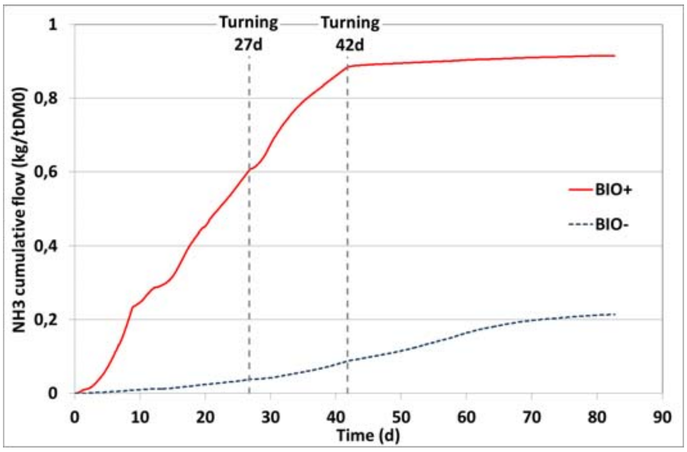

FIGURE 9 **Cumulative flow per mass of initial dry matter for NH_3 obtained for BIO+ and BIO-**

4. DISCUSSION / CONCLUSION

The first part of this work (based on the comparison of composting experiments with various aeration according a experimental design methodology), allowed to define an effective aeration strategy to provide satisfying composting responses. The aeration strategy identified was then used on BIO+ tests to compare the composting responses in such condition with ones with unfavourable aeration conditions (BIO- tests). The study of T°C and O_2 profiles confirmed the efficiency of BIO+ aeration strategy to obtain high temperature without aeration limitation compared to BIO- strategy. Despites the differences in T°C and O_2 profiles, the total amount of organic matter losses is similar for BIO+ and BIO-. Nevertheless, the biodegradation rates are different: they are quicker for BIO+ than BIO-. This point is confirmed by the results of CO_2 production, biochemical fractionation and mineralised carbon on 28d. According to the faster biodegradation observed for BIO+, it would be interesting to sample the matter more frequently to define at what point it no longer changes and maybe to get shorter the process. The study of NH_3 emissions reveals higher production for BIO+ than BIO- which results from the combination of several mechanisms: the degradation of nitrogenous organic matter and the effect of stripping related to temperature and aeration. This study also shows that emissions of CH_4 and NMHC are higher for unfavourable aeration mainly due to anaerobic periods. This work also shows the interest to study the process on long term because high difference can be induced by curing phase. Indeed, the BIO- substrates present their highest emissions in NH_3, CH_4 and NMHC after 54d. Thus, it seems that an optimised aeration process focuses its emissions during the first time of composting, which could be interesting to regroup the treatment system at this stage of the process, whereas a process under unfavourable aeration focuses its emissions during the end of the process.

5. ACKNOWLEDGEMENTS

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(60) NITROGEN DYNAMIC DURING AEROBIC TREATMENT OF ORGANIC WASTES

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EXECUTIVE SUMMARY

Present work investigated the nitrogen dynamic and the evolution of ammonia oxidizers during aerobic treatment of three kinds of organic wastes including municipal solid wastes, mixture of municipal solid wastes and waste activated sludge, digested sludge. Experimental results suggest mineralization of organic nitrogen was correlated to biodegradability and ratio carbon / nitrogen of each type of waste. The mineralization of organic nitrogen following the intense biodegradation stage of organic wastes contributed to ammonia emissions. Although ammonia oxidation occurred in the early phase during treatment of the mixture and digested sludge, the oxidation flow was much lower than ammonia emissions. For each waste, an increase of ammonia oxidation occurred similarly at the end of treatment. Ammonia-oxidizing bacteria were more abundant than ammonia-oxidizing archaea. Only the evolution of ammonia-oxidizing bacteria was correlated to ammonia oxidation flow. These findings suggest only ammonia-oxidizing bacteria were responsible for ammonia oxidation during aerobic treatment of three kinds of organic wastes.

1. INTRODUCTION

Composting is a conventional and appreciated waste management practice (Gajalakshmi and Abbasi, 2008). In aerobic conditions, organic wastes are biodegraded, stabilized and gradually transformed to composts. The composts can be further applied in the agricultural soil as soil conditioner and fertilizers. This application makes nitrogen (N) content an important indicator of the quality of composts. The most part of N contained in the wastes are lost in form of ammonia (NH_3) emissions during treatment (Beck-Friis et al., 2001). These emissions not only reduce the agronomic value of composts, but also cause series of environmental problems since NH_3 is malodorous, toxic, contributes to eutrophication and acid rains.

Many previous works evaluated NH_3 emissions during composting as function of treatment parameters (Beck-Friis et al., 2001, de Guardia et al., 2008, Fukumoto et al., 2003) and characteristics of wastes (Mahimairaja et al., 1994, Martins and Dewes, 1992, Sommer, 2001). However, the process involved in N transfers and transformations during composting are not thoroughly studied, especially N dynamic and nitrification. Nitrification is one of the key processes in nitrogen cycle of our planet. Ammonia-oxidizing bacteria and archaea (AOB, AOA) oxidize NH_3 to nitrite (NO_2^-). The NO_2^- are successively oxidized to NO_3^- by nitrite-oxidizing bacteria (NOB). NH_3 oxidation is the rate-limiting step of nitrification (Spieck, 2005). Concerning composting, the onset and the intensity of NH_3 oxidation is still obscure. The participation of AOB or AOA on NH_3 oxidation requires further investigation.

Present work aimed to study the N dynamic during aerobic treatment of organic wastes. Three kinds of waste were studied including the fine organic fraction of municipal solid wastes (MSW), the mixture (MMW) of MSW with waste activated sludge (WAS) and digestated sludge (DS). N transfers and transformations were monitored. Mineralization of organic nitrogen (Norg), stripping of NH_3 (NH_3 emissions) and NH_3 oxidation were followed for a period of 50 days. Evolutions of groups of microorganisms including AOB, AOA, total bacteria (TB), total archaea (TA) and total fungi (TF) were investigated.

2. MATERIALS AND METHODS

2.1 Treatment reactors and substrates

Experimental device consisted of 10 air-tight cylindrical cells. Each cell was immersed in a bath which was heated by a thermostat. The interior volume of the cell was approximately 10 liters. Wastes mixed with bulking agents were loaded on a grid located 10 cm above the bottom of the cell.

Below the grid, a glass screen supplied by compressed air homogenized the aeration flow. A Pt100 temperature probe was inserted into the middle of the mixture to measure the real time temperature.

MSW were collected at a composting site in France (15.000 tons year-1). In this site, residual household wastes including kitchen wastes, papers, cardboards, textiles and plastic films are firstly mixed and pre-degraded in two rotary tubes. Then, pre-degraded mixture passes through mechanical treatments to remove impurities like metals and glasses. The last step of mechanical treatment consists in sieving at 10 mm. Finally, fraction inferior to 10 mm is mixed with green wastes for composting in windrow. The samples MSW were collected at the outlet of sieving before mixing with green wastes. The WAS was collected at a municipal wastewater treatment plant (WWTP, 1.850 $m^3 \cdot day^{-1}$, 10.000 population equivalent, PE). This plant performs organic C removal via aerobic biodegradation and N removal via nitrification-denitrification in a single tank using intermittent aeration. Surplus sludge discharged from clarifiers passes through a draining table prior to storage. The WAS was collected at the outlet of draining table. The DS was collected at one other WWTP (30 240 $m^3 \cdot day^{-1}$, 140.000 PE). At this plant, primary sludge and WAS are mixed together and treated by anaerobic digestion. Samples of DS were collected at the outlet of the centrifuge after digestion.

2.2 Experimental conditions

The purpose of this experiment was to monitor the aerobic treatment of MSW, MMW and DS for a period of 50 days using 10 identical cells (C1 to C10). We studied successively three wastes in these 10 cells.

For each cell, wastes were mixed with bulking agents and homogenized manually. Bulking agents used in this study were Ø 16mm polypropylene pall rings (PR). The material polypropylene did not absorb water or N materials and neither contained microorganisms. The total volume of the mixture was nearly 10 litters. The masses of wastes and PR were as below: (1) 1 600 g MSW and 512 g PR; (2) 1 000 g WAS, 1 300 MSW and 430 g PR; (3) 4 200 g DS and 517 g PR. The volume ratio of waste/bulking agents was around 1/2.5. The temperature of wastes was controlled constantly at 35 °C. This medium temperature is optimal for aerobic biodegradation (Tremier et al., 2005). At the same time, it avoided inhibition on nitrification at elevated temperatures (Grunditz and Dalhammar, 2001, Myers, 1975). The aeration rate was fixed at 78 $L \cdot h^{-1}$. Every 5 days, one cell among the 10 cells was stopped. The residues of this cell were separated from PR and stored for further analyses. Residues of other cells were turned. 200 g of distilled water were added to keep the materials humid.

2.3 Characterization of materials

Physical and chemical analysis of samples were given in detail in our previous study (Zeng et al., 2012). Samples preparation and real-time PCR analyses were also detailed. Each analysis was practiced at least 3 times. The coefficient of variation (C.V.) was controlled and additional analysis was practiced once C.V. was superior to 5 %. The characteristics of studied wastes and residues are respectively given in TABLE 1 and 2.

2.4 Theory / calculation

N transfers and transformations during aerobic treatment of organic wastes can be summarized through several processes. Quantify N flows through these processes makes meet two difficulties related to respectively Norg mineralization and NH_4^+/NH_3 conversion through nitrification then denitrification. Mineralization of Norg-containing materials releases NH_4^+/NH_3. Initial NH_4^+/NH_3 and released NH_4^+/NH_3 contribute to the pool of free NH_4^+/NH_3. For this reason, mineralization of Norg is also termed as ammonification. However, this process is bilateral. Parts of NH_4^+/NH_3 can be immobilized by biomass to synthesize Norg (Liang et al., 2004, Paredes et al., 2000). Since it was difficult to measure the gross NH_4^+/NH_3 immobilization or gross Norg ammonification flow, biodegradable Norg (Nbio) was calculated by subtracting Norg in final residues from Norg in initial wastes. A positive value of Nbio indicates a net ammonification flow, whereas a negative one indicated a net immobilization flow.

Besides ammonification, Ekliand and Caceres suggested that nitrifying microorganisms can oxidize NH_3 to NO_3^- during aerobic treatment of wastes (Caceres et al., 2006, Eklind and Kirchmann, 2000). The formed NO_3^- and NO_2^- (NOx-) are further transformed to N_2 via denitrification (de Guardia et al., 2010; Mahimairaja et al., 1994). Parts of N are lost in form of N_2O since the latter is a by-product of both nitrification and denitrification. In the present work, since initial and final NH_4^+/NH_3, Nbio and NH_3 emissions were quantified, the amount of oxidized NH_3 was calculated according to the N lacking in NH_4^+/NH_3 balance. Since there was only negligible of leachate (<10 g) in the cells, these trace leachates were not analyzed.

3. RESULTS AND DISCUSSION

3.1 Mineralization of Norg

Above 60 % of O_2 were consumed in first 15 days of treatment of three kinds of wastes. This period corresponded to the intense biodegradation stage of OM in the wastes. From day 15 to day 50, O_2 consumption slow down and remained at relative low level. This period corresponded to the maturation stage of resiudes. FIGURE 1-I presents the amount of Nbio of three kinds of wastes through 50 days' treatment. The most parts of values of Nbio were positive. It indicated the gross mineralization flow was superior to the gross immobilization flow throughout the treatment. Obvious net immobilization could only be observed on MMW from day 25 to 35. The mineralization of Norg in MSW exhibited two phases. The first phase began after ten days and the second began after 25 days. The mineralization of MMW and DS began early and concentrated in first ten days. Nbio of MMW increased from 6 to 9 g $N{\cdot}kg^{-1}$ OM_0 on day 10 and decreased dramatically after day 10. Similar to MSW, MMW exhibited a second phase of mineralization at around day 45. Nbio of DS was the highest compared to MSW and MMW, which attained 20 g $N{\cdot}kg^{-1}$ OM_0 on day 5. It however decreased dramatically from day 5 to day 15 and remained at around 1-2 g $N{\cdot}kg^{-1}$ OM_0 until the end of treatment.

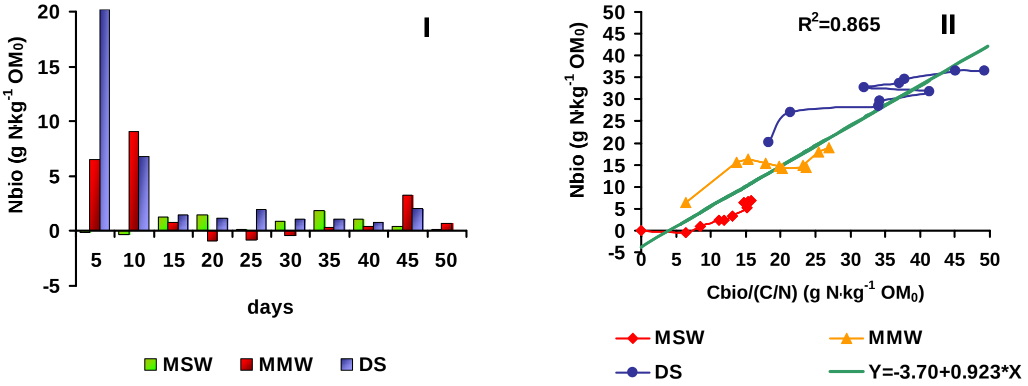
TABLE 1: Characteristics of organic wastes

	MSW	MMW	DS
DM (g·kg-1 OM0)	1354.65	1322.58	1709.99
NH_4^+/NH_3 (g N kg-1 OM0)	4.77	4.67	14.65
KN (g N·kg-1 OM0)	26.12	41.55	93.26
NO_2^- (g N·kg^{-1} OM$_0$)	0.00	0.00	0.00
NO_3^- (g N·kg^{-1} OM$_0$)	0.04	0.01	0.08
TC (g C·kg^{-1} OM$_0$)	510.57	572.15	638.79

TABLE 2: Characteristics of residues of organic wastes

I	Residues of MSW									
	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10
DM (g·kg^{-1} OM$_0$)	1329.44	1143.20	984.86	885.12	873.93	852.02	765.16	739.65	714.79	695.58
OM (g·kg^{-1} OM$_0$)	973.95	777.95	627.06	517.35	504.96	475.34	407.98	384.99	368.40	356.56
NH_4^+/NH_3 (g N kg^{-1} OM$_0$)	4.48	1.65	1.27	2.04	1.37	0.95	0.11	0.06	0.04	0.06
KN (g N·kg^{-1} OM$_0$)	25.94	23.42	21.73	21.08	20.27	18.91	16.25	15.10	14.70	14.62
NO_2^- (g N·kg^{-1} OM$_0$)	0.00	0.01	0.00	0.00	0.00	0.00	0.37	0.69	0.14	0.14
NO_3^- (g N·kg^{-1} OM$_0$)	0.03	0.01	0.00	0.01	0.01	0.01	0.13	0.30	0.04	0.01
TC (g C·kg^{-1} OM$_0$)	511.63	386.86	345.29	292.00	278.52	256.03	215.62	222.56	210.72	203.25
II	Residues of MMW									
	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10
DM (g·kg^{-1} OM$_0$)	1224.24	1106.02	1024.36	958.38	903.41	864.89	854.26	812.22	747.34	718.15
OM (g·kg^{-1} OM$_0$)	890.39	775.88	698.72	626.78	572.58	538.91	508.71	473.20	430.92	409.34
NH_4^+/NH_3 (g N kg^{-1} OM$_0$)	8.70	4.61	0.90	0.65	0.46	0.33	0.59	0.59	0.34	0.11
KN (g N·kg^{-1} OM$_0$)	38.25	25.17	20.86	20.95	21.39	21.63	22.12	21.38	18.37	17.29
NO_2^- (g N·kg^{-1} OM$_0$)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
NO_3^- (g N·kg^{-1} OM$_0$)	0.01	0.01	0.00	0.01	0.00	0.01	0.00	0.00	0.01	0.01
TC (g C·kg^{-1} OM$_0$)	482.87	379.27	355.72	320.31	294.06	287.62	239.21	246.38	213.68	193.26
III	Residues of DS									
	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10
DM (g·kg^{-1} OM$_0$)	1621.32	1539.68	1524.57	1475.29	1457.93	1476.37	1405.04	1409.29	1364.88	1305.97
OM (g·kg^{-1} OM$_0$)	925.45	860.06	824.18	784.70	767.60	769.34	725.42	714.37	682.03	652.59
NH_4^+/NH_3 (g N kg^{-1} OM$_0$)	18.19	15.72	13.13	11.63	10.18	10.45	9.51	9.63	9.25	3.96
KN (g N·kg^{-1} OM$_0$)	76.62	67.32	62.44	60.54	57.15	56.38	54.40	53.68	51.32	45.98
NO_2^- (g N·kg^{-1} OM$_0$)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.07	0.00
NO_3^- (g N·kg^{-1} OM$_0$)	0.04	0.03	0.04	0.04	0.04	0.03	0.10	0.03	0.03	0.03
TC (g C·kg^{-1} OM$_0$)	513.99	491.87	405.97	404.61	355.95	419.98	385.35	380.44	330.10	302.14

Mineralization of Norg resulted from biodegradation of N containing part of organic wastes, that is to say, the biodegradability and the relative N content to C content of organic wastes decided the mineralization of Norg. FIGURE 1-II demonstrates the evolution of Nbio with the value Cbio/(C/N) of each waste through 50 days' treatment. The Nbio was positively correlated with the value Cbio/(C/N). This correlation is statistically significant for three kinds of wastes (R^2=0.865). In spite of this, the composition and biodegradation process of each waste made the Nbio fluctuated around the straight line. The increase of Nbio of DS was significant in first two points. The high Norg-containing part of DS might be mineralized in this period. After day 10, its points approached to the straight line and fluctuated around it. FIGURE 1-II revealed two phases of mineralization of Norg in MSW and MMW. The Nbio of MSW did not increase with the biodegradation in the beginning and began to rise in parallel with the straight line. Although the ratio remained at around 15, an increase of Nbio was still observed at the end of treatment. Compared MMW to MSW, we found MMW had a parallel increase of Nbio with the straight line at the beginning of treatment. The introduction of WAS into MSW might increase the content of easily biodegradable Norg and stimulate the mineralization process.



MSW: municipal solid wastes, MMW: mixture of municipal solid wastes and waste activated sludge, DS: digested sludge

FIGURE 1: I, Mineralization of Norg for each 5 days; II, Evolution of mineralization of Norg with ratio Cbio/(C/N).

The two phase's net mineralization of Norg of MSW and MMW might be explained by three reasons: 1 Parts of Norg were firstly degraded to some simple N-containing compounds and stored in the residues. These compounds were then mineralized after biodegradation slowed down. 2 A slowly biodegradable fraction with high content in Nbio was mineralized after an easily biodegradable fraction with low content in Nbio was mineralized. 3 NH_4^+/NH_3 released from ammonification at the beginning of treatment were firstly immobilized into biomass and re-mineralized once the biomass declined.

3.2 NH_4^+/NH_3 dynamic

Transfers and transformation of NH_4^+/NH_3 pool of three wastes are presented in FIGURE 2.

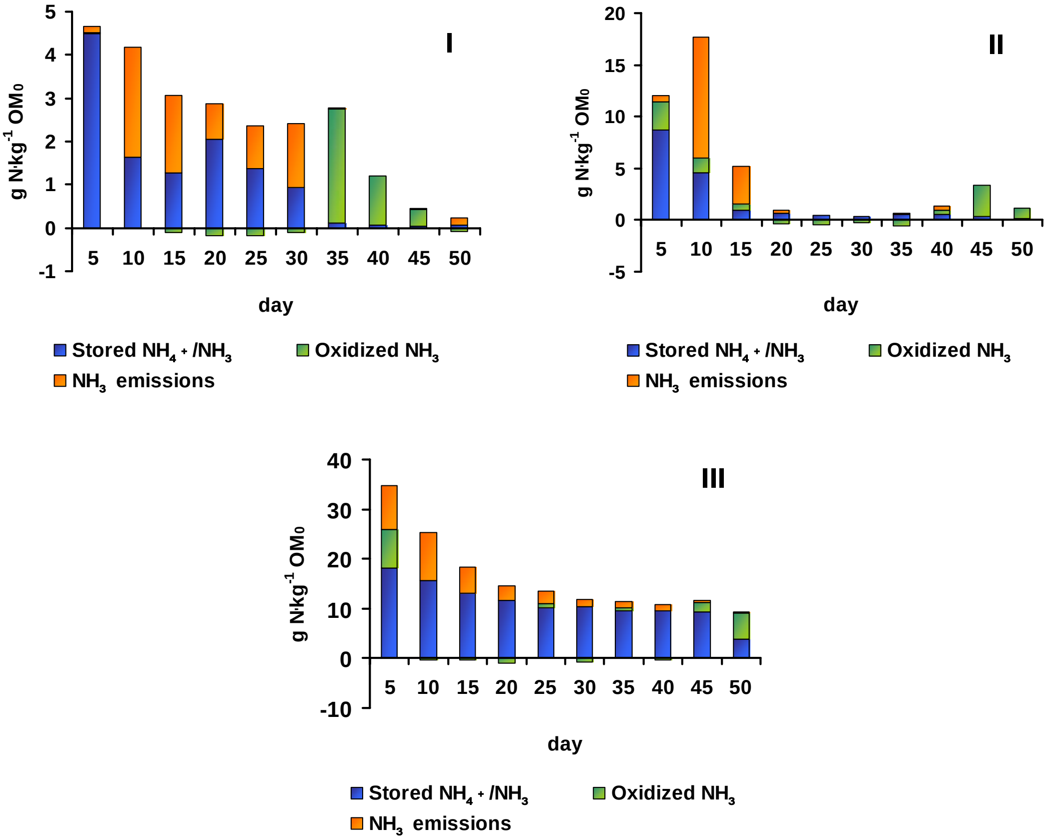
NH_4^+/NH_3 were mainly stored in the materials during the first five days' treatment of MSW. Between day 5 and day 30, although mineralization of Norg supplied the NH_4^+/NH_3 pool, the size of pool reduced gradually. NH_3 emissions were the main sink of NH_4^+/NH_3 pool. The second phase of mineralization of Norg contributed to an increase of NH3 emissions between day 25 and 30. After day 30, NH_3 oxidation occurred and consumed the NH_4^+/NH_3 pool. Both stored NH_4^+/NH_3 and NH_3 emissions decreased to around zero. At the end of treatment, NH_4^+/NH_3 pool decreased to zero since mineralization stopped to supply NH_4^+/NH_3.

Mixing WAS with MSW increased the size of NH_4^+/NH_3 pool. NH_3 oxidation exhibited two phases during the entire treatment. NH_3 oxidation was promoted in first 15 days thanks to introduction of WAS. However, the amount of NH_3 oxidation decreased gradually, which was much less than NH_3 emissions. NH_3 emissions dominated the NH_4^+/NH_3 pool in this period and resulted in the depletion of NH_4^+/NH_3. At around day 40, the second phase of Norg mineralization contributed to an increase of NH_4^+/NH_3 pool. The second phase of NH_3 oxidation occurred synchronously and consumed the NH_4^+/NH_3 released from mineralization. The amount of stored NH_4^+/NH_3 dropped to around zero at the end of treatment.

The NH_4^+/NH_3 pool of DS decreased gradually through the treatment.

Although NH_3 oxidation was observed in first 5 days, NH_3 emissions were still the main sink of NH_4^+/NH_3 pool until day 40. After day 40, NH_3 oxidation occurred, which reduced both NH_3 emissions and the amount of stored

NH_4^+/NH_3 in the final residues. Compared to MSW and MMW, the amount of NH_4^+/NH_3 stored in DS was much higher.



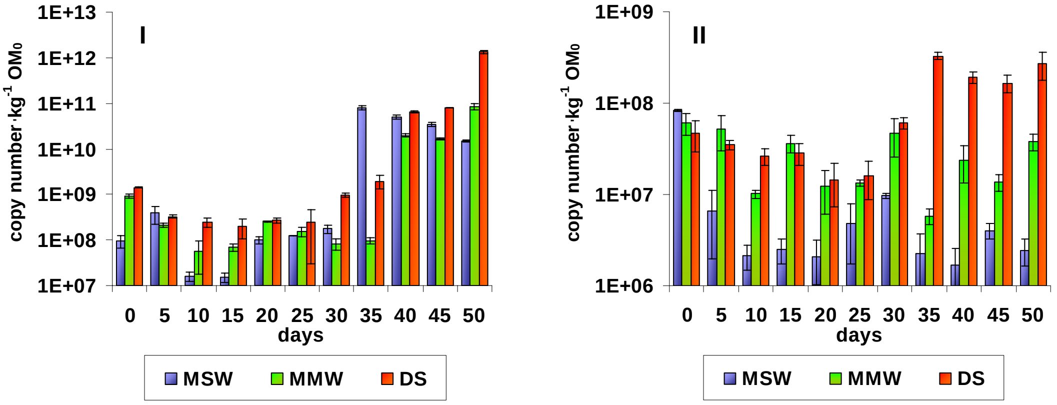
MSW: municipal solid wastes, MMW: mixture of municipal solid wastes and waste activated sludge, DS: digested sludge

FIGURE 2: Transfers and transformation of NH_4^+/NH_3 during aerobic treatment of MSW (I), MMW (II) and DS (III).

3.3 Evolution of groups of NH_3 oxidizers

Evolutions of AOB and AOA are demonstrated respectively in FIGURE 3-I and 3-II. The genes number of AOB in MSW fluctuated in first 30 days, while that of AOB in both MMW and DS decreased gradually in first 15 days and fluctuated at around 10^8 copies number·kg^{-1} OM$_0$. After 30-35 days' treatment, the number of AOB increased dramatically and remained at high abundance till the end of treatment. AOB exhibited the similar tendency during treatment of three wastes: A lag or declining phase followed by a significant multiplication phase.

AOA were less abundant than AOB. The number of AOA declined in MSW and remained at low level till the end of treatment. The number of AOA in MMW fluctuated during the entire treatment. We observed a multiplication of AOA only during treatment of DS, which occurred at around day 35. However, this multiplication was accomplished by an increase of total archaea (TA) (data not shown). This multiplication could hardly be interpreted as a development of AOA group.



AOA: ammonia-oxidizing archaea, AOB: ammonia-oxidizing bacteria, MSW: municipal solid wastes, MMW: mixture of municipal solid wastes and waste activated sludge, DS: digested sludge

FIGURE 3: Evolution of AOB (I) and AOA (II) during aerobic treatment of MSW, MMW and DS.

3.4 Correlation between development of NH_3 oxidizers and NH_3 oxidation flow

In order to investigate the relationship between NH_3 oxidizers and NH_3 oxidation flow, we employed ratio AOB/(TB+TA+TF) and ratio AOA/(TB+TA+TF) to represent the relative abundance of AOB and AOA in three wastes and residues of three wastes. NH_3 oxidation flows, ratios AOB/(TB+TA+TF) and AOA/(TB+TA+TF) were normalized. The correlation between relative abundances of AOB and AOA and NH_3 oxidation flows were analyzed statistically (TABLE 3). Only development of AOB was statistically correlated to NH_3 oxidation flow obtained from characterization of N balance. The significant correlation and the high abundance of AOB in the residues revealed only AOB were responsible for NH_3 oxidation during aerobic treatment of MSW, MMW and DS.

TABLE 3: Correlation between ammonia oxidizers and NH_3 oxidation

		AOB/(TB+TA+TF)			AOA/(TB+TA+TF)		
		MSW	MMW	DS	MSW	MMW	DS
NH_3 oxidation flow	Pearson Correlation	0.746*	0.686*	0.749*	-0.313	0.536	0.419
	Sig. (2-tailed)	0.013	0.029	0.013	0.378	0.110	0.228
	N	10	10	10	10	10	10

*Correlation is significant at the 0.05 level (2-tailed).

4. CONCLUSIONS

Our work investigated the N dynamic and the evolution of NH_3 oxidizers during aerobic treatment of three kinds of organic wastes. Our finding revealed mineralization of Norg was correlated to ratio Cbio/(C/N) of each type of waste. Mineralization of Norg in MSW or MSW mixing with WAS (MMW) exhibited two phases. The mineralization of Norg following the intense biodegradation stage of organic wastes resulted in an increase of NH_4^+/NH_3 pool and hence NH_3 emissions. Although NH_3 oxidation occurred during the intense biodegradation stage of MMW and DS, the oxidation flow was much lower than NH_3 emissions. For each waste, an increase of NH_3 oxidation occurred similarly at the end of maturation stage. Evolution of groups of NH_3 oxidizers and statistical analysis of their relative abundance and NH_3 oxidation flow suggested only AOB were responsible for NH_3 oxidation during aerobic treatment of three kinds of organic wastes.

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(171) OPTIMISATION OF THE AEROBIC MATURATION PROCESS OF THE MSW DIGESTATE ON MONTPELLIER AMETYST PLANT

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EXECUTIVE SUMMARY

Due to an increasing environmental pressure affecting regulation of waste treatment in developed countries, new solutions of collection, specific treatment and recycling of organic waste appear in many countries in order to lower the quantities of organic waste that directly sent towards land filling or incineration. The growing scarcity and increasing price of fossil energy leads towards the more and more systematic integration of an anaerobic digestion stage in the biological treatment of bio waste or mixed waste.

In the European Union, the regulatory context concerning waste management is still in evolution : If Germany has voted a new waste law on a reinforced but not completely mandatory selective collection of bio waste that will enter in force on the 1st June 2012, French big producers of bio waste have to collect them selectively since the 1st January 2012 and to treat them through a biological treatment with agronomic recycling of the end products.

This paper summarizes the main results of an industrial test led on Ametyst plant through a dedicated R&D program led by CIRSEE in partnership with the Ametyst team operating the plant and the municipality. The origin of this test can be found in the conclusion of our previous paper called “Contribution to the agronomic characterization of Methacompost* produced on Monpellier Ametyst plant” where we indicated that the internal bulking agent used in the initial maturation process (the 0-80 mm MSW pre-treated fraction) was not able to give a sufficient porosity to the raw digestate for a good composting and was bringing too much raw cellulose and too many physical impurities to the mix. In this industrial test, 2 different batches are formed : one test batch with the mix of 0-20 mm digestate with an external bulking agent composed of wood pallets and shredded green waste and one reference batch with the mix of the same digestate with the internal bulking agent that is the 0-80 mm fraction.

In the first part, the protocol of the test is described : composition of each batch, process lines applied to each batch and technical follow up applied (temperature of each batch and processed air evolution, mass balance of the 4 weeks ventilated maturation stage applied to each batch with a weekly turning, evolution of the biodegradability of each mixture during the maturation process and final maturity level /agronomic and environmental quality of the Methacompost produced by each batch).

In the second part, some adaptations to the initial protocol and linked to some operational and not foreseeable events affecting more particularly the test batch maturation are mentioned and main results are presented and discussed. The temperature evolution of each batch and total volumes of processed air show rather important differences : the temperature of the test batch is well controlled (average temperature under 40°c) through a total extracted volume in 4 weeks of 131.000 m3 while the temperature of the reference batch is badly controlled and generally too high (average temperature over 50°c) in spite of a total extracted volume of 212.000 m3. The mass balance and the residual biodegradability of the 2 batches confirm the different efficiency of the ventilated maturation process from one batch to the other : the same global loss of weigh (45%) but without degradation/stabilisation of organic matter for the reference batch and final Methacompost that presents a rottegrad 1 and with an effective degradation of organic matter for the test batch and final Methacompost that presents a rottegrad 5 and that complies with the U44051 french standard. In conclusion, the efficiency and industrial interest of this new process of maturation using an external bulking agent is outlined and it is mentioned that it is being operated at an industrial scale on the Ametyst plant.

* Methacompost^{R} is a commercial brand registered by Suez Environment

1 INTRODUCTION

This paper summarizes the main results of an industrial test led on Ametyst plant through a dedicated R&D program led by CIRSEE in partnership with the Ametyst team operating the plant and the municipality.

1.1 Background

Under the growing environmental pressure and taxes that affect in many developed countries as the 27 European Union member states or Australia, the direct land filling or incineration of municipal waste, the emergence of new ways of collection, treatment and organic recycling of domestic, industrial or agricultural waste is confirmed. Due to an additional leverage created by a growing scarcity and increasing price of fossil energy, the integration of an anaerobic digestion stage appears more and more frequently in numbers of existing composting platforms or in new projects that combine renewable energy and renewable fertilisers production from organic waste.

In the European Union, the regulatory context concerning waste management is still in evolution through a stronger application of the 1999 landfill directive and the progressive application of the new 2008 framework directive on waste management : If Germany has voted a new waste law on a reinforced but not completely mandatory selective collection of bio waste that will enter in force on the 1st June 2012, French big producers of bio waste have to collect them selectively since the 1st January 2012 and to treat them through a biological treatment with agronomic recycling of the end products.

In this context, we observe in France more and more various private and municipal projects with AD and dealing with selectively collected bio waste or residual municipal solid waste (MSW).

1.2 Research context and objective

The Montpellier Ametyst plant that we have already described in a previous ORBIT 2010 paper (1) still remains today the biggest French AD plant with a global capacity of 203,000 tpa (170,000 tpa of residual MSW and 33,000 tpa of selectively collected bio waste) and is operated by SITA Méditerranée, a regional subsidiary of SITA France.

The objective of the industrial test led in 2010 with the Ametyst operating team was the technical validation of a new maturation process of the 0-20 mm digestate. It consisted in comparing the initial 0-20 mm digestate maturation process with a new process using a safe and efficient bulking agent and supposed to improve the final quality of the Methacompost produced in 2009 and that had been evaluated in our previous paper.

The original maturation process that had been designed by the study office commissioned by the municipality of Montpellier was consisting in mixing the 0-20 mm raw digestate with the 0-80 mm pre-treated fraction of MSW coming from the rotating tubes. As it was mentioned in the conclusion of our previous paper, the use of this 0-80 mm fraction as bulking agent was bringing to the mixture a lot of physical impurities and too much fresh and cellulose rich organic matter and led on one hand to the physical pollution of the digestate and, on the other, to a too long maturation process that had to be achieved out of the Ametyst plant by creating storage external platforms.

2 METHODOLOGY

It has been decided with the operator to form and to follow the 4 weeks aerated maturation phase of 2 industrial batches of about 500 m3 each by using for each batch only 2 adjacent Ecosilos per batch except of 4 (one per week because of the application of a turning per week and moving of each batch from one silo to another) in order to minimise the impact of the trial on the current operation of the plant and facilitate the technical follow up and more particularly the temperature evolution of each batch through an easier positioning/ removal of probes before and after each weekly turning/moving.

2.1 The 2 studied batches and corresponding process lines

- The test batch

The volumic mixing ratio used for this test batch (compromise between an optimal porosity and a minimal consumption of external bulking agent) will be equal to 1 volume of 0-20 mm raw digestate for 1 volume of shredded wood pallets and 0,5 volume of shredded green waste. The initial mixing will be done directly on the soil of the maturation hall with help of a front loader equipped with a bucket of 3,5 m3 and with an autonomous weighing system. The initial mixture will be placed in the first Ecosilo with the same front loader and the global initial weigh of the batch will be evaluated by using the same weighing system.

A negative and intermittent forced ventilation (managed as for the reference batch through the industrial process and the supervision software) will be applied automatically during the 4 weeks and a pre-refining stage will be applied specifically to the test batch with a 40 mm mesh mobile rotating screen rented for this industrial test in order to separate and recycle the wood pallets. The global process line applied to the test batch is shown on figure 1.

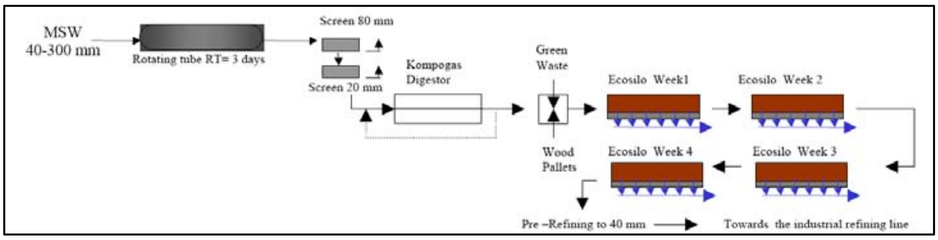

Figure 1 : The global process line applied to the test batch

- The reference batch

For the reference batch, the initial mixing is done as usual with an automatic screw mixer and with the usual industrial volumic ratio : 2 volumes of the pre-treated 0-80 mm fraction for one volume of raw 0-20 mm digestate.

The initial mixture is placed in the first Ecosilo with help of the same front loader used for the test batch. The process line applied to the reference batch is shown on figure 2.

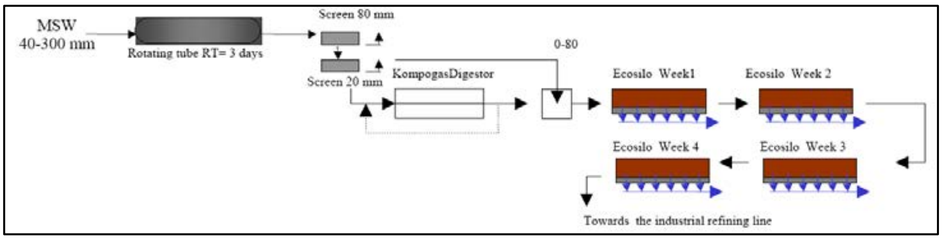

Figure 2 : The global process line applied to the reference batch

2.2 Follow up of the 2 batches during the maturation stage

- Follow up of the temperature

For the batch test, four probes (T1, T2, T3 and T4) of 2 meters long each will be placed in different sectors of the Ecosilo in order to measure continuously (except during each turning) the temperature at the heart of the pile, and for the reference batch, only one probe (T5) will be placed in the same sector of T1 as shown on figure 3. the process air temperature of each batch will be followed and registered too during the four weeks of maturation.

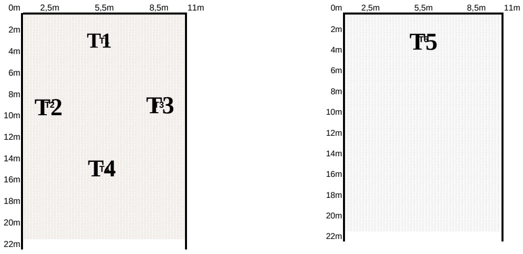

Figure 3 : location of the temperature probes in the test batch (left)and in the reference batch (right)

- Mass balance of the maturation stage including the final refining

As indicated for the initial mixing, the weigh of each batch will be evaluated at each turning and moving from one Ecosilo to another and one sampling with quartering will be done after each turning in order to measure the dry matter content. Concerning the refining line described on figure 4, it will be cleaned before the refining of the 2 followed batches and the weigh of each fraction produced in the 4 silos located under the 8 mm mesh trommel and the densimetric table will be measured in order to know for each batch the quantity of Methacompost produced .

- Biodec respirometric test and Rottegrad or self heating test

The Biodec test (photo 1) already described in a previous paper (2) will be successively applied to 3 samples of each batch (fresh mixture, 2 and 4 weeks matured raw products) while the more simple but less precise rottegrad test that measures during one week the elevation of temperature in the humidified Methacompost (about 50% of water content) placed in a Dewar container of 1, 5 litre capacity (as shown on photo 2) will be applied to each 4 weeks refined product. The maximal temperature measured allows to evaluate the degree of maturation of the compost as indicated on table 1.

Photo 1 : Biodec Test principle

Photo2 :

Table 1 : Rottegrad classification

Maximal Temperature (°C)	Rates of decomposition	Matter Designation
60-70	I	Raw Matter
50-60	II	Fresh Compost
40-50	III	
30-40	IV	Matured Compost
20-30	V	

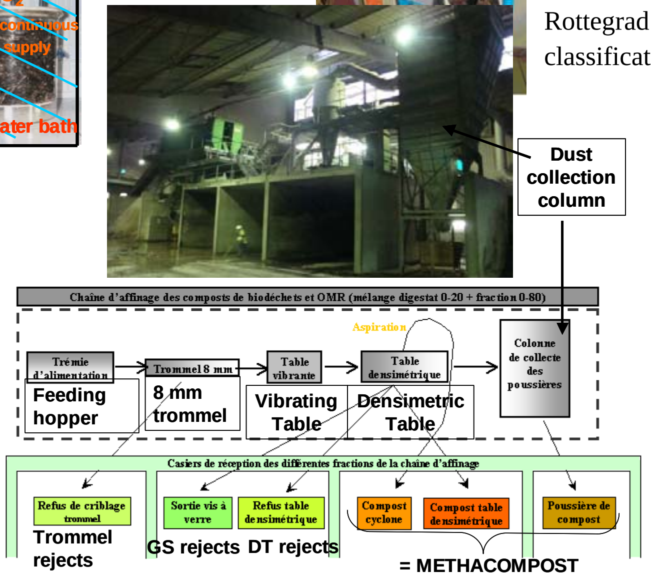

Figure 4 : Description of the Ametyst industrial refining line

- Methacompost compliance with the U44051 french standard

The 2 piles of final Methacompost produced from each batch will be sampled and the 2 samples will be sent to an accredited lab in order to compare their respective quality level and compliance to the new 2006 U44051 french standard.

3 RESULTS AND DISCUSSION

3.1 Realisation of the 2 batches and application of the initial protocol

We can state on table 2 a rather important loss of volume (25%) during the mixture of the test batch while this loss is very little (3%) for the reference batch ; it already shows the better porosity of the bulking agents used for the batch test.

BATCH Test	Components	Initial weigh (in Tons of HM and %)		Volumes (in m3 and %)	
Intial dry matter content : 39,4 %	Wood Pallets	65,5	20%	218,5	38,1%
	Geen waste	55,0	16,8%	148,1	25,8%
	0-20 mm Digestate	207,0	63,2%	207,0	36,1%
	Total	327,5	100%	433,0	75% *

Reference Test	Composition	Initial weigh (in Tons of HM and %)		Volumes (in m3 and %)	
Intial dry matter content : 39,5 %	0-80 mm fraction	255,4	58,5%	382	67,8%
	0-20 mm Digestate	181,2	41,5%	181,2	32,2%
	Total	436,6	100%	545,7	97%*

* : expressed in % of the sum of initial volume of each component

Table 2 : Constitution of the 2 batches

Due to different events linked to the operation of the plant, the reference batch constitution has been achieved on the 18 june 2010, 10 days after the test batch one and some works have been engaged without previous advertising by the supplier of the plant on the ambient air ventilation line of the maturation hall between the 8 june and the 16 june with a general stop of the general ventilation. It has led to the increase of the internal temperature and hygrometry respectively over 30°c and 100% with consequent failing of the ventilators used for the batch test from the 12 to the 16 june.

This rather long stage of no forced ventilation and high hygrometry has probably lowered the performances of the process applied to the batch test concerning its drying and final dry matter content. It will be taken in consideration for the discussion of the results.

Concerning the application of the different weekly turnings, the test batch ventilation has started on the 8 june, with subsequent turning and moving on the 15, 23 and 30 june and final refining on the 7 july while the reference batch ventilation has only started on 18 june, with subsequent turning on 29 june, 9 july and 17 july and final refining on the 20 july only 3 days after the third turning in order to respect the one month maturation global residence time and not too much disturb the general working of the plant.

3.2 Follow up of temperatures of the piles and of processed air

- Test batch

The evolution of the temperature of the four probes located in the test pile and of processed and ambient air is reported on figure 5 .

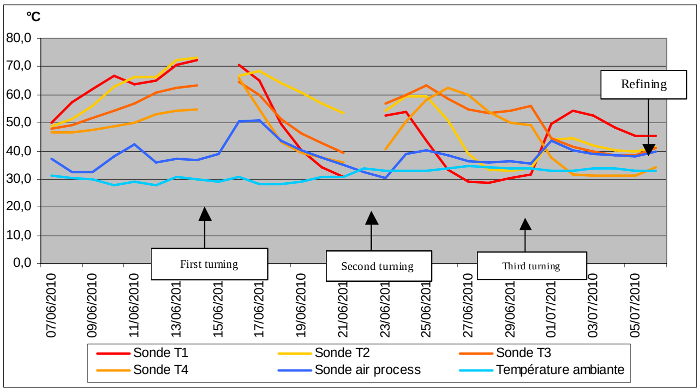

Figure 5 Follow up of the temperature of the Test batch

All the probes except T4 show a quick and important increase of temperature from 50 to 60/70 ° with the start of ventilation while the processed air increases a little later from 32 to 42°c. The accidental stop of the forced ventilation from the 12 to the 16 june leads to a new increase of temperature of all the probes over 55°c by lack of heat extraction while the processed air temperature falls quickly under 40°c until the first turning and the restart of forced ventilation. After the first turning, processed air temperature increases quickly to 50°c and remains 2 days a this level while nearly all the probes temperatures fall from 60/70 °c to 50°c ; then the processed air temperature and the probes temperatures fall progressively to 30°C until the second turning. The same evolutions are observed after the second and the third turning but with a less important fall observed after the peak of temperature of the processed air. These observations seem to show a rather good control of the pile average temperature by the ventilation process that is able (when it works!) to extract heat from the pile and to cool it because of a good initial porosity that is restored after each turning.

During the four weeks of maturation, the average temperature of the processed air remains under 40°c and the total ventilation duration is about 218 hours for a total extracted volume of 130,800 m3 (average airflow of 600 m3/h) and the final dry matter content of the pile is 55%.

- Reference batch

The evolution of the temperature of the single probe located in the test pile and of processed and ambient air is reported on figure 6.

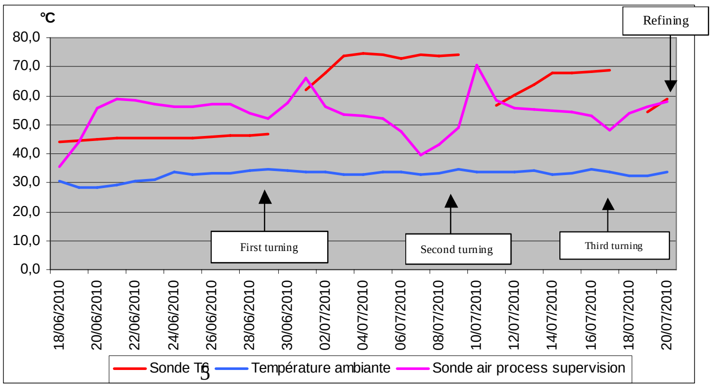

Figure 6 **Follow up of the temperature of the Reference batch**

Contrary to the Test batch and to the T1 probe behaviours, the evolution of the temperature of the single T5 probe doesn't seem to be controlled by the forced ventilation system and remains under 50°c until the first turning while the processed air temperature reaches quickly and remains at 60°c. After the first turning, the temperature of the probe reaches rather quickly 70°c and remains at this level while the processed air shows a quicker increase to 65°c and a peak before decreasing up to 40°c before the second turning. The same evolution is observed after the second and the third turning but with less important levels of temperature for the T1 probe.

These observations seem to show a poor control of the pile average temperature by the ventilation system due to a bad initial porosity that is partially improved after each turning.

During the four weeks of maturation, the average temperature of the processed air remains over 50°c and the total ventilation duration is about 606 hours for a total extracted volume of 212,100 m3 (average airflow of 350 m3/h) and the final dry matter content of the pile is 72%.

3.3 Mass balance

- Mass balance of the ventilated maturation phase

The mass balance of the 2 batches considered from initial mixture to the end of the maturation phase are reported on table 3 and 4.

Date	Humid tonnage	DM tonnage	Water tonnage	DM loss	DM cumulated loss	Water loss	Water cumulated loss	total losses	total cumulated losses
8 june	327,5	129,3	198,1						
15 june	293,7	124,8	168,9	4,5	4,5	29,2	29,2	33,7	33,7
23 june	241,6	111,9	129,8	12,8	17,3	39,1	68,3	51,9	85,6
30 june	199,3	105,2	94,3	6,7	24	35,5	103,8	42,2	127,8
7 july	178,8	98,5	80,2	6,7	**30,7**	14,1	**117,9**	20,8	**148,6**
total losses 1	in % of the initial quantity of the concerned parameter				**23,7%**		**59,5%**		**45,4%**
total losses 2	relative contribution to the total cumulated losses			**20,7%**		**79,3%**			
total losses3	effective contribution to the total cumulated losses			**9,4%**		**36,0%**			**45,4%**

Table 3 Mass balance of the test batch

Date	Humid tonnage	DM tonnage	Water tonnage	DM loss	DM cumulated loss	Water loss	Water cumulated loss	total losses	total cumulated losses
18 june	436,6	172,5	264,1						
29 june	371,9	172,2	199,7	0,3	0,3	64,4	64,4	64,7	64,7
9 july	325,8	172,3	153,5	-0,1	0,2	46,2	110,6	46,1	110,8
17 july	257,4	172,2	85,2	0,1	0,3	68,3	178,9	68,4	179,2
20 july	237,1	170,2	66,9	2	**2,3**	18,3	**197,2**	20,3	**199,5**
total losses 1	in % of the initial quantity of the concerned parameter				**1,3%**		**74,7%**		**45,7%**
total losses 2	relative contribution to the total cumulated losses			**1,2%**		**98,8%**			
total losses3	effective contribution to the total cumulated losses			**0,5%**		**45,2%**			**45,7%**

Table 4 Mass balance of the reference batch

We state that if the global losses are comparable for the 2 batches and represent 45% of the initial humid tonnage, the dry matter losses contribute to nearly 10% of the initial humid tonnage for the test batch and only to 0,5% for the reference batch. It means that for the test batch, the bulking agent mixed to the digestate has allowed a rather well balanced composting associating degradation/stabilisation and drying of the mixture and that for the reference batch, the 0-80 mm fraction mixed to the digestate has only allowed a good drying of the mixture but without real stabilisation.

- Mass balance of the final refining

The mass balance of the final refining stage applied to the 2 batches are reported on table 5 and table 6.

product	humid tonnage (in tons)	in % of (1)	in % of (2)
raw compost (1)	178,8	100	
pre screened compost (2)	111,4	62,3%	
rejects > 40 mm	67,4	37,7%	
rejects > 8 mm	57,9	32,4%	52,0%
glass rejects	5,9	3,3%	5,3%
Methacompost	**47,5**	**26,6%**	42,6%

Table 5 **Mass balance of the test batch final refining**

product	humid tonnage (in tons)	in % of (1)
raw compost (1)	237,1	100
rejects > 8 mm	191,6	80,8%
glass rejects	3,5	1,5%
Methacompost	**40,9**	**17,3%**

Table 6 **Mass balance of the reference batch final refining**

The proportion of Methacompost produced from the test batch is more important (27%) than the one produced from the reference batch (17%) even if the refining line bad tuning to this product that was rather humid was not representative of a routine working while the refining line configuration and flow was well adapted to the reference batch dry product. Reported to the ton of raw digestate treated, the Methacompost production ratio are quite comparable: 229 kg/ton of digestate for the test batch and 226 Kg/ton for the reference batch.

It has to be precised that the more little bring of bulking agent dedicated to the test batch leads to an increase of 45% of tonnage of digestate treated per Ecosilo and that the reference batch process line leads to a production of 1100 kg of ultime rejects per ton of digestate treated against only 400 kg for the test batch process line.

3.4 Biodegradability evolution of the 2 batches during the maturation phase

The results of the 3 successive applications of the Biodec Test to some samples of the test and reference batches are expressed on table 7 in AT4 equivalent that represents the cumulated O2 consumption of the considered samples during the 4 first days (96 hours) of the respirometric test.

	Test batch AT4	Reference batch AT4
fresh sample	33,4	93,5
2 weeks sample	11,9	33,9
4 weeks sample	< 5	13,6

Table 7 AT4 equivalent of different samples of test batch and reference batch (in mg O2/g DM)

It confirms the previous observations done on the follow up of the process and regarding temperature evolution or mass balance of the 2 batches : the test batch mixture presents an initial biodegradability level nearly 3 times lower than the reference batch mixture and reaches in 2 weeks a lower residual biodegradability level than the reference mixture after 4 weeks of ventilated maturation.

The results of the Rottegrad test applied to the samples of the final Methacompost produced by each batch are given on figure 7. These results show for the test batch Methacompost a Rottegrad 5 against a Rottegrad 1 for the reference batch Methacompost and confirm the Biodec test results.

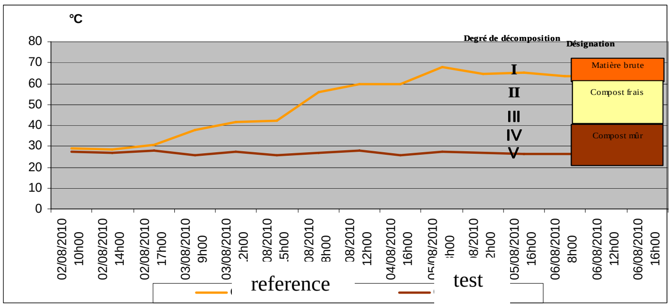

Figure 7 **Results of the Rottegrad test applied to the 2 final Methacompost**

3.5 Methacompost agronomic and environmental quality

The 2 Methacompost produced from each batch are shown on photo 3 : the Methacompost produced from the test batch presents a dark brown colour and a better aspect than the one produced from the reference batch that keeps a grey brown colour given by MSW.

Photo 3 **The 2 Methacompost produced respectively from the test batch (left) and the reference batch (right)**

The main agronomic and environmental quality parameters to be compliant with the new U44051 french standard are presented for the 2 Methacompost on Table 8.

It appears that the reference batch Methacompost doesn't comply with the French standard for the hard plastic parameter (0,9% on DM for a threshold limited to 0,8%) while the test batch Methacompost complies with for all the parameters.

According to the agronomic potential, it is clear that the lower ratio C/N (12 against 22) and the higher fertilising potential in P_2O_5 (1% on HM against 0,7 %) and K_2O (1,4% against 0,9 %) of the test batch Methacompost are mainly linked to the lower dilution of the initial digestate and to the higher fertilising potential brought by a more coarse and effective external bulking agent.

Methacompost Quality	Reference Batch		Test Batch		U44051 / 2006 French standard
	on DM	on HM	On DM	on HM	
Agronomic parameters					
Dry Matter %		65,8		55	>=30 on HM
Organic Matter %		43		27	>=20 on HM
N_{tk} (%)		0,95		1,02	< 3 % on HM
N_{NH4+} (%)		0,13		0,12	
Rapport C/N total		22		12	> 8
Total P_2O_5 (%)		0,68		1	< 3 % on HM
Total K_2O (%)		0,92		1,4	< 3 % on HM
Heavy Metals					
As (mg/kg)	< 2		3,7		18 on DM
Cd (mg/kg)	0,92		1,6		3 on DM
Cr (mg/kg)	47		50		120 on DM
Cu (mg/kg)	190		180		300 on DM
Hg (mg/kg)	0,64		1,1		2 on DM
Ni (mg/kg)	29		29		60 on DM
Pb (mg/kg)	95		120		180 on DM
Se (mg/kg)	< 3		< 3		12 on DM
Zn (mg/kg)	400		490		600 on DM
Physical impurities					
Plastic Films + PSE > 5 mm (%)	0		0		< 0,3 %sur sec
Other plastic > 5 mm (%)	🟥 0,9		0,5		< 0,8 %sur sec
Glass + Metals > 2 mm (%)	0,1		0,3		< 2 % sur sec
Micro organisms					
Salmonella		Absence		Absence	Absence in 25 g

Table 8 **main quality parameters of the 2 Methacompost produced during the industrial test**

4 CONCLUSION

The results of the industrial test led during summer 2010 on the Ametyst plant show the interest of the new maturation process applied to the raw digestate through the use of an external and good quality bulking agent in place of the 0-80 mm MSW pre-treated fraction : much better efficiency of the ventilated maturation process with a good control of the temperature of the mixture, good final maturation level and better agronomic and environmental quality of the final Methacompost. It leads too to an higher capacity of treatment of the maturation hall and to a bigger production of Methacompost that will improve the global diversion rate of the Ametyst plant. That is why this new process has been elected by the Montpellier urban community and is now starting to work at an industrial scale on the Ametyst plant.

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(217) COMPO-BALL: NOVEL ON-LINE COMPOSTING MONITORING SYSTEM. FIRST RESULTS

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EXECUTIVE SUMMARY

Composting is nature's way of recycling organic waste into valuable fertiliser. It is a natural biological process in which microorganisms such as bacteria and fungi break down organic matter. Composting is an easy process but needs to be well controlled to optimise the compost quality and to avoid undesirable odours or germs and loss of nutrients. An optimal composting process will also reduce the volume and weight of organic waste significantly as the composting process converts much of the biodegradable component to gaseous carbon dioxide. The effectiveness of the composting process is dependent upon the environmental conditions present within the composting system, i.e., oxygen, temperature, moisture, material disturbance, organic matter and the size and activity of microbial populations.

Among all the parameters, moisture and temperature are very helpful in process management. The content of water is given in order to allow microbial development, but always keeping aerobic conditions. Temperature is a consequence of the activity of these microorganisms that allows to higienisation of the material limiting the presence of pathogens and weed seeds. While the main biological and chemical parameters affecting the composting process are well known, the technological solutions available for monitoring and controlling the process are very limited. Temperature and moisture in the core of the material that is being composted are currently only measured from time to time and in a number of very limited points. Moreover, temperature is usually manually measured by inserting a probe, and the moisture is measured by extracting samples that need to be analysed in a laboratory.

There is a clear need to provide composting operators with improved process control technology, especially in view of increasingly stringent and evolving regulations. Such regulations, along with market demands for high quality, stable and safe composting are clear drivers for bridging the current gaps in compost monitoring and control technology.

The 3-year research COMPOBALL project (www.sensoball.eu) entitled *Novel on-line composting monitoring system* with grant agreement 243625 is funded by the European Commission and brings together 16 partners from 9 European countries. The aim is to develop a novel on-line wireless system for the measurement of temperature and humidity at various points in the composting material and to bring affordable wireless composting-sensors to the European composting industry.

The COMPO-BALL system is designed for the on-line measurement of the temperature and humidity during the whole composting process. It consists of many autonomous encapsulated sensor nodes, the balls, able to communicate with each other and to transmit the measured data to a pile-hub, which then transfers the information through a gateway into the internet. Real time data of both temperature and humidity of the composting side can be monitored from an easy-to-use user interface. The compoballs contain a rechargeable battery system, which can last up to few months to cover the whole composting process.

The primary results show that it can be an interesting tool for management of composting process and for the whole monitoring of temperature.

1. INTRODUCTION

Composting is nature's way of recycling organic waste into valuable fertiliser. It is a natural biological aerobic process in which microorganisms such as bacteria and fungi break down organic matter. Composting is an easy process but needs to be well controlled to optimise the compost quality and to avoid undesirable odours or germs and loss of nutrients. An optimal composting process will also reduce the volume and weight of organic waste significantly as the composting process converts aerobically much of the biodegradable component to gaseous carbon dioxide. The effectiveness of the composting process is dependent upon the raw materials and the environmental conditions present within the composting system, i.e., oxygen, temperature, moisture, material disturbance, organic matter and the size and activity of microbial populations, where most of them depend on the management.

Composting can treat a wide range of raw materials and is more robust than other biological treatments such as anaerobic digestion.

Organic fraction from municipal solid waste, sewage sludge, manure and agri-food industry wastes are the main groups of material treated biologically, but the characteristics (physical, chemical) of every one of them vary extensively. Aspects like homogeneity, moisture, organic matter, etc, can need of an intensive change on the treatment conditions during composting to optimise product quality and to reduce losses and environmental impact.

Moisture is necessary to maintain the biological activity, but different levels have to be considered depending on the phase of the process. During the decomposition phase of composting, a range between 40-60% of moisture should be kept (Haug, 1993) to help in the maximum degradation, while in maturation phase, water requirements are quite lower. Due to this microbial activity, an increase of temperature is observed as consequence. Then, the following of the temperature can inform about biological activity. In other hand, this increase of temperature allows to higienisation of the material, actuating on the elimination of pathogen and weed seeds.

While the main biological and chemical parameters affecting the composting process are well known, the technological solutions available for monitoring and controlling the process are very limited. Two of these essential parameters, temperature and moisture in the core of the material that is being composted, are currently only measured from time to time and in a number of very limited points. Moreover, temperature is usually measured manually inserting a probe, and the moisture is measured by extracting samples that need to be analysed in a laboratory and the result is obtained after 24 h. The variation of these two parameters can be very wide even in short distance, and this is why can be interesting to be able to monitor them in as much points as possible.

There is a clear need to provide composting operators with improved process control technology, especially in view of increasingly stringent and evolving regulations (Directive 99/31/CEE, Directive 2008/98/CE, COM(2008) 811, Regulation 1774/2002/EC, Regulation 2003/2003/EC). Such regulations, along with market demands for high quality, stable and safe composting are clear drivers for bridging the current gaps in compost monitoring and control technology.

1.1 Research objectives

The 3-year research COMPO-BALL project (www.sensoball.eu) entitled *Novel on-line composting monitoring system*, is funded by the European Commission (grant agreement 243625) and brings together 16 partners from 9 European countries. The overall objective of the COMPO-BALL project is to develop a novel on-line wireless system for the measurement of temperature and humidity at various points in the composting material, which provides affordable wireless composting-sensors to the European composting industry.

2. THE COMPO-BALL SYSTEM

2.1 The base

COMPO-BALL is dedicated to measuring the temperature and humidity during composting processes via autonomous, encapsulated wireless sensor nodes, and is designed for the on-line communication of the sensors. Many autonomous encapsulated sensor nodes, the balls, able to communicate with each other and to transmit the measured data to a pile-hub, which then transfers the information through a gateway into the internet (Figure 1). Real time data of both temperature and humidity of the composting side can be monitored from an easy-to-use user interface.

The system consists of a kit of balls with sensors, the pile-hub and the programme to manage the system (Figure 2). In parallel, a system for charging the balls is needed in order to allow the collection of measures along the time.

2.2 The sensors

Each compo-ball has two sensors inside, one for the temperature and one for humidity. The design of the temperature sensor is based on the use of a modulator sensor of platinum and a sensor-to-microcontroller direct interface, which allows us to reduce the number of components and the power consumption. With this direct sensor-to-microcontroller interface, we have obtained a maximum absolute error of 0.3 °C, and 0.1 °C of resolution in the temperature range from -10 °C to 80 °C. To measure the humidity a capacitive system has been designed, which allow to measure changes in the humidity from 15 %RH to 80 %RH with a resolution of 1 %RH and accuracy of 5 %RH.

2.3 Battery and charging system

The composting process can last up to few months.

In order to be able to monitor the whole process without removing the balls from the compost piles, the balls switch on in regular time intervals, and communicate the measured values into the pilehub. Each of the sensor nodes includes a rechargeable battery that can supply power during each complete composting cycle.

After the end of each cycle, the Compo-Balls are placed in a charging plate where the batteries are charged during 8 hours. The wireless charging system includes a primary coil in the charging plate and a matching coil inside the Compo-Ball to transmit the power from the charging plate into the ball [1,2].

2.4 Communication

The balls contain an antenna that allows them to communicate with each other inside the compost through using a low radio frequency free ISM band and novel communication protocol. The balls transmit the data to the pilehub, which transmit the measured data of temperature and humidity via wi-fi into a web-based database. Operators in the composting plant can see real time data in a friendly web-based user-interface (Figure 3).

2.5 Protection

In order to protect the electronics of the harsh atmosphere of the compost process (physical and chemical aggressions) it is necessary to use a protective material. Rigid-polyurethane has proven to be a good selection as a protective material (Figure 1) in front of other more weak. Tests, both in composting plant and in a composter, have been carried out to validate the capsule mechanical, chemical, electrical and thermal properties. The elements of this material do not imply any risk of contamination of the compost and of reducing its end product quality, since the metal content is lower than the stipulated by European regulations.

3. PRELIMINARY RESULTS

First installations of the COMPOBALL prototype in facilities of the participating countries have already taken place and have given favourable results. These trials were focused in protective material, recovery of the ball, sensor detection and communication.

Protection was essayed with 2 different materials: foamy and rigid polyurethane, with empty balls (e.g without electronics). In the first case, after several turnings with turning machine in composting windrows, the balls were seriously damaged, while in the second case, the balls were undamaged (Figure 4). In relation to recovery, trials were conducted with trommel and star screening, and it was observed that the compo-balls naturally moved into the refuse of both screening systems, but some adjustments to the machinery should be done to improve and facilitate the recovery. The size of the balls (120mm diameter) clearly helped in this task because is bigger than the screening mesh.

In relation to temperature, some delay was observed in laboratory trials due to the protective material, but at last, measured temperature was according to the theoretical one. Calibration trials in field were done to validate the measurement of the sensors by fixing 4 thermocouple probes around one compo-ball and connected to a 2 datalogger (Figure 5). All the system was inserted 40 cm inside the composting material. The values of the temperature of the 4 probes were 41.9 ºC, 41.9 ºC, 42.4 ºC, 43.9 ºC, which a mean of 42.3ºC, while the sensor of the compo-ball showed an output temperature of 42.7ºC. This result indicates in one hand that in a very short distance a variation in temperature can be observed and in other hand that the values showed by the compo-ball are very similar to those of the probes.

The trials for the measurement of moisture were done in composting material. For the validation, one sample was taken on the same point where the measurement was done, and was dried at 105ºC until constant weight. Figure 6 shows the relation with two points of calibration. As it can be seen, the results obtained by the sensors are quite similar to those of the gravimetric method.

The sensor nodes (the compoballs) include communication equipment to send measurements from their sensors to the outside world (pilehub). To reduce power consumption, this is achieved with a mesh network in which the balls send messages to each other until the date reaches the pilehub. Wireless communication depends very much on the conditions of the transmitting media and while the balls are able to communicate to up to few hundred meters in open air, this distance reduces to few meters inside the compost, which determines the number of sensors needed to scan the temperature and humidity of the particular compost pile. The pilehub consists of a receiving antenna on a pole and a head containing external communication and translates the internal data into a more suitable technology to use outside the pile. The gateway device receives the raw information from the pile-hubs and processes it to send it into a server.

COMPO-BALL brings a complete solution not seen so far in the composting industry. The sensors and the communication system are integrated into one simple encapsulation, able to resist the harsh composting environment, and facilitate measuring in multiple points inside the composting matrix without adding more than just one pole.

All this saves time for the operators in order to obtain results of temperature and moisture, which both are necessary to determining the need of oxygen and water.

The compoball system is designed to be installed in the compost pile while real time data of temperature and humidity are displayed in a web-based user interface to be used by the compost plant operator, helping to take management decisions.

By now, more trials are being conducted in real composting plants in order to check the validation of measurements and recovery of the balls.

More than this, this system could be used in other type of industries in the future in which the current system to measure moisture and temperature can be complicated. Depending on the industry, other sensors can be developed to suit industrial needs.

4. ACKNOWLEDGEMENTS

European Commission for the grant agreement 243625 (project *Novel on-line composting monitoring system*). Composting plants participating in the trials (Torrelles de Llobregat-Spain; Komtek-Denmark; Bitterfeld and Tautenheim-Germany; Den Ouden-The Netherlands). We are grateful to the RTD partners O. Casas, P. Ramos, E. Solà, C. Rovira and the Compoball-Project Coordinator M. R. Pinilla for their help in writing this manuscript.

FIGURE 1 **Compo-Ball and Pile-hub.**

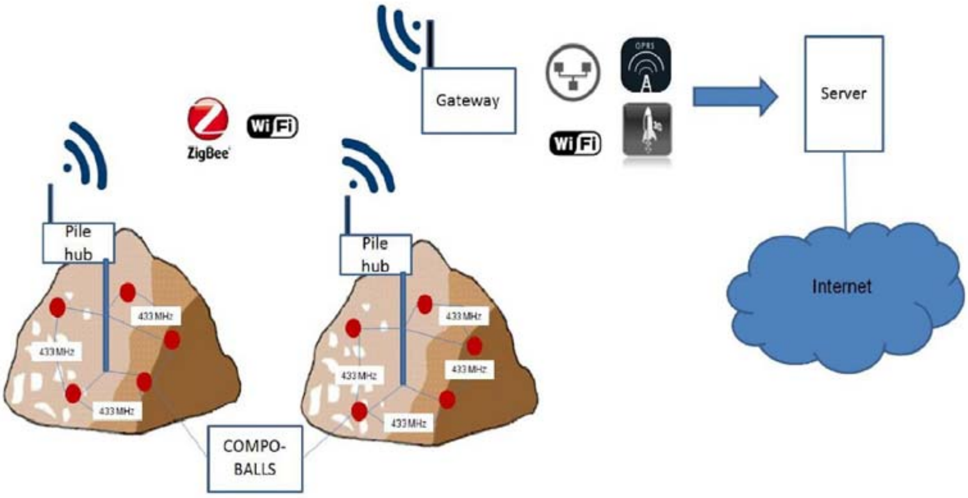
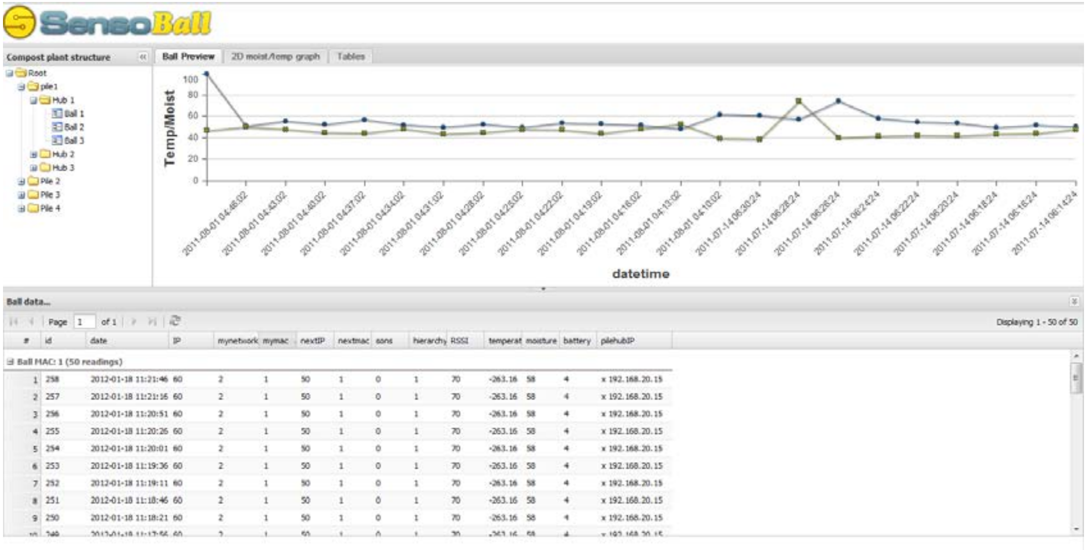


FIGURE 2 **The Compo-Ball system overview.**



Ball MAC: 1 (50 readings)

#	id	date	IP	mynetwork	mymac	nextIP	nextmac	sons	hierarchy	RSSI	temperat	moisture	battery	pilehubIP
1	258	2012-01-18 11:21:46	60	2	1	50	1	0	1	70	-263.16	58	4	x 192.168.20.15
2	257	2012-01-18 11:21:16	60	2	1	50	1	0	1	70	-263.16	58	4	x 192.168.20.15
3	256	2012-01-18 11:20:51	60	2	1	50	1	0	1	70	-263.16	58	4	x 192.168.20.15
4	255	2012-01-18 11:20:26	60	2	1	50	1	0	1	70	-263.16	58	4	x 192.168.20.15
5	254	2012-01-18 11:20:01	60	2	1	50	1	0	1	70	-263.16	58	4	x 192.168.20.15
6	253	2012-01-18 11:19:36	60	2	1	50	1	0	1	70	-263.16	58	4	x 192.168.20.15
7	252	2012-01-18 11:19:11	60	2	1	50	1	0	1	70	-263.16	58	4	x 192.168.20.15
8	251	2012-01-18 11:18:46	60	2	1	50	1	0	1	70	-263.16	58	4	x 192.168.20.15
9	250	2012-01-18 11:18:21	60	2	1	50	1	0	1	70	-263.16	58	4	x 192.168.20.15

FIGURE 3 **User interface.**

FIGURE 4 **Compo-balls after turning (foamy polyurethane on the left and rigid on the right).**

FIGURE 5 **Validation of temperature measurement.**

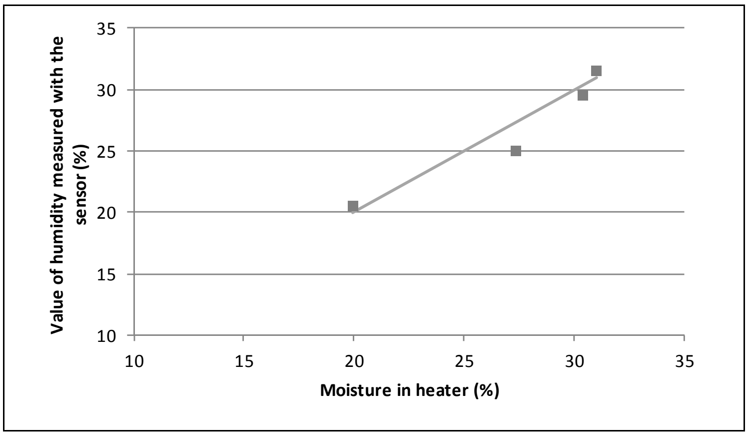


FIGURE 6 **Validation of moisture measurement with 2 points of calibration.**

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Session 7

(98) BIOPLASTIC PRODUCTION BY HYDROCARBONOCLASTIC BACTERIA

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EXECUTIVE SUMMARY

The polyhydroxyalkanoates (PHAs) are polymers that are produced from a variety of bacterial genera as *Pseudomonas*, *Flavobacterium*, *Bacillus*, *Alcanivorax* which are synthesized under conditions of shortage of certain nutrients and excess of carbon source (Anderson *et al.*, 1990). According to research, PHAs, including poly-3-hydroxibutirate (PHB), has been determined to have thermoplastic properties similar to petroleum products, as well by their nature are readily biodegradable. Therefore new trends involve the implementation of these polymers as possible replacements for conventional plastics, but it is necessary solve the problems that limit their large scale production. The aim of this research is isolate, and characterizes at least 3 hydrocarbonoclastic bacteria capable of producing PHB from hydrocarbon-contaminated sites, the results obtained can be used to design a bioreactor for bioremediation applications and utilization of residues form oil refining taking advantage of the production of raw material for biodegradable plastic production.

The bacteria strains were isolate at three stages of the plant for wastewater treatment property of PEMEX located in Salamanca, Guanajuato, México; pH values and oils and grease from the three stages were determined under the conditions described in the current Mexican regulations obtaining the most extreme values of pH (9.83±0.018) and oils (193±2.307 mg/mL) at the beginning of the process; the plate count of total viable aerobic bacteria ranged from $3.1\text{x}10^2$±59.85 to $1.30\text{x}10^6$±$1.68\text{x}10^5$ cfu/mL, the plate count in Bushnell-Haas medium (BHM) supplemented with a 5% v/v of an equimolar mix of octane and hexadecane, of viable hydrocarbonoclastic bacteria growth only in one of the sites.

21 presumptive hydrocarbonoclastic colonies were isolated from the stage with hydrocarbonoclastic bacteria growth which were cultivated again in BHM to the confirmation of hydrocarbonoclastic capability getting 8 bacteria, Gram stain was performed, obtaining 7 Gram-negative strains. To detect PHB-producing bacteria, we performed Black Sudan and Red Nile stains, were used to identify PHB under bright field and fluorescence microscopes respectively; the results for Black Sudan stain indicate the presence of PHB in 5 strains, the results for Red Nile Stain were inconclusive.

For phylogenetic analysis we aplificate a 1543-base pair fragment of gen coding for 16S rDNA from isolated strains. The amplification was performed in a thermocycler, using colony PCR method, PCR products were visualized in agarose gel electrophoresis. PCR products were purified and sequenced.

Edited forward and reverse complementary sequences where aligned using the Blast2sequences algorithm. The complete 16S DNAr sequences where compared to those deposited in the Genbank database using the megablast algorithm for the genera assignation. To identify the taxonomic position of each strain, we download the 16S DNAr sequences of the closest Type-strains to our bacteria sequences; a first alignment of the sequences was done using the ClustalX software and the phylogenetic tree was constructed using the Weighbor alghorithm using the tree builder form the ribosomal database project webpage. We use the 16S DNAr sequence of *Saccharococcus thermophilus* as out-group of the phylogenetic tree; due the highly similitude we propose that 2 strains are *B. muralis* and 1 strain is *B. pumilus*.

1. INTRODUCTION

1.1. Background

PHAs are linear polymers of (R)3-hydroxy acids in which it carboxyl group of a monomer, form an ester bond with the hydroxyl group of the following monomer. Examples of these polymers are PHB, the poli3-hydroxybutyrate-CO-3–hydroxyvalerate (Wang Yuhua, 2005), hidroxyhexonate (3HHX) and 3-Hidroxyeptonate (3HHp) (Chee *et al.*, 2010).

Under culture conditions that favor the accumulation of PHA, the bacteria used for production can be classified into two groups: the first group of bacteria requires essential nutrient limitations; some of the representative bacteria include: *C. Necator*, *Protomomas extorquens* and *Pseudomonas oleovorans*. The second group of bacteria does not require nutrient limitation for the synthesis of PHA and may accumulate during the exponential growth phase. Some of the bacteria included in this group are: *Alcaligenes latus* and *Azotobacter vinelandii* mutant strains and recombinant *E. coli* (Chee *et al.*, 2010).

The synthesis of PHAs is linked to the β-oxidation of lipids, for this reason can be synthesized from fatty acids, carbohydrates (Klinke, 1999) and hydrocarbons (Sabirova, 2010). There is about 150 different monomers that may be constituents of biopolymers, and this composition are defined the rheological properties of polymer.

1.2. Research objectives

The aims of this research includes de phylogenetic identification of at least 3 PHB- producing bacteria capable of degrading hydrocarbons isolated from de wastewater treatment system of RIAMA refinery

2. MATERIALS AND METHODS

2.1. Sampling and determination of physicochemical and bacterial parameters

For isolation of HCB, sampling was performed in the plant for wastewater treatment property of RIAMA refinery, located in Salamanca, Guanajuato, Mexico (20°34'18.06"N, 101°10'29.32"O). The company has a process for water contaminate with oils and other industrial wastes. The principal stages of the process for oily wastewater it is shown in the figure 1, at the same time, we specify the sampled sites. For the identifications of sampled stages, sites were named as follows: API oil water separator: OWS, oxidation ponds: OP, Biological Reactor: BR.

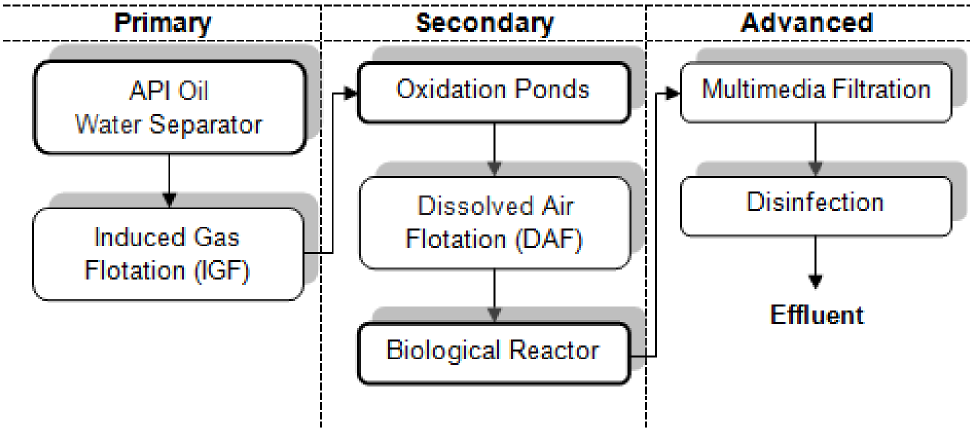

FIGURE 1 Main stages of the treatment process for oily wastewater of the refinery located in Salamanca, Guanajuato, Mexico. The sampling sites are the stages marked with thick line.

Sampling was carried out based on the specifications of the current Mexican standards also for the triplicate determinations of the next physicochemical parameters: temperature, pH, conductivity, turbidity, chemical oxygen demand (COD), total Kjeldahl nitrogen (TKN), nitrates, phosphorus, phenols and oils and grease; at the same time we obtained bacterial parameters by determination of total aerobic bacteria, total coliform bacteria and fecal coliform bacteria under the same specifications as mentioned.

2.2. Isolation of HCB and obtaining axenic cultures

For isolation of HCB, we inoculated 10 mL of the sampled wastewater in flasks with 100 mL of Bushnell-Haas medium (BHM) prepared according to the methodology of Patel and coworkers (2003) and supplemented with 5% v/v of an equimolar mix of octane (≥99%, Sigma-Aldrich) and hexadecane (ReagentPlus® 99%, Sigma-Aldrich), we incubated at 37 °C and 200 rpm during 48 h; after the incubation, serial dilutions in saline solution (NaCl, 3% w/v) were made and inoculated 200 μL aliquots of each them onto BHM agar plates by spread plate technique and incubated 48 h at 37 °C for colony observation and selection, for this last, we selected 21 different colonies (presumptive HCB) and the main factor considered was the repeatability at higher dilutions, as well as shape and color of colony.

To confirm the metabolism of the presumptive HCB, the 21 different colonies were growth onto BHM agar plates by streak-plate technique which were incubated at same conditions as before to evaluate growth, this method was performed twice; from the last plates were we obtained growth, we performed direct observation and Gram staining in the bright field microscope for observation of cellular morphology and axenicity of culture.

For not axenic cultures, they were grown in 5 mL of nutrient broth (NB) at 37 °C during 24 h; serial dilutions in saline solution were made and inoculated (100 μL) onto nutrient agar (NA) plates by spread plate technique and incubated 24 h at 37 °C for colony selection and microscope observation. The method was repeated until obtaining axenic cultures.

2.3. Microscopic visualization of lipid inclusions

To detect PHB-producing bacteria, the lipophilic stains, Sudan Black and Nile Red were used in axenic cultures grown in NB.

2.3.1. Nile Red stain

For this staining, axenic cultures were inoculated in tubes with 5 mL of NB supplemented with Nile Red at a concentration of 0.5 μg/mL-medium then, they were incubated at 37 °C and 160 rpm. The preparations were observed in the fluorescence microscope at 48 and 72 h of growth.

2.3.2. Sudan black stain

Axenic cultures were inoculated in 5 mL of NB and incubated at 37 °C and 160 rpm, after incubation time, heat-fixed samples were stained with Sudan Black solution (0.03 g of Sudan Black dye in 7.5 mL of ethanol 95% and 2.5 mL of water) for 10 minutes, dried and clarified with xylene drops, dried again for 1 minute and added safranin for 5 s and finally washed with distilled water. Observations were made in bright field microscope at 48 and 72 h of growth.

2.4. Phylogenetic identification

2.4.1. Colony PCR for amplification of fragment 8-1942 form bacterial 16S rDNA

Universal primers were used, forward primer (FP): 5’ AGA GTT TGA TCM TGG CTC AG 3’ and reverse primer (RP): 5’ AAG GAG GTG ATC CAN CCR CA 3’ (Giovannoni, 1991) to amplify a 1543-base pair fragment of gen coding for 16S rDNA from isolated strains. The enzymatic amplification was performed in a thermocycler, 50 μL of PCR reaction mix contained 36.5 μLs autoclaved ultrapure water, 5 μL of 10X reaction buffer (Invitrogen), 1 μL of 10mM dinucleoside triphosphate (dNTP) mixture, 1 μL of primer mix (10 μM each), 0.2 μL of Platinum® Taq DNA Polymerase (Ivitrogen) and 5 μL of cells previously rinsed in autoclaved distilled water and centrifuged; the samples were incubated 3 min at 94 °C for DNA denaturation, subsequently 30 cycles were performed each of them under the next conditions: 94 °C (1 min), 60 °C (1 min) and 72 °C (2 min); for an extension final cycle, we added to the protocol a temperature of 72 °C during 7 min (Polz *et al.*, 1997), also we added a positive control (DNA from *E. coli*) and a blank reaction with no DNA. The PCR products were visualized in 0.7% agarose gel using as molecular weight marker 1 kb ladder (Sigma-Aldrich). PCR products were purified and sent (30 μL ≈ 100 ng/μL) to Macrogen in South Korea for sequencing.

2.4.2. Phylogenetic analysis

Electropherograms from the 16S DNAr sequences where qualified and edited using the software Chromas Lite V 2.0, edited forward and reverse complementary sequences where aligned using the Blast2sequences algorithm (Tatsuova and Madden, 1999) available in the National Center for Biotechnology (NCBI) webpage (www.ncbi.nlm.nih.gov/).

The complete 16S DNAr sequences where compared to those deposited in the Genbank database using the megablast algorithm for the genera assignation.

To identify the taxonomic position of each strain, we download the 16S DNAr sequences of the closest Type-strains to our bacteria sequences, also we use the 16S DNAr from distant *Bacillus* species: *Bacillus muralis* (Heyrman *et al*., 2005), *B. simplex* (Priest *et al*., 1988), *B. coahuilensis* (Cerritos *et al*., 2008), *B. clausii* (Nielsen *et al*., 1995), *B. anthracis*, *B. pumilus*, and B. subtilis (Skerman *et al*., 1980).

A first alignment of the sequences was done using the ClustalX software and the phylogenetic tree was constructed using the Weighbor alghorithm (*Bruno et al*., 2000) using the tree builder form the ribosomal database project webpage (www.rdp.cme.msu.edu/). We use the 16S DNAr sequence of *Saccharococcus thermophilus* (Nystrand, 1984) as out-group of the phylogenetic tree.

2.5. Obtaining and quantification of biopolymer

Polymer quantification was performed by the method of Spekley Law. We inoculated 25 mL of NB, incubated for 24 hr at 37 °C and 160 rpm, subsequently used as inoculum for 250 mL of modified MYP medium, and were incubated for 42 h at same conditions as before. For recovery of the biomass, the tubes were centrifuged at 3000 rpm for 5 minutes. The lysis was performed in 20 mL of cells by adding a volume of commercial sodium hypochlorite (1:1) and incubated for 1 h at 37 °C. The PHB precipitate is washed with water, acetone and ethanol, and extraction is performed with chloroform, the pellets subsequently were filtered and allowed to evaporate excess chloroform, PHB becomes crotonic acid by sulfuric acid digestion at 100 °C for 10 min and quantified in the UV-VIS spectrophotometer at 235 nm comparing with a calibration curve of commercial PHB (Sigma-Aldrich).

3. RESULTS AND DISCUSSION

3.1. Bacterial and physicochemical determinations

Table 1 shows the average values for physicochemical measurements in each sampled site, as we describe previously, also we determine some average parameters of bacterial concentration, and they are shown in table 2.

TABLE 1 Mean values of phisicochemycal determinations in OWS, OP and BR sites.

Parameter (units)	OWS	OP	BR
Temperature (°C)	*27.46 ± 0.06*	*27.30 ± 0.00*	*23.03 ± 0.12*
pH	*7.02 ± 0.01*	*7.07 ± 0.01*	*9.83 ± 0.03*
Conductivity (µS/cm)	*1.31 ± 0.01*	*1.32 ± 0.00*	*3.47 ± 0.01*
DQO (mg/L)	*313.66 ± 8.08*	*373.66 ± 8.08*	*784.66 ± 6.51*
Turbidity (%ABS)	*0.667 ± 0.00*	*0.664 ± 0.00*	*0.75 ± 0.00*
TNK (mg/L)	*26.98 ± 1.41*	*27.51 ± 0.88*	*47.64 ± 7.89*
Nitrates (mg/L)	*0.19 ± 0.02*	*0.46 ± 0.04*	*0.81 ± 0.04*
Phosphorus (mg/L)	*0.11 ± 0.01*	*0.21 ± 0.04*	*0.33 ± 0.03*
Phenols (mg/L)	*0.06 ± 0.02*	*0.15 ± 0.05*	*0.98 ± 0.14*
Oil and grease (mg/L)	*27.20 ± 0.46*	*58.27 ± 1.13*	*193.00 ± 3.99*

TABLE 2 Mean values of bacterial determinations in OWS, OP and BR sites.

Site	Total aerobic bacteria (UFC/mL)	Total coliform bacteria (MPN/100 mL)	Fecal coliform bacteria (MPN/100 mL)
OWS	$3.1x10^2 \pm 59.85$	$\leq 2{,}400 \pm 0.00$	$< 2 \pm 0.00$
OP	$3.58x10^5 \pm 4.51x10^4$	$\leq 2{,}400 \pm 0.00$	$< 2 \pm 0.00$
BR	$1.30x10^6 \pm 1.68x10^5$	$\leq 2{,}400 \pm 0.00$	$< 2 \pm 0.00$

Isolation results were only favorable for BR site, which is the only one where growth was obtained in the BHM agar plates, *i.e.*, the 21 colonies selected where isolated from the stage denominate 'Biological reactor'. We can considerer that pH and oil and grease value affects the concentration of bacterial population, even the concentration of hydrocarbon can improve the presence of hydrocarbonoclastic bacteria, we can suggest in this case, that at high concentration (193 mg/L) we have a diminution of tolerance.

3.2. Cellular and colony morphology of hydrocarbonoclastics strains

From the 21 initial colonies we collected the data of colonial morphology; the table 3 shows the characteristics of the isolated strains, the last columns indicate if the strain was able to grow on BHM agar plates in a first (BHM-1) and a second (BHM-2) streak. At the end of the streak number 1 (BHM 1) we had 11 presumptive hydrocarbonoclastic strains, in the next streak (BHM-2) we only obtained 8 strains, which were maintained throughout the whole experiment.

TABLE 3 Characteristics of colonial morphology from the 21 initial strains isolated from BR.

Strain number	Strain name	Color	Form	Elevation	Margin	BHM-1	BHM-2
1	*A*	*Cream*	*Irregular*	*Umbonate*	*Undulate*	*Yes*	*Yes*
2	*B*	*Orange*	*Circular*	*Convex*	*Entire*	*Yes*	*Yes*
3	*C*	*Yellow*	*Circular*	*Convex*	*Entire*	*Yes*	*No*
4	*D*	*Cream*	*Circular*	*Flat*	*Entire*	*Yes*	*Yes*
5	*E*	*Cream*	*Circular*	*Flat*	*Entire*	*No*	-
6	*F*	*Cream*	*Circular*	*Convex*	*Entire*	*No*	-
7	*G*	*Black*	*Circular*	*Crateriform*	*Filiform*	*No*	-
8	*H*	*Cream*	*Circular*	*Crateriform*	*Entire*	*Yes*	*Yes*
9	*I*	*Cream*	*Circular*	*Flat*	*Entire*	*Yes*	*No*
10	*J*	*Cream*	*Irregular*	*Umbonate*	*Undulate*	*Yes*	*Yes*
11	J_1	*Cream*	*Circular*	*Convex*	*Entire*	*Yes*	*No*
12	*K*	*Cream*	*Circular*	*Convex*	*Entire*	*No*	-
13	*L*	*Cream*	*Circular*	*Flat*	*Entire*	*No*	-
14	*M*	*Cream*	*Circular*	*Convex*	*Entire*	*Yes*	*Yes*
15	*N*	*Cream*	*Irregular*	*Convex*	*Undulate*	*No*	-
16	*O*	*Black*	*Circular*	*Crateriform*	*Filiform*	*No*	-
17	*P*	*Black*	*Circular*	*Crateriform*	*Filiform*	*No*	-
18	*Q*	*Orange*	*Spindle*	*Convex*	*Entire*	*Yes*	*Yes*
19	*R*	*Cream*	*Circular*	*Convex*	*Entire*	*Yes*	*Yes*
20	*S*	*Cream*	*Circular*	*Flat*	*Entire*	*No*	-
21	*T*	*Cream*	*Circular*	*Convex*	*Entire*	*No*	-

The bright field microscopies of which are shown in Figure 2, correspond to the 8 axenic strains, where it was possible to determine its cellular morphology and their affinity to the crystal violet. B strain does not present bacterial characteristics, the colony-PCR can help us to confirm this information.

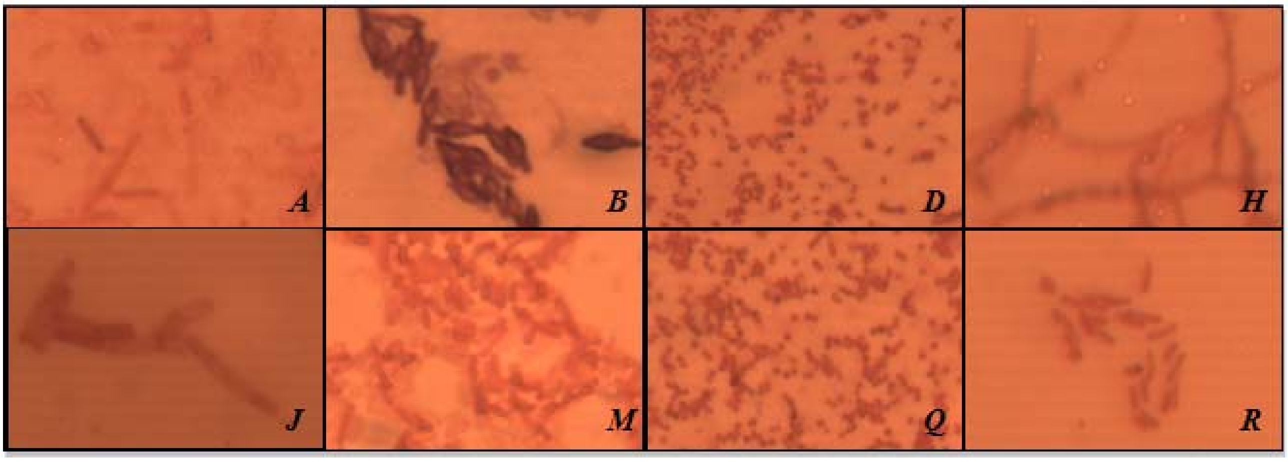

FIGURE 2 Bright field microscopies from axenic strains. Only the B strain shows affinity to crystal violet, also is the only that present a filamentous structure, the rest present rods (A, H, J and M) and cocci (D, Q) as cellular morphologies.

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3.3. PHB-producing strains

For the Black Sudan stain, we show de results in table 4, as we can see only 5 of the 8 strains present lipid inclusions in cytoplasm. The results for Red Nile stain were considered inconclusive.

TABLE 4 Results of the Black Sudan stain at 48 and 72 h of growth.

Time	A	B	D	H	J	M	Q	R
48 h	**No**	**Yes**	**No**	**Yes**	**Yes**	**No**	**Yes**	**No**
72 h	**No**	**Yes**	**No**	**Yes**	**Yes**	**Yes**	**Yes**	**No**

In case of M strain we can appreciate that de PHB production is after 72 h, this is considered normal, this type of microorganisms has a slower metabolism.

3.4. Colony PCR

For the 8 strains, we performed Colony PCR and the fragment amplification only were obtained for 3 strains: H, Q and J, we can say that B strain is not a bacteria, because the controls an blank test were obtained as expected.

3.5. Phylogenetic analysis

All tree sequences had enough quality to proceed to the phylogenetic analysis:

StrainQ, 1434 Base pairs length

StrainJ, 1424 Base pairs length

StrainH, 1458 Base pairs length.

The 16S rRNA gene from the Strain Q and strain J, seems to be related with the *B. muralis* type-strain, and strain H to *B. pumilus* type strain (Figure 3), due the highly similitude we propose that the identity of strain Q and J are *B. muralis* and strain H is *B. pumilus*.

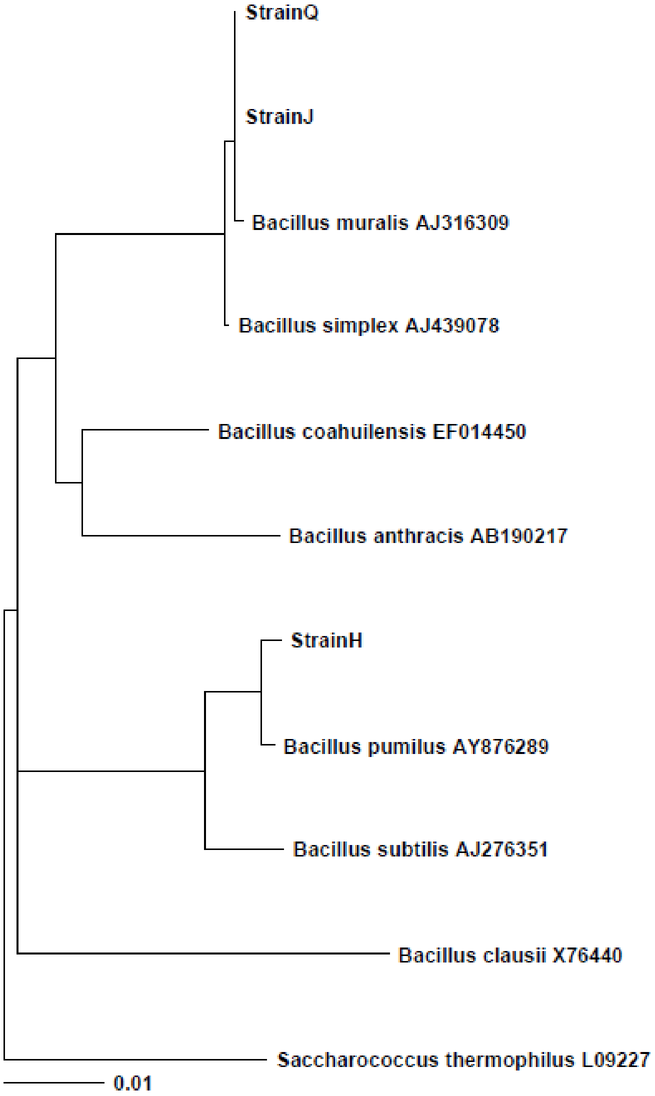

FIGURE 3 Phylogenetic tree based on the 16S rRNA sequences of bacteria isolated from the RIAMA´s wastewater treatment complex, the 16S DNAr sequence of *Saccharococcus thermophilus*, was used as the cluster root. The bar scale represents the average number of substitutions per site.

3.6. PHB Quantification

The values obtained for the quantification of PHB were for B strain: 1.33 ± 0.2 mg/L, H strain: 0.65 ± 0.1 mg/L, J strain: 0.85 ± 0.2 mg/L, M strain 1.60 ± 0.1 mg/L and Q strain: 0.71 ± 0.5 mg/L. It is important indicate that the method has a lot of losses.

4. CONCLUSIONS

Five hydrocarbon-degrading PHB producers strains were determinated, being two Bacillus genera bacteria the major producers, nonetheless the production was inferior to the one reported for bacteria of the same genera, therefore it is important to determine the optimal conditions for a major productivity

5. ACKNOWLEDGEMENTS

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(113) BIODEGRADATION OF BIOPLASTICS AND NAURAL FIBERS DURING, ANAEROBIC DIGESTION AND IN SOIL

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EXECUTIVE SUMMARY

Plastics are increasingly causing pollution problems in natural environments due to their recalcitrant nature. Various new materials have recently begun to be marketed that claim to biodegrade or compost during waste treatment. These materials include conventional plastics amended with additives that are meant to confer biodegradability or compostability as well as plastics made from biopolymers, and natural fiber based materials. Different industries are particularly interested in these materials as alternatives to conventional plastics that are neither compostable nor biodegradable. In this study, the amount of carbon in various commercially available bioplastics and natural fibers converted to carbon dioxide (CO_2) and methane was determined during soil incubation, composting and anaerobic digestion (AD). The materials included biopolymers made from corn starch, polylactic acid and polyhydroxyalkanoate, natural fibers such as coconut coir, paper, dry manure and asphalt and soy-wax coated paper as well as polypropylene (PP) and other conventional plastics amended with additives claiming to confer biodegradability. Controls included cellulose paper, pots made from PP. During the 550 day soil incubation experiment, all materials degraded more slowly than the cellulose positive control. However the biopolymers and natural fiber materials showed substantial degradation during this period. No significant degradation was observed for plastics amended with additives. During anaerobic digestion for 50 days, 75% of the cellulose paper, 20-25% of the biopolymers and less than 2% of the additive containing PP and PETE had been converted to biogas. After 110 days of composting, more than 70% of cellulose paper, 0.6% of an additive amended PP, 51% of a corn starch based bioplastic and 12% of a soy-wax coated paper was converted to CO_2. In conclusion, although certain biopolymers and natural fibers appear to biodegrade to an appreciable extent, none meet ASTM standards for biodegradability or compostability. Conventional plastics containing additives did not biodegrade differently than non-additive containing plastics.

Keywords: bioplastics, composting, soil, anaerobic digestion, biodegradation.

1 INTRODUCTION

In 2010, the United States generated almost 14 million tons of plastics as containers and packaging and almost 7 million tons of nondurable goods such as example plates and cups (EPA, 2010). Only 8 percent of the total plastic waste generated was recovered for recycling. A vast majority of the plastics that are commercially available are made from non-renewable petroleum-based resources and are essentially not biodegradable. Thus, until improvements in recycling programs are made, tons of plastics will accumulate in landfills, minimizing the overall landfill (Hopewell et al., 2009). These materials are also increasingly causing pollution problems in natural environments due to their recalcitrant nature and due to energy use and greenhouse gas emissions during their life cycle (Kyrikou & Briassoulis, 2007). Roadsides, parks, beaches and natural areas are inundated with plastic trash.

One reason for limited plastics recycling is that it is often commingled with organic wastes (food scraps, wet paper, yard trimmings, soil and liquids), making it difficult and impractical to recycle without expensive cleaning and sanitizing procedures. Food scraps and wet, non-recyclable paper comprise an additional 50 million tons of municipal solid waste in the U.S. Composting these mixed organics could potentially recover a sizable portion of the waste stream, dramatically increasing community organics recycling and natural resource sustainability. Biodegradable plastics can replace the non-degradable plastics in these waste streams, allowing mixed wastes to be composted and making municipal composting a significant tool to divert large amounts of otherwise unrecoverable waste.

Various plastic and natural materials have recently begun to be marketed that claim to biodegrade or compost during waste treatment (Song et al., 2009). These materials include conventional plastics amended with additives that are meant to confer biodegradability or compostability as well as plastics made from biopolymers, and natural fiber based materials. However, there is limited information about the extent or rate of biodegradation of these materials.

There is a need to develop novel materials that are designed to fully or partially degrade under different conditions, including soil, composting and anaerobic digestion (AD); the latter mimics landfilling. Advantages of biodegradable materials include the increase in the economic feasibility of the landfill or anaerobic digestors due to methane gas collection for bioenergy production and volume reduction of the waste due to biodegradation during the active life of the of the system (International, 2011). One of the most pressing questions is whether the “biodegradable” plastics and natural fibers available in the market truly degrade. There is a lack of scientific information supporting the claims made by “novel” materials developers.

Whereas some research has focused on the degradation of specific biopolymers and natural fibers that are used to produce bioplastics (Hanley et al., 2000; Ishigaki et al., 1999; Woolnough et al., 2010) there is lack of information on how the “novel” materials that are available in the market will degrade after incorporation into products or under conditions found during waste management or environmental release. These degradation rates are particularly important to studies quantifying the impact of specific products and processes on different environments. The overall objective of this study was to determine the biodegradability of a wide range of materials and additives claiming to be biodegradable or to confer biodegradability to conventional plastics.

2 METHODOLOGY

Laboratory-scale experiments were conducted to study the biodegradability of plastics under soil incubation, composting, AD conditions. The percent biodegradation was calculated by measuring the average carbon dioxide (CO_2) evolved from each treatment, subtracting this from the average CO_2 evolved from negative controls, and dividing this by the total amount of sample carbon added to each treatment. All materials were cut into 1 cm squares and tested based on ASTM international standard test methods.

2.1 Experimental procedure

The compostability of bioplastics samples was determined based on ASTM D5338-98(2003) (International, 2003b). The test determines the percent of carbon in the plastic material that was converted to CO_2 during the course of a composting experiment that mimics the temperature and aeration conditions during full scale commercial composting. Each bioplastic sample (80 g dry) was mixed with a compost inoculum (350 g dry) and the moisture content of the mixture was adjusted to 60% using deionized water. On a wet-weight basis, each vessel contained approximately 1100 g of material.

The compost inoculum was obtained from the Ohio Research and Development Center (OARDC) composting site. This is a full scale windrow composting facility featuring a concrete composting surface and a built in aeration system. The compost contained a mixture of dairy manure and hardwood sawdust (Michel et al., 2004). Compost was then screened to less than 10 mm and large inert items were discarded. Compost was amended with ammonium phosphate to give an overall C:N of 20:1 (by weight), including carbon contained in the test material. Composting was performed using a bench scale reactor system featuring 18, 4-liter capacity reactors placed in an incubator set at 55°C. To maintain aerobic conditions, regulated humidified air at a -40°C dew point entered the vessels at a rate of 100 ml/min (Grewal et al., 2006). Air was exhausted at the top of the vessels through tubes attached to an air outlet and bubbled through flasks in a separate water bath set at 9°C to condense moisture from the off-gas. The off-gas was then analysed for percent CO_2 (Vaisala model GMT 220, range 0 to 20%). Data was automatically recorded on a Campbell Scientific model 23XL data logger for each vessel every hour. Each vessel was also equipped with a K-type thermocouple to measure temperature in the mix near the middle of the compost, and data will be recorded automatically every 12 min. The experiments were conducted over a period of 110 days.

The anaerobic digestion study determined the degree and rate of conversion of plastic materials to CO_2 and CH_4 during incubation under anaerobic conditions based on ASTM D5511-02 (International, 2002). For this study test materials were exposed to an active methanogenic inoculum derived from a full-scale anaerobic digester treating municipal sewage sludge. The test was designed to measure the percent conversion of carbon in the test materials to biogas, a combination of CH_4, CO_2 and trace gases. These conditions resemble those found in high-solids anaerobic digestion systems and in biologically active landfills.

The anaerobic digestion assays were performed using laboratory-scale batch reactors, without solids recycling. Temperatures were maintained at mesophilic (36 ± 1) conditions by means of incubators. The volume of gas produced, and biogas composition were quantified on a daily basis (Gomez et al., 2011). All the reactors were inoculated with methanogenically active sludge obtained from the Akron Waste Water treatment plants full-scale (3000 m3) anaerobic digester. Once inoculated, reactors were sealed to initiate a 7 day start-up phase.

The start-up process involved adapting the digester biomass to the operational conditions of the laboratory-scale reactors. After proper functioning of the reactors was confirmed, sample materials (20 g dry) were added. The experiments were conducted over a period of 50 days.

For the soil incubation experiment the degree and rate of aerobic biodegradability of a plastic material in the environment determines the extent to which and time period over which the plastic may be mineralized according to ASTM D5988-03 (International, 2003a) test method. Agricultural soils were obtained from experimental units at the OARDC. The soil media was a laboratory mixture of 43% certified organic top soil, 43% of no-till farm soil and 14% sand. Soil was sieved to less than 2-mm particle size, plant materials, stones, and other inert materials were removed. The soil was then analysed for moisture holding capacity (MHC) according to ASTM test method D2980. For this experiment, 300 grams of the soil media (dw) were placed in the bottom of the vessels (figure 1). Soil was amended with ammonium phosphate to give a C:N of 20:1 (by weight), including carbon contained in the test specimen. Distilled water was added to bring the moisture content of the mixture to 60% of the MHC. The test samples containing 1000 mg of carbon (~ 2 of sample) were then mixed thoroughly with the soil. A solution containing 20 ml of potassium hydroxide (KOH) 0.5N was placed in each vessel. The experiments were conducted over a period of 550 days.

2.2 Chemical parameters

A variety of analytical measurements were used to monitor the biodegradation of sample materials during composting, AD and in soil experiments. These included CO_2 evolution, total carbon content of the media and plastics, total nitrogen and biogas production (CO_2 and CH_4).

The total carbon and nitrogen contents of the sample materials were measured according to methods developed by the US Composting Council (Composting Council et al., 2002) by the Service Testing and Research laboratory (STAR) at The OARDC in Wooster, OH. A VarioMax Carbon-Nitrogen combustion analyser (Elementar Americas Inc., Mount Laurel, NJ, U.S.) was used to carry out the analyses based on combustion followed by CO_2. Analyses for the complete suite of elements was performed in the same location by ICP analysis after microwave digestion (EPA Method 3051).

The off-gas from the composting experiment was analysed for percent CO_2 using infrared spectroscopy (Vaisala model GMT 220, range 0 to 20%). A polaragraphic oxygen system was used to measure percent oxygen (O_2) (Mine Safety Appliances model “ULTIMA,” range 0 to 25%). Data will be automatically recorded on a Campbell Scientific model 23XL data logger for each vessel every hour. Each vessel was equipped with a K-type thermocouple to measure temperatures in the mixes near the middle of the compost, and data was recorded automatically every 12 min.

Volumetric production of biogas from the AD experiments was measured by collecting the off-gas from the reactors in 0.5 L tedlar bags. The gas volume evolved during the experiments was measured by pumping the gas through a drum type wet-test volumetric gas meter (RITTER®, Bochum, Germany), volumes were converted to standard temperature and pressure (STP) conditions based on the building temperature and pressure at the time of the measurement. The methane content in the biogas was determined using a gas chromatograph (GC) HP 6890 (Agilent Technologies, Santa Clara, CA, U.S.) series equipped with a ValcoPlot VP-Alumina (Valco Instruments Co. Inc., Ontario, Canada) column and a thermal conductivity detector (TCD) operated isothermally at 40°C. Helium was the carrier gas at a flow rate of 20 ml/min.

Carbon dioxide produced in each vessel containing soil reacted with KOH to form bicarbonate (KHCO3). The amount of CO_2 produced was determined by titrating the remaining KOH solution with 0.25N hydrochloric acid to a phenolphthalein end-point. Vessels were incubated at room temperature. KOH traps were removed and titrated before their capacity was exceeded. In addition O_2 content in the vessel was ensured not to fall below 18%. The time to replace a trap varied with test materials. Moreover, a frequency of every 3 to 4 days for the first 2 to 3 weeks and every 1 to 3 thereafter was used. At the time of removal of the traps, the vessel was allowed to sit open for a maximum of 15 minutes to allow for fresh air to get into the system. In addition, distilled water was added accordingly to maintain adequate moisture content. The test was terminated when the CO_2 evolution reached a plateau assuming that all accessible carbon was oxidized.

2.3 2.3 Data analysis

Three independent replicates will be used for each treatment. Analysis of variance (ANOVA) will be calculated for the average cumulative percent conversion for each of the tests. Comparisons for all pairs of bioconversion means will be performed using Tukey-Kramer HSD method. All conclusions will be based on a significant difference level of α =

0.05. The statistical analyses will be performed using JMP statistical program version 9 (SAS Institute Inc., SAS Campus Drive, NC, U.S.).

3 RESULTS AND DISCUSSION

Three materials were tested under composting conditions; corn starch biopolymer Ecotainer®, soy wax coated paper, and polypropylene (PP) with 2% ECM® additive. The positive control used was cellulose paper. The initial rate of degradation was similar for the positive control and the Ecotainer®, samples (Fig. 1). The Ecotainer material continued to biodegrade steadily until the end of the experiment. Little conversion was observed for the soy wax coated paper or the PP with additive. Soy wax coated paper samples reached a maximum degradation after 15 to 20 days of the experiment. The average final carbon conversion for the coated paper and Ecotainer samples was 12.4±0.2 and 51.3±0.2% respectively. Over 70% biodegradation was observed for the positive control. Plastic with ECM additive did not show significant degradation over the period of study (110 days).

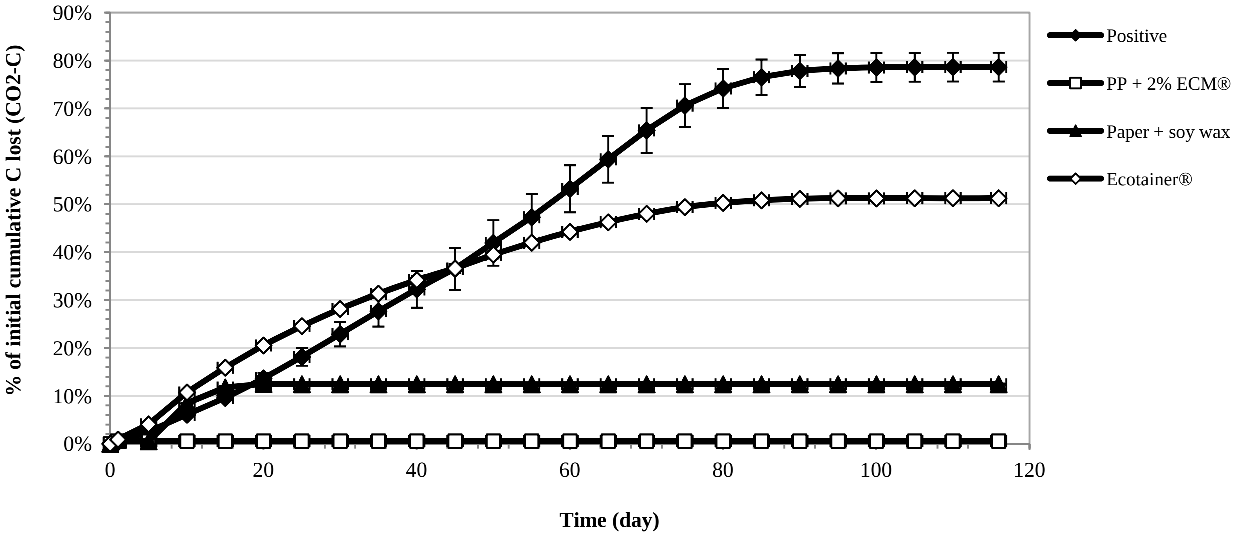

Figure 1. Cumulative carbon loss (CO_2-C) during composting as the percentage of initial carbon (±SD) over time. For some data points SD bars are smaller than markers.

Eleven different materials were tested in soil incubation experiments. The positive control used was cellulose paper. Bioconversion occurred at faster rate initially for most of the materials tested (Fig. 2). PHA and Ecobras®, showed the greatest rate of degradation among the materials tested. This was followed by Cow Pot®, paper + asphalt, and Ecotainer. Materials derived from natural fibers (Coconut coir, Rice hull and Peat Pot®) showed a slower, but significant rate of degradation. No degradation was observed for PP and PETE with additives over the entire period of study. After 550 days, the PHA and Ecobras® sample showed 56.4±0.7 and 53.0±0.8% carbon conversion to CO_2, respectively. Cumulative degradation of the materials made from paper and natural fibers were between 5 and 25%.

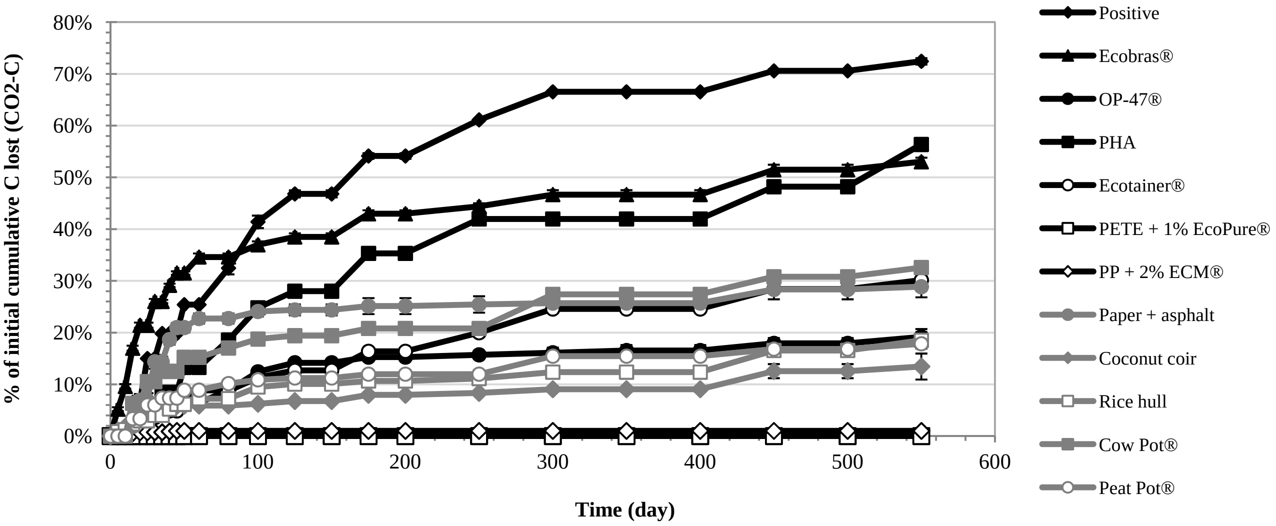

Figure 2. Cumulative carbon loss (CO2-C) during soil incubation as the percentage of initial carbon (±SD) over time. For some data points SD bars are smaller than markers.

Four materials were tested under anaerobic digestion conditions (Fig. 3). The positive control used was cellulose paper. The initial rate of degradation was highest for Ecotainer® and Ecobras® samples. The rate of degradation was steady through day 30 for these two samples and appeared to slow somewhat after this period. Final values for Ecotainer® and Ecobras® were of 26.4±8.0 and 20.2±6.7% over a 50 day period in an anaerobic environment, respectively. For treatments containing PP and PETE amended with additives, no significant degradation was observed over the period of study. Statistical analyses revealed that differences between Ecobras and Ecotainer were not significant.

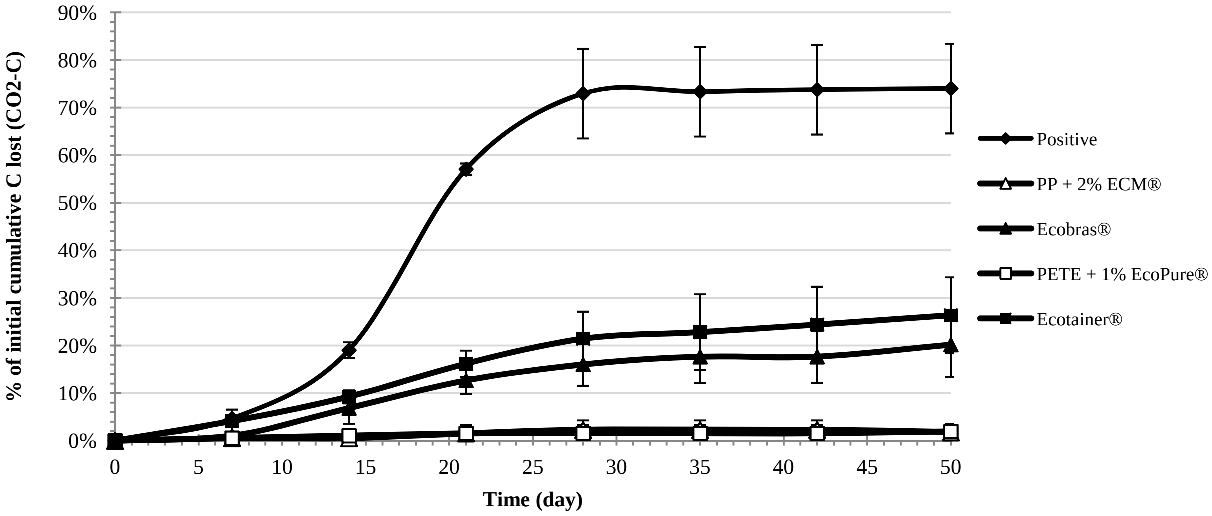

Figure 3. Cumulative carbon loss (CO_2-C) during anaerobic digestions as the percentage of initial carbon (±SD) over time. For some data points SD bars are smaller than markers.

4 CONCLUSION

Although certain biopolymers and natural fibers appear to biodegrade to an appreciable extent during composting, anaerobic digestion and soil incubation, none degrade at the same rate or to the same extent as positive controls. Therefore, none met ASTM standards for biodegradability or compostability. Conventional plastics did not biodegrade differently than non-additive containing plastics and neither biodegraded to an appreciable extent.

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(227) BIOPLASTICS AND COMPOST: THE ITALIAN APPROACH

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EXECUTIVE SUMMARY

Recent times have seen the introduction to the market of products and packaging made of bioplastic materials, such as mulch films, catering products, packaging and waste bags. Bioplastics are a family of materials that are bio-based, biodegradable, or both (conventional plastics do not have these qualities). The annual global production capacity of bio-based plastics is estimated at around 1.000.000 tons, while in 2010 the European market consumption of bioplastics in all application areas was estimated at 100.000 – 150.000 tonnes (European Bioplastic , may 2011). These particular products, when recovered through composting, break down completely allowing the production high-quality compost.

The presence of conventional plastics in the organic fraction of waste collection has always been a problem for the composting sector. Plastic is removed through refining interventions (screening, sorting) and sent for disposal, both of which are operations that strongly influence the cost of treatment of organic waste. The aim of exploiting materials made from bioplastics is the idea behind the new label on Compostability "Compostable CIC", introduced by the Italian Composting Association (CIC) in 2006. The trademark identifies and certifies all those artefacts that decompose naturally in the composting process and that have well-defined characteristics such as biodegradability and disintegrability, all requirements of specific European standards (UNI EN 13432:2002) and Italian law (art. 182 of D.Lgs 152/2006). It is also important to note that in Italy there is a ban on the marketing of non-biodegradable bags (traditional plastic bags) from January 2011.

Compostable CIC wants to be a quality instrument in the hands of all stakeholders who share the same sustainable choice: the producers of products that use biodegradable and renewable organic raw materials, final consumers who prefer the consumption of recyclable materials, products that provide a higher quality of compost and those able to reduce the costs of recovery.

1. INTRODUCTION

1.1 Compostability: the “CIC- compostabile” the labeling program

In 2006, the Italian Composting Association (CIC) introduced in Italy the quality certification programme for biodegradable materials and in particular, compostable products. The compostable certification programme ensures the material’s ability to turn into compost during the industrial composting process. The trademark "CIC Compostable" logo is issued after audits and tests performed by CIC in collaboration with Certiquality, the market leader in certification standards. The CIC compostable certification programme meets the European regulation EN:13432 on compostable packaging. As a result, more than 50 products have successfully passed the certification program (cfr. www.compostabile.com) and more than 30 firms can use the CIC innovative trademark logo “CIC- compostabile” to label authentic compostable items. The “CIC- compostabile logo” was designed to help citizens visually identify products that successfully passed the operational, technical and scientific standards on compostable materials. In addition, only certified compostable products are accepted into composting facilites.

The reasons that led CIC to establish the certification programme on compostable products are briefly listed below:

- The bioplastics world market is evolving rapidly;
- The quality of incoming biowaste into composting facilities strictly depends upon non-compostable materials (NCM) content;
- Impurity content increases biowaste treatment costs
- Biowaste can became an organic fertilizer only if impurities are really low;
- Promoting only authentic compostable bioplatics that meet the European standards

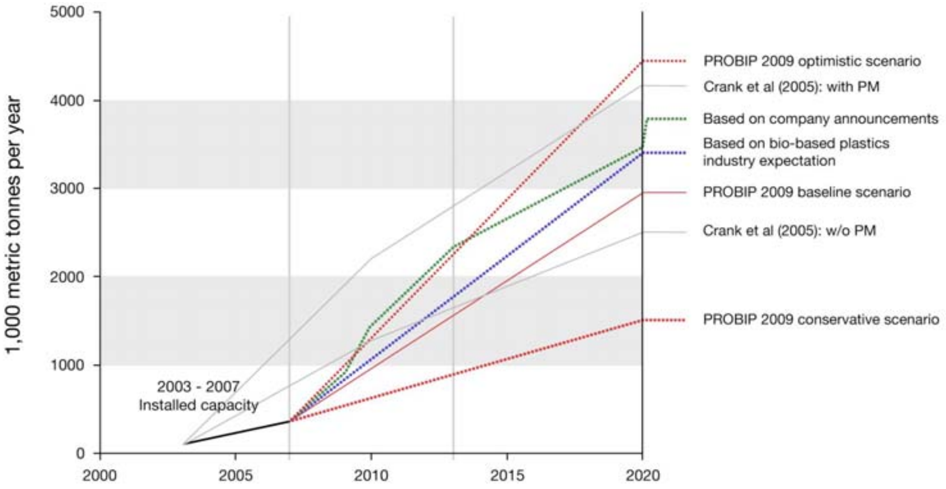
1.2 Bioplastic market worldwide

Despite the fact that the current market relegates bioplastics into a small niche, biopolymers stimulate stronger interest in the packaging industry. There are also some interesting developments in compostable semi-durable goods and medical applications. Bioplastics have been on the market for more than a decade and they are continually growing in film, packaging and consumer durable fields. Nowadays, bioplastics are used in manufacturing mobile phones, cars, toys, medical equipment and textiles. Conventional plastic polymers such as polyethylene (PE), polypropylene (PP), PVC and PET, but also high-performance polymers such as polyamide or polyester have been totally or partially replaced by equivalent bioplastics. Biopolymers are made by renewable raw materials such as sugars and starches, which they can obtain from dedicated crops or from by-products.

In 2009, the “PRO-BIO Study 2009” commissioned by both EB (European Bioplastics) and the EPNOE (European Polysaccharide Network of Excellence), communicated that up to 90% of the current global production of traditional plastic polymers can be converted from oil consumption into renewable raw materials. However, EPNOE also state that because of low oil prices and the high production costs of biopolymers, in the short term, bioplastics are incapable of completely replacing oil-based polymers. On the contrary, CIC suggests that biopolymer production will grow from 360000 tons in 2007 to 2.3 million tons in 2013. This growth is equivalent to an annual increase of 37% a year.[1] Despite the economic and financial crises of 2008 and 2009, the bioplastics sector is conquering new market segments that in the past were held by conventional plastics. This increase in the market is an encouraging sign for the biopolymer industry.

In order to define the potential market growth for bioplastics, the 2009 PROBIO study was performed by analyzing three different scenarios: the baseline scenario, the optimistic evolution and a conservative development. The results of those three market scenarios were then compared to a previous study completed in 2005. The conclusion was that substantial technological progress had been accomplished by the bioplastic industry during the last past five years. Material innovation, the development of new products, as well as crude oil depletion, are all important elements that are promoting new applications of biopolymers made from renewable raw materials.

Figure 1: Forecast on global bioplastics production (source: PROBIO 2009 - European Bioplastics, European Polysaccharide Network of Excellence EPNOE)



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[1] European Bioplastics Association, www.european-bioplastics.org, 2009

The latest market trend for the biopolymers manufacturing sector is to emphasize that since bioplastics are made from renewable resources they reduce global fossil fuel consumption as well as CO2 emissions. On the other hand the plastic recycling sector is concerned about the contamination risks that bioplastics may present if, by mistake, they are collected during traditional plastic post-consumer recycling collections. In fact, even small percentage of bioplastic such as PLA (polylactide) into post-consumer plastic collection, may become a serious contamination problem for plastic recycling sector. However, features such as biodegradability and disintegrability which embrace the "compostable" concept present fundamental advantages. For example, compostability is a key feature for biowaste container collection, food containers, and various other post-consumer packaging that, together with biowaste, can be collected during source separated organic collections. The quality and purity of biowaste (low NCM) represents the key factor for artefacts recoverable through the composting process since the waste management system is strongly affected by the problems related to the incorrect planning, implementation and management of the separate collection of organic waste.

1.3 Quality assurance analysis on biowaste

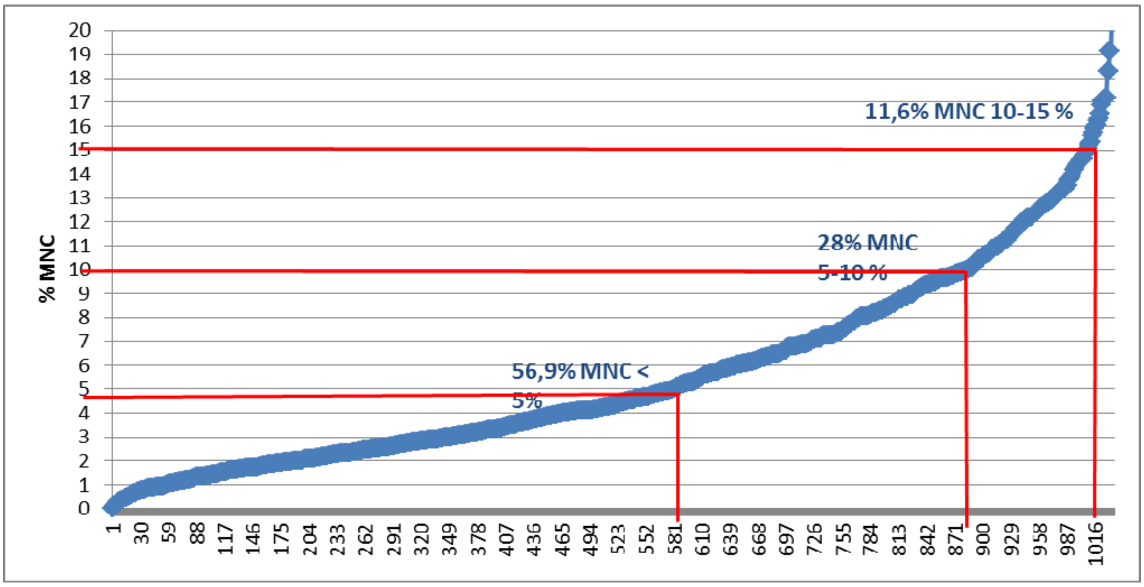
The Italian Composting Association (CIC) has in important role in monitoring and protecting the Italian organic waste sector. In particular, CIC performs hundreds of quality assurance analyses on incoming organic waste across the Italian peninsula every year. The scientific method used to perform analysis on source separated organic waste begins by sampling a representative quantity of biowaste. The sample is then analyzed and divided into six different categories:

- Organic materials (food waste, green waste, paper and cardboard, animal and vegetable fibres, etc.)
- Plastics (plastic films, packaging, rigid plastics, etc.).
- Glass
- Metals (iron, aluminium, etc.)
- Inert materials (ceramics, pottery, stones, bricks, etc.)
- Other (synthetic fabrics, diapers, hazardous and bulky waste, tetrapack, batteries, etc.)

The “organic materials" category represents the "compostable materials” (CM), while the remaining categories are categorized as "non-compostable materials" (NCM). The six different product fractions are then separately weighed and compared to the total weight of the original sample. The main goal is scientifically declare the quality of the incoming organic waste entering composting facilities. Briefly, lower NCM content recorded during the sample analysis would mean better quality for the incoming biowaste.

Figure 2 show the non-compostable material (NCM) content recorded during each quality assurance analysis on source separated organic waste performed of 1016 Italian municipalities. Cases in which NCM content represented more than 20% of the total weight of organic waste are not reported (2 % of the actual data). The percentage of NCM in the total biowaste analyzed has been classified into 3 different classes (<5%, between 5% and 10%, and between 10 and 15% of NCM). 57% of the total analyses presented less than 5% of NC,. while 28% had a NCM content between 5% and 10%, and only 11% of had a NCM content between 10% and 15%.

Figure 2: % NCM content on incoming biowaste between 2009 and 2010 recorded on 1016 municipalities;



1.4 Non-compostable materials (NCM) content increases biowaste treatment costs

Presently, the large quantities of plastic placed on the market is leading to an increase of post-consumer waste. Since plastic materials are resistant to biodegradation, without a dedicated recycling initiative they can become a dangerous environmental threat. The presence of post-consumer plastics by error or negligence into source separated organic waste represents a problem for composting facilities. When machines separate post-consumer plastics, they also remove precious organic waste that is trapped in the undesired materials from composting plants.

It has been estimated (Centemero, 2011) that for each percentage point of NCM content entering a composting facility imposes a drag effect coefficient between 1 to 3. Which means that, for each kg of initial NCM, the composting facility will need to dispose of a further 1 to 3 kg of unwanted materials.

At a national level (see Table 1) the Italian composting industry treated about 4.5 MT of organic waste in 2010 resulting in the production of approximately 1.3 MT of quality compost. . On the other hand, unwanted materials represented about 20.9% which are approximately 730,000 T of non-compostable materials. Plastics are equal to 30-40% which represents 220-300,000 T of plastics that the composting Italian sector need to further dispose. As a result, composting plants have to sustain a cost burden to dispose of plastic waste. The cost burden leads to a tremendous impact on the economy of the entire Italian composting sector.

Table 1: Italian composting sector in 2010
Source: Rapporto Rifiuti ISPRA,2011 - Centemero, 2011

Organic waste treated	**4.5 MT**
Food waste	2.2 MT
Green waste	1.6 MT
sludge	0.4 MT
other	0.3 MT
Quality Compost produced	**1,300,000 T**
Unwanted materials: NCM (estimated)	**120,000 T**
Unwanted materials: plastic bags (estimated)	**35-40,000 T**

In the worst scenario if the organic waste is highly contaminated with post-consumer plastics it could compromise the entire organic treatment process. In fact, by the Italian law (D.lgs. 75/2010) if the plastics content is higher than 0.5% the compost produced cannot be sold. As a result, materials such as polyethylene and polystyrene may compromise the ability of the composting sector to reach quality compost fertilizer parameters set by D. lgs. n.75/10.

Further disposal of those post-consumer plastic packaging strongly effects the Italian composting sector. In particular the disposal cost for 40,000 T of plastic bags is approximately € 4.8 M. In addition to that, we need to add the cost of the disposal of other plastics (approx. € 3.6 M) and extraction costs (pre-treatment, sieving, refining). In order to eliminate plastics from organic waste, the final estimated total cost is 15 million € / year. however, this number doesn't take into account the missing production of biogas during the anaerobic digestion process (plastics does not produce biogas!), the loss of organic matter which could be made into compost and the economic and environmental costs associated with highly contaminated compost.

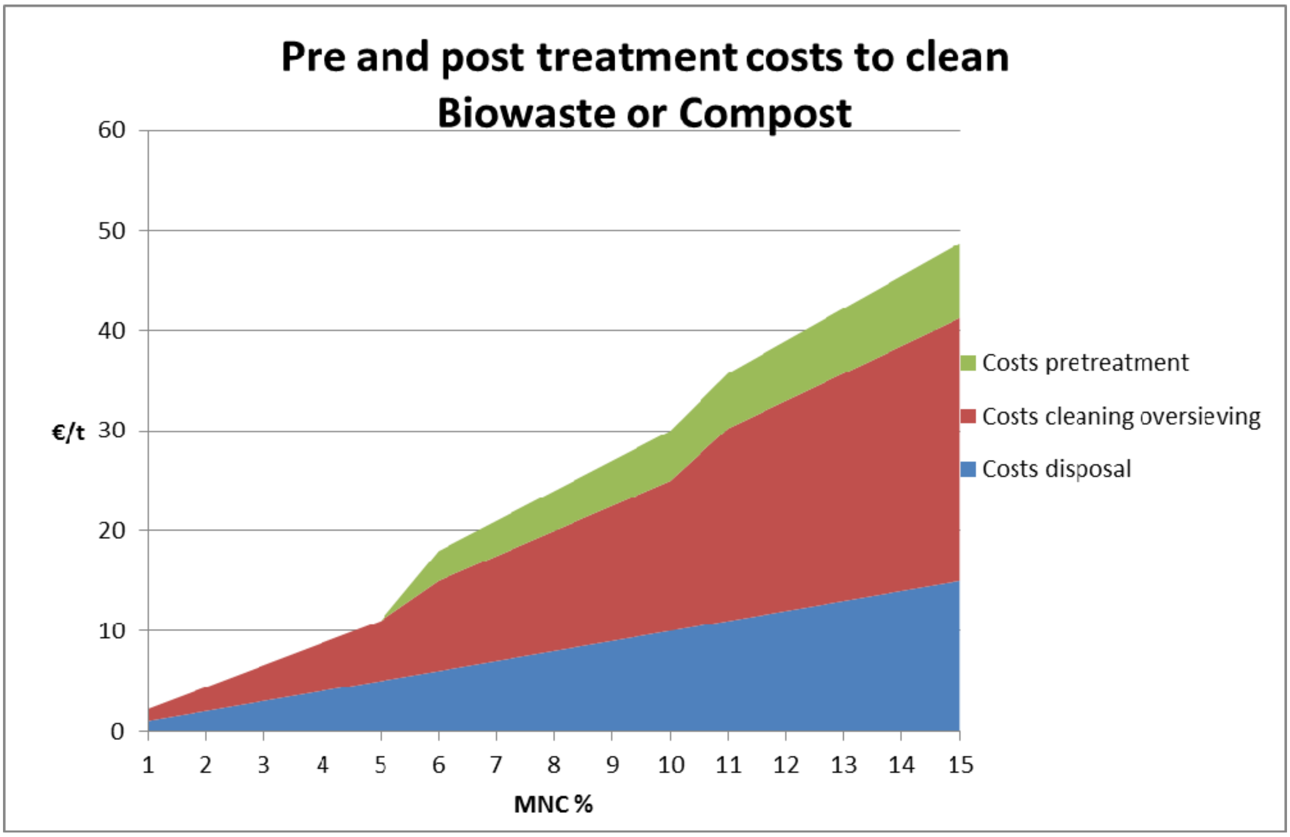


Figure 3: total amount of costs to clean biowaste (pre teatment) or compost (post treatment).

As shown in Figure 3, treatment costs rise exponentially when impurities are above 5%. On the other hand, when NCM is above 10%, the drag effect coefficient becomes equal to 3. Thus, above NCM=10%, treatment costs rises up to €3 per tonne of impurities for each further percentage point of NCM.

1.5 Compost is an organic fertilizer with strict limits on impurities

Post- consumer plastic packaging such as polyethylene, polystyrene, undermines the ability to produce quality compost. In fact, the Italian legislative decree n. 75/2010 that regulates the fertilizer market sets maximum impurity limit in quality composts. Quality compost can be only be sold when its impurity content is below 0.5% on dry matter.

Maximum level of impurities in Italian quality compost (dlgs. 75/2010)		
Parameter	**unit**	**Limit value**
Impurities plastics, glass and metals (size Ø > 2 mm)	% d.m.	< 0,5
Stones (size > 5 mm)	% d.m.	< 5

In Italy, only quality compost that meet the legislative decree 75/2010 is considered as End of Waste.

1.6 Bioplastic recovery meets European Standards

Today, a wide range of consumer products are made of plastic: bags, packaging, disposable tableware, etc.. The greatest commercial success of plastic is due to its versatile features such as low cost, light weight and resistance to degradation. The sectors of plastic applications are wide: cosmetic, pharmaceutical, food, beverage and chemical . Unfortunately, uncontrolled use of plastic packaging, especially disposable plastic goods, contribute to an increase in waste production. In Italy the packaging, recycling, and the recovery sector is implemented by CONAI (Consorzio Nazionale Imballaggi), a private and non-profit organization. The most important field is packaging recovery through a mechanical recycling. In 2006, according to CONAI data, more than 20% of packaging materials placed on the market were collected for recycling.

The most important Italian laws on the packaging sector (production and management of post-consumer packaging) are:

- Directive 2004/12/EC, which replaces the Directive 1994/62/CE;

- The legislative decree n. 152/06

The European Directive applies to packaging production and packaging waste placed inside the European Community. The main purpose of the European Directive is to reduce the environmental impacts from packaging production and packaging waste. The Directive sets limit on unnecessary packaging production while promoting recycle, reuse and recovery of packaging materials. Directive 2004/12/EC defines what "packaging" means (see Annex I).

Annex II of the above European Directive (essential requirements on the composition and the reusable and recoverable, including recyclable, nature of packaging) have been acknowledge by the Italian government through the UNI technical standards listed below:

- UNI EN 13427:2005 – Packaging - Requirements For The Use Of European Standards In The Field Of Packaging And Packaging Waste
- UNI EN 13428:2005 Packaging - Requirements Specific To Manufacturing And Composition - Prevention By Source Reduction;
- UNI EN 13429:2005 – Packaging - Reuse;
- UNI EN 13430:2005 - Packaging - Requirements For Packaging Recoverable By Material Recycling
- UNI EN 13431:2005- Packaging - Requirements For Packaging Recoverable In The Form Of Energy Recovery, Including Specification Of Minimum Inferior Calorific Value;
- UNI EN 13432:2002 – Packaging - Requirements For Packaging Recoverable Through Composting And Biodegradation - Test Scheme And Evaluation Criteria For The Final Acceptance Of Packaging

These Standards have also been implemented by Italian Legislative Decree 152/2006. In accordance with the European Directive, Decree 152/2006 explicitly requires the different management of packaging materials.

1.7 Bioplastic market: clarity needed

Unfortunately, there are still a lot of non-compostable items that are placed on the market as compostable. First of all, we need to distinguish the two different meanings of the words biodegradable and compostable. Most of time, these two words are incorrectly used and as a result, create confusion among citizens, waste collection companies and organic waste facilities. Claiming that a material is biodegradable, doesn’t non necessary means that it is compostable as well. In fact, only authentic compostable materials become compost in industrial compost facilities.

In order to know whether a material has biodegradability and / or compostability features there is an reference European standard. EN 13432:2002 (Packaging Requirements For Packaging Recoverable Through Composting And Biodegradation - Test Scheme And Evaluation Criteria For The Final Acceptance Of Packaging) sets the criteria of compostability. As a result, customers are encouraged to check for the presence of a certified “compostable” label that identifies the material as meeting the UNI EN 13432 standard. The generic “biodegradable” label does not ensure that the materials are compostable.

According to EN 13432, a compostable material must have the following characteristics:

- Biodegradability: the capability for the material to be converted into CO2 under micro-organisms processes. This property is measured using a standard laboratory test method: EN 14046 (also published as ISO 14855: biodegradability under controlled composting conditions). In addition, in less than six months, the material must be biodegraded for more than 90% of its initial total weight..
- Disintegrability, the fragmentation and loss of tested material in the final compost (absence of visual pollution). It is measured in a pilot scale composting test (EN 14045). A sample of material will, together with biowaste, be subjected to a composting process for three months. The final compost is then screened with a 2mm sieve. In order to pass this test, the total tested material residues with dimensions larger than 2mm shall be less than 10% of the original weight.
- Absence of negative effects on the composting process. Verified with the pilot scale composting test.
- Absence of negative effects on the final composting process. Verified with the plant growth test according to OECD 208.

1.8 Italian law: a driving force for bioplastic sector

There have been numerous regulatory interventions in Italy to prevent environmental impact of plastic packaging. In December 2006, through the Economic and Financial Planning Document the Italian government banned non-biodegradable bags from the Italian market. In addition, in 2012 the Italian Government (Law 28/2012) strongly reinforced the ban by defining “biodegradable” as a feature that meet EN13432. It also defines the difference between

single use and reusable bags and set clear system of fines for lawbreakers. As a result, this new bill reinforced the strong synergies between banning non-compostable plastic bag and source separated organic waste collection systems in Italy.
The law n. 28/2012 stipulates two basic concepts:

- the acknowledgement biodegradability as feature that meets EN 13432:2002;
- the requirement of a specific certification (issued form a third accredited party) to attest genuine compostable materials under EN 13432:2002 standard.

Since 2010, in Italy (Decree n.152/2006) the organic waste collection must be carried out using reusable containers or compostable UNI EN 13432-2002 certified bags.
Items that are collected by source separation organic waste must to specific characteristics:

- certified biodegradability and “compostability” characteristics following UNI EN 13432:2002
- The presence of the “compostable” logo issued from an accredited third party (CIC-compostable trademark or similar)

The compostable certification programme on bioplastic products both supports the recovery of those new products and the fight against non-conforming products. For more information please visit www.compostabile.it
The benefits of products certified as "Compostabile CIC" are:

- from the producers of certified compostable goods point of view, the presence of CIC compostable trademark logo allows stronger marketing and promoting,
- from the consumer point of view, the presence of logo encourages them to purchase an environmentally friendly product that can be recovered with their food waste
- from the municipality’s point of view, compostable products help to reach a high standard of source separation collection system
- from the composting plants point of view, the CIC logo identifies genuinely compostable products which help reduce additional disposal costs.

The legal support for the implementation of the Compostable Mark is definitely worth mentioning, the Finance Act of 2007 and the recent Law no. 28/2012 which banned non-biodegradable disposable bags. In addition, the Framework Law on Waste (Environmental Code) defines that "the separate collection of organic waste must be carried in containers with empty reusable or compostable bags certified to UNI EN 13432-2002."
These legislative measures will help narrow products to only those which are actually compostable (biodegradable is not only required but there must be certainty that the product will degrade in a composting process for a maximum period of 12 weeks and to least 90 %). Moreover this will also remove the phenomena of counterfeiting from the market of the supply of bags and artifacts which are not compostable.

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(228) MANAGEMENT OF ORGANIC RESOURCES WASTE IN FRANCE: REGULATIONS AND COMPOSTABLE BAGS

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ABSTRACT

The purpose of this paper is to make a close examination of the various regulations established in France in order to manage the organic waste in a sustainable and efficient way.

The 1st part of the paper will present in grater detail the French initiatives, laws and arguments reflecting the official interests and goals of the public authorities and of the private stakeholders too (e.g. the industry), where prevention is the first and the major step in the municipal solid waste management and material recycling follows suit, with organic waste collection playing a major role in reducing the amount municipal waste sent to disposal. The 2nd part will showcase some case studies of French municipal authorities having adopted the separate collection of biowaste with production of quality compost.

The 1st part focuses on:

- Grenelle law – targets on the reduction of the municipal and big producers organic waste
- French composting norms and bioplastics
- "NF environnement" composting waste bags labelling
- French agreement between government, local authorities, plastics industry and retailers

In the 2nd part, some French case histories are presented.

(234) HOW COMPATIBLE ARE COMPOSTABLE BAGS WITH MAJOR INDUSTRIAL COMPOSTING AND DIGESTION TECHNOLOGIES?

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EXECUTIVE SUMMARY

Industrial composting has widely implemented across in Germany for over 20 years. During the last decade and driven by legislation, the separate collection of organic waste has grown more and more popular. As a result, a broad variety of technologies of industrial composting and anaerobic digestion are in place. According to the *Bundesgütegemeinschaft Kompost's (BGK;* the German Quality Assurance Association for Compost) classification scheme, there are eight major types of process, so-called *'Hygiene-Baumusterkategorien'*, which differ a lot with regard to their technical components and composting/digestion times.

Compostable biowaste bags have been on the market for more than 15 years. As soon as biodegradability and compostability according to DIN EN 13432 or DIN EN 14995 have been demonstrated under laboratory conditions and have then been certified, the product can be labelled with the compostability logo. Nevertheless, compostable biowaste bags have not systematically been field-tested in all of the different types of industrial composting or anaerobic digestion plants until now. As a result, there is still uncertainty among operators and local authorities as to whether or not compostable bags are technically compatible with on-site technology, especially as to whether the bags decompose fast enough within the usual decomposition times. At the same time, the interest in the subject is increasing, as compostable bags may increase the amount and quality of organic waste collected by households.

This paper outlines a project performed in Germany from April 2010 to November 2011. The project is divided into two parts. The scope of the first part was to evaluate the relevant industrial composting and anaerobic digestion technologies for the treatment of organic waste. For this purpose, all plants that are members of the *BGK* were evaluated. The results showed that six types of process cover approximately 50 % of the total number of plants and the annual capacity of all composting and anaerobic digestion plants listed by *BGK*.

In the second part, the specifications of these types of process were first determined by means of telephone interviews. Subsequently, five different kinds of biowaste bags were practically tested, each on one plant of a certain plant design. The biowaste bags for testing were added to the plants' normal bio-waste streams. Samples were taken at different times. Material degradation was documented by photographs and by weight determination.

In summary, it can be said that standard types of bio waste bags quickly achieved high degradation rates in the field test. The test showed that the degradation requirements according to DIN EN 13432 or 14995 were met in nearly all kinds of plants. It can thus be concluded that these bio waste bags do not cause any visible compost contamination or technical problems in all the practice-relevant plant types that were tested.

The recognisability of the biowaste bags tested should be improved – in spite of the compostability logo. All five product types that were tested looked very different. When sorted by hand, they do not clearly differ from conventional plastic bags. So there is room for improvement on the part of the bioplastics' converters. For instance, C.A.R.M.E.N., the Bavarian Agency for Renewable Raw Resources, recommends a standardised, hexagonal design for compostable bio-waste bags. This type of design was introduced by the local waste association in Straubing, Bavaria in 2004. Since then, the number of local authorities using bio-waste bags with the hexagonal design is continuously growing.

1 INTRODUCTION

In 2015 the biobin will be introduced nationwide throughout Germany (BMU 2011). Many people, however, refuse to collect their kitchen waste separately because they consider it unhygienic. Thus, large amounts of valuable biowaste are not utilised for the production of compost and bioenergy. Another problem is that households use conventional plastic bags for collecting their kitchen waste. These are not biodegradable or compostable and can cause technical problems in composting and anaerobic digestion plants and may also contaminate the compost.

1.1 Background

Compostable biowaste bags made of special bioplastics are an alternative to conventional plastic bags. As soon as the biodegradability and compostability is tested under laboratory conditions and certified according to DIN EN 13432 or DIN EN 14995, the product can be labelled with the compostability logo (CEN 2000, CEN 2006; figure 1). Standards require *inter alia* that a maximum of 10 % of the original dry weight of the test material in a sieved fraction > 2 mm can be found after 12 weeks composting or a maximum of five weeks with a combination of anaerobic and aerobic treatment.

Figure 1: Compostability logo

In Germany, there is a large number of different composting and anaerobic digestion processes which are used for recycling organic waste. On the one hand, there are partly enormous procedural differences between these processes. On the other hand, the composting time is typically much shorter in practice than the twelve or five weeks required in DIN EN 13432/14995. Plant operators and representatives of local authorities who are critical of compostable biowaste bags conclude from this that the bags do not meet the requirements of composting practices because they do not degrade fast enough.

The most common composting and anaerobic digestion processes are listed and exactly described (BGK 2010) in the *Hygiene Baumuster-Prüfsystem (HBPS)* (i.e. Hygiene Type Control System) of the *Bundesgütegemeinschaft Kompost (BGK* the German Quality Assurance Association for Compost). Each process is assigned to a so-called type category which is characterised by certain constructional conditions and system components. For further distinction, all category types are divided into a number of type models.

1.2 Research objectives

Even though compostable biowaste bags have been used for the collection of municipal biowaste in Germany for more than 15 years, to date no exact study for analysing the biodegradation times of these products in different kinds of plants has been conducted. The objective of this study was therefore to determine how quickly standard certified compostable biowaste bags degrade under real conditions in composting and anaerobic digestion plants that are relevant in Germany.

2 MATERIAL AND METHODS

2.1 Relevant types of process

The first part of the study was to determine the plant types that in practice are relevant for the valorisation of biowaste in Germany. For this purpose, all plants that are members of the *BGK* (Federal Compost Association) were evaluated. These plants hold approximately a 70% share of all composting and anaerobic digestion plants and a share of 80 % of the input quantities in Germany (Kehres 2010). Of these only plants which utilise biowaste collected in biobins were examined. In the next step, at least three plants of each type were surveyed regarding the specifications of their plant. A field test was then carried out with one plant operator per type. With respect to the spatial acceptance of the results, the field tests were undertaken all over Germany.

2.2 Sampling

It should be possible to take a sample at any time. Therefore, the biowaste bags to be tested were put into a 40-litre nylon mesh bag with a mesh width of 1 to 2 mm. The nylon fabric is not affected by the composting process and thus prevents any loss of the sample material. At the same time, the exchange of gases and liquids is enabled. So nearly the same conditions should be achieved both in the test bags and in the surrounding biowaste. For greater practical relevance, the biowaste bags were filled with biowaste. So the sample material was fully surrounded by biowaste and thus by micro-organisms. The degradation of the material was documented by photographs and by gravimetric weight determination. Before, the (remnants of the) biowaste bags were dried to a constant weight at a temperature of 105°C.

2.3 Test procedure

The filled nylon mesh bags were added to the regular material flow of the biowaste plant. During the turning processes they were temporarily removed to prevent them from being damaged, thus avoiding sample losses. Samples were taken after a third of the composting time, after the intensive composting process at the earliest, after two thirds, and when sifting the compost. In accordance with the standards DIN EN 14995 and 13432, a sample must definitely be taken after 12 weeks in case of aerobic digestion and after five weeks in case of a combined procedure of anaerobic and aerobic digestion (CEN 2000, CEN 2006). If possible, the sampling periods after one third and two thirds of the composting time had been combined with the dates required by the standards.

A sample volume of 30 litres of biowaste per nylon mesh bag was determined. It was assumed that biowaste bags are usually disposed of when they are approximately half-full. Six biowaste bags of a certain type were filled with five litres of biowaste and put into a nylon mesh bag. In order to examine the effect of a knot on the rate of degradation, three bags were fastened by tying knots, the others remained open. Subsequently, a scoopful of biowaste was added in order to cover the biowaste bags evenly with biowaste.

For each type of biowaste bag, a nylon mesh bag with six biowaste bags was provided per sampling operation. An additional nylon mesh bag (including contents) was added to the composting process for safety margin. Since five different products were tested in the field test (see below), a total of 20 nylon mesh bags was thus added to a batch of biowaste per composting plant.

2.4 Sample materials

Four types of standard certified compostable biowaste bags and one T-shirt bag were field-tested. All products are available in German retail stores or online shops.

Table 1: Overview of the sample materials tested

Product type	**Bioplastics type**	**Bioplastics manufacturer**	**Supply source**	**Filling volume [l]**
Wenterra T-shirt bag	Mater-Bi® NF	Novamont (I)	biomasse (D), *retailer of biobased products*	< 10
Profissiomo biowaste bag	Mater-Bi® CF	Novamont (I)	dm-drogerie markt (D), *chemist's shop*	10 +
Biowaste bag	Bioplast®	Biotec (D)	Rewe (D), *grocery store*	10 +
Bio4Pack waste bag	Ecopond Flex®	KingFa (Hong Kong)	www.hygi.de, *internet portal for the purchase of detergents*	< 10
Biowaste bag	Bio-Flex®	FKuR (D)	Real (D), *grocery store*	10 +

3 OPERATIONAL PRACTICES OF THE RELEVANT PLANT TYPES AND TEST PLANTS

Evaluation of the data of the *Bundesgütegemeinschaft Kompost (BGK)* showed that six types of process cover approximately 50 % of the total number of plants and the annual capacity of all composting and anaerobic digestion plants listed at *BGK*. Table 2 lists these plants and some of their specifications. Table 3 shows an overview of the number of plants and annual capacities of these types of process.

Table 2: Practice-relevant types of process with process description (BGK 2010)

Number and type of process	Active Composting time	Turning process	Additional Aeration	Humidification
1.1 Herhof boxes	7 days	not applicable	forced aeration	process water, industrial water
3.6 Horstmann WTT tunnel	7 days	not applicable	forced aeration	process water, industrial water
5.2 Bühler-Wendelin	9 weeks	≥ 9x	forced aeration	industrial water, process water up to 4.5 weeks
6.2 triangular windrow, not covered	6 weeks	≥ not later than once every four weeks	not applicable	During the turning process when required. Industrial and process water up to 3 weeks.
6.3 trapezoidal windrow, open-air (I)	5 weeks	≥ 4x	not applicable	During the turning process when required. Industrial and process water up to 2.5 weeks.
6.8 triangular windrow, covered	4 weeks	wheel loader or compost turner ≥ 1x	not applicable	During the turning process when required. Industrial and process water up to 2 weeks.

Table 3: Number and annual capacity of practice-relevant types of process (plants listed at BGK)

Type of process	6.3	6.8	1.1	5.2	3.6	6.2
Number of plants	22	26	19	8	7	127
Capacity of the smallest plant [t/a]	2,900	4,500	8,000	15,000	10,000	6,500
Capacity of the largest plant [t/a]	50,000	85,000	36,000	80,000	85,000	87,500
Average capacity [t/a]	15,782	13,842	17,924	36,937	34,286	10,392
Total capacity [t/a]	347,199	359,885	340,550	295,500	240,000	1,319,792

From each of the types of process named above one plant operator was chosen for the field test. The locations of the test plants are listed in table 4. Considering the fact that plants are increasingly equipped with pre-fermentation systems such as batch dry digesters, this kind of plant was also included in the study. The type of process 3.6 was included in the study instead of the type of process 3.1 (Gicom) because no plant operator of the type of process 3.1 was willing to take part in the field test. Therefore, an alternative type of process from the same category type was sought. It should also have procedural similarity and a comparable share in the total capacity and the number of plants.

Table 4: Types of process taking part in the field test with locations

Plant	Number and type of process	Location
Plant 1	6.3 Trapezoidal windrow, open-air with pre-fermentation (“combined system“)	Bavaria
Plant 2	6.8 Triangular windrow, not covered	Saxony
Plant 3	1.1 Herhof boxes	Hesse
Plant 4	5.2 Bühler-Wendelin	Lower Saxony
Plant 5	3.6 Horstmann WTT tunnel	Lower Saxony
Plant 6	6.2 Triangular windrow, covered and without forced aeration	Bavaria

3.1 Trapezoidal windrow, open-air (6.3) with pre-fermentation (“combined system”)

The first test was carried out in a composting plant in Bavaria. The aerobic composting process is preceded by a fermentation in a dry batch digester which is loaded intermittently. In the “combined system” of the test the incoming organic waste is temporarily stored in a bunker. The dry batch digesters are loaded with a mixture of old (inoculated) material and fresh organic waste. The digester gates are locked gas-tight by means of an air-pressurized circumferential seal.

The process temperature of approximately 40° C is reached quickly by self-heating caused by the forced input of air. Subsequently, the air input is stopped and anaerobic micro-organisms via percolation are added. Following the three-week anaerobic period, the fermentation substrate is mixed with immature compost and further composted in a trapezoidal windrow for 8 to 12 weeks. In this process, at first the windrow is turned twice a week by means of a self-propelled compost turning machine. 9.5 weeks after the fermenter discharge the windrow is only turned once a week. In the 12th week after the start of the test the compost is screened.

3.2 Triangular windrow, covered and without forced aeration (6.8)

(Triangular) windrow composting is the simplest form of composting. After homogenising the material, windrows are built. They are aerated through regular turning processes. During the interviews the plant operators indicated an average impurity content of 3 to 4 % and a composting time of 4 to 6 months.

In the composting plant in Saxony where the field test was executed, the incoming organic waste is directly piled into windrows. Within the first two months, the windrows are turned weekly using a windrow turner and afterwards only once in three weeks. When required, the windrows are irrigated during turning. After approximately four months, the compost is screened by means of a drum screen to a size of 0 to 10/15/20 mm.

3.3 Herhof boxes (1.1)

In this system, the organic waste is first conditioned, then filled into the Herhof boxes by means of wheel loaders. In the boxes, the composting process is enhanced by active aeration or de-aeration. According to the Hygiene Type Control System (BGK 2010), seven days of composting are required for hygienisation. During the interviews, operators of Herhof plants stated that they practise composting times of 7 to 10 days. The post-composting process takes 56 to 70 days. The average impurity content in these plants is 5%.

In the composting plant in Hesse where the field test was executed, the incoming organic waste was first mixed with fibrous green waste, homogenised and then put into the Herhof boxes. After seven days, the material is piled into windrows by means of a wheel loader. After an eight day maturation the material is screened using a drum screen < 20 mm.

3.4 Bühler-Wendelin (5.2)

In composting plants with the Bühler-Wendelin turning technology, the compost is piled windrows in a composting hall. The turning process is automatic and the substrate is first taken by a paddle-wheel being filled into the next area via conveyor belts. The hygiene control system requires nine weeks for complete hygienisation. Whereas, in the plants that were surveyed, hygienisation time lasts 10 to 14 weeks. The impurity content is 5% on average.

In the plant in Lower Saxony where the field test was executed, the incoming organic waste first passes through a shredder with a bag opener, then through a pre-sorting system with a star screen, where a large part of the films and bags are separated. Mixed with fibrous green waste, the organic waste is piled via a feeder into trapezoidal windrows on a composting field in the composting hall where suction aeration takes place through the floor. After one week, the organic waste in turned to the next field. Six weeks later, the organic waste arrives in the final composting shed, where no active aeration takes place. After a total of 12 to 14 weeks, the compost is sieved.

3.5 Horstmann WTT tunnel (3.6)

For the composting process in the Horstmann WTT tunnel, the conditioned organic waste is filled into the tunnel. After a composting or hygienisation time of at least seven days, the material is discharged (*BGK* 2010). Subsequently, the substrate is finally composted in a windrow. During the interviews the plant operators indicated an impurity content of the input material of 2 to 3%. The main composting process takes 2 to 3 weeks, while the final composting process is finished after an average of five weeks.

The incoming organic waste in the test plant in Lower Saxony is screened with a drum screen to < 60 mm. After removing metallic pollutants with a magnetic separator, oversize material is shredded and again added to the material flow before screening. The screened fraction (< 60 mm) is automatically discharged into the tunnel. The material remains in the tunnel for approximately seven days. The discharge process is fully automatic. The organic waste is drawn from the tunnel on a mesh and then aerated by means of two spade shafts. The material is transported to the final composting shed via conveyor belts. There, the contents of four tunnels are piled into a windrow. The windrow is turned at least once a week to the next zone. After approximately four weeks, the compost is turned to the storage hall. When required, it is sieved to a size of 12 mm.

3.6 Triangular windrow, not covered and without forced aeration (6.2)

Most of the composting plants registered with *BGK* work with an open triangular windrow. The Hygiene Type Control System requires a composting time of at least six weeks for this kind of process (BGK 2010). The average time for composting is about three months. The average impurity content amounts to approximately 15% of the input material. All in all, triangular windrow composting is flexible in composting time and thus in the degree of composting. Typical composting times are one to two months with a preceding intensive composting process and up to six months without a pre-composting process (Bidlingmaier 2000).

In the test plant in Bavaria, the homogenised and conditioned organic waste is pre-composted in Thöni boxes. Since the composting process in a composting plant of the type of process 6.2 (triangular windrow) was to be tested, the incoming organic waste and the filled nylon mesh bags were directly put into the triangular windrow for post-composting. The windrow is turned once a week by means of a self-propelled compost turning machine and irrigated when required.

This test additionally examined whether compostable biowaste bags degrade faster when they are in a biobin before being put into a windrow (corresponding to real conditions). In order to achieve that, the biowaste bags were filled with organic waste, then put into a biobin and left there for a week because scheduled waste collections take place every seven or every 14 days in Germany. Afterwards, their condition was documented by photographs. Subsequently, they were placed into nylon mesh bags and then put into a windrow. For comparison, biowaste bags were freshly filled with organic waste as before, placed into nylon mesh bags and also put into the windrow.

4 DISCUSSION

The field tests show that the majority of samples meet the degradation requirements according to DIN EN 13432 or 14995 in nearly all plant types (figure 2). The red line marks 10% per cent of the original weight of the sample weight which, according to the standards, may still be found after a composting time of twelve weeks in the sieve fraction > 2 mm. The remnants of film which were still present were often knots. However, these were obviously heavily decayed, too, because they could be easily crushed between fingers.

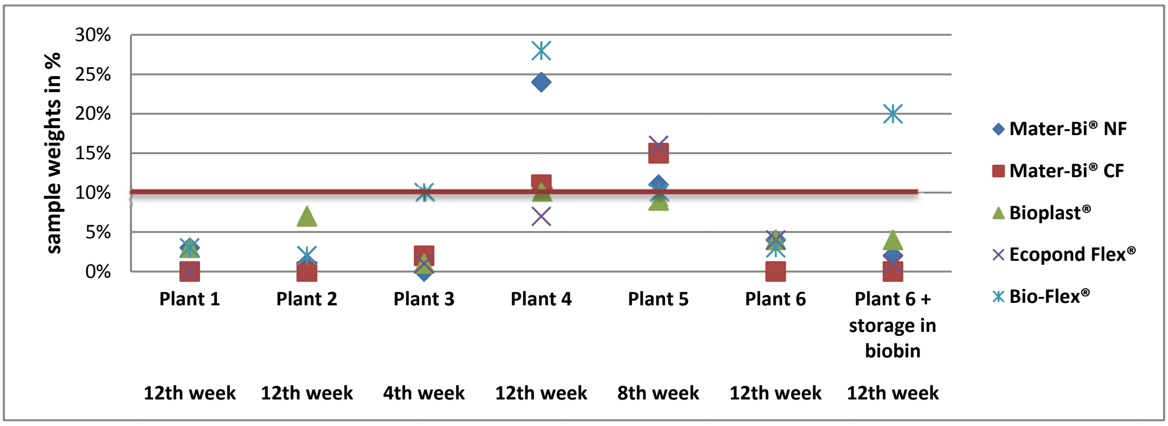

Figure 2: **Overview of the sample weights in % at the time of screening**

In plant 4 (type of process: Bühler-Wendelin), the products made of Bio-Flex® and Mater-Bi® CF did not meet the rates of degradation. Due to the remnants of film that were found, it can be assumed that the biowaste bags were unfilled when put into the nylon mesh bags – against the test protocol. So the empty biowaste bags were crumpled in the nylon mesh bags, thus multiplying the material strength. The rate of degradation directly depends on the sample thickness thus the degradation took considerably longer. Moreover, the low degradation rates in the plant are presumably a result of the special test form (see below). In a written comment, the plant operator also assumes that the nylon mesh bags inhibited the degradation of the biowaste bags and that a much better, possibly a complete degradation would be possible in a test without them.

Furthermore, the analysis showed that that the composting time is much shorter in many plants than the 12 weeks required in the standards. In nearly all cases the biowaste bags were also degraded within the shorter composting times. In plant 3 (type of process: Herhof boxes) the compost was already sifted after four weeks.

Even after this short time, only insignificant remnants of the biowaste bags were found (a maximum of 8% of the original material, mostly even < 5%). In plant 5 (type of process: Horstmann WTT tunnel) only the samples made of Ecopond Flex® and Mater-Bi® CF just dipped below the ten per cent mark.

It can be noted that products made of Bioplast® and Mater-Bi® (both types) – with one exception each – constantly achieved good degradation rates. The bags made of Mater-Bi® CF degraded fastest in three of the six tested plants, the T-shirt bags made of Mater-Bi® NF fastest in plant 3 (Herhof boxes).

In the course of test in plant 6, in which a group of test materials had been stored in the biobin before the test, the following observations were made after a storage time of one week in the biobin: While no or just minor traces of degradation were optically detected on the products made of Ecopond Flex®, Bio-Flex®, and Bioplast®, both products made of Mater-Bi® were already visibly affected. The weight determination of the samples at the end of the test showed the following results: All samples that had been stored in the biobin for one week were clearly more degraded than the samples that had been put directly into the windrow (figure 3). This was especially evident with Mater-Bi® NF (figure 4). The only exception was the sample made of Bio-Flex® although this is obviously the result of a measurement error.

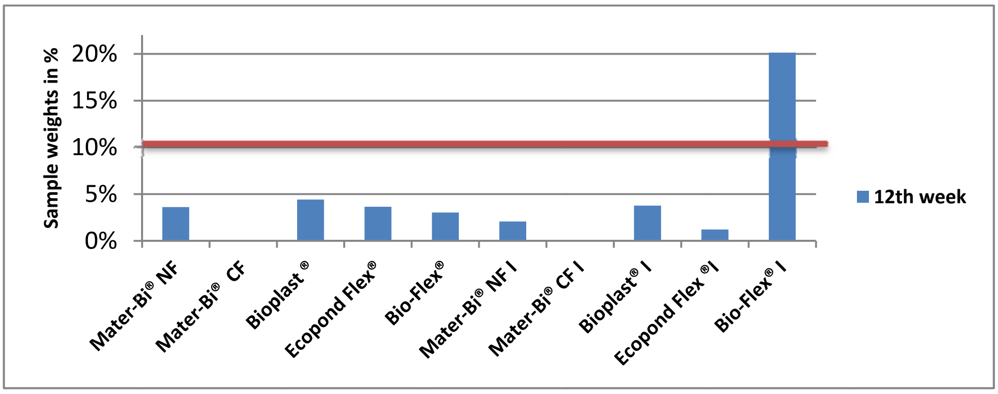

Figure 3: **Dry weights of the samples with (I) and without a one-week storage in a biobin followed by triangular windrow composting (6.2)**

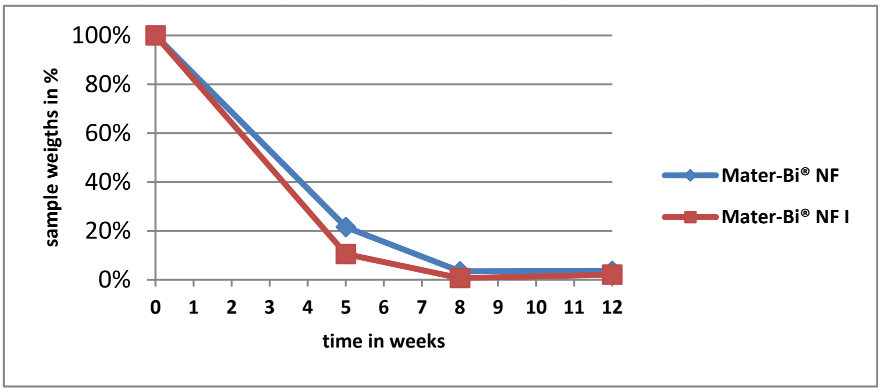

Figure 4: **Degradation of a sample made of Mater-Bi® NF with (I) and without a one-week storage in a biobin followed by triangular windrow composting (6.2)**

Two aspects of the selected test have to be critically discussed in order to put the results into perspective. One is the use and the handling of the nylon mesh bags, the other is the filling of the bags with homogenised biowaste directly before putting them into the windrow.

Both aspects had purposefully been integrated into the test procedure and have not lost their importance when looking back. However, in the course of the project it turned out that both aspects contribute to a deterioration of the degradation conditions of the biobags in comparison to reality: Organic waste (and biowaste bags) in the nylon mesh bags are subject to considerably less mechanical stress during the turning processes than the rest of the organic waste in the plants that were tested.

Especially in processes with a self-propelled compost turning machine, but also in a Bühler-Wendelin unit, strong tearing, shearing and compressive forces have massive effects on the materials because they tear films and bags. In order to avoid damaging the nylon mesh bags, the bags were removed from the windrow before the turning processes started and were put back into the turned windrow afterwards. The biobags underwent less decomposition in comparison to the biowaste that was in the windrow because the biowaste bags offered a smaller contact surface to micro-organisms than the shredded material. Secondly, the samples (partly) cooled down during the turning process. For example, the turning process takes up to four hours in a Bühler-Wendelin plant. And thirdly, the microbial activity was probably relatively lower due to the lower content of oxygen in the nylon mesh bags because the contents did not intermix.

The following indications speak for those three assumptions: In the course of a field test the remains of an almost undecomposed salad leaf were found, in the course of another test a piece of a biowaste bag made of paper was found. Several tests have shown that biowaste in nylon mesh bags was significantly more humid than the rest of the windrow. That means that the pore volume of biowaste is filled with water and therefore no longer available for oxygen.

The second aspect that might have distorted the results as compared to reality is the fact that the biobags to be tested were each filled with biowaste on site in the composting plant and – wrapped in nylon mesh bags – put directly into the biowaste stream. The biowaste bags that get to a plant via municipal biowaste collection are already exposed to degradation processes when they are filled with household kitchen waste. Moreover, waste collection puts additional mechanical stress on those bags. So it can be concluded that the conditions of degradation are considerably better in practice than demonstrated in the study.

5 CONCLUSIONS

In summary, it can be said standard biowaste bags quickly achieved high degradation rates in the field test. So it can be assumed that they do not cause any technical problems in relevant plant types, do not contaminate the compost optically and are thus suitable for municipal biowaste collection. Against the background of a planned increase in biowaste collection and the aspired increase in the rates of food waste capture, the citizen, who is an important link in the chain, must be motivated to participate. Water-proof compostable bags can make an important contribution here. If the consumers fill the bags completely and do not fasten them with a knot, even better degradation rates could possibly be achieved.

In spite of the compostability logo printed on the bags, the recognisability of all tested biobags is difficult amidst the mass of biowaste. The labelling for compostable biowaste bags could be improved. All five product types that were tested look very different. When sorted by hand, they do not clearly differ from conventional plastic bags. So there is room for improvement on the part of the bioplastics' converters. For instance, C.A.R.M.E.N., the Bavarian Agency for Renewable Raw Resources, recommends a standardised, hexagonal design for compostable bio-waste bags. This design is accepted and supported by a growing number of local authorities using bio-waste bags.

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Session 8

(67) LIFE CYCLE ASSESSMENT AS A DECISION TOOL IN LOCAL WASTE MANAGEMENT? A FRENCH CASE STUDY

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EXECUTIVE SUMMARY

Background: It is now a bit more than a decade that LCA is being used as a decision making tool in waste management, in France and other European countries. Among the different tools available for environmental assessment, LCA is expected to fit particularly well the demand for global and exhaustive environmental evaluation of waste management scenarios. While specialised waste LCA tools already exist on the market, the adequate adaptation of the methodology to this particular field is still under discussion. One open question is here how to achieve a “good” integration of LCA results into the decision making process between multi-criteria analysis and simplification.

Objectives: This communication addresses the integration of LCA results into decision making in waste management. It gives evidence of a case study where non-aggregated LCA results were fed into decision making, in an early stage planning process with stakeholder implication. The case has been selected as a particular rich opportunity for studying the question whether and how LCA may be a help in decision making in local waste management. Following the LCA from the very motivation of use to the final decision, we analyse how the multi-criteria character of LCA and its results is fit into the process.

Methods: Our research results have been obtained through a case study, selected from a previous inventory of applications of LCA and similar tools in French waste management. The study is based on a total of ten interviews with the different stakeholders involved, as well as in-depth analysis of documentation on the decision making process and of the LCA. Interpretation of the results was carried out through interdisciplinary dialogue between researchers in LCA methodology and social scientists.

Results and discussion: The integration of LCA into the decision making process happened in different spheres (a sphere of external expertise, an internal decision sphere, a restricted stakeholder sphere and a wider one). LCA has been recognized by all actors as an appropriate tool thanks to its normalized nature. Accordingly, during and shortly after the period of external expertise, LCA results were discussed side by side with other (technical and economic) elements of multi-criteria analysis. The appropriation and comprehension of the non-aggregated LCA results were rather high, also within the stakeholder sphere. Supporting preconditions showed here to be the academic and professional background of the actors and the still reduced size of the stakeholder group. However, rather general LCA results due to early use in the planning phase mostly showed that all scenarios had their advantages and inconveniences. So, while LCA contributed in the eyes of decision makers to a solid decision, this very decision had in fact to be based on other criteria. Moreover, results appeared to concerned stakeholders quite trivial, in contrast to more tangible evidence on existing sites obtained through bio-monitoring and alike. A turning point in the acceptance of LCA was the moment of transformation of the results of external expertise to a synthetic document, integrating all criteria finally to be taken into account in the decision. This document was supposed to represent a consensus within the restricted stakeholder sphere, and was employed as a means for translating the process to the wider public sphere. Yet, the integration of certain criteria judged by stakeholders as “political” ones reduced the credibility and thus interest of the entire exercise for these stakeholders. They abandoned the scene of multi-criteria assessment, the political process was displaced to another scene. So, multi-criteria LCA seemed to offer a basis for discussion on environmental aspects of the scenarios, at least during some time. However, its influence on the final decision was rather a non-influence, showing that the decision had to be taken on other criteria.

In fact, the normalized nature of LCA, considered by the actors a strength in a politically charged decision, showed to be at the same time a means for the decision maker for making the decision non-contestable at least in environmental terms.

Conclusions: The successful integration of non-aggregated multi-criteria LCA results showed in our case not so much to be a problem of comprehensibility, as one might expect. Instead, our case study allows to get a perspective on the complex and in some sort political roles LCA adopts in decision making.

1 INTRODUCTION

Due to the growing awareness of the impact of human activities on health and the environment and the need for their environmental valuation, Life Cycle Assessment (LCA) is applied for more and more technical fields, including waste management. It allows indeed for an integrated vision of the environmental impacts, making explicit the frontiers of the systems and the internal and external flows of matter and energy, especially pollution emission, transfer and avoidance. Nevertheless, this environmental engineering view has to prove its usefulness in the real decision processes, i.e. how the "rational" choices of technical operators are embedded into the local political arena, especially when coming down from county management to local projects. The present study shows through one specific example how a LCA was used in a decision process inside a rural county while some local conflict about a landfill needed to be cope with.

1.1 Background

It is now a bit more than a decade that LCA is being used as a decision making tool in waste management, in France and other countries. Among different tools available for environmental assessment, LCA is expected to fit particularly well the demand for global and exhaustive environmental evaluation of waste management scenarios. While specialised waste LCA tools already exist on the market, the adequate adaptation of the methodology to this particular field is still under discussion. One open question is here to achieve a good integration of LCA results into the decision making process between multi-criteria analysis and simplification.

Waste management has to deal with flows of matter and energy (including avoided ones such as energy and matter recovery) which characterises the final sinks of industrial and domestic metabolism, and some global impacts like climate change are directly linked to (fossil, and possibly organic depending on the time horizons considered) CO_2 and CH_4 emissions. Nevertheless, it is also well known that waste treatment generates local disamenities coming from specific emissions (odours from organic waste for instance) or perceived risks (even without any emission of matter or energy), leading to the famous NIMBY problem and then high transaction costs. Since LCA was not specifically designed to quantify these local perceived risks, the use of this tool for (even environmental) decision-making in waste management needs some care.

Moreover, the often controversial situations about waste management may need the use of an independent arbitrary, which can be a human being (outsider, ombudsman) or non human, like any evaluation tool coming from an external and (still) non refuted knowledge of the normal science (Kuhn, 1996), from which LCA claims its origin and its standardisation (ISO 14140s). Nevertheless, researchers and practitioners recognize the relative (not absolute "scientific truth") of LCA results, depending not only on the quality of the input data but also of the more fundamental assumptions about the time horizon or the limits of the system, which can finally rely on some (often implicit) ethical views (Méry, 2010). Of course, the final phase of LCA which has to rate different (possibly uncommensurable) non aggregated environmental impacts is prone to debate: when comparing waste management scenarios, multi-criteria analysis do not allow itself for a direct rating of scenarios, unless the very lucky cases where there is a full dominance of a scenario for all non aggregated criteria (a review of the literature about uses and values of LCA in a complex and uncertain field of the environment is given elsewhere (Schlierf et al, 2012)).

1.2 Research objectives

Given this background, the objectives of this research is to analyse and interpret field cases where a LCA was used, to contribute to the knowledge about the possibilities, limits and relevance of this evaluation tool for decision aid in waste management. We choose to analyse non aggregated LCA instead of aggregated LCA or other aggregated valuation methods like ecological footprint and monetization (i.e. cost-benefit analysis with internalisation of environmental externalities) since we wanted to understand if detailed technical results (then generally without any direct ranking of scenarios) were understandable and useful inside the different stakeholder spheres. These spheres depend on the size of the stakeholder group: from the lowest to the biggest, these are external experts and their partners, public decision makers, enlarged commissions on waste management, local inhabitants and the general public.

The stakeholder size is an interesting variable for analysing the role of LCA in waste management, then one main research objective was to use it to understand the integration of non aggregated LCA into decision making.

2 METHODOLOGY

LCA is quite recent for waste management (the compulsory character of environmental valuation of waste planning was enforced in France in 2004). An extensive inventory of applications of LCA and similar tools in French waste management was first carried out. The deepened use of this tool was only noticed in a few cases and even fewer when a tangible effect on decision-making was looked for. We selected then an exemplary case in a French rural county which had to update its non dangerous waste regional plan while pending the usual problem of an ending landfill. Interpretation of the results was carried out though interdisciplinary dialogue between researchers in LCA methodology and social scientists.

When trying to elicit the viewpoint of actors to learn some general conclusions on the integration of non aggregated LCA results into decision making, one has to choose cases studies where data are relevant and available. This means that these cases must be not too old (witnesses of the real decision processes are not always found in this working context or may have forgotten some relevant details) but also not on the current stage, especially if they are quite controversial (Voineau, 2010). The review of the different cases study showed that only a few could conform to this requirement.

3 THE CASE STUDY

3.1 Presentation

The chosen case study is situated in a French rural county whose non dangerous waste planning was voluntarily revised in 2005 due to previous difficulties to extend the landfilling of waste, only one existing "provisional" (locally unwanted) landfill being available. At this moment, European and national regulations had just made compulsory the environmental assessment of plans and programs (2001/42/EC directive), and the staff in charge of waste management in this county was managed by an environmental engineer quite interested by LCA, helped by a skilled person in public relations. These facts created a seldom (if not unique in France) opportunity to organise a thorough and possibly operational analysis of the environmental consequences of waste management scenarios. A very precise tender was launched, including high level requirements in technical and democratic terms: the environmental assessment should have been scientifically robust (preventing from critics about its scientific quality), but also made understandable by the public. This challenge is not so easy to reach, since not only the justification and design of each non aggregated criteria of LCA need some scientific culture (hence often limited to a narrow technical sphere), but choosing then between scenarios when there is no dominance of all criteria need a deliberative procedure about the relative importance of each criteria (to be held then in a wider sphere where explicit or implicit values come into the game). LCA needs to be simplified for its comprehensibility and usability (for references about this specific topic, see Schlierf et al, 2012). Only one engineering consultant (well known in France) was able to fit the tender.

Four scenarios were first elaborated in a narrow technical sphere (technical committee) including the waste manager of the county and the consultants, then validated in a wider technical sphere (management committee). This wider sphere includes the local public operator of waste treatment and some elective representatives interested by the topic (among them the mayor of the town where the landfill is settled, who got some skills on waste engineering due to her scientific background in agricultural engineering and her local concernment). These scenarios were incineration, incineration with MBT, landfill with MBT, MBT with compost production (in order to induce a better involvement of local ecologists) and reversible bioreactor landfill (a technical improvement of the current landfill), in comparison with the current landfill situation.

3.2 In the narrow technical spheres : confined strategic use of LCA

Although the waste manager of the county considered himself as a technical staff, he perfectly knew the political game and the strategies of each stakeholder. Indeed, while he succeeded on making acceptance of the LCA tool by all stakeholders, he also knew how these stakeholders would like to use the tool for their own strategies. For instance, the mayor proposed to develop incineration instead of landfill, perfectly knowing that such a scenario would be badly valuated by the LCA without heat use, which means that such an incinerator should be settled elsewhere than in the very rural location of her town. But the county waste manager also knew how to circumvent this reasoning; by carefully using the national energy mix for the avoided emissions of incineration (instead of European mixes which give less weight to nuclear energy).

This is the most obvious epistemic limit of external "neutral" valuation tools: there are sensitive to their input parameters, which are prone to strategic considerations. Despite this first limit, the stakeholder accepted to play the game further since strategies could be hidden by scientific reasons. Note that this game was restricted to the narrowest sphere, and that no public written proofs remained on these technical and sensible aspects. Moreover, some obvious factors in non dangerous waste management that LCA could not easily take into account, like odours disamenities, were integrated more or less implicitly in the valuation process (the tracing back of this evolution was quite fuzzy indeed).

As usual in multi-criteria analyses, no scenario showed dominance on all criteria together. It means that an external "meta-criteria" had to be used to get a full ordering of scenarios. This can be done by a weighing between criteria, which has to be quantified through technical (for instance, monetization or ecological footprint) or political trade-offs. Another possibility is to use outclassing criteria which do not belong to the environmental field of LCA. It can usually be costs, but here the technical knowledge of the county waste manager led him to propose the reliability of waste management, due to the uncertainty of future waste flows for the next 30 years (a voluntarily longer time horizon than strictly necessary in the French regulations). He perfectly knew that only a landfill was able to efficiently cope with variable flows. To increase the acceptability of furthering landfilling, he had proposed an active exploitation (bioreactor landfill by leachate recirculation) and a possible reversibility through landfill mining of the solid refused fuel. But these two improvements were not applied for the LCA (probably due to the difficulty to get all the precise input parameters), which strategically allowed for a conservative landfill scenario. The incineration with MBT was here abandoned on non-environmental criteria: the financial cost. It seemed indeed for the technical committee and the management committee that it was not worth going on applying LCA to this scenario since the costs was clearly higher than others. At this point where a hard decision had to be made, the stakeholder sphere was enlarged from the technical committee (where the mayor of the town with the landfill did not yet fully agree due to the reliability argument) to the management committee with quite a publicisation of the decision criteria and results. A simplified rating of the three remaining scenario (table 1) was then worked out by the technical committee for the wider political sphere (elected representatives, public operators, and the general public). Here, the initial consensus about the use of LCA broke, which was strategically unavoidable since the altered use of LCA change the valuation in a wrong direction for some stakeholders who said it was "just theatre". The initial motive for using LCA, external rational arguments (if not truth), lost its sacred aura inside the management committee.

TABLE 1

Comparison of impact categories valued by LCA (inside the narrow technical sphere) and final three-level criteria used for the wider political sphere, with the ordering of each scenario
BRR : bioreactor landfill
INC : incinerator
MBT-C : mechanical biological treatment and composting of products

LCA environmental aspect	**Scenario ordering (based on quantified indicators of LCA environmental aspects)**	**Translation of LCA environmental aspects into simplified impact categories for the wider public sphere**	**Simplified scenario ordering through three "significant" levels (good, mid, bad) for the wider public sphere**
Consumption of resources : primary energy	INC > BRR > MBT-C	Energy balance	Idem
Consumption of resources : water	BRR > MBT-C > INC	Consumption and water eutrophication	**All the same**
Water pollution : eutrophication	INC > MBT-C > BRR		
Consumption of resources : primary minerals	INC > MBT-C > BRR	Inert materials	Idem
Climate change (100 years)	BRR > INC > MBT-C	Climate change	**"Almost" all the same**
Air pollution : acidification	BRR > INC > MBT-C	Emissions in air (acidification and heavy metals, dust, etc.)	**BRR and MBT-C >> INC** (BRR and MBT-C high, INC low)
Air pollution : metals	BRR > MBT-C > INC		
Air pollution : dust	MBT-C > INC > BRR		
Residual dangerous waste	BRR and MBT-C > INC	Landfill for dangerous waste	Idem
Residual non dangerous waste	INC > MBT-C > BRR	Landfill for non dangerous waste	Idem
		Organic balance	MBT-C > BRR and INC
		Landscape quality and space consumption	INC > MBT-C > BRR
		Others disamenities (odours, birds)	INC and MBT-C > BRR (INC and MBT-C high, BRR mid)

3.3 In the wide political spheres : public use of meta-criteria

While all elected representatives were asked for a weighing between simplified impact categories (table 1), it was clear that the existing landfill (with all possible improvements like the bioreactor technology) gathered most of wishes, due to its lower political cost and its ability not to give too bad results on all criteria. This kind of risk aversion is a classical phenomenon for collective decision-making (Slovic, 1987) and then for elected representative behaviour (Weaver, 1986). Then, following an usual temporal scheme (Richman and Boerner, 2006), the town where the landfill was settled only had to claim for compensations and monitoring, which was accepted by other elected representatives with ease despite increasing costs.

4 CONCLUSIONS

The results of this case study on strategic use of rational arguments will not be surprising those in charge with negotiations about territorial governance and waste management. The need to convince others implies the "civilizing force of hypocrisy" (Elster, 1999), and this is all the more true that the spheres under discussion are large. Then what is the interest of such rational tools like LCA (and other less standardized environmental assessment methods)? The fact that they use stabilised (not (yet) refuted) knowledge limit the strategic use of arguments: lower and upper bounds (and sometimes uncertainty) of input parameters are common knowledge, one cannot say everything without any critical peer review. This worked quite well in the narrow technical spheres of the county waste manager, the external consultant and even the technical committee: typical LCA arguments were mastered for justifying each viewpoint.

More important perhaps is the ability of such tools to generally exhibit the complex (hence controversial) nature of environmental evaluation, especially for waste management. Disagreements are then unavoidable, but there is generally not one unique solution that project leaders or opponents may impose to others (including through sacrificial utilitarianism: the best happiness for the greatest number anyway). Such tools can contribute to deliberative democracy where the preferences of stakeholder may interact and change, which can induce better results than simple win-loose gaming and crude negotiations. The governance of waste then should well include all the necessary transaction costs (including the needed time for deliberative and participative democracy) instead of false hopes of accelerating projects through the use of ex ante evaluations. This induces also prospective and anticipative capabilities, which do not seem so common in waste management: time horizon of one decade for waste planning is probably too short for controversial downstream projects, hence for upstream planning.

5 ACKNOWLEDGEMENTS

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(165) THE WASTE MANAGEMENT SYSTEM ON LANGKAWI ISLAND, MALAYSIA- OPTIONS FOR WASTE TREATMENT

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ABSTRACT

The island of Langkawi, situated in the north west of Peninsular Malaysia, is the one of the major tourist attractions in South East Asia with area of around 360 sq km around 100,000 permanent residents (2010). As a result of tourism the number of visitors to the island could reach more than 2 million a year creating strong pressure on municipal services, especially on the waste management system.

Since February 2010, Langkawi Island has been implementing a new waste management system which involved privatization. The new system includes a numbers of activities and facilities including; collection, transportation, treatment, transfer stations, recycling, and disposal, and the selected private company has a 20 years concession to manage the solid waste.

Currently the majority of the waste goes to a non-engineered landfill and there are plans to build a 100 tonne per day incinerator. Like many other sites in the world, land is scarce on Langkawi Island and landfilling is increasingly becoming an unacceptable option. Options for waste treatment are very restricted due to environmental constraints and limited spaces. Composting, recycling and anaerobic digestion treatments are the possible options to minimise the negative impact of waste on the island. The initial analysis has shown that the most viable options for the organic fraction of the waste are composting and anaerobic digestion. Recycling is likely to be restricted as there are no direct uses for the recycled materials on the island and they will need to be shipped elsewhere.

This paper considers the available waste options, with a focus on the optimization of the current practices on Langkawi . The evaluation of the different management options used the current situation as a baseline. The data used for the baseline study generated information on the Global Warming Potential (GWP), human toxicity via air, water and soil, acidification and spoiled groundwater. These parameters are shown on a common baseline using units of equivalent per person impact (PE).

Different scenarios were reviewed for this study using Life Cycle Assessment (LCA). The initial results show that on low emission the best options are separate collection of organic matter, shipping of selected dry recyclables off the island, anaerobic digestion of organic matter with composting of the digestate and landfilling of the residual waste.

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(199) IMPORT OF BIOMASS OR USING LOCAL BIOMASS TO REACH THE RENEWABLE ENERGY OBJECTIVES?

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EXECUTIVE SUMMARY

Biomass can be used in lots of different products, applications and industries. All European countries need biomass to reach the European goals for renewable energy. So does Belgium. In Belgium waste and energy policy is a competence of the Regions. Belgium has three regions: Flanders, Brussels and Wallonia. More as 50 % of the renewable energy sources have to come from using solid biomass in order to reach the goal of 20% energy from renewable sources in 2020. In Flanders (6 million inhabitants) in the last two decades, the Public Waste Agency (OVAM) has given very active attention with regards to an integrated approach of biomass and biowaste. Prevention measures, the separate collection of good quality biomass and biowaste materials and quality control on the organic-biological treatment chain represent a win win for the local authorities, industries and the environment. Flanders was one of the first EC member states to imply the landfill directive strategically. Since 2007 less than 5% of the industrial biowaste and sewage sludge is disposed of on landfill sites - to reduce green house gas emissions, in order to mitigate climate change.

Nowadays the demand of biomass in the different industries grows quicker as the production, the question rises where and how biomass can be applied the best in the economy.

The OVAM develops a mix of decision tools to guarantee the most sustainable applications for the different biomass materials. In 2012 the new material legislation will be implemented and replaces the waste legislation. This material legislation aims the recycling of the biomass/biowaste e.g. digestion of biowaste and composting the digestate instead of incinerating. Landfilling is no longer admitted. Furthermore, sustainability indicators for solid biomass are being worked out based on the 3 P's (People, Planet, Profit) in order to evaluate the social, environmental and economical impact of local biomass or imported biomass. This analysis is important to decide in which application the biomass can be applied in the most efficient way. The analysis of the local biomass market will be updated each two years to find potential land and potential biomass materials that can be used. For biomass flows which cannot be recycled, incineration plants are build, new technical solutions are in development. To ensure sustainable treatment and the right biomass treatment capacity in Flanders, adequate incentives and coherent regulations from the government are essential. They should create a positive investment climate for a long term, in order to attract and finance the necessary (re)investments.

One of the sustainable targets is reaching good quality of the end products of biological treatment (e.g. of the pelletised digestates), in order that biological nutrients can stay in the organic-biological cycle. 90% of the food industry sludge is of good quality and is recycled/treated in a biogas plant before using in agriculture. Direct and indirect Land use change and greenhouse gas emission reduction are important sustainability criteria for locally produced as well as imported biomass. More as 50% of the wood pellets have to be imported in order to reach the renewable energy targets.

The success of this sustainable cradle-to-cradle-policy and green economy is due to the following:

- a long term coherent and clear policy and framework stimulating the treatment of locally produced biomass and use of local biowaste
- supported by different instruments: sensitisation, different support for building treatment facilities, green power certificates for producing electricity of biogas from digestion and for producing electricity of incineration of not recyclable bio waste.

This biobased economy policy based on the combination of import and locally produced biomass leads step-by-step to a more optimized economical and ecological situation, less depending on the variability of the world market of fuel and biomass.

1 INTRODUCTION

1.1 Background

Biomass can be used in lots of different products, applications and industries. All European countries need biomass to reach the European goals for renewable energy. So does Belgium. In Belgium waste and energy policy is a competence of the Regions. Belgium has three regions: Flanders, Brussels and Wallonia. More as 50 % of the renewable energy sources has to come from using solid biomass in order to reach the goal of 20% energy from renewable sources in 2020. In Flanders (6 million inhabitants) in the last two decades, the Public Waste Agency (OVAM) has given very active attention with regards to an integrated approach of biomass and biowaste. Prevention measures, the separate collection of good quality biomass and biowaste materials and quality control on the organic-biological treatment chain represent a win win for the local authorities, industries and the environment. Flanders was one of the first EC member states to imply the landfill directive strategically. Since 2007 less than 5% of the industrial biowaste and sewage sludge is disposed of on landfill sites - to reduce green house gas emissions, in order to mitigate climate change.

1.2 Research objectives

Nowadays the demand of biomass in the different industries grows quicker as the production, the question rises where and how biomass can be applied the best in the economy.

The OVAM develops together with the stakeholders and e.g. the Flemish Institute for Technological Research (VITO) a mix of decision tools to guarantee the most sustainable applications for the different biomass materials.

2 ACTUAL SITUATION

2.1 Green power capacity

Figure 1 gives an overview of the new installed capacity in Flanders with green power certificates each year till 2012 (VREG, 2012). In 2012 totally 58% of the green power is produced by photovoltaic, 12% by wind, 29% by biomass digestion, (waste)wood combustion and incineration.

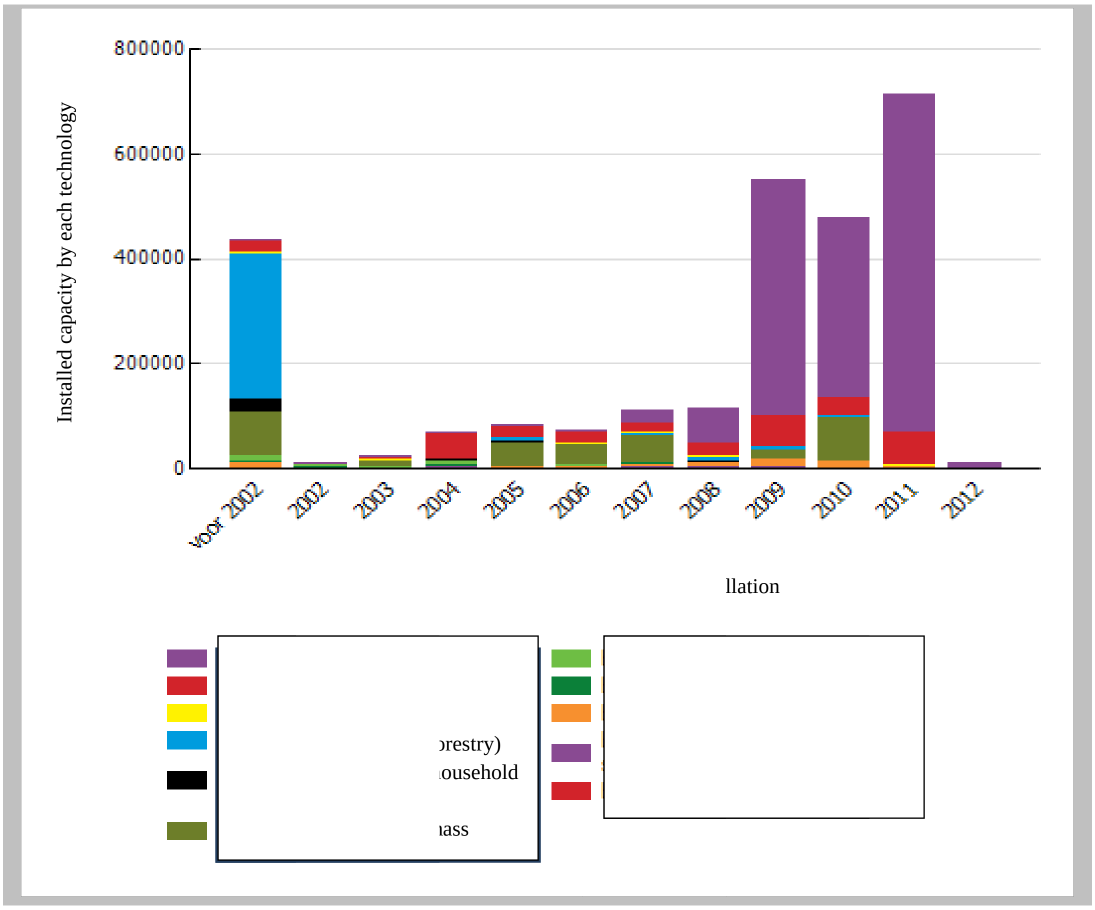

FIGURE 1 **Overview of the new green power capacity in Flanders installed each year up to 2012 (in kWe)**

2.2 Use of local biomass

In the Flemish rural areas about 50 digestion plants are build for the treatment of about 780000 tonnes of food waste, 420000 tonnes of pig manure and 130000 tonnes of energy crops in 2012. Household biowaste (320000 tonnes) and 480000 tonnes of green waste are separate collected and treated in a composting/digestion plant in industrial area since the early 90s. The production of good quality compost and digestate is necessary to close the biological circle. In order to fulfil the target of stimulating the use of compost and digestate efficiently, three main topics are considered to be necessary : quality assurance, research and marketing. Since 1992 the Flemish Compost Organisation (VLACO) is responsible for these topics with good results. VLACO is established as a cooperation between the OVAM, all the Flemish waste intercommunalities, all the public and private composting and digestion plants.

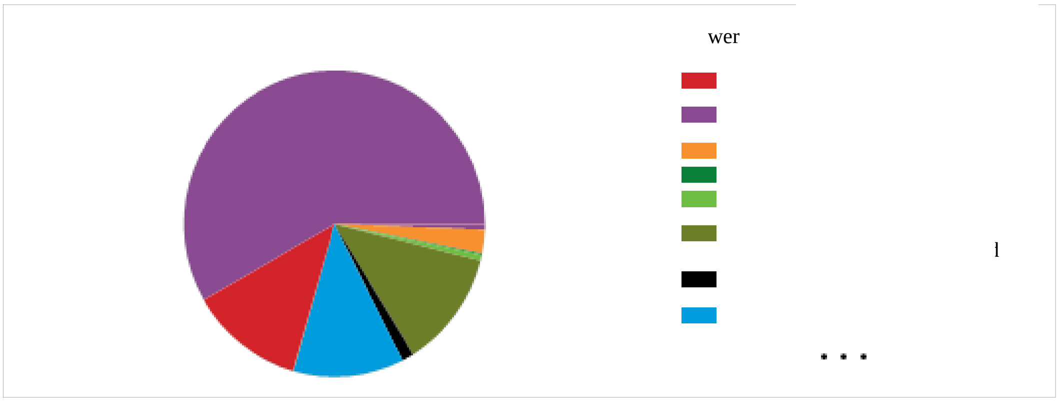

FIGURE 2 **Installed green power capacity for digestion of local biomass and incineration in Flanders in 2012 (VREG, 2012)**

3 DECISION CRITERIA FOR A SUSTAINABLE USE OF LOCAL/IMPORTED BIOMASS

In 2012 the new material legislation will be implemented in Flanders and replaces the waste legislation. This material legislation aims e.g. to close the recycling circle of the biomass/biowaste by digestion of biowaste and composting the digestate instead of incinerating. Per tonne green waste that is composted, instead of incinerated, 624 kg CO_2 is saved. Not only the processing itself but certainly the use of compost contributes to this by carbon sequestration. A lot of research on nutrient recuperation out of waste water and digestate is being done to replace artificial fertiliser. Landfilling of organic waste is no longer admitted. The waste cascade exists, the material cascade is born (figure 3). The focus lays on quality, on reducing the losses in the biological circle and to prevent losses going out of this circle.

Furthermore, sustainability indicators and verifiers for solid biomass are being worked out based on the 3 P's (People, Planet, Profit) in order to evaluate the social, environmental and economic impact of local biomass or imported biomass (OVAM-VITO, 2012). This analysis is important to decide in which application the biomass can be applied in the most efficient and sustainable way. In practice this means pioneering work. Some indicators (the environmental) are relatively easy to quantify, while the economic and social have proven to be difficult to quantify. For the moment international policy makers focus on a better interpretation of sustainability indicators for - mostly - only energy-related applications (GBEP, 2011). In the market it is not obvious to direct the available biomass towards those applications which contribute most towards the more sustainable objectives. This means policy has an important role in setting priorities. The use of biomass for food and feed stands ethically on the first and second place, biomass for energy on the last step. Each chain has its responsibility in reaching a biobased economy. In order to include sustainability criteria in policies, a good understanding is needed of the scale on which different indicators play. While impacts from waste treatment and waste processing are generated on a local level, the avoided impacts often occur abroad.

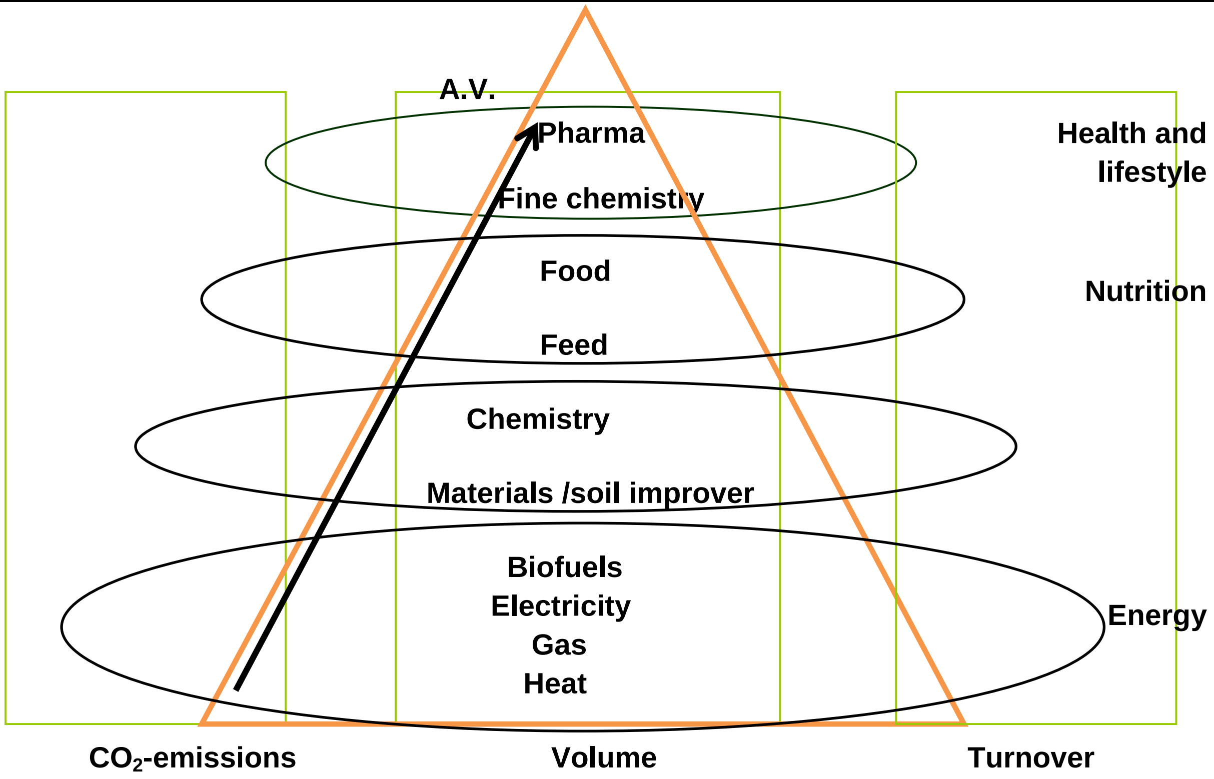

FIGURE 3 **Focus on closing the biological circles with respect for the cascade principle**

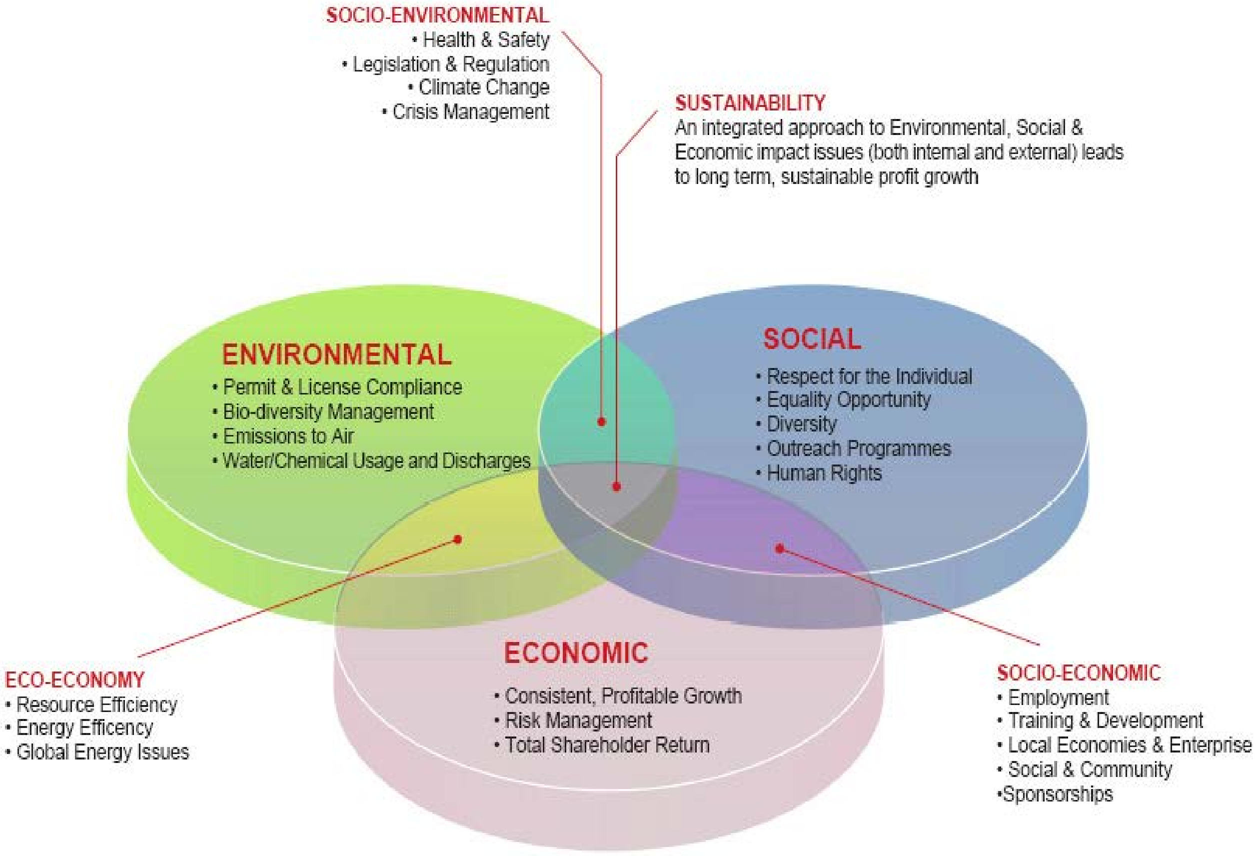

FIGURE 4 **The additional value is based on the Triple-P principles**

4 IMPORT OF BIOMASS TO REACH THE RENEWABLE ENERGY OBJECTIVES

In order to reach the renewable energy objectives in 2020 the most European regions have to import biomass, so thus Flanders. Figure 5 shows that Flanders still has a long way to go, by investigating a lot in wind energy, photovoltaic and biomass applications.

The analysis of the local biomass market will be updated each two years to find potential land and potential biomass materials that can be used for the different applications as shown in figure 6. For biomass flows which cannot be recycled, combustion plants are build, new technical solutions are in development. To ensure sustainable treatment and the right biomass treatment capacity in Flanders, adequate incentives and coherent regulations from the government are essential. They should create a positive investment climate for a long term, in order to attract and finance the necessary (re)investments.

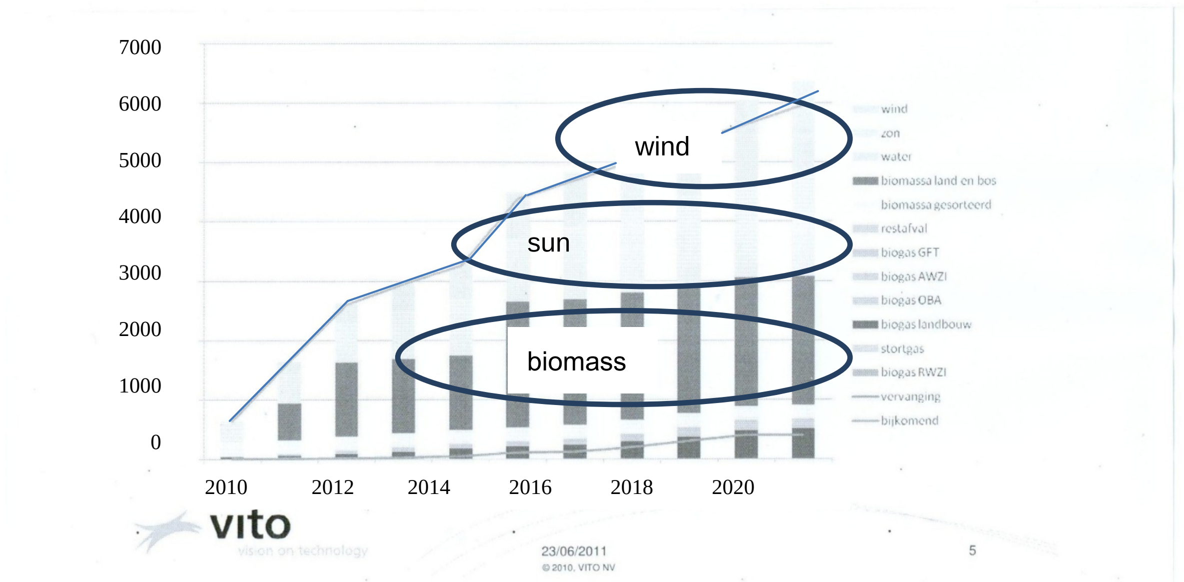

FIGURE 5 **Scenario for new green power potential in Flanders 2010-2020 (in GW) (VITO, 2011)**

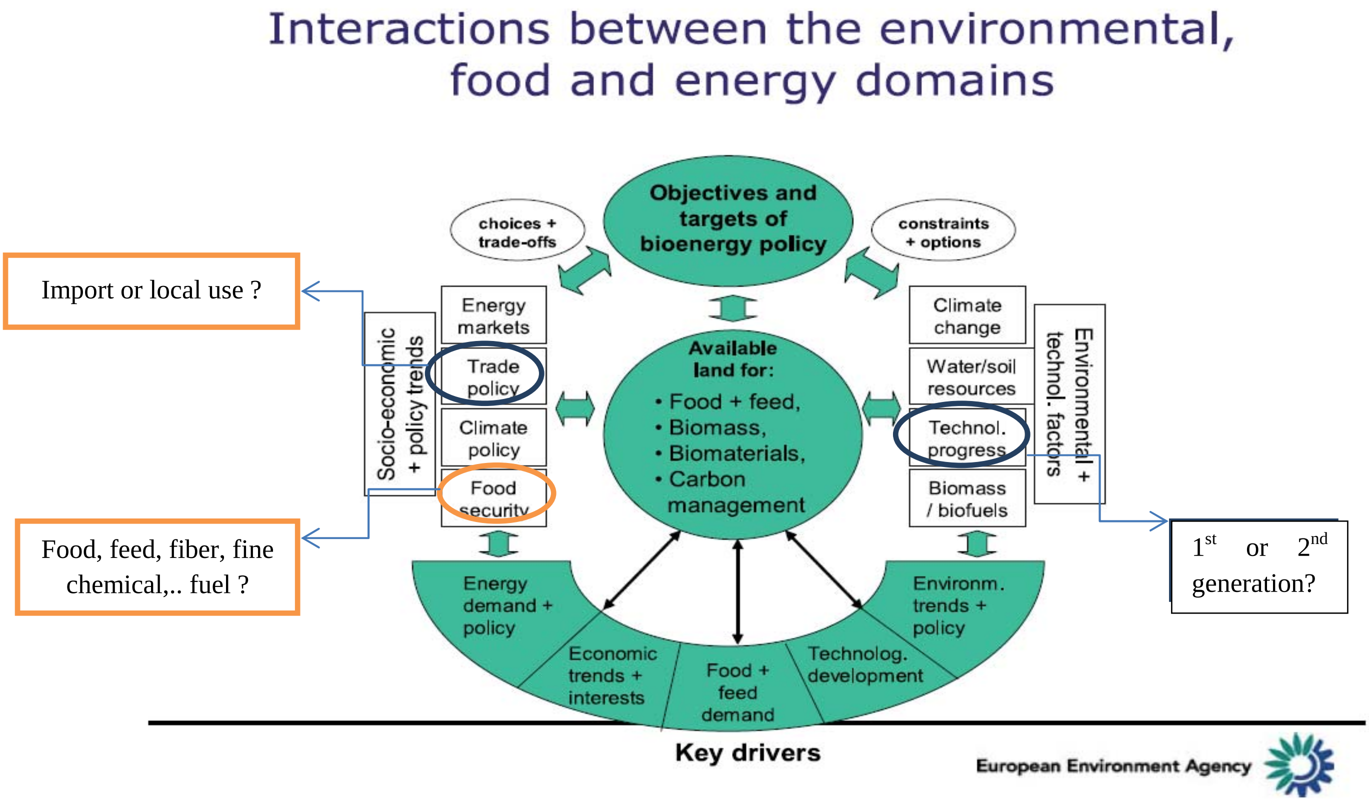

FIGURE 6 **Interactions between the environmental, food and energy domains (EEA)**

In waste policy an important target is the treatment of wet biomass (low energy potential) in the organic-biological cycle to a useful product, and what rests using for energy. Using the best available technology with not exceeding excessive costs means at the moment that for composting you need around 20% wood residues (prunings). Reaching good quality of the end products of biological treatment (e.g. of the pelletised digestates) will always be a key driver, in order that biological nutrients can stay in the organic-biological cycle. 90% of the food industry sludge is of good quality and is recycled/treated in a biogas plant before using in agriculture. Direct and indirect Land use change and greenhouse gas emission reduction stay important sustainability criteria for locally produced as well as imported biomass. More as 50% of the wood pellets have to be imported in order to reach the renewable energy targets in Flanders.

5 CONCLUSIONS

The biobased economy policy in Flanders is growing on the combination of import and locally produced biomass for all applications. This leads step-by-step to a more optimized economic and ecological situation, less depending on the variability of the world market of fuel and biomass.

The success of this sustainable cradle-to-cradle-policy and green economy in the Flemish part of Belgium is due to the following:

- a long term coherent and clear policy and framework stimulating a sustainable management of resources e.g. the treatment of locally produced biomass and use of local separate collected biowaste,
- supported by a mix of different instruments: e.g. sensitisation, different support for building treatment facilities, green power certificates for producing electricity by biomass digestion and for producing electricity by wastewood combustion and by incineration of not recyclable biowaste.

Our Flemish government aims a greener economy based on closing the material and energy cradle until 2014.

Not only renewable energy targets but also targets and green deals with the stakeholders for the use of biomass as food, feed, material,.. are necessary in all countries in order to reach a sustainable level playing field. No time to waste. Let’s start.

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(224) PROVISION OF A WEB TOOL ON BIO-WASTE PREVENTION FOR LOCAL AUTHORITIES IN EUROPE

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EXECUTIVE SUMMARY

The European community cannot address the issue of bio-waste solely in terms of collection and waste management, as such activities generate costs. Today, waste prevention in Europe must involve waste reduction and prevention at a local level, targeting all producers of bio-waste, not only consumers and households, and promoting the use of a number of waste reduction practices including composting, mulching, grinding, use of slow-growing plant species, using bio-waste as animal feed, reducing food wastage and the use of ramial wood chips.

The Miniwaste project, supported by the European Life+ program (2010-2012), aims to provide evidence that it is possible to significantly reduce the production of bio-waste generated in the European Union, as well as provide tools and points of further reflection on bio-waste prevention.

The partnership (Rennes Métropole, Irstea, City of Brno, LIPOR in Porto, and ACR+) wishes to set up a computerised tool for local authorities within Europe. This project will help users to conduct a bio-waste management programme including territorial diagnosis, the implementation and monitoring of specific preventive actions and the evaluation of these actions.

The overall structure will be based on three different modules:

1-A decision making assistance module that includes a territorial diagnosis chart offering several scenarios associated with different potential possible actions in the field of bio-waste prevention:

Composting (home, community, apartment blocks, vermi-composting, catering services), food waste (household, restaurant), smart gardening and animal feeding.

The territorial diagnosis is produced on the scale chosen by the user in order to get homogeneous data. It could be a neighbourhood, district, town, etc.

For a given sector, the decision making module consists in entering data of 15 territorial indicators in terms of population, current waste management and current preventive actions on the territory, etc. Meanwhile, a calculation of potential waste reduction is processed by the computer using an algorithm.

The result of the diagnosis is displayed for the whole territory and for each sector according to the list defined. Corresponding to the results, synthetic worksheets are proposed as a real decision making tool with the following information: target, type of waste concerned, linked indicators, main actions, participants involved, materials necessary, personnel necessary, an estimate of cost, and expected results.

With such information (diagnosis and worksheets), the users are able to choose one or several preventive actions to implement in their territory.

2-Some guidelines to implement the actions and especially some data sheets proposing activity indicators and impact indicators: depending on the preventive actions chosen, the user can download the corresponding procedural sheet which explains step by step how to implement the actions in detail. Moreover, a data sheet is available with a list of relevant indicators (awareness, number of composters, etc.)

3-A results display module, producing graphs to help visualise the results.

This web tool will be available on the Miniwaste website by subscription, from 2013 (www.miniwaste.eu). More information will be provided and a tool demonstration will be organized during the final conference scheduled in Rennes in November 2012.

1. INTRODUCTION

1.1 Background

The local authorities of Rennes Métropole (France), Brno (Czech Republic) and LIPOR (Serviço Intermunicipalizado de Gestão de Resíduos do Grande Porto, Portugal), together with ACR+, the European network on waste management, and Irstea, are joining forces in a common project (Miniwaste) in order to find, test and disseminate the appropriate tools to enable good implementation and monitoring of organic waste reduction actions.

This three year project was submitted to the European Life+ programme in November 2008.

1.2 Objectives of the Miniwaste project

The main objective of Miniwaste is to demonstrate, in accordance with the Directive 2008/98/EC, that it is possible to significantly reduce the amount of organic waste (food and green waste) at the source in a controlled and sustainable way.

This reduction will be obtained by limiting food waste and by encouraging the practice of group or individual composting of food and green waste (grass, leaves and small branches). For that, awareness is implemented in three different European states (France, Portugal, Czech Republic), both at the pilot (Brno) and larger scale (Rennes Metropole and Lipor), and to get a large part of the population involved in the organic waste reduction action.

A second objective will be to develop and implement a set of monitoring protocols in order to assess the quantity of composted waste and the quality of the compost produced in compliance with European standards and to compare the efficiency of different possible kinds of composting bins. The main idea here is to provide citizens and local authorities with flexible solutions well suited to their needs.

The last objective is to produce and disseminate recommendations and guidance notes for other European cities, showing the easy duplication of the Miniwaste successful and original actions for organic waste minimisation (namely separate collection at the source by motivated citizens enabling the amount of waste to be processed to be reduced and high quality compost to be produced in an economic and eco-friendly manner).

1.3 Objective of the computerised tool

Experience shows that local authorities do not have enough structured technical resources at their disposal to design and implement an appropriate waste reduction management plan and prevention programme as well as an effective control system using measurable indicators to monitor whether the objectives are met. The objective of the computerised tool is to fill this gap by implementing a knowledge and feedback database to identify relevant models of action and also to have at one’s disposal measurable indicators of targets, actions and results.

2. METHODOLOGY

2.1 Defining the functions of the tool

The partners discussed the overall structure of the IT tool based on three different modules:

- A decision making assistance module that includes a territorial diagnosis chart comprising several scenarios suggesting different possible actions in the field of bio-waste prevention.
- An implementation and monitoring module including a variety of indicators.
- A results display module, producing graphic displays to help visualise the results.

In order to define precisely the functions of the tool, a workgroup comprising Rennes Métropole (both the project team and the computing department) and Irstea (responsible for the protocols whose results will be integrated within the tool) met on October 2010.

This meeting was able to:

- Define the indicators to be integrated in the tool as well as methods of calculating the values.
- Propose a method for the creation and implementation of the tool.

A second meeting with the whole partnership was organised in order to:

  - Validate the work of the previous meeting,
  - Define the structure of the tool per module,
  - Discuss ways to integrate the results of the protocols established by Irstea

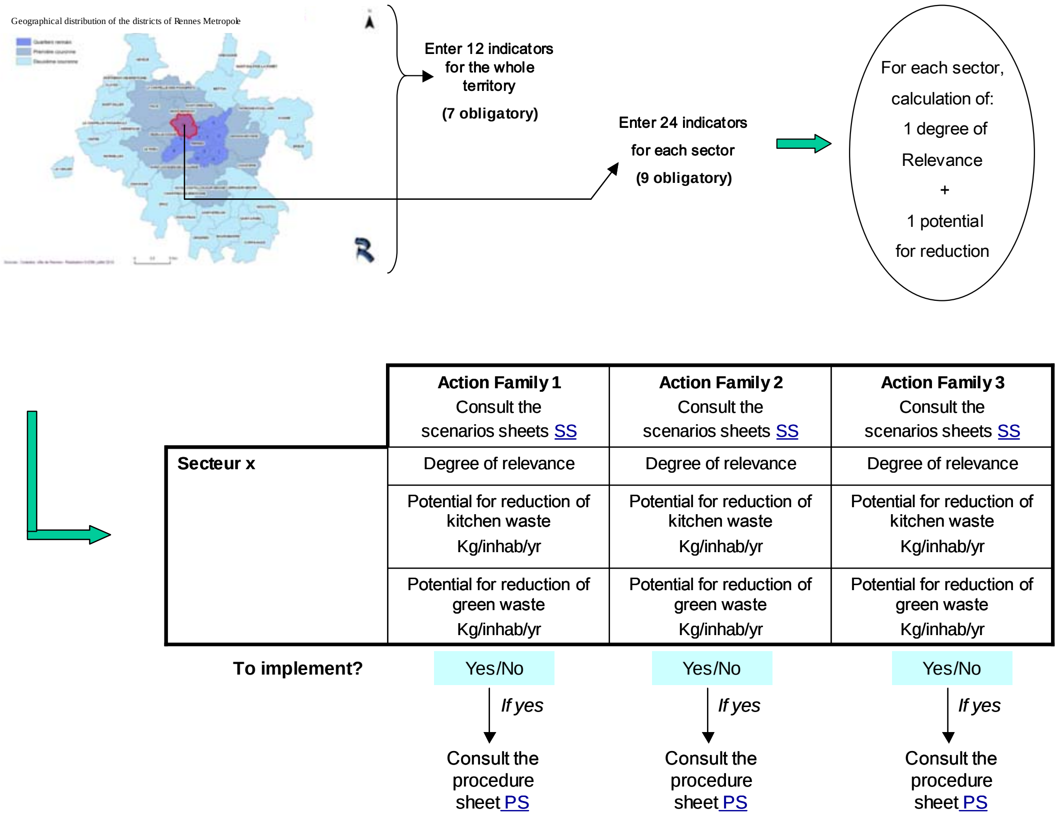
2.2 Building of computing tool

Rennes Métropole's team drew up the specifications for a public tender which was launched in January 2011. The objective of this consultation was to define European indicators in terms of information and calculations in order to monitor composting operations from the beginning to the end.

On the basis of defining European indicators in terms of information and calculations, a second consultation was launched in order to build the computerised tool that will take these items into account.

The figure 1 gives the global functioning of the module 1:

- a territory diagnosis with entries of indicators
- a display of results by sector with links to procedure sheets (PS) e.g entry to module 2



	Action Family 1 Consult the scenarios sheets SS	Action Family 2 Consult the scenarios sheets SS	Action Family 3 Consult the scenarios sheets SS
Secteur x	Degree of relevance	Degree of relevance	Degree of relevance
	Potential for reduction of kitchen waste Kg/inhab/yr	Potential for reduction of kitchen waste Kg/inhab/yr	Potential for reduction of kitchen waste Kg/inhab/yr
	Potential for reduction of green waste Kg/inhab/yr	Potential for reduction of green waste Kg/inhab/yr	Potential for reduction of green waste Kg/inhab/yr
To implement?	Yes/No	Yes/No	Yes/No

Note:
5 action families described in 5 procedure sheets PS1 to 5
7 types of action for households described in scenario sheets SS 1 to 7
4 types of action for non-households described in scenario sheets SS a to d.

FIGURE 1: Block diagram of tool

2.3 Transferability of the tool

A preliminary version of the IT tool will be tested by the 3 partner authorities of the project and other local authorities in Europe who have volunteered to test it with their own local data.

At the end of the Miniwaste project, the final version of the tool will be made available for all European local authorities.

3. RESULTS AND DISCUSSION

3.1 General features and constraints of the tool

The principles of implementation adopted by the partners in the project during the preparation meetings are:

- To design the tool to be multilingual; it will be available in English, French, Czech and Portuguese.

- A fully configurable tool (ability to translate headings, changing the reference values, thresholds and scales, changing the calculation formulae, etc.).
- Fully self-contained use by any European community wishing to use it: it was therefore decided to have a downloadable tool developed on the Miniwaste site which could then be used offline.
- The tool will be based on tools of the spreadsheet type with very wide international coverage, this choice enabling it to be modified to give access to the largest number of users.
- The tool will be developmental, and new data sheets will expand it as experiments are conducted.

The tool will have effective help available in both the initial versions (English and French) and other languages will be added in the future.

3.2 The functions of the tool

3.2.1 A decision making assistance module.

Module 1 of the computerised tool provides help in the decision making phase. It consists of two parts, the entered part, then the action recommendation part which will display scenario sheets suitable for the territory.

Entering the indicators

To establish the diagnosis, the user must enter the 12 territory indicators (of which 7 are obligatory) that concern the geographic and demographic factors, the quantities of waste collected, and the initial state of the community concerning the prevention of bio-waste.

He must then enter the 24 indicators (of which 9 are obligatory) for each of the sectors on which he wishes to work from the following categories of indicator:

population
economic activity
data on waste collected (quantities, method of collection used, characterisation)
type of housing
characteristics of gardens
behaviour of the inhabitants (e.g.: initial state of domestic waste management, potential for equipment)

The smaller the geographical boundary, the more refined the diagnosis will be and the more recommendations will be relevant.

The user will have information to aid entry (info-bubble, comments) telling him the unit or type of information expected as well as information on how to obtain this data (source column).

Some of these indicators are obligatory and others are optional; a calculated default value must sometimes be used if the user has not entered anything (e.g.: calculations based on the French national MODECOM).

When entering data, in the case of selective collection of green waste or food waste, a message will alert the user, saying: “A selective collection may harm the actions for the promotion of domestic management of this waste”.

The entry will also be checked, for example by comparing the total quantity of waste collected by the community with the sum of the various methods of collection.

An error message will invite the user to correct his entry if it is inconsistent.

3.2.2 The decision algorithm

This algorithm is calculated on the scale of one sector and is used to determine a degree of relevance for each action and a potential for the reduction of waste.

Calculating the degree of relevance

For each sector, a degree of relevance is calculated for actions intended for households:

composting in an individual house
community composting for individual houses
animal food in an individual house
shared composting in an apartment block
vermicomposting
sustainable gardening for households
food waste in households (this action is not the subject of a degree of relevance)

The degree is obtained by means of a list that allocates points by comparing the indicator value entered with the reference values defined by the partners that can be changed by the user if required.

For example, a ‘population density’ indicator of less than 300 inhabitants/km² will give one point for an ‘individual composting’ action and 0 point for ‘group composting’ action while on the other hand a density of more than 2000 inhabitants/km² will give 0 point for ‘individual composting’ and 1 point for ‘group composting’.

The total degree of the action is obtained by dividing all the points thus obtained by the maximum potential points for the action in order to weight it if all the indicators are not entered.

An indicator is considered to be entered even if a default value is calculated (optional indicator).

The degree of relevance is then calculated by action family and is equal to the average of the degrees of each action of the family.

The 5 action families that correspond to the 5 procedure sheets (PS) of module 2 are:

PS 1. domestic waste management in an individual house
PS 2. domestic waste management in an apartment block
PS 3. domestic management of green waste
PS 4. food waste in households
PS 5. prevention of bio-waste from non-household producers (not the subject of a degree of relevance)

Actions intended for non-households (the 5th family) are considered relevant actions since the corresponding relay structures exist in the sector studied. This information is obtained from the type indicators: in this sector, is there a food shop (a garden centre), etc.?

The action family for non- households consists of 4 types of action corresponding to 4 scenario sheets (SS):

SS a. composting in group catering
SS b. food waste in a restaurant
SS c. food waste in food shops
SS d. relaying information by sustainable gardening, composting, animal feed by gardeners and gardening clubs.

potential for reduction

The potential for reduction of green waste and kitchen waste is calculated by family of action (see list above).

It is calculated on the basis of the quantity of waste collected by the community.

If the community carried out a campaign to characterise its Residual House Waste (MODECOM), the percentage of kitchen waste of RHWs was expressly entered, if not, the rates of the national MODECOM will be applied to the total quantities collected.

This enables the quantity of green waste and kitchen waste produced to be calculated.

The quantity potentially avoided is then obtained by applying the percentages of reduction for kitchen waste on one hand and green waste on the other hand, according to the following table 1:

TABLE 1 : Potential for reduction depending in the family of actions

% reduction	Domestic waste management in an individual house	Domestic waste management in an apartment block	Domestic management of green waste	Food waste
Kitchen waste	70%	70%	-	100% of food not consumed
Green waste	30%	-	70%	-

The potential for reduction for the bio-waste prevention family for non-household producers was not calculated.

This algorithm may be refined depending on the results of the experiments conducted.

Displaying the results of the diagnosis

A summary will give recommendations for each sector.

A double entry table (sector/family of action) will show the degrees and potential for green waste and kitchen waste reduction by sector and by family of action, like the example in table 2:

TABLE 2: Type of display of the results of the territory diagnosis for one sector

	Action Family 1 Consult the scenarios sheets SS	**Action Family 2** Consult the scenarios sheets SS	**Action Family 3** Consult the scenarios sheets SS
Secteur x **% of total inhabitants** **% of whole territory**	Degree of relevance	Degree of relevance	Degree of relevance
	Potential for reduction of kitchen waste Kg/inhab/yr	Potential for reduction of kitchen waste Kg/inhab/yr	Potential for reduction of kitchen waste Kg/inhab/yr
	Potential for reduction of green waste Kg/inhab/yr	Potential for reduction of green waste Kg/inhab/yr	Potential for reduction of green waste Kg/inhab/yr

In order to make the extent of the actions undertaken relative, the percentage of inhabitants in the sector in relation to the total for the territory will be announced.

SS1, SS2, … SSd are links for displaying the scenario sheets in PDF format.

In the case of a non-household producer family, only the sheets corresponding to the participants present in the sector are offered (replies to the questions in the ‘presence of xxx’ diagnosis).

Depending on the actions chosen, the user will have free access to the procedure sheets (PS) by means of the links given in the table of results.

3.2.3 Guidelines for implementing the actions

Module 2 is a tool for carrying out and monitoring actions; it is independent of module 1 and the user can use it directly without having established the territory diagnosis.

If the territory diagnosis has been carried out, the data corresponding to the initial situation will be taken from module 1; otherwise they must be entered.

The user must periodically enter the items used to quantify the progress of the actions taken and the data concerning the quantities of waste collected.

A data chart can be used to monitor the indicator values, whether they are indicators of the quantity of waste collected, or indicators used to measure the actions implemented or the rate of progress in relation to a defined objective.

3.2.4 The results display module

Module 3 plots the results obtained by completing the data chart of module 2.

Each indicator will be associated with a graph to display the results, like the examples in figure 2.

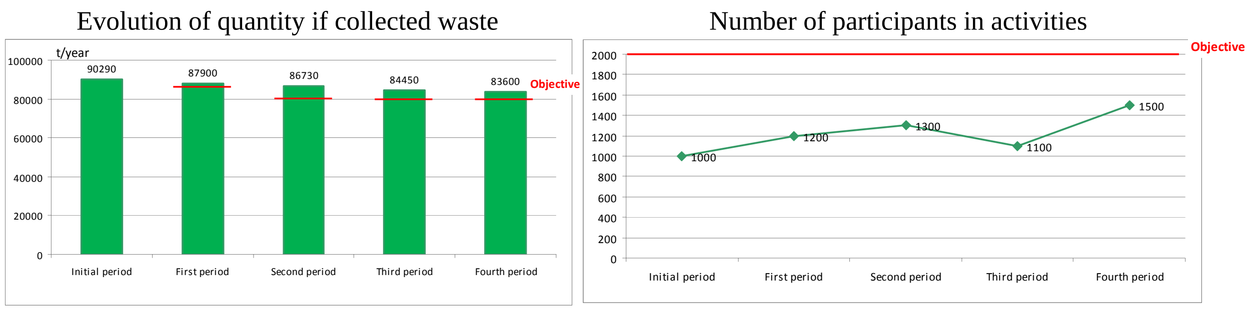


FIGURE 2: Examples of graphs of the results display module

4. CONCLUSIONS

This computer tool encompasses all the tools developed by the partners in the Miniwaste project:
the collection of best practice data sheets concerning the prevention of bio-waste,
the communication tools developed,
protocol sheets to evaluate the preventive actions taken in the territories,
certain ratios identified by the measures taken for territories of the partnership in order to monitor the impact of the actions.

A methodological guide will be made available on the site of the Miniwaste project at the end of the project to guide users in setting up and monitoring the actions (miniwaste.eu).

Session 9

(125) POTENTIAL OF PRODUCING BIO-ETHANOL FOR USE AS E10 IN TRANSPORTATION SECTOR FROM LOW COST LIGNOCELLULOSIC GREEN WASTE IN MAURITIUS

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EXECUTIVE SUMMARY

Bio-ethanol production from biomass is attracting attention all over the world in view of its use as an alternative source to petrol or in blends with petrol for clean energy technology in the transportation sector. The commercial feasibility of bio-ethanol production from locally available renewable lignocellulosic resources depends both on its ease of availability and its low cost. Moreover, with the intensive urge in having a clean environment for the present and future generation, the Government of Mauritius has adopted a strategy of Building a Green future for Mauritius through the Maurice Ile Durable (MID) concept through a shift to renewable sources of energy from imported fossil fuels. Among the strategies, is the concept of E10, that is, using 10% of bio-ethanol blend with gasoline in the transportation sector in all vehicles in Mauritius by end of 2012. Mauritius being an agricultural island has a great potential for easily available lignocellulosic biomass. Sugar cane cultivation occupies the highest function in the agricultural sector, with more than 70% of arable land under cane in Mauritius. The major biomass formed is bagasse, which is currently being burnt to produce energy. Furthermore, the molasses formed are already used in Mauritius for bio-ethanol manufacture. However, molasses being limited, other sources such as agricultural wastes (700 tons/day) which are highly abundant in the local context were being looked into. Thus, to be in line with the Government strategy set out on E10 for 2012 and to seek environmental-friendly alternatives in waste disposal, bio-ethanol production potential were investigated from low-cost local feedstock through the acidic hydrolysis. Five types of feedstock from waste streams were studied: peels of cane stalk, cane tops and leaves, elephant grass, coconut husks and acacia residues. One of the major challenges in hydrolysis technologies is to optimize the combination of process engineering, fermentation technology and metabolic engineering. Consequently, this study is based also on investigating the optimum conditions required for acidic hydrolysis to attain highest bio-ethanol yield for each feedstock. During the dilute acid hydrolysis, the feedstock was hydrolysed at 121 ^{0}C in an autoclave at atmospheric pressure using different concentrations of dilute Sulphuric acid at various reaction times, to obtain the available sugars which were then fermented at optimal conditions. Maximum yields of 145 Liter bio-ethanol per tonne cane stalk hydrolyzed for 1 hour at 2 % sulphuric acid, 118 Liter bio-ethanol per tonne cane tops and leaves hydrolyzed for 3 hours at 4 % sulphuric acid, 104 Liter bio-ethanol per tonne elephant grass hydrolyzed for 3 hours at 4 % sulphuric acid, 87 Liter bio-ethanol per tonne acacia hydrolyzed for 1 hour at 2 % sulphuric acid and 82 Liter bio-ethanol per tonne coconut residues hydrolyzed for 2 hours at 2 % sulphuric acid were obtained. It was concluded that lignocellulosic biomass such as cane tops and leaves, elephant grass, Acacia leaves which are normally thrown away have a very good potential of being converted to bio-ethanol which can be used as E10 blends as transportation fuel in Mauritius to meet the demand set out, by end of 2012.

1 INTRODUCTION

With the ever increasing concern over the speed at which fossil fuel reserves are being depleted and the damage that burning them does to the environment, the development of sustainable fuels has become an increasingly attractive topic. In this spectrum, alcohol manufacture from biomass has attracted a large attention all over the world which could be used as an alternative source to petrol or in blends with petrol (Chandrakant and Bisaria, 1998; Gong, 1999). Moreover, with emerging environmental concerns, the government of Mauritius has set a commitment to reduce its dependency on fossil fuels in the recent Budget (Budget Report, 2008). As, the automotive industry is a major consumer of fossil fuels, the government of Mauritius has mounted a national framework so as to promote the use of renewable energy while reducing the import of conventional fuels by boosting the extensive use of ethanol in motor vehicles. So far, ethanol has proven to be a very reliable auto motive fuel in countries like Brazil, Canada, India and the United States. And today, gasoline replacement by liquid ethanol produced from renewable resources is also becoming a high-priority goal in many other countries and is the only proven commercial-scale renewable transportation fuel that has the potential to replace at least 10 percent of the nation's gasoline supply (Dawson and Boopathy, 2006). According to Berg (1999) and Hahn-Hagerdal et al (2001), ethanol blends of 15 – 20% can be used in automobile engines with essentially no engine modifications. The commercial feasibility of ethanol production from locally available renewable lignocellulosic resources depends both on its ease of availability and its low cost (Nigam, 2000). Hence, locally produced renewable fuel; ethanol, has the potential to broaden the energy portfolios, lower dependence on foreign oil and to improve trade balances in oil-importing nations (Cheng, 2001). Thus, starting with available resources in Mauritius, a list of the most potentially promising crops was developed which consisted of cane tops and leaves, elephant grass, acacia, coconut husk and peels of cane stalk.

1.1 Background

Lignocellulosic biomass such as agricultural, forest products (hardwood and softwood) and their residues are renewable resources of energy (Wyman, 1996). Approximately 90% of the dry weight of most plant material is stored in the form of cellulose, hemi-cellulose, pectin, and lignin. Conversion of cellulose and hemi-cellulose from waste materials to sugars provides a feedstock for the production of fuel ethanol and substantially reduces the amount of wastes that would otherwise exert pressure on municipal landfills (Jeffries, 1994). The production of ethanol from lignocellulosic biomass results in a no net contribution to global warming, since the carbon dioxide produced by the combustion of ethanol is consumed by the growing raw material (Sivers & Zacchi, 1995; Galbe et al, 2005).

1.2 Research Objectives

The objectives of the research consisted of determining the optimal conditions for concentrated acid hydrolysis and dilute acid hydrolysis to produce bio-ethanol and to compare both technologies.

1.2.1 Concentrated acid hydrolysis technology

The primary benefits of concentrated acid processes are that they can convert a wide variety of biomass feedstocks into sugars for fermentation into ethanol (Goldstein and Easter, 1992). Moreover, as concentrated acid hydrolysis normally takes place at low temperatures and pressures, they tend to minimize the degradation of sugars (Goldstein et al, 1983). As a result, concentrated acid processes achieve high yields of ethanol vital to economic success. Hydrolysis of cellulose with almost 100 % yield are normally achieved with 40 wt% hydrochloric acid, 60 wt% sulphuric acid or 90 wt% hydrofluoric acid at around 100ºF for 2 to 6 hours in a hydrolysis reactor (Grohmann et al, 1985). However, concentrated acid hydrolysis requires large amount of acid which is very costly, thus for this technology to be economically viable, a considerable amount of these acids have to be recovered.

1.2.2 Dilute acid hydrolysis technology

Dilute acid hydrolysis is known to degrade lesser amount of the fermentable sugars to fermentation inhibitors (furfural, and hydroxymethyl furfural) than do other methods (Nguyen et al; 1998). It uses low concentration acids of the order of 1-5% and high temperatures in the range of 160 – 250°C to process the lignocellulosic biomass within seconds or minutes. Hemicellulose are normally broken down at temperatures of around 160°C (320°F) to form principally xylose and arabinose. The remaining solids that is, cellulose and lignin, go through a second hydrolysis stage where the cellulose is converted to glucose with approximately 2 % acid in liquid at up to 240 ^{0}C (Nguyen et al., 1998). This results in a mixture of sugars with xylose as the major component which can be effectively converted to ethanol. However, these high temperatures are severe enough to degrade xylose and glucose into hydroxymethyl- furfural (HMF), which in turn degrades to form tars and other undesirable co-products (Burnhardt and Ingram, 1992) resulting in a subsequent ethanol yields of around 50-60% (Wright, 1983; 1988).

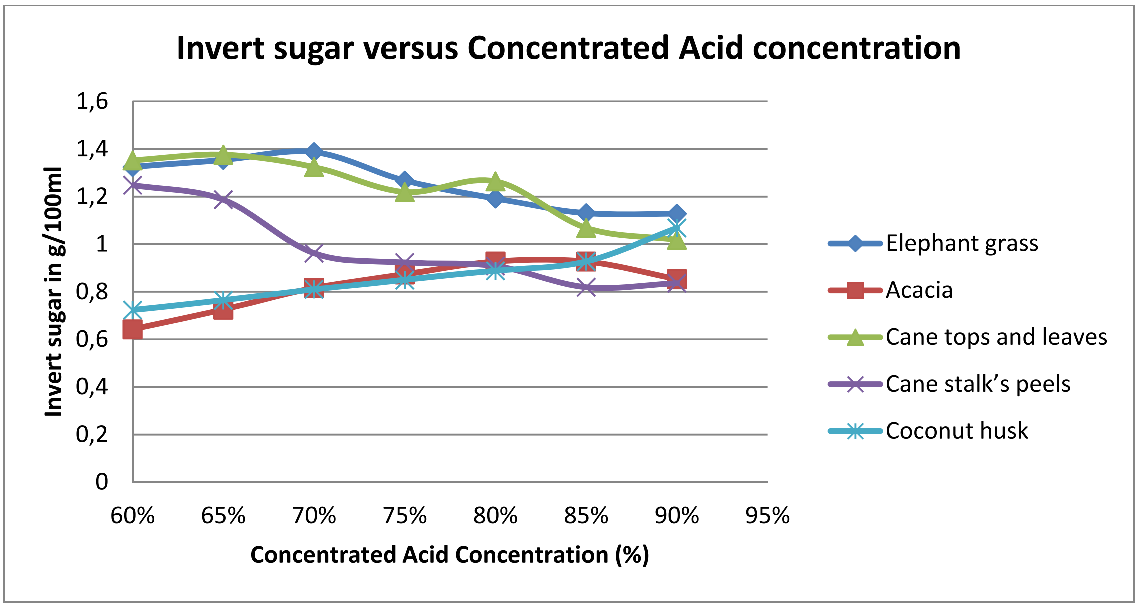
2 METHODOLOGY

For the purpose if this study, five different feedstock were used namely cane tops and leaves (CTL), Acacia leaves, elephant grass (EG), coconut husk (CH) and peels of cane stalk (PCS). These were hydrolyzed under concentrated acid and dilute acid and thereafter fermented to determine the optimum conditions for acidic hydrolysis technology for each feedstock to attain maximum ethanol yield.

3 RESULTS AND DISCUSSIONS

3.1 Concentrated acid hydrolysis of lignocellulosic feedstock

3.1.1 Optimum concentration for concentrated acid



* Values are for Invert sugar in g/100ml (by Lane & Eynon Method)

Figure 1: Fermentable sugars from concentrated acid hydrolysates

ANOVA analysis was performed to determine the most favorable acid concentration. However, the analysis revealed that an increase in acid concentration resulted in an insignificant increase in amount of fermentable sugar being released as the p value was greater than 0.05 (p = 0.792). Consequently, the lowest concentration of 60 % was chosen as from an economic point of view with the feedstock elephant grass giving the highest yield.

3.2 Dilute acid hydrolysis of lignocellulosic feedstock

3.2.1 Optimum dilute acid concentration

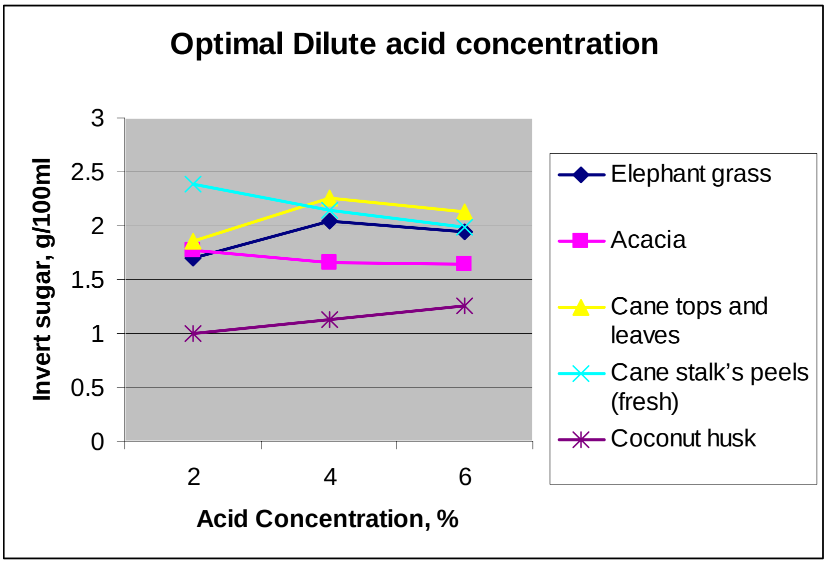
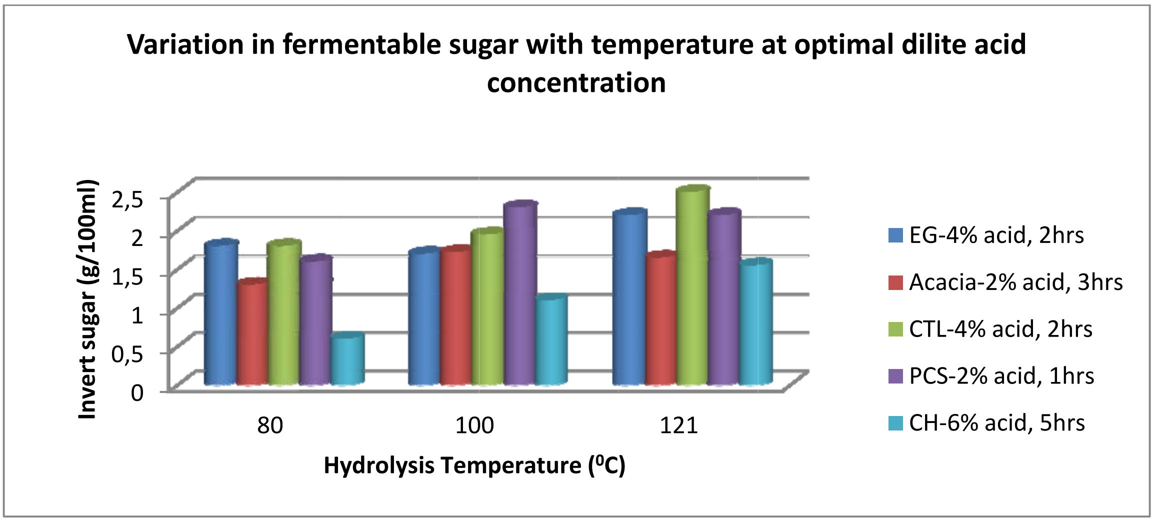


Figure 2: Fermentable sugars from dilute acid hydrolysates

The optimum acid % for elephant grass and CTL were found to be 4 %, that of acacia and peels of cane stalk were 2% and that of coconut husk was 6 %. The highest amount of sugars released was from peels of cane stalk followed by cane tops and leaves, elephant grass, acacia and finally coconut husk. This is due to the fact that peels of cane stalk already have some readily available sugars which required a lower concentration of acid to break the cellulose and hemicelluloses bonds. As regards CTL and elephant grass, higher acid concentration was required to obtain the best yield as the fermentable sugars are more difficult to access. For coconut husk, an even higher concentration of 6% was needed to obtain highest amount of sugars as it has wood like structure which makes accessibility to the fermentable sugar even more difficult. ANOVA single analysis for all the feedstock gave a p value < 0.05 implying the ratio of change in acid concentration to increase in fermentable sugar liberated is significant.

3.2.2 Optimum hydrolysis temperature

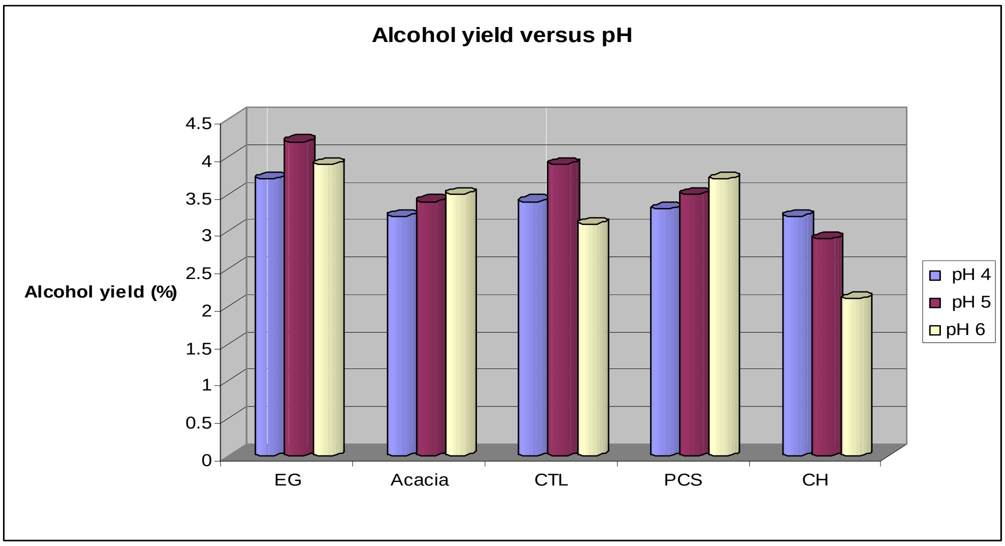


(EG-Elephant Grass, CTL- Cane tops & leaves, PSC- Peels of cane Stalk, CH- Coconut Husk)
Figure 3: Variation of Fermentable sugars with Temperature

When hydrolyzed at temperatures below 121^{0}C, a lower amount of sugars were released from the respective feedstocks EG, CTL and CH. This was due to a lower percentage of degradation of cellulose and hemicelluloses at lower temperatures indicating that, higher thermal energy is required to break the non-lignin fraction. In contrast, a decrease in sugar content was observed for Acacia and PCS when hydrolyzed at 121 ^{0}C as compared to 100 ^{0}C which was caused by the thermal degradation of the additional sugar released into undesirable noxious products resulting in a decreased value of sugar content.

3.3 Optimal fermentation conditions

3.3.1 Optimum pH



(EG-Elephant Grass, CTL- Cane tops & leaves, PSC- Peels of cane Stalk, CH- Coconut Husk)

Figure 4: Variation of bio-ethanol yield with pH

Highest alcohol yield was obtained at pH of 5 for EG– 4.2 % and CTL– 3.9% after 48 hours of fermentation, pH of 6 for Acacia– 3.5 % and PCS– 3.7 % after 72 and 48 hours of fermentation respectively and pH of 4 for CH– 3.2 % after 48 hours of fermentation. These pH values were subsequently used for the rest of the fermentation process. The lignocellulosic feedstock EG obtained the maximum ethanol yield of 4.2 % when fermented for 72 hours from concentrated acid hydrolysates. As regards the concentrated acid hydrolysates from CTL, PCS and CH, they reached their highest ethanol concentration after 48 hours of fermentation at 3.9 %, 3.7 % and 3.2 % respectively.

The topmost ethanol % obtained was from the biomass CTL (6.7 %) followed by PCS (6.4 %), EG (6.1 %), Acacia (4.9 %) and CH (4.0 %). It has been found that a fermentation period of 72 hours was required to reach the highest yield for all the lignocellulosic biomass except for PCS that needed a fermentation period of only 24 hours. Thereafter, a decrease in the yield by 4.41 % ad 6.15 % was observed with 48 hrs and 72 hours of fermentation respectively which was due to fungal invasion detected by formation of white patches.

4 ACKNOWLEDGEMENT

The authors are very grateful to the Danisco Division for providing the corresponding author with the enzyme ACCELERASE 1000.

5 CONCLUSION

The ethanol yields obtained from the various technologies were different from each feedstock. This implies that different feedstock behaves differently with changing hydrolysis technology. This may be due to the structure of the lignocellulosic feedstock where the cellulose and hemicellulose fraction some feedstock are readily reachable with just dilute acid whereas others needed a more intense treatment to reach their non-lignin structure. For concentrated acid hydrolysis, the feedstock EG gave the highest ethanol yield, while that from dilute acid hydrolysis was CTL. The ethanol yield from dilute acid hydrolysis was 1.6 times, 2.1 times, 2.3 times, 2.3 times and 2. 6 times that of concentrated acid hydrolysis for the lignocellulosic feedstock EG, Acacia, CTL, PCS and CH respectively. Out of the two technologies, the dilute acid hydrolysis technology produced the most ethanol. In contrast, concentrated acid hydrolysis is known to give the highest ethanol yield which was not the case in this study. Moreover, it can also be concluded that, out of the five types of feedstock utilized for study, PCS (145 L/ ton dry biomass)and CTL (118 L/ton dry biomass) have been found to be the most favorable lignocellulosic biomass for both acids hydrolysis technologies both producing. Since Mauritius is an agricultural land and 70 % of the land is under cane cultivation, producing bio-ethanol via acidic hydrolysis for use as a fuel blend is very promising and the target set for end of 2012 can be easily met in the near future.

FIGURE

Figure 1: Fermentable sugars from concentrated acid hydrolysates
Figure 2: Fermentable sugars from dilute acid hydrolysates
Figure 3: Variation of Fermentable sugars with Temperature
Figure 4: Variation of bio-ethanol yield with pH

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(155) ETHANOL PRODUCTION USING COMPLEX ANAEROBIC INOCULUM: EFFECT OF PH ON THE FERMENTATION PROFILE OF GLUCOSE AND XYLOSE

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EXECUTIVE SUMMARY

The regulatory and political context in France and Europe gives strong incentives for the development of transportation biofuels during the ten coming years (EU Directive 2003/30/CE). The environmental benefit of using first generation biofuels (agro-fuels) is however questionable (Kalogo *et al*., 2007). In this framework, the development of bioethanol from other organic resources such as household waste has been reported to be more economically and environmentally attractive. We are therefore working on the coupling of an ethanol production reactor to existing anaerobic digestion processes. Household waste being a complex and heterogeneous matrix, yeast fermentation would require energy-intensive pretreatments. We have consequently focussed on ethanolic fermentation by complex anaerobic microbial communities which are able to cope with a wider range of substrates without pretreatment. In particular, lignocellulosic waste constitute a major fraction of household waste. It is composed of cellulose and hemicellulose. The first one is a polymer of glucose (C6) and the second one contains many different sugar monomers, xylose (C5) being present in the largest amount. In this study, we focus on the influence of pH, being reported in the litterature as a key element for ethanol production by mixed anaerobic cultures (Ren *et al.* ,2001 and Temudo *et al*., 2007), on the fermentation profile of these two substrates.

Experiments were carried out in batch with glucose or xylose under anaerobic conditions. A concentration of 4 g/L was used for the two sugars. We have tested six different pH, in triplicate (ranging from 4.5 to 7). We have shown that the production of ethanol was possible from glucose and xylose. For each sugar, the greatest ethanol concentration was obtained at pH 7 at day 2: around 500 mg/L which represents 12.5% of the initial carbon. From day 3, ethanol is degraded and at day 7 no ethanol can be quantified anymore. We have also observed that ethanol degradation is slower for pH under 6. Moreover, according to pH, we have seen that the microorganisms' populations have evolved differently which could explain the differences in ethanol production and degradation. This knowledge could be very useful to adjust parameters to optimize ethanol production.

Ethanol production from simple sugar is one step of the development of biofuels from household waste. Experiments are under progress to study optimal pH operation strategies, evolution of microbial populations and their functionalities.

1 INTRODUCTION

The regulatory and political context in France and Europe gives strong incentives for the development of transportation biofuels during the ten coming years (EU Directive 2003/30/CE). The objectives of incorporation are 10% in 2020. French government has even taken stronger measures: 10% is targeted for 2015 (Loi d'orientation agricole 2006). The environmental benefit of using first generation biofuels (agro-fuels) is however questionable (Kalogo *et al*., 2007). Indeed, they require important surface areas for cultivation and large amount of water and fertilizers. In this case, the production of first generation ethanol would be in competition with the production of food for arable land use. Moreover, when the sugar market price is high, it is more profitable for farmers to produce sugar than ethanol.

This could have consequences on incorporation of biofuels rates: in France in 2010 the objectives were 7% and they reached only 6.3% notably because of sugar price. Concerning biofuels of second generation, that's to say using lignocellulosic materials, it appears that the treatments to release sugars for yeast fermentation are heavy and very expensive (Ogier *et al*., 1999). Furthermore, yeasts cannot ferment xylose to ethanol and so a part of ethanolic potential is lost.

The development of bioethanol from household waste has been reported to be economically and environmentally attractive. We are therefore working on the coupling of an ethanol production reactor to existing anaerobic digestion processes. Household waste being a complex and heterogeneous matrix, yeast fermentation would require energy-intensive pretreatments. We have consequently focussed on ethanolic fermentation by complex anaerobic microbial communities which are able to cope with a wider range of substrates without pretreatment. We have been particularly interested in lignocellulosic biomass because it is the main component of household waste: more than 50% of the organic matter in household waste (Barlaz *et al.*, 1989). It is composed of cellulose and hemicellulose. The first one is a polymer of glucose (C6) and the second one contains many different sugar monomers, xylose (C5) being present in the largest amount.

It has been reported in the litterature that pH is a key element for ethanol production by mixed anaerobic cultures (Ren *et al.*, 1997 and Temudo *et al.*, 2007), on the fermentation profile of these two substrates. However, different results have been obtained: in the first study, a pH of 3.5 has been defined as optimal whereas in the second one the optimum pH was 6.9. That's why, we have focused on the influence of pH in our study in order to find the adapted pH to produce ethanol from glucose or xylose in our system.

2 METHODOLOGY

2.1 Inoculum

The inoculum comes from a mesophilic anaerobic digestor treating municipal solid waste. It was sifted to remove big debris. Afterwards the inoculum has been stabilized anaerobically for one month and a half to exhaust remaining endogeneous degradable substrates. The degradation proceeded to carbon dioxide and methane (no dissolved VFA could be quantified at the end of the stabilization process). Finally, the sludge was centrifuged, aliquoted in 50 mL screw cap tubes, and pellets were stocked at -80°C until used.

2.2 Medium

In order to stabilize the pH, a phosphate buffer has been used (buffer strength 200mM). We have tested six different pH, in triplicate (ranging from 4.5 to 7). Nutrients were provided in the medium, according to the concentrations used in a standard BMP-test medium: NH_4Cl 0.53 g/L ; $CaCl_2,H_2O$ 0.075 g/L ; $MgCl_2,6H_2O$ 0.10 g/L ; $FeCl_2,4H_2O$ 0.02 g/L ; $Na_2S,9H_2O$ 0.1 g/L and $MnCl_2,4H_2O$ 0.5 mg/L ; H_3BO_3 0.05 mg/L ; $ZnCl_2$ 0.05 mg/L ; $CuCl_2$ 0.03 mg/L ; $Na_2MoO_4,2H_2O$ 0.01 mg/L, $CoCl_2,6H_2O$ 0.01 mg/L, $NiCl_2,6H_2O$ 0.1 mg/L, Na_2SeO_3 0.05 mg/L. Two different sugars were used at the concentration of 4 g/L (1.6 gC/L): glucose and xylose.

2.3 Experimental system

Inoculum (1.72 g, 35.65 %TS, 14.22 %VS, VS/TS : 39.89 %) and medium (140 mL) have been mixed in 330 mL glass bottles. Bioreactors were hermetically closed with a screw cap and a septum. Headspaces were flushed with molecular nitrogen at the beginning of incubation (O_2 percentage lower than 0.5). Reactors are placed at 35 °C ± 2 °C with 110-120 rpm agitation.

2.4 Analytical methods

2.4.1 Gas analysis

Head-space gas analysis is performed using a micro GC (CP4900, Varian). The gas measured were: O_2, N_2, CH_4, CO_2, N_2O, H_2S and H_2. To measure gas production, the biogas is assimilated to an ideal gas: between times t and t+1, biogas accumulates in the headspace. As the volume of the headspace is constant, the ΔP can be converted into a volume of biogas produced at standard pressure.

In the reactor:

$$P_h \times V_h = nRT_i \qquad \text{so} \qquad n = \frac{P_h \times V_h}{RT_i}$$

$$V_{pb} = V_m \times (n(t) - n(t-1))$$

P_h corresponds to the pressure in the headspace (mbar) ; V_h, volume of headspace (L) ; n, amount of gas (moles) ; R, gas constant (L.mbar.K^{-1}.mol^{-1}) ; Ti, temperature of incubation (K), constant at 35°C (308.15 K) ; V_{pb}, volume of biogas produced, V_m, molar volum 24.79 L.mol^{-1}.

All the results below are thus given at STP (Standard Temperature and Pressure) conditions: P = 1013.25 mbar and T = 0 °C (298.15 K).

2.4.2 Leachate sampling

Leachate samples are collected from the reactors twice a day (2 + 1 mL) through the septum with a syringe fitted with a 0.6 mm needle. The pH is immediately measured after sampling. The samples are centrifuged at 10 000 g for 10 minutes and the supernatant and the pellet are recovered and stored at -20 °C.

2.4.3 VFAs

VFAs concentrations are measured using ion chromatography (DIONEX DX 120, column IONPAC® ICE-AS1 (9*250 mm)).The mobile phases are heptafluorobutyric acid (0.4 mmol.L^{-1}) and TBAOH (5 mmol.L^{-1}). Acetic, propionic, butyric, lactic, formic and valeric acids can be quantified from 10 mg/L to 500 mg/L.

2.4.4 Ethanol

Ethanol is quantified using headspace gas chromatography – mass spectrometry (Trace GC Ultra and DSQ II from Thermo and the column used is TR-WAX : 30 m length, 0.25 mm intern diameter). The vial was sealed with a rubber cap and an aluminium crimp seal and incubated for 10 min at 90 °C. After this, 1 mL gas aliquot was withdrawn through the rubber cap and injected directly into the gas chromatograph (the syringe was equilibrated at 100 °C, in order to prevent condensation on the walls). Linearity and calibration curves were performed in water with ethanol standard to obtain concentrations of 0.1, 0.25, 0.5, 1, 5, 10 and 25 mg/L.

2.4.5 Analysis of bacterial diversity

Sample pellets were extracted with MOBIO kit (PowerSoil™ DNA Isolation Kit) and amplified with bacterial primers ITSF (5'-GTC GTA ACA AGG TAG CCG TA-3') and ITSR eub (5'-GCC AAG GCA TCC ACC-3') (Cardinale *et al.*, 2004) to target ITS between 16S DNA and 23S DNA sequences. PCRs were performed in a thermal cycler (Eppendorf ep silver, AG, Germany) in a reaction mixture containing, in a final volume of 25 µL : 2.5 µL of PCR buffer ; 0.15 µL of Taq polymerase (Thermo Start) ; 0.25 µL of each deoxynucleoside triphosphate (C=10 mM) ; 0.5 µL of each primer (C=10 µM) ; 1.5 µL of $MgCl_2$ (C=25mM); 1 µL of DNA. The mixture was held at 94°C for 15 min, followed by 35 cycles of 94 °C for 1 min, 55.2 °C for 1 min, 72 °C for 2 min and a final extension at 72 °C for 10 min. ARISA (Fisher *et al.*, 1999)) of the PCR product was carried out using the Agilent DNA 1000 Kit (Agilent Technologies Inc., CA) and then analyzed by an Agilent 2100 Bioanalyzer (Agilent Technologies Inc.).

3 GLUCOSE AND XYLOSE FERMENTATION

3.1 Biogas and leachate analysis

Thanks to the phosphate buffer we succeeded in stabilizing pH for glucose and xylose fermentations (range of 4.7 to 6.9). However we have observed a decrease of ½ unit pH between the first day and the end of incubation (Figure 1).

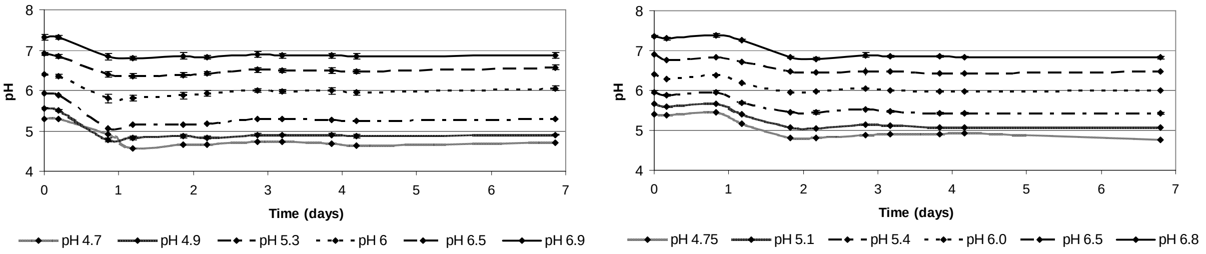


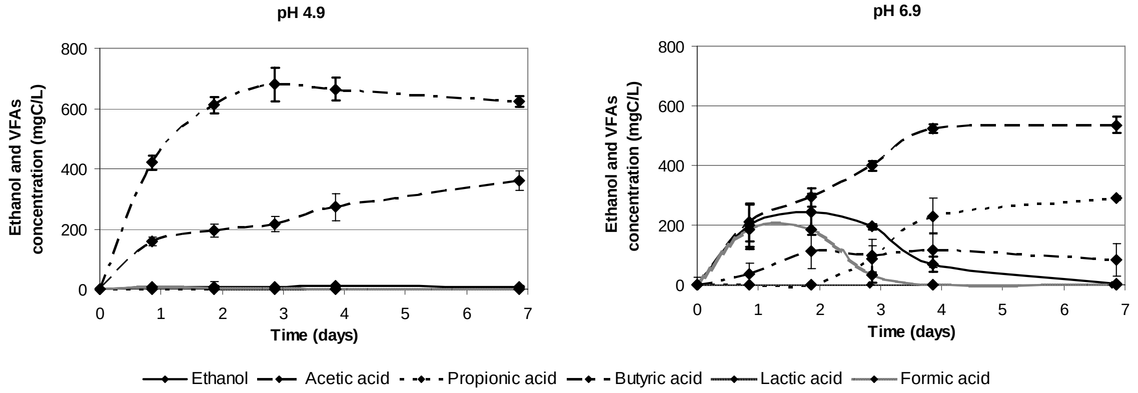
FIGURE 1 **pH stabilization during glucose (left) and xylose (right) incubations**

Thanks to the analysis of VFAs and ethanol two pH-dependent fermentative pathways were evidenced. However, the switch of fermentation pathways happens at different pH depending on the sugar used. In glucose fermentation we have noticed that VFAs predominantly produced are butyric acid and then acetic acid from pH 4.7 to pH 6.5 (Figure 2). The maximum of ethanol production has been observed at pH 6.9, with the switch of fermentation pathway (Figure 2). At this pH acetic acid is predominantly produced. We can also observe an accumulation of propionic acid from day 3. In xylose fermentation the switch of fermentation pathways appears at pH 6.5 (data not shown). Either side of this pH we have observed the same profiles of VFAs production as for glucose fermentation with one additional day of lag-time before the onset of the fermentation process: neither VFAs, nor ethanol could be quantified at day 1 (Figure 2). We have also analysed sugar concentration. It happens that xylose is not degraded at all during the first day contrarily to glucose (data not shown). This could be explained by the fact that pentose fermentation is less widespread than hexose fermentation (as glucose) and so a longer latency is required for the biomass to adapt to this substrate. Once this latency period passed, it is however remarkable that xylose fermentation could proceed similarly to glucose. Concerning ethanol production we have obtained the most important concentration at pH 6.8 at day 2 as for glucose. Moreover we have obtained the same conversion rate of sugar in ethanol, i.e. 12.5% of initial carbon at day 2. Nevertheless, we can notice that an ethanol production occurs even at low pH from xylose.

We can notice that formic acid production and ethanol production are correlated with a maximum at pH 6.9 (or 6.8). This result reminds those of Temudo on glucose fermentation (Temudo *et al.*, 2007): at increasing pH values the butyrate, CO_2 and H_2 yields decreased whereas ethanol, acetate and formate yields increased.

The fermentation goes with important production of H_2 and CO_2 (Figure 3). As expected, the decrease of pH is associated to a higher production of hydrogen. The hydrogen produced is then consumed.

If we look at the different molecules produced we can notice that some of them are rapidly consumed: it is the case of ethanol, lactate, formate and H_2 contrarily to acetate, butyrate, propionate and CO_2 which accumulated. This last point can be notably explained because of the short monitoring of incubations and the fact that methanogenesis did not totally started as evidenced by the low methane concentrations recorded in the biogas (data not shown). We can add that when ethanol is produced at low pH (under 6), it seems that its degradation is slower than at pH close to 7.



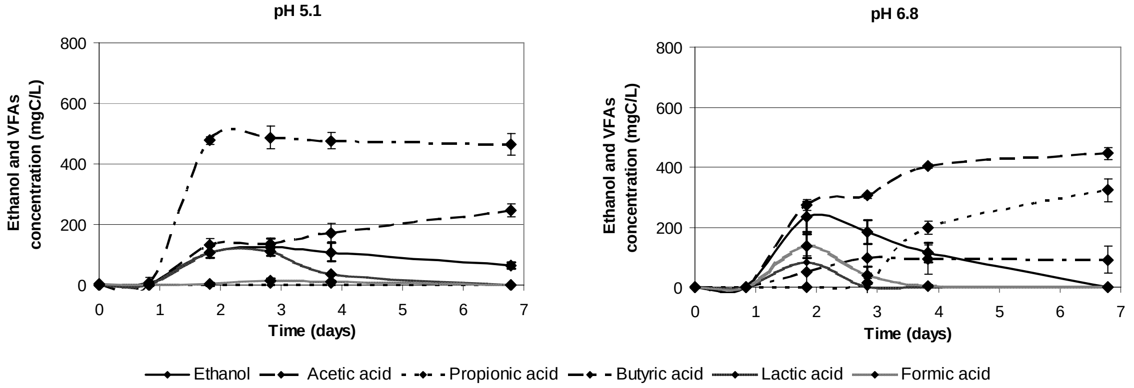


FIGURE 2 **Ethanol and VFAs production from glucose or xylose at different pH**

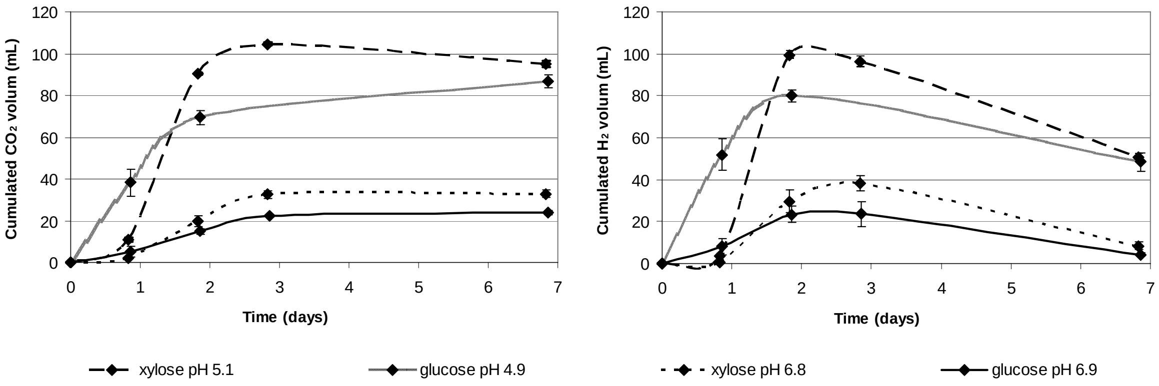


FIGURE 3 **Production of CO_2 and H_2 from glucose or xylose at different pH**

3.2 Bacterial diversity

Thanks to leachate sampling we have followed the evolution of bacterial populations in our batch incubations. We have confirmed that the bacterial populations were virtually identical at the beginning of each incubation (data not shown). Important diversity changes occurred in the incubations between day 0 and day 2. After this period ARISA profiles remained almost unchanged.

We have observed three different profiles depending on pH for glucose fermentation: 4.5 to 5.5, 6.0, 6.5 to 6.9 (Figure 4). For xylose fermentation the bacterial populations seem to be selected not by the pH but by the substrate. Indeed we can observe that the profiles are very similar with any pH (Figure 4).

It's interesting to see that even with the same bacterial population we do not obtain the same pattern of chemical molecules. Indeed the switch of fermentation pathways in glucose fermentation is observed at pH 6.9 and the switch of populations is observed at pH 6.0. Moreover in xylose fermentation, we have seen that with the same bacterial community we did not obtain the same concentration of VFAs and ethanol and biogas production depending on pH. This confirms the functional plasticity of complex microbial communities and the importance of pH to steer bacterial metabolism.

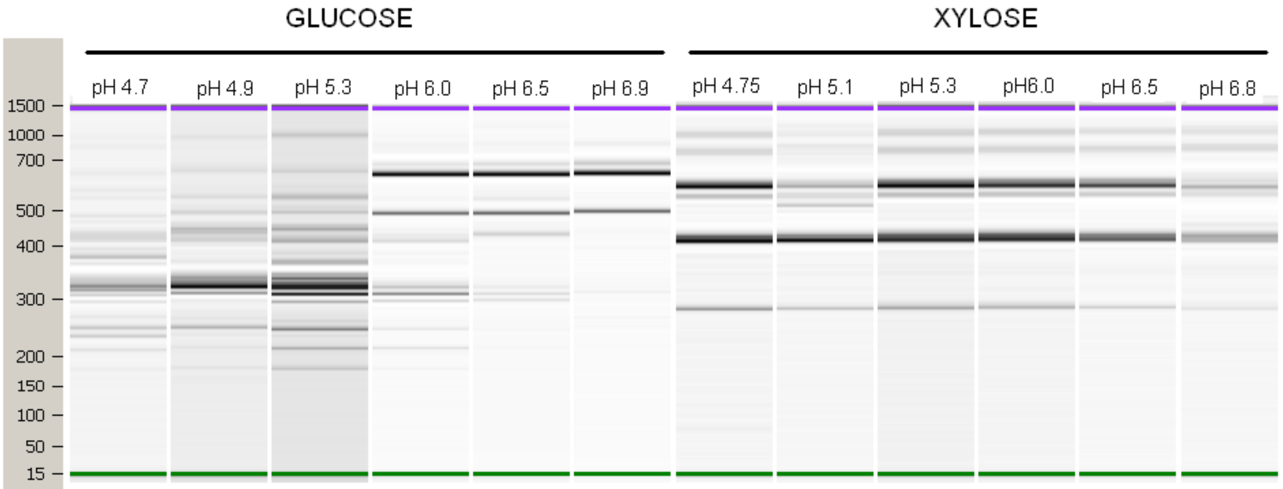


FIGURE 4 **ARISA profiles of bacterial populations in glucose (left) or xylose (right) incubations at different pH (day 2)**

4 CONCLUSIONS

Previous studies have shown that pH has a key role on the fermentation profile of sugars. Using a concentration of 4 g/L of glucose or xylose we have obtained the most important concentration of ethanol at pH 6.9 (or 6.8 for xylose) at day two. The value of optimal pH could be due to the origin of our inoculum: an aerobic digestor which works at pH close to 7. With both sugars, the rate of conversion sugar/ethanol is 12.5% (in term of initial carbon). However the ethanol is rapidly degraded from day 3 and at day 7 no ethanol can be quantified. We can add that at pH under 6, when a production of ethanol can be observed, the degradation is slower than at pH near 7. This could be explained by the fact that methanogenesis is inhibited at pH under 6. Concerning microbiology analysis we have observed two different bacterial selections: one by the pH in the case of glucose fermentation and another one by the substrate in the case of xylose. By choosing an inoculum coming from an anaerobic digestor, we know that we have microorganisms enable to degrade household waste, we can thereby avoid pretreatments to release sugars. We have now shown that it allows the conversion of glucose and xylose in ethanol, contrarily to yeasts which could only ferment glucose in ethanol. Studying microbial populations responsible for ethanol production (and degradation) is a way to optimize the production of ethanol from household waste.

5 ACKNOWLEDGEMENTS

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(160) MICRO-GAS GRIDS – AN INNOVATIVE APPROACH FOR OPTIMIZING ENERGY EFFICIENCY FROM BIOGAS PRODUCTION

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EXECUTIVE SUMMARY

Against the background of increasing governmental pressure to generate energy from biomass without compromising environmental sustainability, the University of Stuttgart investigates the intelligent concept of micro-gas grids in the context of the EU Central project "SEBE - Sustainable and Innovative European Biogas Environment".

The concept is based on the fact that biogas production is decoupled from biogas utilization allowing for (i) high flexibility regarding substrate charge and location of the biogas unit (ii) increased cogeneration of heat and energy and (iii) accelerated utilization of natural gas and biogas.

The overall objective of the research is to assess the status quo of application of the concept in Germany and identify the potential of micro-gas grids for optimization of conventional biogas production pathways. More specifically, it is to be investigated under which technical and economical framework conditions the concept features of micro-gas grids can optimize the (a) biogas to CHP and biogas to (b) bio-methane utilization pathway.

Regarding methodology a combination of quantitative and qualitative research methods such as operator questionnaires, semi-structured expert interviews and the Cumulative Energy Demand (CED) are being applied.

The preliminary results of the status quo analysis in Germany show that the predominant type of application is the micro-gas grid with the distributional function allocating biogas to heat sinks. Usually this type encompasses a centralized biogas plant that is connected to (one or more) satellite CHPs. Despite being a promising concept for accelerated utilization of bio-methane from small-sized biogas plants, micro-gas grids for collection of biogas from decentralized plants for central upgrading has not been implemented in Germany yet. Supportive framework conditions for implementation of this concept have found to be: high plant density, well developed gas grid infrastructure, proximity to natural gas fuel stations as well as beneficiary topographic conditions. On micro level, potential operators for this type of concept are usually found to be in dissimilar planning phases which make decision-making in defined timeframes and the creation of entrepreneurial units for the concerted concept implementation difficult. Regarding technical aspects, biogas quality i.e. composition, accounting of the partial biogas volume and provision of constant biogas volumes are found to impede the implementation of the concept in practice.

For the investigation of technical parameters a model of calculation of the CED has been developed. The assumptions of the model parameters are based on literature review and expert interviews. With the model it is to be investigated how the technical parameters e.g. biogas volume demand, transport distances, substrate quantity input affect the optimization efficiency.

Against the background of rising energy crop prices and rising environmental challenges of high share of energy crop feedstock for biogas production new optimization strategies for bio-methane production need to be developed. Micro-gas grids are an innovative approach for adding value to biogas based on local production as they allow for transportation of gas instead of substrate. To what extent the latter can contribute to increasing integration of biogas production based on local biomass and therefore increase the share of agricultural and other bio waste products into the bio-methane market is to be investigated.

1 INTRODUCTION

Against the background of increasing governmental pressure to generate energy from biomass without compromising environmental sustainability, the University of Stuttgart investigates the intelligent concept of micro-gas grids in the context of the EU Central project "SEBE - Sustainable and Innovative European Biogas Environment". The approach is based on the fact that it is not necessary to make use of biogas directly at the production site.

Overall efficiency can be increased by diverting biogas to where best use can be made of its energy content. The aim of the research is to investigate the optimization potential of micro-gas grids for conventional biogas pathways. This research is separated in multiple parts. In this paper, the focus is on the conceptual characteristics of micro-gas grids and the model characteristics for calculation of the cumulative energy demand of the micro-gas grid system for the biogas to bio-methane pathway.

1.1 Problem definition

For the utilization of biogas different utilization pathways are in place. Up until now the combustion of biogas in CHP constitutes the predominant utilization strategy. Thereby the generated electricity is injected into the national electricity grid. Due to the fact that majority of biogas plants operate in isolation in outskirts of settlements, heat recovery that makes up to 2/3 of the overall energy content in biogas, is insufficiently taking place. Considering the rising costs for energy crops and the related resource demand for its cultivation, it becomes clear, that strategies for increasing energy efficiency from biogas utilization will become a pressing need for further dissemination of the technology.

One strategy for addressing the issue of increasing energy efficiency from biogas utilization is the spatial and temporal decoupling of biogas production and utilization sites. This allows for biogas use at a location where an energetically optimized application is feasible. From an energy efficiency point of view, the decoupling brings about several advantages such as:

- Utilization of full energy potential of biogas and regulation of biogas supply according to demand without seasonal and daily constraints.
- Application of biogas plant sizes and substrate types that are in line with local conditions, reducing transport distances and enforcing the utilization of waste products.

Biogas upgrading to bio-methane quality has in the past years become a popular technology for enforcing the decoupling of biogas production and utilization. The upgrading to natural gas quality and injection into the natural gas grid makes the utilization of biogas independent from spatial and temporal demand. The injection into the natural gas grid allows for comprehensive utilization of energy content of biogas and therefore has highly been supported on policy level. Nevertheless the technology is not without energetic and therefore economic pitfalls, as recent project failures have shown.

As a result of high energy demand for upgrading and injection, the biogas production for upgrading usually has to fulfil following prerequisites to be economically viable: (a) availability of large amounts of energy crops[1] for production of huge biogas volumes and (b) proximity to natural gas grids.

These requirements constitute significant (energetic) barriers for biogas regions with small-scale agricultural structures that are characterized by smaller sized biogas plants with a higher share of waste products as substrate input for participating in the bio-methane market.

The "decoupling approach" of micro -gas grids increases the flexibility regarding substrate type and quantity and location of biogas plant. With the application of micro-gas grids, the necessary biogas volume for upgrading is generated by decentralized collection of partial biogas volumes from different biogas locations. Micro-gas grids allow for transportation of gas instead of transportation of substrate and therefore support the decentralized production of biogas based on local biomass potential.

Against this background it is believed that the application of micro-gas grids can alleviate the above mentioned barriers and contribute to speeding up the integration of decentralized small-scale biogas plants into the bio-methane market.

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[1] Large and constant biogas volumes can due to its characteristics (high energy value, storable, easy for transportation, constant availability) mostly be provided by a high share of energy crops (ca.90%) contrary to waste products.

1.2 Research objectives

The overall objective of the research is to identify the potential of micro-gas grids for optimization of conventional biogas production pathways. More specifically, it is to be investigated under which technical framework conditions the concept features of micro-gas grids can energetically optimize the (a) biogas to CHP and biogas to (b) bio-methane utilization pathway.

The overarching research questions are:

1. What type of MGG asserted oneself in Germany and what future developments are to be expected?
2. Under which framework conditions is the integration of micro- gas grids for the optimization of energy efficiency of conventional biogas production pathways feasible?
3. To what extent can the concept of micro-gas grids contribute to speeding up the integration of decentralized biogas production from small-scale plants into the bio-methane market?

The following paper focuses on the investigations related to the second question.

2 METHODOLOGY

For investigation of the research quantitative and qualitative research approaches have been applied. Due to lack of data on micro-gas grids and therefore the need to obtain data in a standardized format, operator questionnaires have been send to all federal states of Germany. Furthermore semi-structured expert interviews have been undertaken with designed interview guideline. For the investigation of technical parameters of the concept based on energetic performance of the micro-gas grid concept, the quantitative methodology Cumulative Energy Demand (CED) approach is to be applied.

2.1 Model for Cumulative Energy Demand (CED) calculations

In the first step of the research, the model for investigation of the optimization potential of micro-gas grids for the bio-methane pathway was designed. Therefore a (I) micro-gas grid decentralized model that is connecting 6 small-scale plants is compared with a (II) reference model encompassing a big centralized biogas plant producing biogas for upgrading and injection (Figure 1). Both scenarios produce the same raw biogas volume.

Based on the results of the CED calculation, the aim of the designed model is to provide answers to the following question: “*What biogas production and utilization parameters are particularly affecting the energy improvement efficiency of micro-gas grids in the context of the bio-methane production pathway?*”

More specifically, following questions are to be answered with the developed scenarios:

(a) To what extent does the biogas quantity demand for upgrading influence the energetic feasibility of the installation of micro-gas grids?

(b) At what distances is the transportation of gas energetically more feasible than the transportation of substrate and how does influence the viability manure and biowaste utilization for bio-methane production?

(c) What is the energetic payback period of the micro-gas installation when compared to transportation of substrate?

The assumptions of the model parameters are based on literature review and expert interviews. The calculated figures related to the model (see Table 1 and Table 2) are based on these assumptions.

Table 1: Calculated parameters for Model I

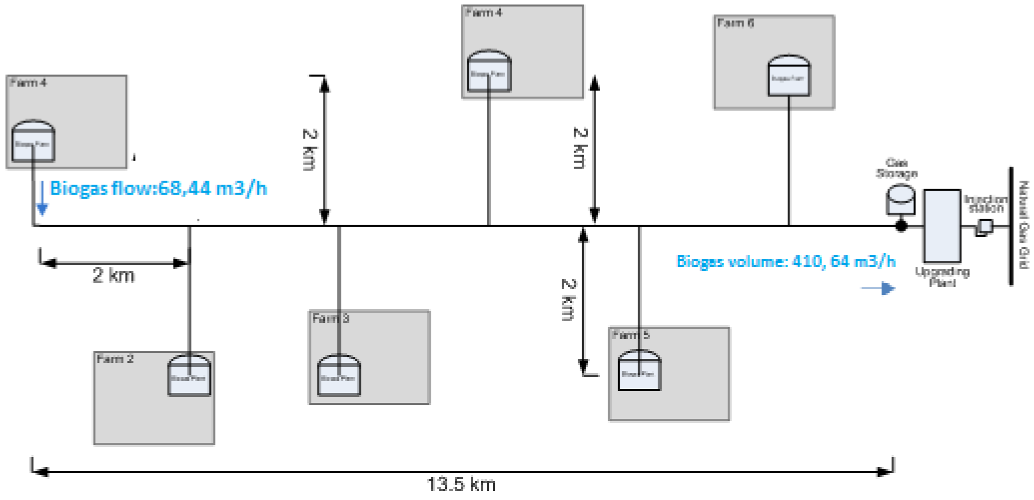
Scenario I	6 dairy cow farms with equal type of digester technology, substrate mix and substrate input quantity. Although not reflecting the reality, the similarities of biogas production for each plant were assumed to simplify the CED calculations. The size of the farms reflects an economically feasible dairy farm. It is assumed that manure is completely provided by the respective farms. Maize silage is been supplied externally. Biowaste is been supplied from a distance of 15 km.
Biogas plant owner	Dairy farm 210 GVE = 175 cows Area = 2,10 km^2 Manure production: 8,75 t/day
Biogas plant	Co-fermentation; substrate mix: 50 % manure, 25 % biowaste, 25 % silage Substrate input = 17 t/day per plant (8,75 t manure/day + 4,375 t biowaste + 4,375 t maize silage

Biogas production from substrate	8,75 t manure = 263 m^3/d 4,375 t maize silage = 826,875 m^3/d 4,375 t biowaste = 656,25 m^3/d = 1699 m^3/d
Biogas production per plant	Raw biogas per plant = 70,79 m^3/h Partially upgraded = 68,44 m^3/h (Reduction of H_2S from 2,85 % V to 0,15 % V[2]; reduction of H_2O from 0,25 % V to 0,00035 %V[3])
Micro- gas grid length	Main pipeline: 13,5 km Each feeder i.e. transfer pipeline: 2 km
Bio-methane production	6067, 206 m^3/day = 252,8 m^3/h

Table 2: Calculated parameters for Reference model

Reference Scenario	One biogas plant is mainly fed with maize silage. Maize silage is being obtained from a farm that is 20 km away. Manure is supplied from 10 km distance.
Biogas plant owner	Energy provider that obtains substrate input from contracted farmers.
Biogas plant	Co-fermentation, substrate mix: 90 % maize silage, 10 % manure Substrate input = 2,31 t (maize silage: 52,91 t /day; 5,29 t manure/day)
Biogas production from substrate	5,29 t manure = 90,98 m^3/d 52,91 t maize silage = 10.000 m^3/d
Biogas production per plant	10.090,98 m^3/d
Bio-methane production	6306.86 m^3/day = 262.79 m^3/h

Figure 1: Model 1



3 RESULTS

Prior to the energy-related evaluation of the potential of micro-gas grids for optimization of biogas utilization pathway (a) CHP and (b) bio-methane could take place, it was necessary to scrutinize the conceptual parameters of the micro-gas grids due to lack of theoretical embedment of the concept. Therefore in the following section some selected results regarding the general overview of the concept characteristics and the technical components of the micro-gas grids are given. In the course of completion of this paper the results of the CED calculations could not be included as they were not finalized yet.

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[2] Limit for H_2S in biogas

[3] Limit for H_2O in biogas

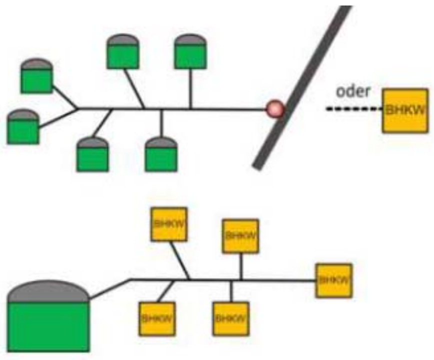
3.1 The concept of micro-gas grids

Literature review has shown that the terminology of micro-gas grid encompasses a myriad of different definitions as a result of lack of institutional embedment of the concept. Hence different terminology for addressing this energy concept is broadly used (e.g. local/regional biogas grids, biogas distribution/ collection networks etc.)

MGG is a local low pressure energy supply (Bärrntaler et al. 2008; Erler, 2009), serving for direct transfer of upgraded or marginally enriched biogas to private or public consumers. MGG can have either a *distributional function* to proceed biogas to heat sinks or have a gas *collection function*, merging smaller sub-volume biogas flows (see Figure 1). Hence the innovation potential of the concept is based on the fact that biogas production and utilization is decoupled. The latter is believed to be the basis for the optimization potential of micro-gas grids for conventional biogas production pathways as the decoupling approach allows for:

- high flexibility regarding substrate charge and location of the biogas unit,
- increased cogeneration of heat and energy and
- accelerated utilization of natural gas and biogas.

Figure 2: Micro-gas grid concept (Panic et al., 2011)



Micro-gas concepts may vary in following characteristics (changed after Bärnthaler et al., 2008):

- centralized or decentralized biogas production (single or multiple producers),
- autarkic or demand-driven supply[4] (island network or connection to the grid),
- consumer structure (private households or industry),
- gas quality (partly or fully upgraded).

The preliminary results of the status quo analysis in Germany show that the predominant type of application is the micro-gas grid is in combination with the CHP biogas utilization pathway. The decoupling approach of the micro-gas grid is mainly being used to distribute the centrally produced biogas to different CHP plants that are located in heat sinks (Figure 1, type B). Usually this type encompasses a centralized biogas plant that is connected to (one or more) satellite CHPs. In practice these micro-gas grids are being installed as *refitting measurement* to complement the missing heat utilization concepts of older biogas plants or as a (B) *subcomponent of a comprehensive energy supply concept* from Renewable Energies.

Despite being a promising concept for accelerated utilization of bio-methane from small-sized biogas plants, micro-gas grids for collection of biogas from decentralized plants for central upgrading has not been implemented in Germany yet. In this research phase only general assumptions regarding the possible barriers and drivers concerning the application of the concept for bio-methane production can be provided.

Supportive local conditions for implementation of this concept have found to be:

- high plant density well developed gas grid infrastructure,
- proximity to natural gas fuel stations and
- beneficiary topographic conditions for installation of micro-gas grid pipelines.

Regarding barriers some local and technical impediments could be identified. On local level, potential operators for this type of concept are usually found to be in dissimilar planning phases which make decision-making in defined timeframes and the creation of entrepreneurial units for this type of concept implementation difficult.

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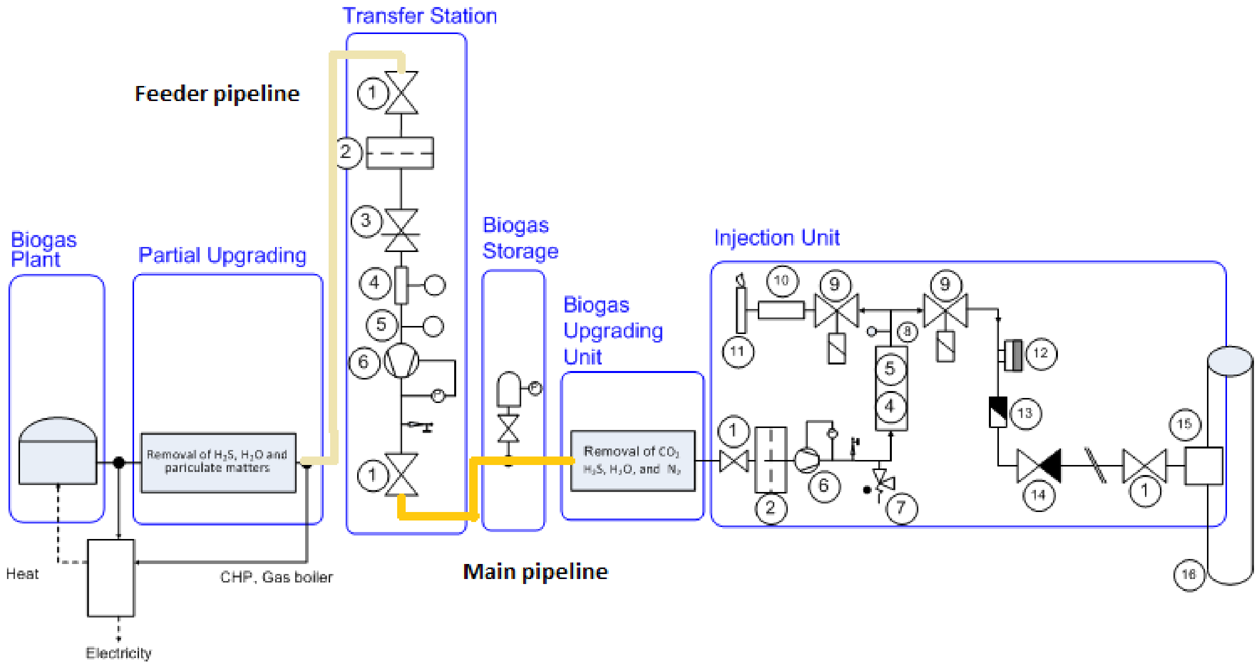
[4] The focus of the respective research is micro-gas grids for demand-driven supply.

Regarding technical aspects, biogas quality[5] the accounting of the partial biogas volume and provision of constant biogas volumes are found to impede the implementation of the concept in practice.

3.2 Technical components of a micro-gas grid concept for bio-methane production

In Figure 3 technical components of a micro-gas grid concept for upgrading and injection are displayed. The concept includes a biogas plant, partial upgrading, transfer station, biogas storage, upgrading unit and the injection point, after which biogas is injected into the natural gas grid. The partial upgrading and transfer station are connected via *feeder pipeline* or *transfer pipeline* that is usually smaller (e.g. dn 25 - dn 110) in diameter than the *main pipeline* (dn 165-dn 315) of the micro-gas grid. The *partial upgrading* is necessary for the removal of H_2S and H_2O to avoid corrosion, biofouling and therefore congestion of the pipelines. Substance concentrations of $H_2S \leq 5$ mg/m^3 biogas and $H_2O \leq 40$ mg/kg biogas have to be achieved in order to safeguard smooth transport of biogas. The *transfer station* is the technical and legal system boundary between the biogas supplier and the grid operator. It is responsible for the control of the injection pressure, quantity and gas quality. The pressure level of the grid should be as low as possible, as this is reducing the operating costs as well as costs for the different facilities and components of the micro-gas grid system. After the transfer station, the partially upgraded biogas is injected into the *main pipeline* that constitutes the collection system of the partial biogas volumes from different plants. Depending on the characteristics of the grid, additional compression facilities need to be installed for safeguarding the biogas flow within the grid up to the upgrading unit. The micro-gas grid concept also might require a *storage system* that acts as a buffer if one of the connected biogas plants fails to provide regular biogas quantities as the uupgrading unit requires relatively constant biogas input. Another two components constitute the upgrading and injection unit. In the upgrading unit components such as H_2S, H_2O and N_2, but mainly CO_2, are being removed in order to reach natural gas quality. Depending on the upgrading technology methane contents between 96-99 % can be achieved. The *injection unit* is composed of equipments for the final assurance of gas flow into the natural gas grid. For safety reasons, here also odorization of the gas takes place.

Figure 3: Components of a micro-gas grid concept for upgrading and injection



1. Valve
2. Filter
3. Safety valve
4. Gas quality measurement
5. Net calorific measurement
6. Pressure regulator
7. Safety outlet valve
8. Temperature probe
9. Automatic valve
10. Valves for gas flare
11. Flare
12. Odorization
13. Gas amount measurement
14. Backflow assurance
15. Gas mixing and flow regulating facility
16. Natural gas grid

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[5] Biogas contrary to natural gas contains corrosive elements such H_2S, H_2O, CO_2 etc. that affect devices in the grid and also cause congestion.

4 CONCLUSIONS

Against the background of rising energy crop prices and rising environmental challenges of high share of energy crop feedstock for biogas production new optimization strategies for bio-methane production need to be developed. Micro-gas grids are an innovative approach for adding value to biogas based on local production as they allow for transportation of gas instead of substrate. To what extent the latter can contribute to increasing integration of biogas production based on local biomass and therefore increase the share of agricultural and other bio waste products into the bio-methane market is to be investigated.

5 ACKNOWLEDGEMENTS

The concept of micro-gas grids is being investigated at the University of Stuttgart in the framework of the EU-Central project Sustainable and Innovative European Biogas Environment (SEBE).

Figure

FIGURE 1 **Model I**

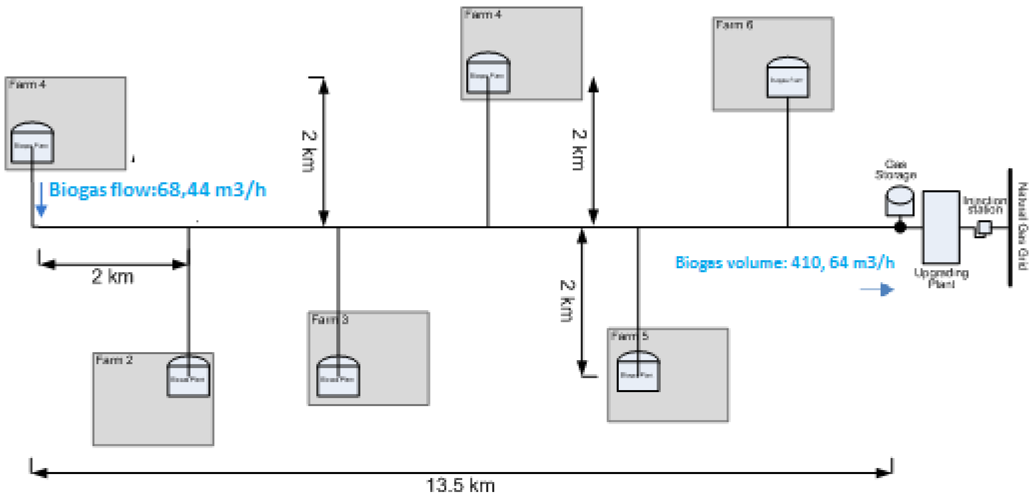
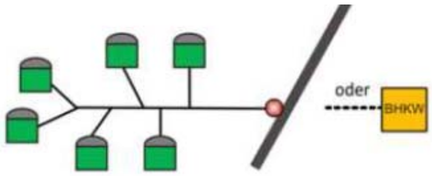


FIGURE 2 **Micro-gas grid concept (Panic et al., 2011)**



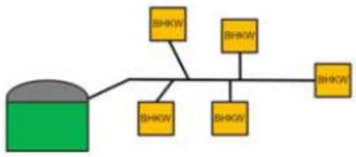


FIGURE 3 **Components of a micro-gas grid for upgrading and injection**

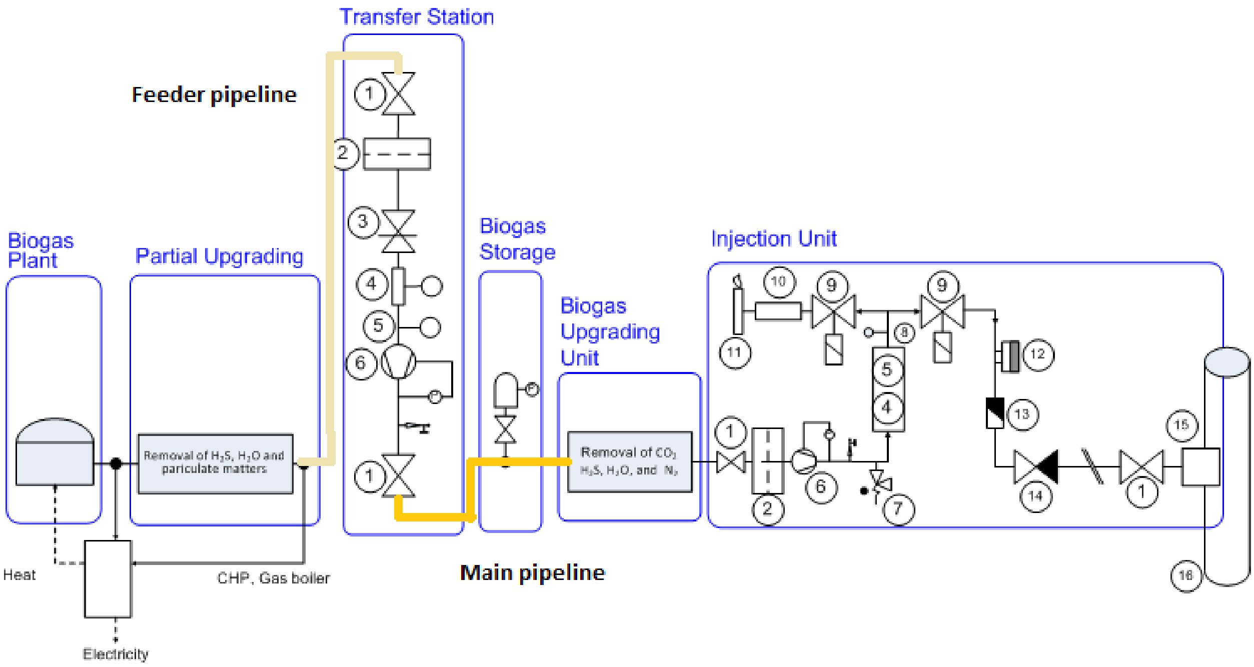


TABLE 1: Calculated parameters for Model I

Scenario I	6 dairy cow farms with equal type of digester technology, substrate mix and substrate input quantity. Although not reflecting the reality, the similarities of biogas production for each plant were assumed to simplify the CED calculations. The size of the farms reflects an economically feasible dairy farm. It is assumed that manure is completely provided by the respective farms. Maize silage is been supplied externally. Biowaste is been supplied from a distance of 15 km.
Biogas plant owner	Dairy farm 210 GVE = 175 cows Area = 2,10 km^2 Manure production: 8,75 t/day
Biogas plant	Co-fermentation; substrate mix: 50 % manure, 25 % biowaste, 25 % silage Substrate input = 17 t/day per plant (8,75 t manure/day + 4,375 t biowaste + 4,375 t maize silage
Biogas production from substrate	8,75 t manure = 263 m^3/d 4,375 t maize silage = 826,875 m^3/d 4,375 t biowaste = 656,25 m^3/d = 1699 m^3/d
Biogas production per plant	Raw biogas per plant = 70,79 m^3/h Partially upgraded = 68,44 m^3/h (Reduction of H_2S from 2,85 % V to 0,15 % V[6]; reduction of H_2O from 0,25 % V to 0,00035 %V[7])
Micro- gas grid length	Main pipeline: 13,5 km Each feeder i.e. transfer pipeline: 2 km
Bio-methane production	387, 766 m^3/h

TABLE 2: Calculated parameters for Reference model

Reference Scenario	One biogas plant is mainly fed with maize silage. Maize silage is being obtained from a farm that is 20 km away. Manure is supplied from 10 km distance.
Biogas plant owner	Energy provider that obtains substrate input from contracted farmers.

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[6] Limit for H_2S in biogas

[7] Limit for H_2O in biogas

Biogas plant	Co-fermentation, substrate mix: 90 % maize silage, 10 % manure Substrate input = 2,31 t (maize silage: 52,91 t /day; 5,29 t manure/day)
Biogas production from substrate	5,29 t manure = 90,98 m^3/d 52,91 t maize silage = 10.000 m^3/d
Biogas production per plant	10.090,98 m^3/d
Bio-methane production	6,306.86 m^3/d

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(195) ETAMAX: DRIVING WITH BIOGAS FROM BIOWASTE

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EXECUTIVE SUMMARY

The utilization of vegetable biomass for the recovery of bioenergy – power, heat or fuels – plays an exceptional role as a sustainable alternative to conventional energy carriers. Biogas, a mix of energetically usable methane and carbon dioxide, is created during the anaerobic digestion of organic matter. In conjunction with the combined heat and power generation, biogas generation is considered as a technology with a high net energy yield and a high CO_2 avoidance potential.

This project has focused its activities on easily fermentable, low-in-lignocellulose, wet biomass – low-cost biowaste and residual algal biomass which constitute no competition for the production of foodstuffs – with a combined, modular process under maximum energy recovery. The aim is the complete conversion into biogas and the simultaneous closing of all materials cycles. The main focus is on the regional creation and utilization of bio-methane. The biogas is to be purified by separation of the carbon dioxide with membrane technology so that the bio-methane can be used as fuel for vehicles which are operated on compressed natural gas (CNG). The potential of biomass for the creation of biogas has so far been insufficiently exploited in general and hardly at all in vehicles. To ensure that a digestion plant can convert the different substrates as efficiently as possible into biogas, the process technology for the individual substrates is specifically adapted by means of a flexible multi-substrate high-load digestion plant. Only by this the substrate can be converted into methane with the maximum degree of efficiency. During the investigations unsorted market waste from the Stuttgart central market was provided, disintegrated and digested. Our investigations showed that the fluctuations in the composition of the substrate could only be compensated by means of intelligent process control: by adding substrate portions of different digestion stages via several pre-digestion tanks, we can ensure a continuous biogas production with few variations despite the great fluctuations in the substrates. The investigations in the pilot plant enabled us to determine successfully permissible values such as the minimum detention period and maximum volume load and parameters for the ideal feed of different substrate compositions. In a high-load digestion process developed by the Fraunhofer IGB the solid matter biowaste fractions which are low in lignocellulose are almost completely converted into biogas within a hydraulic retention time of only 8.5 days. During the first year the process parameters were determined in a digestion plant at pilot plant scale (2 x 30-l-reactors) at the Fraunhofer IGB. The two-stage pilot plant produced 850 l of biogas from central market waste per total volatile solids (TVS). In terms of the existing reactor volume this equates to an average of 190 l biogas per day at a volume load of 7 g TVS/ld. Based on those results the demonstration plant scale (2 x 3.5 m³) was designed. Additional wet, low-lignocellulose biomass for multi-substrate high-load digestion is contributed by means of residual algal biomass. Energy recovery from algal biomass is already possible in a highly efficient manner today thanks to a photobioreactor platform developed by the Fraunhofer IGB. The algae in the reactors grow to high cell densities using only sunlight as an energy source and carbon dioxide as a carbon source plus inorganic nitrogen and phosphate. The aim is to find robust algae which grow rapidly in flue gas, and in the changing seasonal light and temperature conditions in central Europe. EtaMax utilizes carbon dioxide which is created as a co-product during the digestion process and during the combustion of biogas as a source for the cultivation of algae. Current investigations have shown that the inorganic nutrients necessary for growth are contained in sufficient quantities in the filtrate of the digestion plant and can be used for the cultivation of algae. Expensive nutrients will therefore not be necessary. For small amounts of digestion residues which cannot be further decomposed in an anaerobic environment, the catalyst-supported hydrothermal gasification under high pressure and high temperature is examined. Here the same products are created as during the digestion process: carbon dioxide and methane. The joint factor of all individual components to be utilized is the minimum energy input for the implementation of the individual tasks. In the end of 2011 the findings determined at pilot plant scale were converted to a demonstration plant on the site of the EnBW combined heat and power station in Stuttgart-Gaisburg where they will be tested. In a future full scale plant it would be possible to generate 300,000 cubic meters of methane gas per year from the municipal biowaste from the city of Stuttgart. After purification it can be used as vehicle fuel for a small fleet of collection vehicles which run on natural gas. This would also be beneficial for local air quality.

1 INTRODUCTION

1.1 Background

The utilization of vegetable biomass for the recovery of bioenergy – power, heat or fuels – plays an exceptional role as a sustainable alternative to conventional energy carriers. Biogas, a mix of energetically usable methane and carbon dioxide, is created during the anaerobic digestion of organic matter. In conjunction with the combined heat and power generation, biogas generation is considered as a technology with a high net energy yield and a high CO_2 avoidance potential.

1.2 Research objectives

This project has focused its activities on easily fermentable, low-in-lignocellulose, wet biomass – low-cost biowaste and residual algal biomass which constitute no competition for the production of foodstuffs – with a combined, modular process under maximum energy recovery. The aim is the complete conversion into biogas and the simultaneous closing of all materials cycles. The main focus is on the regional creation and utilization of bio-methane. The biogas is to be purified by separation of the carbon dioxide with membrane technology so that the bio-methane can be used as fuel for vehicles which are operated on compressed natural gas (CNG). The potential of biomass for the creation of biogas has so far been insufficiently exploited in general and hardly at all in vehicles.

2 METHODOLOGY

The principle of the EtaMax-concept (figure 1) is to use easy fermentable, low-in-lignocellulose, wet biomass – low-cost biowaste and residual algal biomass with a combined modular process under maximum energy recovery. The biowaste is crushed by a specifically developed macerator which is able to crush all kind of fruits and vegetables in particles under 5 mm particle size under highly reduced energy demand. After crushing the biowaste is pumped into storage tanks (pre-stage) where a first degradation step is proceeding. Depending on the pH the stored biowaste will be pumped in specified amounts into the anaerobic fermentation tanks (central step) where the biomass is finally converted in methane and CO_2. The characteristic of the high load digestion process is the combination of the reactor with a membrane system, which concentrates the microorganisms and the biomass in the system. This leads to higher degradation rates and therefore to higher biogas yield per time.

The added value of the whole process is a result of a membrane cleaning process of the biogas, the gasification of the digestate by hydrothermal gasification and the disinfection of the filtrate from the bioreactor. The methane is directly used as biofuel in CNG-cars. The CO_2 of the cleaned biogas and the filtrate of the fermentation reactors which contains nutrients and salts are used for the cultivation of microalgae. The process conditions are adjusted that the microalgae produces oil (triacylglycerides). After separating the oil fraction, the residual algae biomass is added to the pre-stage of the biogas process. In every process step the relevant parameters for running the facilities automatically and also to determine the quality of processes and products like e.g. pH, temperature in the reactors, biogas yield, methane concentration etc. are measured.

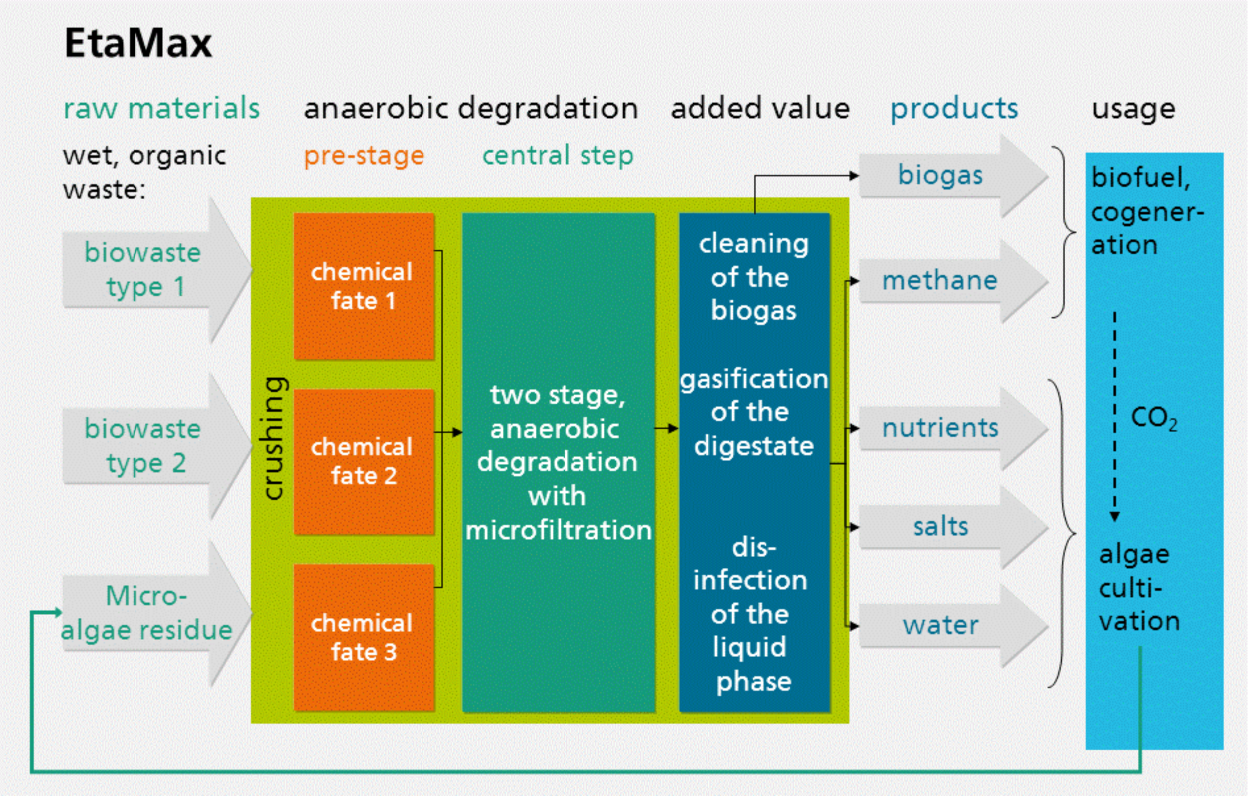

Figure 1: Principle of EtaMax project

3 RESULTS AND DISCUSSION

3.1 Digestion process

In the high-load digestion process developed by the Fraunhofer IGB the solid matter biowaste from the wholesale market Stuttgart (figure 2) which is low in lignocellulose are almost completely converted into biogas within a hydraulic retention time of only 8.5 days. The fruit and vegetable distribution was determined by taking spot tests over one year (figure 3). Depending on the season, the distribution was very different and not predictable. This leads to changing initial conditions for the system operation of the digestion plant.

Figure 2: Unsorted biowaste from wholesale market Stuttgart

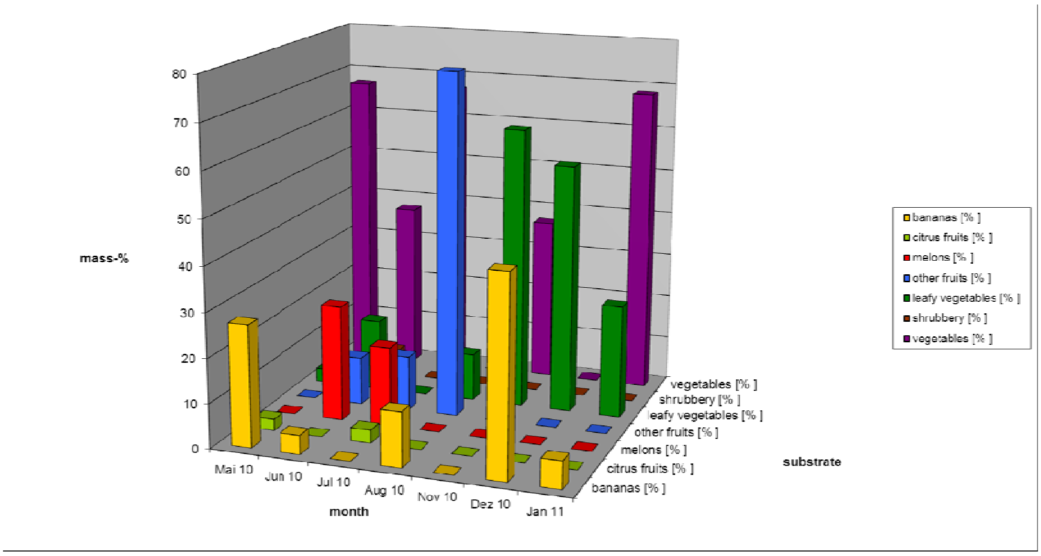

Figure 3: Fruit and vegetable distribution as a function of time

During the first year the process parameters were determined in a digestion plant at pilot plant scale (2 x 30-l-reactors) at the Fraunhofer IGB (figure 4) with real unsorted wholesale market waste and residual microalgae biomass. The two-stage pilot plant produced 850 l of biogas from wholesale market waste per total volatile solids (TVS). In terms of the existing reactor volume this equates to an average of 190 l biogas per day at a volume load of 7 g TVS/ld. About 90% to 95% of organic load was degraded. Adapted from those results the demonstration plant scale (2 x 3.5 m³) was designed (figure 5).

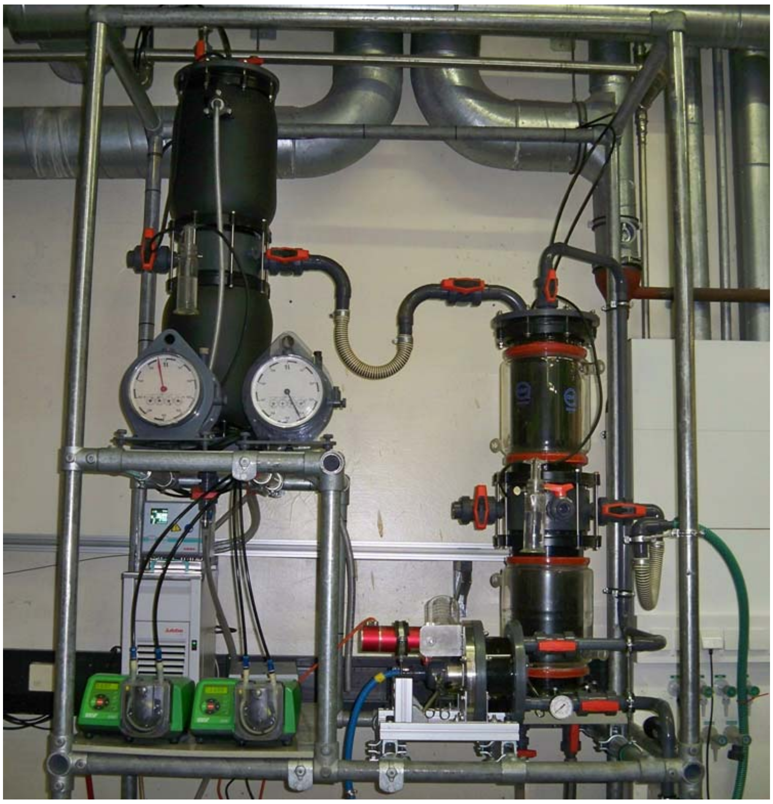

Figure 4: Fermentation plant (pilot scale, 2 x 30-l-reactors)

Figure 5: Fermentation plant (demonstration scale, 2x3-5m³-reactors)

To ensure that the digestion plant can convert the different substrates as efficiently as possible into biogas, the process technology for the individual substrates is specifically adapted by means of a flexible multi-substrate high-load digestion plant. Additional to the fruits and vegetables wet, low-lignocellulose biomass for multi-substrate high-load digestion is contributed by means of residual algal biomass out of the cultivation of oil producing microalgae cultivation.

3.2 Hydrothermal gasification

For small amounts of digestion residues which cannot be further decomposed in an anaerobic environment, the catalyst-supported hydrothermal gasification under high pressure and high temperature is examined. Here the same products are created as during the digestion process: methane and carbon dioxide. Previous experiments showed that the specific gas yield by using a catalyst is nearly two times higher and the methane yield is about five times higher than without using a catalyst. Focus of the ongoing experiments is to find and develop a suitable and stable catalyst material which is able to convert the organic components of the digestate into the product methane for continuous operational mode.

3.3 Microalgae cultivation

Energy recovery from algal biomass is already possible in a highly efficient manner today thanks to a photobioreactor platform developed by the Fraunhofer IGB. The algae in the reactors grow to high cell densities using only sunlight as an energy source and carbon dioxide as a carbon source plus inorganic nitrogen and phosphate. EtaMax utilizes carbon dioxide which is created as a co-product during the digestion process and during the combustion of biogas as a source for the cultivation of oil producing microalgae. Current investigations have shown that the inorganic nutrients necessary for growth are contained in sufficient quantities in the filtrate of the digestion plant and can be used for the cultivation of algae. Expensive nutrients are not necessary. The reached biomass productivity is 20% higher than with synthetic nutrient solution.

For the experiments flat panel airlift photobioreactors are used and the experiments are done in laboratory with artificial light as well as in a greenhouse and in outdoor photobioreactors both with natural daylight (figure 6).

Figure 6: Greenhouse with flat panel airlift photobioreactors

The optimum light intensity for microalgae cultivation is 1/10 up to 1/5 of sunlight intensity. The reached oil content in cultivated Chlorella vulgaris was about 45w-%, in different Nanochloropsis types it differed between 30w-% up to 50w-% under laboratory conditions (figure 7).

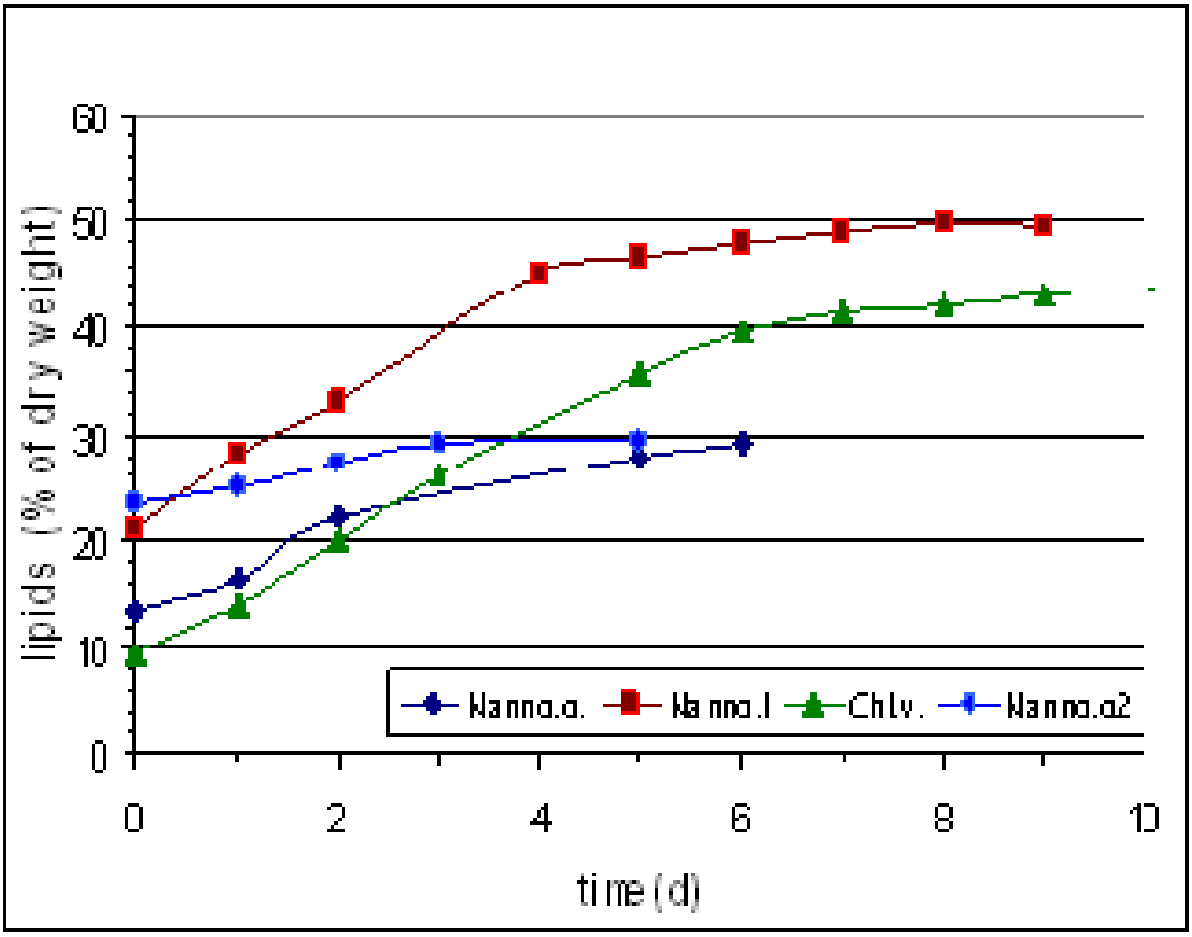

Figure 7: Lipid productivity of different Microalgae in function of time (under laboratory conditions)

For the above described algae types, the lipid content increases for 2-4% in dependence of the relative light availability from 0,3 up to 1,1 E/(gDW*d).
The main fatty acid is oleic acid $C_{18:1}$ (figure 8). The fatty acid content can reach up to 50-70% of dry weight under laboratory conditions.

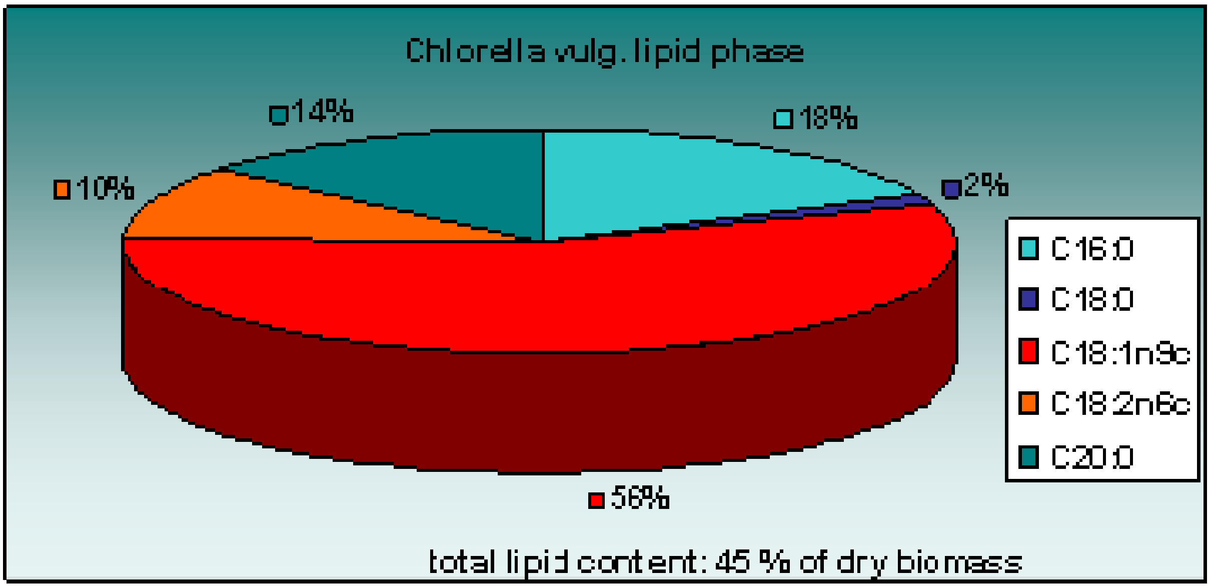

Figure 8: Fatty acid distribution in Chlorella vulgaris (under laboratory conditions)

Under outdoor conditions, the fatty acid content reaches up to 45w-%. Furthermore the concentration of polyunsaturated fatty acids increases.

3.4 Biogas cleaning and biogas utilisation

For the use of biogas as biofuel in CNG-cars the biogas has to be dried and desulphurized before the methane is concentrated by membrane technology (capacity of 13 m³/h) up to the necessary content of 94 vol-% at a working pressure of 4-5 bar. The start of operation of the plant will be in Mai 2012. The membrane technology is the only methane separation technology which is available in small demonstration scale. Furthermore the modularity of the membrane technology is a big advantage in the achievement of small, local plants. The expected methane slip will be about 3% (0,7kg methane/d). After gas cleaning the methane will be compressed in pressure cylinders and will be available for filling of the car tank directly at a local installed service station.

4 CONCLUSIONS

In the end of 2011 the findings were determined at pilot plant scale and were converted to a demonstration plant scale on the site of the EnBW combined heat and power station in Stuttgart-Gaisburg. In a future full scale plant it would be possible to generate 300,000 cubic meters of methane gas per year from the municipal biowaste from the city of Stuttgart. After purification it can be used as vehicle fuel for a small fleet of collection vehicles which run on natural gas. This would also be beneficial for local air quality. The joint factor of all individual components to be utilized is the minimum energy input for the implementation of the individual tasks.
The biogas demonstration plant is completely closed in terms of odour emissions. This will show the possibility to site this kind of plant at sources of waste generation e.g. in settlements directly.

5 ACKNOWLEDGEMENTS

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Figure

Session 10

(4) ANAEROBIC DIGESTION OF BIOGENIC RESIDUES IN GERMANY

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EXECUTIVE SUMMARY

The anaerobic digestion (AD) has reached a very high level in Germany. By the end of 2011 nearly 7.000 AD plants had an installed capacity of more than 2.700 Megawatt supplying 5 million households with electricity. Besides of generating electricity the energy carrier *biogas* is able to supply households, industries, stables, swimming pools and other buildings with heat and even substitute natural gas and fuel when it is upgraded to biomethane. But also the material benefit from biomass, using the digestate as valuable nutrient-rich fertiliser plays an important role, especially regarding the shortage of phosphor. The production of biogas combines the energetic and the material use of biomass. The cycle of nutrients is closed, saving fossil and mineral resources and green-house-gas emissions, especially with the treatment of organic residues and manure.

The above mentioned 7.000 AD plants run mainly on the base of energy crops and manure. But the use of biogenic residues in AD plants is also of great importance in Germany. For 2009, the German Federal Office of Statistics lists 13.2 million tonnes of separately collected biowaste in Germany with a share of 7.8 million tonnes of biowaste from households. Approximately 4 million tons of this separately collected biowaste in addition of two million tons of animal by-products (ABP) are treated in 969 biogas plants in Germany.

The European Directive 2008/98/EC on waste (Waste Framework Directive) includes ABP which are destined for the use in AD plants into its scope. The Waste Framework Directive is to be implemented into national law. On the 1st of June 2012 the German Material Cycle and Waste Management Act came into force. As a disastrous change manure as an ABP falls under the scope of the waste regime though the competent authority may decide if manure is a waste or not on a case-by-case basis. This unclear guidelines form the EU Commission is a very unsatisfying situation. Classifying manure as waste would be a serious discrimination since manure that is directly used in agriculture will not be waste. The future use of manure in AD plants could be significantly reduced if it is classified as waste.

The strong development in the area of AD and of Renewable Energy in general could only be achieved thanks to the German Renewable Energy Sources Act (EEG), the most powerful instrument promoting renewable energy. On the 1st of January 2012 the new EEG 2012 came into force installing a new concept for compensating the electricity production from biogas. In the “old” EEG of 2009 with different bonuses, manure was classified as renewable resource. Thus, manure was also used in agricultural plants substituting energy crops. With the commencement of the “new” EEG on the 1st of 2012 there will be a separate compensation for AD plants treating 90 % of municipal biowaste (16 ct/kWh < 500 kW, 14 ct/kWh < 20 MW). For all other plants there will be no separation between waste and energy crops. All kind of feedstock can be used in every new plant. However there will be an additional compensation for different feedstock. For energy crops there will be an additional compensation (6 ct/kWh < 500 kW/kWh, 5 ct/kWh < 750 kW, 4 ct/kWh < 5 MW) and for manure and other recoverable material like the cup-plant 8 ct/kWh until 5 MW. This additional compensation is only paid for the energetic part of each feedstock. There is only one additional bonus available left for the injection of upgraded biomethane into the natural gas grid.

This shows a new way of compensation in contrast to the EEG 2009. For small scale AD plants treating energy crops the compensation of the EEG 2012 will be lower in comparison to the EEG 2009 and higher for large scale plants upgrading biogas. A much higher compensation can be reached for the electricity production of AD plants treating waste which will lead to lower revenues for the acceptance of waste at the AD plant. This is a big problem for existing waste treatment plants falling under the compensation scheme of the EEG 2009 that cannot change into the new compensation scheme. The realisation of new projects will show if the new concept of compensation energy from biogas leads to a further development in the biogas sector of Germany in the next years.

1 INTRODUCTION

In 2011, renewable energies covered 12.2 % of German primary energy supply and 20.0 % of German electricity production (BMU, 2012). This goal could only be achieved due to the German Renewable Energy Sources Act (EEG), the most powerful instrument promoting renewable energy. The EEG demands a share of renewable energy at the electricity production of 35 % by the end of 2020 with an increase every decade up to 80 % by the end of 2050.

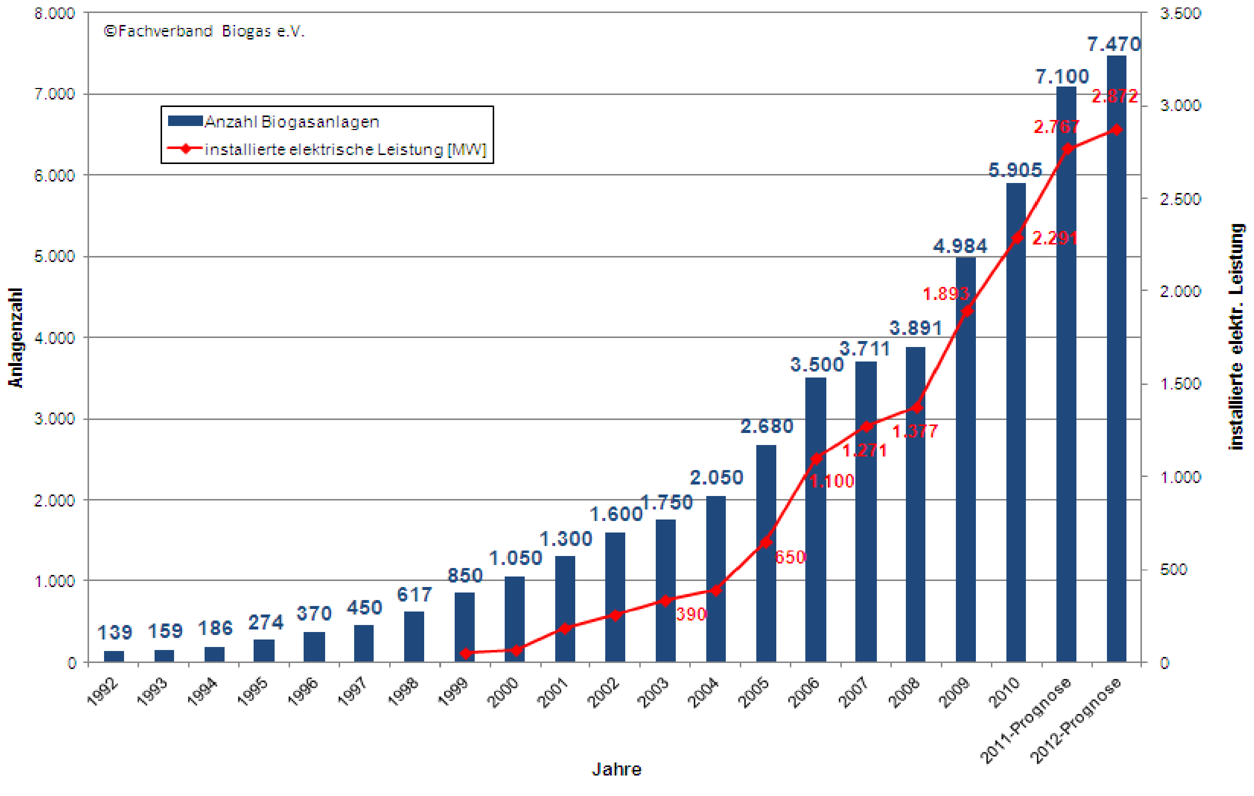
In 2011, biogas had a share of 14.4 % at the electricity production of renewable energies and a share of 2.4 % at the total electricity production as well a share of 1.0 % at the heat production in Germany. The most common feedstock for biogas plants are energy crops and liquid manure in Germany. But also the use of organic residues like plant remains, grass, overlaid food, leftovers, separated municipal biowaste, residuals from industries and agriculture, fats, flotation tailings, slaughterhouse waste and other animal by-products etc. are fermented in German biogas plants. The digestate is used as organic fertiliser mostly in agricultural application. The possibility of upgrading digestates like separating, composting, pelletizing and filtering (UF, RO) reduces the costs of transportation and can lead to new markets.

2 BIOGAS PRODUCTION IN GERMANY

Biogas is an all-rounder among renewable energies. Organic matter can be converted to electricity and heat or be used as a fuel and natural gas equivalent via biogas. Biogas plants improve the independence and economic strength of municipalities and regions. Germany is the undisputed market leader in this versatile and forward looking industry.

Since 1992 the numbers of biogas plants and their installed capacity is increasing constantly. By the end of 2011 approximately 7.000 biogas plants with an installed electric capacity of 2.700 Megawatt supply German households, industry and public facilities with 18 billion kWh electricity and heat. Upgraded biomethane can be fed into the natural gas grid or used as fuel in natural gas-powered vehicles without technical modifications. By the end of 2011, about 60 AD plants fed approximately 260 million cubic metres of biomethane into the German gas grid.

Thus, the production of biogas contributes with a yearly saving of nearly 12 million tons CO_2-equivalent effective to the climate protection. The following graph shows the development of the German biogas market with the number of biogas plants in blue columns and the installed electricity capacity as MW in the red line.



Number of biogas plants & installed electricity capacity in Germany in MW

3 CYCLE OF ORGANIC RESIDUES

According to the German Federal Statistical Office, 13.2 million tons of German biowaste were separately collected in 2009 with a share of 7.8 million tons of biowaste from households (biobin). Four million tonnes of this separately collected biowaste were digested in AD plants. The group of animal by-products (ABP) which were until now not legally classified as biowaste in German legislation is currently not yet included in this figure. Estimated two additional million tonnes of ABP are digested in German AD plants.

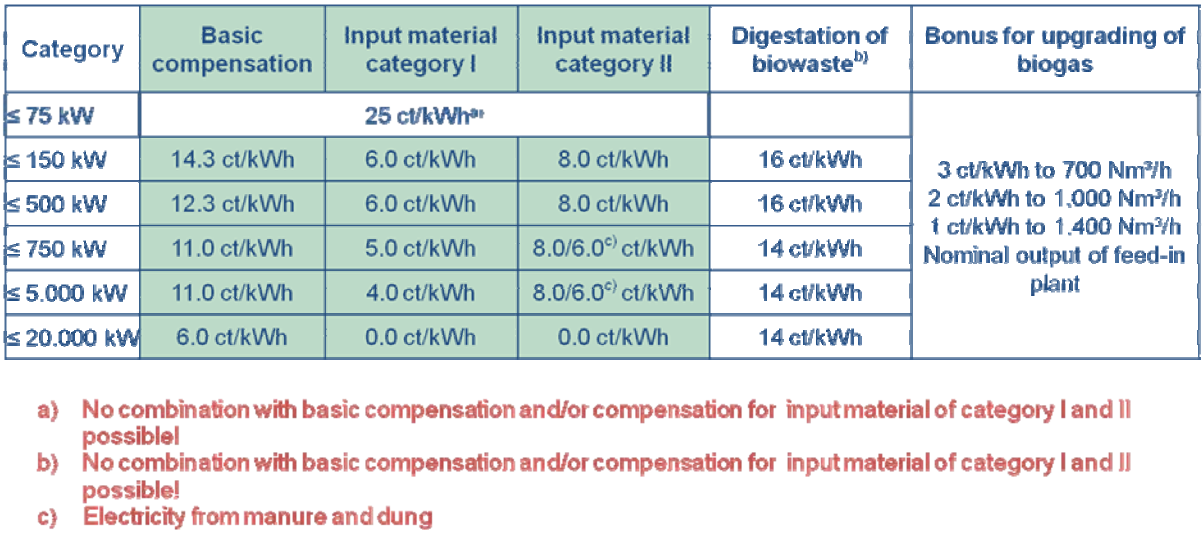
Thus, six million tons of biogenic residues (not including manure) from biowaste and ABP are treated in nearly 1.000 AD plants with an installed capacity of 520 MW. In Germany approximately 20 million tonnes of manure (15 % of all accruing manure in Germany) is treated in AD plants with an installed capacity of 230 MW. The use of manure in the AD process has the second highest energy potential after energy crops in Germany. But the digestion of household wastes and landscape material has also a big potential in Germany.

The use of the digestate as valuable fertiliser displays an important part in the contribution to the climate protection. Replacing one kilogram of mineral fertilizer saves over six kilograms of CO_2 which would be released by the production of the mineral fertilizer. The production of biogas describes the conjunction of energetic and material use of biomass. The nutrient cycle is closed, resources are conserved saving fossil and mineral materials and green-house-emissions, especially with the treatment of organic residues and manure.

4 GERMAN RENEWABLE ENERGY SOURCES ACT (EEG)

The EEG creates a stable reliable legal and economic framework for sustainable growth of renewable energy mostly in the electricity sector. It guarantees connection to the electricity grid for all facilities producing electricity from renewable energies, as well as allowing them to feed in and sell the electricity they generate for a period of 20 years at guaranteed minimum rates specified in the Act. These tariffs vary depending on the technology used and the size of the plants. With the commencement of the “new” EEG on the 1st of 2012 the compensation for electricity produced from biogas has changed mainly from a system of different bonuses to different input material categories. Also two new separate classes of special compensation were created. One is the higher compensation of 25 ct/kWh for AD plants using 80 % manure with an installed capacity up to 75 kW. The other is a higher compensation for plants treating 90 % of municipal biowaste with 16 ct/kWh up to 500 kW and 14 ct/kWh up to 20 MW installed capacity.

For the basic compensation there is no separation between biogas plants treating waste or energy crops anymore. All kind of feedstock can be used in every new plant. However there will be an additional compensation for different feedstock. For energy crops there will be an additional compensation (6 ct/kWh < 500 kW/kWh, 5 ct/kWh < 750 kW, 4 ct/kWh < 5 MW) and for manure and other recoverable material like the cup-plant 8 ct/kWh until 5 MW. This additional compensation is only paid for the energetic part of each feedstock. There is only one additional bonus available left for the injection of upgraded biomethane into the natural gas grid. The following graph shows the tariffs for electricity from biogas in €-Cent per kWh up to a capacity of 20 MW.



Category	Basic compensation	Input material category I	Input material category II	Digestation of biowaste[b)]	Bonus for upgrading of biogas
≤ 75 kW	25 ct/kWh[a)]				3 ct/kWh to 700 Nm³/h 2 ct/kWh to 1.000 Nm³/h 1 ct/kWh to 1.400 Nm³/h Nominal output of feed-in plant
≤ 150 kW	14.3 ct/kWh	6.0 ct/kWh	8.0 ct/kWh	16 ct/kWh	
≤ 500 kW	12.3 ct/kWh	6.0 ct/kWh	8.0 ct/kWh	16 ct/kWh	
≤ 750 kW	11.0 ct/kWh	5.0 ct/kWh	8.0/6.0[c)] ct/kWh	14 ct/kWh	
≤ 5.000 kW	11.0 ct/kWh	4.0 ct/kWh	8.0/6.0[c)] ct/kWh	14 ct/kWh	
≤ 20.000 kW	6.0 ct/kWh	0.0 ct/kWh	0.0 ct/kWh	14 ct/kWh	

a) No combination with basic compensation and/or compensation for input material of category I and II possible!
b) No combination with basic compensation and/or compensation for input material of category I and II possible!
c) Electricity from manure and dung

Fe

Feed-in tariffs for electricity produced from biogas in plants with a capacity of up to 20 MW

5 BIOGAS PRODUCTION IN EUROPE

According to EurObserv'ER, biogas has reached a European production of primary energy of 10.9 million tons of oil equivalents (toe) and a European production of electricity of 30.3 TWh in 2010. The share from agricultural biogas units constituted 6,938 toe. Here the feedstock are energy crops, liquid manure but also co-digestion plants, using organic waste in combination, are included in opposition to urban and industrial waste treatment plants, which are listed in the sludge gas plants with a total production of primary energy of 1,075 toe in 2009. The turnover was at over € 4 billon and counts roughly 53,000 jobs in Europe.

6 GERMAN BIOGAS ASSOCIATION (FACHVERBAND BIOGAS E.V.)

The German Biogas Association unites owners, manufacturers and planners of biogas plants, representatives of science and research as well as interested parties from all over Germany. Since it was formed in 1992, the Association has grown into Europe's strongest biogas organisation with a membership of over 4,500.

The German Biogas Association promotes the extensive use of biogas technologies at European, national and regional level, and to this end acts as a powerful political pressure group. In addition, the Association promotes the exchange of experience in the biogas sector, e.g., by collecting and communicating scientific findings and practical experience as well as organizing conferences, exhibitions and other events.

Through participation in European projects and its membership of the European Compost Network (ECN), the European Biogas Association (EBA) and of the European Renewable Energies Federation (EREF) umbrella organization, the German Biogas Association is an active initiator and promoter of the international pooling of experience.

The German Biogas Association is represented by a committee which is elected by the general meeting of members. Over 4,500 members of the Association in Germany are organised in 23 regional groups. This ensures efficient networking of competent contacts regionally, nationally and internationally.

7 EUROPEAN BIOGAS ASSOCIATION

The European Biogas Association (EBA) was founded in February 2009 as a Belgian non-profit organisation aiming to promote the deployment of sustainable biogas production and use in Europe. This covers all energetic applications of biogas like heat, electricity and automotive fuel.

Three years after foundation, EBA's membership comprises 49 national biogas associations, institutes and companies from 18 countries all across Europe. The member associations cover the majority of producers, companies, consultants and researchers in the field of biogas within Europe.

The growing membership provides a well-established network and communication platform for exchanging information and expertise. The network is basis to provide information on market opportunities and future market development in the different countries. EBA unites Europe's most experienced biogas experts and has highly educated and skilled staff providing policy advice, know-how and information to promote beneficial legislation and framework conditions in the field of biogas. EBA is member of the European Renewable Energies Federation (EREF) and the European Biowaste Alliance.

EBA's strategy defines three priorities: Establish biogas as an important part of Europe's energy mix, promote source separation of household waste to increase the gas potential and support the production of biomethane as vehicle fuel. For more information please visit: www.european-biogas.eu

(133) COMPOST & BIOGAS MARKET IN FRANCE : CURRENT STATE & TRENDS FOR ORGANIC WASTE MANAGEMENT (sewage sludge, MSW, agroindustrial residues & farm residues...)

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ABSTRACT

Given the European context on end-of-waste criteria on biodegradable waste subject to biological treatment, but also the national context on fertilization & renewable energy production from biogas, the purpose of this paper is to address current and future management options developed in France, a major agricultural country, for urban sewage sludge, mixed and sorted municipal solid waste, agro-industrial wastes and farm residues.

Continuously produced by our modern society, those organic and wet biowaste demand efficient management in order to prevent nuisances, but also require special attention to ensure proper fertilization of crops.

By analyzing, at both national and regional levels, the different management routes including direct landspreading, but also composting & anaerobic digestion, this research deals with the variety of solutions that characterize the organic sector.

Based upon various ongoing industrial experiences, a special attention is paid to agronomic recycling and its potential effects on human health but also to the positive effects of compost application.

Besides, in regards to the Aarhus Convention adopted on 25th June 1998 on Access to Information, Public Participation in Decision-making and Access to Justice in Environmental Matters, this paper also addresses public participation dealing with biodegradable waste management routes (recycling, incineration, landfilling).

To some extent, the economic context is presented for the French context.

Eventually, this paper considers 3 major options for the future of biodegradable waste management market with its pros & cons.

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(150) COMPOST AND FERMENTATION RESIDUES USED AS LITTER MATERIALS IN DAIRY FARMING

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EXECUTIVE SUMMARY

In dairy farming, litter materials have an essential influence on health and production performance of animals and therefore, on the quality of the comestible milk. As straw has recently become an essential cost factor in dairy farming, alternative options are in demand. In this study, selected materials were tested for their hygienic-microbiological quality, i.e. compost barns, litter materials from dry fermentation residues as well as composted fermentation residues, composted garden and forest waste.

Representative collected composite samples from the litter materials are taken from commercial farms. From compost barns 20 collected composite samples (10 from surface and 10 in a depth of 40 cm) were taken. From fermentation residues five collected composite samples of the fresh material and five of the dry material were analyzed. These materials were not used as bedding material at the time-point of sampling. From composted fermentation residues five representative samples were taken from the cubicals equally from composted garden and forest waste. In addition, three collected composite samples were tested from this fresh compost material. All samples were quantitatively tested for coliform bacteria, enterococci, salmonellae, streptococci and *staphylococci.*

Investigated materials showed varying results. The average bacterial numbers for *E. coli* and coliform bacteria in compost ranged between 10^5 and 10^6 CFU/g, respectively. In fermentation residues the numbers were considerable lower. In fresh material coliform bacteria were below the limit of detection and in dry material below 1.44 CFU/g. In used bedding material enterococci were verified in average between 10^3 and 10^5 CFU/g. In comparison, 10^1 CFU per gram of enterococci were found in fresh fermentation residues and 10^4 CFU per gram in dry fermentation residues. Salmonellae were not found in any samples. There were only a sporadically appearance of streptococci and staphylococci.

In summary, the content of *E. coli* and enterococci was quite high in all compost material. Therefore, this material can be recommended in a limited way as litter material. The variation of the results might be due to the sampling scheme since contamination with animal’s excrements could not be avoided.

Dried fermentation residues seem to be more promising, but more hygienic and microbiological analyses of the already interspersed litter material are necessary. From the view of prevention for epizootic diseases some more requirements and arrangements should be taken in account. In any case the material should be in a safe condition for reasons associated with hygienic control of epidemics. This means, that the fermentation residues should either come from a thermophilic actuated biogas plant or that the substrates are pasteurized before or the anaerobic fermentation. A separation of the fermentation residues after mesophilic digestion, followed by pasteurizing of the liquid phase and appropriate composting of the solid matter should also be possible. Beyond salmonellae, the deactivation of *Mycobacterium spp.*, mycoplasmas and coxiella should be assured. For that purpose further research efforts are needed.

1 INTRODUCTION

1.1 Background

In dairy farming the bedding material is very important. However, economy, working aspects, animal welfare, and hygiene must be optimized in each farm. There are a lot of possibilities, e.g. traditional straw, mixtures of different other organic materials, but also practices without any bedding materials. One common aim of all these methods is to provide a long time of recumbency for the cows. This is very important for a high milk production. Moreover, it should be not too expensive and the workload should not be too high. Last but not least a high hygienic quality is critical for healthy animals and subsequently for secure and faultless comestible milk.

Price of straw is increasing constantly. In addition, there are also a lot of problems in systems without bedding materials in cattle husbandries. Therefore, some alternatives are searched.

In this project selected alternative materials were tested particularly with regard to their microbiological contamination and hygiene.

The first husbandry system uses a continuing composting process, so-called compost barns. These barns should combine consumer requirement for animal welfare with high demand for milk quality and limited workload.

Another innovative possibility is the usage of dried fermentation residues from digester. Through the increasing number of digester, there arise high amounts of fermentation residues. Possibilities for the usage of these materials are searched. One possibility is to use the dried material as bedding material in cow barns.

Furthermore, there is a look at other compost materials, i.e. composted garden and forest waste as well as composted fermentation residues of communal biological waste recycling. The possibility of these materials as bedding material will be examined.

All putative bedding materials were tested for their content of coliform bacteria, enterococci, salmonellae, streptococci and staphylococci.

1.2 Research objectives

1.2.1 Compost barns

In literature, there is a continuing composting process in the bedding of compost barns. The natural composting process is used for optimal cow comfort, prevention of injuries, improvement of milk quality and better hygienic conditions of the comfort area.

This system worked with very different litter materials and the management must be adapted for adequate climatic and economically conditions. Thereby, there are a lot of different keeping barns, which are all called `compost barn´.

In general compost barns are a loose housing system with a bedded pack area and a concrete feed alley. There is a wall for separating these areas with several walkways to avoid carrying over bedding material from the lying area to the feed alley.

Different materials can be used for the bedded pack, e. g. wood shavings or wood chips, but also different agricultural and industrial waste, e. g. oat hulls and strawdust or residuals from the paper industry. The pack is stirred twice a day to mix manure and urine on the surface into the bedded pack and provide a good aeration and drying of the pack (JANNI, RENEAU 2005; JANNI et al. 2006; KLAAS et al. 2010).

This project looked at two compost barns. One of these uses wood chips and the other wood shavings.

To avoid endangering the composting process, 7.4 up to 15 m² bedding area per cow are necessary according to compost barn system and circumstances. It is important that there are not too many cows in these barns. Besides this, moisture ingress from outside the barn should be avoided and good ventilation is needed (JANNI, RENEAU 2005; JANNI et al. 2006; KLAAS et al. 2010).

1.2.2 Fermentation residues

The number of biogas plants is increasing; therefore a reasonable usage of fermentation residues is important. One possibility as aforementioned is their usage as litter material. The dried material can be used loose or compressed to pellets in cubicals. It can be used for high bed as well as for low bed cubicals; though in low bed cubicals the required amount has to be higher. Field tests of different research institutions have shown a good absorbing capacity of the loose material and a high acceptance by the cattle. However, cows carry a great amount of the loose bedding material out of the cubicals which requires a lot of supply. The pelletized fermentation residues have not been accepted in the same way; in addition there have been problems with the absorbing capacity (ROHWEDER 2010).

One very important condition for the use of fermentation residues must be the epidemiological safety of the products.

1.2.3 Compost

Several farmers need compost from different basic materials as litter material in cubicals. Thereby composting garden and forest waste as well as composting fermentation residues come into consideration. Both possibilities are analyzed in this examination.

A producer composts garden and forest waste by himself and scatters the prepared compost which is mixed with feed lime as litter material.

Another farmer purchase composted fermentation residues of communal biological waste recycling.

2 METHODOLOGY

2.1 Sampling

Sampling was performed on a total of five commercial farms. The samples from compost barns were taken at two farms in Austria, i.e. 20 collected composite samples. In each case these are mixtures of five primary samples. Ten are taken from surface and ten in a depth of 40 cm. As sampling location there were selected representative areas all over the bedding area.

One of these farms needs the barn in winter month, about October till May, and needs as base wood chips. The other farm needs the barn all over the year and uses wood shavings.

Fermentation residues were derived from one biogas plant in Baden-Württemberg. Five representative samples consisting of five single samples were collected from fresh fermentation residues and from dried fermentation residues, respectively. These materials were not used as litter material, so far.

Composted fermentation residues from a loose-housing stable with cubicals in Switzerland were collected (five gross samples) and tested.

Ready compost derived from garden and forest wastes were mixed with feed chalk and used as litter material in cubicals in Switzerland. In this farm, samples were taken accordingly from the cubicals as well as from the fresh compost outside the stable.

2.2 Coliform bacteria

20 g of each sample was mixed with 180 ml sodium chloride solution (0.85 %). 1 ml hereof was used for a tenfold serial dilution. After inoculation of each dilution into Flourocult Bouillon and incubation at 37°C for 48 hours building of gas, fluorescence and production of indol was evaluated. If all factors are positive, *E. coli* is verified.

2.3 Enterococci

20 g of each sample was mixed with 180 ml sodium chloride solution (0.85 %). 1 ml hereof was used to inoculate 9 ml AD-Bouillon. After 48 h incubation at 37 °C it was applied on Kanamycin Aesculin Azide Selective Medium and after further 48 h subcultivation was performed to identify enterococci.

2.4 Salmonellae

50 g of each sample was mixed with 450 ml buffered peptone water containing novobiocin and incubated overnight. 100 µl each were transferred to 10 ml Rappaport-Vasiliadis bouillon and incubated for 24 h at 37° C and 43°C, respectively. Afterwards, subcultivations were performed on XLD and BPLA agar plates. Colonies indicative for salmonellae were further differentiated serologically and biochemically.

2.5 Staphylococci/ Streptococci

A solution of the sample will be applied on Columbia Agar containing sheep blood. After incubation pure cultures from colonies indicative for streptococci or staphylococci are produced. Further differentiation is done by gram staining as well as test of catalase and cytochrome oxidase activity

TABLE 1 **Schema of farms, litter materials and samplings**

Farm	**Litter materials**	**Samples**
Compost barn 1	Wood chips	20 gross samples • 10 surface samples • 10 in a depth of 40 cm
Compost barn 2	Wood shavings	20 gross samples • 10 surface samples • 10 in a depth of 40 cm
Biogas plant	Dried fermentation residues	10 gross samples • 5 fresh material • 5 dried material
loose-housing stable with resting pens 1	Compostet fermentaion residues	5 gross samples
loose-housing stable with resting pens 2	Composted garden and forest waste	8 gross samples • 3 fresh compost • 5 from boxes

3 RESULTS

3.1 Compost barns

The contamination of the tested compost barns with *E. coli*/ coliform bacteria and *enterococci* is seen in table 2. In compost barn with wood shavings the average contamination with *E. coli* was 4.44 x 10^{6} CFU/g within the surface samples and 2.0 x 10^{6} CFU/g within the samples taken in 40 cm depth, respectively. Therefore, it is slightly higher than in the compost barn with wood chips where average contamination was 1.09 x 10^{6} CFU/g on the surface and 5.14 x 10^{5} CFU/g in 40 cm depth.

The bacterial counts for enterococci were lower. Average numbers ranged between 8.59 x 10^{3} CFU/g and 3.25 x 10^{5} CFU/g. Thereby, accounts in the barn with wood chips were also lower in comparison to the barn with wood shavings.

TABLE 2 **Contamination with *E. coli* and *enterococci* in two compost barns (in CFU/g)**

Litter material		**Average**	**Minimum**	**Maximum**	**Median**
	E. coli				
Wood chips	Surface (n=10)	1.09x10^{6}	2.4x10^{4}	9.3x10^{6}	4.3x10^{4}
	40 cm depth (n=10)	5.14x10^{5}	2.4x10^{3}	4.3x10^{6}	2.4x10^{4}
Wood shavings	Surface (n=10)	4.44x10^{6}	3.8x10^{5}	1.5x10^{7}	2,4x10^{6}
	40 cm depth (n=10)	2.0x10^{6}	2.4x10^{4}	1.5x10^{7}	4.3x10^{5}
	Enterococci				
Wood chips	Surface (n=10)	1.62x10^{4}	1.5x10^{2}	7.5x10^{4}	6.4x10^{3}
	40 cm depth (n=10)	8.59x10^{3}	2.3x10^{1}	4.3x10^{4}	6.4x10^{3}
Wood shavings	Surface (n=10)	3.25x10^{5}	2.3x10^{1}	1.1x10^{6}	1.95x10^{5}
	40 cm depth (n=10)	6.5*10^{4}	9.3*10^{3}	2.1x10^{5}	3.35x10^{4}

Salmonellae could not be detected in any of the samples.

In the barn with wood chips *staphylococci* were detected in two samples on the surface and one sample in 40 cm depth. *Streptococci* were unverifiable.

In the barn with wood shavings *staphylococci* were detected in two samples on the surface only. *Streptococci* were unverifiable, too.

3.2 Fermentation residues

The results of the tested fermentation residues are summarized in table 3. In fresh material *E. coli* were unverifiable and *enterococci* were verified to a amount of 2.4 x 10² CFU/g. After drying it increased.

TABLE 3 **Contamination with *E. coli* and *enterococci* in fermentation residues (in CFU/g)**

	Average	**Minimum**	**Maximum**	**Median**
	E. coli			
Fresh material (n=5)	n.n.	n.n.	n.n.	n.n.
Dry material (n=5)	1.44×10^{0}	n.n.	3.6×10^{0}	n.n.
	Enterococci			
Fresh material (n=5)	5.17×10^{1}	n.n.	2.4×10^{2}	3.6×10^{0}
Dry material (n=5)	5.09×10^{4}	4.3×10^{2}	2.4×10^{5}	4.3×10^{3}

Salmonellae were not detected.

In fresh fermentation residues, neither *staphylococci* nor *streptococci* were detected. In two samples of the dried material staphylococci, but no streptococci could be proved.

3.3 Compost

Table 4 shows the results of the composted fermentation residues. The average contamination with *E. coli* was 6.54 x 10^{5} CFU/g and with enterococci 1.98 x 10^{4} CFU/g.

TABLE 4 **Contamination with *E. coli* and *enterococci* in compost from fermentation residues (in CFU/g)**

Average	**Minimum**	**Maximum**	**Median**
***E. coli* (n=5)**			
$6{,}54 * 10^{5}$	1.5×10^{2}	2.4×10^{6}	4.3×10^{5}
***Enterococci* (n=5)**			
$1{,}98 * 10^{4}$	2.3×10^{2}	4.6×10^{4}	2.1×10^{4}

In table 5 the results of composted garden and forest waste are shown. In composted, but unused material *E. coli* was detected amounting between 9.2 x 10^{0} CFU/g and 4.3 x 10^{1} CFU/g. In used material the average content was 2.83*10^{5} CFU/g.

In unused compost the average content of *enterococci* was 2.24 x 10² CFU/g and in used compost 8.85 x 10^{5} CFU/g.

TABLE 5 **Contamination with *E. coli* and enterococci in Compost from garden and forest waste (in CFU/g)**

	Average	**Minimum**	**Maximum**	**Median**
	E. coli			
Compost outside the cow barn (n=3)	2.51×10^{1}	9.2×10^{0}	4.3×10^{1}	2.3×10^{1}
Compost inside the cow barn (n=5)	2.83×10^{5}	1.5×10^{3}	9.3×10^{5}	4.3×10^{4}
	Enterococci			
Compost outside the cow barn (n=3)	2.24×10^{2}	9.2×10^{1}	4.3×10^{2}	1.5×10^{2}
Compost inside the cow barn (n=5)	8.85×10^{5}	4.3×10^{3}	4.3×10^{6}	4.6×10^{4}

Salmonellae could not be detected verify in any of the samples.

In one sample of composted fermentation residues streptococci could be verified. Staphylococci were detected in one sample of the composted garden and forest waste. Neither streptococci nor staphylococci could be detected in any of the other compost samples.

4 DISCUSSION

The tested bedding materials vary considerably in hygienic quality. There is a big range in the results of the different gross samples, with the exception of the fresh fermentation residues. This suggests a different contamination and disposition with microorganism. Because of the way of sampling it was inevitable that the samples of used litter are mixed with fresh feces. Thereby the content of tested germs increases considerably.

4.1 Compost barns

The contamination with enterococci and *E. coli* in the tested compost barns is relatively high, because the composting process is insufficient. The machining depth is too low, particularly in the barn with wood shavings, thereby the oxygenation is too low. Thus the activity of the microorganisms, which are important for the composting process, is blocked. So, the composting process and with it the disinfection of the material fall through.

In the barn with wood chips 43 °C were measured at a depth of 40 cm. In the compost barn with wood shavings the temperature increases hardly above 26 °C. For an adequate disinfection, temperatures over 55 °C for any length of time are needed, so the temperatures in both barns are assessed as too low.

According to KRISTULA et al. (2005) coliform bacteria $>10^6$ CFU/g in bedding material is a risk. So both compost barns are on average near this limit, some samples already above it.

The content of enterococci are altogether lower. However this genus is estimated as inapplicably for assessment of the pathogenic potential of dairy husbandries.

It`s positive that there were not detected any *salmonellae* and *streptococci* in this two barns. *Staphylococci* were detected just sporadically and these are also expected to be found in used litter material.

4.2 Fermentation residues

In the fresh fermentation residues *E. coli* are below limit of detection and the content of enterococci were very low. Salmonellae, staphylococci and streptococci were also unverifiable. This can be traced back to the heating during the fermentation process. The contamination of the dried material was slightly higher, although there was a second heating during the dehydration. This indicates a contamination during transport or storage. It`s possible that the tractor bucket was contaminated after carrying manure. When the fermentation residues were carried with the same tractor bucket, the material will be contaminated, too. Another possibility is a contamination by manure near the storage area.

But also the content of the dried material with the tested microorganism is so low that they can used as bedding material without difficulty. But to assess this certainly, analysis of the used material are required. Only then a realistic comparison with the other materials is possible. When the substrate of the biogas plant includes cattle, hog or poultry excrement, the fermentation residues have to come under a treatment to inactivate salmonellae as well as *Mycobacterium spp.*, mycoplasmas and coxiella. This proceedings have to be subject to validation.

4.3 Compost

The microbial contamination of the composted and interspersed fermentation residues lies in a similar range as the compost barns. But the composting process occurs in composting firm, hence it is assumed that the composting process is proceeded accurately and thereby the disinfection is achieved. So the high contents are caused by a later contamination during the storage near the barn and in the cubicals.

The material, which the farmer composted by himself has low contents of *E. coli* and enterococci when it`s still outside the barn. Besides, staphylococci, streptococci and salmonellae were unverifiable. From this, it is assumed that there was a good composting process. The used compost is comparable to the other samples, which were taken from the used bedding materials in cow barns. Thus the higher contents can only be explained by contamination with excrement of the animals. Also these contents are in an area, which is acceptable for dairy husbandry.

5 CONCLUSIONS

Generally, all analysed materials are acceptable for dairy husbandry. Certainly all materials which were inside the barn are near the limit to be risky for udder health. It was unable to test dried fermentation residues, which were used as bedding material. Here akin results are due.

From the view of a safe condition for reasons associated with hygienic control of epidemics the compost materials must be complete sanitized and in particular the fermentation residues, which were used as litter material, have to come from a thermophilic actuated biogas plant or from such digester in which the substrates are pasteurized before the anaerobic fermentation. Only in this way it can be prevent that pathogens spread out unregulated. Particularly, this applies to the selling from fermentation residues as bedding material, because thereby separate farms will connected.
When animal byproducts are used as substrate in biogas plants, it`s important that the inactivation of salmonellae *Mycobacterium spp.*, mycoplasmas and coxiella are secured. For that purpose further research efforts are needed.

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(170) MANEV LIFE+ PROJECT FOR MANURE MANAGEMENT IN EUROPE: COMPOSTING IN THE REGION OF MURCIA, SPAIN

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EXECUTIVE SUMMARY

The project LIFE+ *Evaluation of manure management and treatment technology for environmental protection and sustainable livestock farming in Europe* - MANEV is included in the area Environment Policy and Governance. It involves partners from 8 regions in Europe from Spain, Italy, Denmark and Poland. The main objectives are:

- To improve environmental protection including the contribution on the GHG emissions reduction, and the sustainability of the pig farming sector, by promoting the use of manure treatment technology in different livestock saturated or surplus zones of Europe;
- To unify criteria for the evaluation of different manure treatment technology systems and management schemes;
- To develop a common protocol for all the European zones, for the evaluation of manure treatment technology and management schemes, considering environmental, technological, energetic, economic, and legal factors;
- To develop a decision supporting and planning tool to evaluate different manure treatment and management strategies for being used by institutions, associations, administration and also to provide information for farms;
- To define the fertilising properties of directly applied manure and of every treatment effluent with the aim to get a real value in the market;
- To balance the know-how related to the treatment technologies and management methods, as well as its own strengthens and weaknesses, within the different countries and areas in Europe;
- To disseminate the knowledge for manure treatment methods and technologies among farmers and all stakeholders.

In order to reach those objectives, 13 different treatment technologies and manure management schemes (full-scale), located in surplus zones will be evaluated in Europe, including: 3 separation systems; 9 anaerobic digestion; 6 nitrogen removal technologies; 2 nitrogen recovery technologies; 2 composting; 1 direct application of manure in soils.

Every partner will monitor and evaluate treatment plants and manure management schemes according to the common protocol. As a result of this analysis the Life Cycle Assessment of every scheme will be created, which includes the three pollution potentials: global warning, eutrophication and acidification, as well as energetic and economic aspects.

The action being carried out in the Region of Murcia consists on the monitoring and evaluation of manure management strategies based on composting. The work is designed in the following steps:

- Evaluation of the pig slurry distribution in the Region of Murcia and identification of the surplus areas;
- Identification of appropriate agricultural soils for pig slurry application according to the crop and nutrient balances;
- Establishment of the technical requirements for pig slurry composting: the feasibility of composting as an alternative for recycling pig manure at the farm level in the Region of Murcia will be determined, according to the characteristics of the pig slurry produced, the available solid-liquid separation systems, and the bulking materials available in the surplus areas;
- Identification of the benefits and constraints in manure and slurry composting: economic, environmental and agricultural;
- Feasibility of the combination of anaerobic digestion and composting of the digestate for manure management;
- Environmental and quality indicators of composting technology;
- Implication of composting on the carbon cycle: C-conservation and sequestration.

During the first year the work has been developed in the first 3 steps. The results are integrated into a geographical distribution of pig slurry production and agricultural crops, using GIS. The main area of pig slurry production has been also identified as a vulnerable zone for nitrate pollution, which conditions the management of pig slurry.

Differences in pig slurry composition have been revealed according to the kind of animal and storage system. The composting system has been established in a selected farm with a solid-liquid separation system.

INTRODUCTION

1.1 Background

The environmental impact of high density intensive pig farming zones leads to qualitative deterioration of air (ammonia, nitrous oxide and methane), water (by nitrates, phosphates and organic matter) and soil (phosphates and heavy metals). Of particular importance are nitrous oxide (N_2O), methane (CH_4) and ammonia (NH_3) emissions to the atmosphere, and nitrate (NO_3^-) leached to watercourses. Agriculture is a major source of the three gases, for which national ceiling targets (NH_3) or target emission reductions (CH_4 and N_2O) have been established (Petersen et al. 2007). Nitrate leaching contributes to eutrophication and may pose a threat to drinking water quality. Furthermore, there are also the related disease risks especially when pig slurry is incorrectly handled (Burton and Turner, 2003).

The substances that most contribute to the environmental impact from the pig product chain are nitrous oxide (53%) and methane (10%) for global warming potential, nitrate (63%) and ammonia (30%) for eutrophication potential, ammonia (84%) for acidification potential. These indicated contributions to global warming, eutrophication and acidification arise from production of feed and handling of manure. The latter is specified in two steps as the pig housing and during storage. Furthermore, when manure and slurry are applied to fields it has been shown to make a significant contribution to eutrophication potential (Dalgaard et al., 2007; Flessa et al., 2002). An increasing number of livestock farms become specialised with insufficient land for efficient use of manure nutrients for crop production, use of manure as feed increases hygienic risks, and ineffective manure handling on small-holdings can lead to excessive losses (Petersen, et al. 2007).

Efforts to recycle nutrients in livestock manure for crop production will contribute for reducing environmental pollution problems, providing nutrients to crops, and reducing the need for mineral fertilisers, although general solutions are difficult to devise in view of the diversity in production systems, management strategies and legislation between countries and regions (Petersen et al. 2007). Also, large production units can also lead to high energy use for transport of livestock manure to be recycled in crop production, and the risk of disease spreading among livestock will increase.

1.2 Research objectives

The objective of the project is to demonstrate that both the use of treatment technology and an environmentally correct management scheme of pig manure can contribute to a reduction of GHG emissions and simultaneously improve the farmers' sustainability.

For that the criteria for the evaluation of different manure treatment technology systems and management schemes (mainly for the pig sector) will be unified in the different zones of Europe; a **common evaluation and monitoring protocol** for all the different European zones, which defines the evaluation of manure treatment technology and management schemes taking into account environmental, technological, energetic, economic, legal and health factors will be developed, as well as a **decision supporting and planning tool** to evaluate different manure treatment and management strategies for being used by institutions, associations, administration and also provides information for individual farms.

Every partner monitors and evaluates a treatment plant and manure management scheme according to the common protocol. As a result of this analysis the Life Cycle Assessment of every scheme will be created, which includes the three pollution potentials: global warning; eutrophication; acidification, energy balancing and economic aspects.

2 METHODOLOGY

In order to demonstrate the contribution on the reduction of the GHG emissions and to improve the farmers' sustainability, 13 different treatment technologies and manure management schemes (full-scale), located in 8 different surplus zones in 4 different countries, will be evaluated in Europe. The different treatment technologies and management schemes to be evaluated across Europe, involve: separation systems; anaerobic digestion; N removal technologies, composting and direct use to agricultural soil for providing plant nutrients. The action being carried out in the Region of Murcia by the CEBAS-CSIC consists in monitoring and evaluation of manure management strategies based on composting.

2.1 Common evaluation and monitoring protocol

The common evaluation and monitoring protocol (CEMP) is a guideline to be followed by all partners in order to obtain comparable results in the monitoring of each technology and develop a software tool which covers all these useful information to assess and make decisions about the most appropriate management and technology solution for a local problem. This protocol will specify parameters, evaluation procedures and functional units agreed by all partners to get the maximum profit of their research and assessment in this software tool.

Functional unit of all the assessments will be m^3 of pig slurry which can be also easily transformed into Mg of manure using density parameter. Monitoring period of every technology will include a whole natural year in order to obtain a whole assessment of how these systems answer different weather conditions.

Performance of every technology will be related to the following minimum parameters: main nutrient content (total-N, NH_4-N, P, K), organic matter (VS and COD), pathogen agents (*E. coli* and Salmonella), pH, electrical conductivity and CH_4 content in biogas produced in the case of biogas plants. Every technology will have its own sampling protocol in which will be included the most relevant parameters in each case.

Also, an environmental monitoring is going to be carried out in every scenario in order to check and evaluate the direct consequences of the implementation of the new manure management regarding GHG and ammonia emissions, water quality (nitrate pollution), soil quality (nitrogen, phosphate, potassium and organic matter) and other common and specific indicators.

The assessment will start when the manure is taken from the collection pit of the sewerage farm system before the real end storage tank of the farm. End-products of the project can whether be used in the local area within the system boundaries or exported out of the system assessed as a valuable product

2.2 Monitoring and evaluation of composting manure management strategy

Within the specific action being carried out in the Region of Murcia, the work is designed in the following steps:

- Evaluation of the pig slurry distribution in the Region of Murcia and identification of the surplus areas;
- Identification of appropriate agricultural soils for pig slurry application according to the crop and nutrient balances;
- Establishment of the technical requirements for pig slurry composting: the feasibility of composting as an alternative for recycling pig manure at the farm level in the Region of Murcia will be determined, according to the characteristics of the pig slurry produced, the available solid-liquid separation systems, and the bulking materials available in the surplus areas;
- Identification of the benefits and constraints in manure and slurry composting: economic, environmental and agricultural aspects;
- Feasibility of the combination of anaerobic digestion and composting of the digestate for manure management;
- Environmental and quality indicators of composting technology;
- Implication of composting on the carbon cycle: C-conservation and sequestration.

3 RESULTS AND DISCUSSION

3.1 Development of the common evaluation and monitoring protocol

The components of the CEMP have been defined in the following terms: criteria, indicators, parameters and reference units (Fig. 1):

- Criteria: the categories in which the evaluation and monitoring is carried out. The defined criteria for the CEMP are: Environment, Agronomy, Energy, Economics, Social Aspects, Animal & Human Health and Legislation. The criterion Environment comprises four sub-criteria: Climate Change, Water, Air and Soil Pollution.
- Indicators: Every criterion (or sub-criterion) will have one or several indicators that express a certain effect on it. For example, Global Warming will be used as the indicator of the sub-criterion Climate Change, and it will be expressed by kg CO_2 equivalents.
- Parameters: A set of parameters is needed to evaluate different manure treatment technologies and management systems, as well as the methodology. For the different evaluation media, such as pig slurry (and liquid derivate), solid fraction (and other solid manure), soil, water, gas emissions and individual set of parameters has been proposed.
- Reference units: Not a single reference unit will be used and equivalences between them will be established: Mg of pig manure, Livestock Unit, ha of land surface, kg of meat product.

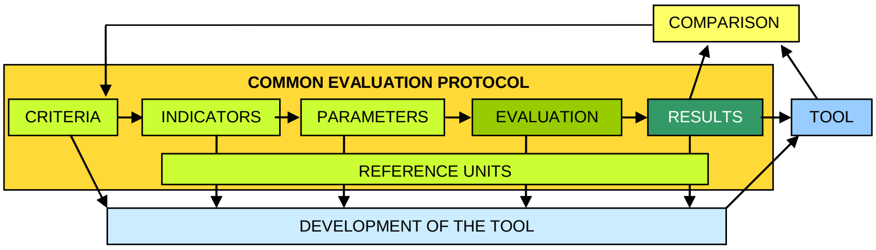

FIGURE 1. **Schematic approach of the Common evaluation and monitoring protocol.**

Environmental criteria: Climate change is indicated by global warming potential, expressed by kg CO_2 equivalent, described by the parameters: CO_2, CH_4, N_2O and NO_x emissions, and CO_2 savings. Acidification is the indicator for air pollution, expressed as kg SO_2 equivalents by the parameters NH_3 and SO_2 gas emissions. Water pollution indicator is eutrophication, being evaluated by the total-N, total-P and NO_3^- parameters, and expressed in kg NO_3^-, kg PO_4^{3-} and O_2 equivalents as the units. Soil pollution is divided in subcriteria: salinity, evaluated by the parameter electrical conductivity in dS/m; metals with the parameters Cu and Zn concentrations in kg; and excess of nutrients with the parameter total-P in mg/kg.

Energy criteron: A balance between energy production and energy consumption is considered as the indicator, with the parameters: Energy spend of the facility; Energy potential -Energy content of biogas production; Electrical potential – Electrical energy produced after transformation; Heat potential - Heat produced after transformation, all expressed in kW.

Economy criterion: Benefit and loss account, depreciation and financing costs are the indicators, with the parameters incomes and expenses, expressed in euros.

Social criterion: Odour, noise, visual impact and impact in local activity are the indicators considered which will be determined by the parameters olfactometry; direct measurements or technical data of equipment and jobs created.

Agronomic criterion: Livestock density and fertilising units will be included as the indicators with the parameters, N, P and K balances.

Animal and human health criterion includes *E. coli* and Salmonella as the indicators and parameters to evaluate.

Legal framework includes the indicators of European regulation, National regulation and Regional/local regulation.

At the moment the integration of all the parameters for the development of the CEMP is under progress.

3.2 Evaluation of the pig slurry distribution in the Region of Murcia

Taking into account the information collected from the regional agricultural database of 2009 (Conserjeria de Agricultura y Agua de la Region de Murcia; CARM), the main characteristics of the farms have been described and located. The vulnerable areas to nitrate pollution from agricultural sources in the Region de Murcia were designated (BORM, 2001; BORM, 2009), which need to be taken into consideration for the development of this project. Then, a map showing the vulnerable zones to nitrate contamination in the area has been made using the data from the "geo-catalogue" from CARM (Fig. 2).

The analysis of the local data shows the limited spatial distribution of the farms in the Region. More than 50% of the farms are located in one area, the "comarca del Guadalentin". This area holds 65.09% of all the pig farms of the Region, the pig slurry production can account for 2,510,221 m^3 per year, which is equivalent to 54 % of the total pig slurry production in Murcia Region. At municipal scale, Lorca has 46 % of the total pig farms in the region; followed by Fuente Álamo (area of Cartagena) with 14.8 % of the total regional amount. Therefore, the slurry production is highly concentrated in some municipalities: Lorca with 1.5 million m^3/year; Fuente Álamo with 1.0 million m^3/year (Fig. 2B).

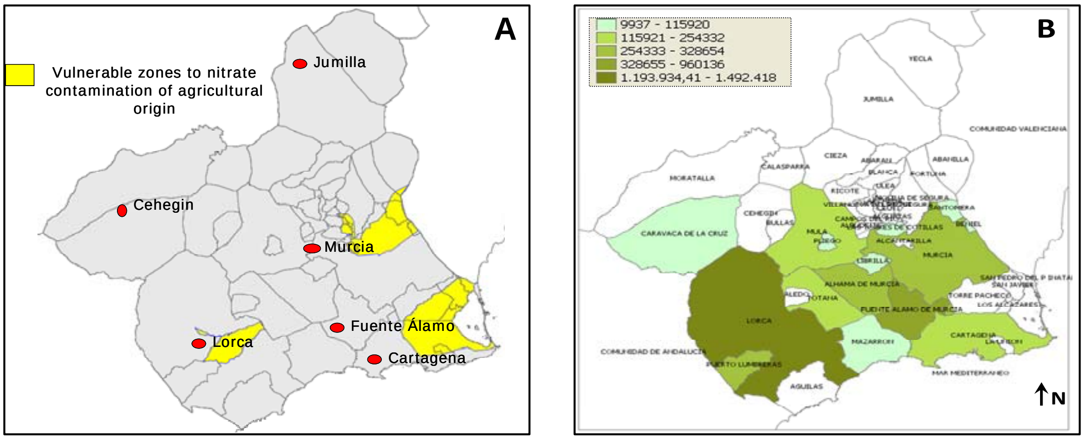

FIGURE 2. **Vulnerable zones to nitrate pollution from agricultural sources in the Region of Murcia (A); and Distribution of pig slurry in the Region of Murcia (m^3/year) (B).**

The average available agricultural land estimated per farm at regional scale, according to the 2009 database is 12.39 ha, which should be consider in the first instance for the agricultural use of the pig slurry. The use of slurry as a fertiliser is highly dependent on the distance between the source of production and the receiving area, as the cost of transport can make this practice unprofitable for both parts in most cases, except for those farms surrounding the pig slurry-production farms. For the time being, both farmers and livestock farmers agree the pig slurry removal at zero cost. The livestock farmers do not have any guarantee for this practice being permanent; sometimes they have to pay the farmers for removing it from the pig farms for soil application. Some farms from Lorca are located within the limits of the vulnerable zone to nitrates contamination (Figure 2), where the maximum input of nitrogen to the soil is 170 kg per ha and year (Council Directive 91/676/CEE; Spanish Real Decreto 261/1996). For an efficient management, the use of slurry as fertiliser should be solved by transporting the slurry from the surplus areas to N-deficient areas. The use of slurry in agricultural soils outside the vulnerable zones may be a solution, while its use in vulnerable areas needs a careful management strategy.

The crops existing in the main area for pig production (Lorca) were identified according to the information collected from the local Government (CARM) and the statistical indicators for the Region of Murcia. According to such data, the amount of N required for the major crops in the municipality of Lorca was calculated, taking into account the maximum N doses recommended in Annex IV of the Code of Good Agricultural Practices in the Region of Murcia and considering the limitation of N application in vulnerable zones. So the total N requiring crops can be estimated to be 4500 Mg/y. If the average concentration of N of the pig slurry is 3.15 kg m^{-3}, as indicated in the Spanish legislation setting the basic rules of management of pig farms, that amount would be equivalent to 1.4 million m^3 per year of pig slurry. Using the pig slurry for fertilising the crops, it could provide more than 96 % of the annual requirements for N, the main part to horticultural crops and cereals. About 3.34 % of the pig slurry (almost 50,000 m^3 per year), generated annually would be in excess to the N requirements for crops, which would require an alternative management.

From these data, several farms located in Lorca, the area with the highest slurry production and inside the vulnerable zone, have been selected for further studies on pig slurry composition and its composting, as feasible way for slurry agricultural management. The composting would allow the safe export of the excess of slurry (and nitrogen) as a regulated fertiliser from the vulnerable zones to areas with nutrients and organic matter requirements.

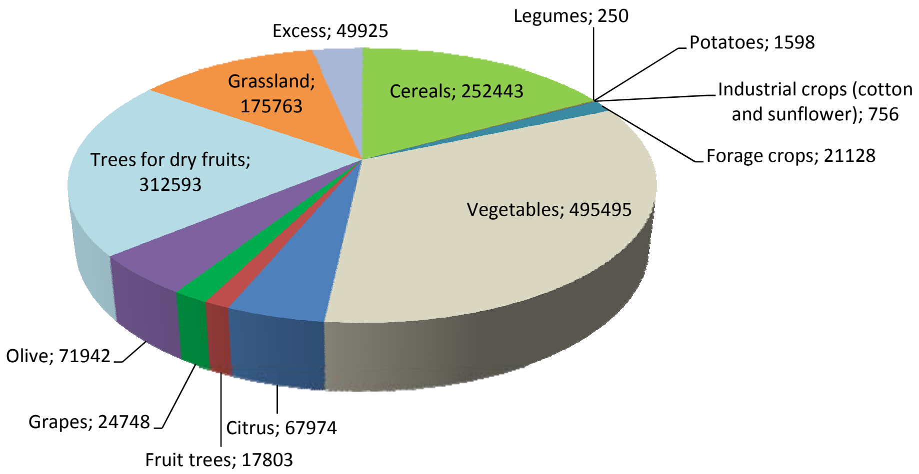

FIGURE 3. **Amount of pig slurry which can be used in the different crops in the municipality of Lorca (m^3).**

3.3 Pig slurry characteristics for composting

The composting strategy at the farm level has been considered the most adequate due to the relevance of the organic matter of the pig slurry for the agricultural soils from the semi-arid climate. The centralised installation is not feasible up to now, since the transport of the slurry in trucks is very expensive and with high environmental risks.

Also, the composting of pig slurry requires a previous solid-liquid separation and the farmers could manage the liquid phase in their agricultural soils. The composting will obtain a stabilised product with high agricultural value in the agriculture of the area, with high demand in organic matter for the soil and also substrates for soilless cultivation . The product can also be transported in a safe way to other agricultural areas.

As previously reported, several farms located in the highest slurry production area inside the vulnerable zone in the Region of Murcia (Spain) were selected for studies on pig slurry composition and its composting, as a feasible way for slurry agricultural management. In order to detect differences in the pig slurry composition according to the type of animal, 18 pig slurry samples were divided into four groups according to the type of farm and slurry origin: Full cycle (slurry from gestating and lactating sows, piglets and fatteners collected together); sows; piglets (from 6 to 20 kg); and fatteners (pigs from 20 kg to slaughter).

The main differences on the pig slurry composition according to the type of animal were found with respect to the electrical conductivity, with the highest values in the slurry from fatteners, which also had the highest concentration of total organic-C (TOC), total-N, P and K, indicating the greatest agricultural value (Table 1). However, the concentrations of heavy metals (Cu and Zn) were the highest in slurry from piglets, due to the use of diet complements rich in those elements for health and welfare reasons, or as growth promoters. Copper is added to pig diets as an anti-bacterial agent in the gut and Zn is also used in pig diets for the control of post-weaning scours (Petersen et al., 2007). The variability of the results in the different animal slurry revealed the influence of the installations including slurry management system in the pig slurry composition (Moral et al., 2005).

In general, pig slurry shows several characteristics that strongly influence the design and development of the composting process such as (Bernal et al., 2009):

- Neutral-alkaline pH values, which do not suppose any problem for the composting process. However, in the wastes with pH values higher than 7.5, N can be lost by NH_3-volatilisation during aeration of the composting mass;
- High electrical conductivity values, which can reduce the quality of the final compost obtained;
- Low C/N ratio, which implies N losses by ammonia volatilisation or by leaching from the composting mass;
- High moisture content, which makes difficult composting without bulking agent;
- Presence of contaminants, such as heavy metals.

Therefore, composting of pig slurry has several constrains which must be overcome for obtaining quality compost (Bernal et al., 2009): Pig slurry should be previously separated into a solid and a liquid phase in order to obtain a solid material; bulking agent is required to adjust the moisture content and C/N ratio for composting. The selection of the bulking agent is also a key factor for controlling and reducing N-losses during composting, mainly associated to ammonia volatilisation. Such losses reduce the agronomical value of the compost having a high environmental impact.

Other additives can be considered for improving the development of the composting process or/and for reducing NH_3-losses. The presence of certain heavy metals (Cu and Zn) in the pig slurry can limit the agronomic quality of compost and therefore its final use.

TABLE 1 **Analysis of the pig slurry according to the type of animal.**

Parameters	Full cycle	Sows	Piglets	Fatteners
pH	7.7 ± 0.44	7.7 ± 0.34	7.2 ± 0.10	7.1 ± 0.41
EC (dS/m)	27.2 ± 6.26	19.3 ± 3.54	21.2 ± 7.45	36.5 ± 7.73
Moisture (%)	96.86 ± 3.51	96.27 ± 4.02	97.55 ± 1.12	97.13 ± 2.04
Dry matter (%)	3.14 ± 3.51	3.73 ± 4.02	2.45 ± 1.12	2.87 ± 2.04
Total-N (g l^{-1})	0.98 ± 1.21	0.83 ± 0.72	0.83 ± 0.23	1.32 ± 1.03
Total organic-C (g l^{-1})	8.60 ± 11.19	7.37 ± 7.22	7.50 ± 4.11	12.69 ± 12.97
Total-P (g l^{-1})	0.38 ± 0.63	0.55 ± 0.59	0.33 ± 0.15	0.71 ± 0.62
K (g l^{-1})	1.68 ± 0.21	1.39 ± 0.32	1.39 ± 0.16	2.60 ± 1.31
Ca (g l^{-1})	1.18 ± 1.72	1.26 ± 1.20	0.66 ± 0.24	1.21 ± 0.95
Na (g l^{-1})	0.95 ± 0.42	0.85 ± 0.31	0.40 ± 0.05	0.84 ± 0.43
S (g l^{-1})	0.29 ± 0.32	0.38 ± 0.09	0.35 ± 0.10	0.43 ± 0.28
Mg (g l^{-1})	0.41 ± 0.39	0.37 ± 0.36	0.21 ± 0.06	0.60 ± 0.41
Cl- (mg l^{-1})	2019 ± 727	480 ± 432	837 ± 156	1076 ± 564
NO_2^--N (mg l^{-1})	< 0.1	1.0 ± 0.3	< 0.1	1.3 ± 2.2
NO_3^- -N (mg l^{-1})	1.9 ± 0.7	1.1 ± 0.1	1.9 ± 1.3	1.2 ± 0.5
PO_4^{2-}-P (mg l^{-1})	24 ± 11	16 ± 2.3	29 ± 9.5	31 ± 21
Cu (mg l^{-1})	9.5 ± 11	6.9 ± 8.5	27 ± 28	8.0 ± 5.0
Fe (mg l^{-1})	142 ± 263	51 ± 55	52 ± 20	57 ± 58
Mn (mg l^{-1})	17 ± 25	14± 14	8.7 ± 3.1	18 ± 16
Pb (mg l^{-1})	1.1 ± 1.10	0.37 ± 0.05	0.58 ± 0.18	1.5 ± 1.46
Zn (mg l^{-1})	108 ± 150	83 ± 0.16	398 ± 207	40 ± 31

4 COMING RESEARCH ACTIONS IN COMPOSTING

A composting pile was established in a pig farm based on a simple turning system using the solid fraction of pig slurry from sows. The farm has a screw press solid-liquid separator, with an aerobic pre-treatment of the slurry and two aerated tanks for aerobic treatment of the liquid fraction, which is later stored for using as irrigation water in the citrus trees of the farm. The solid fraction has 10 % dry matter content, alkaline pH (8.1), the dry matter is rich in organic matter (74 %), TOC (403 g kg^{-1}) and total-N (13.9 g kg^{-1}) with a C/N ratio of 29, adequate for composting. However the excess of moisture make it necessary to add a bulking agent to provide porosity and aeration to the composting mass.

Then a composting pile of about 4.8 m^3 was established with maize straw as the bulking agent, at the proportion 2:1 (in volume) solid fraction of pig slurry to maize straw ratio.

The temperature evolution indicated a slow development of the composting process, requiring 3 weeks to reach thermophilic temperatures. The aerobic treatment of the slurry before separation could have partially degraded the labile organic matter, this together with an excess of moisture in the pile at the beginning of the process can be responsible for the slow increase of the microbial activity. However, after the first turning, the temperature of the pile increased quickly to 55 ºC, remaining stable for more than three weeks. Several samples of material have been taken during the composting process, which are being analysed.

The results indicated that the pig slurry was partially degraded during the aerobic pre-treatment, then, composting requires a co-composting material for microbial inoculation and activation of the microbial activity. Other waste materials produced in the farm to be tested include solid pig manure and addition of fresh pig slurry (without the aerobic pre-treatment) during the composting process.

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(230) ADMINISTRATIVE AND ECONOMIC TOOLS FOR PROMOTING BIODEGRADABLE WASTE DIVERSION FROM LANDFILL

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EXECUTIVE SUMMARY

During the last decade the waste management landscape in European Union (EU) Member States (MS) is being reconstructed to a lesser or greater extent, due to the pressures exercised by the EU policy and legislation. This “reconstruction” is quite radical in MS that did not already have in place complex material and resource recovery systems by the mid 1990s. The European Landfill Directive (LD -1999/31/EC) is placed among the most influential documents, as it sets increasingly demanding diversion targets for the biodegradable fraction (BMW) of municipal solid wastes (MSW) and requires from MS to adopt policies together with administrative and economic tools for the diversion of the biodegradable fraction of municipal waste. MS like Greece and Cyprus that were heavily relying on landfilling, for more than 80% of their MSW, did not share the same starting point with states that have made significant steps towards diversion systems; therefore they had the option to postpone each of the LD diversion targets by a maximum of 4 years. Using this allowance, Greece and Cyprus should meet the respective diversion targets by 2010, 2013 and 2020. Moreover, the Waste Framework Directive (WFD - 2008/98/EC) sets requirements for the promotion of source separation and recycling for biowaste, the “most difficult” fraction of the biodegradable municipal waste (BMW), although these requirements are not defined in quantitative terms.

In 2010 Greece was short of reaching its 1,1 million tn BMW diversion target by about 15%; since then a very modest progress has been made in infrastructure development, with new facilities still being at various stages of planning and/or tendering procedures. It is unlikely that any substantial increase in BMW treatment capacity will be available by the year 2013, when the next diversion target, a foreseen 1,9 million tn, should be achieved, risking a considerable increase of the deviation from the diversion target. Cyprus also did not meet the 2010 diversion target. In order to reverse the existing situation and comply with the LD and the WFD, the entire waste management sector in Greece and Cyprus should be subjected to radical and rigorous changes. Purcell and Magette (2011) indicated that targeted intervention strategies designed for specific geographical areas should lead to improved diversion rates of BMW from landfill.

In the present study the accumulated experiences of different biowaste diversion efforts worldwide are analyzed with ultimate goal to assist decision-makers in Greece and Cyprus to successfully implement the LD and WFD through the adoption of the appropriate administrative and economic tools. Results would also be useful for a number of other MS that still need to make progress towards achieving BMW diversion targets. The research involved: a thorough literature review of the implemented biowaste diversion practices worldwide, with emphasis upon EU MS, and 65 interviews with stakeholders (central and local government waste management officers, academics, policy makers, consultants and NGO’s) in Greece and Cyprus, using structured questionnaires.

The analysis of the collected data delineates the stakeholder’s viewpoint on the biowaste diversion potential of the existing waste management in Greece and Cyprus and the options for improvement. Overall, their view is that the existing legislative framework, based on the transposition of the LD fails to promote biowaste diversion from landfill in both countries. This could be reversed through setting specific targets for biowaste source separation systems, establishing appropriate waste acceptance criteria for landfilling, and fully implementing landfill charges based on the waste quantity disposed by the municipalities (“pay-as-you throw” for the municipality). Hinged upon these tools, an integrated waste management system is expected to be established, addressing the targets set until 2020 in the two countries.

1. INTRODUCTION

During the last decade the waste management landscape in European Union Member States (EU - MS) is being reconstructed to a lesser or greater extent, due to the pressures exercised by the EU policy and legislation. This “reconstruction” is quite radical in MS that did not already have in place complex material and resource recovery systems by the mid 1990s (Lasaridi, 2009). The European Landfill Directive (LD -1999/31/EC) is placed among the most influential documents, as it sets increasingly demanding diversion targets, and requires from MS to adopt policies, administrative and economic tools for the diversion of the biodegradable fraction (BMW) of municipal solid waste (MSW). MS like Greece and Cyprus that relied heavily on landfills in 1995, for more than 80% of their MSW, may postpone each of the targets by a maximum of 4 years. Using this allowance, Greece and Cyprus should meet the respective diversion targets by 2010, 2013 and 2020. Moreover, the Waste Framework Directive (WFD - 2008/98/EC) sets requirements for the promotion of source separation and recycling for biowaste, although these are not defined in quantitative terms.

In 2010 Greece was short of reaching its 1,1 million t BMW diversion target by about 15%; since then a very modest progress has been made in infrastructure development, with new facilities still being at various stages of planning and/or tendering procedures. It is unlikely that any substantial increase in BMW treatment capacity will be available by the year 2013, when the next diversion target, a foreseen 1,9 million t, should be achieved, risking a considerable increase of the deviation from the diversion target. Cyprus also did not meet the 2010 diversion target although considerable treatment capacity has been installed since.

Back in 2003, a study analysing the gained experiences of different waste diversion efforts (Fehr, 2003) showed that a landfill diversion model would not be efficient unless it: achieves the maximum feasible amount of recycled material with minimum feasible source separation effort; avoids thermodynamic and administrative contradictions; makes the best use of existing equipment infrastructure and human resources; actively involves the population with long-term goals; does not target in short-term results; and builds and uses exemplary pilot projects to achieve public participation. According to Lasaridi (2009), Greece failed to fulfill the aforementioned conditions. In order to reverse the existing situation and comply with the LD and the WFD, the entire waste management sector in Greece should be subjected to radical and rigorous changes. Purcell and Magette (2011) indicated that targeted intervention strategies designed for specific geographical areas should lead to improved diversion rates of BMW from landfill.

This is in agreement with the current view of the Commission (2008/98/EC) that there is no single environmentally best option for the landfill diversion and management of BMW. The environmental benefits and costs of the different options available depend on a number of local factors, including collection systems, waste composition, climate, potential compost uses and their contribution to combating soil erosion etc. It is recommended, therefore, that BMW management strategies should be determined by MS using “life-cycle thinking”, although it is still not clear how this principle will be utilised, especially in those MS with less developed waste management systems and traditions.

1.1 Research objectives

The present study details the administrative and economic tools that are employed worldwide (though emphasis is given in EU MS) for the biowaste diversion from landfills (Trois & Simelane, 2010; Reichenbach, 2008; Morrissey & Philips, 2007; Price, 2001), with ultimate goal to showcase examples of good practice as well as to support decision-makers in Greece and Cyprus to successfully adopt the LD and the WFD. In order to adequately comprehend the current situation in the Waste Management in these countries, an elicitation survey (interviews) was carried out among Greek and Cypriot waste management experts and decision-makers.

2. ADMINISTRATIVE AND ECONOMIC TOOLS FOR BMW DIVERSION FROM LANDFILL

The main alternatives to landfill for BMW include: (a) waste reduction, (b) source separation and recycling for paper, (c) source separation and composting / anaerobic digestion (AD) for biowaste and some types of paper, (c) mechanical-biological treatment for commingled MSW, and (d) thermal treatment for commingled MSW or the solid refuse fuel derived from MBT. A detailed description of these options can be found in the relevant technical literature (Bardos 2004; Velis et al. 2009). It is of paramount importance to understand that there is strong interrelation between the technologies selected and the systems of provision and everyday practices. The latter, especially regarding the implementation or not of source separation, determine which technologies may be applicable in each specific case, as some options may be suitable only for source separated BMW. Costs (absolute and relative to landfill), public acceptance and markets for the outputs of the different BMW treatment processes will define the options selected for each region and their potential for successful implementation (Bulkeley and Askins 2009; Lasaridi 2009; Price 2001).

In order to drive effective implementation of BMW diverting some strong policy instruments are required (EEA 2002; 2007; Togia et al. 2006). This is demonstrated by the practices of the MS achieving large diversion rates (Table 1). In most cases, a combination of economic, such as the introduction of a landfill tax, and administrative instruments, such as the ban of BMW from landfill, has been used by those countries who have achieved compliance with the LD targets.

TABLE 1 **Policy instruments for the diversion of waste from landfilling in the different EU member states (adapted from Lasaridi 2009)**

	Market based instruments		**Administrative instruments**		
Country	**Landfill tax**	**Tax, other**	**Landfill ban**	**Packaging collection systems**	**Separate collection other BMW**
Group 1: Countries with high material (>25%) and high incineration (>25%) rates					
Belgium-Flanders	1990		2005	1990-1994	1990s
Belgium-Wallonia	Yes		2004-2010	Yes	2002-2003
Denmark	1987	1978/1998: packaging	1997	Glass:1990	2004
France	1992		2002	1992	1999
Luxembourg					Yes
Netherlands	1996		1996	1997	1994
Sweden	2000		2002	1994	1994
Group 2: Countries with high material (>25%) and low incineration (<25%) rates					
Austria	1989		2004	1990/1992	1995
Finland	1996		2005	1995/1997	1998
Germany			2001*	1991	
Hungary		1995: product fee	2002	2001	2001
Italy	1996		2003/2007	1998	1997
Ireland	2002	2001: plastic bags		1997	
Spain				1997	
United Kingdom	1996	2005: LATS		1997	2003
Group 3: Countries with low material (<25%) and low incineration (<25%) rates					
Cyprus				2005	2000
Czech Republic	1991: fee on landfill		1997: paper 2004:BMW	1999	1999
Estonia	1990: fee on landfill	1997: product fee (packaging)	2008	2004	
Greece				2001	
Latvia		2002: product fee (packaging)		2003/2005	2006-2012
Lithuania		2003: product fee (packaging)			
Malta					
Poland				2002	
Portugal				1998	
Slovakia	1992: fee on landfill	2001: recycling fund		1998	
Slovenia	2001		2001	2001	2001

* Prior to the landfill ban for waste with high organic content, there was a requirement for BMW pre-treatment up to specific quantitative criteria (since 1998).

3. METHODOLOGY

The data used in the present paper were collected as part of a study conducted during the period April 2011 to March 2012, in Greece and Cyprus. The study was conducted in two stages. At first, a state-of-the-art review on the administrative and economic tools for the effectively diversion of biodegradable municipal waste from landfilling, was held. The review contained the identification and evaluation of the aforementioned tools, on the basis of their reported results achieved.

During the second stage, an elicitation survey among "key-persons" in the waste management sector was conducted in Greece and Cyprus to investigate their insights towards the effective implementation of LD and WFD, as well as to the drawbacks of the current situation and their causing routes. Totally, 65 interviews (47 in Greece and 18 in Cyprus) with stakeholders in waste management sector (central and local government waste management officers, academics, policy makers, consultants and NGO's) were obtained. The interviewees were called upon to answer the same set of questions, irrespectively of their background and job title.

3.1 The interview design

The interview was based on a structured questionnaire, which comprised of 15 closed or open text questions. The questionnaire design was based on the relevant waste management literature as well as a questionnaire designed for a similar EU consultation study (Anon, 2010). In order to shed light on stakeholder's viewpoint on the BMW diversion potential of the existing waste management systems in Greece and Cyprus, the questions were categorised into the following sections:

- Knowledge and comprehension of the current waste management system in the study areas;
- Opinion on the implementation of administrative tools for the diversion of BMW from landfilling;
- Opinion on the implementation of economic tools for the diversion of BMW from landfilling; and
- Suggestions about the administrative and/or economic tools.

4. RESULTS AND DISCUSSION

During the period April 2011 to March 2012, 65 structured interviews using a purpose designed questionnaire were obtained by the research group from stakeholders in the waste management field from Greece and Cyprus. All interviewees were actively involved in the field of waste management, at different sectors that all influence decision making in their respective country; central and local government waste management officers: 55%, consultants: 34%, academia: 8% and NGO's specialising in waste management: 3%. There was a bias in the sample towards sexes (males: 60%, females: 40%), which reflects the under-representation of women in the waste management sector. Based on professional organisations membership and participation to national thematic conferences and exhibitions, the overall pool of relevant stakeholders in the two countries is estimated to be around 1,000 persons (800 in Greece, 200 in Cyprus).

By the design of the study, all the respondents had a good knowledge of the national legislation framework and were well informed about the waste management practices implemented. Overall, their viewpoint was that regarding the implementation of the Landfill Directive, the existing legislative and organizational framework fails to achieve the BMW landfill diversion targets. The majority of the respondents (81% in Greece and 61% in Cyprus), stated that *"the existing waste management system was not effective for the diversion of biodegradable waste from landfilling"* (Figure 1).

A closer look at the composition of these two "denial" group, is quite illuminating: 50% of them in Greece and 82% in Cyprus were employed in the central and local government waste management sectors, meaning that – at least at the time of the survey - they were playing a role in the implementation of the central or local waste management system and/or decision-making and had a clear picture of the existing situation. Interestingly, almost all the Greek respondents who affirmed the effectiveness of their national legislative framework commented that the implementation of it was not sufficient, mainly, due to inadequate planning, political cost considerations and public opinion opposition. On the other hand, the Cypriot respondents stated that failure of implementation was mainly due to public opinion opposition. The climate of mistrust to the implementation of the waste management policies and legislation is strongly enhanced by the answers to another relevant question. When asked *"Do you believe that the compulsory pre-treatment of the biodegradable waste stream is effectively implemented in your country?"* the overwhelming majority (all, but two Cypriot responders) stated categorically *"No"*.

When asked whether *"the Greek/Cypriot legislation, driven by the WDF, prioritizes recycling or incineration"*, 81% of the Greeks and 72% of the Cypriots responded *"recycling"* of biodegradable waste (e.g. by composting). Nine (9%) and six (6%) percent of the Greek and Cypriot respondents, respectively, chose *"incineration"* (Figure 2).

The next group of questions aimed to investigate the viewpoint of stakeholders on the implementation of administrative and economic tools for the diversion of the biodegradable waste stream. More specifically, it investigates how the respondents would implement certain tools that are widely used in other MS (Table 1). Regarding the implementation of *compulsory biowaste source separation targets*, over 70% of the Greek stakeholders and all Cypriots shared the view that at least 10% of biowaste should be source separated – the target being compulsory by law.

About 53% of the Greek stakeholders responded that the diversion rate target should be set at 20% or 30%, 13% answered < 10%, while 16% did not answer this question, stating that this tool could never have been implemented under the given circumstances of the waste management system. In Cyprus, the responses were more positive to this administrative tool; 61% would opt for a compulsory biowaste source separation target of at least 30% (Figure 3).
The introduction of a landfill tax is considered to be an effective economic tool to bridge the cost difference between the lost cost option of landfill and the higher cost alternative treatment methods (Bulkeley and Askins 2009; EEA 2007; Lasaridi 2009). Greek and Cypriot stakeholders seem to share this belief; 68% of the Greeks and 61% of the Cypriots stated that a landfill tax would be a useful economic tool to the municipal solid waste management system (Figure 4). When asked *"whether the landfill tax could be implemented during the economic recession"*, the majority (84% and 82%, in Greece and Cyprus, respectively) stated *"yes"*. However, 56% of the Greeks and 55% of the Cypriots believe that the imposed landfill tax should be less than 10€ per tonne for non-toxic waste.

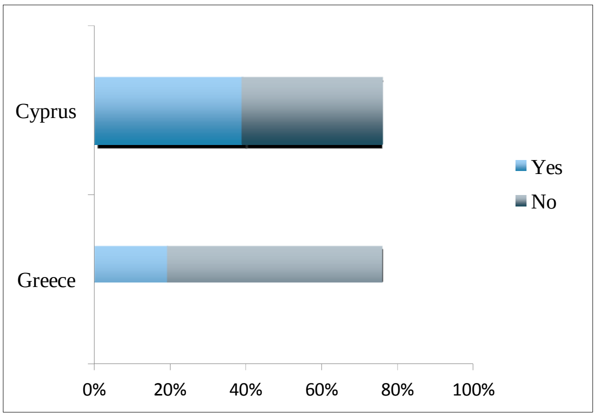

FIGURE 1 **"Do you consider the existing legislative framework in your country (Greece/ Cyprus) effective for the diversion of the biodegradable waste from landfilling?"**

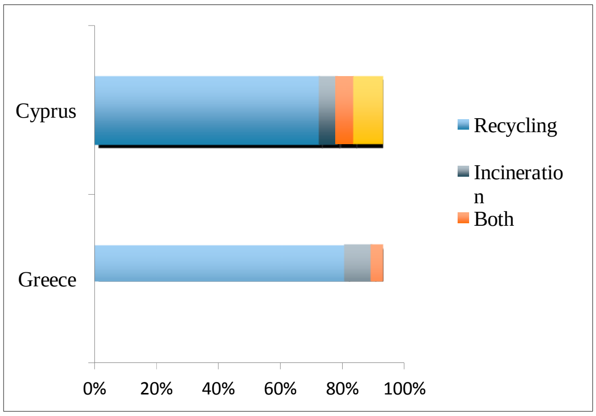

FIGURE 2 **"Do you think that the existing legislative framework, based on the WFD, promotes: recycling, incineration or both of them?" (N.A.: Not Answered)**

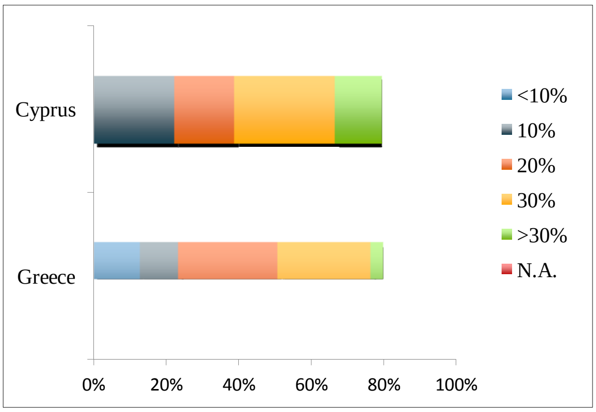

FIGURE 3 **"If the national legislation adopted compulsory source separation of biowaste, which rate would you prefer? <10%, 10%, 20%, 30%, >30% (N.A.: Not Answered)**

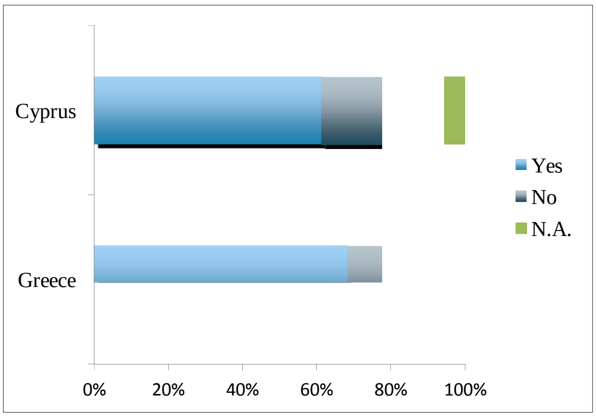

FIGURE 4 **"Would you consider the imposition of a landfill tax to be a useful economic tool for MSW management in your country? (N.A.: Not Answered)**

The majority of the respondents (68% of the Greek and 72% of the Cypriot) believe that an extra landfill ban would have a positive impact on the improvement of the biodegradable diversion rate, which is in accordance with the findings of Anon (2010) (Figure 5). Almost all of those that opposed such a landfill ban justified their choice stating that since the existing landfill diversion target was not implemented properly or the benefits of it were negligible, such a strategy would ultimately have a negative impact on public opinion and would - in long term - favor the thermal treatment of municipal solid waste.

Although Greek and Cypriot stakeholders agreed in the previous question, they were diverged when asked at what level – EU or national – such a BMW landfill ban should be taken. Fifty three percent of the Greek respondents replied "on national level", while 67% of the Cypriots "on EU level" (Figure 6).

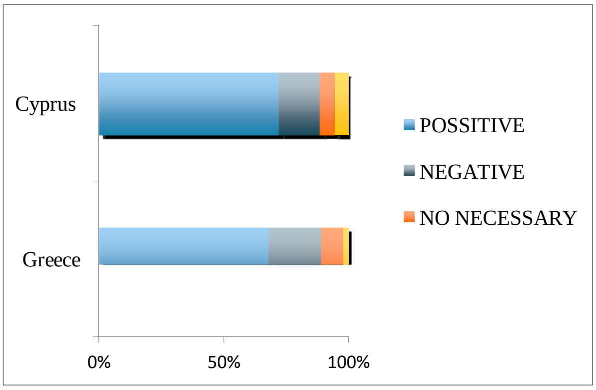

FIGURE 5 **"Do you believe that the impact of a specific BMW landfill ban, beyond the diversion targets of the LD, would be: i. positive, ii. negative, iii. unnecessary, iv. I do not answer / I do not know"**

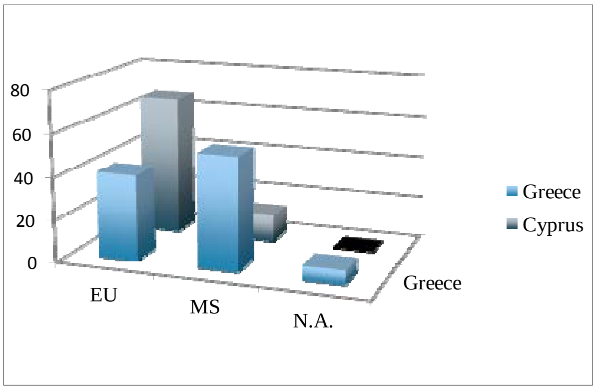

FIGURE 6 **"Do you believe that extra specific BMW landfill ban measures should be taken at European Union (EU) or national (MS) level?"**

Based on the literature review, the economic tools that are commonly employed for the landfill diversion of BMW are: the imposition of landfill tax (in addition to gate fees), quantity based gate fees, "Pay-as-you throw" systems, and Waste Management Authorities landfill allowance trade schemes. The analysis of the data in the present study (Table 2) showed that Greek stakeholders favor the second of the aforementioned tools (34%) as well as "Pay-as-you throw" systems (34%), while Cypriots prefer "Pay-as-you throw" systems (55.6%). The high preference for the "quantity based gate fees" in Greece is explained by the prevailing charging system, according to which Local Authorities are charged for the use of the landfill according to a fixed rate, calculated on the basis of their state financial support and not related to the amount of waste they dispose to the landfill. Obviously, this suppresses any economic motives for diverting waste from landfill – either dry recyclables or biowaste – and is contradictory to the basic "polluter pays" policy principle. A recent law (3854/2010) included measures to end this distortion from 1/1/2011, but due to inadequacies and opposition to the measure from some large Waste Management Authorities, full implementation has not been achieved yet. In Cyprus, landfill charges are based on the waste quantity disposed. "Pay-as-you-throw" systems at the household level exert a strong attraction to stakeholders in both countries. However, tools for their implementation are not readily available, especially in Greece, due to the type of temporary storage and collection system adopted (central, open access bins) and fears for fly tipping, as supervision and control measures are weak.

A common belief in both Greece and Cyprus, shared by about 80% of the respondents, is that *"energy recovery through thermal treatment of biodegradable waste should be part of in an integrated waste management system"*. It is worth to mention that in Greece 90% of the consultants and 62% of the central/local government waste management officers responded positively to this question (data not shown).

Moreover, both Greek and Cypriot respondents shared the same view about the three most suitable administrative and economic tools that should be implemented in order to achieve the BMW diversion targets: Source separation; Pay-as-you-throw systems; Landfill acceptance criteria. Landfill tax and high gate fees, came 4th and 6th among Greeks and Cypriots, respectively. The other options provided were: Compulsory waste pre-treatment; Landfill ban; Quality specifications for waste derived "products"; Local Authorities landfill allowance trade schemes; and Green procurement.

TABLE 2 **"In your opinion which of the following economic tools would be implemented successfully in your country?" (only the percentage of those that have declare each option as first choice is noted)**

	Greece **%**	**Cyprus** **%**
Landfill tax	12.8	11.1
Quantity based gate fees	34.0	16.7
"Pay-as-you-throw" systems on households	34.0	55.6
Landfill allowance trade schemes	4.3	5.6
Other	0.0	5.6
N.A. / did not prioritize	14.9	5.6

5. CONCLUSIONS

Both Greece and Cyprus did not fully meet the 2010 BMW diversion target, set by the Landfill Directive, while it is unlikely that the more demanding 2013 diversion will be met, as progress towards developing the necessary waste treatment infrastructure has been slow. Structured interviews with decision makers in the two countries highlighted their view that the implementation of the national legislative framework was ineffective mainly due to inadequate planning, political cost considerations in Greece and public opinion opposition in both countries. Opinion makers in both countries felt that the new WFD clearly prioritizes recycling of biowaste, but national implementation tools are weak. Among the proposed tools, compulsory biowaste source separation targets were favored by the majority (>70%) of the respondents, with Cypriots opting for higher targets.

The utility of ladfill tax as an efficient economic tool was recognized by over 60% of the respondents, who did not consider the financial crisis to be a sufficient reason to deter from its use. However, they opt for a modest level of up to 10€ per tonne of MSW. However, when asked to rate the three most suitable tools, opinion makers form both countries agree on the following ranking: Source separation; Pay-as-you-throw systems; and Landfill acceptance criteria, leaving landfill tax in the 4th and 6th position in Greece and Cyprus, respectively.

6. ACKNOWLEDGEMENTS

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Session 11

(27) PREDICTING URBAN SOIL SUSTAINABILITY: IMPACT OG ORGANIC MATTER ON SOIL WATER TRANSFER

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EXECUTIVE SUMMARY

The physical properties of urban soils often limit tree development. Urban soils are compacted; as a result, water and air infiltration are reduced. The available soil volume for root colonisation is usually limited by the underground infrastructure. Exogenous organic matter has been shown to have a positive impact on the physical properties of agricultural soils. In some urban situations, the wide-scale availability of organic matter and the need for important soil restructuring have led to the application of large quantities of organic matter. We studied soils reconstituted with high levels of organic matter, with the following aims: (1) measuring the effects of organic matter decomposition on the evolution of soil physical properties, and (2) assessing the sustainability of the soil system by modelling water balance on a long-term period (i.e 20 years after the treatment was applied).

Throughout a 5-year long experimental study (i.e: from 2005 to 2010), we monitored the physical properties - dry bulk density, soil water retention and permeability - of urban soils placed in 600-L containers. In addition, the soil volumetric water content was continuously measured with TDR probes. The top layer was a sandy loam amended with 40% v/v of organic compost – sewage sludge wood chip compost (SW) or green waste compost (GW) – lying on a layer of sandy loam. The control container was composed of two layers of sandy loam. Organic matter had significant effects on the soil physical properties compared to the control, with a bulk density in amended soils. Organic matter modified aggregate organisation and pore distribution, which contributed to a better infiltration rate. Within five years, bulk density in the top layer increased from 0.88 to 1.51 g cm^{-3} in SW, from 0.89 to 1.57 in GW whereas it increased from 1.25 to 1.55 to g cm^{-3} in the control soil. In the same top layer, measured hydraulic properties, yielded a significant negative correlation between the hydraulic conductivity at saturation (Ks) and time. In the second soil layer, no significant change in the soil hydrodynamic properties was observed.

This 5-year long experiment made it possible to model the soil volumetric water content satisfactorily over time. The HYDRUS model was used in a first step for calibration, using data from 2005. Thereafter, it was validated over a whole year, using data from the 2008-2009 period. These good simulations were confirmed by low root mean square errors, whatever the treatment. Using the correlation equations described above, it was possible to extrapolate and estimate the soils' hydraulic properties 20 years later. Then, we simulated the water-filled pore space (WFPS) in the three treatments, with the 2008-2009 climate database (reference). The control and SW treatments displayed very low differences between the reference and 20 years later. The control treatment presented higher WFPS values, with an important risk of transient anoxia. Sewage sludge can therefore maintain soil hydraulic properties over long periods of time. The GW treatment displayed the highest changes in WFPS – an average 10% during dry periods – induced by the decrease in total porosity from 47 % to 42% and in Ks from 3.4 10^{-6} to 0.92 10^{-6} m s^{-1}. Thus, the stability of soil mixed with green waste can be questioned.

1 INTRODUCTION

The physical properties of urban soils often limit tree development (Jim, 1998; Rossignol, 2001). Urban soils are quickly compacted by mechanical stresses, which result in reduced water and air infiltration. The available soil volume for root colonisation is usually limited in part by the underground infrastructure (Lindsey and Bassuk, 1991). The supply of high levels of organic matter can considerably improve the physical properties of urban soils (Tejada and Gonzalez, 2003; Grosbellet, 2011) and tree root development (Watson, 2002). However, the sustainability of such amended soils is questioned. Indeed, high amounts of organic matter are mixed into the soil only once, before planting. Then, what of the soil hydraulic properties after several years?

To answer that question, a five-year long experiment was carried out to model the hydraulic properties of soils amended with organic matter, without planting. The objectives of the present study were: (1) to model and to extrapolate the hydraulic properties of soils amended with green waste compost (GW) or sewage sludge woodchip compost (SW), and (2) to simulate such properties 20 years after organic matter was applied.

2 METHODOLOGY

The study site was located in north-western France, in Angers (47°28' N, 00°36' W). In October 2004, twenty-five 600-L containers were filled with two layers of soil and placed under natural conditions. The underlying layer (25- to 53-cm deep) consisted of sandy loam, classified as Fluvic Cambisol (WRB, 2006). The topsoil layer (0-25 cm) was enriched with organic matter (40% v/v). Two organic products were tested (**Table 1**): sewage sludge woodchip compost (SW) and green waste compost (GW). The control container was composed of two layers of sandy loam. Holes were drilled in the bottom of the containers and a 5-cm gravel layer covered with geotextile ensured drainage.

Soil volumetric water content was measured using TDR probes placed 7-, 17-, 27- and 37-cm deep (4 replicates *per* depth). It was monitored in 2005, and then in 2008 and 2009. For each sampling date, (5, 12, 24, 48 and 60 months after the experiment started), soil physical properties were measured – dry bulk density, soil permeability – in triplicate for each treatment. Saturated hydraulic conductivity (Ks) was measured in the two soil layers of the containers with a minidisc infiltrometer (Decagon Devices, Inc; Pullman, WA, USA). In addition, soil water retention curves and soil bulk density (ρ_d) were also assessed – in triplicate. Water retention curves were modelled using Van Genuchten equation (1980).

Volumetric water contents were modelled using the HYDRUS 1D model (Simunek *et al.*, 2005). In a first step, the model was calibrated for each treatment, using the volumetric water content data obtained 7-, 17-, 27- and 37-cm deep in 2005. Then, the model was validated based on the same measurements over a whole year, from June 27th, 2008 to June 26th, 2009. Meteorological data were obtained from the Beaucouzé weather station of Météo France, located less than 1 km from the experimental site. Soil evaporation was not measured, but was estimated from potential evapotranspiration (PET) data. Following Miralles et al. (2011) study, we supposed that evaporation represented 7% of PET. After that, we were able to simulate water content values 20 years later, i.e. by 2024. To that purpose, correlation equations were derived from our 5 measurement campaigns by linking (1) soil bulk density to time, and (2) hydraulic conductivity at saturation to time. As the HYDRUS model required the modelling parameters of the soil water retention curves, we had to estimate them from correlation equations. The water content value at saturation was obtained by calculating the total porosity (P_t) of the soils in 20 years' time based on the bulk density value (ρ_d) expected in 20 years' time.

$$\mathrm{Pt} = \frac{\rho_s - \rho_d}{\rho_s} \qquad [1]$$

where P_t is total porosity ($m^3\ m^{-3}$), ρ_s is soil particle density ($g\ cm^{-3}$) and ρ_d is bulk density ($g\ cm^{-3}$).

Thus, it was possible to re-estimate and fit the parameters of Van Genuchten equation according to the estimated water content value at saturation. In order to compare treatments between them, the water-filled pore space (WFPS) was calculated using the following equation:

$$WFPS(\%) = \frac{\theta}{1 - Pt} \times 100 \qquad [2]$$

where θ is the soil volumetric water content ($m^3\ m^{-3}$).

3 RESULTS AND DISCUSSION

The evolution of bulk density in the 0- to 28-cm layer throughout the first 5 years after organic matter was incorporated displayed contrasting results depending on amendment nature (**Table 2a**). It increased by 23% in the control, whereas it increased by 47 and 26% in GW and SW, respectively. A positive correlation with time was observed, and was significant whatever the treatment (**Table 2a**). After 5 years, although amended soil densities remained lower than in the control soil, bulk density values in green waste compost soil were not as low as in pallet chip compost soil. In the 29- to 57-cm deep, amendment-free layer, densities remained comparable between treatments and increased by 27% within the 5 years. Then they reached values beyond the root growth threshold, which is 1.6 g cm^{-3}. Hydraulic conductivity at saturation (Ks) tended to decrease with time in the 0- to 28-cm layer (**Table 2b**). The input of organic matter at the beginning of the experiment caused that value to drop, as compared to the control, 40-fold and 18-fold for GW and SW, respectively. After 5 years, GW and SW Ks values were relatively close to control values, which had not changed significantly. It was possible to derive correlation equations only from amended treatment values. In the 28- to 57-cm layer, the mean Ks value was 0.32 10^{-6} m s^{-1} and did not vary over time.

The parameters of the retention curves according to Van Genuchten model are presented in **Table 3**. Telling from root mean square error (RMSE) values, the modelling of experimental points for all treatments and in the 2 soil layers for the year 2009 was relevant. In the first layer throughout that same year, water content values at saturation (i.e. total porosity values) were highest in SW, followed by GW, and then by the control. These figures result from the effect of organic matter, which highly contributes to increasing soil water retention, but also from the nature of the organic amendment, which keeps its potential for water retention to different extents depending on the way it evolves over time. In the 28- to 57-cm layer, although there was no organic matter added, the same ranking was observed concerning water retention contents at saturation. However, differences were much lower. Based on the correlation curves described above, new retention curves were estimated between now and in more than 20 years' time. In the 0- to 28-cm layer, water retention values at saturation in GW and in the control were very close, whereas they slightly decreased in SW. Hydraulic conductivity also decreased for all treatments, and reached close values in the control and in GW. SW kept a Ks value 1.7-fold higher than GW.

Thanks to these hydro-dynamic characteristics taken together, obtaining a satisfactory simulation of soil volumetric water contents on the full height of the containers, i.e. in the 2 soil layers, was possible. The validation of our model is presented in **Figure 1**, for the SW treatment and at 7- and 37-cm depths only. The simulation period spanned a whole year, from June 27th, 2008 to June 26th, 2009. Throughout that period, total rainfall was 621 mm and PET was 804 mm, therefore 57 mm estimated evaporation. Whatever the depth, the model succeeded in reproducing experimental data. For SW, RMSE values were 11, 13, 16 and 13% at 7-, 17-, 27- and 37-cm depths, respectively. The values were slightly higher for GW, i.e. 20, 17, 23 and 22% at 7-, 17-, 27- and 37-cm depths, respectively. Water content peaks resulting from rainfalls excluded, we can note that the baseline remains relatively constant over time. The absence of plants mainly accounts for that observation. The evolution of water contents in 20 years' time was expressed as a function of soil water-filled pore space (WFPS) in order to make comparisons between treatments easier. Moreover, estimated WFPS values are compared to the reference scenario issued from the validation of our model in the 2008-2009 period (**Figure 2**). Whatever the treatment, a decrease in WFPS values "20 years +" later is observed, especially during soil drying-out periods. This can be explained by the decrease in water retention capacity which goes on 20 years later. Later, comparing treatments shows that control and SW display similar curves in the reference scenario compared to the "20 years +" scenario. However, it is important to note that WFPS values are generally higher in the control, especially during rainy episodes, within a 3- to 8% range depending on rainfalls. This means that in the absence of organic matter input, soils may become water saturated and therefore run a higher risk of anoxia. The most important differences were observed with GW, in which case the sharp decrease in water contents at saturation in the "20 years +" scenario (**Table 3**) resulted in the highest WFPS curve gap with the reference scenario. Moreover, the minimum values for WFPS curves were around 2% lower than with SW.

Our simulation shows that although organic matter input makes it possible to maintain good soil physical quality in the medium term, longer-term evolution can prove less beneficial. Conditions would be ideal if soil water retention could remain stable without favouring risks of anoxia. The WFPS criterion is frequently used in soil microbiology, as some of its threshold values are favourable to such or other micro-organism families.

It is commonly accepted that a WFPS value of 60% is optimal for the functioning of heterotrophic micro-organisms, whereas a WFPS value in the 85-90% range is favourable to anaerobic micro-organisms (Linn et Doran, 1984). All our values are above 70%, whatever the scenario and the treatment. This can be accounted for by the low Ks value ($3.2\ 10^{-7}$ m s^{-1}) and the high bulk density (1.74 g cm^{-3}) of the 28- to 57-cm layer. Yet, WFPS values around 70-75% remain acceptable for ensuring aerobic conditions to the soil. In the year of the “20 years +” simulation, the numbers of days when WFPS values were between 70 and 75% were 113, 81 and 15 for GW, SW and the control, respectively. In the same manner, the numbers of days when WFPS values were above 85% were 74, 20 and 14 for the control, SW and GW, respectively. Our results were obtained from bare soils; the presence of plants such as trees is bound to strongly contribute to decreasing these values, during evapotranspiration phases only.

4 CONCLUSIONS

Based on the acquisition of soil hydro-dynamic measurements after organic matter input over a 5-year period, we simulated the evolution of the same parameters over a 20-year period. The main conclusions of our work are the following:

- The use of a water balance simulation model made it possible to get a good representation of the evolution of volumetric water contents in the soils,
- The input of high quantities of organic matter has a significant effect on soil physical properties and is linked to compost nature and maturity. Palette chip compost displays better stability over time than green waste compost, as regards its better water retention capacity.

In the long term, palette chip compost displays the best hydro-dynamic characteristics, even if the deep layer does not allow a good outlet of drained water and can thereby cause transient anoxia periods.

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Table 1: Main characteristics of the sandy loam and the three organic products.

Particle size	> 10 mm	5-10 mm	2-5 mm	0.2-2 mm	0.05-0.2 mm	0.002-0.05 mm	< 0.002 mm
				/ g kg^{-1} dry mass			
Soil	-	12	10	262	323	280	113
SW	114	82	258	312	74	160	-
GW	173	65	163	74	128	173	-

	Particle density	Bulk density	Water pH	Conductivity /µS cm^{-1}	Total C	Total N	Total C-CO$_3$
	/ g cm^{-3}					/g kg^{-1} dry mass	
Soil	2.58	1.25	6.3	123.2	7.4	1.5	<0.1
SW	1.8	0.31	6.15	2320	253.2	25.2	<0.1
GW	1.76	0.29	9.00	816	260.1	15.9	0.65

Table 2a: Correlation equations between bulk soil density (ρ_d, g cm^{-3}) and time (T, years). Numbers between brackets represent standard deviations.

	Control	**GW**	**SW**
0- to 28-cm layer			
ρ_d after 5 months	1.25 (0.04)	0.89 (0.03)	0.88 (0.01)
ρ_d after 12 months	1.43 (0.07)	1.11 (0.07)	1.07 (0.04)
ρ_d after 24 months	1.48 (0.06)	1.21 (0.05)	1.04 (0.02)
ρ_d after 60 months	1.54 (0.02)	1.31 (0.01)	1.11 (0.02)
equation	$0.08\times\ln(T\times12)+1.21$	$0.15\times\ln(T\times12)+0.73$	$0.03\times\ln(T\times12)+0.99$
R^2	0.9884, P<0.001	0.9761, P<0.001	0.653, P<0.01

28- to 57-cm layer	mean values for all the treatments
ρ_d after 5 months	1.28 (0.03)
ρ_d after 12 months	1.50 (0.07)
ρ_d after 24 months	1.56 (0.05)
ρ_d after 60 months	1.63 (0.04)
equation	$0.06\times\ln(T\times12)+1.41$
R^2	0.78, P<0.01

Table 2b: Correlation equations between hydraulic conductivity at saturation (Ks, 10^{-6} m s^{-1}) and ρ_d in the 0- to 28-cm layer. Numbers between brackets represent standard deviations.

	Control	**GW**	**SW**
0- to 28-cm layer			
Ks after 5 months	5.0 (0.3)	200 (57)	90 (4.7)
Ks after 12 months	1.7 (0.04)	19 (2.5)	19 (1.0)
Ks after 24 months	2.8 (0.7)	2.9 (1.1)	6.3 (1.6)
Ks after 60 months	4.1 (0.3)	3.4 (0.3)	3.7 (0.2)
equation	--	$10^{-4}\times Db^{-9.72}$	$10^{-5}\times Db^{-14.68}$
R2	--	0.73, P<0.01	0.7, P<0.01

Table 3: Hydrodynamic properties of the control soil, the green waste compost (GW) and the sewage sluge woodchip compost (SW) treatments in 2009 and 20 years later (+ 20 y). Soil volumetric water contents (*θs*) correspond to the calculated total porosity. Soil residual water content (*θr*) parameters *α* and *n* are fitted values obtained from Van Genuchten model. Model performance was assessed by calculating the root mean squared error (RMSE). Ks is the soil hydraulic conductivity at saturation

	Control		**GW**		**SW**	
	2009	+20 y	2009	+20 y	2009	+20 y
0- to 28-cm layer						
θs (m^3 m^{-3})	0.41	0.35	0.47	0.38	0.55	0.54
θr (m^3 m^{-3})	0.18	0.1	0.14	0.1	0.2	0.1
α (m^{-1})	20.75	22.3	45.04	3.36	37.66	5.69
n	1.38	1.3	1.25	1.35	1.38	1.21
RMSE (m^3 m^{-3})	0.03	0.04	0.04	0.06	0.04	0.05
Bulk density (g cm^{-3})	1.55	1.67	1.33	1.53	1.10	1.14
Particle density (g cm^{-3})	2.58	2.58	2.46	2.46	2.46	2.46
Ks (10^{-6} m s^{-1})	4.1	0.8	3.4	0.92	3.7	1.54
28- to 57-cm layer						
θs (m^3 m^{-3})	0.39	0.31	0.42	0.31	0.45	0.31
θr (m^3 m^{-3})	0	0.1	0	0.1	0	0.1
α (m^{-1})	12.28	10.34	20.90	1.56	21.01	1.37
n	1.1	1.23	1.11	1.24	1.12	1.27
RMSE (m^3 m^{-3})	0.02	0.04	0.03	0.05	0.02	0.03
Bulk density (g cm^{-3})	1.65	1.74	1.57	1.74	1.51	1.74
Particle density (g cm^{-3})	2.58	2.58	2.46	2.46	2.46	2.46
Ks (10^{-6} m s^{-1})	1	0.32	4.5	0.32	4.6	0.32

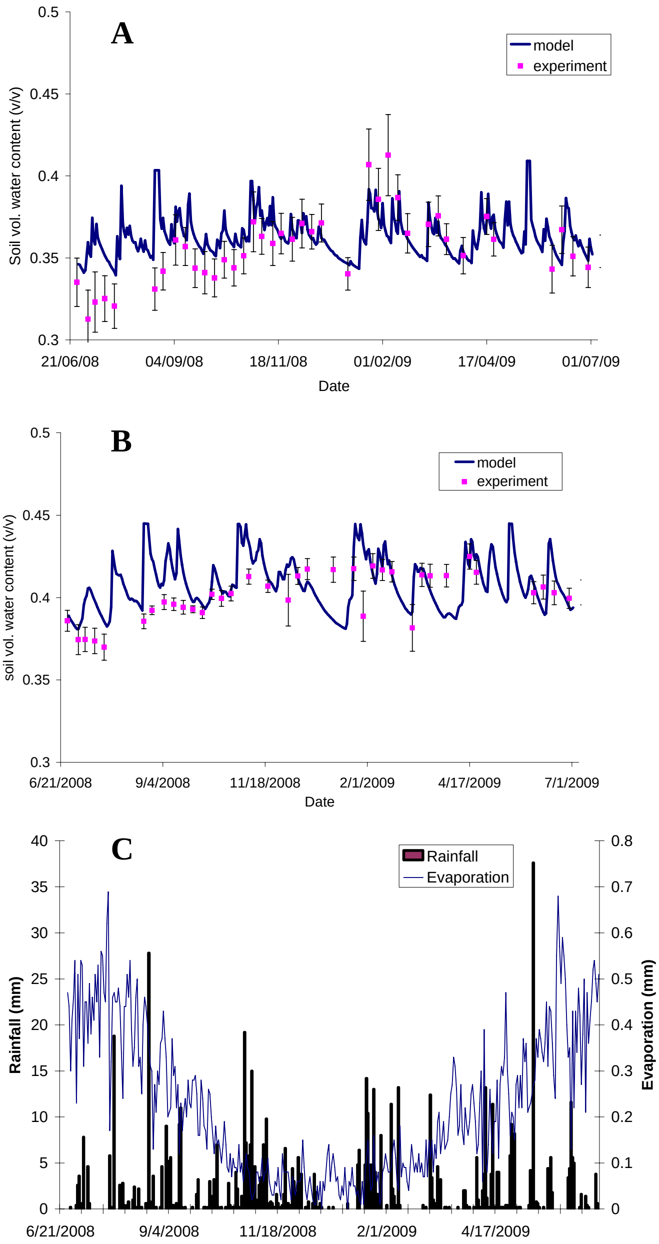

Figure 1: model validation for the simulation of soil water contents in SW treatment 7-cm (A) and 37-cm (B) deep, during the 2008-09 climatic period (C). Bars represent standard errors.

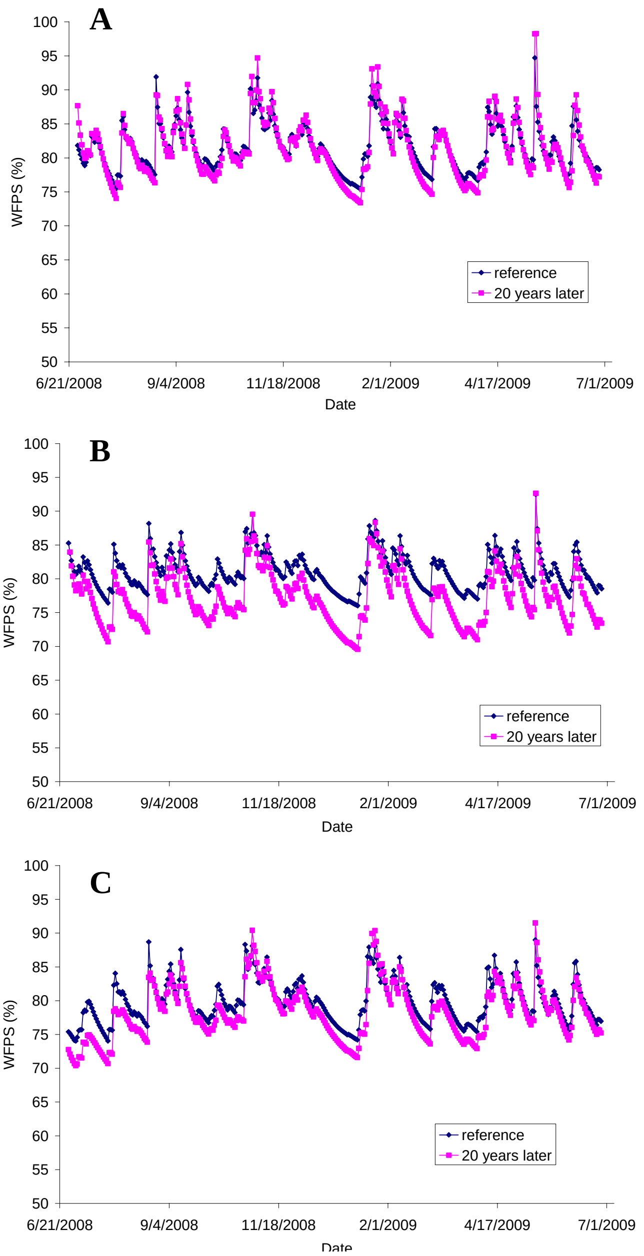

Figure 2: water-filled pore space (WFPS) scenarios in the Control (A), GW (B) and SW (C) treatments in the present context and in 20 years' time, using the same 2008-09 climatic data

(92) MALAGASY URBAN AND LIVESTOCK WASTE: CHARACTERIZATION AND MODELLING OF THEIR TRANSFORMATION IN SOIL

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EXECUTIVE SUMMARY

The need to effectively deal with disposal of human and animal residues continues to be a challenge as population increases. Known as Exogenous Organic Matter (EOM), these residues represent unused resources that can be transformed into an opportunity to create a new supply, turning an issue into a solution. In agriculture, the practice of using EOM as a crop fertilizer can be ecologically sound, both solving a waste management problem and reducing the cost of chemical fertilizer. The management of EOM and valorization is based on the understanding of EOM composition by its categories, biochemical and physicochemical characteristics. Simultaneously, this work aimed to classify the Malagasy EOM into different categories and to predict in controlled conditions their transformation in soil in order to define a tool improving their management through agricultural recycling.

Twenty (20) organic materials originating from Madagascar agricultural, urban and industrial activities were studied in terms of chemical and biochemical contents and for their C and N mineralization during incubation in a typical Ferralsol of Malagasy upland known as "Tanety" according to AFNOR XPU 144-62 (2009) and XPU 144-63 (2009). The IROC, index of potential residual organic C after EOM application to soil, developed by Lashermes et al. (2009) was used to evaluate the proportion of EOM remaining in soils over the long-term after application. The typology of EOM based on biochemical data and N-mineralization (Lashermes *et al.* 2010) allowed a classification of Malagasy EOM. The TAO (Transformation of Added Organic materials) model calibrated on EOMs and substrates of temperate areas (Thuriès *et al.* 2001) was used to assess the transformations in soil of carbon and nitrogen forms of EOMs. Predictions of the transformations of C (very labile, resistant and stable organic C) and N forms (very labile, resistant and stable organic N, produced and immobilized inorganic N) can be made with TAO, driven by EOM biochemical data.

The large range of initial C and N contents and biochemical characteristics induced a wide panel of IROC values. When compared with Malagasy EOM data of residual C measured in French metropolitan experiments, most of animal waste match with the characteristics of the main types of EOM applied in soil. It is not the case for the urban waste, which not meet their supposed category. According to the typology based on N-mineralization the majority of our EOMs presents a low N mineralization and may involve N immobilization in soil. The N-mineralization diagram confirms this categorization. Without any change in the calibration formulae, TAO predicted accurately the EOM transformations in the Malagasy context; three EOM are given as examples.

The IROC index and TAO model both appear as promising tools to optimize the management of urban and livestock waste but complementary calibrations are suggested to fit with the tropical areas.

1 INTRODUCTION

1.1 Background

Growing populations and the increasing use of existing resources has led to growth in organic waste emissions. Therefore, a sustainable approach to managing this waste has become a major concern in densely populated areas (Odlare *et al.* 2011). An increasing need to find a sustainable use for this material is inevitable. Known as Exogenous Organic Matter (EOM), organic waste from agricultural, urban and agro-industrial activities, used 'as is' or after transformation (e.g. compost) can be a valuable fertilizer for agricultural soils. Soil application of EOM could both contribute to environmental issue of improving C sinks to mitigate CO_2 emissions and to the restoration of soil properties related to soil organic matter content (Loveland *et al.* 2003). Tools for predicting the effect of EOM application on soil organic C status would contribute to the improvement of their management in cropping systems and to develop their use in agriculture (Lashermes *et al.* 2009). On the other hand, organic farming systems mainly rely on organic fertilizers such as compost, slurry, and animal or green manures to maintain nitrogen (N) nutrition of the crops (Nett *et al.* 2010).

EOM decomposition is classically assessed during soil laboratory incubation via a characterization of short- and medium-term effects of EOM input under controlled conditions (Thuriès *et al.* 2002; Morvan *et al.* 2006). The efficiency of EOM in increasing soil organic matter is also dependent on its degradation kinetics following its application on soils. This efficiency can be determined through a biochemical characterization of EOM using the Van Soest fractionation procedure. This method has been applied to the characterization of EOM such as animal manures and their composts (Charest *et al.* 2002; Thuries *et al.* 2002; Morvan *et al.* 2006; Francou *et al.* 2008), wastewater sewage sludges (Parnaudeau *et al.* 2004) and is now standardized in France (AFNOR 2009). A recent indicator of the fraction of added OM that might remain in soils has been defined, based both on the quantification of Van Soest biochemical fractions and on the mineralization of added carbon during short-term incubations (Lashermes *et al.* 2009): the IROC and is now standardized in AFNOR (2009).The IROC gives an indication about the stock of organic carbon in soil in order to improve the organic management in soil and the EOM organic N content permit to classify them into six categories (AFNOR 2011) .

In order to link EOM biochemical data to EOM transformations in soil predictive models were needed. TAO (Transformation of Added Organic materials) model was proposed to predict the AOM transformations: C and N mineralization, N immobilization and re-mineralization of immobilised N (citation). TAO is entirely driven by AOM biochemistry. The TAO-C structure fractionates EOM into three compartments which decompose in parallel like input compartments of most of the more complex soil organic matter models (Pansu *et al.* 2004) . It was simplified to use only two parameters which defined the very labile and stable fraction of EOM, and had been linked to EOM biochemical properties (Thuriès *et al.* 2002). TAO-N was defined using addition to TAO-C of two further parameters (Pansu *et al.* 2003), and had been again linked to EOM properties (Pansu *et al.* 2003). TAO was built up on EOMs from organic fertilizer industry of the temperate industrial zone in a South France Fluvisol (FAO-IUSS-ISRIC 2006)and was recently tested in an arid Ferric Lixisol of Ouagadougou (Kaboré *et al.* 2011).

1.2 Research objectives

The objectives of this study are (1) to test if an existing French typology based on the IROC and N contents allows a classification of selected Malagasy EOM and (2) to predict the transformations of three most used organic fertilizers in a Malagasy Ferralsol by using TAO model.

2 METHODOLOGY

2.1 EOM characterisation

The EOM used in this experiment included a range of fresh and transformed organic materials from agricultural, urban and agro-indrustrial waste applied in agriculture.

All EOM samples were oven-dried at 40°C and finely ground (<1 mm) before analysis. Total organic matter (TOM) content was determined by loss on ignition at 480°C, total organic C (TOC) content by dry combustion using an Elemental Analyser after the dissolution of carbonates with hydrochloric acid and total N (totN) content also by dry combustion .

The biochemical composition of EOM was determined using the Van Soest method (Van Soest 1963; Van Soest *et al.* 1967) as modified according to the French standard XP U 44-162 (AFNOR, 2009a).

2.2 Incubations in controlled conditions

The top layer (0–10 cm depth) of a Ferralsol from Lazaina (located in 47° 32’ 3”E and 18° 46’ 54” S) was used for the incubations. The main characteristics (on a dry matter basis) of this soil are : coarse sand 48.1%; fine sand 10%; coarse silt 2%; fine silt 3.5%; clay 32.9%; organic carbon 21.55 g kg^{-1}; total nitrogen 1.46 g kg^{-1}; C/N ratio 14.8; total phosphorus 275 g kg^{-1}; available phosphorus 9.6 g kg^{-1}; CEC 8.88 meq.$100g^{-1}$; pH (H_2O) 7.21. The collected soil sample was air-dried and sieved to 2 mm.

Incubation experiments has been driven according to the French standard XP U 44-163 during 182 days at 28 ± 1 °C in a dark room at 66% soil water-holding capacity. Mixed samples of 30 g air-dried soil, and 0,5 g of EOM (dry matter basis) has been used.

2.3 EOM Classification

2.3.1 Index of remaining organic carbon (IROC)

The indicator of remaining organic carbon (I_{ROC}) estimates the stable pool of EOM-TOC potentially remaining in soil over the long term after application. It is calculated using the SOL, CEL and LIC fractions of the Van Soest fractionation and the proportion of EOM-TOC mineralized after 3 days of incubation (C_{3d}), according to the formula:
$IROC = 44.5 + 0.5\, SOL – 0.2\, CEL + 0.7\, LIC – 2.3C_{3d}$ (gC.$100g^{-1}$EOM)
It is expressed as a percentage of TOC. (Lashermes *et al.* 2009).

2.3.2 N typology

Figure 1 summarizes the classification of EOM according to the N typology described by Lashermes *et al.(2010).*

2.4 TAO model

TAO-C is a parallel three-compartment model using only two parameters (very labile (P'_L) and stable (P_S) fraction of EOM) to predict C mineralization.

TAO-N uses two additional parameters to predict N transformations (fraction P_{im} of transformed EOM which is reimmobilized, rate k_{remin} of remineralization of this immobilized fraction). For few N-rich EOMs a further parameter was used, the rate k_V for gaseous losses of produced inorganic-N. Thuriès *et al.* (2001) showed that the mineralization rates of very labile (k'_L) and resistant (k_R) compartments of AOC could be considered as constants in these incubation conditions with $k'_L = 0.4 \pm 0{,}15\ d^{-1}$ (half life $T_{1/2} = 1{,}7$ d) and $k_R = 0.012 \pm 0.003\ day^{-1}$ ($T_{1/2} = 58$ d). The mineralization rate of the Stable compartment [S] was set to 0 for a 6-(or 3-)month experiment.

So the residual AOC fraction (RAOCF) after C mineralization was given at a known time of incubation (t) from time of EOM application (t_0) by:

RAOCF = $P'_L e^{-0.4(t-t0)} + (1 - P'_L - P_S)e^{-0.012(t-t0)} + P_S$ where $(1 - P'_L - P_S)$ corresponded to the fraction of intermediary resistant compounds.

From Pansu and Thuriès (2003), the RAONF could be approximated knowing only C-to-N ratio of EOM (C/N_{EOM}) by:

$$RAONF = \frac{1}{EOM}\left(P'_L e^{-0.4(t-t0)} + (1 - P'_L - P_S)e^{-0.012(t-t0)} + P_S\right)$$

TAO was programmed on VENSIM 4.2 (Ventana systems Inc., Harvard, MA, USA). Simulation was run without any modification of the initial model equations and parameter estimations (Thuriès et al., 2002; Pansu et al., 2003). The significance of TAO predictions comparatively to mean of measured values was tested by:

$$F = \frac{\sum_{i=1}^{p}(\bar{y}_i - \bar{y})^2}{\sum_{i=1}^{p}(\bar{y}_i - \hat{y}_i)^2}$$

where i = 1 ,. . ., p was the number of sampling occasions, $\bar{y}_i$ the mean measured value at i of MAOCF or inorgN, $\hat{y}_i$ the corresponding predicted value by TAO and $\bar{y}$ the mean of the data series.
F value was compared to F tables for bilateral test at 1%, 2% and 5% risk.

3 RESULTS

3.1 EOMs classification

The results of the characterisation of MOEx: C contents, N contents, the index of stable organic C in Malagasy EOMs based on their biochemical fractions and the N class according to Lashermes et al. (2009) are summarized in table 1.

Diversity of EOM are shown by the variability of (i) C contents (range of 4.55 to 42.26 gC.100g^{-1}dm for TrT and Vgt, respectively);(ii) N contents (0,24 to 14.19 g.100g^{-1}dm for TrT and SpA, respectively); (iii) IROC values between 7.5 and 75 gC.100g^{-1}EOM.

The typology established by Lashermes et al., (2010) allowed a classification of Malagasy EOM according to their N content and their biochemical fraction. Slaughterhouse waste (SpA) and poultry droppings (FiP) belonged to Class 1; they are supposed to have very high N mineralization potential. The zebu ground horn (SpZ) is categorized in class 2 i.e. an EOM with high mineralization. The groundnut cake (SpH) is considered as an EOM with a medium to low N mineralization potential, class 4. Most of the Malagasy EOM studied here (80%) have a low N mineralization and present a risk of N immobilization in soil: class 5. For the Malagasy EOM tested here, this category regroups as well as animal and urban materials (composted or not)

3.2 C and N mineralization; their modelling

Three EOMs (CoM, FiV and FuBb) are presented here as they are representative of wide-spread fertilizers in Madagascar. Figure 2 presents both the C- and N- mineralization measured in laboratory and their predictions obtained by using TAO. The dynamics of C decomposition in soil were different. Decomposition of carbon from the dairy manure FuBb was lower than the 3 EOMs. Only 32% of C added are mineralized during the experiment. CoM is a urban compost obtained from market waste. During the 182 days of incubation, 52% of carbon are mineralized. Decomposition kinetics were rapid for the FiV, a poultry dropping: 82 % of added carbon was transformed. TAO-C estimate accurately the C mineralization for these three EOMs. The F-tests results show any significant different between the measured and predicted values (interval confidence of 95%) The organic N of the EOM considered here was slowly and weakly mineralized. Until 28 days of incubation, immobilization of N can be observed for the 3 EOMs. The curve of N mineralized estimated by TAO-N does not show accurately this immobilization but in general the patterns of predicted values fit with our observation. The F-tests confirms that the measured data of N mineralization were predicted accurately at 5% risk by TAO for the 3 representative EOMs.

4 DISCUSSION

4.1 Classification of Malagasy EOM

The IROC of Malagasy composted materials are low, 22%, as well as for urban and manure composts. According to Lashermes *et al* (2009) the IROC of urban manures varies between 30 and 80 percent of EOM added and the IROC for composted manure is about 45 to 87 percent of EOM added. This difference can be explained by (i) the characteristics of raw materials composted, (ii) the process of composting and (iii) the product maturity (Paillat *et al.* 2004). For the animal dejection without litter, a quantity between 40 to 80 percent of EOM added can remain in the soil (Lashermes *et al.* 2009). If the dairy manure is correctly classified, it is not the case for the pig slurry which IROC is only 29%. This fact can be related to the variation in composition with type and age of animal, food, water content, storage and handling, climate, and amount of particulate material (Prairie Swine Centre Incorporation 2000)existing between temperate and tropical environments. Lashermes *et al.*(2009) reported that for animal dejections with litter, the IROC median is 50%. So, in general Malagasy manure considered here are similar to the French EOM. Three of the four animal by-products (SpA, SpH, SpP) considered in this study match with their supposed category because the IROC values varied between 25 to 75 %, only the zebu ground horn (SpZ) is excluded.

EOM are in general classified in the category 5 because of their low nitrogen content (< 6,5%) for 80% EOM and the importance of resistant fraction (LIC>20%). The reluctance of N mineralization can be justified in this way. The EOM which have a very high potential of N mineralization, class 1, are the slaughterhouse waste (SpA) and one of the poultry droppings (FiP). According to Morvan *et al.*(2009), due to their high N- and labile fraction- contents, a rapid transformation of the organic N into mineral N has occurred. The EOM with a potential of medium to low mineralization are categorized in class 4, it is the case for SpH which is essentially a groundnut cake so it is a product with a high protein contents.

Using the IROC and N-typology based on French EOM to classify Malagasy EOM can be considered as a first approach to assess their behavior in soil. The N classes and IROC values are similar for animal dejections and by-products but need to be adjusted for composted materials and urban waste. The establishment of tropical EOM typology is suggested for the composted materials and urban waste because their composition varies according to cultural habits, economic status, urban structure, density of population, and extent of commercial and industrial activity. A comparative study confirms the numerous results obtained on lifestyle differences of the two population types, the role of weather (higher production of fruits and vegetables in summer), food processing activities and socio- cultural status.(Guermoud *et al.* 2009).

4.2 Prediction of EOM transformation in soil using TAO model

Predictions with TAO focused on 3 materials: an urban compost (CoM) , a poultry dropping (FiV), and a dairy manure (FuBb). Even though, the mineralization patterns of manures might be more difficult to predict than those of plant residues, as the heterogeneity of manures derives not only from the biological origin and biochemical quality of the feed but also animal species, manure handling and storage, and stage of decay (Bruun *et al.* 2005)good modelling of the dynamics of C and N predicted by TAO can be reported in this study.

TAO-C applied in the selected EOM here is performant as confirmed by the F-test. As written by Kaboré *et al.*(2011)TAO-C predicted accurately the C-mineralization of raws materials and has a tendency to overestimate the C-mineralization of composted materials. FiV and FuBb are both raw materials, so modelling the transformation of C in soil can be easily directed by TAO. Even though CoM was an urban compost, a good prediction of his C transformation can be observed here.

The transformation of added organic N was well predicted by TAO for FuBb which give an immediate net production of inorganic N. The two another materials FiV and CoM presented N immobilization during the first month. For this period, TAO-N slighty overestimates the inorganic N production. During the last five months, because the measured inorganic N was relatively constant, TAO-N allows a better estimation of N dynamics.

4.3 Elaboration of Malagasy EOM typology

As can be seen in this study, building a Malagasy EOM typology seems necessary to better characterize them and predict their transformations in soil. To do that, a database regrouping the main characteristics of Malagasy EOMs: C contents, N contents, biochemical fractions are taken in consideration. To elaborate the typology, the chemical composition of EOMs will be carefully revised in each cluster in order to define (1) thresholds for each composition criterion associated to their transformation in soil; (2) the decision tree used to attribute each EOM to only one class according to its behavior. Each step of the decision tree was based on the comparison between the chemical composition of an EOM and the thresholds defined in the decision tree. Thus, each EOM was progressively attributed to a single class of nutrients availability. A multiple factor analysis coupled with an ascending hierarchical classification will be carried out on a subset of the calibration data collection that had C and N mineralization value in define date respectively, to better define the criteria used for their discrimination. Finally, once the composition criteria and the decision tree were defined for all classes, the proposed typology will be tested back with the EOMs used for its definition and validated with another data collection.

Such strategy needs a large panel of EOM in order to have a robust classification. Because traditional laboratory analyses are expensive in terms of time and resources, a rapid estimation of EOM composition and transformation have to be identified. On the one hand, the near infrared spectroscopy (NIRS) technique will be chosen to allow a rapid and low cost determination of several parameters of EOM. On the other hand, TAO model will be adopted to predict the behavior of EOM in soil.

5 CONCLUSIONS

If most of Malagasy animal waste match with the characteristics of the main types of French EOM applied in soil, the urban waste and composted materials do not meet their supposed category. For the typology established by N-mineralization, the majority of our EOMs presents a low N mineralization and may involve N immobilization in soil. The classification and index based on the French EOM can be taken as a first approach to categorize Malagasy EOM. The establishment of typology is suggested to allow more genericity of results in order to improve the prediction of their effects in soil.

Although the initial TAO model calibration (Thuriès *et al.* 2002) was made using organic materials from temperate zone incubated in Fluvisol, the present study used organic materials from tropical waste incubated in a tropical Ferralsol, it can be a promising tool to predict the transformation of Malagasy EOM in soil.

6 ACKNOWLEDGEMENTS

This work was funded by the Reunion Island Region and the European Union (FEDER:Fonds Européen de Développement Régional) according to the “Programme Opérationnel de Coopération Territoriale”. We thank also the International Foundation for Science to their financial support for laboratory and field equipements.

Figures and Tables

TABLE 1 **Characterisation of MOEx**

Nature	**Code**	**C (% d.m)**	**N (% d.m)**	**IROC (%OM)**	**N Class**
Mixed compost	CoA	15,73	1,37	27,12	Class 5
Composted fermentable waste	CoM	21,47	1,11	22,17	Class 5
Composted fermentable waste	CoV	20,35	0,83	22,18	Class 5
Marketed compost	Vgt	42,26	2,47	55,04	Class 5
Poultry droppings	FiP	40,60	9,02	52,21	Class 1
Poultry droppings	FiV	25,83	2,44	36,24	Class 5
Dairy slurry	LiB	29,34	2,15	52,05	Class 5
Pig slurry	LiP	16,85	0,92	29,19	Class 5
Dairy manure	FuBb	33,20	2,79	54,10	Class 5
Dairy manure	FuBp	31,06	2,35	46,58	Class 5
Rabbit manure	FuL	15,40	1,61	40,07	Class 5
Goat manure	FuO	37,90	2,51	38,20	Class 5
Pig manure	FuP	14,29	0,79	27,26	Class 5
Slaughterhouse waste	SpA	41,99	14,19	62,38	Class 1
Oil mill waste	SpH	27,77	5,83	75,05	Class 4
Fishery waste	SpP	30,80	5,13	32,18	Class 5
Zebu ground horn	SpZ	17,70	6,81	48,68	Class 2
Potting soil from landfill	TrA	12,65	0,56	27,38	Class 5
Potting soil from landfill	TrI	5,79	0,38	14,10	Class 5
Potting soil from landfill	TrT	4,55	0,24	7,24	Class 5

FIGURE 1 **N typology (Lashermes et al., 2009)**

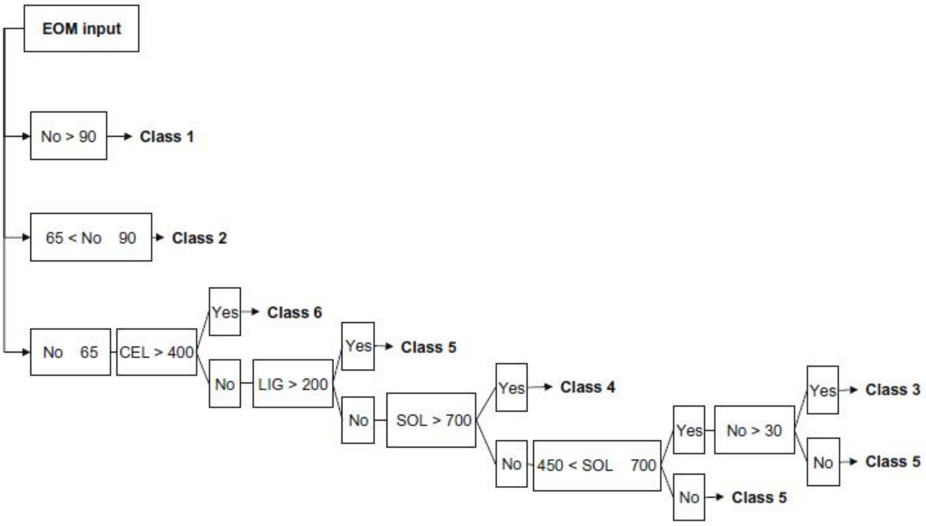
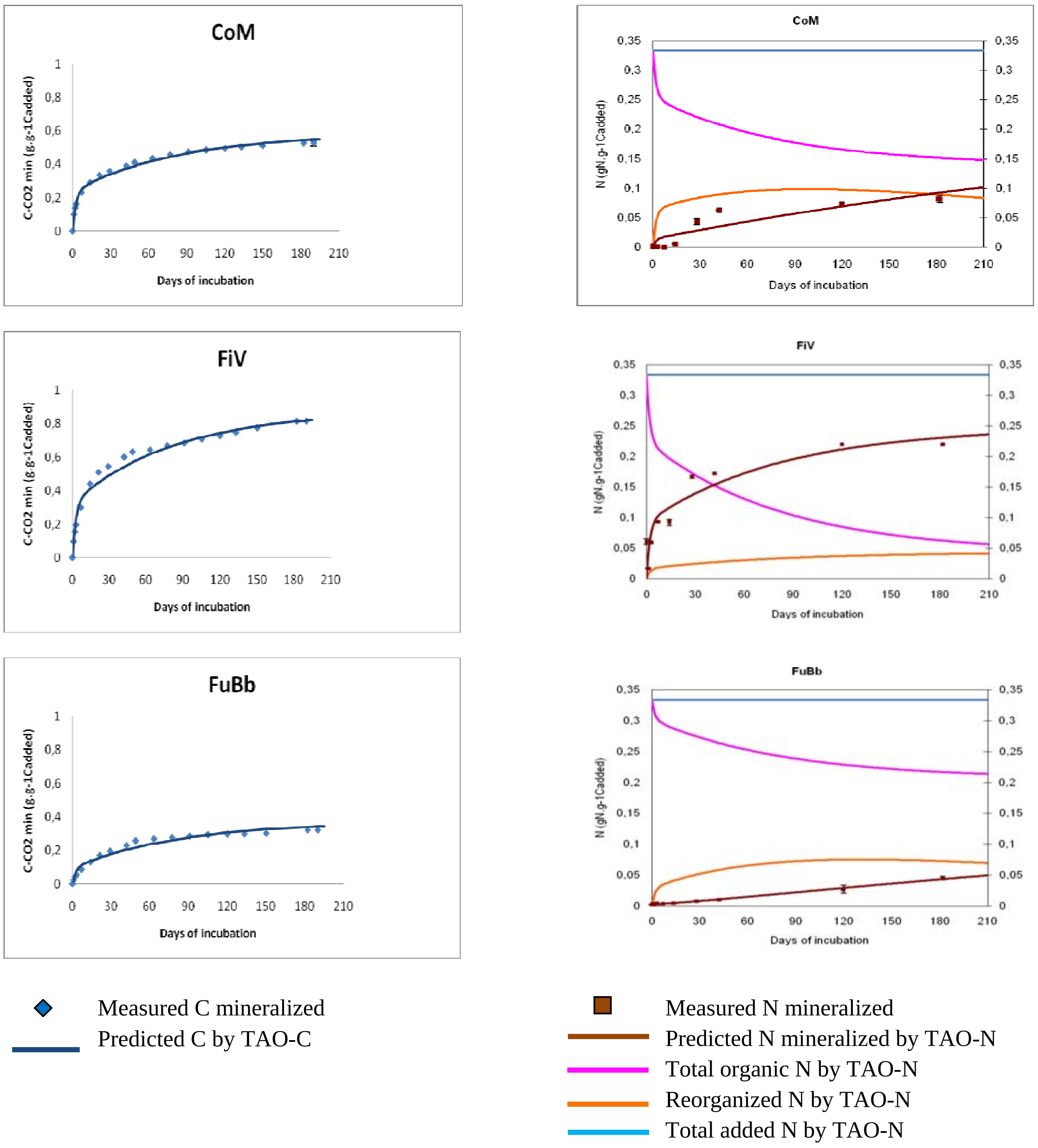


FIGURE 2 **C & N mineralization: measured in laboratory and predicted using TAO**



Carbon mineralization

Nitrogen mineralization

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(146) USE OF A UV- VISIBLE SPECTROSCOPY METHOD TO DETERMINE THE ORGANIC MATTER CONCENTRATION IN ANDISOL SOILS AND SOILS TREATED WITH ORGANIC AMENDMENTS OF CHILE

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EXECUTIVE SUMMARY

The central role of soil organic matter in maintaining soil functions and plant productivity in agroecosystems has long been recognized. Soil organic matter (SOM) content is considered a soil quality indicator, whose advantage is to be simple and sensitive to measure environmental stresses and, consequently, may provide a rapid and accurate assessment of changes in soil. There are many methods for measuring SOM, of different complexity, requiring various pieces of equipment. The goal of this research was to evaluate a quick and simple method based on UV-visible spectroscopy to estimate SOM fractions, including total and soluble, in Andisols and carbon-amended soil samples. For this, 95 Andisol and 64 C-amended Inceptisol soils samples were selected. Samples were analyzed for SOM through the Walkey-Black method and for water-soluble carbon (WSC). The UV-visible spectroscopy method consisted on extracting the soil with 0.5M NaOH in a 1:10 soil to extractant relation, centrifuged by 5 min at 9000 rpm, and reading the supernatant at 465 nm and 665 nm. Correlation and regression analyses were performed to determine the performance of the method against standard ones. Results demonstrated that there was a significant and positive relationship between SOM and the absorbance at 465 nm and 665 nm (R^2=52% and R^2=50%, respectively). However, SOM by the Walkey-Black method did not increase with the rate of C applied at amended soils (R^2=3%); but this increase was observed when using the UV-visible spectroscopy method (R^2= 80% at 665 nm and R^2= 29% at 445 nm). When evaluating the performance of UV-visible spectroscopy method against WSC very poor correlations were observed indicating that the extracted fraction does not represent this C pool. The way of using this index would be as follows: A composite sample is analyzed for SOM by the Walkey Black (WB) method as well as by the UV-visible spectroscopy one; individual samples are analyzed by the UV-visible spectroscopy method; readings are transformed to SOM values by using the quotient between WB and the UV-visible spectroscopy method. Thus, it was possible to conclude that the spectroscopy method is an economical, rapid and useful tool to quantify and estimate the organic matter content in Andisols and temporal and management-induced SOM changes in soils.

1. INTRODUCTION

Almost all soil and crop management practices have implications for Soil organic matter (SOM), including tillage and planting techniques, methods of handling crop residue, application of organic amendments, crop rotations, and use of cover crops. Soil organic matter (SOM) corresponds to soil organisms, simple organic compounds, large and complex humic substances (HS), as well as relatively fresh residue in various stages of decomposition that are influenced by soil and crop management practices (Magdoff &Weil, 2004).

The SOM content in a soil is considered an indicator of soil quality, which is able to determine and show, early and sensitively, changes or stresses in a given ecosystem; its evaluation in a soil has been related to soil productivity, even in well-fertilized soils; soil productivity is usually reduced by loss of SOM, because it is related with the efficiency of agroecosystems (Magdoff &Weil, 2004).

The content of organic matter in soils ranges from 2g kg^{-1} in some desert soils to more than 800 g kg^{-1} in some Histosols; however, usually in cultivated systems SOM contents are from 10 to 40 g kg^{-1} in the A horizon (Magdoff & Weil, 2004).

Physical, chemical and biological processes occurring in soils are greatly influenced by soil organic carbon (SOC) content. Estimates of SOC are often required for a wide variety of agricultural, environmental and engineering applications. Quantitative and semiquantitative methods are available for SOC determination, although the former are more accurate than latter ones, they are also typically more expensive and time consuming. For example, the wet oxidation method (Walkey-Black method), is generally suitable for the analysis of a small number of samples; however for precision agriculture applications where a large number of samples is analyzed, the method becomes wasteful and time consuming. For that reason it is necessary to develop other techniques that are cheaper, easier and reliable to estimate SOC, allowing estimating and modeling the spatiotemporal variability in SOC across small as well as large geographic areas; besides performing cross study comparisons and the development of more consistent databases of SOC pools under different land uses and management practices (Harvey *et al.*, 2008).

Soil organic matter has very complex and heterogeneous composition and is generally mixed or associated with the mineral soil constituents to form soil aggregates. The SOC dynamics is mainly influenced by its recalcitrance and accessibility, and its interactions with soil components. Recalcitrance is the inherent molecular level resistance of a molecule to microbial breakdown. Interactions of organic substrates with organic or inorganic molecules may alter the degradation rate of the substrates. Several studies have shown that in Al-rich soils, such Andisols, chemical stabilization is the determining process for carbon mineralization. However the exact role of SOC and Al in the process of chemical stabilization, and which kind of interactions occurs among the different soil components is not yet fully understood (Huygens *et al*., 2005). Meanwhile, labile SOM can be assessed effectively by its characterization, which is strongly influenced by soil management, because it has a higher proportional response to management as compared to other measures of labile SOM. The major portion of SOM usually consists of a conglomeration of relatively recalcitrant organic molecules termed humus. The humic substances (HS) are understood to include a continuum of complex biogenic amorphous heterogeneous molecules that are chemically reactive and refractory in nature and that are ubiquitously formed through random chemical alteration of diverse precursor molecules, and they are divided into labile and recalcitrant fractions based on the ease with which they can be removed from soil (Magdoff &Weil, 2004).

A common method to estimating SOM is the determination of total organic C by wet oxidation with sodium dichromate in strong acid. When SOM is reported by this method, the value determined for organic C must be multiplied by a standard factor designed to account for the method’s incomplete oxidation of SOC (traditionally assumed to be 77%, however, for a range of tropical Alfisols, Inceptisols and Vertisols, the value can be close to 100%) and then by another factor based on questionable assumption that the SOM always contains 58% C. Although most laboratories use the same standard factors for these purposes, the factors are certainly not equally appropriate for all soils (Magdoff & Weil, 2004).

A second commonly used method estimates total SOM from the weight lost when a dry soil sample is ignited at a temperature high enough to volatilize all organic material. Various loss on ignition (LOI) methods use different durations and temperatures for the ignition, but usually give values of SOM about 25% higher than those by Walkley Black method (Magdoff & Weil, 2004).

The term HS is a generic name used to describe colored material or its fractions including humic and fulvic acids. Humic substances are the dark-brown, fully decomposed remains of plant and animal organic matter. They are the most chemically active compounds in the soil, with cation exchange capacities far exceeding those of clay. The genesis of HS is one of the least understood subjects of humus chemistry. In natural processes HS are formed through the process called humification; this is a natural process of changing organic matter such as leaves into HS by geomicrobiological mechanisms. This process leads to the formation of complex chemical structures, which are more stable than the chemically structures of the starting material and provide a concentrated and economical form of organic matter that can replace humus depletion caused by conventional soil management practices (Mema, 2006, Giovanela *et al.,* 2010).

- Research objectives

The goal of this study was to evaluate a method based on UV-visible spectroscopy to estimate SOM fractions, including total and soluble, in Andisols and carbon-amended Inceptisol soil samples.

2. METHODOLOGY

2.1. Soil Sampling

We evaluated 95 Andisol soils samples from the Bío-Bío region of Chile (37º13’52’’S, 71º54’6’’W) and 64 C-amended Inceptisol soils sample from the Coquimbo region (29º54’28’’S, 71º15’15’’W), whose treatments were based on the addition of different concentrations of organic matter to the soil, using humic substances, at rates of 0, 125, 250, and 500 kg C ha^{-1}, which was sampled after two years of the application of the organic amendments.

2.2. Determination of soil organic carbon fractions

Samples were analyzed for SOM through the wet oxidation method, and for water-soluble carbon (WSC) in a 1:10 soil to water relationship. The determination of C was performed colorimetrically following the method described by INIA (2006) for Chilean soils.

2.3. Development the UV-visible spectroscopy method

The UV-visible spectroscopy method consisted on extracting soil previously sieved (2 mm) with NaOH 0.5M in a 1:10 soil to extractant ratio and agitated for 30 minutes in a reciprocal shaker. Then, this suspension was centrifuged for 5 min at 9000 rpm, and finally the supernatant read at 465 nm and 665 nm. For Andisol soils samples the adequate dilution to read the absorbance was 1:20, while for C-amendment Inceptisol soils samples was not necessary to do any dilution of the extracts.

2.4. Statistical analyses

Correlation and regression analyses were performed to determine the performance of the proposed method against standard ones.

3. RESULTS AND DISCUSSION

Soil organic matter is non homogeneous and not well defined, chemically, material; it cannot be measured directly as a specific substance. Instead, methods used routinely for its analysis generally measure some proxy for SOM, such as total organic C, total C, or weight loss on ignition. Unfortunately, the methods used to determine SOM vary from laboratory to laboratory. Each method in common use has its own limitations and inaccuracies, and the estimate of SOM obtained by one method can differ significantly from that obtained by one of the alternative methods. Making comparisons between some values obtained by different soil test labs or by different pieces of research can be fraught with ambiguity. However, most soil test labs routinely analyze for SOM and therefore information on SOM content is widely available and used for many purposes. Therefore, it is important that users appreciate the differences among the methods in common use (Magdoff & Weil, 2004).

Nondestructive spectroscopic techniques provide valuable information on the molecular structure and chemical of functional properties of HS and are widely used as indicators of changes in composition induced by management practices, based in the dynamic of the HS. Specifically, the E4/E6 ratio (ratio of the absorbance at 465 nm and at 665 nm) is often used to characterize the HS fraction and it is considered to be inversely related to the degree of humification; it is also suggested that this ratio is governed mainly by molecular sizes and weights. Furthermore, E4/E6 ratios appear to be strongly influenced by the type of soil management, as the ratios for samples from conventional management soils are lower (Magdoff & Weil, 2004).

In the present study the Walkey Black method was defined as the standard methodology to quantify SOM, widely used for this objective. This method was established, by the Chilean National Commission of Accreditation (CNA, for its abbreviation in Spanish), as the standard method for Chilean soils. Meanwhile, the WSC quantification was performed through the extraction of the fraction in a 1:10 soil to water relationship, whose estimation was based in the wet oxidation and colorimetry.

The results of the present study demonstrated that in soils with high levels of organic matter (Andisol soils), the HS extraction with an alkali solution and its absorbance value at 465 nm and at 665 nm (UV-visible spectroscopy method), is a fast, sensitive and economic methodology to determine SOM content. The method is highly correlated with SOM by Walkely and Black at both wavelengths, 465 nm and 665 nm (R^2=52% and R^2= 50%, respectively) (Figure 1)..

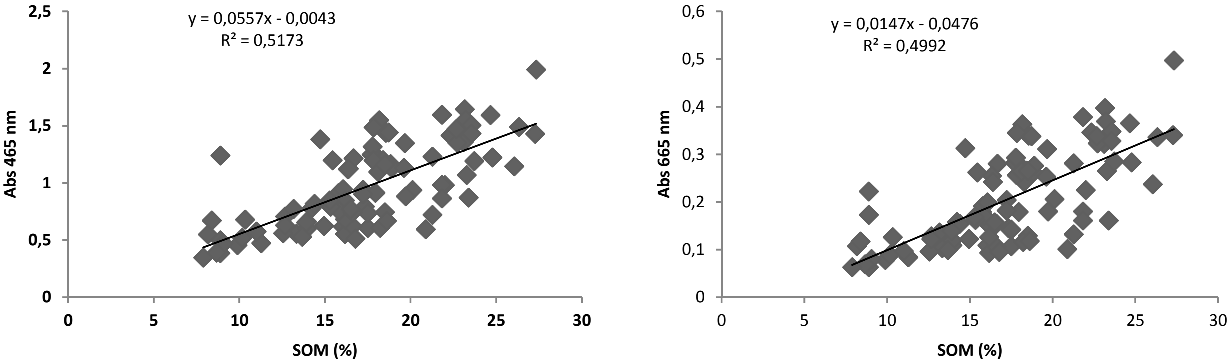

Figure 1. Correlation between SOM content determined by Walkey Black method and UV-visible spectroscopy method in Andisol soils. **A.** 465 nm and **B**. 665 nm.

Andisol soils develop from volcanic ash and are characterized by high soil organic matter content, good physical properties, and high potential anion or cation exchange capacity. Their clay fraction consists mainly of poorly crystalline allophone and oxides and oxyhydroxides of Fe and Al. Allophane is a group of clay minerals made up of silica, aluminum and water. This chemical composition, which favors the sorption by the formation of metal-humus complexes, would act as binding agent between aggregates of soil particles. In these metal-humus complexes, SOC can be complexed by allophone through ligand exchange between functional groups, through cation bridging, water bridging, hydrogen bounding and van der Waals forces (Huygens *et al*., 2005). Thus, SOM mineralization in Andisol soils is influenced by the intrinsic chemical characteristics of the substrate, which do not allow a continuous degradation of the organic matter, ensures the aromatic structure's stability and therefore its characterization on UV-visible spectroscopy analysis is possible, being homogeneous and accurate for all evaluated Andisol soils; its molecular characterization does not depend on its concentration but its quality, stability and purity of the aromatic groups, whose absorbance, in the UV region, is due to the contribution of $\pi - \pi^*$ electron transitions in the phenolic arenas, aniline derivatives, polyenes and phenolic aromatic hydrocarbons with two o more rings. Hence, the loss of aromatic structures of humic acid results in significant changes in the UV-visible spectrum of these substances and an increase in the degradation of aromatic structures causes a decrease in extinction values (Mema, 2006; Magdoff & Weil, 2004). Now, the same explanation sustains the low correlation between WSC and the UV-visible spectroscopy method (R^2=17% y R^2= 22%, respectively) (Figure 2); the low mineralization rate in these soils does not generate stable results in WSC. Although it is possible to infer that SH extracts contain WSC, the SOC fraction estimated by UV-visible spectroscopy method does not correspond to the WSC content of the Andisol soils.

Thus, the way of using this new technique would be as follows: A composite sample is analyzed for SOM by the Walkey Black (WB) method as well as by the UV-visible spectroscopy one; individual samples are analyzed by the UV-visible spectroscopy method; readings are transformed to SOM values by using the quotient between WB and the UV-visible spectroscopy spectroscopy method.

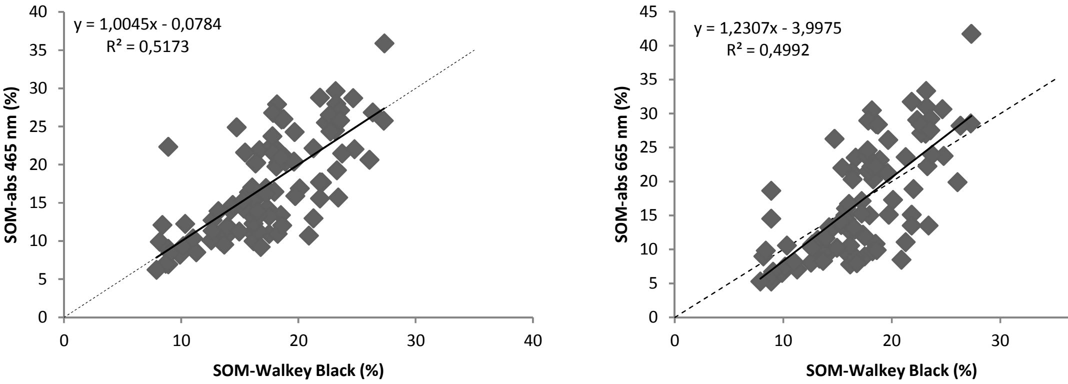

Figure 2. Results of the regression line of the spectroscopy method at 465 nm and 665 nm, comparing SOM concentration obtained by spectroscopy procedure with the results calculated by the WB method.

Method performance was evaluated by calculating the standard error of prediction and comparing, through a regression line, the results obtained by the procedure above described with the results calculated by the WB method, evaluating R^2 and testing whether the intercept=0 and the slope=1. The results demonstrated that the method is valid at both wavelengths since the confidence intervals contained the values of 1 for the slope and zero for the intercept, respectively (Table 1, Figure 2).

Table 1. Results of the standard error of prediction and regression line parameters of the spectroscopy method at 465 nm and 665 nm

Parameters		**Abs 465 nm**	**Abs 665 nm**
Intercept	**Coefficients**	-0,078	-4,00
	Lower 95%	-3,66	-8,55
	Upper 95%	3,5	0,55
Slope	**Coefficients**	1,00	1,23
	Lower 95%	0,80	0,98
	Upper 95%	1,20	1,48
R^2		0,52	0,50
Standard error of prediction		1,9%	2,2%

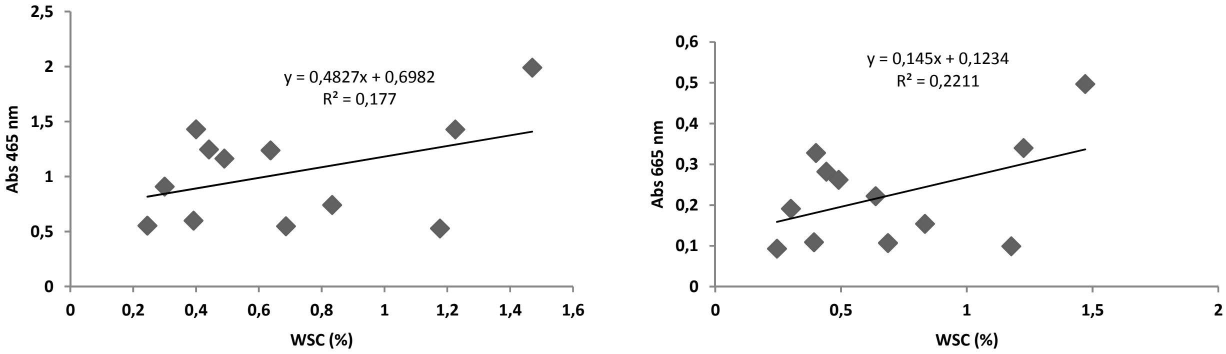

Figure 3. Correlation between WSC content and the UV-visible spectroscopy method in Andisol soils. **A.** 465 nm and **B**. 665 nm.

On the another hand, evaluating the carbon-amended Inceptisol soils, it was possible to find a low correlation between UV-visible spectroscopy method and SOM content in these soils, with R^2= 17% at 465 nm and R^2= 1,5% at 665 nm, respectively. However, it is important to note that the SH extraction and characterization is a good indicator of the influence of the type of soil management. In a study carried out by Aranda *et al.* (2011) it was determined that both structural and composition differences observed in HS were related to the type of management (organic vs. conventional) and soil type, which were related to the microbial transformations of plant waste, input of microbial metabolites and enrichment in chemically stable molecules. That explanation reflects the behavior of the results of this study; the decomposition of the organic materials added to the soil generates a change on the humic structures and its absorbance in the UV-visible spectroscopy method, which will depend on the soil specific chemical, biological and biochemical characteristics, resulting in a low correlation. Also, this explanation sustains the low level of correlation between WSC content and UV-visible spectroscopy method (R^2=0,4% y R^2= 0,1%, at 465 nm and 665 nm, respectively), which indicates that the results obtained by the UV-visible spectroscopy method do not correspond to the soluble fraction of SOC. However, in a study performed by Harvey *et al.* (2008) it was demonstrated that NaOH alone was not an efficient extractant in Histosol soils, due to the fact that clay and $CaCO_3$ can physically protect organic matter from extraction, which may in turn explain the results obtained in the present assay in the Inceptisol soils.

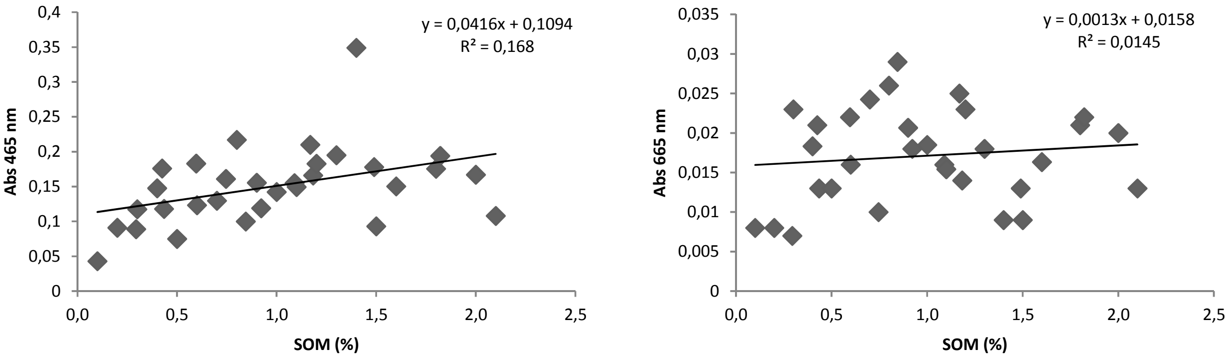

Figure 4. Correlation between SOM content determined by Walkey Black method and UV-visible spectroscopy method in C-amended Inceptisol soils. **A.** 465 nm and **B**. 665 nm.

Nevertheless, knowing that the SH characterization of soils can predict crop management, two regression analyses were performed: a) UV-visible spectroscopy method on C rate, added to the soil in the form of liquid hunus, and b) SOM content estimated by the Walkey Black method on C rate. These analyses demonstrated that the UV-visible spectroscopy method is more sensitive to changes in soil by agronomic practices, along time, that the SOM determination by Walkey Black method (Figures 5 and 6).

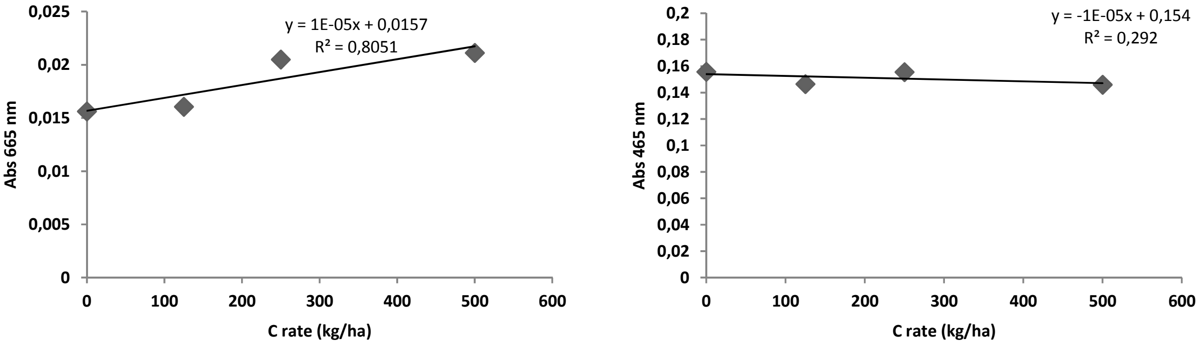

Figure 5. Correlation between C amendment concentrations added to Inceptisol soils and UV-visible spectroscopy method in C-amended Inceptisol soils. **A.** 465 nm and **B**. 665 nm

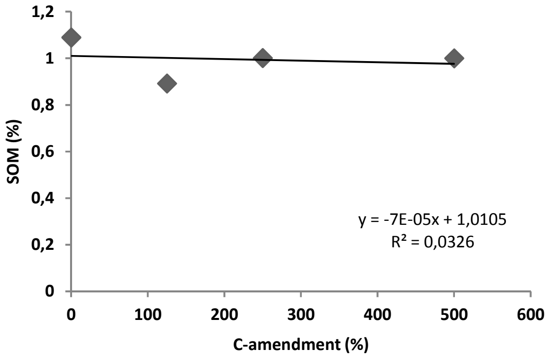

Figure 6. Correlation between C amendment concentrations added to Inceptisol soils and SOM content determined by Walkey Black method.

4. CONCLUSION

The spectroscopy method is an economical, rapid and useful tool to quantify and estimate organic matter content in Andisols and temporal and management-induced SOM changes in Inceptisol soils.

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(168) USE OF COMPOST AND DIGESTATE IN ITALY: QUALITY REQUIRMENTS, LABELLING AND USE IN SOUTHERN EUROPEAN SOILS

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The Europe has reached excellent levels of compost production. The total amount of compost quality in EU is estimated about 12 mln tons di compost in 27 European countries. Does ‘n exist an European legislation on quality criteria for compost. Every countries sets rules for compost production and use. Is necessary to establish laws or regulations to harmonized the quality levels of compost. In southern Europe is very important to find new sources of organic matter to improve the C levels in the soils. The agricultural techniques and the low availability of manure caused a strongly decrease of organic matter in soil mainly in southern Europe. Scope of the paper is to analyse the agronomic characteristics of the different qualities of compost produced and the way they fulfil the quality requirements needed by the potential end users (e.g. farmers, landscapers, plant breeders, vine-growers, etc.). This paper shows the Italian agronomical trials of compost use. The main result is that the compost may satisfy the needs of the farmers and nurseryman. Finally the document provides suggestions on what has to be done to regulate, promote and develop the compost market: labels, quality limit values according to specific features in the different end-use sectors.

(220) EVALUATION OF STANDARDS AND PROCEDURES TO REGULATE PRODUCTION AND QUALITY OF MUNICIPAL COMPOST IN THE PHILIPPINES

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EXECUTIVE SUMMARY

In emerging economies were municipal solid waste typically consists of a high proportion of organic residues which amounts up to 60% mass or more composting is a favorable technology to transfer that part of waste into a marketable product for agriculture or gardening to improve soil fertility especially in nutrient deficient and/or devastated areas through its humus and mineral content. However, application potentials including marketing opportunities depend on high quality of the produced composts which term refers to valuable criteria such as organic matter content or plant nutrients, as well as precautionary environmental criteria such as hygienic aspects and the content of potentially toxic substances.

This is of highest importance in the case organic components of municipal solid waste (MSW) are used for composting. Only source separated organics must be applied for compost production since the quality of the compost by no means will be better than the raw material was. Good composting practice is another pre-condition for a good result. Low quality compost will become rejected by potential users, and a composting industry eventually cannot be established or successfully sustained in this way.

For proper composting and high quality products a specific legislation is necessary which may consider best international practices for composting adapted to the concrete situation on site and the amount to be treated. However this is in many cases missing or differs from values given by best composting practices after long term international experiences.

In the Republic of the Philippines composting is on its way to become a part of MSW management systems on all levels from Municipalities to Provinces. Standards for composting are under development as part of the Organic Agriculture Act established in 2010. This situation may be seen as typical, and therefore it can be used a case study, for the situation in many other emerging economies where establishing of green technology becomes a mayor political issue.

In the Philippines the Organic Agricultural Act favors the usage of compost as a soil improving agent and a plant fertilizer. Amongst sources of raw material for composting besides agricultural residues, MSW components were also listed. A specification of quality parameters was detailed in the National Standard by 2008.

The evaluation of the standards results in the following statement: The regulation is not really clear and easily applicable. A “positive list” of material suitable or accepted for composting and production of fertilizers is missing and should be prepared after regional substrate availability or waste produced. Moreover, the values allocated for quality parameters are in many instances far from what is proven internationally e.g. by ECN network or by long term tried and tested national regulations. This will make it difficult to establish a compost industry and introduce compost into agriculture supporting green technology. A part of the values seem very strict. An example is the number of foreign material which after the Standard has to be totally removed. ECN proposals include a practical value in case of pieces larger than 2 mm. In case of heavy metals some values exceed the ECN proposals by more than fivefold.

It is proposed to consider the typical situation for small composting plants which are typical for the treatment of organics separately collected in most of the Municipalities and which will not exceed an amount of 6,000 tons per year. A simple but highly effective process control especially with respect to hygienisation involves the temperature measurement in the composting pile. Same can be considered proven if the temperature will exceed 55°C over 10 days or 65°C over 3 days. A sophisticated sanitary test procedure is also not necessary in case proven technologies were applied such as windrows. Quality of the end product may be defined also after its maturation state which term refers to the degree of decomposition of the organic matter. Here it is not necessary to totally decompose the organics as it is ruled in the Standard but to determine the maturation degree after the simple self heating test in a Dewar flask.

1. INTRODUCTION

1.1 Background

The Philippines belongs to a group of states which are most vulnerable to climate change impacts. Therefore the country supports the trend of green technologies on the low carbon development path as one of the cornerstones for future sustainable economic development. This situation coincides with another urgent problem the Philippines is faced with: The large amount of organic residues produced by industry and agriculture, and also organic, which make up a large proportion of municipal solid waste (MSW) which in most cases are deposited on landfills or on dump sites causing huge amounts of climate relevant emissions of Methane and other burdens on the environment. The proportion of organics in MSW is about 66% and amounts to about 7 Mio tons annually (see figure 1; Soyez, 2010).

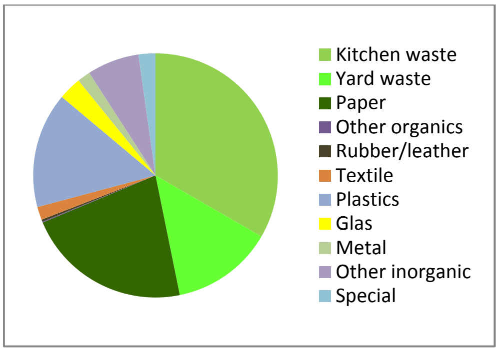

FIGURE 1 Waste composition (%)

This also means that resources of organic and inorganic matter are wasted instead of their usage. Since the economy of the country is by 75% dominated by agriculture it is obvious that production of organic fertilizers such as compost could improve the humus status of the soil and substitute mineral fertilizers and therefore benefit the nation significantly. Making use of the organics therefore combines climate mitigation with economic and ecological effects.

As a result of such considerations the so-called Organic Agriculture Act of 2010 was issued by the Philippines Parliament which intends to promote, propagate, develop further and implement the practice of organic agriculture (RA, 2010).

Within the Act organic fertilizers such as compost produced by farm operations and others are in the focus as part of soil fertility management and its production by means of recycling wastes of plant and animal origin in order to return nutrients to the land, thus minimizing the use of non renewable resources.

In summary following aspects of fertilizer production, using organic waste components, also including MSW, are approached by this Act:

- Organic fertilizers are considered an important means of organic agriculture amongst which compost is an example, and nationwide use and processing are main topics of practical implementation of the Act.
- Waste of plant and animal origin are to be recycled to return nutrients to the land as a measure of reduced use of non renewable resources and thus, enhancement of re-source efficiency and climate impact reduction.
- A list of biodegradable waste components accepted for compost and/or organic fertilizer production is formulated but not limited to given examples (see table 1).

TABLE 1 Bio-waste as defined by Organic Agriculture Act

Source	Specification
Kitchen/household	Food leftovers, vegetables and fruit peelings and trims, fish/fowl cleanings, seeds, bones, soft paper used as food wraps
Yard or garden	Leaves, grass, weeds and twigs
Market	Wilted, decayed or rotten vegetables and fruits, fish/fowl clean-ings, bones
Farm	Grass clippings, dead or decayed plants, leaves, fruits, vegetables, branches, twigs and the like

- Community garbage and waste which are components of MSW, as well as industrial waste, are both seen raw materials for organic fertilizer production and therefore collection and dispose of such material to be organized in such a way that it is usable under the high quality constraints for organic agriculture.

- To prevent that waste is only segregated at dumpsites instead of segregation at source, recycling and composting and termination of burning practice at household level is requested so that production of compost for organic fertilizer can be implemented.
- Research and development as well as education and capacity building in the field of organic fertilizer production including compost is high ranking task and to be executed on all levels including university schedules.
- A national certification of qualities of organic fertilizers production is demanded and its international recognition considered necessary. Therefore quality standards to be implemented following international standards of organic fertilizers including compost qualities have to be applied as adapted to conditions in The Philippines.
- A network of organic fertilizer production related bodies to be implemented amongst which a standardization body which rules product qualities for improved marketing of such organic fertilizers as well as marketing of agricultural products thereof should especially be considered.

Relevant standardization of the Act is after Philippine National Standard (PNS, 2008). It was prepared in 2008 which means before the Act came into power but already covers a variety of items demanded by the Act. It was formulated with respect to organic fertilizer production only based on “pure” agricultural residues though this is not definitely stated and only demands “the original materials (to be) biodegradable”. The standard involves a comprehensive list of such quality parameters which are typically used in organic matter characterization. It seems that concrete values for these parameters are specified after the conditions in The Philippines. In several cases they differ from internationally accepted values. Quality management and analytical methods are missing.

1.2 Research objectives

The objective of this study is to evaluate in which way the standards and procedures formulated after Organic Agricultural Act cover the regulatory needs for good compost production in case MSW components are used as substrates, and if, would it still correspond with internationally proven quality assessment systems for composting such as the ECN guideline? This comparison will concentrate on the most relevant topics especially the choice of substrates, applied process as well as quality management with respect to hygienic and its certification.

2. METHODOLOGY

International compost regulations for usage of MSW components, e.g. from European Composting Network Association (ECN), will be characterised, and afterwards compared with the regulation in power in the Philippines. Deficits will be clarified and measures proposed to enhance the country's regulation to comply with international standards.

3. FEATURES OF QUALITY ASSESSMENT SYSTEMS

A quality assessment system should comprise a number of features characterizing quality assurance needs at several levels. Following, the regulation proposed by ECN (Siebert, 2010) for the proposed European composting standard is used as an example. It involves:

- Establishment of a comparative list of waste materials allowed for the production of compost (“Positive list”).
- Regular assessment of production facilities by the national Quality Assurance Organization.
- Regular sample taking and analysis of the final product considering relevant quality parameters through independent, acknowledged laboratories, and additionally, the evaluation of the results by the national Quality Assurance Organization.
- Declaration of the product by the national Quality Assurance Organization including information about the quality properties of the product, legal requirements, the necessary compost declaration and information about use and application rates according to good practice.
- Awarding of a Quality Label to composting plants by the national Quality Assurance Organizations.

For practical monitoring ECN proposed a process control model using the Critical Control Point (CCP) which consists of seven CCPs.

CCP1 refers to the quality of the input material and involves the acceptance or rejection of the waste and the separation of contaminants. This is based on the fact that the quality of the input material is decisive in production of organic fertilizers and soil improvers: High quality value of compost can only result if proper input materials were used. The process itself cannot improve the material. This fact is obvious especially in case of heavy metals and similar substances which will become part of the product.

Therefore analytical control of the input material and documentation of the values after a certain schema are both indispensible and certain inputs which do not fit into the quality schema have to be sorted out and must not be used as composting substrate. Thus, a most important measure of that process step is to separate and reject unsuited input materials especially in case of inputs used from MSW. Only source separated waste materials be used as input. Mixing with other components of MSW will negatively influence the quality and will result in an useless product meaning all efforts used for production becomes useless. Mixing is therefore not an authorized measure. In case of product residues from industrial or agricultural processes the quality of such substances has generally to be tested before qualifying it an organic fertilizer production input. As a result of such tests a “positive list” of substances can be established. Such a list was already elaborated and is under permanent control including its extension to new potential materials. On EU level it contains more than 100 materials (Barth, 2008).

For proper composting it is necessary to obtain mixtures of different input materials so that the moisture content, the structure, as well as Carbon to Nitrogen ratio at process start are in optimum range. In practice however, only certain materials may be available und mixing impossible at a time. In such a case the storage of the material must be considered and storage capacity made available.

In summary: Only source separated organics must become part of the composting for high quality organic fertilizer, and sewage sludge, mixed municipal waste, (large amounts of) paper, natural textiles and processed wood to be excluded. If this is not considered by no means high quality compost for use in organic farming especially for human crops will result. Such kind of treated organic material is not to be named “compost” or “organic fertilizer” but as “rotting product”. It could find its way into other applications such as for oxidation layers of landfills if appropriate.

CCP2 refers to pre-treatment and involves all process steps which aim to best composting conditions, such as shredding, screening, mixing, conditioning or homogenization.

CCP3 refers to equipment set up and the type of the equipment and its specific layout to be defined. In case of windrow composting it involves the shape and dimension of the heap, and other technological details which are of importance for characterization of the product quality.

CCP4 refers to the following decomposition and maturation which is supported by measures such as regular turning, aeration, and watering for good process condition of microbial activities. This is to be documented daily in an operational diary.

CCP5 refers to hygienisation which means reduction of human, animal and plant pathogens. It is well established that the temperature profile during processing is a suitable criteria. In case of a closed system 3 days at 60°C are suitable. Not so for open windrow systems: the temperature of the material shall exceed 55°C over a period of 10 days or, as an equivalent, a period of 3 days with temperatures of 65°C or more. Each portion of the windrow must undergo this temperature profile including peripheral areas of the heap. Therefore mixing of the material by its turning is essential. Temperature has to be measured and results documented.

CCP6 refers to the degree of maturation which includes the formation of humus-like substances. Product of composting may be a fresh or a finished (“matured”) compost, each for special applications. Determination of the maturation status is defined after the maximum temperature level measured in a standardized test (in a Dewar flask). In case of mature compost temperature will not exceed 30°C.

CCP7 refers to refining and storage. Product is screened and product type classified after intensive quality control and monitoring. Compost quality regulation comprise the methods of analyses and the concrete limits which define the “quality” of compost as the marketable end product. For analysis, standard methods have to be applied. It is recommended that samples are taken by 100% by independent institutions, or up to 50% by the plant manager. The analyses must be carried out by an accredited laboratory. Analyses frequency which is the number of analyses per year depends on the amount of input and is calculated by dividing the amount by 10,000 (tons) plus 1 limited to 12. That means 4 analysis in case of 30,000 t input annually.

Compost quality is decisive for marketing under the aspects of its use as soil improver or fertilizer (“Valuable Criteria”) as well as how it affects people and environment (“Precautionary environmental criteria”). These two aspects are presented in figure 2.

Compost value defining criteria can be grouped into four categories which are i) soil improvement by organic matter and lime; ii) fertilizing by their content of Nitrogen, Phosphorus, Potassium, and Magnesium; iii) material properties such as maximum particle size, bulk density, dry matter content, salinity and pH value and iv) biological parameters such as aerobic biological activity and plant response. Precautionary parameters include the ingredients which are not desired. These components only can be reduced by input control and the usage of source separated material, together with an optimized process management.

Good hygienic standard; low content of heavy metals and impurities like plastics, metals and glass; germinable seeds and plant parts are of special importance. Sanitization will be proven if no Salmonellae can be detected (in a 25 g dry matter standard probe). For heavy metals it is to be noticed that Copper and Zinc are also trace elements important for plant growth. Values after ECN proposals are given in Table 2 as compared with the Philippine standard.

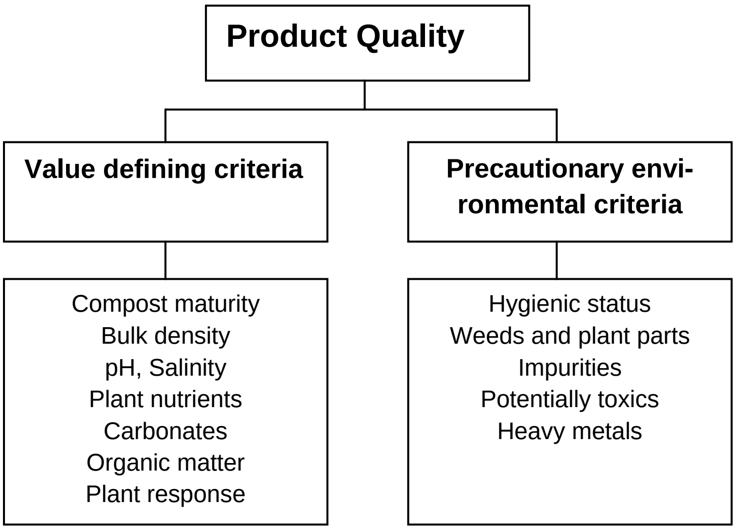

FIGURE 2 Product quality defining features (after Siebert, 2010)

Compost value defining criteria can be grouped into four categories which are i) soil improvement by organic matter and lime; ii) fertilizing by their content of Nitrogen, Phosphorus, Potassium, and Magnesium; iii) material properties such as maximum particle size, bulk density, dry matter content, salinity and pH value and iv) biological parameters such as aerobic biological activity and plant response. Precautionary parameters include the ingredients which are not desired. These components can only be reduced by input control and the usage of source separated material, together with an optimized process management. Following items are of special importance: i) good hygienic standard, ii) low content of heavy metals and impurities like plastics, metals and glass and iii) germinable seeds and plant parts are of special importance. Sanitization will be proven if no Salmonellae can be detected (in a 25 g dry matter standard probe). For heavy metals it is to be noticed that Copper and Zinc are also trace elements important for plant growth. Values after ECN proposals are given in Table 2 as compared with the Philippine standard.

4. CRITICAL REVIEW OF THE COMPOST STANDARD OF THE PHILIPPINES

The details of the quality regulations by the Philippine standard are summarized and compared with ECN proposals, in Table 2, and some of them afterwards commented in more detail.

TABLE 2 Quality parameters demanded by Philippine standard and ECN proposals

Quality parameter	Demand after ECN proposals	Demand after Philippine standard
Type and quality of input	Positive list of input material including source separated biowaste from MSW	Biodegradable material in general
Hygiene standard	For used model of technology of composting, hygiene effectiveness has been generally proven after standardized by authority approved methods. General testing of hygiene hazards in models of facilities.	No such type of regulation
	Documentation of temperature profile during composting (10 days >55°C or 3 days >65°C) in open windrow systems	Not included
	Maximum two germinating seeds per liter product	Included but not yet defined

	No Salmonella detectable in 25 g d.m.	Salmonella: 0; Infective parasites: 0; Faecal streptococci and total coliforms, each: <500 cfu/g
Foreign matter	Maximum 0.5% mass foreign matter with dimension >2 mm detectable in dry substance. Matter <2 mm not considered	0; foreign materials to be totally removed
	If amount >0,1 Mass-% a specified test using the total area of foreign matter applied; largest dimension of particles measured after their area needed. Criteria: Total area <25cm^2/ l fresh substance	
Plant compatibility	No phytotoxic substances present	Not ruled
	No Nitrogen fixing present	
Maturation level	IV or V	Not clearly ruled
Moisture content	Loose compost: max 45% mass	Maximum 30% mass
	Wrapped compost: max 35% mass	
	Special values for organic fertilizers having more than 40% incineration loss	
Organic	Minimum 15% in dry mass	Minimum 20% in dry mass
Heavy metals	Lead: 150 (EU:130)	750-500
	Cadmium: 1.5 (1.3)	5
	Chromium: 100 (60)	150
	Mercury: 1.0 (0.45)	5
	Nickel: 50 (40)	50
	As: not ruled	5
Micronutrients	Copper: 100 (200)	300
	Zinc: 400 (600)	50

Comments on some of the specifications are as follows:

- The number of analysis is not ruled, and the qualification of test laboratories not defined, which both are but important pre-conditions for a control system of compost quality and its acceptance in agriculture and gardening. With reference to the input material only a rough list is presented (see table 1) which has to be specified and extended after the situation prevailing locally or nationally.
- Maturation as a decisive factor in quality assessment and application should be defined after the Dewar test resulting in Maturation stages of I to V with IV and V characterizing mature compost. The statement "an organic fertilizer must be substantially decomposed" is not sufficient.
- "C:N ratio must not be higher than 15:1": This seems not decisive. At process start a C:N ratio of 25-30:1 has to be established through clever mixing of substrates for good composting effects, and in such a case a proper compost will result.
- “The temperature of product must not be higher than 30°C”: Definition of a temperature will only be useful if the maturation stage of the organic fertilizer was in question
- “Organic fertilizer must be free of pathogens”: The Standard defines levels for fecal streptococci and total coliforms (both <500 colony forming units, cfu) which is not “free” of pathogens. A test procedure including test organisms such as *Salmonella senftenbergii* is to be defined and standard test conditions formulated. Such type of test is but not practicable for everyday process control especially in small plants. Proof of temperature level exceeding 65°C over 3 days or 10 days 55°C (in open windrow systems) or 3 days 60°C in closed systems is sufficient and easily applicable in small plants also. Temperature profiles must be recorded for evidence.
- “Allowable levels of heavy metals in organic matter”: Values given in table 3 are between 1…5 times the internationally approved levels and seem problematic especially in case of lead and mercury.
- “Plastics, aluminum wrappers, stones and other inert material must be totally removed from the product”: Best control of such substances is for the input material. In case of agricultural waste as input material small soil particles

such as stones are unavoidable and belong to the natural system. Particles smaller than 2 mm cannot be avoided and do not significantly influence the application of the organic fertilizer.

- After international experiences foreign matter with dimension larger than 2 mm is tolerable up to 0.5 Mass%. A specific test procedure is available (see table 2). For substrate components even higher values are acceptable.
- "Material, organic fertilizer weighing more than 5 kg must be packed in woven propylene sacks etc.": Packing in closed containers or sacks is only advisable in case of matured products since not matured organic fertilizers tend to emit gas. In case of large scale application of compost in organic agriculture direct transport from the compost facility to the farm will be practiced in which case packaging is useless and would result in extra costs, non renewable resources consumption and in littering which all should be prevented. Selling of organic fertilizer amounts of less than 5 kg seems not useful and should not be considered,

5. CONCLUSIONS

In the Philippines a so-called Organic Agriculture Act was enacted as part of the nation's activities to tackle with raising amounts of waste and of depletion of natural resources and soil degradation. Its focus amongst others is on the production of compost using MSW components. Existing ordinances such as the National Standard for composting mostly refers to the use of agricultural residues as composting raw material. To produce high quality composts from organic MSW components a specification of such a standard is needed. As a model the ECN Quality assessment system (ECN-QAS) is available which includes a 7 step Critical Control Point (CCP) approach. Improvement of the standard must preferably refer to good quality raw material and rejection or separation of contaminants at the start of the process chain. Hygienic control by temperature profile recording is preferred instead of direct microbial detection which is too expensive for daily tests. Maturation status should be controlled by the Dewar test and a classification of composts after maturation stage in fresh and mature compost is advisable to support marketing and application. Impurities larger than 2 mm in an amount less that 0,5% are considered acceptable, as well as impurities of less than 2 mm. Acceptable concentration ranges of heavy metals seem in many cases too high as compared with EU regulations.

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Session 12

(8) THE CIVILISATION BIOREFINERY- INVENTORIES FOR EFFICIENT UTILIZATION OF LOCAL WASTE AND WASTE WATER BASED BIORESSOURCES FOR MATERIAL AND ENERGY GENERATION

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EXECUTIVE SUMMARY

Background: Since fossil resources are limited, more and more focus is laid on the utilisation of organics for energy generation. But these bioresources are also valuable for food, feed and material production. Biorefineries are complex and integrated systems of processes and facilities with the purpose to transform primary bioresources into a multitude of energetic and material products. They are expected to be the step forward into a bio-based economy. Civilisation biorefineries expand this goal by the efficient utilization of local resources which are generated as secondary or tertiary bioresources in form of waste, waste water or residues e.g. of landscape care.

Objectives/Methods/Techniques: Civilisation biorefineries contain several parts – inventory, collection, transport of the local bioresources, their conversion via utilization cascades in a network of centralized and decentralized units into various products and the product utilization. These parts are introduced in a general way. Ways to reach a civilization biorefinery were studied more detailed on examples for the part of the inventory. Scientific basics to available regional waste and waste water based bioresources were investigated on the example of the District Hamburg-Bergedorf located in the North of Germany (120,000 inhabitants, area of 155 km²). In the BERBION-project 14 partners develop an integrated approach for the complete utilization of all municipal biogenic resources for the selected district Hamburg-Bergedorf. One major part focuses on basic scientific questions regarding inventory studies; therefore studies were carried out by a multitude of methods. A practice relevant inventory was carried out for toilet water as co-substrates for another example – Hamburgs city quarter Jenfelder Au (1,800 inhabitants, area of 0.35 km^2). In the KREIS-project, the new city district shall be developed using modern technologies for covering the regional energy demand. A first bioresource inventory for the Jenfelder Au was done using general information. In a second step it was made more accurate using the BERBION-findings. The biogas, electricity and heat potential were judged by basic literature data, the BERBION-findings and specific practical investigations. Although several utilization pathways for the bioresources are possible in a civilization biorefinery, the focus is laid in this paper on transformation into biogas by anaerobic digestion. Using anaerobic fermentation process to biogas as central conversion process, the interfaces of a civilisation biorefinery were shown on the example of the Jenfelder Au. They include e.g. new collection systems, substrate-specific pre-treatments, energy conversion and utilization steps and extension of the cascades to material products.

Results/ Discussion/Conclusions: The bioresources from the 2.5 half-year BERBION-inventory were distinguished in solid and liquid bioresources from different sources (kitchen, garden waste, tree clippings, woody, herbaceous materials, mixed green, grass, leaf, horse manure, food residues from restaurants and companies, waste water, residual fat, special fractions; in total 84,000 Mg/a solids; 560,000 m³/a liquids for the district Bergedorf). Quantitatively most important from the solid sector were the green waste fractions and from the liquid the toilet wastes. For utilization of the substrates for anaerobic treatment following criteria are most important: 1) type and quality, 2) amount and availability over the year, 3) the origin of the bioresoure; 4) interfaces. To simplify future inventories, key data and inventory guidelines were elaborated. A first and second "quick" inventory for the Jenfelder Au resulted in a suitable estimation of theoretically available bioresources and their energetic potential on seasonal basis for use in an anaerobic pre-treatment facility with biogas as a product. The considered bioresources were: 1) toilet waste water, 2) lawn cuttings of public and of private areas, 3) kitchen waste from households, 4) fat separator residues, 5) fruit waste residues. Other fractions are available as well and have to be quantified in 3rd inventory step. Also the interfaces to other technical processes in the neighborhood such as geothermic and photovoltaic systems have to be elaborated in the next step to allow facility planning. The local available bioresources can significantly contribute to the local energy mix.

1 INTRODUCTION

Since fossil resources are limited, more and more focus is laid on the utilisation of organics for energy generation. These resources are also valuable for food, feed and material production, e.g. in pulp, paper and chemical industry. In general bioresources can be categorized as follows: 1st generation includes agricultural plants such as feeding beet, wheat, rye, maize (grain, whole plant, silage), 2nd generation are lignocellulosic primary resources such as wood and other linocellulosic plants, 3rd are marine resources such as micro- and macroalgae. But it exist also another category with secondary and tertiary bioresources - the urban wastes and waste waters such as mixed municipal waste, source separated organic waste, agricultural waste, green lignocellulosic waste, toilette waters.

The 1st to 3rd generation substrates are considered as input of biorefineries. Biorefineries are complex and integrated systems of processes and facilities which are supposed to transform primary bioresources into a multitude of energetic and material products. Civilisation biorefineries expand this goal by the efficient utilization of locally available secondary or tertiary bioresources. The civilisation biorefinery is still a vision today, but examples exist, which demonstrate their potential & feasibility.

The goal of a civilization biorefinery is defined by Körner (2011) as system for most complete and efficient material and energy utilization of secondary and tertiary urban bioresources. The approach is holistic and integrative. Civilization biorefineries contain 3 major parts connected via logistic (Figure 1). These are the aspects of inventory and collection of the local bioresources, their conversion via utilization cascades in a network of centralized and decentralized units into various energetic and material products as well the aspects of the utilization of the products in the region. Only high-value-added products shall be a topic of sub-regional exports.

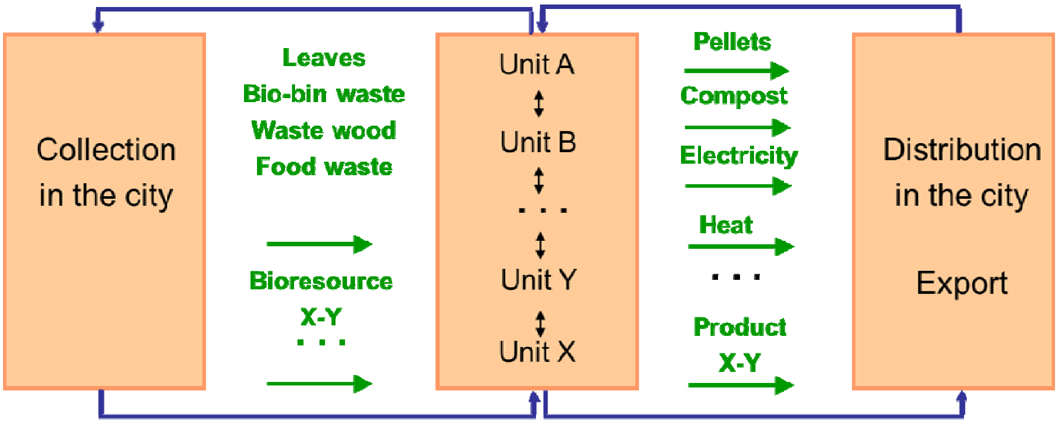

FIGURE 1: Principal Set-up of a civilisation biorefinery (Körner, 2011)

The basic to reach the vision of a civilization biorefinery is a profound knowledge on regional available bioresources. The goal of this paper was to elaborated basic procedures for inventories.

2 METHODOLOGY

Two case studies were chosen and studied in different ways in order to elaborate the basics to carry out regional bioresource inventories in an easy and quick, but comprehensible and situation adapted way.

Hamburg's district Bergedorf:

It is located in the North of Germany with approximately 120,000 inhabitants and an area of 155 km². It consists of a multitude of different area structures including rural, urban and commercial areas, making it suitable model case. In the BERBION-project 14 partners develop an integrated approach for the utilization of municipal biogenic resources for the district. One major part focuses on basic scientific questions regarding inventory studies. Inventory data were elaborated by a multitude of methods (see also Adwiraah et al. 2012):

a) Data provided by companies

b) Information and materials provided by public bodies

c) Literature data

d) Information aroused by questionnaires

e) Information gained by personal interviews
f) Data from evaluation of Aerial views
g) Data from own waste sorting and waste analysis

Hamburg's Jenfelder Au neighbourhood:

It will be a new residential area of 0.35 km^2 in construction for 1,800 inhabitants, located at the eastern part of the city of Hamburg. The homes will be terraced and town houses. Approximately 20 % of the area will be public green areas and private gardens. Other 20 % are assigned for neighbourhood business. A combination of renewable energy and innovative wastewater systems (Hamburg Water Cycle®, HWC) will be applied. The HWC contains separate pathways for grey- and blackwater collection and treatment. In the KREIS-project (KREIS, 2012), following inventory methods were used so far:

a) "Quick-and-dirty" inventory
b) "Rapid decision" inventory
c) Interface inventory

3 RESULTS AND DISCUSSION

The inventory results documented here focused on selected aspects only. Important elaborated data are shown in Tables 1 and 2.

- *The yearly amounts:* For any kind of planning the knowledge on the theoretical potential of the resource is the first parameter to know. However, these potential may not be completely usable, since not all resources can be collected or losses occur.
- *The provision time:* Some regional bioresources are generated more or less constantly over the year; these were the bioresources which can be related to the number of inhabitants. Others were strongly season depending, these were the bioresources on plant matter basis, which were related to the area of growing.
- *The composition:* Some basic qualitative types of regional bioresources were distinguished: 1) Liquid, moist, dry; 2) rich, poor in inorganics; 3) lignocellulosic, herbaceous, rich in carbohydrates, proteins or fats; digested materials. Each kind is different suitable for the different utilization options.
- *The generation location:* Also if a bioresource has a similar composition it may be suitable for different utilization pathways, since collection systems are location focused, e.g. collections from households, form industry, from public or from private areas of different types and collection as well as conversion steps often are closely related.
- *The interfaces:* For the product generation from a bioresource commonly a multitude of options exist. For deciding about the most suitable option for the region a lot of factors have to be considered; e.g. the political frame the existing infrastructure, the involved player and their intentions.

Table 1: German estimates on urban bioresource generation data – totals and seasons approximate share of the overall generation

Bioresource-Fraction	**Amount Min-Max**		**Winter Dec-Feb**	**Spring Mar-Mai**	**Summer Jun-Aug**	**Autumn Sep-Nov**
Black water from vacuum toilets	8.3[3]	L FM/I&day	25 %	25 %	25 %	25 %
Household kitchen waste	1.3-1.6[1]	kg FM/I&Week	25 %	25 %	25 %	25 %
Lawn cuttings	0.6-3.0[1]	kg FM/m²&a				
from public areas[2]	1.7[2]	kg FM/m²&a	0 %	33 %	39 %	27 %
private areas[1]	0.9-1.2[1]	kg FM/m²&a	0 %	29 %	46 %	25 %

1 Source: BERBION-project; based on literature and own measurements
2 Source: Bocksch (2006)
3 Source: Wendland (2008)
I-Inhabitant, FM-Fresh matter

Table 2: Urban bioresource composition data and their methane formation potential by anaerobic digestion

Bioresource-Fraction	Water Content (% FM)	Organic matter content (% FM)	Ash Content (% FM)	Methane Potential m³/Mg FM
Black water	99.4[1]	0.7[1]	negligible	1,7 [1]
Kitchen waste	50[2]	45[2]	5[2]	57[4]
Lawn cuttings from public areas	70[2]	26[2]	4[2]	100[4]
Lawn cuttings from private gardens	68[2]	29[2]	3[2]	100[4]
Wood cuttings from public areas	40[2]	53[2]	7[2]	-
Wood cuttings from private areas	40[2]	53[2]	7[2]	-
Mixed green from public areas	44[2]	46[2]	10[2]	43[4]
Mixed green from private areas	44[2]	44[2]	12[2]	43[4]
Fat separator residues	90[3]	4[3]	negligible	15[4]

1 Source: Wendland (2008)

2 Source: BERBION-project; values chosen for calculations; based on literature and own measurements
3 Source: Deegener (2012)

4 Source: BiomasseV (2012); declared as food residues, grass resp. green cuttings from garden and park care

3.1 Hamburg's District Bergedorf

The inventory works in the BERBION-project were carried out over a period of 2.5 years and are now in the evaluation phase. Various inventory methods were applied and delivered the following data types:
a) Data provided by companies: The project partners from the communal waste and waste water companies delivered data from their own collections regarding residual waste, biobin waste from communal collection, green waste delivered in recycling centres, public street leave litter collection resp. total communal waste water as well as waste water amounts delivered at a tipping point; all on time-depending basis. Some less detailed data were provided by other companies, which were not included in the project e.g. from local acting green waste and food collectors.
b) Information and materials provided by public bodies: From the district administration Bergedorf the amounts of various green waste types collected from public areas were gained e.g. regarding floating refuse from water bodies. From public offices like district Bergedorfs registry office, Hamburgs state office on geoinformation and topographical survey, regional and federal offices on statistics information regarding inhabitant numbers, DSGK- and ALKIS-maps (see chapter f), and general waste collection data were received. Furthermore Hamburg's authority in city planning and environment information – e.g. their biowaste study 2009 as well as card materials from biotopes and data about fat separator residues.
c) *Literature data:* A detailed literature study was carried out using very different sources - scientific literature, statistical materials, region-related or company related studies, waste-sorting studies, surveys from lobby and public organisations and more. The information were very numerous, complex and broad. They are general quite difficult to summarize.
d) *Information aroused by questionnaires:* Questionnaires were elaborated for different situations and answers collected by different means. 164 questionnaires were filled out directly by private deliverers at a recycling centre; they answered questions regarding their green and garden waste disposal and utilization practice. 99 questionnaires were filled out by inhabitants of two different types of residential areas; they received them per post. It was asked for the garden and kitchen waste disposal and utilization practice of the inhabitants. Further questionnaires were distributed by post, by direct interviews or both - 24 questionnaires were received from farmers, 18 from tree nurseries and gardening companies and 30 from restaurants.
e) *Information gained by personal interviews:* Key persons from the region were intervied regarding specific questions focused on their company or organisation.
Examples are persons from a local biomass heating company, from the forestry office of district, from a nearby fruit processing company as well as from Hamburg's nature conservation union.

f) Data from evaluation of aerial views: The ALKIS provides the actual land use data in a codified way as a combination of area type and function attributes (e.g. sport, leisure and recreation areas with attributes for green areas and for parks; agricultural areas with attributes as farmland, green area, garden areas and tree nursery). DSGK is the habitat cadastre. It gives more detailed information about the green areas, and enables the assignment e.g. of different growth rates for grass cuttings. Otho pictures from the Hamburg's state office on geoinformation and topographical survey were evaluated by area measurements and counting. The results were data about garden structures, public and private lawn areas, bush land and trees (see also Adwiraah et al. 2012).
g) *Data from own waste sorting and waste analysis:* Waste sorting analyses were carried out at two different types of residential areas for households with residual waste bin only as well as for households with biowaste bin together with residual waste bin. Residual waste bins (134) were sorted into 15 fractions: food processing waste, food leftovers, original food packings, opened food packings, unpacked original food, waste wood, paper & cardboard potted plants with root, herbaceous garden waste, woody garden waste, plastics, wood organics < 40 mm, mixed organics < 40 mm, , other waste < 40 mm and other waste. The biobins (116) waste was sorted into 26 fractions. The first 7 fractions listed for the residual bin were also sorted for the biobin, but the organics were distinguished more detailed into following additional fractions: plant residues with roots, softwood hedge clippings, hardwood hedge clippings, branches, trunks, windfall, leaves > and < 40 mm, grass clippings > and < 40 mm, further plant residues without roots, kitchen waste < 40, mixed garden waste < 40, waste wood < 40 mm, mixed organics between 10 and < 40 mm as well as < 10 mm, woody organics < 40 mm, paper and cardboard < 40 mm, other waste < 40 mm. From selected fractions chemical analysis were carried out regarding water content, organic content and ash content. From specific samples furthermore the heating value and a Van Soest component analytics was carried out.

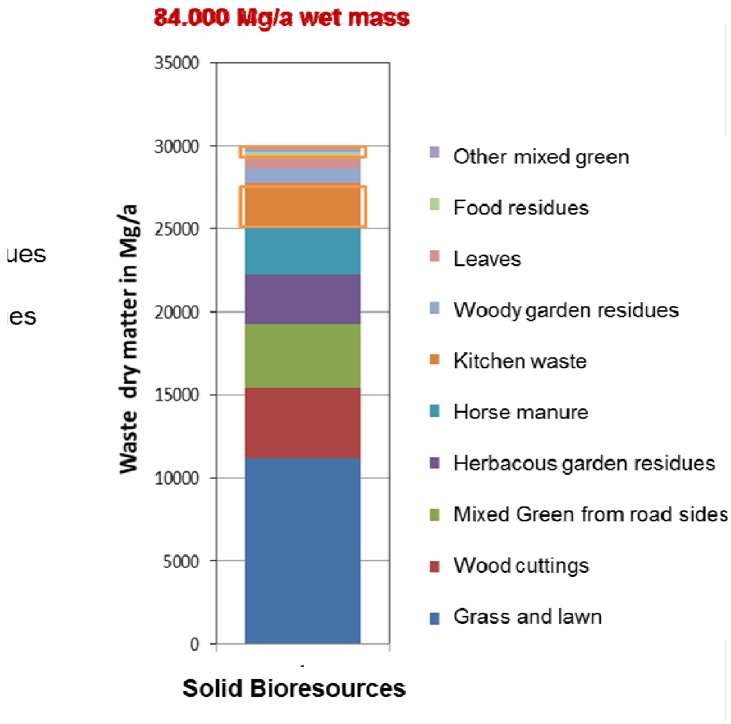

FIGURE 2: Results of the detailed bioresource inventory for the district Bergedorf (Körner, 2011)

Not all of the previously listed inventory methods were used to determine the amounts and their availability over the year. The result was a combination of a lot of methods. The overall inventory results are given in Figure 2. They were primarily distinguished into solid and liquid bioresources secondarily regarding different types. The detailed locations of generation are known as well, but not included here. From the quantitative point of view most important from the solid sector were the green waste fractions and from the liquid the toilet wastes.

3.2 Hamburg's Jenfelder Au neighbourhood

In the KREIS-project the first "quick and dirty" inventory focused on fractions only which are considered as usable for anaerobic digestion, since it is planed to build an anaerobic pre-treatment facility for black water from vacuum-toilets and co-substrates in the neighborhood. Therefore bioresources with a major part on lignocelluloses were excluded, since they are considered as not digestable (Table 2) and kitchen wastes were excluded, since they will not be available on short term because they go to incineration or anaerobic digestion into another facility. After this exclusions, only the major fractions which were elaborated in the BERBION-inventory (Figure 1) were considered – grass and lawn cuttings as well as private waste waters (but only the black water fraction from the toilets; since for these a separate collection system will be installed). The results are shown in Figure 2. This inventory was carried out with in a time frame of days. Only a few basic data from easy available sources were chosen:

- Number of potential inhabitants (1800) and size of green areas (10.000 m² public lawn; 50.000 m² private lawn): from the "Masterplan Jenfelder Au" (KREIS, 2012; Bezirksamt Wandsbek, 2012a, b)
- Black water generation in vacuum toilettes (8,3 L/I & a): from a pHD-thesis (Wendland, 2008)
- Lawn cutting generation rates (1.7 kg/m² & a for public lawn, 1.4 kg/m² for private lawn): from webpage of a lobby organization (Boksch, 2006)
- Biogas potentials: Biogas calculator from internet; missing values from a diploma thesis (KTBL, 2012; Westphal 2010)

This first inventory gives an impression of the basically available substrates and their efficiency. For the Jenfelder Au it means that these main substrates deliver not enough biogas. The planning's for the neighborhood foreseen a concept were about 60 % of the heat demand of the neighborhood is delivered by a combination of the biogas facility, geothermic, photovoltaic systems and eventually a biomass heating system. For that reason, in the 2nd inventory additional bioresources for use in the anaerobic fermenter were included. Beside total amounts also the delivery according the year was considered. Furthermore, the accuracy of data from the 1st inventory was increased.

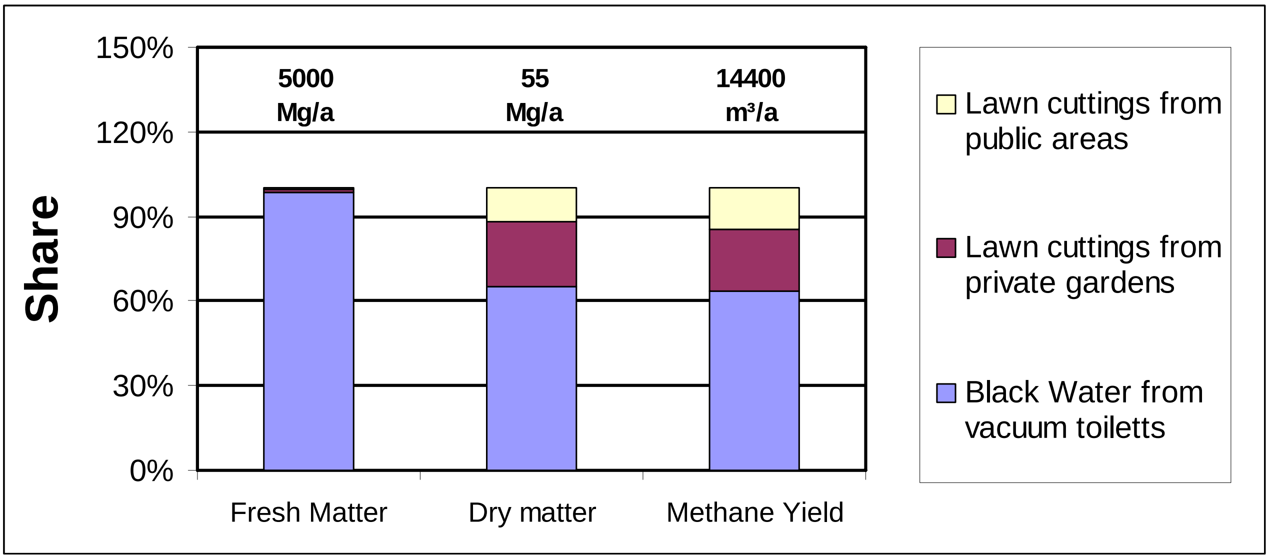

FIGURE 3: Results of the "Quick-and-dirty" Inventory of the district Jenfelder Au (1st inventory)

For this "rapid decision" inventory, the summarized findings from the BERBION-project and theoretic biogas generation values from a law (BiomasseV) were included. Furthermore it was considered, that people "delivered" 40 % of their black water not in their homes. The data from Figure 3 are now presumable more accurate. The total calculated methane generation potential changed from 14000 m³/a to 12000 m³/a). But the overall change was not so significant, since for some substrates higher, for the other lower corrected estimates were calculated.

Since the 1st inventory showed clearly, that available bioresources are by far not enough to reach the goal of heat delivery, other bioresources were considered. One option is the utilization of external bioresources. Fat separator residues from all Hamburg and surrounding could be provided by the public waste water company; 10 Mg d or more would be possible.

Furthermore, in the close surrounding of the Jenfelder Au a fruit processing company is located, which delivers their residues actually to more fare away facilities. It was assumed, that 30 % of these residues could made available for the neighborhood anaerobic treatment facility. It was assumed too, that all of the kitchen waste of the new houses could be used.

Under consideration of Table 1, the generation of black water, kitchen waste and also the deliveries from the fruit company should be more or less continuous over the period of one year. But the provision of lawn cuttings is season depending. Since it could be favorable, that the anaerobic treatment facility works with more are less constant output on biogas, the seasonal differences were equalized by variable delivery rates of the fat separator residues. The results are shown in Figure 3. With these assumptions the methane generation rates of the anaerobic treatment facility would be in a suitable range.

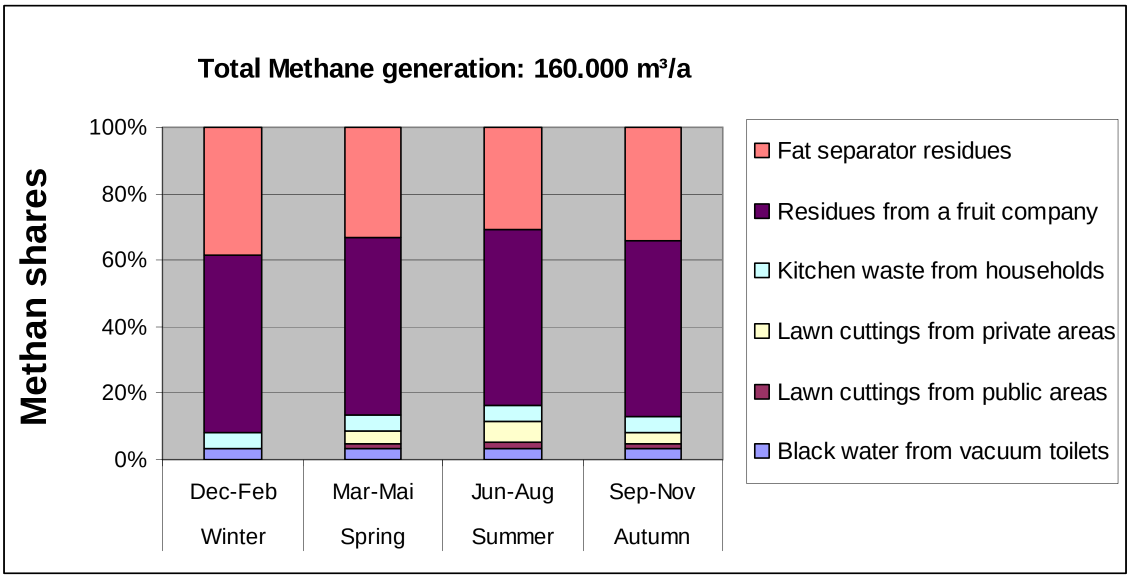

FIGURE 4: Results of the “Rapid application” Inventory of the district Jenfelder Au (2nd inventory)

Some of the bioresources from Figure 4 are actually not available, since they are going somewhere else for treatment, but it would make sense to re-direct from the technical point of view. Some of the bioresources are from external areas and considering regionalism it would be preferable to include much as possible internally available substrates. For that reasons further types of bioresources which could eventually made available in the Jenfelder Au and in the close surrounding are listed in the following:

Smaller fraction inside the Jenfelder Au: e.g. further green waste fractions, restaurant waste, commercial waste, residues from grey water cleaning, kitchen waste from existing households, residues from waterways and pond cleaning.

Larger fraction in the close sourrounding of the Jenfelder Au: lawn cuttings, kitchen waste, eventually other commercial wastes.

They shall be estimated quantitatively in a 3rd inventory step. In a 3rd inventory step also the frame conditions have to be clarified, under which the additional substrates from figure 3 (fat separator residues, kitchen wastes, fruit residues) could be included on the short or the long term or if they have to be excluded completely. Furthermore the interfaces have to be designed more concrete. The most important interfaces are so far the following: energy deliveries from the photovoltaic and geothermic systems, necessity of an additional biomass heating plant, technical connections with the combined heat and power plant; treatment options for transformations of digestates into usable products. Based on all of this information more clear goal definitions are possible and with it the fixing of methane delivery goals. Based on this, planning’s for construction on the anaerobic pre-treatment facility can be carried out.

4 CONCLUSIONS

Following major conclusions could be drawn from the case studies regarding inventories of regional waste and waste water based bioresources for efficient application for material and energy generation in civilisation biorefineries:

- A multitude of bioresources from different locations and different qualities exist in one region. As single amount they are rather small, but accumulated **they could contribute significantly as a source for material and energy delivery**.
- An easy, quick and comprehensive inventory could be carried out in all regions. Some general data has to be known. The basics were elaborated in the BERBION-project. Other data are region-specific and a recherché has to be carried out in the region. To allows country wide regional planning's the following strategy is suggested:
- Suitable estimates, valid within a country should be provided by statistical offices. Following parameters are essential: inhabitant related generation rates for kitchen and toilet waste and waste waters; area related estimates for generation rates of different green waste fractions (lawn, woody residues, mixed green; each separately for gardens and public areas), average composition estimates of the fractions.
- Regional basic data should be available by regional public offices. Geo-information on the number of inhabitants and on area structures. The specific amounts and types of bioresources produced, collected, treated, utilized on the various locations within a city should be raised by the authorities on yearly basis to allow self-formation of efficient local utilization networks.

5 ACKNOWLEDGEMENTS

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(29) BRAZILIAN'S BEEF CATTLE FEEDLOT MANURE MANAGEMENT: A COUNTRY REPORT

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EXECUTIVE SUMMARY

Brazil has been the largest beef exporter in the world with almost 10% of its 40 million slaughters in 2010 coming from feedlots. The Brazilian beef feedlot industry has grown 60% in the last 8 years driven by increase in the external market demand for fed cattle. High concentration of manure is an inevitable by-product in feedlots that must be managed as a resource rather than waste in order to avoid environmental pollution. Thus, knowledge on manure management is necessary for properly advices and estimates. However no inventory of beef cattle feedlot manure management is available for Brazil. In order to fill this gap, a survey was carried out covering 73 feedlots in seven Brazilian states (São Paulo, Goiás, Mato Grosso, Mato Grosso do Sul, Paraná, Minas Gerais and Rondônia). The feeders were asked about characteristics of the herd, the diet offered to the animals and the handling of manure since its deposition in the pens till its final disposal in the year of 2010. This survey concerned about 1 million animals, representing 30% of beef cattle confined in Brazil in this year. Results indicated that *Bos Indicus* breed (Zebu) steers with mean age of 24 months have predominantly been fed in Brazilian feedlots (80% of responses). Half of these animals remained about 90 days in pens with no cover, bare soil on the ground and stoned or concreted in feed bunk line. Corn was the primary source of grain used in feedlot diets (80%) and the level of concentrate inclusion was around 80% (31%). Soybean meal, cottonseed, soybean hulls and citrus pulp were the primaries concentrate co-products used (60%). The most representative manure management practice consisted in manure cleaning from pens at the final of the feed cycle (57.5%) with subsequent disposal in heaps during 2-6 months (47%) and with final application in crops (mainly maize) and pastures (90% both). Slurry, formed mainly by rainwater, was stocked in tanks (86.7%) and also used in agriculture (50%). Part of these crops and pastures biomass productions was directed to cattle feeding and provided at least 50% of the demand for silage to the own feedlot. When using manure as fertilizer, feeders related savings in the purchase of synthetics fertilizers, and they also noticed similar or even higher crop and pastures yields associated with an increase in soil organic matter and structure. However, 80% of the respondents did not carry out chemical analysis of the manure and did not know exactly the amount of manure excreted in the pens as well as the amount applied in the field. Although widely used, lack of information about practices that improve the efficiency of manure handling and utilization were reported by 90% of the respondents, so that only 5% of the feeders practiced composting. Most of them (83.5%) reported having information in relation to generation of energy and bio-fertilizer through biodigesters, but only one single feedlot had already such technology. Through digesters, it can be estimated that the manure from beef cattle feedlots alone have the potential to generate around 0.47 million MWh, i.e.0.12% of all energy consumed in Brazil in 2010. Since the Brazilian government has recently launched a national program (Low Carbon Agriculture) that fosters the development of more sustainable practices in agriculture, this work presents pioneer information from which assessments can be drawn to fit local needs for improvement, while implementing better manure management practices, such as composting and anaerobic digestion, in the growing beef cattle feedlot industry in Brazil.

Key words: Survey, manure handling, livestock, beef production, Brazil

1 INTRODUCTION

Since 2003 Brazil has been one of the largest beef exporter in the world with the largest commercial cattle herd (FAOStat, 2008). In 2010, almost 10% of the 40 million slaughters in Brazil came from feedlots (Anualpec, 2011). Brazilian cattle are fed in feedlots as strategy to maintain a constant beef supply mostly during the dry season when pasture availability is decreased and has grown 60% in the last 8 years, from 1.96 in 2002 to 3.05 million heads in 2010, as the external market demand for fed cattle has increased (Anualpec, 2011; Millen et al., 2009).

High concentration of livestock results in greater local emissions of pollutants from manure during housing, storage and land application (Petersen et al 2007). These pollutants are mainly linked to greenhouse gases and ammonia emissions and eutrophication of water bodies (Chadwick et al 2011). If livestock intensification continues, there is a need for development of technology and strategies to control the associated environmental problems (Petersen et al 2007).

Nevertheless, general solutions are difficult to devise as systems for livestock manure management are extremely diverse. For example, in parts of Europe, recycling on the farm effectively reduces the need for mineral fertilizers whereas in other regions most livestock farms handle the manure as dilute slurry that is stored in lagoons and frequently applied to spray fields. Thus, priorities of farmers will be very different and call for different strategies, requiring that whole manure management chain (from excretion to land application) should be analyzed in order to more sustainable practices be developed and adopted (Petersen et al. 2007).

In this way, Brazilian government created in June 2010, the ABC program (Low Carbon Agriculture), which estimulates the development and adoption of more sustainable agricultural practices combining food production and bioenergy. One of the six basic initiatives is animal manure treatment through anaerobic digesters (AD). Also, manure management has been supported by Brazilian National Waste Management Policy (Política Nacional de Resíduos Sólidos – PNRS) that was approved in August 2010 (Brasil, 2010b), based on the concepts of shared product responsibility, product life cycle and the reverse logistics for several sectors. According to this law, producers and government authorities must prepare plans that provide diagnosis of the situation, scenarios, reduction, reuse and recycling goals and targets for decreasing disposal in landfills.

However, no inventory of beef cattle feedlot manure management and use is available for Brazil, which may slow down the knowledge on the scientific field on the impacts of the manure on the environment and in identification of management practices that lower emissions towards better agricultural practices.

Given the need for information, our objectives were (1) outline an overview of manure handling in feedlot beef cattle in Brazil and, (2) collect and summarize survey data that will facilitate the design of introducing environmental-friendly and assessments practices.

2 METHODOLOGY

2.1 Data Collection

A hundred and twenty commercial feedlots in Brazil were inquired in the survey. These feedlots were accessed with predominant aid of National Association of Brazilian Cattle Feeders (ASSOCON), reaching feedlots in seven Brazilian states. The feedlots were interviewed in 2011 on information from 2010. These commercial feedlots were contacted by e-mail or telephone regarding their interest in participating in the survey, in which 73 agreed to participate. The questions in the survey were divided into: animal, diet and feedlot information; questions about the manure management from excretion on pens to final disposals; questions about the manure field application; and final questions regarding benefits and challenges faced by manure management and use. This survey was conducted using similar methods to those of Vasconcelos and Galyean (2007) which included a Web-based survey tool (http://www.surveymonkey.com). The 73 feedlots who agreed to participate in this survey received preliminary instructions about participating in the survey. The participants were guaranteed anonymity and asked to complete the survey as quickly as possible.

2.2 Data Analyses

The dataset was separated by question and was input in the SAS using IMPORT procedure (SAS, Inst. Inc., Cary, NC). Statistical analyses were performed using MEANS and FREQ procedures as SAS Macro in order to build a parsimonious programming friendly procedure. First of all, parametric answers (e.g. animal's age and adaptation period to the final diet) using MEANS procedure in order to calculate the number of observations, sum, mode, mean, SD, minimum and maximum values and atypical data were analyzed. Sequentially, non-parametric answers (e.g. main grain used in the diet and soil texture) using the FREQ procedure in order to calculate frequency statistics was performed.

3 RESULTS

3.1 General Information

The number of feedlots accessed in this work was 73, which were located in the states of São Paulo (27), Goiás (15), Mato Grosso (14), Mato Grosso do Sul (11), Minas Gerais (4), Paraná (1) e Rondônia (1) (Figure 1). From these feedlots, it was possible to range a database of 831,450 animals, around 30% of the whole beef feedlot industry in Brazil in 2010, according to the suggested data by the most popular Brazilian source of information (ANUALPEC, 2011).

The Nellore breed was the most frequent cattle type, comprehending 80% of the animals ranged in the current research, followed by the crossbred Nellore (22%) and other breeds (5%). About 95% of these animals were males. The typical initial and final body weight were 350 and 493kg and 276 and 390kg for males and females, respectively.

Corn was the primary source of grain in the accessed animal's diet (76%), followed by sorghum (21%) and oat (3%). Corn silage was the primary roughage source (40%), followed by sugarcane bagasse (27%), sorghum silage (12%), grass silage (11%), fresh chopped sugarcane (6%), sugar cane silage (2%), and millet silage (2%). Soybean meal was the primary co-product used in the finishing diets, related by 33 (18.6%) of the feeders, followed by whole cottonseed by 31 (17.5%), citrus pulp pellets by 29 (16.4%), soybean hulls by 26 (14.7%), cottonseed cake by 18 (10.1%) and corn gluten feed by 11 (6.2%) of the participants. Others co-products in proportions small than 2% were also cited. Daily dry matter intake averaged 11.3kg and 8.2kg for males and females, respectively. The level of concentrate (grain plus other concentrate ingredients) included in finishing diets, 38, 18, 12 and 10% of the animals were offered diets with a level ranging from 80 to 90%, 71 to 80%, up to 55% and 56 to 70% of concentrate. The nutrient compositions of the finishing diet related by the feeders averaged 72.7% of total digestible nutrients and 13.4% of crude protein.

Most of these 831.450 animals were kept in farms where either one (24%) or two (50%) finishing diets (cycles) were carried out in 2010. Only 17 and 9% of the animals were fed in places with three cycles and continuous cycles during 2010, respectively. About 40% of the animals were confined during a period from 71 to 86 days, 39% from 87 to 100 days, 18% from 101 to 135 days and only 4% from 55 to 70 days. The average of all the periods is 90.7 days of confinement. Approximately 50% of the animals were fed in feedlots on clayed soil texture, followed by feedlots on medium soil texture (33.8%) and sandy soil texture (16.5%). Most of the animals was confined in pens with ground soil with concrete by the feed bunk line (29%), followed by pens with total area ground soil (25%), totally stoned (24%), stone and concrete by the feed bunk line (10%), ground soil and stones by the feed bunk line (9%). About 85% of the animals were kept in pens with no type of covering (except by the feed bunk line for some).

3.2 Manure management

Fifty four of the respondents (74%) said they did not have an estimate of the amount of manure produced in their feedlots. These feeders represented 61.2% of the animals accessed in this research (Table 1). The other 19 interviewees (26) related an average amount varying from 2.0 to 4.0 kg of dry manure excreted by each animal daily, being in line with an average of 3.2 kg related in literature (ASAE, 2003).

The manure produce, were withdrawn only once right after their respective feeding cycles by 42 of the respondents (57.5 %), 11 (15.1%) feeders said twice per feeding cycle, 5 (6.8%) related every 10 days, 5 (6.8%) cited only at the final of the year of 2010 and 3 (4.2%) said every 20 days. Seven of the respondents (9.6%) related that they did not withdrawn manure from pens and they would not (Table 1).

Of these 66 feedlots that removed the manure from pens, 58 of the respondents (87.9%) reported storing the manure in heaps. Six of the participants reported (9.1%) have composted, one feeder (1.5%) said it had applied directly into pasture, and another one (1.5%) said it was directed to anaerobic digesters (Table 1).

The formation of liquid manure occurred in 37 (50.7%) through water dilution of solid manure (from pens and heaps) through the action of rain and were directed to stabilization ponds in 33 (86.8%) of these farms. Other three feedlots (7.9%) had channels that manure flowed directly to crops and pastures and only one feeder (2.6%) reported they have dumped manure directly into waterway. The single farm (2.6%) that generated liquid manure by cleaning the pens, refers to the feedlot that made use of digesters (Table 1).

The remaining solid manure in piles before taking another destination of 3-6 months was the period most cited by our respondents, 16 (25.0%). Periods of 2-3 months, up to 15 days, between 15 and 30 days, between 6 and 12 months and from 1 to 2 months were reported by 14 (21.9%), 12 (18.8%), 8 (12.5%), 7 (10.9%) and 3 (4.7%) of the respondents. Only 4 participants (6.2%) reported that the waste disposed in heaps remain indefinitely in this way and that would not be used for another purpose (Table 1).

Slurry remained in tanks indefinitely in 15 (44.0%) of 34 feedlots. Feedlots where slurry remained between 6-12 months before taking another destination was the response of nine of the respondents (26.5%), six (17.6%) answered within 30 days and between 1-3 months for 3 (8.8%) Only one feeder (2.9%) reported that it took between 3-6 months (Table 1).

After the period in heaps, 23 of the respondents (38.3%) said they used solid manure as fertilizer in crops only, 20 (33.4%) in crops and pastures, and 11 (18.3%) only in pastures. Only four (5.1%) and 2 (2.5%) of the participants sold and donated these manure, respectively.

All of the 19 respondents who said give another destination to slurry after period in tanks, 4 (21.1%) related used as fertilizer in crops and pastures, crops only by 5 (26.4%), pastures by 2 (10.5%). Eight of the participants (42.0%) did not answer this question.

Corn was the primary crop fertilized with manure, related by 26 of the respondents (32.1%), followed by soybean for 17 (20.9%), sugar cane for 13 (16.0%), sorghum for 9 (11.2%), beans for 5 (6.2%) and coffee for three (3.7%) of the participants. The crops of cotton, rubber, tomatoes, wheat and orange three were also cited in minor proportions. Pastures fertilized with manure belonged to the genus of Brachiaria, Panicum, Pennisetum and Cynodon, reported by 20 (54.1%), 11 (29.7%), 5 (13.5%) and only one (2.7%) of the respondents, respectively.

Thirty-one of the respondents (52.5%) were unaware of the amount of manure that was applied to the field. Of those who knew this amount, 17 of the participants said they applied up to 10 ton per hectare, four between 11 and 30 ton per hectare and only two responded more than 30 ton per hectare. Five (8.5%) did not answer this question. With respect to manure chemical composition, only 10 of the respondents (13.7%) said they had sent samples to specialized laboratories and that manure contained percentages of nitrogen, phosphorus and potassium from 1.1 to 1.5% for each element. Compared to the use of synthetic fertilizers, the yield of these crops fertilized with manure was reported as higher, similar and lower for 32 (54.2%), 15 (25.5%) and 12 (20.3%) of the respondents, respectively.

When asked about the percentage of the demand for silage for the feedlot itself extracted from the production of crops fertilized with manure, 18 of the respondents (30.5%) reported that produce 100%, 16 (27.1%) said around 50% and other 16 (27.1%) answered zero, three (5.1%) responded around 25% and only 1 (1.7%) said they did not known.

Purchase of synthetic fertilizers was the economical benefit more related by the feeders, 54 of the respondents (71,0%). Other 13 feeders (17.2%) answered that increased their incomes through better crop productivity using manure as fertilizer. Only one feedlot said has sold carbon credits by manure management. Six producers did not answered this question.

Increasing of soil organic matter was related by 30 of the respondents (48%) as the main environmentl benefit of using manure as fertilizer. Fourteen participants (22.6%) said that no envirnmental benefits were reached, 11 (17.7%) cited reduce environmental impacts by decriasing synthetic fertilizer use, 7 (13%) related have had increased soil structure and humidity.

The need for information related to the handling, treatment and use of manure still is the main concern of the feeders that were interviewed (87.8%) when questioned about the governmental technical support. Seventeen of the respondents (26.5%) said they did not face difficulties and 6 (8.2%) did not answer to this question. It is important to mention that only one feedlot possessed digestors for manure treatment out of the 61 feeders (83.5%) that mentioned having the knowledge of the possibility for producing energy and high-quality fertilizer through the use of this technology. Furthermore, a hundred percent of the respondents mentioned having interest in programs that aggregate value to the meat through manure management.

4 SUMMARY AND CONCLUSION

The present survey provides an overview of manure management currently applied by farmers in 73 Brazilian feedlots, generated by 831.450 animals, predominantly Nellore cattle, fed with a corn-based high-energy diet. After a period of time, feeders removed this dry manure from the pens that was typically held in a manure stockpile area followed by spreading as an organic fertilizer on agricultural land. This model of manure handling has also been observed in feedlots in countries such as Canada and the U.S., which possesses a higher percentage of cattle fed in feedlots than Brazil (Beauchemin et al., 2010, Kissinger et al., 2007).

That manure use as fertilizer was important input for rural properties in the aggregation of economical benefits, mainly by saving synthetically fertilizers purchase for crops that, most of the times, were destined to the production of silage to be used by the farm itself. In the other hand, lack of information about practices that improve the efficiency of manure handling and utilization were reported by 68.5% of the respondents (87.8% of the animals accessed), what reflected in leaving manure in an extend variation of time in the pens, heaps and tanks, which are not desirable ways of decomposition, and that were predominately applied without any previous treatment or knowledge regarding its chemical characteristics and quantity.

This kind of handling has also been focus of environmental concerns and impact assessments looking at feedlot operating emissions, because over time manure breaks down releasing nutrients to the atmosphere - methane (CH_4), carbon dioxide (CO_2), ammonia (NH_3) and nitrous oxide (N_2O) - and into the ground - potassium (K), phosphorus (P) and nitrate (NO_3^-) run-off (Chadwick et al., 2011; Beauchemin et al., 2010; Gilley et al., 2010). The emission of these substances anthropically might cause local and global environmental damages once they are directly related to the eutrophization of water bodies, contamination of waterways, acid raining and global warming (Chadwick et al., 2011; Cornejo and Wilkie, 2011; Kissinger et al., 2007).

The percentage of these nutrients that is lost during its excretion and management is dependent on several factors, such as: animal type, ratio nutrient concentration, pH, moisture content, temperature, and frequency between manure management stages and events (Mathot et al., 2012). Although, still currently little reported data available on nutrient emissions from beef cattle feedlot manure worldwide. On the U.S. feedlot, Farran et al. (2005) reported manure N losses of 45.1 and 25.2% of the N excreted when pens were cleaned monthly and at the end of a 166-day feeding period (corn-based) of steer calves, respectively. On the other hand, care needs to be taken to ensure that the saved nutrients from pens are not immediately lost in the subsequent manure stockpile/composting process (Hao et al., 2001) and by field application (Chadwich et al., 2011).

Thus, introduction of better manure management practices would avoid in a great extend emissions of pollutants through the management chain, bringing higher nutrients availability to be used as fertilizer in crops. Besides environmental aspects, a conscientious recycling of the excreted nutrients by the beef cattle in feedlots would take to an improvement in this system, once the manure reflects the diet offered to these animals, which represents around 70% of the total cost production in Brazil (Restle & Vaz, 1999).

Composting and anaerobic digester (AD) are two excellent techniques to handle with manure treatment (Kaparaju & Rintala, 2011; Shen et al., 2011). Composting is a low cost technique once is needed a place where manure are aerated and watered for some weeks. During composting, biodegradable material is mineralized by the microbial communities present in the waste. The organic material is transformed primarily into water, carbon dioxide (CO_2) and after a period of time a mature, humus-rich material (compost) is produced (Rico et al., 2011). While AD is a process by which organic matter is broken down by bacteria in the absence of oxygen, resulting in biogas, containing methane (CH_4), CO_2 and digestate. The majority of the nutrients entering the process are contained in the digestate, making it a valuable source of bio-fertiliser (Marañón et al., 2011). The biogas produced can be used to replace fossil fuels for heat and/or electricity generation, that if sufficient quantity is produced, farms can become energy self-sufficient or even become a net electricity producer (Karapaju ad Rintala, 2011).

The volume of CH_4 produced from AD is based on the amount of volatile solids in the manure. Each beef cattle in feedlot would excrete daily about 2.5kg of VS (IPCC, 2006), that is able to generates around 0,57m^3 of biogas (around 60% of methane) and that might produce 0.85 and 1.54 kwh of electricity when converted to a low (25%) and high (40%) end efficiency in small generator, respectively (Cuéllar & Webber, 2008). Not considering losses, the manure come from feedlot cattle in Brazil in 2010 (3.05 million heads) would have a theoretical potential of generating biogas to a proportion of 3,704 and 6,710 MWh per day (at a low and high-end efficiency, respectively), or 335.979 and 608.597 MWh during only 90 days of feeding cycle (currently in Brazil). This amount represents around 0.12% of the total annual Brazilian's electricity consumption in 2010 (BRASILc, 2011).

These estimative suggest AD as a promising means to the manure handling. However, it is evident that in a short term all the manure come from feedlot beef cattle in Brazil will be digested or even composted, because of existing large differences among feedlots and their farmers' skills, environmental conditions and up to that moment there was no knowledge upon the destiny of the manure produced in these places. These factors may make the introduction of any technique costly with low return on the investment for many feedlots, mainly for small producers (Baylis and Paulson, 2011; Hayes et al., 1980).

The ABC Program and the Brazilian National Waste Management Policy may strengthen the introduction of practices for the treatment of manure in Brazil. Once that, together, they finance and stimulate more sustainable production practices (BRASIL, 2010a,b). Although, lack of studies and projects at a regional or country level showing the effect of different manure handling systems in feedlot beef cattle in Brazil and their agronomical, economical, environmental and social impacts may prevent helping programs from being effective and being adopted by producers.

This survey of manure management practices in beef cattle feedlots should aid in the development of research for the feedlot industry in Brazil as well as provide data to facilitate broader application of future manure management models.

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FIGURE 1 **Locations of the Brazilian beef cattle industry accessed in this study concerning its manure management and use.**

TABLE 1 **Beef cattle feedlot manure storage by 73 Brazilian feedlots surveyed.**

Item	n^o of responses	% of responses
Knowledge about the amount of manure excreted (n=73)		
No aware	54	74.0
Aware	19	26.0
Frequency of pens cleaning (n=73)		
At the final of feed cicle	42	57.5
Twice per feed cicle	11	15.1
There was no pens cleaning	7	9.6
Every 10 days	5	6.8
At the final the year	5	6.8
Every 20 days	3	4.2
Type of storage of manure after being cleaned from pens (n=66)		
Heaps	58	87.9
Directly to land application	1	1.5
Pasture	1	100
Crops	0	0
Composting before heaps	6	9.1
Tank before biodigestor	1	1.5
Slurry formation (n=73)		
Due raining	37	50.6
There was no slurry formed	34	46.6
Water cleaning	1	1.4
No answer	1	1.4
Knowledge about the amount of slurry formed (n=38)		
Not aware	35	92.1
Aware	3	7.9
Type of storage of slurry after leaving the pens (n=38)		
Tanks	33	86.7
Directly to land aplication (crop/pasture)	3	7.9
Pasture	3	100
Crops	0	0
Tank before anaerobic digestion	1	2.6
Watercourse	1	2.6

(36) VALORISATION OF BIOMASS WASTE STREAMS IN LOCAL ENERGY CONVERSION PARKS

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EXECUTIVE SUMMARY

Biomass is considered as one of the main alternatives for the use of fossil fuels. Current production of bioenergy is strongly driven by policy support and subsidies. Focus mostly lays on the use of clean biomass streams like wood or food crops, often imported from the other side of the world. Energy production plants are specifically dedicated to one specific biomass product with one conversion technology and with one specific output (electricity, or heat, or biofuel). On the other hand, biomass (waste) streams are available at local level, like green household waste, cuttings and mowing residues, by-products of agriculture and industry. These streams are hardly or inefficiently used, due to limited and often costly processing possibilities. Nevertheless these biomass residues are present in substantial amounts and markets are looking for solutions offering higher economical added value for these biomass streams. Within the project ‘Energy Conversion Parks’ a consortium of Belgian and Dutch research institutes is analyzing whether an economically viable concept can be achieved to valorise these locally available biomass streams, through the use of synergies and clusters between different biomass streams and conversion technologies. An Energy Conversion Park (ECP) is a multi-dimensional, synergetic concept, converting multiple local biomass streams into useful energy and other bio-based products, through a combination of conversion processes.

The project develops a technological concept and a business plan for five pilot Energy Conversion Parks in the South of the Netherlands and in Flanders, Belgium. The five cases have different starting points, like the types and availability of biomass streams, the presence of other industrial activities that can be linked to, and potential energy exchange with nearby companies. For developing the five pilot plants, a stepwise approach is taken. First an inventory is made of the locally available biomass, followed by first ideas of a technical concept. Different concept and scenarios are developed depending on the local conditions, and this will crystallize out along the project. For the most appropriate technical concept an economical and technical feasibility study will be made, together with a sensitivity analysis for the most important parameters. Finally the interest of potential investors will be checked as well as societal feasibility to come to a fully developed business case. Important to know is that stakeholder participation has already started from the beginning of each pilot study. This approach is chosen very consciously because of the objective to embed the ECP in the local region. It is envisaged that the technical concepts and business plans developed for each site, will serve as a starting points for investors on these locations.

The project is on-going and first results are showing that the bottom-up approach in the five different pilots are evolving spontaneously in a similar way, although the speed of development largely depends on the support and willingness to participate of local parties. All developments are going through a learning process. This process and the experience will be logged in a public manual and knowledge system. The knowledge system will collect the main background data on biomass streams and processing methods, and suggest the optimal approach and points of attention to develop a local energy conversion park. This will help other initiatives to elaborate a similar concept in other locations and contribute to a more sustainable and climate neutral energy supply.

1 INTRODUCTION

1.1 Background

The growth of renewable energy largely depends on the use of biomass for energy. Current production of bioenergy is strongly driven by policy support and subsidies. Commercial focus mostly lies on the use of clean biomass streams like wood pellets or food crops, often imported from large distances. Biomass conversion plants are often one-dimensional, meaning that they are specifically dedicated to one specific biomass input product, relying on the most appropriate conversion technology for that type of biomass, and producing a specific output like biofuels, or electricity and/or heat. On the other hand, biomass residues and waste streams are available at local level. Typical examples are green household waste, cuttings and mowing residues or by-products of agriculture and industry. These streams are hardly or inefficiently used, processing possibilities are limited and often very costly. Nevertheless these biomass residues are present in substantial amounts, their energy potential is considerate, and markets are looking for solutions offering higher economical added value for these biomass streams.

1.2 Research objectives

Within the project ‘Energy Conversion Parks (ECP)’ a consortium of Belgian and Dutch research institutes is analyzing whether an economically viable concept can be achieved to valorise these locally available biomass (waste) streams, through the use of synergies between different biomass streams, conversion technologies and outputs. The idea is to maximize the valorisation of intrinsic values of the biomass and thereby improving its business case.

A biomass energy conversion park (ECP) is defined as a synergetic multi-dimensional biomass conversion site with a highly integrated set of conversion technologies in which a multitude of regionally available biomass (residue) sources are converted into energy and materials. Important starting point is the presence and availability of biomass streams (types of biomass and availability in the region of the pilot), other industrial activities that can be linked to or other nearby companies that can exchange energy. Both in biomass resources, conversion technologies and applications, smart combinations and links can be achieved, leading to very efficient and cost effective solutions.

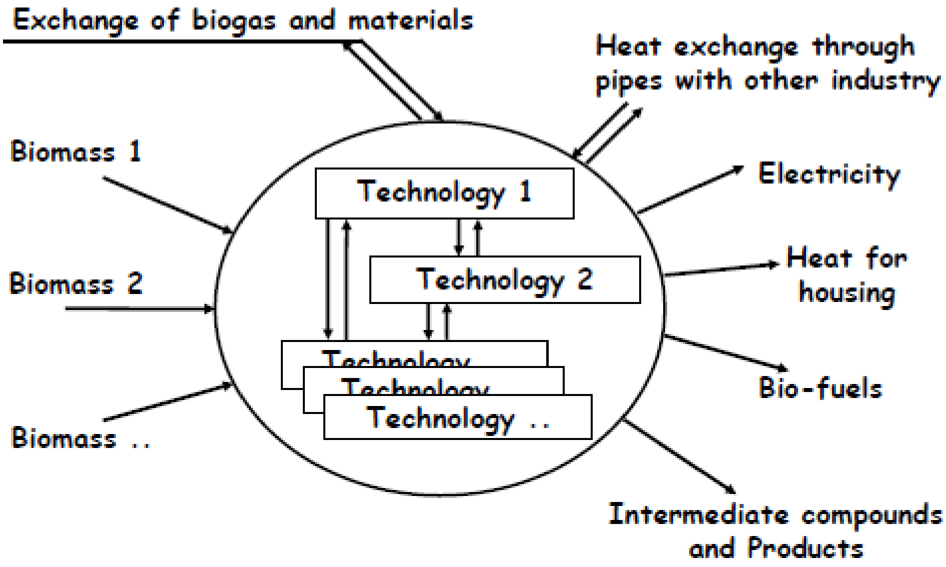

FIGURE 1 **Schematic of the ECP concept**

Through the use of local biomass, and converting this into energy and/or products that can be used in the region itself, not only fossil energy will be saved (with accompanied greenhouse gas savings), but also transport distances will decline, with reduced costs and pollution as a result. In addition the local use of biomass provides a contribution to the local economical climate, which is enforced because the focus lies on waste streams which are currently not or inefficiently used, often with high processing costs. This way these biomass streams can generate extra added value for the local community.

The project started in April 2010 and will last three years until March 2013. The goals to be reached are the following:

- demonstration of the economic advantage (feasibility) of an integrated/synergetic utilization of locally available biomass residues,
- development of a concept for five Energy Conversion Parks (ECPs) as pilots in South Netherlands and Flanders (Belgium). The development of an ECP concept implies a preliminary design will be developed with a study on the technical feasibility and a full business plan.
- development of a knowledge platform to help further initiatives getting started. The platform will include the main background data on biomass and conversion technologies, an optimal approach with respect to the local conditions, and the logging of the learning processes in the five pilots.
- further dissemination of the ECP concept.

2 METHODOLOGY AND RESULTS

2.1 Pilot studies

When demonstrating the ECP concept it is necessary to apply it in more than one case study as the conditions of resources, processing and utilization (with the possibility of linking to neighbouring consumers) may be very different. This requires a different approach with different choices, and consequently a broader experience needs to be built up. A technological concept and a business plan will be developed for five pilots. In general the pilots will be dimensioned for around 50,000 ton biomass (dry mass). Depending on the local conditions a combination of electricity, heat, biogas/biofuels and other material production will be the result. The pilots are situated in the North of Belgium and the South of the Netherlands. Because of the interaction between the five pilots the methodology will build on the experience gained in every situation.

Figure 2 shows the chosen locations for the pilots. From West to East: Sluiskil, Moerdijk and Breda in the Netherlands, and Beerse-Merksplas and Lommel in Belgium.

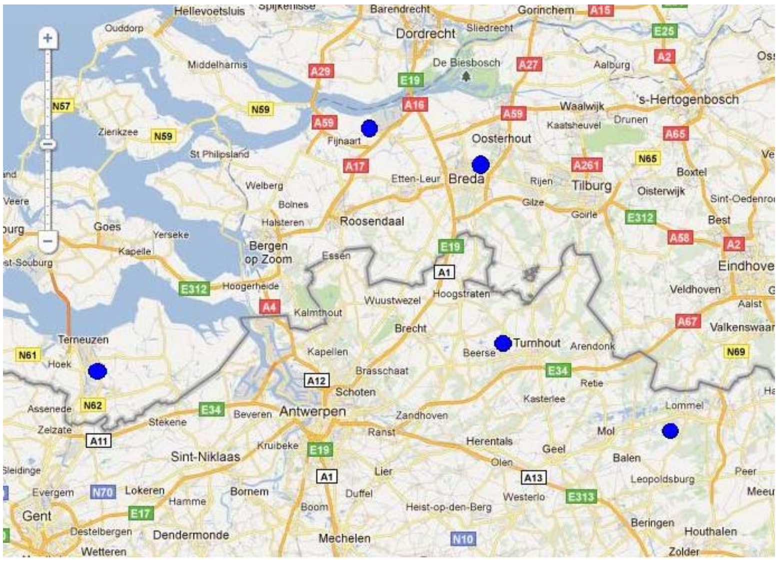

FIGURE 2 **Location of the five ECP pilots (blue dots, the grey line is the border between Belgium in the South and the Netherlands in the North)**

2.2 Steps in the implementation process

An important learning process in the project is to go through the implementation process of each of the pilots. The implementation process is often a long and difficult journey that has to be undertaken step-by-step. However every step of the implementation of a technically and economically viable project can be hindered or even obstructed by various obstacles. The project experiences in the implementation process of the five pilots will be registered and shared with future initiatives.

The first step of the implementation approach is the determination of the **location**. Often the location is decided at early stage by the party which takes the initiative (in terms of available space and location of related activities). When the location is not yet decided, various side conditions may play, and at early stage a detailed analysis of all influencing conditions is not feasible. In the 5th case (Lommel) a multi-criteria analysis was performed for the entire province of Limburg. Four main criteria were taken into account for every community in the region: (i) local biomass potential, (ii) local energy demand and exchange possibilities, (iii) existing biomass conversion installations and (iv) accessibility, industrial space and the willingness of local communities. Figure 3 shows the subcriteria used in the multi-criteria analysis. These criteria (filled with proxies) were weighted against each other, to come to an indicative comparison between the different communities. At the end the city of Lommel in the North of the province has been chosen as location for the 5th ECP in consultation with local stakeholders. What also played a role was the availability of historically contaminated soils around Lommel, which could be dedicated for biomass production for energy production (Schreurs *et al.*, 2011).

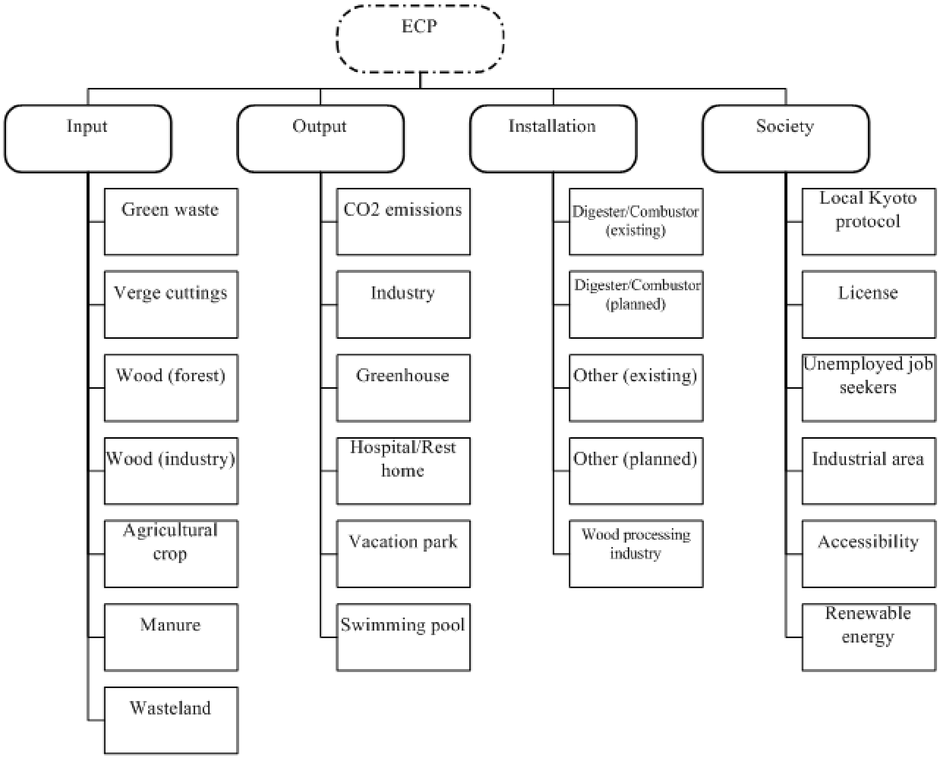

FIGURE 3 **criteria used in the analytical hierarchical process to determine the optimal location in the province of Limburg (BE) (Van Dael *et al.*, 2012)**

The second step in the implementation process (after the location has been selected) is to make a more detailed screening of the region in terms of **biomass availability**, coming from the waste sector, agriculture, forestry and industry. Potentials from literature are mostly theoretical and do not take into account local (practical) characteristics so these figures are less usable. It is necessary to talk to people and sectors that control these biomass streams to check how much is actually available and under which conditions.

Local energy demand is scanned in a third step. Crucial for the economic viability of an ECP concept is the valorisation of waste heat coming from the different conversion processes. Electricity can relatively easy be injected in the grid, upgraded biogas could potentially also be injected in the gas grid. Heat is more difficult to transport and therefore it is best to find a local market. For other output (biofuels, bio-products) the logistic situation (connection to roads, railways and waterways) is of high importance.

The fourth step is to derive first ideas for **technical concepts** starting from the local biomass streams, and the potential connection to existing facilities. These first ideas are then checked with local interested parties and first economic indications are also necessary at that stage.

In the ECP project a **sounding board** is created that unites the local interested parties. These parties result from interviews in preceding steps and are willing to take an active role in the next steps of the process. They have to be carefully selected considering the major impact they will have on the remaining implementation steps. The concepts are further developed in cooperation with the sounding board and take into account many different local parameters (biomass availability, exchange/clustering possibilities, access to grids and transport infrastructure) that were determined earlier.

In the following step the most interesting concepts are further screened using a **techno-economic evaluation** tool to result in a draft ECP concept for the site. Some choices are also to be decided by the investors and stakeholders, and some technical choices are still open for the base engineering.

When the draft concept is clear, a first **business plan** is made to determine the economic viability and fix the individual roles of the different parties. This will be a crucial step towards the financing of the project.

The project should also be communicated to the local community. The importance of the **communication** strategy should not be minimised since many projects have been stopped due to protest of the local community.

Important to know is that every ECP pilot development is approached as a **participative process**, where stakeholders are involved from the beginning. This approach is chosen very consciously because of the objective to embed the ECP in the local region. The stepwise approach should include **feedback loops** as several choices are interdependent.

The actual implementation and realisation of the local energy conversion parks, with base engineering, license applications, contract negotiations with suppliers, finance discussions with banks and investors, the building and commissioning part will not be part of the ECP project. Realisation is foreseen within a few years, earliest around 2014.

2.3 Example

Most of the pilot studies in the ECP project are currently in the stage of techno-economic evaluation of a number of concepts. In this chapter we will show the ideas around the concept in Beerse-Merksplas, where the ECP can build on an existing composting plant and a gas engine running on landfill gas.
Figure 4 shows a general overview of the biomass input and the potential conversion techniques and outputs involved. Solid lines are rather sure; dotted lines are suggestions or options.
Quite soon there was agreement with the owner of the site to focus on a VFG (vegetable, fruit and garden waste) digester to produce biogas, coupled with composting of the digestate. The existing landfill gas engine will also remain operational in the next years.

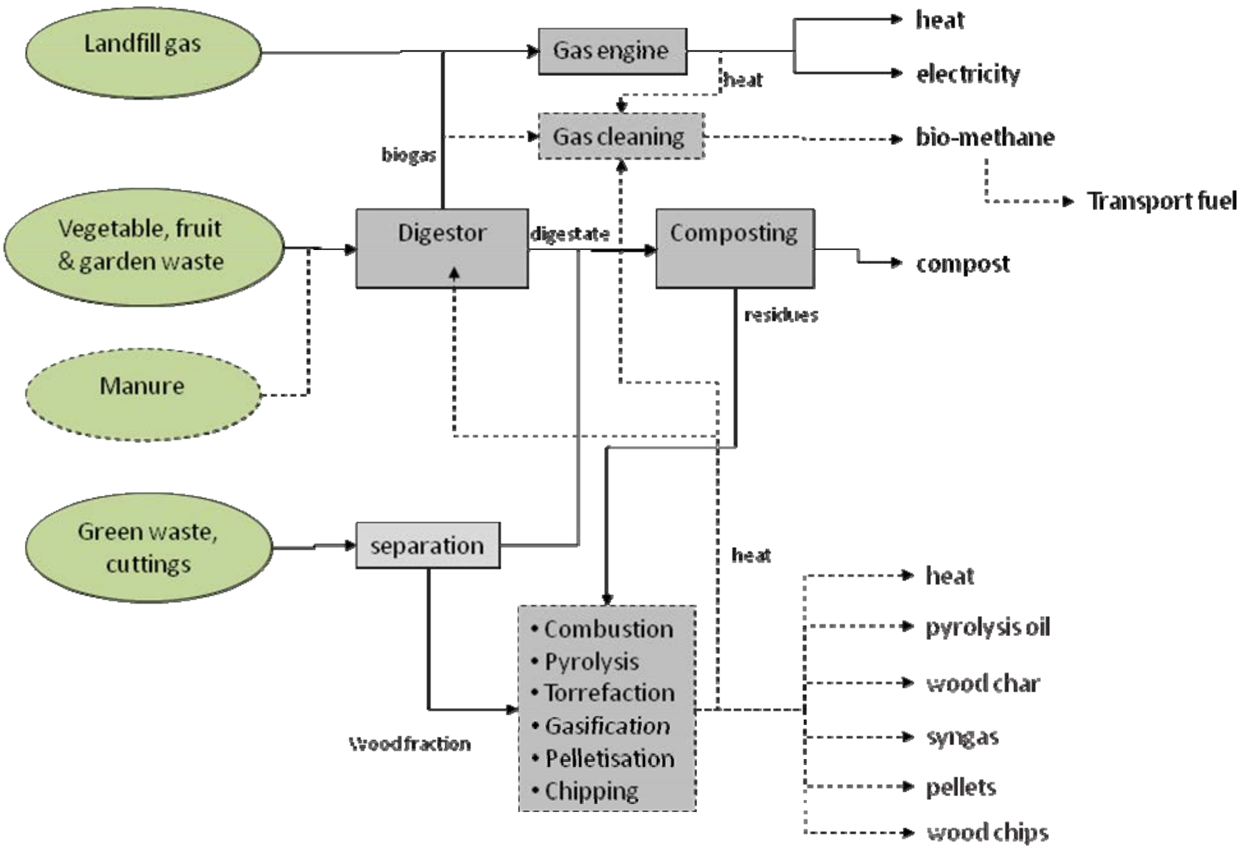

FIGURE 4 **First ideas for the pilot in Beerse-Merksplas (Belgium)**

The main uncertainties in the above scheme were what to do with the biogas, and what to do with the woody fraction of the biomass input, and how to deal with the balance of plant and the way heat is provided within the site. The on-site heat demand is limited (some could be used in the digester and for gas cleaning). There are no nearby neighbour companies who could use heat. Input of manure was rejected by the site owner.

Considered options/scenarios for biogas are (1) on-site electricity production and limited use of heat, (2) on-site electricity production and export the heat through pipes, (3) export of biogas through pipes to a CHP site, (4) upgrading of biogas to bio-methane and injection to the natural gas grid, (5) upgrading of biogas to vehicle fuel quality, which could serve as fuel for waste collection lorries and (6) upgrading of the biogas to LBM (liquid bio-methane) which can be sold to end consumers, or used in vehicle fleets. These options are actually valid for whatever biogas production site. The optimal choice will depend on local conditions and the legislative framework.

Considered options for the woody fractions are (1) combustion to heat which can be used on site in the digester and for gas cleaning, (2) pyrolysis to oil which can be sold to industry as fuel, (3) torrefaction and conversion to tradable torrified pellets, (4) gasification to syngas, which can be sold to industry, (5) chipping or pelletisation of the woody fraction to wood chips or pellets which can be sold to electricity and/or heating installations.

The different scenarios have been discussed with the involved stakeholders. At this stage preference seems to go to grid injection of bio-methane, which could later be used as vehicle fuel at a different site. Most of the woody fraction will be sold as wood chips; part will be used for heat production in a wood boiler, needed for upgrading of the biogas to biomethane. The following figure shows an overview of the concept with mass and energy balances (annual figures).

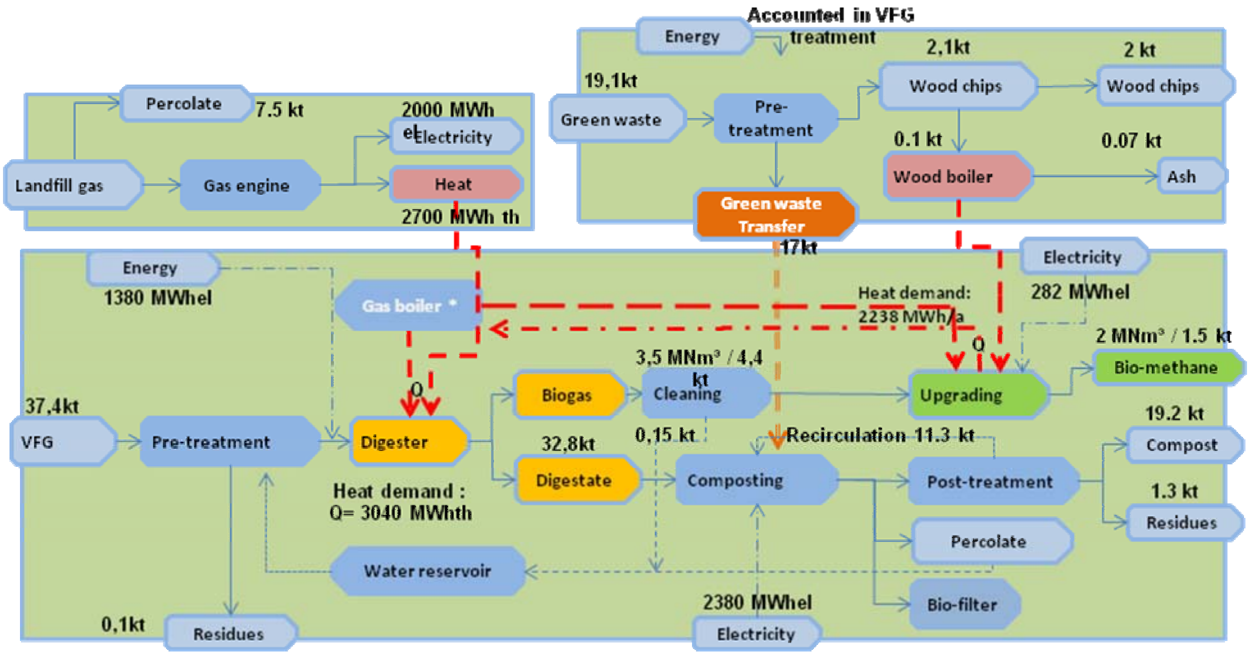

FIGURE 5 **selected concept for the pilot in Beerse-Merksplas (Belgium)**

3 LESSONS LEARNED SO FAR

The project is over halfway and concepts are taking shape. The bottom-up approach in the five different pilots is spontaneously evolving in a similar way. The following

For the screening of available **biomass** the pilots learned that the theoretical potential that can be calculated with statistical data is largely overestimating the actual contractible biomass streams (on the basis of inquiries). Demand for woody biomass as an energy resource is steadily increasing, so large competition with other energy initiatives can be expected. Most of the focus for these local initiatives will be on wet biomass, which is more difficult to transport.
It is very difficult to have an indication about present and future costs of biomass. For some waste biomass types gate fees are applied, but this may not be the case in the future.

In terms of location choice, the search for a well-suited **location** with optimal conditions is not straightforward. Having a starting point (e.g. existing conversion facilities) and a leading partner makes a big difference. Enough attention should also go to the accessibility and logistical part of the site for the required biomass streams. Demand for output (heat, residues) in the immediate surroundings should be explored; especially for heat it is not easy to find consumers in the surroundings. Symbiosis with existing nearby activities must be searched for. The methodological choice of location (Limburg) was a fresh view for local parties who often chose their location depending on only one criterion.

In the five pilots the consortium tries to introduce an **innovative** approach. This may conflict with existing plans of companies, which try to stick to known technologies and concepts. A balance should be found between the innovative character and the economical feasibility and practicability. It should be kept in mind that installations built around 2015 will actually be operated at least until 2030. So we may need to include some room for innovations to be included later on when commercially available (e.g. in terms of output valorisation or feedstock pre-treatment). Moreover a crucial point in an energy conversion park is **clustering**. If this involves different parties, this very soon raises questions concerning the financing model: who will invest, who will take risks, etc. Also the dependency between companies is an issue that gives great concerns.
Technological options and choices are now often determined by existing legislation and subsidy systems. This may divert the system away from the most efficient and economical solutions. In Flanders there is an effective green power certificate system. The result is however that most bioenergy systems only focus on electricity production and use of

heat is less relevant. There is also no system to support green gas injection into the natural gas grid, or to support the use of biomethane in vehicles. So in fact the suggested system for Beerse-Merksplas (as presented above) is currently not attractive because of gaps in legislation and distortion by the current power promotion system. One of the actions in the project is to bring this to the attention of policy and administrations and to lobby for solutions by the time the ECP would be operational.

4 FURTHER STEPS

The consortium will further work on the technological concepts in the five locations, leading to a technical feasibility study and a full business plan. It is envisaged that these plans will serve as a starting point for investors on these locations.

Next to the five local studies, a public handbook and knowledge system will be developed which can help develop similar ECP concepts in other locations. The knowledge system will draw on the experience gained in the five local studies, and contain insights, aids, useful information and calculation tools to find the best route towards a well functioning ECP, without reinventing the wheel. All information will be available on the project website www.ecp-biomass.eu.

The local initiatives and further steps for a broader application of the ECP concept totally fit in the policy of the Netherlands and Flanders to join forces and develop local initiatives towards a greener economy.

5 ACKNOWLEDGEMENTS

The project is funded by the Interreg IVa – Flanders-Netherlands programme from the European Fund for Regional Development that stimulates cross border projects, in this case between the northern part of Belgium and the southern part of the Netherlands. Also the Dutch Ministry of Economic Affairs, the Flemish Government, the Provinces of Noord Brabant (NL), Zeeland (NL), Limburg (BE) and the partners themselves are co-financing the project. The project is coordinated by VITO, a research institute in the Flemish region; University of Hasselt is the second Belgian Partner. The Dutch partners are Avans University of Applied Sciences, Wageningen University and Research Centre (WUR) and HZ University of Applied Sciences. ERAC and BTG have joined the project as subcontractors for project coordination and for technical support respectively.

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(147) SUPPLY OF METHODS AND DATA FOR MUNICIPALITIES WISHING TO SET COLLECTIVE COMPOSTING PROJECTS

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EXECUTIVE SUMMARY

Municipalities wishing to develop collective composting i.e. at the bottom of collective buildings need methods and data to implement and evaluate such operations. These methods and data are for instance the production of organic waste (o.w.) in collective home per inhabitant and per year and the amounts of o.w. really diverted from the usual waste collection following the implementation of a collective composting operation. This paper aimed to propose such methods. These were tested at the territory of Rennes Métropole.

Since there was no selective collection of o.w. at RM, measure organic waste production imposed to collect residual household waste (r.h.w.) then to quantify o.w. within. It also required to select the area of sampling, size the sample i.e. the number of apartments which production of o.w. should be collected, take into account the week frequency of usual collection then apply sampling at every season. The defined area corresponded to one part of urban area. It excluded city center in which economic activities concentrate in collective buildings. Taking into account the r.h.w. production at Rennes Metropole was 211 kg /inhbt /y in 2010 and that every waste sample should be at least equal to 2 tons, a sample of 610 apartments i.e. 1123 inhabitants was selected randomly from a list of 2500 addresses included in the defined area. The selected buildings were not equipped with collective composter. Since r.h.w. collection was practised twice a week on the selected area, i.e. on Monday and Thursday, two samples should be collected per week in parallel to usual collection. Four sampling campaigns were applied corresponding to winter, autumn, spring and summer. Then, the samples were characterized according to the French procedure MODECOM. The mean quantity of r.h.w. in collective homes was found around 145 kg/inhbt/y. and varying from 124 to 168 kg/inhbt/y. according to the season. Discard with global r.h.w. production i.e. 211 kg/inhbt/y should be investigated especially regards to its impact upon estimation of potential waste diversion through collective composting. The mean quantity of o.w. was found around 42.1 kg/inhbt/y. with a little variation over the seasons i.e. we found 10% of variation. The part of o.w. in the r.h.w. was found around 29% with a variation over the season of 10%. In spite of this low seasonal variation, the reduce of the number of sampling should be considered carefully.

Three methods were tested in order to estimate o.w. diversion following the implementation of a composting operation. The first method (M1) was based on the weighing of all the o.w. from the building by a volunteer resident of the composting site. Thus, three collective composting sites were equipped with similar composters and monitored from 6 to 12 months. The second method (M2) consisted in the individual weighing of o.w.. Thirty households from different residences of Rennes Metropole weighed their o.w. at home, before the supply to the composter. A third method (M3) was based on the conservation of the mass of mineral matter during the composting process. Applying methods M1 and M2 led to respectively 43.5 and 47.4 kg/inhbt/y. Although, these are not significantly different from o.w. production (42.1 kg/inhbt/y.), it should be kept in mind that volunteers involved in the methods M1 and M2 might have higher production of o.w. due to their food habits. This assumption was supported by the results from the study carried out in the same framework at individual homes. Such assumption led to propose some rules allowing to estimate o.w. production thanks to whether M1 or M2 or on basis of sampling campaigns were proposed. M3 was found responsible for large errors in the estimation of waste diverted.

1 INTRODUCTION

Many municipalities wishing to develop operations of in-situ composting come up against the difficulty that there is a lack of data and methods to implement and optimize the operations and further to evaluate their interests and limits, especially for collective composting. The methods or data required should allow for instance to i) quantify the amount of organic waste produced on a territory, ii) quantify the amount of organic waste composted i.e. diverted from usual collection, iii) choose the types of composters (ergonomy, treatment capacity, behaviour in composting etc), iv) apply the “best” composting process (bulking agent to waste ratio, residence time in fermentation then maturation etc), v) dimension composters as function of waste diversion expected, vi) be able to monitor composting treatment (measurement of temperature, odours, compost quality etc), vii) evaluate satisfaction of people taking part to the composting operation, viii) provide to the municipality elements which allow the evaluation of the operation (costs, inhabitants satisfaction…). The development of such methods, i.e. focused on collective composting, took part to a more global project named Miniwaste aiming to reduce organic waste production through several ways as for instance collective or individual composting or the reduce of food wasting. This project was financed by the European Life + Program and gathered five partners i.e. Rennes Métropole, Lipor, Brno, ACR + and Irstea. This paper is focused on the set-up of the methods allowing to quantify organic wastes production and organic wastes diversion (following the implementation of a collective composting operation). This set-up was performed at the territory of Rennes Métropole.

2 METHODOLOGY

To help municipalities implement and assess their collective composting operations, it is necessary, firstly, to know the quantity of organic wastes (o.w.) in the residual household waste (r.h.w.). This data represents the maximum amount of o.w. to be processed by the collective composting. Secondly, it is necessary to check what part of o.w. is really diverted from the usual collection by implementing a collective composting operation. That allows, before the start of the project, to size the composting site and after, to assess the efficiency of collective composting.

2.1 Assessment of the quantity of organic waste produced on a territory

The method developed aimed to allow to quantify organic wastes produced at collective homes in a defined area. The method was inspired by the one given by Fangeat (2005) and Ademe (2005). Sampling was applied in agreement with standard methods XP X30-413 and XP X30-422. Characterization was practised on moistened household wastes in agreement with standard method XP X30-408 named also MODECOM.

At Rennes Métropole (R.M.), r.h.w. from individual and collective homes are collected through two different collects. Three geographical areas were defined for collective homes, each having its own collect. Among these 3 areas, one was excluded since it is dedicated to city center in which several economic activities also take place in collective buildings (i.e. not only people living). The selected sector was collected twice a week. According to the sampling standard method XP X30-413, every sample of household wastes collected should be at least 2 tons. Based on the production of r.h.w. at R.M., i.e. 211 kg/inhbt/y. in 2010, we estimated a necessary number of inhabitants in the sample of at least # 1100 inhabitants. Assuming a mean value of # 1,8 inhabitants per apartment, the sample of buildings should account for # 600 apartments. The mean of apartment per building at Rennes being about # 15.6, it would lead to sample # 39 addresses. From a list of 2500 addresses corresponding to the selected area, we extracted, firstly, 200 addresses randomly. Secondly, for 155 addresses it was clear that establish a reliable correspondence between bins presented for collection and the number of inhabitants who supplied their wastes in the bins was impossible. Thus, 45 addresses were kept. Then each was investigated to identify if its wastes storage organization allowed to establish the correspondence between bins and apartments providing in bins. At the end of this work, the sample was constituted of 21 sites of collection including 41 addresses and 610 apartments. Thanks to a request near the syndics to estimate as accurate as possible the number of inhabitants living in the sampled addresses, we obtain a sample constituted of 1123 inhabitants.

The sampling plan took into account that there were 2 collects per week and the fact that we wanted to check the influence of the season on the production of o.w. Thus, four campaigns were organized in a year i.e. September 2010, December 2010, March 2011 and June 2011 with 2 collections per campaign. The sample was taken with a rented garbage truck and the help of rippers. It was weighed before and after collection meaning the amount of waste collected on the defined area was accurately known after each collection campaign. One day was necessary to collect each sample (5 buildings were collected every hour - 1 every 12 mn - and the collection lasted around 8 hours per day).

The sub-sample (500 kg of household waste) was obtained by quartering in series of the amount of waste loaded in the truck at each collect (from 1 to 2 tons). Quartering was practiced after the unloading of the truck, by mixing the sample thanks to a loader, then choose and sample one area-fraction to 500 kg sub-sample. One day more was necessary for characterization of each sub-sample. The sub-samples were characterized in agreement with MODECOM procedure.

More, on our studied area the bins were equipped with electronic identification chips and the trucks with weight sensors. The weight sensors (w.s.) installed on trucks should allow to follow waste production of each building whereas the weighing of the truck give a global production of waste on all the collected area. This w.s. should enable us to monitor the quantities produced in our sample throughout the year. Thus, we can avoid the usual method which consists in monitoring quantities for 3 consecutive weeks. In this usual method, the halfway week is reserved to the characterization. However, in March 2011, some weight sensors were still out of service meaning only masses measured at every sampling campaign could be used. An alternate method was tested to follow the quantity of waste produced at each sampled building in case trucks are not equipped with weight sensors. Then, the production of organic wastes was estimated by a rough estimation of the volume of wastes in every bin collected. Indeed, sum the volumes and then multiply the total volume by the density of wastes in bins could lead to estimate the mass produced provided the density of wastes is measured at every season or territory.

2.2 Quantification of organic waste diverted from the usual collection

The existing data do not make it possible to know the quantity of o.w. which will be really diverted from the household waste following the installation of a collective composter. It is thus necessary to develop methods which permit to know the participation rate to the composting operation and the percentage of o.w. composted i.e. diverted from the usual collection. Determine the percentage of o.w. diverted requires know the quantity of o.w. supplied to the composter and the residual amount of o.w. put in the r.h.w. This determination was performed for individual homes. It is impossible at collective homes since in a collective bin it is impossible to distinguish the wastes supplied by households who compost from the wastes supplied by households who do not compost. So, the determination of the diversion rate cannot be done otherwise than by comparing the mean production of a population (MODECOM) to the diverted quantity i.e. the weighing of o.w. supplied to the composter.

Thus, a first method (method 1) consisting to ask people to bring their wastes in a first container and find a volunteer to weigh the wastes brought in the container was set-up. The main drawback of this method lies in the change of practice usually applied by inhabitants taking part to the composting operation (since they do not directly bring their wastes into the composter but into a transient container). The second drawback of this method is to know precisely the number of inhabitant taking part to composting. In our case, the number of inhabitants supplying o.w. in the composter was estimated through a survey of the volunteers. Figure 1 shows the fitting-out of the composting site. Next to the composter, the inhabitants insert their wastes in a bucket used as a transient container. Every three or four days, the wastes loaded by inhabitants in the transient container are weighed by the volunteer who transfers them into the locked composter. The bracket (c) consists of two parts: an upper part which is movable and a bottom part which is fixed. The upper part can be removed in order to close the composter. Thus, the composting site keeps a normal appearance.

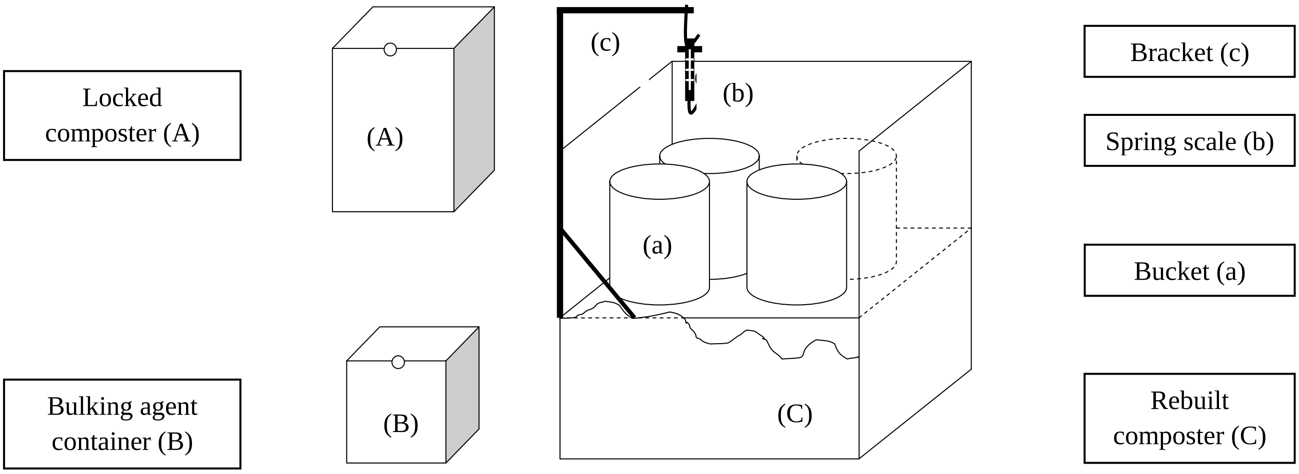

FIGURE 1: Area for composter

A second method (method 2) consisted to select a panel of about thirty inhabitants from various residences who accepted to weigh individually the amount of organic waste they brought to the collective composter.

A third method (method 3) funded on the theory of the conservation of the mineral matter (m.m.) was also set-up. This method consists to weigh initial waste in the composter, take a sample and measure its content in m.m.. When turning of composting material or when compost is extracted, few weeks later, the mass of wastes is measured, a sample is extracted and its content in m.m. is measured. Assuming some mean values of moisture and m.m. content in biowaste, the increase of mass of m.m. allows to determine the mass of wastes brought to the composter. In case of addition of b.a. every time o.w. is brought in the composter, the same procedure has to be applied to the b.a. i.e. the mass of b.a. has to be determined whereas its contents in moisture and m.m. have to be measured at the beginning and every time compost is turned or extracted (this supposes to control b.a. feeding in the container dedicated to its storage). The mass of b.a. introduced is obtained by subtracting the mass in the storage container at the start of the cycle from the mass in the storage container at the end of the cycle. This method requires the determination of dry matter and organic matter (o.m.) contents of wastes and composts samples at a chemical laboratory. More, the method may be difficult to apply by inhabitants without any support (composting guide). It was proposed here mainly to check if it led to results similar to the ones obtained by the method 1. If this method is validated, it could be applied without the assistance of a volunteer to weigh the o.w.

3 RESULTS AND DISCUSSION

These methods were tested in order to be applied or readapted in the territories of the project Miniwaste partners i.e. Rennes Métropole, Lipor and Brno or other interested municipalities. The purpose of this study is therefore, in view of the results, to validate or disprove some of the methods tested to obtain a clear and adaptable protocol.

3.1 Assessment of the quantity of organic waste produced on a territory

For every sampling, the mass of the waste collected was measured. Through the application of French standard XP X30-408, eight samples of about 500kg were characterized. Thus, this method gave two types of result: a percentage of o.w. in the r.h.w. and a quantity of o.w. produced by a known number of inhabitants. Table 1 gives these results for every week sampling. The part of o.w. in the r.h.w. of collective homes in Rennes is similar to the national mean which is about 31% (Ademe, 2007). Table 1 also shows that the season did not significantly influence the part of o.w. in the r.h.w. since CV was inferior to 10%. By the same way, the quantity of r.h.w. did not seem to vary with the season except summer. The mean value was around 145 kg/inhbt/y i.e. significantly below the mean of Rennes. Indeed, in the city of Rennes, the measured quantity of r.h.w. is about 231 kg/inhbt/y (Rennes Métropole, 2010) calculated on all the household waste and related waste i.e. with small economic activities. The origin of this discard should be investigated especially regards its potential impact upon estimation of o.w. diversion following the implementation of a collective composting operation.

TABLE 1
Results about the part and the quantity of organic wastes in the residual household wastes

	Automn	Winter	Spring	Summer	Mean	CV%
Organic wastes (%)	25,80	32,51	28,89	29,72	29,23	9,45
Organic wastes (kg/inhbt/y.)	43,20	47,41	40,93	36,88	42,11	10,45
Total r.h.w. (kg/inhbt/y.)	167,59	145,45	141,45	123,65	144,54	12,49

The low variation of o.w. percentage in r.h.w. seems claim for a possible reduction of the number of characterization. However, such reduction should be decided carefully taking into account for instance discards between autumn and winter. Given our results of characterization were similar to the national mean, use directly national data may also be one way to estimate o.w. production.

Monitor the quantities outside the week of characterization can be performed through the applying of three methods. If the bins are equipped with identification chips, then it is easily possible to follow the quantities produced address by address. The second solution is to rent a garbage truck with a crew that will collect only the addresses of the sample. However, if these two methods lead to reliable results, their applications are still expensive. A third solution, based on a mean density of waste, can be used to estimate the produced quantities of the sample at a lower cost. The eight samples collected allowed to define a mean density of 95 kg/m^3 as presented in the Table 2. The sample 4 was removed of this calculation because the filled volumes were not trustworthy due to a lack of data.

The season had not a significant influence on this density and then it was possible to rely on this density to estimate the quantities of waste presented for collection. However, apply this last method imposes to re-determine the mean density since it may be specific to each territory. The calculation of this density can be carried out the week of characterization.

TABLE 2
Results about the calculation of a mean density

	Automn		Winter		Spring		Summer		Mean	CV%
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8		
Calculated density (kg/m3)	115	89	92	220	87	93	95	97	95	10,45

The Table 3 presents the masses obtained by the utilization of each of these three methods. Except for the sample 4, all the result between the three methods are similar with a variation lower than 11% and for five of them, the coefficient of variation between the results is less than 3.5%. Thus, the use of these three methods to determine the quantity of waste produced is possible. The choice of one of them is left to the municipality.

TABLE 3
Results about the measured quantities according to the method of evaluation

	Automn		Winter		Spring		Summer	
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
Mass obtained by weighing the truck (kg)	2080	1180	1880	880	1840	1060	1600	1080
Mass obtained by weight sensors (kg)	1753	1214	1858	673	1740	1052	1605	1063
Mass obtained by a mean density of 95kg/m3 (kg)	1730	1260	1941	381	2024	1092	1609	1060
Mean (kg)	1854	1218	1893	645	1868	1068	1604	1068
CV %	10,6	3,3	2,3	38,8	7,7	2,0	0,3	1,0

3.2 Quantification of organic waste diverted from the usual collection

Three methods were carried out. These methods must allow to define a mean participation rate to the composting operations and to approach a reasonable maximum rate of diversion per inhabitant. Reasonable, because all the produced o.w. are not supplied to the composter given the restrictions advocated (e.g. meat) and regularity of the household to compost.

In the case of the method 1, three sites of collective composting were monitored. The results of weighing were expressed relating to the number of participating inhabitants in the aim to compare them with the o.w. production i.e. 42 kg/inhbt/y for the collective homes of Rennes. The results about the method 1 are presented in the Table 4. We obtained a mean participation rate of 18.5%, similar to the mean of R.M. which is about 19% (Society Eisenia, service provider of R.M.). The diverted quantity of o.w. measured with the method 1 (43.5 kg/inhbt/y) is similar to the produced quantity. In that case, it is difficult to establish a diversion rate. The similarity between these two data led to think about two hypotheses: an underestimation of the number of participating inhabitants (H1) and/or a bias relating to a behavior different for composting and weighing households (H2). To have a surer data, we have calculated an o.w. quantity diverted at the scale of a building and thus based on the number of residents. We obtain a diverted quantity around 8kg/inhbt/y and to compare with 42 kg/inhbt/y, it represents a diversion rate of 19%. This rate is similar to both the mean rate of participation that we estimated and the mean rate of participation at R.M. These similarities show that the estimation of the number of participating inhabitants by a request near the composting guide is rather correct. The uncertainties about the hypothesis H1 become thus insignificant.

TABLE 4
Results obtained by a collective weighing (Method 1)

	Number of resident of the equipped building	Number of inhabitant taking part in composting (request)	Rate of participation (%)	o.w. composted per year (kg/y.)	o.w. composted per inhabitant participating (kg/inhbt/y.)	o.w. composted per resident (kg/resid./y.)	Rate of diversion per residence (%)
Method of calculation	D	E	E÷Dx100	G	G÷E	H=G÷D	H÷42.1x100
Composting site 1	49	10	20,41	386,59	38,66	7,89	18,74
Composting site 2	356	51	14,33	2326,91	45,63	6,54	15,53
Composting site 3	97	20	20,62	926,38	46,32	9,55	22,68
Mean			**18,45**	1213,29	**43,53**	**7,99**	**18,98**

To check if the hypothesis H2 is admissible, the result of the method 1 was compared with the result obtained by the method 2. Table 5 summarizes the different results obtained in Rennes by both methods of weighing regards to the production of o.w.. With a CV of 6%, there is not a significant difference between these three results. Thus, at this stage we can not validate or reject H2.

TABLE 5
Quantity of organic wastes produced and composted

	OW produced non-composting people	OW composted Method 1	OW composted Method 2
Method	Characterization and quantity monitoring	Collective weighing	Individual weighing
Data (kg/inhbt/y)	42,11	43,50	47,40

To have a better comprehension of these results, we need to rely on the data obtained at individual homes (IH). This study carried out on R.M. territory led us to suppose that the behavior regards composting and food habits are similar between IH and collective homes (CH). The method applied in IH allowed to know for the same category of household both o.w. amounts supplied in the composter and amounts put in the r.h.w.. The sample consisted of three main categories of population: Composting households volunteers to weigh theirs o.w. (CW), Composting households non-weighers and Non-composting households.

The results obtained show that one of the category of CW composts 75% of its production. Regarding its behavior in composting, it is the category nearest to the CH. Indeed, the o.w. put in the composter are the same than the CH due to restriction in compostable wastes in CH. Without further information about a difference of behavior between IH and CH, it is the rate of 75% which is applied. Thus, the maximal diverted quantity hoped for a mean population is 31.5 kg/inbt/y i.e. 75%*42 kg/inhbt/y.. The difference between 31.5 kg/inhbt/y and 47.4 kg/inhbt/y becomes significant. That confirms the hypothesis H2: there exists a different behavior in food and composting habits for weighing volunteers. The weighing and composting households seem more aware and more attentive to their wastes, and thus they will have quantities of waste composted larger than the mean of the composting population.

Then, the results showed that recruit volunteers to weigh can create a bias. In the case of R.M., an overestimation of the quantity potentially diverted becomes significant when the participation rate exceed 20%. It is confirmed by the study in IH i.e. the CW category produces the highest quantity of o.w. i.e. 80kg/inhbt/y. against nearly half for non-composters. Then, the assessment of the diverted quantity of o.w. by the method 2 named B must be corrected by a coefficient named C according to the general expression:

$$\text{Diverted quantity in kg/y} = B \times C \times \text{number of inhabitant}$$

Let A be the amount of o.w. in the r.h.w. and γ be the participation rate. The term "$B \times C$" is framed as follows:

- $B \times C \approx B$ when γ approaches 0 (0% of participation)
- $B \times C \approx 0.75 \times A$ when γ approaches 1 (100% of participation)

The table 6 gives the framing of C as function of the participation rate named y.

TABLE 6
Framing of the coefficient C as function of the participation rate

When the participation rate (y) approaches	0	1
B × C =	B	0.75 × A
C =	1	(0.75 × A) ÷ B

As we do not know the specificities of the whole population, we expressed the coefficient C as a linear equation which can be posed as follows: C = a×y + b

- When y=0 thus C=1 (Table 6). The equation a×y + b becomes a×0 + b, thus b=C=1.
- When y=1 thus C=(0.75 × A) ÷ B. The equation becomes a×1 + 1, thus a = C-1 = ((0.75 × A) ÷ B) - 1.

Thus, we can define C as function of the participation rate as follow:
C= a×y + b = 1 + (((0.75 x A) ÷ B) – 1) × participation rate
The calculation of the diverted quantity becomes:

- Diverted quantity in kg/y = B × (1 + (((0.75 x A) ÷ B) – 1) × participation rate) × number of inhabitant

If the municipality does not want to implement weighing, it is also possible to rely on the waste characterization to estimate the amount of o.w. that can be diverted. Thus, the municipality considers that the behavior of the population is uniform i.e. there is no difference between a volunteer to compost and the rest of the population. The calculation of the diverted quantity becomes:

- Diverted quantity in kg/y = 75% × A × number of inhabitant

If the municipality wants to rely on weighing, it shall not exceed six months because beyond, the volunteers and the inhabitants participating can grow weary of weigh or supply the waste under the conditions of the experiment. To avoid resorting to volunteers to weigh the o.w., a third method was tested. It is based on the theory of the conservation of the m.m. mass during the composting process. The results of this method 3 were compared with the results of the method 1. These results are presented in Figure 2 a. and we note that this method gave results much more important than method 1. Figure 2 b. allows to check if the weighing done by volunteers is trustworthy. Indeed, the b.a. were weighed by themselves and volunteers. Figure 2 b. shows that the results are close. Thus, the method 1 gives more accurate results than method 3.

This difference is due to inaccuracy of the method 3. Indeed, this method imposes assumptions of moisture and organic matter contents of the waste supplied. Although, o.w. supplied were sampled and analyzed once per month, their real characteristics remain unknown. If uncertainties for moisture do not lead to significant discards in the calculation of o.w. supplied thanks to method 3, uncertainties for o.m. content lead to high under or over estimation compared to data obtained by applying method 1. Indeed, a measurement error of less than 2.5% on the amount of o.m. in the incoming waste increase the quantity of waste supplied to the composter of nearly 200 kg.

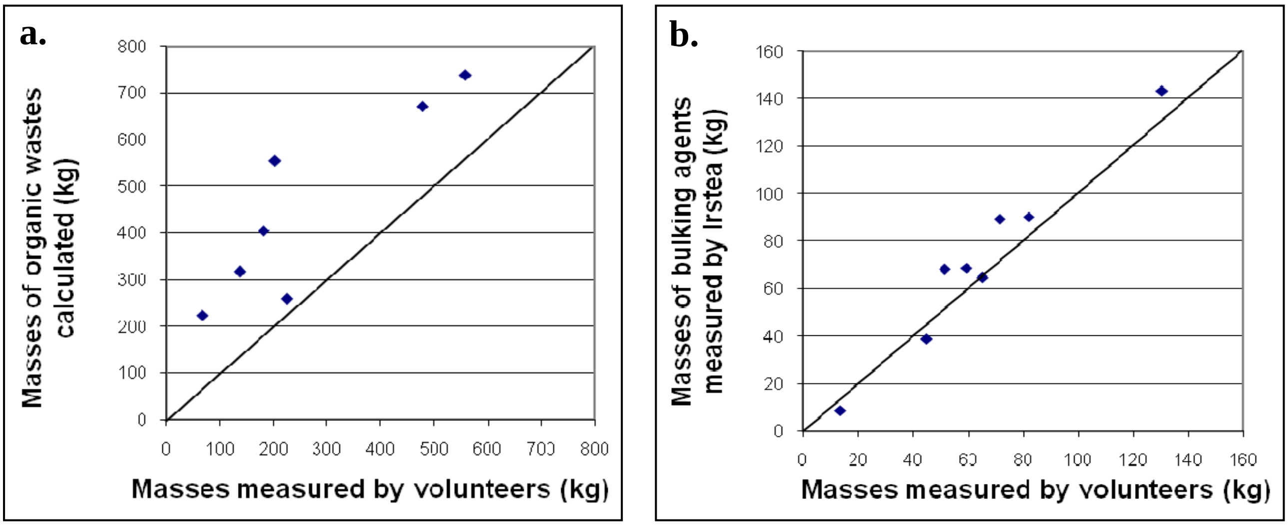

FIGURE 2. Comparison between the results of method 1 and 3

Thus, if a municipality wishes to measure the quantity of diverted o.w. from the usual collection by composting, it must be able to recruit volunteers to weigh the waste or rely on the quantities produced by applying factors.

The choice of the method of weighing i.e. method 1 or 2 is left to the municipality. However, it is important to remember that the method 1 allows to obtain a more reliable result than the method 2. Indeed, the method 1 is a method of weighing which allows to measure a real diverted quantity at the scale of a building, to assess the efficiency of composting and to evaluate the room of progress relating to the rate of participation.

4 CONCLUSIONS

The study conducted within the project Miniwaste allowed to collect data specific to apartment buildings. This work will enable municipalities to optimize the management of o.w. produced in collective homes. Two types of protocols were developed: one which allows a scientific evaluation of the o.w. quantities and the other an estimation of these quantities at a lower cost.

So, in a case of a scientific evaluation of the quantity of o.w. in the r.h.w., the municipality must complete an assessment of the quantities produced on a sample of the population living in collective home on at least 3 consecutive weeks in the year. It will spend the halfway week at the characterization of waste from this sample according to the MODECOM procedure. The approach of the quantity diverted will be through the implementation of weighing of the waste supplied to the collective composter. However, these studies are unwieldy to implement. It is therefore possible to approximate the quantities of o.w. produced and composted by simplified methods. The quantity of o.w. in r.h.w. can be obtained on the basis of national mean of the composition of the bin in % and a quantitative monitoring of 3 consecutive weeks. In the same way, the composted quantities can be evaluated by applying a rate of 75% to the quantity of o.w. in r.h.w.. These proposed protocols should be tested on the territories of the Miniwaste partners before being validated.

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(225) VEGETABLE WASTES CHARACTERIZATION FOR FEED PRODUCTION IN THE BASQUE COUNTRY

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EXECUTIVE SUMMARY

Almost three-quarters pairs of vegetable wastes generated from food surplus finish in a landfill, in spite of having the potential to be valued as raw material for animal feed if they are managed properly. In addition, the dependence on the traditional raw materials by feed manufactures makes necessary to look for alternatives. In this sense, this study, as a part of a European LIFE 09 ENV ES 473 Project, is located in the Basque Country (north of Spain) and has been firstly focused on vegetable wastes characterization. Since the use of food by-products as animal feed feasibility depends on each by-product characteristics, all factors of influence must be identified and analysed in order to assure the developing of an effective and viable solution for their addition in feed formulations.

To get it, all vegetable producer sectors have been identified and the quantity of each by-product has been estimated by consulting previous studies, public and private databases and by making a questionnaire to the associations and main representative enterprises of each sector. Also, the relation between Composition and Technical, Market and Legal needs has been studied listing all requirements and legislation and making a complete list of analytical parameters that have been divided in nutritional composition and undesirable substances. It has also been studied the Seasonal Variability of the composition of Market sector by defining a Sampling plan, which has been divided in three different periods throughout the year: one in spring, the second between summer and autumn and the last one in winter, with the aim of analysing how the composition of each sample varies depending on the month. In order to ensure the representativeness of the samples and to allow comparison between different samples analytical results, it has been designed a Sampling protocol, which guarantees that all samples have been taken in the same conditions.

The vegetable producer sectors identified in Basque Country have been: Markets, Potato, Cider, Wine, Horticulture, Bread and Coffee and the total amount quantified has been about 25,000 tons. In case of Cider and Wine, it has been found a remarkable seasonal variety, only producing at a certain time of year, whereas in other sectors, such as Potato, Bread, Coffee and Horticulture, a seasonal stability has been found. In nutritional composition of Markets sector, substantial differences in the seasonal variability have not been found. In general, vegetable wastes moisture has been so high to advise reducing the water level and therefore bulkiness, by developing a drying process. However, the cost of drying them can be so high to make the treatment infeasible, unless the drying process would be highly efficient. In this sense, according to the EPEA (Feed Manufacturers Association of the Basque Country), the high Volume respect to low Protein level limits the inclusion of this raw material in feed formulations to a low percentage, between 3 and 6%. The most interesting vegetable wastes have been Bread and Coffee by-products due to their percentage of protein, of 10 and 17% respectively. Respect to undesirable substances, almost all them are below maximum legal limits, except coffee and wine by-products which have levels of nitrate above maximum legal limit, 33,3 and 28 ± 28 mg/kg respectively, being the maximum legal 15. In addiction, to minimize the contamination and guarantees their feasibility for feed formulation, it is necessary to design an Action Plan to manage them properly.

As a conclusion, vegetable wastes have potential to be valorised as animal feed due to their characterization results but, there are some critical keys that they are necessary to solve to make them useful for animal feed manufactures, such as their high level of moisture and the cost of the drying process.

1. INTRODUCTION

The fact that the population in the Earth has been growing continuously in the last years and, therefore the need for natural sources associated with them, have caused that natural resources have been become increasingly scarce. In this sense, it is necessary to optimize the use of them in order to make them more sustainable.

In addition, humanity generates thousands of organic wastes tons every day that finish in a landfill, so the use of these wastes as raw material for feed formulation is an interesting alternative that it is necessary to analyse because, as well as reducing the environmental effect associated to its production, they can reduce the cost of animal feed (Myers R.O. et al, 1999; Westendorf, M.L. et al., 1998; Westendorf, M.L. et al., 2000).

However, the use of vegetable wastes as raw material for animal feed formulation has some difficulties that can limit its feasibility. Its high moisture level, in more cases above 80%, makes more difficult the handling and can accelerate the development of microbiological contamination (Garcia, A.J. et al., 2005), which advices previously subjecting to a drying process. Also, their composition can vary extremely throughout the year (Westendorf, M.L. et al., 2000), which involves that animal feed manufactures have to change their feed formulations depending on their composition.

In this way, there are some experiences that show the feasibility of using vegetable wastes in animal feed. A study about organic solids wastes generated in Salamanca city (Garcia, A.J. et al., 2005) analyzed different organic wastes, such as meat waste, fish waste, fruit and vegetables waste, restaurant waste and house hold waste, determining the chemical composition, microbiological characterization, dioxins, furans, PCB, sand mineral content for every type of waste fraction. The conclusion was that it is possible to use vegetable and fruits by-products in the feed formulation for animals.

A subsequent study in the same way (Esteban, M.B. et al., 2006) analyzed these by-products as alternative for traditional raw materials for pigs feed and the influence of the following treatment in its digestibility. This showed that the fruit-vegetable waste contained a high moisture level of 88.12 ± 1.84%, which involves the need of developing a drying process. The nutritional composition on a dry matter basis showed 65% nitrogen free extract, 13% crude fiber, 12% crude protein, 8% ash and 2% ether extract. To sum up, they were mainly composed of carbohydrates (about 65%), which are the most important source of energy in the swine metabolism, but they had a high fiber level content which can be a limitation because it reduces both the digestibility and the available energy in pig diets. However, a minimum level of fiber is necessary for the digestive tract, being the typical contain in pig diets is about 5% of fiber. Consequently, a diet based on vegetable wastes it can be formed and it is necessary take into account other ingredients.

Taking into account these previous experiences, the aim of this study has been to develop a full characterization of the different vegetable wastes generated in the Basque Country to determinate the feasibility of their inclusion in feed formulation. This characterization has involved making a full inventory of the reusable vegetable fractions and, since all fractions are identified, taking samples of each by-product to send them to analyse in laboratory. It has been made a physic-chemical analysis to proof the hygienic aspects of the new raw material and the profitability to be converted into animal feed. To achieve it, it has been necessary to take representative samples so it has been developed a sampling plan and a sampling protocol. All samples has been taken on site and analysed, mostly, in the laboratories of AZTI.

After achieving the aim of this study, proving the feasibility of using vegetable wastes as raw material for animal feed, it will be designed a full Action Plan which guaranties an appropriate management which avoids any change in the nutritional composition or the production of undesirable substances. It will involve collection, transport and centralization of all vegetable by-products in a treatment plant. Also, it will be developed a drying process to reduce the water content, and therefore the volume of vegetable wastes, which makes them more stable and profitable for using them as animal feed. This drying process should be energy efficient enough to ensure the economical viability of its implementation and it should also guaranty that it doesn’t generate any change in the nutritional composition or additional undesirable substances.

2. METHODOLOGY

In order to obtain a feasible valorisation alternative for the actual problem of vegetable wastes in the Basque Country, first of all, an identification of each vegetable waste producer sector and quantification and characterization of them have been done. Once the characterization of by-products was done, the relation between by-products nutritional quality and technical requirements with feed-market needs and Legal requirements was studied, comparing nutritional parameters with the feed-market requirements and undesirable substances with legal obligations.

2.1 Identification, quantification and characterization of vegetable wastes

For the identification of vegetable producer industries, an exhaustive consulting of previous studies has been done, searching in public and private databases. Since all industries have been identified, the amount of all different vegetable by-products has been quantified by contacting with the associations that represent all sectors and by making a questionnaire to the main representative companies of each sector in the Basque Country.

Beside, for the characterization of vegetable by-products, a full sampling plan have carried out based on the differences between vegetables on previously identified sectors. This sampling plan involves visiting two or three of most representative enterprises of each sector in order to take representative samples of each by-product. In addition, to study the Seasonal Variability of Market sector, the sampling plan has been divided into three different periods throughout the year: one in spring, the second between summer and autumn and the last one in winter, with the aim of analyse how the composition of each sample varies depending on the months. Also, in order to ensure the representativeness of the samples and to allow comparison between different samples, a sampling protocol has also designed in order to guarantee the fact that all samples have been taken in the same conditions. Following the protocol is described:

- The most relevant companies of each sector have been sampled. The relevance is based on data of public databases and on the consulting made to the associations which represent them.
- In each company, 150 kg of vegetable waste has been sampled to ensure to its representativeness. From each sample, two simple samples of about 10 kg have been taken: A sample has been analysed and B sample has been frozen in case it would be necessary to repeat the analysis.
- Market sector samples have been divided into citric fruits, non-citric fruit and other vegetables in order to study the suitability of separating the different products at source.
- The density of each by-product has also been quantified in order to evaluate the bulkiness they occupy in a hypothetic future transport.
- To ensure the traceability of all samples, they are labelled with the following dates: name of enterprise and industrial sector where the sample is taken, name of vegetable by-product, date when the sample is taken, sample weight, if the sample is A or B and the person who is responsible of sampling.
- If samples are going to be analyzed bellow, they are sent to the laboratory, whereas if they are going to be analyzed later, such as sample B, they are frozen to 20 degrees below zero until they are to be analyzed. Before sending samples to the laboratory, all of them must be homogenized by crushing them.

2.2 Measuring viability

For measuring the viability of using vegetal wastes in animal feed, first of all, an exhaustive literature search has been done in order to explore feed-market requirements and, secondly, these requirements have been contrasted with the EPEA (Feed Manufacturers Association of the Basque Country), that represents to 11 feed manufactures placed in Basque Country. As conclusion, a list of analytical parameters which must be measured has been made.

On the other hand, a full search of legal requirements has been done in order to know which the forbidden substances for raw materials for animal feed are and which their maximum legal limits. The list of these undesirable substances that must be analysed has been made with the help of the Foundation ELIKA; a Basque technological center scoped to food and feed security.

3. FULL INVENTORY OF VEGETABLE WASTE PRODUCTION:

The level of concreteness of this study includes data of all enterprises of each sector: names, contact data and their production data. However, due to the confidentiality required by the Date Production Law, it only can be published data with a level of detail related to the sector, so the results reported below are limited to each sector.

The different vegetable wastes sectors identified have been Markets, Potato, Cider, Wine, Horticulture, Bread and Coffee and the vegetable by-products that are produced by each sector are fruit-vegetable rest, potatoes rest, pressed-apple, grape-pressed and grape-bunch, vegetable cuts, bread rest and coffee husk.

The geographic dispersion of vegetable by-products production has been studied by the number of centers of each sector in which the quantity of each fraction is divided (Table 1). The need of development of a collection Plan makes this geographic dispersion very important and it is one of the key to profit the reuse of these vegetable wastes as raw material for animal feed due to the high cost of transportation nowadays.

On the other hand, the quantity of each vegetable fraction has been estimated (Table 1) and, in general, the total volume generated in Basque Country is more than 25,000 tons.

TABLE 1 **Vegetable Waste Production**

Sector	**Number of centres of Each Sector**	**Vegetable Wastes tones**
Markets	857	4201
Potato	27	7500
Cider	133	3.626
Wine	209	6981
Horticulture	64	200
Bread	305	3275
Coffee	24	37

In most sectors, the vegetable waste production is stable throughout the year, but there are two sectors in which this production is limited to a few months per year: the Cider sector limits its production from September to December and the Wine sector is limited from September to October, so these by-products are only available at that time.

TABLE 2 **Seasonal variability of Cider and Wine Sector vegetable wastes production**

Sector	**September**	**October**	**November**	**December**
Cider	880,6	1215,6	1215,6	314
Wine	3650	3331	-	-

4. ANALITYCAL CHARACTERIZATION OF VEGETABLE WASTES FRACTIONS:

A full list of parameters, divided in undesirable substances and nutritional parameters, has been analyzed for the characterization of vegetable wastes fractions.

On the one hand, undesirable substances are also divided in groups with the following analytical results:

- ***Nitrites levels*** are above the maximum legal limit for raw materials for animal feed formulations in two cases: in coffee husk with 33,3 mg/kg and grape pressed and grape bunch with 28 mg/kg ± 28, being the legal maximum limit of 15 mg/kg, so it is necessary to control these parameters exhaustibly if they are going to be used in animal feed. For the remaining sectors, all nitrite levels are below the maximum legal limit.
- ***Myco-toxins*** analyzed are: Aflatoxin B1, B2, G1 y G2; Zearalenone; Deoxynivalenol; Ochratoxin A; Fumonisin B1 y B2; T-2 toxin; HT-2 toxin and Patuline. All results show low levels below maximum legal limits. In addition, all of them are also below the detection limit of the method, except in case of Markets sector in which Patuline level is 43,28 µg/kg ± 254,71.
- ***Pesticides*** analyzed are: Aldrin; Clordane; DDT; Endosulfan; Endrin;, HCH; Hexachloro cyclohexane; Heptachlor and Hexachloro benzene. All results show low levels below maximum legal limits. In addition, all of them are also

below the detection limit of the method, except in case of Cider and Wine Sectors in which Hexachloro benzene levels are 5,0 x 10^3 mg/kg and 2,5 x 10^2 mg/kg respectively.

- ***Heavy metals*** analyzed are: Lead; Cadmium; Arsenic and Mercury. All results are below maximum legal limit.
- ***Microbiological analyzes***: Salmonella and Listeria are absence; *Escherichia coli*, *Staphylococcus aureus*, *Clostridium perfringes* and Sulphite Reducing Clostridiums are below the detection limits of the method; Aerobic mesophilics, Enterobacteriaceae, Total coliforms and Molds-Yeast are below maximum legal limits.
- ***Dioxins*** have been analysed in a sample made of a mixture of all by-products and all results are below maximum legal limits.
- On the other hand, a complete list of nutritional parameters has been analyzed, being the following results the most important ones:
- ***Moisture***: Markets and Horticulture's by-products have the highest level of moisture with more than 85%, followed by Cider and Potato's with about 75% and Wine's with more than 65%. However, Bread and Coffee's by-products have lower levels with about 20% and 3,5%, respectively.
- ***Ash*:** All vegetable by-products ash levels are below 0,7%, except Bread and Coffee's, with a level of ash of more than 2%, and Wine's, with 1,5%.
- ***Protein***: Coffee and Bread' by-products protein levels are the most interesting with 17% and 10% of protein, followed by Wine's with 3%. The other sector's protein levels are below 1,5%.
- ***Fiber***: Coffee, Wine and Cider's by-products fiber level are 37,68%, 11,12% y 7,31% respectively, being the other by-products levels below 1,5%.
- ***Starch***: Potato's by-product starch level is 12, 6%.
- ***Fat***: Wine and Cider's by-products fat levels are about 2,5 %, followed by Bread with 1,2%. The other by-products fat levels are below 0,7%.
- ***Sugars***: Markets and Cider's by-products sugars levels are about 5%, followed by Wine and Bread's with 3,5% and 1,7 % respectively. The other by-products sugars levels are below 1%.
- ***Caffeine***: Coffee's by-product caffeine level is about 1,21%.
- ***pH***: Bread, Coffee and Potato's by-products pH levels are about 5,5 units, Markets and Cider's about 3,5 units and Horticulture and Wine's about 4,5 units.

5. CONCLUSIONS

Before drawing conclusion, it is necessary to take into account that analytical results have been obtained from fresh samples with different moisture level depending on each by-product. In this way, according to the Technical Committee of the EPEA (Feed Manufacturers Association of the Basque Country), for including vegetable by-products in feed formulations it is necessary to reduce the moisture below 10%, which means that composition can vary because of drying process, making necessary that the conclusions that have been obtained are related to the moisture content.

The full inventory has concluded that the amount of vegetable by-products produced in a year is big enough to supply to the Basque animal feed market, considering its inclusion in feed formulation limited to 3-6%. However, it is necessary to take into account that geographic dispersion of their production is important and makes necessary to develop an efficient management plan which includes collection, transportation and centralizing them in a treatment plant. Also, it must minimize the contamination with external substances and guarantee their feasibility for feed formulation.

According to analytical results of undesirable substances, it can be concluded that, in spite of the fact that almost all vegetable by-products are nowadays stored for several days in containers outdoors without any refrigeration, the microbiological analyzes and myco-toxins and dioxins levels are below maximum legal limits, as well as pesticides and heavy metals levels. However, high nitrites levels in Coffee and Wine's by-products have been found, making necessary to control nitrites levels in these by-products. In general, it can be concluded that it is feasible to use these vegetable by-products as raw materials for animal feed considering that they set the maximum legal limits.

The nutritional composition has concluded that there are important differences depending on each by-product. In general, it can be concluded that, according to the Technical Committee of the EPEA, the great bulkiness of vegetable wastes respect to the percentage of most important nutritional parameters, such as protein, limits the inclusion of this raw material in feed formulations to between 3 – 6%. Any way, in general, the vegetable wastes moisture is too high to include them in feed formulations, so it is necessary to develop a drying process that reduces water level and bulkiness.

Analyzing each by-product separately, it has been concluded that Bread and Coffee by-products are the most interesting due to their nutritional composition. According to the Coffee husk, it is highlighted its protein and fiber level, of 17% and 37% respectively, related to its moisture level of 3,5% which makes no necessary to submit them to a drying process. However, its caffeine level of 1,21% and the quantity of coffee husk in Basque Country limited to 37 tons a year, according to the full inventory made in its characterization, limits its availability for feed manufactures. On the other hand, Bread rests are interesting due to their protein and sugars levels of 10% and 1,7% respectively related to its moisture level of 20%. In addition, unlike coffee husk, the quantity of Bread rest produced during a year in Basque Country is of about 3275 tons, which makes them more feasible for feed manufactures.

Other sectors, such as Wine, Cider, Markets, Potatoes and Horticulture, are also interesting for feed manufactures. Wine's by-product composition is interesting due to its protein level of 3%, completed with 11% fiber level, 2,5% fat level and 3,5% sugar level, and other by-products presents about 1,5% protein level but related to a high moisture level of about 85%, in case of Markets and Horticulture's, and about 75% in case of Potato and Cider's by-products. In this sense, these high moisture levels make necessary to submit them to an exhaustive drying process, which can increase the final cost of the use of these raw materials as animal feed, making them less feasible for feed manufactures.

To sum up, it can be concluded that, in general, it is feasible the valorisation of vegetal by-products as raw material for feed formulation but, however, there are some critical points that it is necessary to solve to make them useful for animal feed manufactures, such as their high level of moisture and the cost of the drying process, which can make the treatment economically infeasible unless the drying process would be highly efficient, and nitrites levels of Coffee and Wine's by-products, which is necessary to control.

6. ACKNOWLEDGEMENTS

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Session 13

(2) EFFECTS OF ALKALINE PRETREATMENT ON THE ANAEROBIC DIGESTION PROCESS OF VEGETABLES WASTES

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EXECUTIVE SUMMARY

Methane is produced by methanogenic bacteria from acetic acid, hydrogen and carbon dioxide and from other substrates of which formic acid and methanol are the most important (Bouallagui et al., 2005) during the anaerobic digestion of organic biomass. The objectives of this study consisted in setting up laboratory–scale biochemical methane potential (BMP) assay digesters at mesophilic conditions to assess the effectiveness of alkaline (NaOH) pretreatment in improving the anaerobic digestion process for biogas production from mixed vegetable wastes. The parameters monitored were cumulative biogas generation, pH, temperature, Total COD, Soluble COD, total solids (TS), volatile solids (VS) and volatile fatty acids (VFA) with time. Mixed vegetable wastes (120.6±6.00g) were subject to anaerobic digestion after alkaline pretreatment with NaOH(aq). A total solid (dry matter) to liquid ratio was 10.09±0.14% (w/w) and inoculum to wet feed ratio 30.14±1.67% (w/w). Digesters (run in duplicates) were incubated at 30±0.1ºC. Waste activated sludge was used as inoculum in this study and had been taken from the wastewater treatment anaerobic reactor in a textile factory. Controls (pH 7.01±0.67) showed a decrease in VFA to 378±25 meq/L, a decrease in COD from 159,420±8,306 mg/L to 143,478±8,306 mg/L and a 38.9±19.7% decrease in TS. The other two experiments were A1 7% NaOH(aq) (w/w) and A2 12% NaOH(aq) (w/w). For A1 at an average pH of 6.28±1.14, a 34.2±10.2% decrease in COD from 160,870±3,682 mg/L, 18.8±7.56% decrease in SCOD, 85.9±6.8% decrease in VFA from 1,128±416 meq/L, 50.5±5.8% decrease in TS and 52.9±14.6% decrease in VS were recorded. For A2 at an average pH of 6.25±1.12, a 19.1±9.52% decrease in COD from 166,667±15,868 mg/L, 12.6±8.04% decrease in SCOD, 88.9±36.6% decrease in VFA from 1,125±412 meq/L, 45.2±25.6% decrease in TS and 50.0±16.1% decrease in VS were deduced. The average cumulative volumes of biogas produced after 35 days of anaerobic digestion were 285±7.0 mL for the control, 234±5.0 mL for A_2 and 650±11 mL for A_1. In this study, it has been found that chemical treatment of vegetable wastes with alkali ($NaOH_{(aq)}$) enhanced biogas yield over a 35–day retention period of anaerobic digestion. A limitation which has been observed to the anaerobic digestion of the fruit and vegetable wastes studied here was a high volatile fatty acids production, which may have stressed and inhibited an optimum activity of the methanogenic bacteria (Ghasimi et al., 2009; Schievano et al., 2010). On the whole, it may be deduced that alkaline pretreatment with NaOH has had a positive effect in improving the rate and yield of biogas production (López Torres and Espinosa Lloréns, 2008; Lin et al., 2009).

1. INTRODUCTION

The anaerobic digestion (AD) of biomass for biogas production could eventually substitute fossil fuel–derived energy and reduce environmental impacts by providing a clean fuel from renewable feedstocks, such as energy crops, organic fractions of municipal solid wastes, and a variety of agro–industrial wastes (Borjesson and Mattiasson, 2008). AD is characterised by a series of biochemical transformations mediated by different consortia of bacteria. Organic materials of the substrate–like cellulose, hemicellulose, lignin must be liquefied by extracellular enzymes, then is treated by acidogenic bacteria; the rate of hydrolysis depends on the pH, temperature, composition and concentration of intermediate compounds (Fantozzi and Buratti, 2009). Then soluble organic components including the products of hydrolysis are converted into organic acids, alcohols, hydrogen and carbon dioxide by acidogens. The products of the acidogenenesis are converted into acetic acid, hydrogen and carbon dioxide.

Methane is produced by methanogenic bacteria from acetic acid, hydrogen and carbon dioxide and from other substrates of which formic acid and methanol are the most important (Bouallagui et al., 2005) during the anaerobic digestion of organic biomass.

The process is catalysed by a consortium of microorganisms (inoculum) that converts complex macromolecules into low molecular weight compounds (methane, carbon dioxide, water and ammonia). However, recalcitrant organic substrates are normally pretreated in several ways to increase the bioavailability of the readily hydrolysable fractions and thus increase the rate of the rate determining hydrolysis step. In this context, the objectives of this study consisted in setting up laboratory–scale biochemical methane potential (BMP) assay digesters at mesophilic conditions assess the effectiveness of alkaline (NaOH) pretreatment in improving the anaerobic digestion process for biogas production from mixed vegetable wastes. Acid or alkaline hydrolysis can be applied as a pretreatment to enhance the anaerobic biodegradability of a recalcitrant waste. Alkaline hydrolysis at ambient temperatures has been proposed as a chemical pretreatment more compatible with the AD process, since the bioconversion generally requires an adjustment of pH by increasing alkalinity. The aim has been to obtain and process sets of data in order to identify and analyze the major variations of physical process parameters namely biogas generation, pH, temperature, Total COD, Soluble COD, total solids (TS), volatile solids (VS) and volatile fatty acids (VFA) with time.

2. MATERIALS AND METHOD

2.1 Experimental BMP assays

The Biochemical Methane Potential (BMP) test is one of the most relevant tests for assessing the biodegradability of waste materials (Lesteur et al., 2010; Paepatung et al., 2009). The BMP test is run under anaerobic conditions, using bacteria populations, which makes it very time consuming. The BMP test is the most popular but, as with any simple batch test that is intended to provide information on the likely performance of a full–scale continuously operated process, the results of the BMP require interpretation with caution. Three anaerobic biochemical scale reactors (or, BMP assays) were set up. Nutrient broth of trace elements was supplied for stable digestion. The first set up was a control reactor representing the typical anaerobic digestion (TAD) of solids and the other two were additionally involved chemical pretreatment with alkali (NaOH) at different concentrations (**Table 1**). Sodium hydroxide (pH 12) at concentrations of 7% and 12% (w/w) were used to pretreat 120 g substrate at a high solids loading rate. Pretreatment assays were carried out in a 2–L reactor using a solid (dry matter) to liquid ratio (distilled water) of 10% (w/w) and inoculum of 30% (w/w) added to the digesters. Each digester contained 120±6 g of substrate mixed with water, inoculum and sodium hydroxide at the respective concentrations. One digester was labeled as control with only substrate, water and inoculum. The bioreactor was a plastic–made airtight system of volume of 2L. **Figure 1** shows the schematic representation of BMP–assay bottle with the biogas collection system. After filling, the head space above the slurry of substrates in the assay bottles was purged with nitrogen gas to ensure as anaerobic as practicable condition in the bottles. The digesters were wrapped with black paper to prevent sunlight penetration (Wu et al., 2008).

TABLE 1 **Composition of each BMP assay**

	Control	**A_1**	**A_2**
Substrates mix used (g)	120±6	120±6	120±6
$NaOH_{(aq)}$ used (w/w)	No NaOH	7%	12%
Temperature/°C	29.7	29.8	30.1
% Inoculum used (w/w)	30±1.5	30±1.8	30±1.7
% Water used (w/w)	10±0.5	10±0.5	10±0.5

2.2 Substrates and inoculum

Vegetable wastes, from a market in Port Louis (Mauritius), were collected as substrates for the anaerobic digestion experiments. The substrates used in this study consisted of carrot tops, beetroot (top and flesh), cabbage and cauliflower leaves. Samples of each type of waste at 40±3 g were brought up to 120±6 g in each of the bioreactors and were thoroughly mixed. The substrates were mechanically crushed prior to characterization and the alkaline pretreatment in the biochemical potential assays.

For new substrates with unknown degradation characteristics, a number of different dilutions of the substrate (with distilled water) are required.

Dilutions ensured that the methane potential of the substrates would not be underestimated due to overload or potential inhibition. When the maximum methane potential is the same in at least two different dilutions of the dilution series, it can be assumed that the inoculum is not overloaded or inhibited.

If the specific potential continues to increase with increasing dilution (decreased concentration of substrate), additional dilutions are required.

Samples of each waste at 40±4 g were brought up to a total mix of 120±6 g in each of the bioreactors and were thoroughly mixed. The substrates were mechanically crushed prior to characterisation and the alkaline pretreatment in the biochemical potential assays.

Waste activated sludge was used as inoculum in this study and had been taken from the wastewater treatment anaerobic reactor in a textile factory. Immediately after sampling, the sludge was dispensed into 500 mL plastic bottles, and then stored at –4 ºC. The sludge was collected a day before the BMP assay set up and the required portion of the sludge was thawed at room temperature and used in the experiments as inoculum.

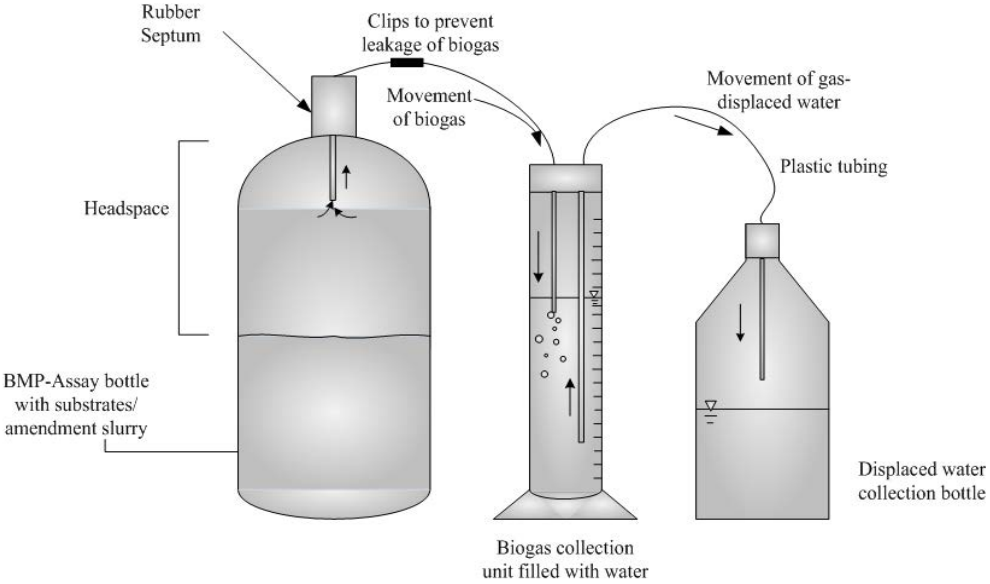

Figure 1 **BMP assay bottle with biogas collection system**

2.3 Analytical methods

Total solids (TS) of samples was determined according to procedures in APHA (1999) section 2540 D. Volatile solids (VS) content was determined following the loss on ignition method BS1377 (i.e. ignition of samples at 550 ºC for 3 hours in a muffle furnace). pH was read using a pH probe and was read off directly from the pH display screen. Temperature also was read off directly from the display screen of the pH meter. Volatile fatty acids (VFA) levels were determined using a combination of volumetric (titration) analyses and the TITGRAM software. After titration analyses the data were entered in the pc program TITGRAM to be able to determine the values of VFA (Heerah et al., 2008). Total chemical oxygen demand (TCOD) was determined following the standard method from APHA (1999). Soluble COD (SCOD) was measured in the same manner as total COD (TCOD), except that samples were centrifuged at 2000 rpm for 30 minutes (centrifuge model Denley BS400), then filtered through a 0.45–μm filter before analysis and the filtrate was also diluted. All analytical tests were conducted in triplicates. The liquid displacement method was employed for measuring the volume of biogas produced. The biogas from the reactor passed to this liquid filled vessel thus displacing an equivalent liquid volume. The amount of biogas was then determined from the graduation marks on the measuring cylinder.

3. RESULTS AND DISCUSSIONS

The reactor fed with 120±6 g substrate maintained at room temperature failed to continue with biogas production after 10 days where high volatile fatty acids (VFA) formation lowered pH of slurry to <5.00 and ultimately lead to formation of thick scum layer on the top of the substrate. For stable digestions, it is imperative that a satisfactory ratio be maintained between VFA and alkalinity levels (Rao and Singh, 2004).

At high organic loading it was very difficult to avoid forming a thick scum layer of undigested solids on the surface of the digesters. Therefore, in the present study, inadequate mixing of substrate and presence of higher concentrations of volatile fatty acids in the reactor might have caused formation of thick scum layer of undigested solids on the surface of the reactor (Rao and Singh, 2004).

Degradation of substrate started almost immediately for reactor A_1 (7% (w/w) $NaOH_{(aq)}$) and A_2 (12% (w/w) $NaOH_{(aq)}$) and proceeded without problems in the reactors maintained at ambient temperature. However, for the reactor without pretreatment, it took about 7 days for initiation of biogas production.

The cumulative biogas production at different sodium hydroxide, $NaOH_{(aq)}$ concentration maintained at room and ambient temperature along with the control. The rates of biogas production differed significantly according to the concentrations. It has been observed that bulk of substrate degradation took place up to a period of 10 days suggesting that the digesters should preferably be run at a digestion time close to 10 days for optimum energy yield.

3.1 Total solids and volatile solids

Preliminary analyses indicated that the ratio of water, bulk substrate and inoculums to be added should be in a 1:1:1 (v/v) ratio in order to achieve a total solids content of 6–12% (Heerah et al., 2008). Conventional anaerobic digesters require feed material with total solids content below 10%. Modern systems can deal with >20% total solids content in the feed. Anaerobic digestion in semi–dry (total solids content of 10–20%) and dry conditions is considered capable of producing an inert bio–solid product with higher methane production (Forster–Carneiro et al., 2009). At the start of the experiments, the control (C_v) had a higher %TS of 8.18±1.61 whereas A_1 and A_2 had %TS of 6.46 and 6.20 respectively. The %TS shooted on the 8th and 17th day and A_1 had the highest %TS on day 17 at 10±2.31 (**Figure 2**).

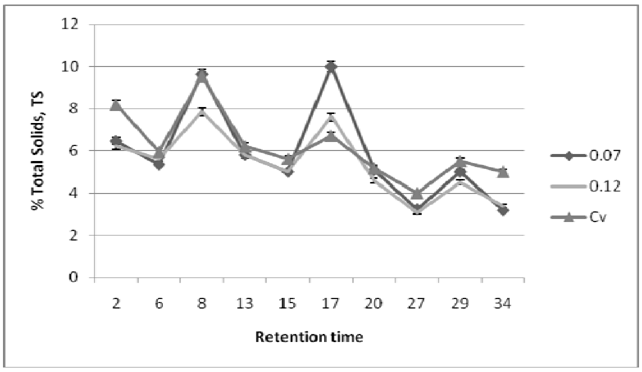

Figure 2 **Variation of total solids**

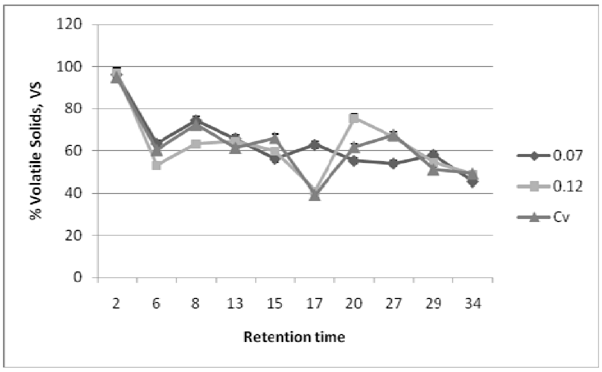

Figure 3 **Variation of volatile solids**

The volatile solid content can give an estimate on the amount of substrate that can potentially be turned into methane. It only gives an estimate because volatile solids are made up of different organic compounds that have varying degradability speeds. **Figure 3** shows the fluctuations of volatile content value. Based on observation until 34th day, it can be seen that there was decrease of volatile content value. At the start the %VS was relatively high for the three assays with 96.2%, 97.0%, 95.0% for A_1, A_2 and control respectively (**Figure 3**). The reactor A_1 had the best efficiency of volatile content decrease with 52.9%, followed by A_2 50.0% and control 48.4%. High volatile content in biological process is good because it guarantees the material stock as an energy resource and microorganism's growth. Waste characterised by high VS and low non–biodegradable matter is best suited to AD treatment. VS are an important parameter for measuring biodegradation, which directly indicates the metabolic status of some of the most delicate microbial groups in the anaerobic system. Bouallagui et al. (2005) reported that fruit and vegetable wastes (FVW) contain 8–18% total solids (TS), with a total volatile solids (VS) content of 86–92%. Higher VS concentration in the effluent (sludge) should possibly be related to a certain accumulation of VFA in the effluent, especially propionate, which degradation tends to be slower than the rest (Pind et al., 2002). Apparently, the higher the VS fed, the higher the

VS removed, and the higher the biogas production. The same pattern described for biogas production applies to methane production.

3.2 Volatile fatty acids (VFAs)

VFAs accumulation can lead to a drop in pH which inhibits the microorganisms. A continual drop in pH can ultimately cause failure in an anaerobic digester. VFA are needed in small amounts as part of an intermediary step for the metabolic pathway of methane production by the methanogens. Lower values for biogas production could be related to process inhibition caused by initial accumulation of VFA.

The concentration of VFA in the slurry 2 days of alkaline pre–treatment was remarkably high (1,128.0 meq/L for A_1 at pH 4.78, 1,125.3 meq/L for A_2 at pH 4.62 and 1,068.7 meq/L for control at pH 4.85) (**Figure 4**). The three experiments showed a decreasing trend. A_1, A_2 and control ultimately decreased to 159.2 meq/L, 124.6 meq/L and 377.9 meq/L respectively. The graph shows an increasing trend during the first 8 days. The VFA for A_1 increasing from day 2 to 1,220.1 meq/L then showed a small decrease to 1,205.3 meq/L on day 8 which differed from that of A_2 and control.

A_2 and control had an increasing trend in the values during these days, VFA increased from 1,191.5 meq/L to 1,215.2 meq/L and 1,098.3 meq/L to 1,275.4 meq/L, respectively. The graph then showed a sharp decrease on the 13th day and this decreasing trend continued till the last day of retention. This trend contradicted the expectations that greater bioavailability of soluble organic substances would result in higher VFA concentrations. The data obtained here compete with the results reported by Park et al. (2005), who found that enhanced anaerobic digestion resulted in higher VFA production. However, the VFAs produced in digestion are generally utilised by the methanogenic bacteria, so it is possible that a portion of the VFAs produced were utilised and did not remain available for measurement. Song et al. (2004) also reported that VFA concentrations decreased in line with CODs during mesophilic sludge digestion and stoichiometrically linked their VFA concentrations to an increase in methane production.

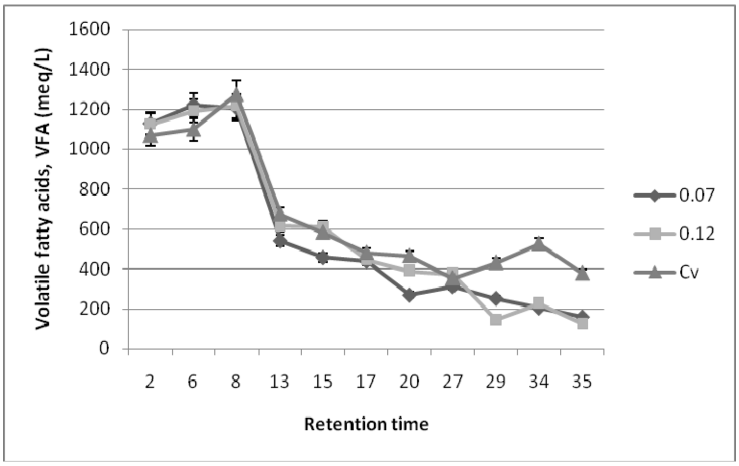

Figure 4 **Evolution of the volatile fatty acids levels in experimental runs**

3.3 pH

During the first few days decrease of pH was noted, it showed there were accumulated volatile acids in high amount in the reactors. A decreasing pH can point toward acid accumulation, which typically occurs if there is an overload of volatile acids in the digester. The acidogenic bacteria then thrive, producing more organic acids and lowering the pH. On day 0, the pH was 6.28, 6.25 and 7.01 for A_1, A_2 and control, respectively. On day 1 it decreased to 6.01, 5.88 and 5.86, respectively which further decreased to pH <5.00 on day 2 (**Figure 5**). After 6 days all of reactor did not stable for reactors with overall range pH 4.63–4.77. It indicated that concentration of volatile acids was still high and afterwards the decreasing concentration indicated possible acidification process will end and going to methanogenesis process.

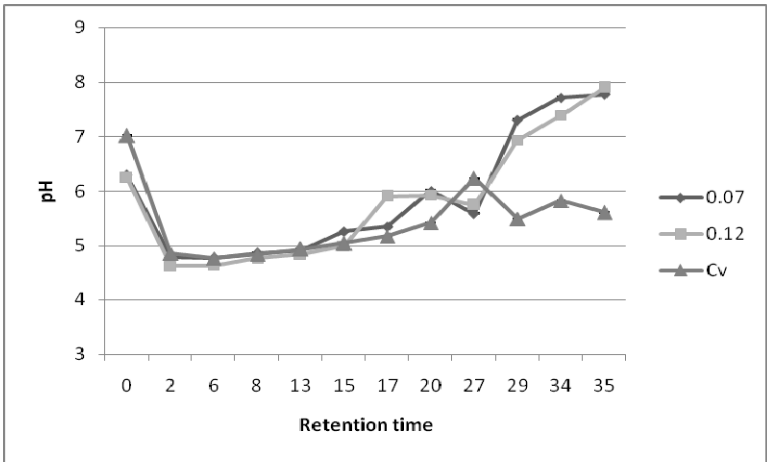

Figure 5 **Evolution of pH for the BMP assays**

3.4 TCOD and SCOD

TCOD concentration for all reactors was high at 166,088–212,438 mg/L (**Figure 6**). A large concentration of TCOD was noted because the substrates used fresh organics so there was no degradation process started yet. Both TCOD and SCOD for the three assays showed a general decrease with time (**Figures 6** and **7**). For A_1, an initial TCOD from 160,870±53,682 mg/L started decreasing to reach 105,797 mg/L on day 34; for A_2 it decreased from 166,667 mg/L to 134,783 mg/L and for the control (Cv) it reached 143,478 mg/L around day 27–29. SCOD also showed a decreasing trend. A decrease of 18.8% in SCOD was determined for A_1, 12.6% for A_2 and 9.43% for control. However, it was seen that there was a decrease of COD concentration in all the reactors. The degree of solubilisation of a substrate/mixture of substrates can be estimated from the SCOD to TCOD ratio (Ryu et al., 2008). The SCOD/TCOD value was high at 0.797 on day 13 for the control. Set up A_2 showed the lowest SCOD/TCOD ratio on day 15 at 0.588. Set up A_1 had a SCOD/COD in the range 0.634–0.785.

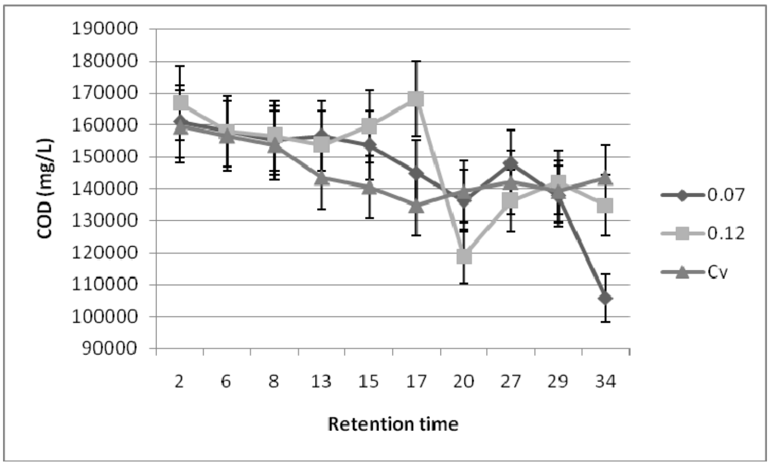

Figure 6 **Variation of TCOD**

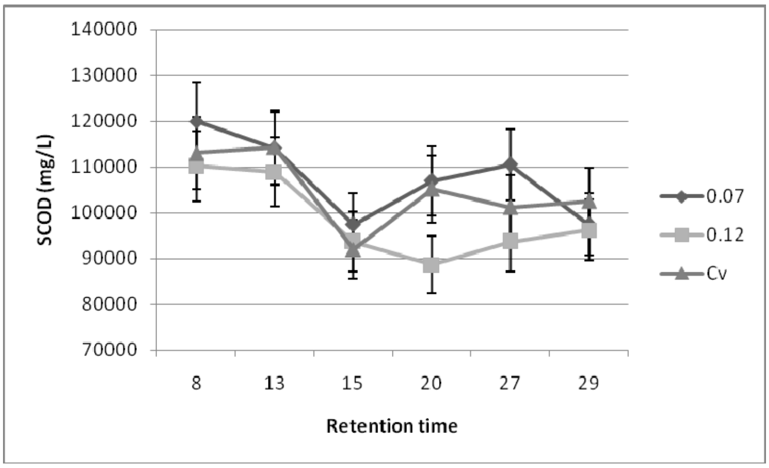

Figure 7 **Variation of SCOD**

3.5 Biogas generation

Methane gas is a major by-product of anaerobic degradation of organic solid and liquid wastes. Cumulative biogas production occurred for all three assays but there were differences in the yields, lag times, and rates (**Figure 8**). The initial biogas production rate (indicated by the slope of the curve) up to day 2 was highest for A_1, and was 7 days for the control set up. Set up A_1 had the highest biogas production of 650 mL. Set up A_2 yielded 234.5 mL of biogas. Comparing the pretreated systems, the set up with 7% (w/w) $NaOH_{(aq)}$ had produced a higher biogas yield than with 12% (w/w) $NaOH_{(aq).}$

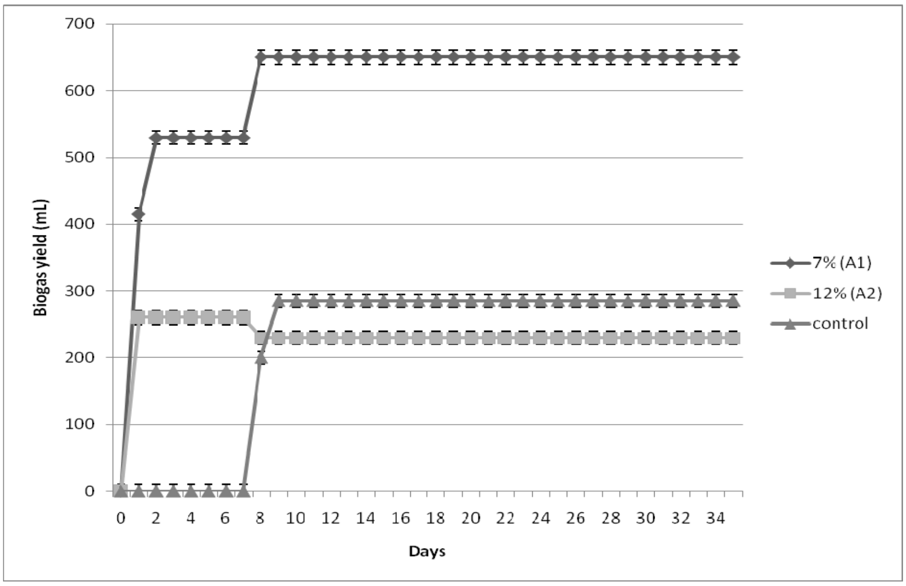

Figure 8 **Cumulative profile of biogas production for 34 days**

4. CONCLUSION

The anaerobic digestion of organic material is a complex biochemical process, involving many intermediate compounds and reactions, each of which is catalyzed by specific enzymes or catalysts (Malakahmad et al., 2009). In this study, it has been found that chemical treatment of vegetable wastes with alkali ($NaOH_{(aq)}$) enhanced biogas yield over a 35–day retention period of anaerobic digestion. A limitation which has been observed to the anaerobic digestion of the fruit and vegetable wastes studied here was a high volatile fatty acids production, which may have stressed and inhibited an optimum activity of the methanogenic bacteria (Xu et al., 2010). On the whole, it may be deduced that alkaline pretreatment with NaOH has had a positive effect in improving the rate and yield of biogas production.

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(77) CAN TWO-STAGE ANAEROBIC DIGESTION IMPROVE ENERGY RECOVERY FROM BIOMASS?

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EXECUTIVE SUMMARY

Biogas production from agriculture, farming and food-industry residues and waste is getting more and more important in the field of renewable energy sources. The two-stage anaerobic digestion (AD) allows producing both biohydrogen and biomethane (Wang et al., 2003) and shows to have other advantages compared to the AD process (one-stage). For example, it promises to be a possibile solution to increase the overall energy recovery from biomass, as shown in studies carried out on carbohidrate-rich substrates (Kreamer & Bagley, 2005). It, also, has been proven that two-stage AD, thanks to different conditions, allows a better reduction of pathogenic bacteria in the output (Bendixe, 1994). In this work, an attempt of comprehensively compare the two- and one-stage AD processes was done. Using different organic matrices we simulated the operation of a two-stage plant for biogas production. The aim of this work was to compare the two-stage and the one-stage processesin termsofoverall energy recovery. .

For this purpose we used four organic matrices: corn silage (CS), rice middling (RM), olive pomace (OP) and fruit/vegetable wastes (FVW), diluted with pig slurry. The two-stage process was simulated by semi-continuous bioreactors; batch methanogenic reactors were run to measure the potential methane production of the output of the hydrogenic reactors and, in parallel, of the raw feeding mixtures.

Higher energy recovery was obtained by the two stage systems as compared to the one-stage, for three of the matrices analyzed: 11%, 23% and 39% higher respectively for CS, RM and FVW. Olive pomace gave null productivity in the hydrogenic stage, and therefore was not further considered. .

From the results obtained, we can state that the two-stage anaerobic fermentation can beinteresting as an alternative to the classic single-stage fermentation process, as a possivle way to increase energy recovery.. However, the two-stage process can be influenced by the characteristics of the feed mix (chemical composition, quality and accessibility of organic matter…) and the process conditions, expecially in the hydrogenic stage.

This preliminary study lays the basis for further work on the improvement of the two-stage AD process. A deeper physical and chemical characterization of the feeding biomass and of the hydrogenic stage output will be required, because it is a highly sensitive process influenced by several variables. As we demonstrated, different combinations of process conditions may lead to completely different energy recovery yields of the whole two-stage anaerobic digestion process.

Other important aspects for future development should be the effectiveness of pre-treatment strategies (thermal, chemical, physical and enzymatic) to increase the availability to microbial activity of the most recalcitrant fractions of organic matter, such as ligno-cellulose, and the characterization of the microbial composition of the consortia in the two stages.

1 INTRODUCTION

Growing energy needs and attention to renewable energy sources caused biogas production (both biohydrogen and biomethane) to become increasingly important, giving a chance to exploit organic wastes from different productive sectors (Tinche, 2008).Biomethane has widely been used to produce electricity and thermal energy through cogeneration. It can also be used as fuel for cars and household warming.

Something different must be said regarding biohydrogen. In fact, despite hydrogen use and management still show some technological issues to be solved, it has a very high environmental compatibility (its energetical conversion comes with almost no dangerous emissions) and calorific power, thus representing one of the most promisin energetical vectors. Hydrogen can be used as fuel in fuel cells to produce electricity with high conversion yields, or mixed with other fuels to improve combustion efficiency.

While one-stage anaerobic digestion (AD) is widely used to produce biomethane, a different process (two-stage AD) allows the combined production of biohydrogen too (Wang et al., 2003).

Biohydrogen production takes place in an acidogenic fermentation stage, in which some microorganism species metabolize simple sugars to produce volatile fatty acids (mainly acetic and butyrric acid) and molecular hydrogen (Redwood et al., 2009). This process takes place in conditions easily obtained in biodigestors similar to the traditional ones (55°C, initial pH 5.5) (Guwy et al., 2011).

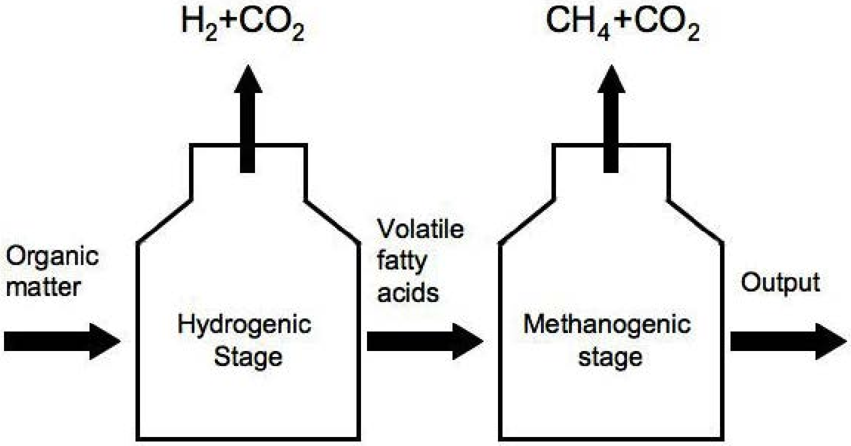

FIGURE 1 **Diagram of the anaerobic digestion process.**

At the end of the hydrogenic stage, the substrate still has a high VFA content, thus representing an ideal substrate for a methanogenic process.

Two-stage AD was reported to allow some benefits compared to one-stage. Hawkes et al. (2002) proved that separating acidogenic and methanogenic stages and using the output from a first hydrogenic reactor to produce methane improved reaction rate and gas production (Blonskaja et al., 2003; Mataalvarez et al., 1993). Subsequent studies on sugar-rich substrates (e.g. glucose) showed that two-stage AD may have higher total energy recovery than one stage (Kraemer & Bagley, 2005)..

1.1 RESEARCH OBJECTIVES

This study aims to take a deeper look at two-stage AD, energy recovery efficiency as compared to the one-stage.

2 METHODOLOGY

To analyze the potential of two-stage process we used different organic matrices: corn silage (CS), rice middling (RM), olive pomace (OP) and fruit/vegetable wastes (FVW),. These materials were mixed with pig slurry to reach a TS concentration of 69-71%. Each waste material has been characterized to evaluate the content of total solids (TS), organic matter (VS), organic carbon (TOC) and pH (TABLE 1).

The two stage AD system was simulated by a semi-continuous bio-reactor for hydrogen production, with hydraulic retention time of around 3 days.

TABLE 1 **Characterization of feed mixtures.**

Mix	**Matrix**	**HRT (d)**	**Mix w/pig slurry (%)**	**TS (g kg^{-1})**	**VS (g kg^{-1})**	**pH**
A	*Corn silage*	3	24	69.32 ±5.50	64.79 ±5.10	5.4 ± 0.21
B	*Rice middlings*	3	12	71.69 ±4.52	75 ± 6.24	7.23 ± 0.06
C	*Olive pomace*	3	28.5	69.31 ± 3.97	118.06 ± 4.82	6.85 ± 0.13
D	*Fruit/vegetables wastes*	3	35	68.65 ± 4.33	52.23 ± 3.74	7.48 ± 0.15

2.1 PRODUCTION OF BIOHYDROGEN

We simulated the functioning of a two-stage plant by Wheaton batch bioreactors (500 ml) with an operative volume of 300 ml, fed once/twice per day depending on HRT. Those reactors has been ermetically closed, linked to bags for gas collection and agitated in a Dubnoff bath at 55°C. We daily checked each reactor pH, gas volume and hydrogen percentage.

2.2 PRODUCTION OF BIOMETHANE

We checked ABP (anerobic biogasification potential, to simulate the functioning of one-stage plants) and rABP (residual anerobic biogasification potential, standing as the second reactor of a two-stage plant). To test ABP we re-created the different feed mixes; 33 g of each were placed in 500 ml vials with 200 ml of inoculum. To test rABP we used outputs from the first stage as alimentation.

Vials were incubated at 55°C; we daily checked the volume of produced biogas and methane concentration.

We analyzed VS, TS, TOC, buffer capacity and VFA on first-stage outputs.

3 RESULTS

TABLE 2 **Volumetric biogas production in the two processes**

	Two-stage			**One-stage**	
	H_2 (L kg_{VS}^{-1} d^{-1})	*CH_4 (L kg_{VS}^{-1} d^{-1})*	*Energy recovery kJ kg^{-1}*	*CH_4 (L kg_{VS}^{-1} d^{-1})*	*Energy recovery kJ kg^{-1}*
A	41.6 ± 1.2	413 ± 4	15066.87±144.9	379 ± 24	13341.28±82.25
B	44.9 ± 3.3	316 ± 3	11688.74±524.37	256 ± 45	9016.62±1593.14
D	96.7 ± 16.9	428 ± 62	16272.69±2391.05	283 ± 14	9945.7±671.33

Analyzing hydrogenic stage productivity, we found that A has an energy recovery of 529 ± 14 KJ kg_{VS}^{-1},B of 571,99 ± 50 KJ kg_{VS}^{-1}, while C produced only 33,77 ± 5,99 $kg_{VS}^{-1}$and D 1232 ± 216 KJ kg_{VS}^{-1}. Regarding methanogenic stage of two-stage process, A had an energy recovery of 14537 ± 130 KJ kg_{VS}^{-1}. Tes B produces 11116 ± 104 KJ kg_{VS}^{-1}, while D 15041± 2175 KJ kg_{VS}^{-1}. Chemical-physical characteristics of olive pomace caused, in the first stage, a production of gas with low content of hydrogen and moderate percentages of methane (10-30%), proving the matrix not worth of a deepening of the comparison between two- and one-stage process.

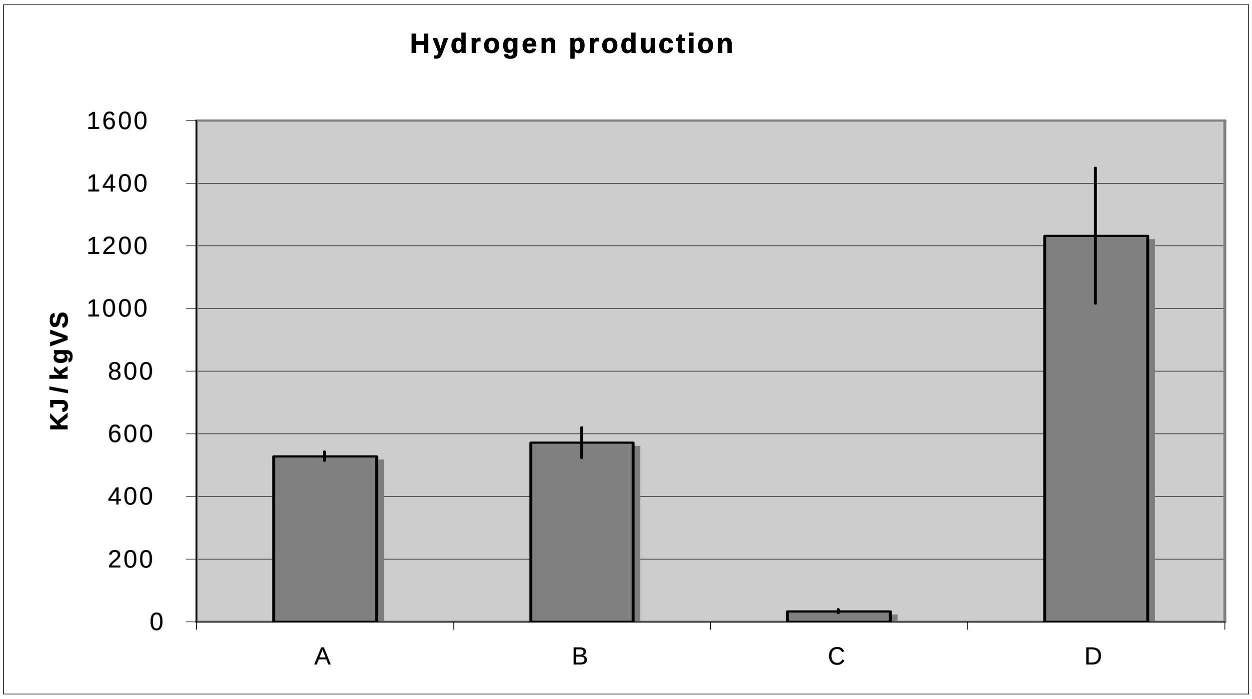

FIGURE 2 **Comparison of the best energy yields in the hydrogenic stage for the used matrices.**

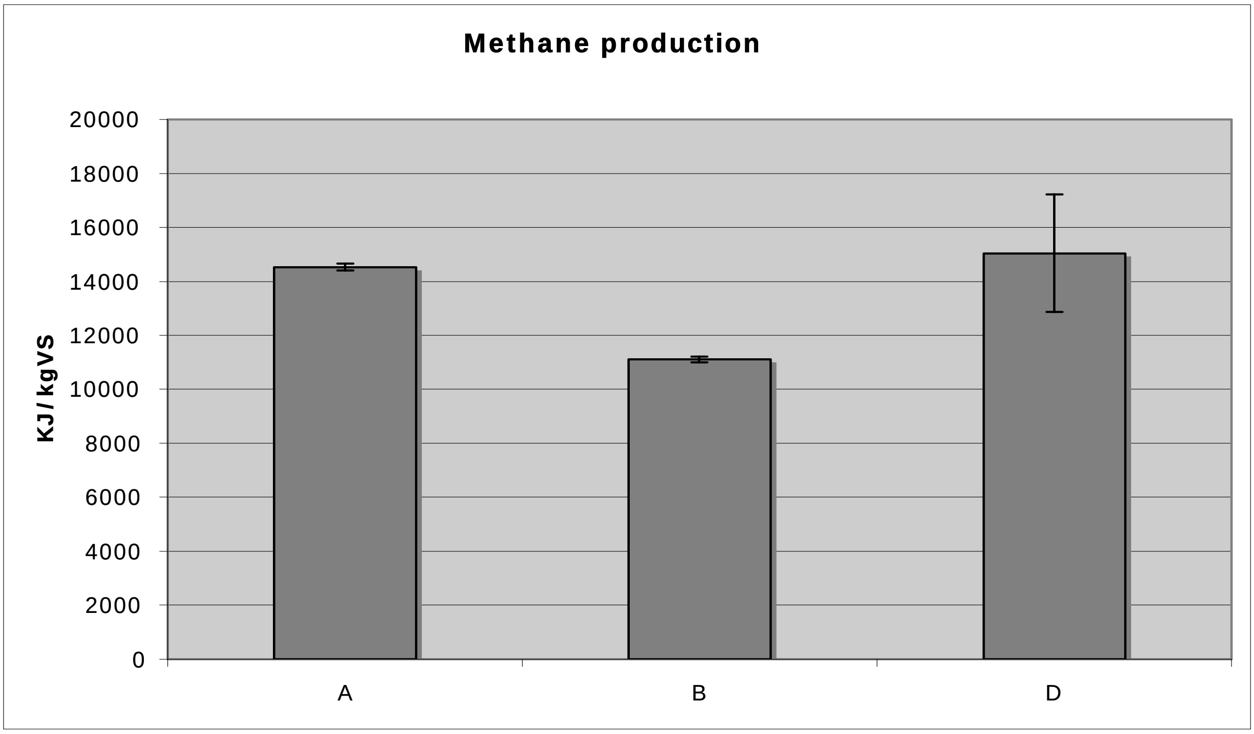

FIGURE 3 **Comparison of the best energy yields in the methanogenic stage for the used matrices.**

If we compare total energy recovery on the two-stage process with that of one-stage, we find that in all tests two-stage allows higher yields than one-stage fed with the same mixes.

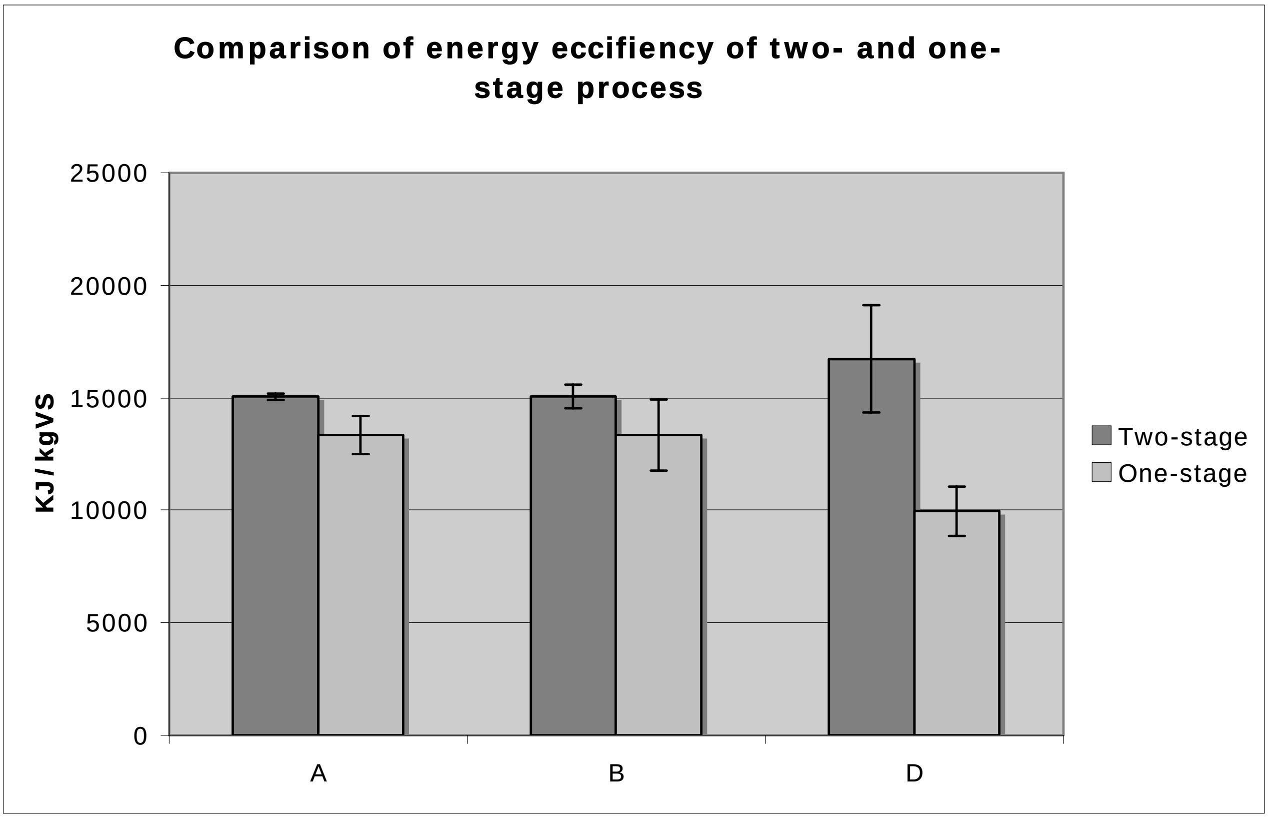

FIGURE 4 **Comparison between energy efficiency of two- and one-stage process**

We therefore can say that, according to obtained results, two-stage anaerobic digestion can represent, for the analyzed data, a good alternative to classic one-stage process to increase energy recovery. This can depend on the fact that the conditions created in the first stage can improve the idrolitic process, givin methanogen bacteria a more readily usable organic matter in the second stage.

This higher yield depends on many factors, especially on the characteristics of feed mixes (matrix used, VS content) and retention time.

4 CONCLUSIONS

This preliminary study can be seen as a base for further analyses on how to improve anaerobic digestion. At a lab scale it will be required a careful chemical-physical characterization of feeding matrices used (both organic matrices and slurry) (Tenca et al., 2011) and outputs of hydrogenic phase. It would also be useful, in order to better monitor the fermentative process, a characterization of the bacterial consortia in the two different stages. Being the hydrogenic stage such a delicate and easily influenced process, a particolar attention in this phas will be needed. In fact, different combinations of the two analyzed variables (HRT and composition of feed mix) lead to very different fermentative processes, going from good production to inhibition, also influencing the following methan production and thus the whole energy production from the organic substrate.

A further step on this study will be the analysis of possible pre-treatments (thermical, chemical-physical and with enzymes) both of the inoculum (Kreamer & Bagley 2007) and of the matrices, to increase the organic matter's availability and so improve produtcivity too.

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(183) COMPARISON OF BATCH AND SEMI-CONTINUOUS ACIDOGENIC PROCESS OF FOOD WASTE ANAEROBIC DIGESTION

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EXECUTIVE SUMMARY

During food waste anaerobic digestion (AD), higher rate of acidogenesis than the methanogenesis affects the stability of the reactor in a single-phase AD system. To decouple the acidogenesis from methanogenesis with the purpose to optimize each reactor separately, in recent years, a two-phase AD is proposed, especially to treat high solid food waste. In the two-phase system, leach bed reactor (LBR) as the first phase, i.e. hydrolysis-acidogenesis, of the two-phase system has gained more attention due to its operational simplicity and efficiency for organic wastes with high solids content. In most cases, the LBRs were operated in batch mode and its efficiency in a continuous loading has not been studied in detail. LBR batch system usually requires longer retention time to completely exhaust the methane production potential; and also requires a large volume of inoculum per batch, which reduces the effective reactor volume resulting in higher operational costs. To overcome this, LBR can be operated in semi-continuous feeding mode and connected to a high rate methanogenic reactor. Since acidogenesis and methanogenesis are mediated by different groups of microbes with different ecological requirements, an understanding of these microbial dynamics offers information to significantly improve the methane production and overall operation. Therefore, the aim of the present study is to examine the stability and sustainability of a two-phase LBR-UASB system operated in semi-continuous feeding mode with different feeding frequencies.

Two LBRs were operated separately with semi-continuous (LBR1) and batch (LBR2) mode of feeding. Initially, 500 g of food waste (FW) (38.5% of total solids), 100 g of AD sludge as inoculum and 0.5 L of water were added to both of the reactors. After 4 days, 50 g of food waste was fed into LBR1 every two days, while 500 g of food waste was fed into LBR2 after 16 days of digestion (two cycles). Leachates were collected at the bottom of the LBR; of which, 75%, was used to feed the UASB while the remaining was recharged to the LBR with pH being adjusted to 6.0 using Na_2CO_3. Periodically, COD, VFAs in the LBR, VFA composition of the leachates and methane production from the UASB were analyzed while the TS removal efficiency and methane yield were calculated at the end of the experiment. Generally, yields of COD (0.86 g COD/g VS_{added}) and VFA (0.33 g COD/g VS_{added}) were higher in LBR1 compared with LBR2. Similarly acetic acid represented 61.4% in the leachates, which were comparatively higher than the 31.9 and 55.2% observed in LBR2 in the first and second cycles, respectively. However, yields of COD (0.84 g COD/g VS_{added}), VFA (0.30 g COD/g VS_{added}) and their corresponding leaching rates were comparatively higher during the second cycle of LBR2 than the yields of COD (0.81 g COD/g VS_{added}) and VFA (0.27 g COD/g VS_{added}) observed during the first cycle; indicating the advantage of digestate as the inoculum over anaerobically digested sludge. Furthermore, the superiority of continuous feeding mode over batch mode was evidenced not only by the increased hydrolysis rate but also the distribution of volatile fatty acids (VFA) composition. UASB1 connected with LBR1 produced higher volume of methane gas than UASB2. The bacterial diversity in LBRs was investigated by PCR-DGGE. *Lactobacillus* sp. dominated during the initial stage of LBR2 and increased diversity with more versatile species appeared in the later stage of digestion. However, the genus *Clostridium* sp., *Acetobacter* sp., and *Bifidobacterium* sp. appeared in LBR1 persistently, which led to the production of extracellular hydrolytic enzyme and yielding acetate as the predominant acid. Results of overall TS removal efficiencies (82.2% in LBR1; 73.3 and 84.1% in 1st and 2nd cycles of LBR2) indicate that the decomposition was almost similar in the 2nd cycle of LBR2 and LBR1).

To conclude, results indicate that leachate with high acetic acid from the semi-continuously fed LBR resulted in higher methane production than the batch mode; however, successive batch operation tended to increase the efficiency of the acidogenesis phase to a level similar to that of the continuous operation.

1. INTRODUCTION

To decouple the acidogenesis from methanogenesis with the purpose to optimize each reactor separately, in recent years, a two-phase leach bed reactor (LBR) coupled with a methanogenic reactor is proposed, especially to treat solid substrates like grass, sugar beet, fruit and vegetable waste, maize and municipal solid waste (Cirne et al., 2007; Cysneiros et al., 2011; Rubio-Loza and Noyola, 2011). A growing interest has been found on the adoption of LBR as the hydrolysis-acidogenic reactor, due to its operational simplicity and efficiency for organic wastes with high solids content (Wang et al., 2005; Park et al., 2008; Selvam et al., 2010). In most of the previous studies, LBRs were operated in a batch mode (Silvey et al., 2000; Nopharatana et al., 2003), which usually requires longer retention time to completely exhaust the methane production potential and also requires a large volume of inoculum per batch that reduces the effective reactor volume resulting in higher operational costs (Nizami et al., 2011) . To overcome this, LBR could be operated in sequential or continuous mode. However, to our knowledge, no trials have been reported on this aspect regarding improving the volumetric capacity and yields of LBR and the impact of various operational conditions on the major functional bacterial community in LBRs. Since acidogenesis and methanogenesis are mediated by different groups of microbes with different ecological requirements, an understanding of these microbial dynamics offers potential information that can be used to devise methods to significantly improve the methane production and overall operation. Therefore, the aim of the present study is to examine the stability and sustainability of a two-phase LBR connected with an upflow anaerobic sludge blanket (UASB) system operated in semi-continuous feeding mode and successive batch mode with different feeding frequencies.

2. METHODOLOGY

2.1. Experimental design and operation

Two sets of laboratory scale LBR-UASB system were used in this study and the detailed configurations of the reactors could be found in our previous report (Xu et al., 2011). Two LBRs were operated separately with continuous (LBR1) and batch (LBR2) mode of feeding. Initially, 500 g of food waste (FW) (38.5% of total solids, TS), 100 g of anaerobically digested (AD) sludge as inoculum and 0.5 L of water were added to both of the reactors. After 4 days, 50 g of food waste was fed into LBR1 every two days, while 500 g of food waste was fed into LBR2 after 16 days of digestion (two cycles). Leachates were collected at the bottom of the LBR; of which, 75%, was used to feed the UASB while the remaining was recycled to the LBR with pH being adjusted to 6.0 using Na_2CO_3. Periodically, chemical oxygen demand (COD), volatile fatty acids (VFAs), VFA composition, TKN (total Kjeldahl nitrogen), NH_4^+-N (ammoniacal-N) of the leachates and methane production from the UASB were analyzed while the TS removal efficiency and methane yield were determined at the end of the experiment. The detailed analytical methods were described previously (Xu et al., 2011).

2.2. Microbial community analysis

Genomic DNA was extracted from leachate samples of LBRs at the determined points, i.e. LBR1 (Day 1, 5, 9, 16, 18, 23, 31, 38, 44, 56) and LBR2 (Day 1, 5, 9, 16, 24, 32) and then used for the bacterial community analyses. The variable V3–V5 region of bacterial 16S rRNA was amplified by PCR using the domain-level universal primers BAC338F (5 -ACTCCTACGGGAGGCAG-3) and BAC805R (5 -GAC TACCAGGGTATCTAATCC-3) (Yu et al., 2005). The 5 end of the forward primer was capped with a GC-clamp (5 -CGCCCGCCGCGCGCGGCGGGCGGGGCGGGGGC ACGGGGGG -3) (Muyzer *et al.*, 1993). DGGE was performed with 7% (w/v) acrylamide gel containing a 40–60% denaturant gradient and run at 60ºC, 75V for 16 h in 1X TAE buffer. Bands of interest were excised from the gel and then cloned into the pGEM-T Easy vector (Promega, USA). The sequences of cloned 16S rRNA gene fragments were analyzed using a 3730XL DNA Analyzer (Applied Biosystems, Foster City, CA) and compared with available sequences in GenBank database using the BLAST program (http://www.ncbi.nlm.nih.gov/BLAST). Neighbor-joining trees were constructed for phylogenetic analysis using MEGA 4 software (Tamura et al., 2007).

2.3. Principal components analysis

Principal components analysis (PCA) was applied to profile the covariance of microbial community structure and reactor performance, i.e. pH, concentrations of COD, acetate, butyrate and propionate, removal efficiencies of VS, TOC and TKN. Proportion of variance represented by each axis is given in parentheses.

3. RESULTS AND DISCUSSION

3.1. Hydrolysis and acidogenesis performance

The pH of LBR leachates was affected by the acidification of substrate and the alkalinity input through the addition of buffering reagent. The initial pH values of both LBRs were close to 5.0, and decreased to around 4.0 as the acidification processed (Fig. 1). Due to the continuous feeding of FW into LBR1, the pH was in the range of 4.0-5.0. In LBR2 with sequential feeding operation, pH varied with typical cycle pattern. It dropped to ~4.0 or even lower at the starting of each cycle and then increased gradually close to 5.0-5.5 at the end of each cycle. Similarly, COD leaching also exhibited a cyclic pattern in LBR2, with a peak value of 60.0 g/L in the first day of each cycle and gradually decreased to lower than 15.0 g/L at the end of each cycle. The average COD leaching rate of LBR1 was 0.43 g COD/g VS_{added}/d, which was higher than 0.32 and 0.33 g COD/g VS_{added}/d in first and second cycles of LBR2. Similar to COD, the initial leaching rates of TKN and NH_4^+-N were relatively high ranging 847-1118 mg/L and 123-216 mg/L, respectively. After that, TKN and NH_4^+-N concentrations also varied with typical cycle pattern in LBR2, whereas they were relatively stable in the range of 47.5-74.4 mg/L in LBR1. The decomposition of protein would be retarded by the acidic pH and the presence of carbohydrate (Miron et al., 2000). Thus the decompositions of protein in both LBRs were found to be limited compared to COD leaching.

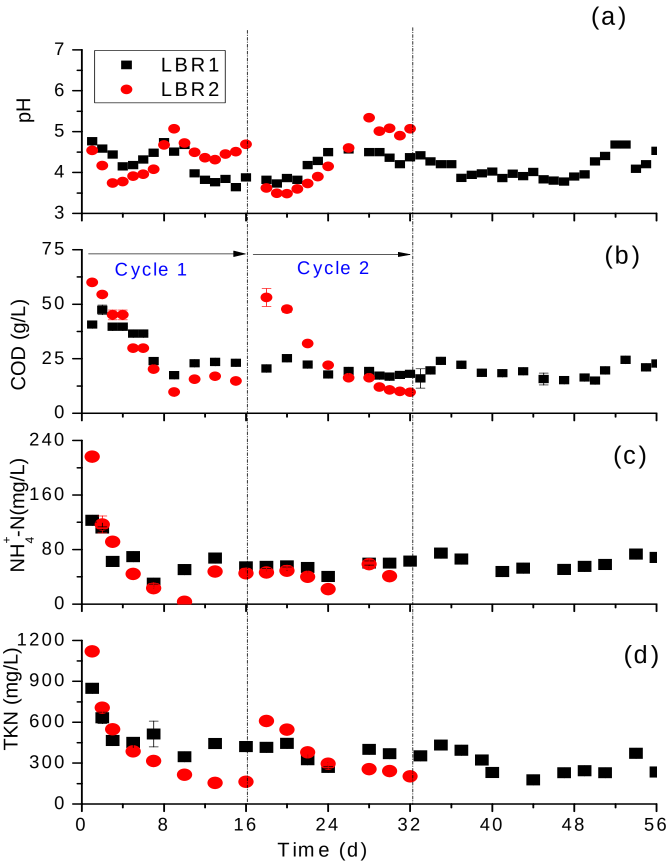

FIGURE 1 Characterization of leachates in LBRs: (a) pH, (b) COD, (c) NH_4^+-N and (d) TKN concentrations.

3.2. Evolutions of VFAs in LBRs

Hydrolytic microorganisms and acidogens efficiently solubilized the complex solid organic matter in food waste to simple organic compounds and their acidification to monomers (VFAs and alcohols), respectively, in LBRs. The variations of VFAs are showed in Fig. 2. Overall, acetate and butyrate acids were the predominant VFAs in the acidogenic leachate collected from both LBRs. Similar observations during the digestion of food waste and/or organic fraction of municipal solid waste were reported previously (Kim et al., 2008a; Dogan et al., 2009; Shin et al., 2010).

In LBR1, butyrate predominated in first 10 days and then was replaced by acetate in the continuous feeding period. Nevertheless, the predominance of acetate and butyrate alternated along with digestion in LBR2. Generally, butyrate dominated in the first Cycle of LBR2, while acetate dominated in the second Cycle. The VFAs composites in the acidogenesis process are affected by the pH (Ye et al., 2007; Kim et al., 2008b), hydraulic retention time (Maharaj and Elefsiniotis, 2001), temperature (Komemoto et al., 2009) and the nature of feedstock (Lü et al., 2009). With repeated batch operation, the conditions of pH and substrate in LBR might be more fluctuant, which could lead to the variation of reactor performance. However, this situation could be alleviated by increasing the loading frequency, i.e. semi-continuous operation. Furthermore, only a low level of propionate, i.e. 1.0-16.5 mM, was found in LBRs throughout digestion. When easily degradable carbohydrate-rich substrates were used, the more acetate was produced and when the lignocellulose-rich or protein/lipid-rich substrates were used, higher concentration of propionate was produced (Lü et al., 2009). This result correlated with the low decomposition rate of protein, as indicated by low concentrations of TKN and NH_4^+-N.

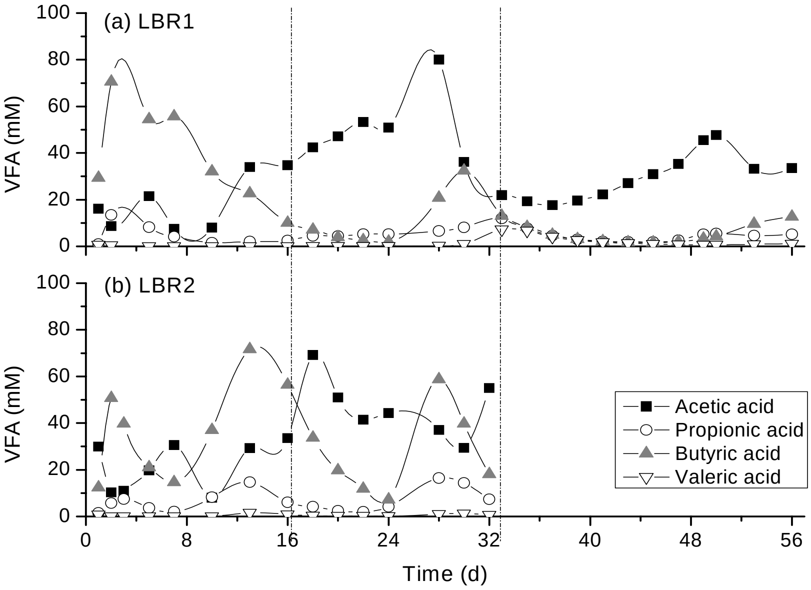

FIGURE 2 Profiles of VFA speciation in LBR1 and LBR2 along with digestion.

3.3. Evaluation of overall efficiency of LBR-UASB system

The performance of solubilization, acidification and methanogenesis of substrate are illustrated in Table 1. The practical organic loading rates (OLR) were 2.34 g TS/L/d for LBR1 and LBR2. The decomposition rates of food waste showed no significant difference between LBR1 and LBR2, as indicated by the TS removal efficiencies, i.e. 82.2% and 84.1%. The COD and VFAs yields in LBR1 with continuous feeding operation were higher than the LBR2 with batch feeding mode; however, the difference was insignificant. Nevertheless, yields of COD (0.84 g COD/g VS_{added}), VFA (0.30 g COD/g VS_{added}) and their corresponding leaching rates were comparatively higher during the second cycle of LBR2 than the yields of COD (0.81 g COD/g VS_{added}) and VFA (0.27 g COD/g VS_{added}) observed during the first cycle; indicating the advantage of digestate as the inoculum overAD sludge.

TABLE 1 Comparison of acidogenic and methanogenic efficiencies between LBR1 and LBR2.

		LBR1	LBR2	
			Cycle 1 (1-16 d)	Cycle 2 (17-32 d)
FW loading frequency		50 g/2 d	500 g/16 d	500 g/16 d
OLR (g TS/L/d)		2.34	2.34	2.34
TS removal efficiency (%)		82.2	73.3	84.1
Average COD leaching rate (g COD/g VS_{added}/d)		0.43	0.32	0.33
Average VFA leaching rate (g COD/g VS_{added}/d)		0.31	0.25	0.27
COD yield (g COD/g VS_{added})		0.86	0.81	0.84
VFA yield (g COD/g VS_{added})		0.33	0.27	0.30
VFA compositions	Acetic acid	61.4	31.9	55.2
(%, mol basis)	Propionic acid	8.0	9.1	8.6
	Butyric acid	16.6	56.2	31.9
Methane yield (g COD/g VS_{added})		0.20	0.16	0.17
Methane yield (g COD/g $VS_{removed}$)		0.30	0.25	0.26

Furthermore, UASB connected with LBR1 produced higher methane yield (0.30 g COD/g $VS_{removed}$) than that of LBR2 (0.25 g COD/g $VS_{removed}$). It might be attributable to the quality of acidogenic leachate. Acetate constituted 61.4% of the total VFAs produced in LBR1, whereas it was only 31.9% and 55.2% in the first and second cycles of LBR2.

3.4. Bacterial community in LBRs

3.4.1. Dynamics of bacterial community in LBRs

DGGE and subsequent phylogenetic analysis were conducted to analyze the metabolically active bacteria in the continuous acidogenic process, as shown in Fig.3a. For easy tracking of the dynamics of active bacteria, ImageJ was used to semi-qualitatively analyze the DGGE profile (Fig. 3b). A total of 27 bands (A1-27) were excised and sequenced from DGGE gel (Table 2). The phylogenetic affiliations of bacterial community included four phyla, Firmicutes, Proteobacteria, Actinobacteri and Bacteriodetes, and 5 bands could not be identified. Among the identified bands, major bacterial groups belonged to two orders: Lactobacillales (13 bands) and Clostridiales (14 bands).

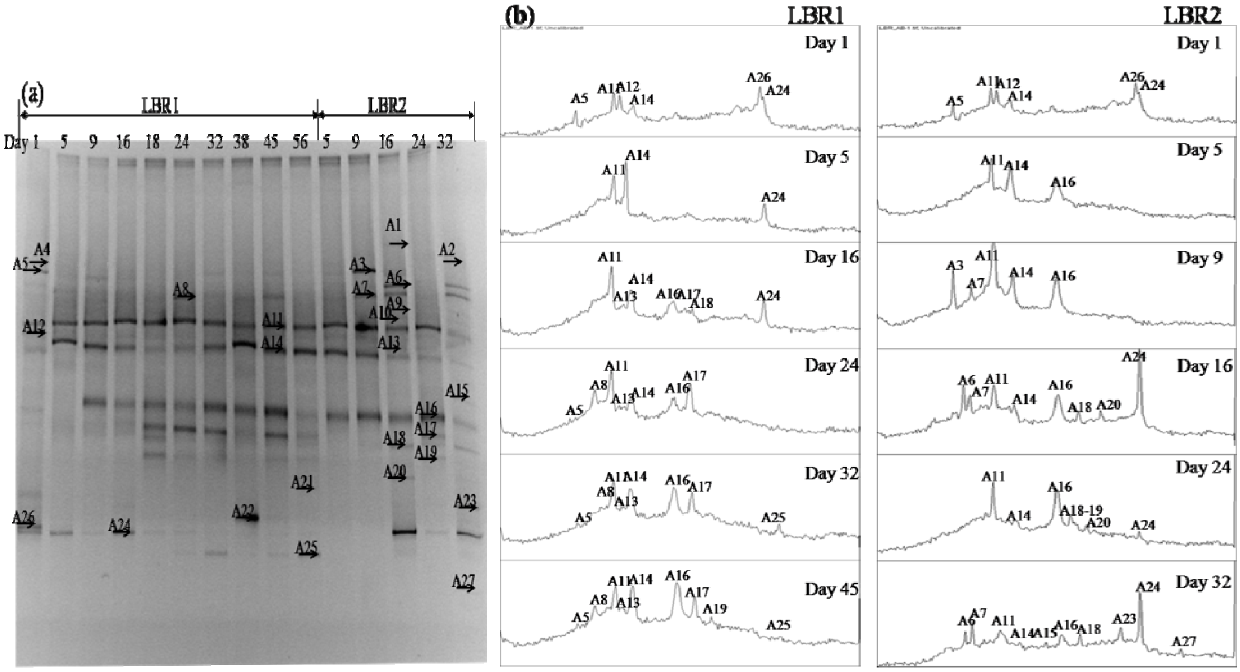

FIGURE 3 PCR-DGGE profiles (a) and relative enrichment of bacterial community as revealed by ImageJ analysis (b).

The active microbial population observed on the first day of digestion was related to the inoculum. The Day 1 samples were clustered to *Clostridium* (bands A4), *Lactobacillus* (bands A5, A11, A14), Alphaproteobacteria (A12), Gammaproteobacteria (A26) and *Bifidobacterium* (A24). Specifically, *Lactobacillus amylovorus* (A5), *Lactobacillus fermentum* (A11), *Lactobacillus mucosae* (A14) and uncultured *Clostridium* (A4) dominated in LBR1.

Lactobacillus amylovorus is a fermentative bacterium which produces lactic acid and small amounts of acetic acid from sugars (Nakamura, 1981). *Lactobacillus mucosae* is closely related to *L. reuteri*, and *L. fermentum* (Roos et al., 2000), which are classified as obligate hetero-fermentative group that metabolize the substrates through the phosphoketolase pathway, and produce significant amounts of other products such as ethanol, acetate and CO_2 in addition to lactic acid (Årsköld et al., 2008). *Clostridium butyricum* (Wang et al., 2009; Luo et al., 2011) along with *C. acetobutylicum*, *C. saccharoperbutylacetonicum* and *C. pasteurianum* are often used for hydrogen production (Pattra et al., 2010). Butyric acid, acetic acid and minor levels of lactic acid are the main by-products of food waste acidogenesis. Despite the same inoculum, the bacterial community structure in LBR was different from the first day of digestion due to the different operations, like pH and water regime. However, due to their similar metabolic pathway, the combination of different bacterial species could produce acetate as the main soluble metabolites as seen in Fig. 2.

3.4.2. Ordination analyses

PCA score plot is used to visualize the patterns in bacterial community structure across the sample set as shown in Fig. 4. Microbial structures in LBR1 and LBR2 reactors (Fig. 4a) along with digestion period were clearly separated. They were located at opposite ends of Axis 1A, which explained 21.2% of the variability in community structures. As the digestion processed, bacterial community moved to the location of the opposite side of Axis PC1. Simultaneously, the bacterial community also migrated along with digestion period from the upper side of Axis PC2 to the lower side, with 19.7% of the variance. Parameters of pH, ethanol, butyrate, COD and COD leaching rate were positively correlated to Axis PC1, while parameters of VS removal efficiency and acetate were more correlated to Axis PC2. As shown in Fig. 4b, the increased abundance of species A21, A25, A18, A19, A16, A22, A17 and A8 were positively correlated to acetate concentration, while species A2, A13, A14, A7, A20 were associated with the increasing in propionate.

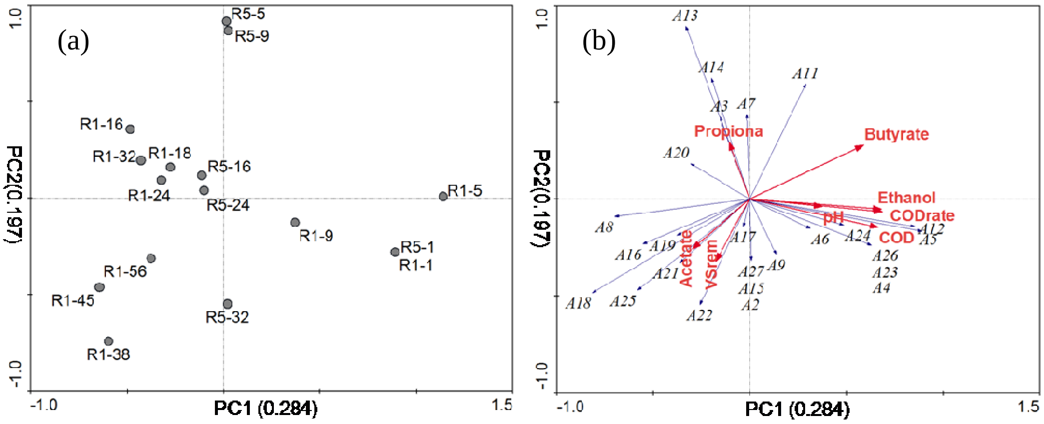

FIGURE 4 **PCA score plot from dataset of DGGE bands alone (a) and the covariance datasets of bacterial species and reactor performance (b).**

TABLE 2 **Phylogenetic identities of the 16S rRNA sequence from DGGE bands.**

OTUs	Accession no.	Taxon	% Similarity
A1	FR668370.1	Uncultured Clostridiaceae bacterium	91
A2	FR716435	Uncultured bacterium clone XMF58	97
A3	GQ477905.1	Uncultured bacterium clone MS-146	100
A4	GU227148.1	*Clostridium tyrobutyricum* strain A1-3	100
A5	FR683089.1	*Lactobacillus amylovorus* strain DSM 20531T	100
A6	JF312646.1	*Clostridium* sp. enrichment culture clone NHT4	100
A7	EU828368.1	Uncultured bacterium clone T4_1_28	99
A8	M59113.1	*Clostridium tyrobutyricum*	97
A9	FJ393107.1	Uncultured *Acidovorax* sp. clone MFC-B162-E05	99
A10	GU583828.1	Uncultured *Caloramator* sp. clone BB20	95
A11	EU626018	*Lactobacillus fermentum* strain KLDS 1.0733	99
A12	GQ240640.1	*Acetobacter pasteurianus* strain DSM 2006	98
A13	FR693800.1	*Lactobacillus mucosae* isolate LB2	99
A14	JN092131	*Lactobacillus mucosae* strain FSL-04	97
A15	JF793969	*Acetobacter peroxydans* strain LMG 1635	99
A16	GQ332261.1	Uncultured *Acetobacter* sp. clone V45	93
A17	HM217933	*Acetobacter fabarum* strain G2-5	100
A18	NR_026310.1	*Lactobacillus panis* strain DSM 6035	100
A19	GQ505035.1	Uncultured bacterium clone TKW-HPB-19	99
A20	AB437362.1	*Bifidobacterium thermacidophilum.*	99
A21	JF032229.1	*Uncultured bacterium* clone ncd323f05c1 16S	100
A22	JN208201	*Enterobacter* sp. DG10	94
A23	EU887962.1	Uncultured Clostridia bacterium clone S4	99
A24	HQ851038.1	*Bifidobacterium thermophilum* strain TEEV 25 II	98
A25	AY278612.1	*Bifidobacteriaceae* genomosp. C1	97
A26	HQ677189.1	*Citrobacter freundii* strain QN4.3.2	96
A27	AB597003.1	*Lactobacillus reuteri*	99

The genus *Lactobacillus* (A11 and A14) got enriched due to the presence of easily degradable fraction of the substrate and the low pH conditions (Enfors and Häggström, 2000). With the gradually vanishing easily degradable organic matter in the reactor, bacterial community became more diverse and even. With the appearance of *C. tyrobutyricum* (A3), *Acetobacter* (A16) and *Bifidobacterium* (A20, A24, A25), the enrichment of those fermentative bacteria decreased correspondingly. Under the operation of semi-continuous feeding, *Lactobacillus* alone dominated in the starting stage (Day 1 to 5); whereas along the digestion, a more diverse bacterial community appeared with higher diversity. The fermentation bacteria *Lactobacillus* and *Clostridium*, co-existed with these species, have more versatile capacity to decompose of complex polymers. *Clostridium* and *Lactobacillus* had significantly higher representation among the detected OTUs throughout the digestion, which might be due to their specialized metabolic nature of producing fatty acids that make their populations less vulnerable to the competition from functionally redundant OTUs. *Bifidobacterium thermophilum* could survive and grow at relatively low pH (4.5) and the fermentation products (from glucose) are acetic and lactic acid at a molar ratio of 2.46–2.72 (Dong et al., 2000). *Acetobacter* is a genera of acetic acid bacteria, which is characterized by converting ethanol to acetate in the presence of oxygen (De Vero and Giudici, 2008). Nevertheless, use of digestate as inoculum is a potential advantage of the sequential LBR as seen in Fig. 4. The higher diversity of bacterial community was observed in Cycle 2 than Cycle 1. Although the DGGE pattern still varied from the start to end of digestion, the variation decreased abundantly. Bands A11, 16, 18 and 24 appeared persistently and thus led to the better performance of organic matter solubilization and higher acetate yield in Cycle 2 of LBR2.

4. CONCLUSIONS

The yields of COD (0.86 g COD/g VS$_{added}$) and VFA (0.33 g COD/g VS$_{added}$) were higher in LBR1 compared with LBR2. The superiority of continuous feeding mode over batch mode was supported not only by the increased hydrolysis rate but also by the volatile fatty acids (VFA) composition. Acetic acid represented 61.4% of VFAs produced in the leachates, which were comparatively higher than the 31.9 and 55.2% observed in LBR2 in the first and second cycles, respectively. PCR-DGGE profiles indicated that *Lactobacillus* sp. dominated during the initial stage of LBR2 and the diversity increased with more versatile species appeared in the later stage of digestion. However, the genus *Clostridium* sp., *Acetobacter* sp., and *Bifidobacterium* sp. appeared in LBR1 persistently, which led to the production of extracellular hydrolytic enzyme and yielding acetate as the predominant acid.

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(185) BIO-HYDROGEN AND METHANE PRODUCTION USING DARK FERMENTATION

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EXECUTIVE SUMMARY

Hydrogen is regarded as an energy source of the future. Currently hydrogen is predominantly produced by electrically driven electrolysis of water or by steam reforming. Both methods base on fossil fuels. These days the production of hydrogen by biological processes has become a matter of global interest and attention (Levin et al., 2004). Alongside photo-fermentation and bio-photolysis, dark-fermentation is a possible biological method to produce hydrogen. In order to fit every day energy requirements, those methods have yet to be enhanced and adapted to an industrial scale.

Previous research of a two stage test set up with bio-hydrogen production as first stage, followed by a "conventional" anaerobic digestion step with a tenfold volume showed, that it is possible to combine these processes. By doing so, it is important to strictly divide both steps from each other because the methanisation stage can affect the hydrogen production negatively.

This paper deals with first lab-scale test series in order to define suitable substrates for a pilot plant (volume of hydrogen and methane tank 1 m^3 each). Simultaneously, effects of disturbances are observed by simulating a leakage of the reactor as well as a failure of the heating system.

The continuously stirred hydrogen reactor has a volume of 4 L and is running at mesophilic conditions around 35 - 36 °C. Digested sludge from a nearby waste water treatment plant (WWTP) was used as seed sludge and pretreated by heating up to 70 °C for an hour. In order to give the microorganisms time to adapt, 2 L seed sludge are mixed with 2 L of the respective substrate solution and stirred for a period of 48 hours. Afterwards the continuous experiment was started.

Polluted industrial sugar and bakery wastes have been used as substrates. Both substrates have been diluted with tap water until a concentration of 10 g volatile solids (VS)/L was reached. The reactor is running semi-continuously by feeding the substrate once an hour.

Testing of sugar-solution as substrate lead to average biogas productions of 971.86 mL/(L_R*d). Maximum production rates of 1,392.70 mL/(L_R*d) could be obtained. Furthermore, the arithmetical mean of hydrogen-yields was 77.84 mL/g VS_{added}. Hydrogen content of produced biogas was varying between 36.8 % and 44 %.

Experimental set up with bakery wastes showed comparable results regarding the amount of produced biogas. Peak level was at 1,448 mL/L_R*d. Hydrogen contents from 30.2 to 52.3 % could be found. Hydrogen-yields alternated between 49.1 and 205.8 mL/g VS_{added}. Although the breadcrumbs-fed reactor ran at distinctly shorter HRT´s, sugar and breadcrumbs both lead to comparable amounts of produced biogas. This outcome can be ascribed to the fact, that bread or rather carbohydrates are not as biologically available as plain sugar.

The simulation of a failure of the heating showed, that low temperatures induce inhibited biological activity whereas metabolic processes themselves remain the same. An aeration of the reactor lead to increased production rates of biogas and hydrogen yields. Both simulated disturbances do not seem to harm the process irreversibly, if adjusted promptly.

The combination of the two treatment steps bio-hydrogen production and "conventional" anaerobic digestion is a feasible option for example for the treatment of mono-charges, which is otherwise ensured by cost intensive enlargement of digester volume or massive reduction of organic load rate (OLR). In the context of the pilot scale research, it has to be evaluated, if energy recovery by the combination of hydrogen- and methane-production with fuel cell is comparable to the conventional anaerobic digestion which uses combined heat and power plants (CHP) to produce electricity.

1. INTRODUCTION

Nowadays, the importance of renewable energy sources is tremendous, especially since fossil fuels are limited and the reliability of atomic power plants is highly controversial. Among wind and solar energy, hydrogen is regarded as one of the most important energy sources to be. Currently hydrogen is predominantly produced by electrically driven electrolysis of water or by steam reforming. Both methods base on fossil fuels. These days the production of hydrogen by biological processes has become a matter of global interest and attention (Levin et al., 2004). Alongside photo-fermentation and bio-photolysis, dark-fermentation is a possible biological method to produce hydrogen. In order to fit every day energy requirements, those methods have yet to be enhanced and adapted to an industrial scale.

1.1 Background

Dark fermentation processes have been examined in the course of different research projects (e.g. Krupp, 2007; Rechtenbach, 2009; Brunstermann, 2010; Redondas et al., 2012). A variety of different reactor types has been reported. Alongside continuously stirred tank reactors (CSTR) other reactor types like upflow anaerobic sludge blankets (UASB), carrier-induced granular sludge bed (CIGSB) and upflow fixed bed reactors (UFBR) have been used for biological hydrogen production (Redondas et al., 2012; Camilli et Petdroni, 2005; Spagni et al., 2011; Lee et al., 2006). While -due to the adhesion forces between the fest bed material and the microorganisms- fest bed reactors can be run at lower hydraulic retention times (HRT), CSTR profit from the benefits of a thorough merging.

Besides advantages like short hydraulic retention times and high metabolic rates, incomplete transformation of the organic components into various organic acids can be named as a disadvantage. Therefore, a second process step is required to ensure optimal energy efficiency. The combination with a methane reactor, fed by the effluent of the hydrogen step, is a very economical solution.

Previous tests by Brunstermann (2010) have been carried out in order to develop a system which allows an efficient combination of hydrogen and methane production by dark fermentation at mesophilic conditions. The continuously operation of the combined hydrogen and methane production out of carbohydrate rich biological waste was examined in a lab scale reactor of 4 L in case of hydrogen production followed by a 30 L reactor for the methane production unit. Digested sludge of a wastewater treatment plant, which was boiled at 70 °C for one hour, was used as seed material for the hydrogen step. The methane reactor was started by using the same digested sludge without any pre-treatment. A solution of a waste sugar with a concentration of 10 g/L and 15 g/L repetitively was used as a carbon or rather hydrogen source in the hydrogen reactor. The methane reactor was fed with a mixture of water and breadcrumbs. After both reactors ran steadily, they were combined by replacing water by the effluent of the hydrogen stage as mixing liquid for the breadcrumbs. This research resulted in different findings. On the one hand increasing the sugar concentration lead to a slightly increased hydrogen content within the produced biogas. At the same time the production rate of biogas decreased to a significant extend. Thus the reactor-specific hydrogen yield sank from 0,22 NL/g volatile solid (VS) (sugar concentration of 10 g/L) below 0,1 NL/g VS (sugar concentration of 15 g/L). The reduction of hydraulic retention time induced increased hydrogen-yields. The methane tank benefited from the combination with the hydrogen step. The replacement of water as feed substrate by effluent of the hydrogen tank led to escalated reactor-specific gas-yields and substrate-specific methane-yields. By providing the tank with hydrolytic microorganisms and organic acids, hydrolysis of co-substrate (breadcrumbs) is enhanced. This could also enable shortened HRT´s within the methane tank. Simultaneously marginal reductions of methane-concentrations within the biogas (from 58,5 to 56 %) could be observed.

1.2 Research objectives

This paper deals with a combination of bio-hydrogen and methane production. According to previous studies, the combination of both steps can enhance the microbial conditions of the methane tank. The goal of this research is to elaborate the conditions and limitations of a pilot scale plant. The hydrogen tank is going to contain a volume of 1,000 L, followed by a methane reactor which has a capacity of 1,000 L as well. A long-term objective is to commute hydrogen into electricity by using a fuel cell. The power efficiency of such a combined system and the possible application at existing waste water treatment plants have to be evaluated. As one of the first steps, possible substrates have to be tested in terms of their gas production potential on a lab scale basis. Simultaneously, effects of disturbances are observed by simulating the failure of the heating system as well as a leakage.

2 METHODOLOGY

Figure 1 shows the schematic assembling of the lab scale experiments.

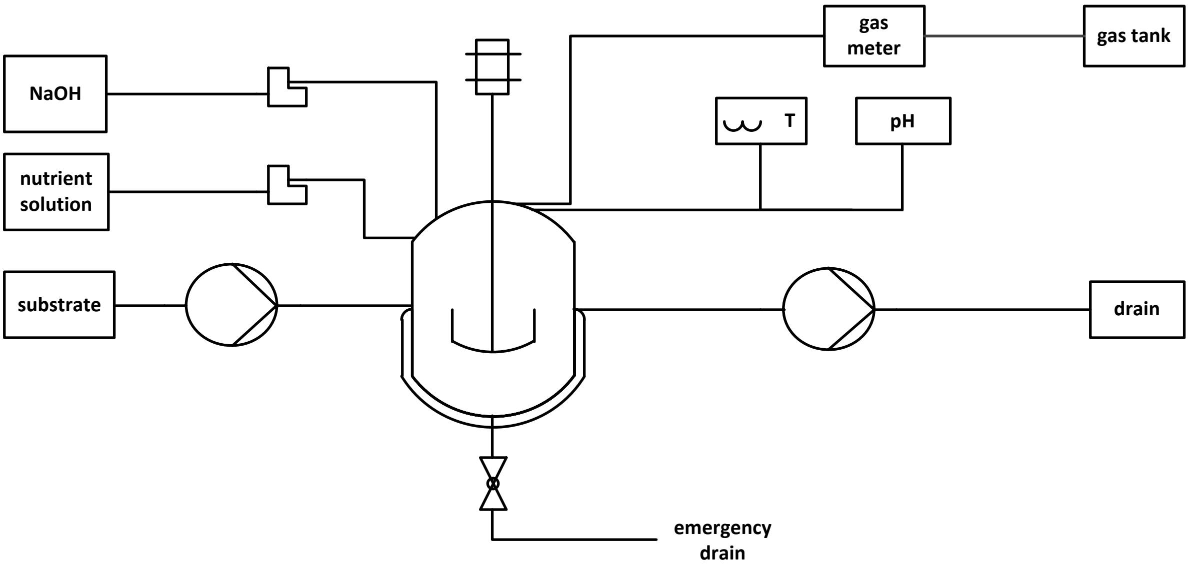

FIGURE 1 schematic assembling of lab scale experiments

The continuously stirred hydrogen reactor has a volume of 4 L. It is double-walled, a water jacket keeps the temperature within the reactor at mesophilic conditions around 35 ± 1 °C. According to previous researches, digested sludge from a nearby WWTP was used as seed sludge and pretreated by heating up to 70 °C for an hour. In order to give the microorganisms time to adapt, 2 L seed sludge are mixed with 2 L of the respective substrate solution and stirred for a period of 48 hours. Afterwards the continuous experiment was started.

Polluted industrial sugar (reactor 1/R1) and bakery wastes (below named breadcrumbs, reactor 2/R2) have been used as substrates. Both substrates have been diluted with tap water until a concentration of 10 g VS/L was reached. The substrate is fed via flexible tube pumps once an hour. The analogous amount of effluent is pumped simultaneously. Therefore the reactor is run semi-continuously. The hydraulic retention time can be controlled by choosing different operating times for the flexible tube pumps. The reactor which was fed with sugar ran at a HRT of 48 hours, the one with breadcrumbs was operated at HRT = 32 hours. The cycle control is operated using LabVIEW. In order to monitor the actual hydraulic retention times, feed and drain tank were weighed on a daily basis.

To every liter substrate 5 mL 10-fold concentrated nutrient solution according to Hussy (2003) are added to the reactor by using a metering pump. Temperature and pH-value have been monitored on-line. The pH-value was regulated by adding a 1 M solution of sodium hydroxide to the reactor, every time the pH sank below 4.5 the proportioning pump ran until the pH was set to 4.55.

The produced biogas was stored in gas sampling bags after being measured by a gas meter. The hydrogen content within the sampled gas probes was analyzed using a thermal conductivity detector (Conthos 2, LFE GmbH &CoKG, Maintal), carbon dioxide-, oxygen- and methane-concentrations of the biogas were measured via landfill gas analyzer (Ansyco GA 2000, Analytische Systeme und Komponenten GmbH, Karlsruhe).

The reactor which was fed with sugar was operated for 29 days, the one with breadcrumbs as substrate ran for 8 days.

In order to simulate a possible failure of the heating system, the water jacket of reactor 1 was not heated for a period of two days (experimental days 18 and 19). The simulation of a leakage was performed by pumping air into R2 on experimental day 6.

A sample of reactor 1 had been taken on experimental day 2 in order to perform Fluorescence in-situ Hybridisation (FisH). FisH has been carried out according to Pernthaler et al. (2001), Küsel et al. (1999), Amann et al. (1990) and Wallner et al. (1993). Samples were hybridized with CLOST I (Probe: Cy3/ 5´-TTC-TTC-CTA-ATC-TCT-ACG-CA-3´) as well as EUB 338 (Probe: Cy3/ 5'- GCT GCC TCC CGT AGG AGT -3'), NON 338 (Probe: 6-FAM/ 5´-ACT-CCT-ACG-GGA-GGC-AGC-3´). Afterwards the samples have been counter stained with DAPI. Evaluation of samples was done with an Axio Imager M2 microscope by Carl Zeiss, equipped with the camera AxioCamMR3. Photomicrographs were taken at 100-fold magnification (objective lens: EC Plan-Neofluar 100x/1.30 Oil M27).

In order to detect Cy3-signals, filter No. 43 is used. Filter No. 49 is used for DAPI- and filter 38 EndowGFP for 6-FAM-signals.

3 RESULTS AND DISCUSSION

3.1 Lab scale research

Figure 2 shows registered experimental conditions like temperature, pH-value, HRT and organic loading rate (OLR).

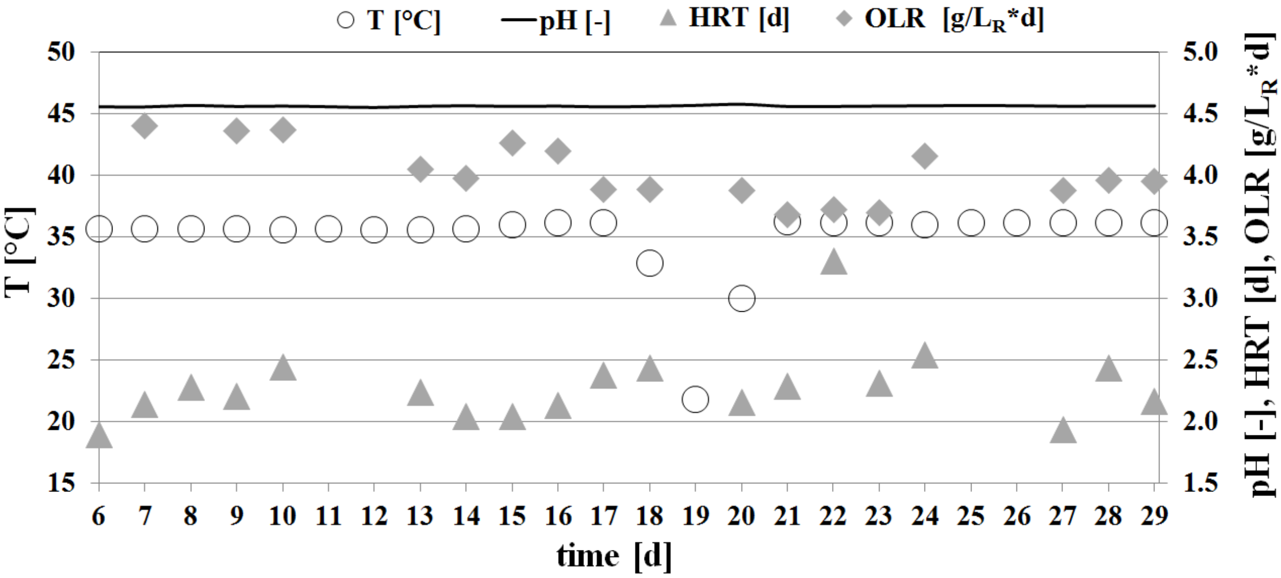

FIGURE 2 **Registered temperature, pH-value, hydraulic retention time and organic load rate/sugar as feed-substrate**

While the pH-value remained mostly constant by varying between 4.55 and 4.58, the other parameters showed considerable fluctuations. Since the delivery rate of the peristaltic pumps varied insignificantly, HRT´s from 46.1 up to 79.2 hours could be recognized. On experimental day 18, temperature sank to 32.9 degrees, on day 19 a temperature of 21.8 °C was observed. After restarting the heating system, the regular working temperature was reached on day 21. OLR varied from 3.03 to 5.21 g/L$_R$*d. Average OLR added up to 4.34 g/L$_R$*d.

Figure 3 shows the gathered results regarding gas production rate (GPR), hydrogen content (HC) and hydrogen yield (HY) alluded to the added amount of volatile solids (VS).

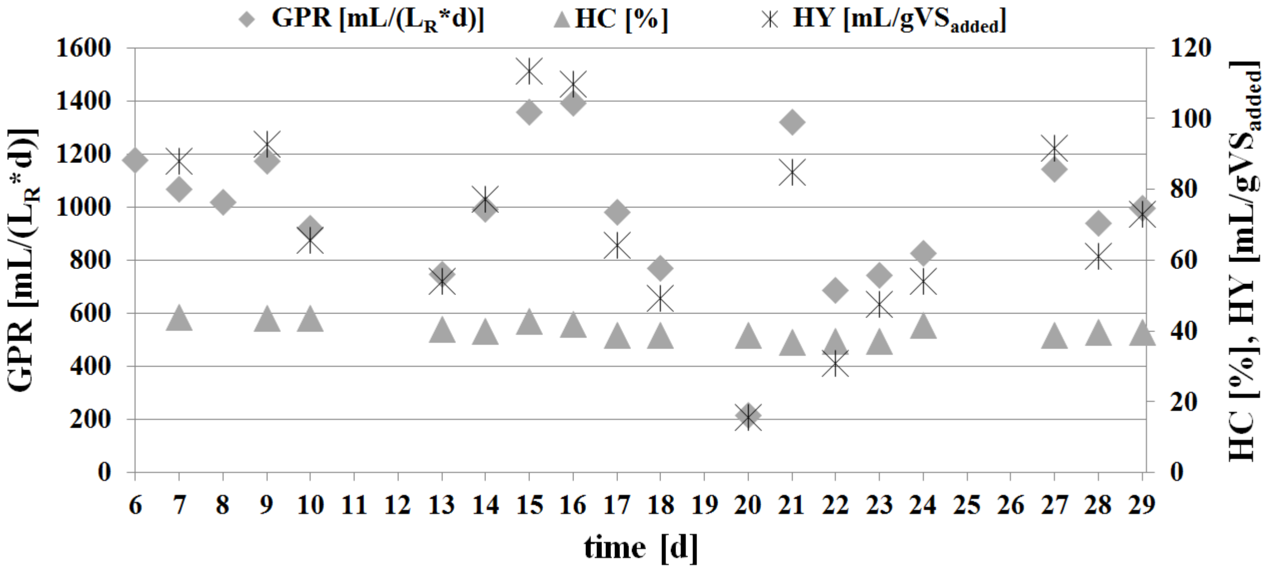

FIGURE 3 **Registered produced biogas, hydrogen content and hydrogen yield/sugar as feed-substrate**

According to figure 3, different conclusions can be drawn. Hydrogen content of the biogas varied only slightly (36.8 vol-% to 44 vol-%) over time. Even the period with considerably low temperatures (days 18 to 20) did not have a noticeable effect on the amount of relatively evolved hydrogen. During regular operating state, the quantity of built biogas fluctuated between 668 mL and 1,393 mL/(L_R*d). When the temperature sank significantly below working temperature, biogas production was inhibited, only 217 mL/L_R*d were rendered. As soon as the temperature was back to normal conditions, biogas-production spiked to 1,320 mL/L_R*d (day 21). A longer period of anew adaption does not seem to be needed. Same could be observed regarding the hydrogen-yield. After dropping down to 15.64 ml/g VS_{added} on day 20, it rose to 84.84 ml/g VS_{added} on day 21.

Experimental conditions of the second test-run with breadcrumbs as substrate are displayed in figure 4.

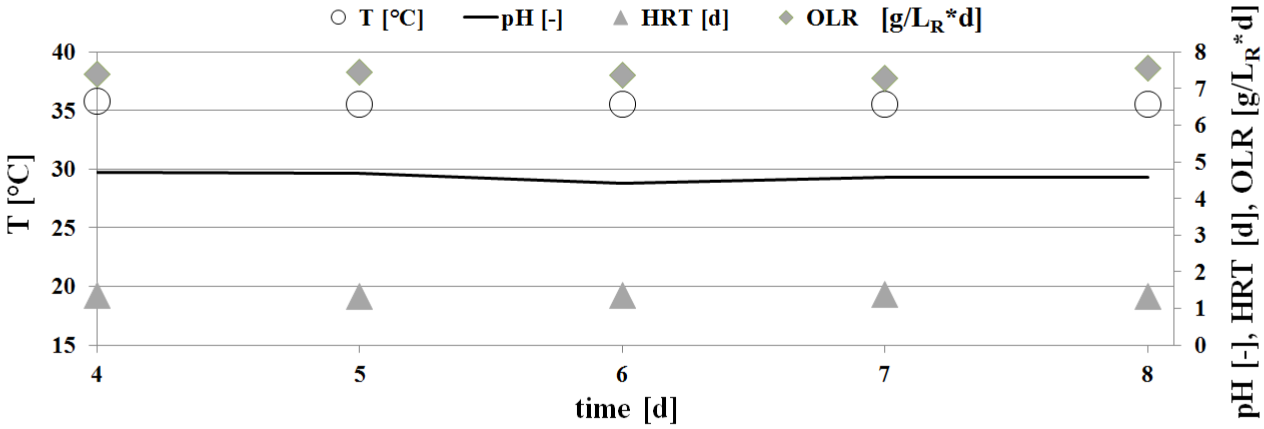

FIGURE 4 **Registered temperature, pH-value, hydraulic retention time and organic load rate/breadcrumbs as feed-substrate**

The different parameters stayed mainly constant. Temperatures from 35.5 °C to 35.8 °C and pH-values between 4.41 and 4.72 were observed. Hydraulic retention times of 31.8 to 33 hours lead to OLR from 7.27 to 7.55 g/L_R*d.

The experimental outcome is shown in figure 5.

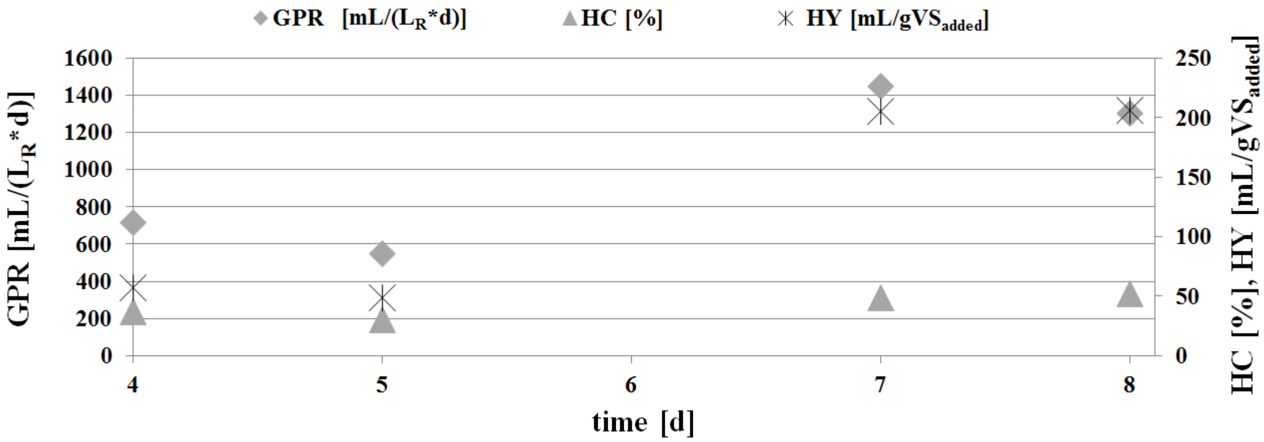

FIGURE 5 **Registered produced biogas, hydrogen content and hydrogen yield/breadcrumbs as feed-substrate**

The amount of produced biogas varied between 547 and 1,448 mL/L_R*d. Hydrogen contents from 30.2 to 52.3 % could be found. Hydrogen-yields alternated between 49.1 and 205.8 mL/g VS_{added}. On day 6, dosing pump pressed air into the reactor, therefore measured parameters could not be recorded. Oxygen content within the biogas was 2.0 % on day 7, on experimental day 8, oxygen was not detectable anymore.

While hydrogen content rose insignificantly after aeration, biogas-production and hydrogen-yield escalated to a remarkable extend.

At day 7, hydrogen-yield spiked to 205.56 ml/g VS_{added}, the amount of produced biogas to 1,448.49 ml/L_R*d. The comparison of day 5 and day 7 shows, that hydrogen yield is increased by a factor of 4.19, the amount of produced biogas by a factor of 2.65.
Accompanied with the fact, that reactor 2 shows approximately twice as high organic load rates (due to shorter retention times) also specific hydrogen yields of R2 are roughly two times higher compared to those of reactor 1.

Figure 6 shows photomicrographs which are taken from reactor 1 on experimental day 2.

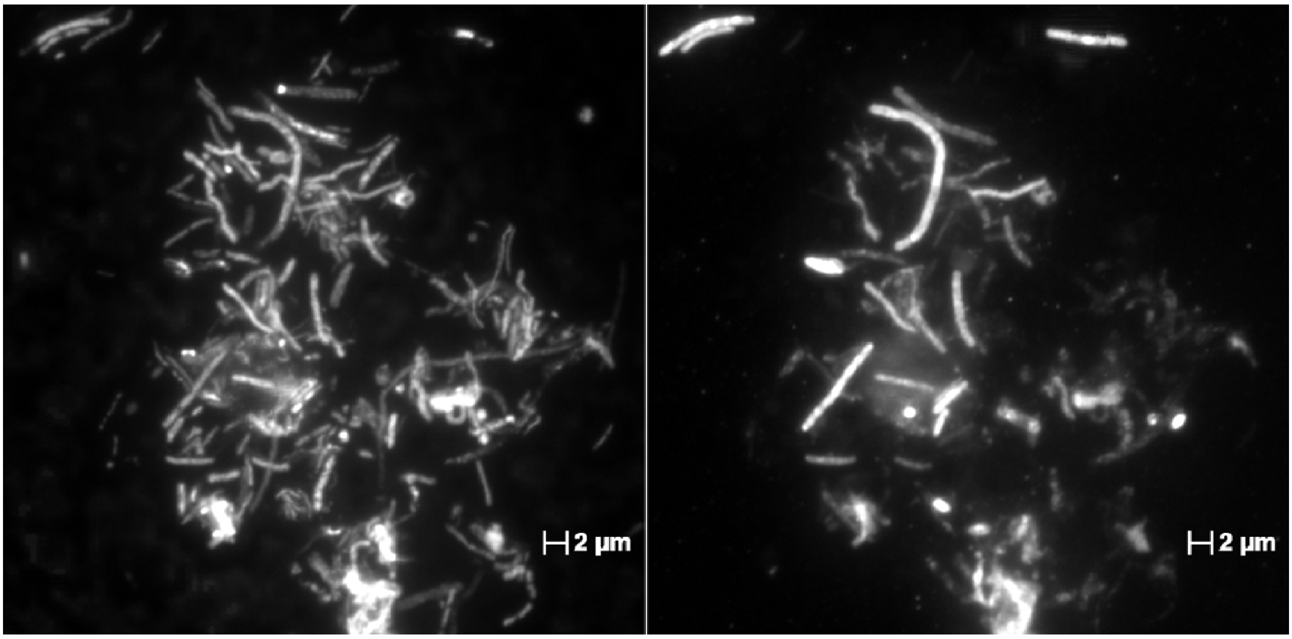

FIGURE 6 FisH of reactor material taken from R1 on experimental day 2 (left: detected DAPI-signals; right: Cy3-signals by Probe CLOST I)

Validity check with NON 338 is not pictured, since the probe did not attach to any genetic material. Accuracy of the FisH performance is also given by the fact of a higher amount DAPI-signals than probe-signals. In addition also the physical shape of the probe-signals corresponds to the characteristics given in literature (Küsel et al. 1999). As the photomicrographs show, genetic material in large part consists of *Clostridia*.

3.2 Pilot scale research

At the moment, the project status of the pilot plant is at the end of the planning phase, fist plant sections are being built. Technical build-up is similar to Figure 1. The effluent of the hydrogen step is going to be used as substrate for the methane tank. Since both tanks are going to be carried out with volumes of 1 m³ each and HRT´s in the methane-reactor are significantly higher than the ones used for hydrogen-production, only a branch current of the effluent is going to be used as feed material. The remaining part is going to be tested as a carbon source for denitrification-processes. Produced hydrogen is going to be impinged on a fuel cell with a capacity of 1 kW.

4 CONCLUSION

Photomicrographs of FisH, show a noticeable amount of *Clostridia* after an adaption phase of two days.
The simulation of a failure of the heating system resulted in the following findings: When the temperature dropped down to 21.8°C (day 19), both the biogas production and the hydrogen yield decreased while the hydrogen content stayed stable. Therefore low temperatures induce inhibited biological activity whereas metabolic processes themselves remain the same. After re-heating the hydrogen tank, microbiologic activity is back to the regular level at once.
An aeration of reactor 2 lead to increased production rates of biogas and hydrogen yields. Only two days after simulation of leakage, anaerobic conditions were reconstituted. Both simulated disturbances do not seem to harm the process irreversibly if adjusted promptly.
Breadcrumbs as well as industrial sugar resulted in acceptable hydrogen contents within the produced biogas. Average hydrogen contents of both reactors are almost identical. Notwithstanding reactor 2 yields higher maximum hydrogen contents.

Although R2 ran at distinct shorter HRT´s, sugar and breadcrumbs both lead to comparable amounts of produced biogas. This outcome can be ascribed to the fact, that bread or rather carbohydrates are not as biologically available as plain sugar. In all likelihood reactor-specific hydrogen yield of R1 can be improved by lowering HRT´s and therefore increasing OLR. Since the testing period is very short, the reliability of the drawn conclusions is limited, further test series are necessary.

The combination of the two treatment steps bio-hydrogen production and "conventional" anaerobic digestion is a feasible option for example for the treatment of mono-charges, which is otherwise ensured by cost intensive enlargement of digester volume or massive reduction of organic load rate (OLR). In the context of the pilot scale research, it has to be evaluated, if energy recovery with the combination hydrogen and methane with fuel cell is comparable to the conventional anaerobic digestion which uses combined heat and power plants (CHP) to produce electricity.

5 ACKNOWLEDGEMENTS

Special thanks go to the European Regional Development Fund and the NRW Ziel 2-programme without whose funding this research would not be possible. Sincere gratitude is given to the Emschergenossenschaft, who allocates substrates as well as a site and technical staff on one of their waste water treatment plants.

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Session 14

(13) INFLUENCING THE GERMINATION OF DIFFERENT CROPS BY LIQUID BIOGAS DIGESTATE

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EXECUTIVE SUMMARY

Anaerobic digestion (AD) of solid and liquid organic wastes is vastly increased world-wide. This process has a lot of positive effects on the environment, among others the use of AD's by-product, the digestate, in the agriculture. Digestate could be a liquid or solid material with high nutrient and organic matter content which is appropriate to, at least partly, substitute for artificial fertilizers because of its favourable effect on the development and increase of plants.

Digestate can be used before sowing and in this case, it has effect on the germination of crop sown. Therefore our aim was to study the effect of a liquid digestate on the germination and early development of different plants grown in our region. We examined the modification effect of soil type and application times on the effect of applied digestate and we studied the differences among the tested plant species.

Germination experiment was carried out in Petri dishes (Ø=8.5 cm) filled with 80 g sand and loamy meadow soil substances which were watered to the 60 % of their field water holding capacity. Tested concentrations of digestate were 25%, 50% and 100% (v/v) and tap water was used as control. Treatments were done on the day of sowing, 5 days and 21 days before sowing. Tested plants were soybean (*Glycine soja* L.), sweet corn (*Zea mays* convar *saccharata*), rye (*Secale cereale* L.), winter wheat (*Triticum aestivum* L.) and sunflower (*Helianthus annuus* L.).

The number of seeds tested depended on the size of the seed, exactly 10 seeds of soybean, sweet corn, 15 seeds of sunflower and 50 seeds of rye and, winter wheat were used in the Petri dishes. The irrigation of seedlings was done in a case of need when seeds began to show wilting symptoms.

The germination rate of tested plants was measured on the 10th day after sowing, according to the Hungarian Standard.

For toxicity tests the germination index (GI%) and the relative growth index (GRI) were used to calculate the phytotoxic effects. We modified these indexes by using the dry mass of root and shoot of plants. The roots and shoots were measured on the 10th day after sowing.

Treatments were carried out in four replications. The statistical analysis of the measured values was done by one-way ANOVA following with Tukey's test for all tested plants, respectively with the SPSS 13.0 statistical program.

The effect of digestate on the germination of seeds depends on the type of soil, plant species (perhaps variety, but it was not examined), doses of digestate, period of digestate application before sowing.

Overall, the most sensitive plants were winter wheat and sunflower in both soil substances. For these plant species, only the smallest digestate dose (25%) not caused inhibition, or the germination of these species could be increased in a small rate. The number of germinated seeds and living plants were increased in the case of non-sensitive plant species.

Application of digestate for improving the germination rate of crops plants is more effective on sandy soil substance. To take into account the properties of this soil type, the use of digestate as a plant nutrient is very important not only in the point of the environmental protection but also in the point of the sustainable crop production.

Generally, early application of digestate causes the loss of nutrients and water and therefore its positive effects are decreased. On the other hand, the high doses of digestate applied near the sowing results the decrease of germination. On loamy meadow soil substance, the inhibitory effect of digestate application was experienced more often, the tested species seemed to be more sensitive than on sandy soil substance, where the treatments increased the germination in a higher rate, comparing to the controls.

1 INTRODUCTION

Anaerobic digestion (AD) of solid and liquid organic wastes is vastly increased world-wide. This process has a lot of positive effects on the environment, among others the use of AD's by-product, the digestate, in the agriculture (Tambone et al., 2010). Digestate could be a liquid or solid material with high nutrient and organic matter content (Möller et al., 2008) which is appropriate to, at least partly, substitute for artificial fertilizers because of its favourable effect on the development and increase of plants (Stinner et al., 2008; Bachmann et al., 2010).

Beside of these positive effects of digestate, it could have some negative effects on plants, too (Pfundtner, 2002). In the germination phase of plants the presence of ammonia, salts, heavy metals and/or organic acids could cause phytotoxicity (Komilis and Tziouvaras, 2009). The degree of phytotoxicity could be measured by the germination index (GI) introduced by Zucconi et al (1981). Using this index for testing the composting process Tiquia et al. (1996) observed significant differences among the types of seeds.

The quality of different soil types could be modified the effect of digestate used as a plant nutrient source in land.

1.1 Research objectives

Digestate have to use before sowing in Hungary therefore it effects on the seeds germinated in the soil. Our aim was to study the effect of digestate on the germination and early development of crops sown. We examined the modification effect of soil type and application times on the effect of applied digestate and we studied the differences among the tested plant species.

2 METHODOLOGY

2.1 Quality of biogas digestate

Liquid digestate was collected from a regional biogas plant located in Nyírbátor, Hungary. The average retention times in mesophilic and thermophilic digestors are 21 days. Digestate is stored in open-air tanks before using in plant production as a nutrient source. The average quality of the used digestate can be seen in Table 1.

TABLE 1 **Quality parameters of the used digestate**

Parameter	Mean value
pH	8.11
Density (kg m^{-3})	1025.75
Dry matter content (m/m % fresh matter)	2.003
Total Nitrogen (m/m % fresh matter)	0.361
Total Phosphorus (mg kg^{-1} fresh matter)	260
Total Potassium (mg kg^{-1} fresh matter)	473
Total Magnesia (mg kg^{-1} fresh matter)	62
Total Calcium (mg kg^{-1} fresh matter)	358
Total Sodium (mg kg^{-1} fresh matter)	263

The ingestates are plant and animal residues and cow manure therefore toxic elements content of the used digestate is low.

2.2 Soil properties used in the experiment

The sandy soil substance has slightly acidic pH with low humus and potassium content while the status of phosphorus is good. The loamy meadow soil is slightly alcalic with higher humus and good macronutrient content (Table 2)

TABLE 2 **Some chemical characteristics of the used soil substances in the germination experiment**

Soil substance	pH_{H2O}	pH_{KCl}	Y_1	$CaCO_3$ %	Humus %	NO_3-N mg kg^{-1}	P_2O_5 mg kg^{-1}	K_2O mg kg^{-1}
Sandy	6.27	4.91	7.19	0.00	0.9	8.88	202	81.5
Loamy meadow	8.13	7.38	0.00	2.29	1.8	7.53	218	159.8

Used soil substances represent the main soil types of the Nyírség region. Especially the sandy soil is frequent but its low organic and mineral colloid content caused weak water and nutrient supply. This is unfavourable for the plant production and its negative effects could be come forward at the germination of seeds.

2.3 Germination test

Germination experiment was carried out in Petri dishes (Ø=8.5 cm) filled with 80 g sand and loamy meadow soil substances which were watered to the 60 % of their field water holding capacity. Tested concentrations of digestate were 25%, 50% and 100% (v/v) and tap water was used as control. Treatments were done on the day of sowing, 5 days and 21 days before sowing. Tested plants were soybean (*Glycine soja* L.), sweet corn (*Zea mays* convar *saccharata*), rye (*Secale cereale* L.), winter wheat (*Triticum aestivum* L.) and sunflower (*Helianthus annuus* L.). These plants were investigated in field plot experiments earlier and were grouped into sensitive and non-sensitive groups on the bases of their reactions to the subsequent delivery of nutrients by digestate (Makádi et al., 2008).

The number of seeds depended on the size of them, exactly 10 seeds of soybean, sweet corn, 15 seeds of sunflower and 50 seeds of rye and, winter wheat were used in the Petri dishes. The irrigation of seedlings was done in a case of need when seeds began to show wilting symptoms.

The germination rate of tested plants was measured on the 10th day after sowing, according to the Hungarian Standard.

For toxicity tests the germination index (GI%) according to Zucconi et al. (1981) and the relative growth index (GRI) according to Alvarenga et al. (2007) are used to calculate the phytotoxic effects. We modified these indexes and used them according to the following equations:

$GI\%_R=(RWS \times GSS \times 100)/(RWC \times GSC)$ and $GI\%_S=(SWS \times GSS \times 100)/(SWC \times GSC)$

$RGI_R=RWS/RWC$ and $RGI_S=SWS/RWC$

where RWS/SWS is the root/shoot weight of the sample, RWC/SWC is the root/shoot weight of the control, GSS is the number of germinated seeds in the sample and GSC is the number of germinated seeds in the control. R and S indexes mean root and shoot. The roots and shoots were measured on the 10th day after sowing.

To evaluate the GI results we used the followings:

- GI% < 80 means inhibition,
- 80 ≤ GI% ≥ 120 means no significant effect,
- GI% > 120 means stimulation effect.
- Similarly, the RGI values were grouped into the three categories (Young et al., 2012):
- RGI < 0.8 means inhibition,
- 0.8 ≤ RGI ≥ 1.2 means no significant effect,
- RGI > 1.2 means stimulation effect.

2.4 Statistical analysis

Treatments were carried out in four repetitions. The statistical analysis of the measured values was done by one-way ANOVA following with Tukey's test for all tested plants, respectively. The SPSS 13.0 statistical program was used for the analysis.

3 EFFECT OF DIGESTATE APLICATION ON THE START OF THE PLANT'S LIFE

Different additives could effect on the quantity and on the quality of the test plants. Quantity means in our case the number of germinated seeds while the quality could be indicated by the growing and developing ability of the plants which are characterized by GI and RGI indexes. These two terms could be different as it can be seen in our results.

3.1 Results of germination on sandy soil substance

On sandy soil substance the best application forms were the 50% and 25% of digestate applied 5 days before the sowing while high concentration of digestate or application by 21 days before sowing result the decrease of germination (Table 3). Lower concentrations of digestate applied 5 days before sowing resulted in very good germination of all tested species.

For the germination of soybean the nutrient and the water seemed to be very important as our results indicate. The best germination was caused by the 100% and 50% of digestate applied 5 days before sowing. Good results of the 25% digestate and the water treatments by 5 days before sowing indicate the importance of water. High concentration of digestate (100% digestate at sowing) resulted a strong inhibition of the germination.

The germination of sweet corn was very good in all treatments, we have not found any significant treatment effect. The highest numbers of germinated seeds were resulted in the treatments before 5 days of sowing.

Similar results were found in the case of rye and winter wheat. Germination of sunflower was inhibited by the treatments of 21 days before sowing. It seems to be that this plant needs high concentration of water for the germination but the direct treatment by 100% digestate decreased its germination.

TABLE 3 **Number of germinated seeds on the 10th day after sowing on sandy soil substance**

Treatment*	**Soybean**		**Sweet corn**		**Rye**		**Winter wheat**		**Sunflower**	
	Mean	**S.D.**	**Mean**	**S.D.**	**Mean**	**S.D.**	**Mean**	**S.D.**	**Mean**	**S.D.**
Control, 21	1.00 a	1.00	6.33 a	1.15	21.00 abc	4.36	39.33 c	2.08	14.33 b	1.15
Control, 5	6.67 bc	1.53	9.67 a	0.58	40.67 de	1.53	41.67 c	1.53	13.00 b	1.00
Control, 0	5.00 abc	1.73	6.00 a	5.19	7.00 ab	6.56	35.00 bc	2.00	12.67 b	0.58
100% digestate, 21	6.33 bc	1.15	8.67 a	1.53	27.67 cde	5.51	17.67 ab	19.35	7.00 a	1.73
100% digestate, 5	8.33 c	1.15	9.33 a	0.58	40.00 de	2.65	38.67 c	4.93	13.67 b	0.58
100% digestate, 0	1.67 a	1.15	8.00 a	2.65	13.33 abc	4.04	15.33 ab	2.08	7.00 a	0.00
50% digestate, 21	1.33 a	1.15	6.67 a	2.52	6.33 a	3.21	8.67 a	1.53	6.67 a	0.58
50% digestate, 5	9.00 c	1.00	10.00 a	0.00	42.67 e	4.16	44.00 c	1.73	14.33 b	0.58
50% digestate, 0	5.33 abc	1.53	7.67 a	2.52	24.33 bcd	4.51	38.67 c	6.03	12.67 b	1.53
25% digestate, 21	2.67 ab	1.53	9.00 a	1.73	23.33 abcd	1.53	29.67 bc	8.50	11.67 b	4.16
25% digestate, 5	6.67 bc	1.53	10.00 a	0.00	42.33 e	2.08	45.33 c	3.51	13.00 b	1.00
25% digestate, 0	6.33 bc	2.52	9.67 a	0.58	21.67 abc	16.26	41.33 c	2.52	13.67 b	1.53

* 21: 21 days before sowing, 5: 5 days before sowing, 0: at sowing. S.D. means Standard Deviation. Red colour: the smallest germination rates. Green colour: the highest germination rates.
a,b,c,d indexes indicate statistically significant differences among the treatments at $P<0.05$ according to Tukey's test.

The intensive germination of soybean was started on the day 6 and the treatment effects were strong. The sweet corn germinated quicker and the treatment effect was less expressed. Rye germinated more or less slow with expressed treatment effect, while the winter wheat germination was the best in a control treatment until the day 7. After that the digestate treated plants started quick germination. The germination of sunflower was similar to the winter wheat but the intensive phase started on the day 5.

The computed indexes (GI%, RGI) also indicated the hazardous property of the digestate applied at high rate and in the sowing time (Table 4). The RGI was more sensitive than GI%, according to the results of Young et al. (2012). Therefore we present only the values of the computed RGI in the tables.

On the bases of these indexes the winter wheat and sunflower are very sensitive plants for the digestate treatment in their germination period. The relative growth index shows that the root elongation and growth of winter wheat is the most sensitive among the tested plants. Only the 25% digestate treatment resulted in a small increase of its root. The digestate treatments were indifferent or had negative effect in some cases on the growth of the root and shoot of sunflower.

The less sensitive plant was soybean followed by sweet corn and rye. The results proved that the similar plant species – in our case the rye and winter wheat – could have different reactions to an environmental impact.

The germination of sweet corn was very good and the development of its root and shoot were also stimulated or not effected by digestate. In the case of rye the germination was not too succeed but the development of the plant was very intensive.

TABLE 4 **Relative growth index of root and shoot of test plants on the 10th day after sowing on sandy soil substance**

Treatment *	**Soybean**		**Sweet corn**		**Rye**		**Winter wheat**		**Sunflower**	
	RGI_R	**RGI_S**	**RGI_R**	**RGI_S**	**RGI_R**	**RGI_S**	**RGI_R**	**RGI_S**	**RGI_R**	**RGI_S**
100% digestate, 21	1.7 abc	2.7 a	1.6 bc	1.1 ab	1.8 d	1.3 ab	0.3 ab	0.7 ab	1.1 a	0.7 a
100% digestate, 5	2.0 abc	2.8 a	0.8 ab	1.1 ab	0.8 abc	1.1 ab	0.6 abc	0.6 ab	0.9 a	1.2 a
100% digestate, 0	0.9 abc	0.2 a	1.0 ab	1.5 ab	0.8 ab	1.1 ab	0.3 ab	0.8 ab	0.4 a	0.8 a
50% digestate, 21	0.6 a	0.7 a	0.4 a	0.8 a	0.4 a	0.4 a	0.2 a	0.3 a	1.1 a	0.7 a
50% digestate, 5	2.8 c	8.5 b	2.3 c	1.7 ab	1.0 abcd	1.2 ab	0.7 abcd	0.8 ab	0.6 a	1.2 a
50% digestate, 0	1.5 abc	0.4a	0.8 ab	1.2 ab	1.7 d	2.3 ab	0.7 bcd	2.1 c	1.3 a	1.2 a
25% digestate, 21	0.8 ab	1.2 a	1.7 bc	1.1 ab	1.4 bcd	1.6 ab	0.7 bcd	0.9 ab	0.8 a	1.1 a
25% digestate, 5	2.8 c	11.3 b	1.4 abc	1.9 b	1.6 cd	1.3 ab	1.2 d	1.1 b	1.0 a	1.2 a
25% digestate, 0	2.7 bc	0.8 a	1.7 bc	2.0 b	1.1 abcd	4.5 b	1.1 cd	1.9 c	0.9 a	1.2 a

* 21: 21 days before sowing, 5: 5 days before sowing, 0: at sowing. Red colour means inhibition. Yellow colour means no effect. Green colour means stimulation.
a,b,c,d indexes indicate statistically significant differences among the treatments at $P<0.05$ according to Tukey's test.

In sandy soil substance the digestate application before sowing have not caused dangerous decrease in plant germination and in the development of plants. The best application form is the low concentration applied some days before sowing.

3.2 Results of germination on loamy meadow soil substance

Germination of soybean was better and quicker on loamy meadow soil than on sandy soil substance but high digestate dose and the earlier application caused strong reduction in germination. On loamy meadow soil substance the sweet corn germination were increased by digestate, but in a smaller rate than on the sandy soil substance. Treatment effect has not found but also the lowest doses were more effective. The rye and winter wheat germination were more favourable when digestate treatments were done 21 days before sowing. Similar results were found in the case of sunflower. The germination of these three species was worse in this soil type.

The computed relative growth index (Table 6) shows stronger inhibition effect of digestate on the development of root and shoot of tested plants comparing to sandy soil. Although the most favourable application method was the 50% digestate applied by 5 days before sowing, similar to the results obtain in the sandy soil. Generally, the tested plant species are more sensitive in loamy meadow soil to the digestate treatment. The development of sweet corn differs from this statement because its shoot development was stimulated by digestate treatments. For its root development the 25% and 50% of digestate applied 5 days before sowing were the best treatment, too.

TABLE 5 **Number of germinated seeds on the 10th day after sowing on loamy meadow soil substance**

Treatment*	**Soybean**		**Sweet corn**		**Rye**		**Winter wheat**		**Sunflower**	
	Mean	**S.D.**	**Mean**	**S.D.**	**Mean**	**S.D.**	**Mean**	**S.D.**	**Mean**	**S.D.**
Control, 21	7.67 abc	1.53	9.33a	0.58	38.67 c	2.08	37.00 b	10.58	13.00 b	1.73
Control, 5	8.33 bc	0.58	9.33 a	0.58	14.00 ab	7.81	7.00 a	1.00	13.00 b	1.73
Control, 0	9.67 c	0.58	10.00 a	0.00	25.00 bc	2.00	5.67 a	0.58	14.33 b	1.15
100% digestate, 21	7.33 abc	0.58	9.00 a	1.00	36.67 bc	2.08	37.33 b	2.31	13.33 b	0.58
100% digestate, 5	8.33 bc	0.58	7.33 a	2.31	0.33 a	0.58	3.33 a	2.31	5.67 a	2.08
100% digestate, 0	5.00 ab	1.00	9.67 a	0.58	15.33 ab	1.53	3.67 a	0.58	5.33 a	4.73
50% digestate, 21	3.67 a	2.31	9.67 a	0.58	32.67 bc	2.31	35.00 b	5.20	13.33 b	1.15
50% digestate, 5	8.00 bc	1.00	9.33 a	0.58	30.67 bc	5.86	7.00 a	4.36	5.00 a	2.00
50% digestate, 0	10.00 c	0.00	10.00 a	0.00	18.67 abc	2.08	4.00 a	1.73	13.00 b	0.00
25% digestate, 21	3.67 a	3.51	9.00 a	1.73	28.67 bc	23.97	18.33 ab	25.70	13.33 b	1.53
25% digestate, 5	9.67 c	0.58	9.33 a	0.58	16.67 abc	5.03	1.33 a	2.31	9.00 ab	5.20
25% digestate, 0	9.67 c	0.58	10.00 a	0.00	16.33 abc	0.58	3.00 a	1.73	13.00 b	2.00

* 21: 21 days before sowing, 5: 5 days before sowing, 0: at sowing. S.D. means Standard Deviation. Red colour: the smallest germination rates. Green colour: the highest germination rates.

a,b,c,d indexes indicate statistically significant differences among the treatments at $P<0.05$ according to Tukey's test.

TABLE 6 **Relative growth index of root and shoot of test plants on the 10th day after sowing on loamy meadow soil substance**

Treatment *	**Soybean**		**Sweet corn**		**Rye**		**Winter wheat**		**Sunflower**	
	RGI_R	**RGI_S**	**RGI_R**	**RGI_S**	**RGI_R**	**RGI_S**	**RGI_R**	**RGI_S**	**RGI_R**	**RGI_S**
100% digestate, 21	🟩 1,9 b	🟥 0,6 abc	🟨 0,9 a	🟩 1,3 a	🟨 1,0 a	🟩 1,2 b	🟨 0,8 a	🟨 1,1 ab	🟨 0,9 abcd	🟨 1,1 c
100% digestate, 5	🟥 0,3 a	🟨 0,8 bc	🟨 0,8 a	🟨 0,8 a	🟥 0,0 a	🟥 0,0 a	🟥 0,1 a	🟥 0,2 a	🟥 0,5 abcd	🟥 0,4 a
100% digestate, 0	🟥 0,5 ab	🟥 0,2 a	🟨 0,9 a	🟩 1,3 a	🟥 0,5 a	🟥 0,6 ab	🟨 0,8 a	🟨 1,1 ab	🟥 0,1 a	🟥 0,5 abc
50% digestate, 21	🟥 0,5 ab	🟥 0,2 ab	🟥 0,7 a	🟩 1,3 a	🟨 0,8 a	🟨 1,0 ab	🟨 0,8 a	🟨 1,1 ab	🟩 1,3 d	🟨 1,1 c
50% digestate, 5	🟩 1,2 ab	🟥 0,5 abc	🟨 1,1 a	🟩 1,3 a	🟩 2,7 b	🟩 2,9 c	🟩 1,2 a	🟩 3,2 b	🟥 0,3 ab	🟥 0,4 ab
50% digestate, 0	🟨 0,8 ab	🟥 0,7 abc	🟥 0,7 a	🟩 1,8 a	🟥 0,6 a	🟨 0,9 ab	🟥 0,6 a	🟩 1,8 ab	🟨 1,1 bcd	🟨 0,8 abc
25% digestate, 21	🟥 0,2 a	🟥 0,1 a	🟥 0,6 a	🟩 1,2 a	🟥 0,7 a	🟥 0,7 ab	🟨 0,8 a	🟥 0,6 a	🟩 1,3 cd	🟨 1,0 bc
25% digestate, 5	🟨 1,1 ab	🟨 1,0 c	🟨 1,1 a	🟨 0,9 a	🟩 1,2 a	🟩 1,4 b	🟥 0,1 a	🟥 0,2 a	🟥 0,3 abc	🟥 0,6 abc
25% digestate, 0	🟨 0,9 ab	🟨 0,8 c	🟨 1,0 a	🟩 1,6 a	🟨 0,8 a	🟨 1,0 ab	🟥 0,7 a	🟩 2,0 ab	🟨 1,0 abcd	🟨 0,8 abc

* 21: 21 days before sowing, 5: 5 days before sowing, 0: at sowing. Red colour means inhibition. Yellow colour means no effect. Green colour means stimulation.

a,b,c,d indexes indicate statistically significant differences among the treatments at P<0.05 according to Tukey's test.

In loamy meadow soil substance the best digestate application method is lower concentrations near sowing in the case of soybean and sweet corn while 50% and 100% digestate applied 21 days before sowing in the case of rye, winter wheat and sunflower.

4 DISCUSSION

Application of digestate on field is restricted before sowing in Hungary therefore the digestate could has strong effect on the germination of which rate determines the efficiency of crop management. This effect could be positive (stimulation) or negative (inhibition). The main reasons of the inhibition of germination could be the concentration of organic matter (Bohórquez-Echeverry and Campos-Pinilla, 2007), salinity (Tam and Tiquia, 1994), heavy metal content (Tiquia et al., 1996). The digestate is a very complex material therefore the correct identification of its toxic factor(s) is difficult (Tam and Tiquia, 1994). Gulyás et al. (2012) identified the ammonium content of digestate as a main factor of its toxicity.

Marchiol et al. (1999) found that the sensitivity of seeds is decreased by the order of legume seeds, grasses seeds and wild herb seeds when they assessed the impact of municipal solid waste compost and soil leachate. Our results also proved that the sensitivity of germination depends on plant species, but this sensitivity is modified by the soil type. The soybean utilized well the digestate in the sandy soil substance while it was sensitive in the loamy meadow soil substance. Gell et al (2011) also found that pig and cow digestate has no phytotoxic effect on sandy soil when they used lettuce, radish and summer wheat seeds in germination tests. On the other hand, the number of germinated seeds was higher in the loamy meadow soil substance which means that the fewer germinated seeds on sandy soil substance resulted higher shoots and roots. Generally, the application of digestate on loamy meadow soil with higher colloid and organic material content seems to be most hazardous for the germination.

Plants could be sensitive for the digestate in their full of life and this sensitivity also depends on the plant species. In our earlier study the plants were grouped to sensitive and non-sensitive categories when digestate was used as a nutrient source in a developing crop (Makádi et al., 2008). Soybean and sunflower were also very sensitive for the digestate treatment.

5 CONCLUSIONS

The effect of digestate on the germination of seeds depends on the

- type of soil,
- plant species (perhaps variety, but it was not examined),
- doses of digestate,
- period of digestate application before sowing.

Overall, the most sensitive plants were winter wheat and sunflower in both soil substances. For these plant species, only the smallest digestate dose (25%) not caused inhibition, or the germination of these species could be increased in a small rate. The number of germinated seeds and living plants were increased in the case of non-sensitive plant species.

Application of digestate for improving the germination rate of crops plants is more effective on sandy soil substance. To take into account the properties of this soil type, the use of digestate as a plant nutrient is very important not only in the point of the environmental protection but also in the point of the sustainable crop production.

Generally, early application of digestate causes the loss of nutrients and water and therefore its positive effects are decreased. On the other hand, the high doses of digestate applied near the sowing results the decrease of germination. On loamy meadow soil substance, the inhibitory effect of digestate application was experienced more often, the tested species seemed to be more sensitive than on sandy soil substance, where the treatments increased the germination in a higher rate, comparing to the controls.

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(84) CORRELATION BETWEEN PHYTOTOCIXITY AND TOTAL AND BIOAVAILABLE HEAVY METALS OF COMPOSTS

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EXECUTIVE SUMMARY

Composting is one of the methods mostly used for organic solid wastes recycling. The compost is mainly used as a soil conditioner due to its carbon and nitrogen content. Although such applications are beneficial to soil, concern about possible environmental hazards, like odor, water pollution, veterinary hazards, and contamination by heavy metals have to be considered. The main limiting factor, in order to use compost as soil amendment, is the total concentration of heavy metals. Therefore, it is important to understand the heavy metals fractionation and their bioavailability to the soil–plant system, which may be correlated with the composts phytotoxicity.

The purpose of this work was to determine the content of six heavy metals (Zn, Pb, Ni, Cu, Cr and Cd) and their bioavailability in composts commercialized in Portugal and correlate with their phytotoxicity.

Four commercial composts, two from non-separated municipal solid waste (MC1, MC2), one from source-separated municipal solid waste (MC3), and one produced from poultry litter (PC), were analysed. The stability of composts was characterised using the self-heating test. The fraction of heavy metals was evaluated following the sequential BCR (Community Bureau of Reference) extraction; the composts were treated with a chain of reagents to quantify the elemental concentration in 4 different fractions (exchangeable/bioavailable, reducible, oxidiseable and residual). The phytotoxicity tests were performed using two different types of seeds: cress (*Lapidium sativum)* and radish (*Raphanus sativus*).

All the composts presented organic matter content higher than 35% and pH values between 8 and 9. The MC3 and PC composts showed the lowest heavy metal content. Zn was the heavy metal present in the highest concentration (861 mg/kg dry matter), while Cd was the least representative (3.0 mg/kg dry matter). When using a sequential extraction method, only Zn (9% - 23%) was bound to the exchangeable fraction (bioavailable form). The highest proportion of the heavy metals were associated with the fractions more resistant to extract, namely to the residual fraction. The compost MC3 was the most stable, and PC compost presented the highest germination index (72%), which suggests that it was the only compost without phytotoxicity.

A correlation analysis indicated that the chemical properties of the composts, such as the organic matter content, can influence the germination index, besides the Cr content in the oxidisable and reducible fractions. These results showed that others parameters besides the bioavailable heavy metals might influence the phytotoxicity of the composts.

1 INTRODUCTION

1.1 Background

Composting is one of the methods mostly used for organic solid wastes recycling. Several characteristics determine the compost quality for land application, such as particle size distribution, moisture, organic matter, carbon and nitrogen content, concentration and composition of humus-like substances, forms of N, P and K, heavy metals content, salinity and its nature, cation exchange capacity, water holding capacity, porosity and bulk density, inert contaminants, pathogens, and state of maturity or stability (Lasaridi, 1998). However, concern about possible environmental pollution hazards, for example, odor, water pollution, veterinary hazards, and contamination by heavy metals need to be considered (Baveye et al., 1999). The assessment of composts toxicity by chemical characterization and biological testing is therefore extremely important for screening the suitability of composts for land application as soil amendments. Assessing the phytotoxicity of composts is one of the most important criteria being used to avoid environmental risks before these composts can be recycled back to agricultural soils (Tiquia at al, 1996). A widely used test is the germination index (GI) that quantifies seed growth upon the application of compost liquid extracts to the seeds (Komilis et al., 2009). Phytotoxicity usually arises from the presence of ammonia, salts, heavy metals and/or organic acids in the compost extract. The total concentration of heavy metals is the main limiting factor, for any use that the compost may have (Epstein, 1997). The determination of the total metal concentration may indicate the overall level of metals in compost, but provides little information about the forms in which heavy metals are present, or about their potential for mobility and bioavailability in the environment (Cai et al., 2007). The heavy metals distribution in organic products is influenced by the pattern of organic matter mineralization or its pH, since the metal solubilization is affected by the pH decrease, metal biosorption by the microbial biomass or metal complexation with the newly formed humic substances, among others (Hsu et al., 2001). Their mobility and bioavailability in a given medium are very important, and thus the assessment of concentrations and chemical speciation of heavy metals in composts enables to evaluate their bioavailability and suitability for land application. Therefore, it is important to know the distribution of the heavy metals in the different compost fractions to assess their bioavailability to the soil–plant system, given it may be correlated with the phytotoxicity of composts.

1.2 Research objectives

In this study, it was intended to assess the quality of four composts commercialized in Portugal. The physical and chemical properties, including the heavy metals content in the exchangeable, reducible, oxidisable and residual fractions were correlated with their phytotoxicity.

2 METHODOLOGY

Four commercial composts, two from non-separated municipal solid waste (MC1, MC2), one from source-separated municipal solid waste (MC3) and one produced from poultry litter (PC), were analysed. The physic and chemical characteristics (pH, electrical conductivity, moisture, organic matter, total carbon, total nitrogen and N-NH_4^+) of compost samples were determined according to standard procedures, as reported by Silva et al. (2009). The germination bioassays were made using two different types of seeds: cress (*Lapidium sativum*), and radish (*Raphanus sativus*). Germination percentage and root length were measured after an incubation period of two days and were expressed as a percentage of the corresponding control values. According to the above description, germination index (GI) was calculated based on the following formula (Zucconi et al., 1981):

$$\text{Germination index (\%)}=\frac{\text{Seed germination (\%)}\times\text{root length of treatment}}{\text{Seed germination (\%)}\times\text{root length of control}}\times 100$$

The ability of the material to reheat was evaluated using a Dewar self-heating test. Vessels (1.5 L) were filled with compost samples with a standardized moisture content of 35%. Temperature evolution was measured until heat production had ceased. According to Laga-Merkblatt M10 (1995), the class of stability was attributed by taking into account the maximum temperature reached in each vessel: class I –temperature >60 °C; class II –temperature between 50 and 60 °C; class III –temperature between 40 and 50 °C; class IV –temperature between 30 and 40 °C; class V –temperature <30 °C. Heavy metals content (Zn, Cu, Ni, Cd, Cr and Pb) were analysed by flame atomic absorption spectrophotometry (Perkin Elmer Atomic Absorption Spectrophotometer) after samples digestion with “aqua regia”, followed by filtration (EN 13650, 2001).

Metal speciation was performed using the modified BCR (Community Bureau of Reference) sequential extraction (three step) procedure (Rauret et al., 2000): first step – extraction with 0.11 M acetic acid to obtain exchangeable, water and acid soluble species; second step – solubilization using 0.5 M hydroxylamine hydrochloride to obtain metals associated with the reducible phases, e.g. bound to Fe/Mn oxyhydroxides; third step – oxidation with 8.8 M hydrogen peroxide, followed by extraction using 1.0 M ammonium acetate to obtain the oxidisable metal fraction, e.g. bound to organic matter or sulphides and the residual fraction.

2.1 Statistic Analyses

All analyses were conducted in triplicate and data were subjected to one-way analysis of variance (ANOVA) and the Newman-Keuls test was used to separate the means. Correlations (Pearson correlation) analysis was also determined. These analyses were carried out using Statistica 6.0.

3 RESULTS AND DISCUSSION

Generally, it is recognized the relevance of physical and chemical characteristics on compost quality. The acceptable values for each parameter may depend on the intended applications of the product namely, if the compost is to be used as landfill cover or in agriculture. Based on the European Union (EU) working document "Biological Treatment of Biowaste" (DG ENV.A.2, 2001), some guidelines for a Portuguese regulation of compost quality have been proposed for composts to be used in agriculture (Anonymous, 2008) and those guidelines are followed in the present discussion. The EU eco-label criteria for soil improvers (EC, 2006) are also explored in this discussion.

The physical and chemical properties of the analysed composts are shown in Table 1. Moisture content varied from 13% to 53% for all composts. Among the analysed composts, only MC2 exceeded the Portuguese limit of 40% moisture, the standard set an upper limit value for moisture content to prevent "selling water" and the development of anaerobic conditions during storage (Lasaridi et al., 2006). The organic matter (OM) content of the analysed composts had a rather wide variation, from 43.6% for PC to 71.6% for MC2. Although OM content is strongly influenced by raw materials and by the effectiveness of the microbial degradation along the process (Epstein, 1997), all the analysed composts were higher than the OM minimum recommended values, 30 % and 20 %, according to Anonymous (2008) and EC (2006), respectively. To receive the EU eco-label, products should contain less than 3% N total. This value was met by all samples examined. MC3 and MC2 had the highest (2.4%) and the lowest N content (1.6%), respectively. Corresponding C/N values ranged from 8.7 to 20.3 for PC and MC2, respectively. The composts presented a pH value within the recommended range for agriculture applications (pH 5.5-8.5) (Anonymous, 2008), except the PC compost. The neutral to slightly alkaline characteristics is an indicator of stable composts. Nevertheless, in the poultry manure compost (PC), the high pH combined with elevated concentrations of ammonium-nitrogen, may lead to the formation of toxic levels of ammonia (NH_3), inhibiting microbial and vegetal development. The electrical conductivity of composts reflects their salt content and may be important at several levels, namely the inhibition of microbiota, the leaching of salts with the consequent environmental contamination, and the inhibition of crops germination, if compost is to be used in agriculture (Epstein, 1997; Komilis et al, 2009). All composts presented electrical conductivity values higher than 1.5 mS cm^{-1} and 4 mS cm^{-1}, which are considered the upper values for growing media (EC, 2006), and tolerable by plants of medium sensitivity (Lasaridi et al., 2006), respectively.

TABLE 1 **Physical and chemical properties of the composts.**

	MC1	MC2	MC3	PC
Moisture (%)	13.1±0.5a*	53.7±1.0b	13.6±0.2a	40.0±2.7c
Organic matter (% d.m.)	44.5±0.3a	64.5±1.1b	52.3±1.4c	33.2±2.0d
Total carbon (% d.m.)	22.2±0.2a	32.2±0.6b	26.2±0.7c	16.6±1.0d
Total nitrogen (% d.m.)	1.9±0.1a	1.6±0.1b	2.4±0.1c	1.9±0.1a
NH_4^+-N (mg kg^{-1} d.m.)	1210±78a	2417±65b	1576±157c	7160±225d
C:N ratio	11.7±1.0a	20.1±1.6b	10.9±0.5a	8.7±0.5c
pH	8.4±0.1a	8.0±0.1b	8.4±0.1a	9.0±0.0c
Electrical Conductivity (mS cm^{-1})	6.5±0.1a	4.9±0.1b	8.1±0.6c	5.6±0.0d

d.m- dry matter; *values in a row followed by different letters are statistically different ($p<0.05$)

Although physical and chemical parameters are key features for compost, the main requirement for a compost to be safely used as soil conditioner or fertilizer is its degree of stability and maturity, which implies stable organic matter content, the absence of phytotoxic compounds and plant or animal pathogens (Bernal et al, 1998). Compost is considered stable when submitted to the self-heating test, in Dewar vessels, and its temperature does not exceed 40 ºC, which corresponds to stability degrees IV and V. According to Table 2, stability degree V was observed for compost MC3, indicating its high stability. In opposition, compost MC2 reached a maximum temperature of 70°C and was classified with the stability degree I, indicating that it was not stable.

TABLE 2 **Class of stability and germination index results.**

	MC1	MC2	MC3	PC
Class of Stability	III	I	V	III
GI (%) Radish (*Raphanus sativus*)	33.6±15.6	0	42.4±3.6	49.1±16.5
GI (%) Cress (*Lepidium sativum*)	29.5±11.7	0	43.9±25.2	71.9±22.3

The phyto-inhibitory/phytotoxicity is reflected in the germination index. When using radish and cress seeds, PC compost presented the highest germination index (average of 49% and 72%, respectively). However, the GI values for the other composts were below to 60% indicating that they are phytotoxic (Pera et al., 1991). The high NH_4^+-N content of compost PC may be responsible for the partial inhibition of seeds germination. The total inhibition of seed germination by compost MC2 emphasizes its non-stability/maturity.

While the above mentioned parameters assume great relevance if the compost is to be used in agriculture, the presence of heavy metals and their leaching potential, leading to the contamination of surrounding environment is of surmount importance for any use that the compost may have (Epstein, 1997). According to Ramos (2006), continuous compost application to soil leads to an increased metal bioavailability, which may represent a potential risk of metal toxicity. In general, the MC1 compost showed the highest content in heavy metals (Table 3). Indeed, this compost exceeds the Cu upper limit content, according to the Portuguese regulation proposal for class III composts. MC2 is classified as class III compost because the Cr content is higher than 150 mg/kg dry matter. Therefore, it can only be applied in areas not devoted to crops for food and feed. MC3 and PC composts comply with the limits defined in the Portuguese regulation proposal (Anonymous, 2008) for class I and in the eco-label standard (EC, 2006) being possible to use them as soil amendment.

TABLE 3 **Total heavy metals content of composts and limit values for classes I, II and III proposed in the Portuguese regulation proposal (Anonymous, 2008).**

Heavy Metals (mg/kg d.m.)	MC1	MC2	MC3	PC	Class I	Class II	Class III
Zn	860.9±24.5a*	389.2±4.3b	132.3±1.4c	177.7±7.0d	200	500	1500
Cu	749.1±26.5a	141.2±2.5b	41.2±3.8c	76.6±3.5d	100	200	600
Ni	73.8± 6.8a	30.6±3.3b	17.7±0.5c	15.4±1.1c	50	100	200
Cr	84.9±5.5a	180.2±3.2b	45.6±4.2c	89.4±1.3a	100	150	400
Cd	3.0±0.1a	1.3± 0.1b	1.0±0.1c	0.8±0.0d	0.7	1.5	5.0
Pb	242.8±15.4a	143.0±3.4b	36.2±5.5c	20.8±0.3c	100	150	500

d.m- dry matter; *values in a row followed by different letters are statistically different ($p<0.05$)

Zn and Cd were the heavy metals present in the highest (861 mg/kg dry matter) and the lowest (0.8 mg/kg dry matter) concentration, respectively. As stressed above, it is also important to study the heavy metals leachability. Metals in the exchangeable and reducible fractions are relatively labile and may be potentially bioavailable, while metals in the oxidisable fractions (organically bound) and residual fractions are relatively immobile and may not be readily bioavailable (Hsu et al, 2001; Fuentes et al, 2004).

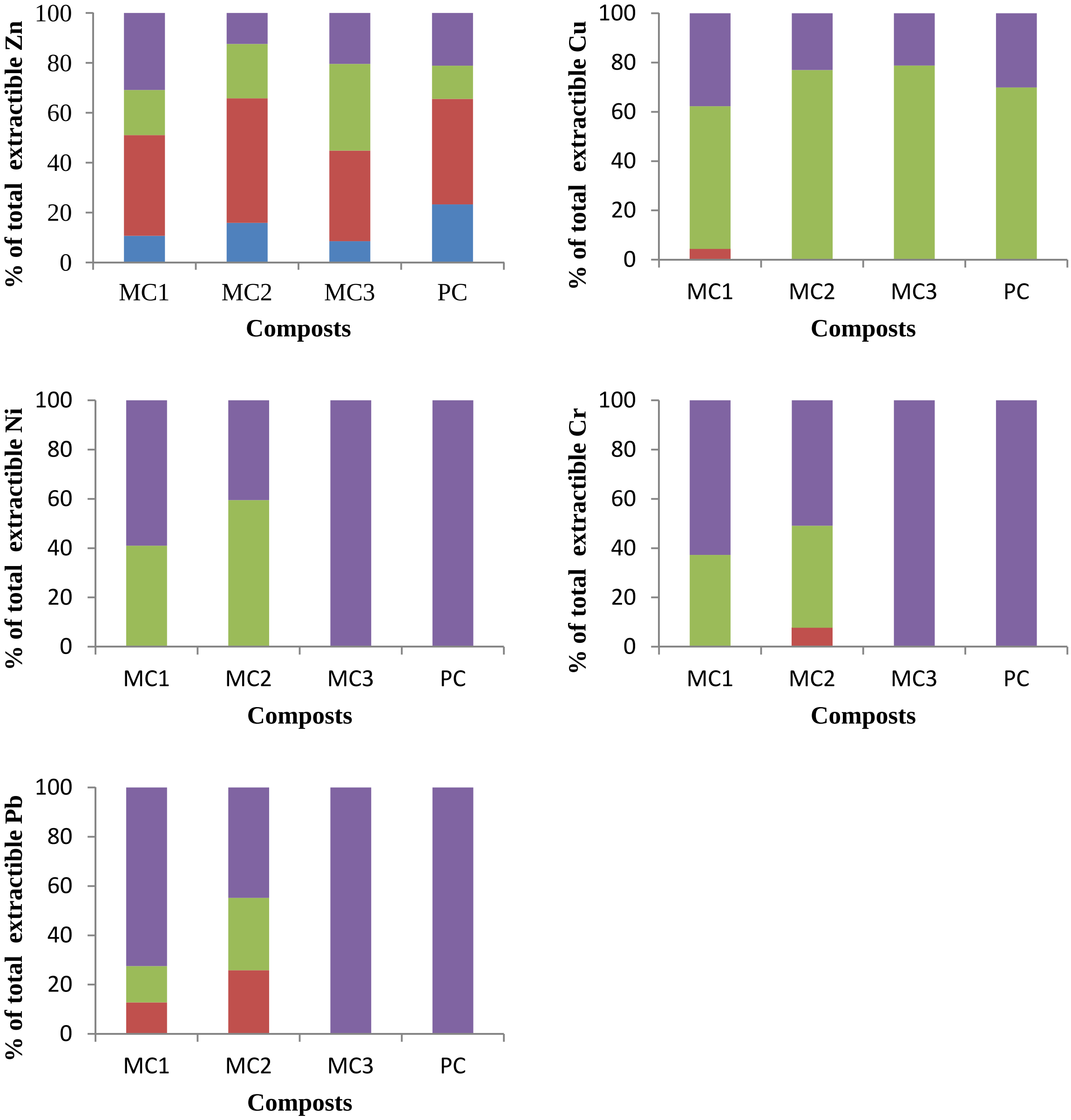

FIGURE 1 **Sequential extraction of heavy metals** (🟦Exchangeable 🟥Reducible 🟩Oxidisable 🟪Residual).

Zn showed the greatest degree of mobility, and consequently of potential bioavailability, as seen from the high proportion of this metal in the exchangeable (9-23%) and reducible (36-50%) fractions (Figure 1), a result also found by Fuentes et al. (2004). Cu was mainly associated with the organic matter (OM) of the composts, given the highest proportion of this metal was detected in the oxidisable fraction of all composts. Given Cu produce stable complexes with OM, these results were not unexpected. The sum of the last two fractions accounted for more than 95% of the total Cu, in all the composts, which indicated that Cu was associated with strong organic ligands and probably occluded in minerals like quartz, and/or feldspars. Ni was associated mainly with the residual fraction (40-100%) and oxidisable fraction (0-60%). Although present at high concentrations, Cr was principally distributed between the oxidisable (0-49%) and residual (51-100%) fraction. In MC3 and PC composts, 100% of Pb was found in the residual fraction. However in the less stable composts (MC1 and MC2), which contained higher concentration of Pb than composts MC3 and PC, this metal was found in reducible fraction (13-25%), suggesting that it has some potential of leaching. Cd was not extractible by the sequential extraction process.

Although the composts are classified according to the total heavy metals content, our results show that, excepting Zn, only a small fraction of the metals analyzed are bioavailable (present in the exchangeable and reducible fraction).

To determined if and how the key parameters affect the compost phytotoxicity, a Pearson correlation analysis was used to correlate the germination index with physical and chemical characteristics of the compost (Table 4 and 5). Negative correlations between germination index and the proportion of the heavy metals in the exchangeable, reducible, and oxidisable fractions were observed. Cr correlated negatively with the germination index irrespective of the degree of its mobilization in the compost. This fact can be related with the absence of germination seed for the MC2 compost, as this compost showed the highest total Cr content and the one that presented about 8% of its reducible fraction.

TABLE 4 **Correlation matrix between the germination index and bioavailable heavy metals of composts.**

	Zn Exc	Zn Rd	Zn Ox	Zn Rs	Cu Exc	Cu Rd	Cu Ox	Cu Rs	Ni Ox	Ni Rs	Cr Rd	Cr Ox	Cr Rs	Pb Rd	Pb Ox	Pb Rs
GI (%) Radish	-0.79**	-0.68*	-0.64*	NS	NS	NS	NS	NS	-0.59*	NS	-0.95**	-0.99**	-0.86**	-0.75**	-0.74**	NS
GI (%) Cress	-0.80**	-0.77**	-0.81**	NS	-0.62*	NS	NS	NS	-0.71*	NS	-0.80**	-0.91**	-0.66*	-0.82**	-0.81**	NS

** Correlation is significant at the 0.01 level (2-tailed); * Correlation is significant at the 0.05 level (2-tailed); NS - not significant; Exc – exchangeable; Rd- reducible; Ox-Oxidisable; Rs - residual

Other factors that affected negatively the germination index were the organic matter content and the C/N ratio, which can indicate that the degree of stabilization of organic matter is positive for the germination. The Cr content correlated negatively with other physical and chemical characteristic, mainly with the C/N ratio (data not shown). A positive correlation was obtained between the germination index and the pH. This correlation was expected because matured composts tend to have pH alkaline which can avoid the bioavailability of heavy metals. In fact, the significant correlations found between total heavy metals, their fractions and compost pH were always negative (data not shown).

TABLE 5 **Correlation matrix between the germination index and the physical and chemical properties of composts.**

	Zn	Cu	Ni	Cr	Cd	Pb	OM	pH	EC	NH_4^+-N	C/N
GI (%) Radish	NS	NS	NS	-0.90**	NS	NS	-0.85**	0.88**	NS	NS	-0.98**
GI (%) Cress	NS	NS	NS	-0.70*	NS	-0.63*	-0.90**	0.98**	NS	0.687*	-0.92**

** Correlation is significant at the 0.01 level (2-tailed); * Correlation is significant at the 0.05 level (2-tailed); NS - not significant

In summary, the correlation analysis suggests that bioavailability of heavy metals influenced the phytotoxicity of the composts. However, given the heavy metal content of some composts were compatible to their use in agriculture, other parameters may have contributed for their phytotoxicity.

4 CONCLUSIONS

The physical and chemical parameters analysed revealed that among the commercially available composts, the compost from source-separated municipal solid waste (MC3), fulfilled most of the recommendations proposed for compost use in agriculture, except for the electric conductivity. The others, particularly MC2, composts failed criteria related with heavy metals content, self-heating potential and germination index. Moreover, evidence has been gained that stability and maturity are distinct properties, which are very important in the assessment of compost quality. In fact, some composts falling in the same class of stability, revealed different levels of maturity evaluated through the germination index. PC compost (from poultry litter), presented the highest germination index, which suggests it was the only compost without phytotoxicity. Zn was the element present in the highest concentration and higher mobility in all composts. In opposition, Ni presented the lower availability. A correlation analysis indicated that heavy metal content in the exchangeable, exchangeable, reducible, and oxidisable fractions contributes for phytotoxicity of the composts. Among the heavy metals determined, Cr seems to be the most toxic. Other factor that promoted phytotoxic characteristics was the organic matter content, which can indicate that the degree of stabilization of organic matter is positive for the germination.

Finally, the physical, chemical, stability and maturity characteristics of studied commercial composts emphasizes the necessity of an adequate management of composting process, mainly in what concerns the selection of biowastes, and the control conditions of active and curing phases in order to improve their quality.

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(116) FATE OF SOIL-APPLIED OLIVE MILL WASTEWATER AND POTENTIAL PHYTOTOXICITY ASSESSED BY TWO BIOASSAY METHODS

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EXECUTIVE SUMMARY

Controlled land spreading of untreated olive mill wastewater (OMW) has been widely practiced as a mean of their disposal and is now permitted by law in several countries. In this respect, OMW is considered as a natural fertilizer, which, at proper application rates, is not harmful to crops and can be disposed of without causing environmental damage. Still, the inherent phytotoxicity of OMW is of major concern and requires careful management. Multiple studies based their phytotoxicity assessments on soil extracts, while it is questionable whether extract-based bioassays reflect reliably the actual phytotoxicity of the soil *in situ*.

The present study investigates the fate of soluble OMW constituents (dissolved organic carbon, DOC and total phenols, TP) and the dynamics of their phytotoxicity during soil incubation. Equivalent application doses of 80, 160 and 320 m^3 ha^{-1} were applied to two soil types: Vertisol (57% clay) and Loess (75% sand), representing two main geographical regions in Israel that are not considered hydrologically sensitive and thus relatively safe for OMW spreading. Soil phytotoxicity was assessed using cress (*Lepidium sativum* L.) both on the soil extracts and by applying a new whole-soil bioassay (modified from Weiss et al., 2006 and Prasad et al., 2010). Major differences were found between the results of these two methodologies: At time 0, the phytotoxicity of the Vertisol soil was shown to be substantial at all doses by the extract-based bioassay, whereas the whole-soil bioassay showed phytotoxicity at 320 m^3 ha^{-1} only. Such differences were less substantial for the Loess soil. On the other hand, after 5 days of incubation, during which the DOC and TP substantially reduced, the whole-soil bioassay generally showed only slight negative effect or almost full recovery, whereas the soil extract showed opposite effect (seed growth stimulation) at 80 and 160 m^3 ha^{-1}.

Overall, with respect to soil phytotoxicity, the results support the safe application of commonly permitted doses in the range of 50-100 m^3 ha^{-1} with only short-term (days) phytotoxic effects. Out of the two tested bioassay methodologies, the extract-based bioassay seems to over-estimate both the negative and positive potential effects of OMW application. Possible artefacts associated with extract-based bioassays may be related to sorption processes and/or inefficient extraction. On the other hand, the newly developed whole-soil bioassay seems useful for assessing the dynamics of potential phytotoxic effects that are associated with various application doses, different soil types and under varying environmental conditions.

1 INTRODUCTION

The extremely high organic load and the toxic nature of olive mill wastewater (OMW) prevent their direct discharge into domestic wastewater treatment systems (Laor et al., 2011). Controlled land spreading of untreated olive mill wastewater (OMW) has been widely practiced as a mean of their disposal and is being permitted by law in several countries. E.g. the Italian law permits annual spreading of up to 50 or 80 m^3 ha^{-1} for OMW generated by press or continuous centrifugation method, respectively (Rinaldi et al., 2003). In Israel, the Ministry of Environmental Protection has been involved in spreading activities in the range of 50-100 m^3 ha^{-1} with some reservations regarding hydrologically sensitive areas. Besides being a cheap and viable solution, the OMW is considered as a natural fertilizer, which at proper application rates is beneficial to crops and can be disposed of without causing environmental damage Still, the inherent phytotoxicity of OMW is of major concern and requires careful management (Aviani et al., 2009, 2012, Piotrowska et al., 2011). Multiple studies based their phytotoxicity assessments on soil extracts, while it is questionable whether extract-based bioassays reflect reliably the phytotoxicity of the soil *in situ*.

1.1 Research objectives

The aim of the present work was to investigate the dynamics of OMW phytotoxicity after soil amendment by two bioassay methodologies (*soil-extract* and a newly modified *whole-soil* bioassay) and to follow the concomitant disappearance of soluble organic matter and total phenolic compounds.

2 METHODOLOGY

2.1 Soils and OMW samples

Two different soils representing regions of potential OMW application in Israel were used: A Vertisol-type soil (fine-clayey, montmorillonitic, thermic Chromic Haploxerert) from Newe Ya'ar Research Center in Jezre'el Valley, northern Israel (32°42'N 35°11'E; 580 mm rainfall) and a Loess-type soil (sandy loam) from Revivim at the Negev, southern Israel (31°02'N 34°43'E; 115 mm rainfall). OMW generated by the three-phase extraction process was kindly received from the olive mill of Iksal (near Nazareth, northern Israel).

2.2 Equivalent application doses of OMW

Soil samples were amended with OMW based on equivalent field application doses, considering the following: 1. the applied OMW is homogenously mixed with the top 15 cm soil layer. This assumption is valid for cases where surface spreading is followed by tillage. This practice (when possible) can reduce the risk of contaminants leaching (Laor et al., 2011). 2. After OMW application, the soil would reach a certain level of water field capacity. Based on field moisture conditions and the OMW dose, we calculated the amount of water that had to be added in order to reach this desired moisture level. Control soil samples were amended with water to achieve the same moisture conditions. After addition of OMW and water, the soils were thoroughly mixed to achieve homogeneous mixtures. Chemical analyses and phytotoxicity bioassays (Section 2.3) were conducted at time 0 (soil samples were extracted immediately after OMW application) and after 5 days of incubation at 25°C.

2.3 Soil chemical analyses and phytotoxicity bioassays

Soil paste extracts were prepared by saturating about 450 g of soil with deionized water, overnight incubation at 4°C (to minimize microbial degradation), and subsequent vacuum extraction of the liquid phase. Dissolved organic carbon (DOC) and total phenols (TP) were analyzed in these extracts. DOC was analyzed by a total organic carbon analyzer (Shimadzu TOC-5000) after samples acidification ($pH<2$; using 2N HCl). TP were analyzed with slight modifications after Box (1983), involving 0.5 ml Folin-Ciocalteau reagent (Merck) and 3 ml of 20% sodium bicarbonate in a total volume of 10 ml. Absorbance was measured at 725 nm (Cary 50 Bio, Varian) against a blank of deionized water and Folin reagent. TP concentrations were determined on the basis of a calibration curve obtained for caffeic acid (Merck).

Soil phytotoxicity was determined by two bioassay methodologies (Fig. 1): The *soil-extract* bioassay was conducted by placing four millilitres of the soil paste extract on glass microfibre filters (GF/A; Whatman, Brentford, UK) and inserted into 90 mm glass Petri dishes. Ten seeds of cress (*Lepidium sativum* L.) were placed in each dish, using three dishes per sample. Germination and root length were recorded after 3 days under darkness, at 25°C. Control dishes were prepared with the extract of control unamended soils. The *whole-soil* bioassay was modified from Weiss et al (2006) who developed this assay to assess the phytotoxicity of pesticides in a sand matrix, and from Prasad et al (2010) who demonstrated a similar test to assess the phytotoxicity of composts and other growing media. We optimized these protocols for soil media (instead of sand or growing media) in terms of sufficient moisture and oxygen for roots development. To provide efficient oxygen diffusion, the lids of the dishes were perforated. A 80% level of water field capacity was found appropriate. Control soils were prepared separately by applying water only to reach the same level of moisture. Three replicates were prepared for each treatment.

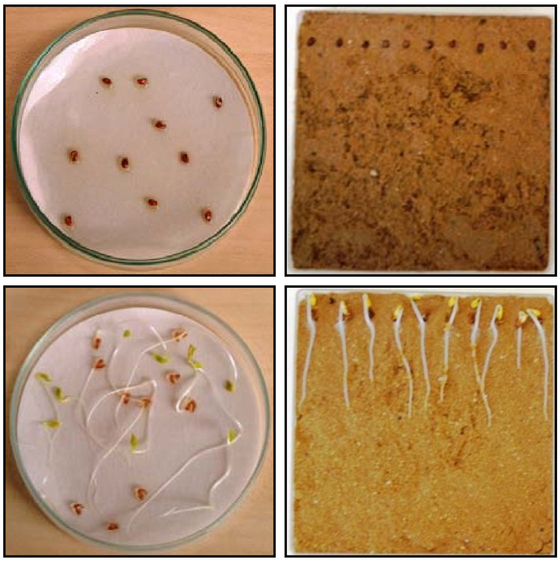

Figure 1. *Soil-extract* (left) and the *Whole-soil* (right) bioassays. Top photos show phytotoxic conditions, which completely suppress seed germination (taken after 3 days of incubation).

In both bioassay methodologies, the *Phytotoxicity Index* was calculated from Eq. 1:

Phytotoxicity Index = [1-(treated soil/control soil)] (1)

Where for both treated and control soil samples, the average root length was recorded. Non-germinated seeds were considered as length = 0. The form of Eq. 1 yields a range of values between (-1) and (+1), whereas positive sign means negative (toxic) effect and negative sign means positive (stimulatory) effect.

3 RESULTS AND DISCUSSION

The initial DOC and TP concentrations measured in the soil paste extract at time 0 and after 5 days of incubation were positively related to the application dose (Fig. 2). Although both soils were applied with the same equivalent OMW doses, the concentrations of DOC and TP at time 0 were higher in the Loess soil as compared to the Vertisol soil. This is the result of a higher dilution of the OMW with the soil water in Vertisol (which has higher water content in the soil paste and at a certain fraction of field capacity). Presumably, it also resulted by more intense adsorption processes in the clayey soil. After 5 days of incubation, both the DOC and TP reduced substantially in both soils. This disappearance is attributed to biodegradation processes.

A comparison between the two bioassay methodologies is presented in Fig. 3. For the Vertisol soil, the phytotoxicity at time 0 for doses of 80 and 160 m^3 ha^{-1} was substantially higher in the soil extract than in the whole soil bioassay. In fact, in the lowest dose, no phytotoxicity but stimulatory effect was observed in the whole soil. At the highest dose of 320 m^3 ha^{-1}, both bioassays showed very high toxicity, but some less severe effect was shown with the soil extract (at this dose seeds development was completely inhibited in the whole soil bioassay). After 5 days of incubation, no significant phytotoxic effect was shown in the Vertisol at all application doses. The differences between the two bioassays were also insignificant. For the Loess soil, the phytotoxicity at time 0 was significantly different between the two bioassays only at 160 m^3 ha^{-1}. At this dose, although the difference was less substantial than that observed in the Vertisol, the same trend was observed, with higher phytotoxicity in the extract as compared to the whole soil bioassay. After 5 days of incubation, in contrast to the Vertisol soil, significant differences were observed between the two bioassays, with significantly higher phytotoxicity shown in the whole-soil bioassay. Moreover, the soil extracts in this case showed opposite effect (seed growth stimulation) at 80 and 160 m^3 ha^{-1}.

This reduction of phytotoxicity during such a short incubation period can be related at least partly to the decrease of toxic phenolic compounds (as expressed by TP disappearance) or other toxic organic compounds (grossly expressed by the disappearance of DOC).

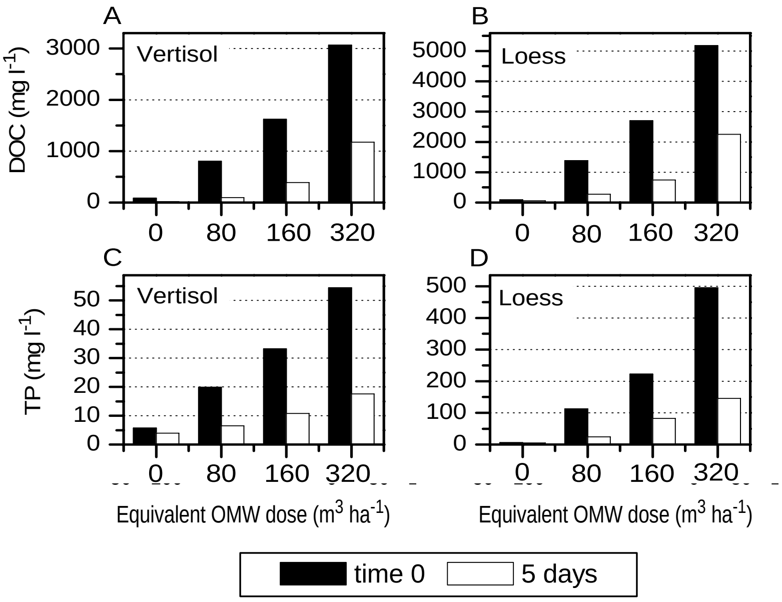

Figure 2. Dissolved organic carbon (DOC) and total phenols (TP) measured at time 0 and after 5 days of incubation in Vertisol and Loess soils amended with equivalent OMW doses of 80, 160 and 320 m^3 ha^{-1}.

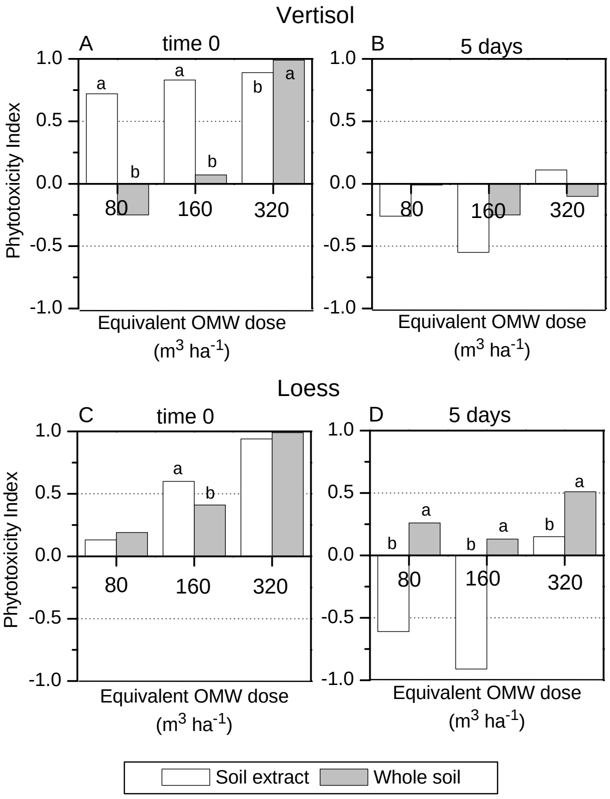

Figure 3. Soil phytotoxicity obtained by two bioassay methodologies. The *soil-extract* and *whole-soil* bioassays were conducted at time 0 and after 5 days of incubation following OMW application of equivalent doses of 80, 160 and 320 m^3 ha^{-1}.

4 CONCLUSIONS

Out of the two tested bioassay methodologies, the extract-based bioassay may over-estimate both the negative and positive potential effects of OMW application. Possible artefacts associated with extract-based bioassays presumably be related to sorption processes and/or inefficient extraction. On the other hand, the newly developed whole-soil bioassay seems useful for assessing the dynamics of potential phytotoxic effects that are associated with various application doses, different soil types and under varying environmental conditions.

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(211) ECOTOXICOLOGICAL IMPACTS OF BIODEGRADABLE POLYMERS IN AGRICULTURAL SOILS

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EXECUTIVE SUMMARY

The market relevance for biodegradable polymers has been increasing for a few years. A benefit for the reduction of landfill-waste, a high public acceptance and a positive company image are the driving forces behind their soar. Biodegradable polymers can reach agricultural soils either by composting flow (as bags or packaging) or as mulching film.

The aim of this study was to investigate the innocuousness of biodegradable polymers in agricultural soil. In a first stage, we did a review of bioassays used for the last ten years to evaluate the toxicological effects of these products. In a second stage, tests were performed with agricultural soils to assess the impacts of plastics as a mulching film on the biological soil properties for a sustainable agro-ecosystem. Biodegradable and non-biodegradable materials were tested in agricultural soils.

Most of the scientific studies focusing on biodegradable polymers conclude to their innocuousness only because of the well biodegradation of the tested products. Few of them use ecotoxicological tests to study their environmental impact and some show toxic impacts during or after the degradation on life, growth or reproduction of bacteria, plants and animals used as bioindicator. *Vibrio fischeri* is the main bioindicator used (in more than 90% of the publications issued on the subject). This organism is a marine bacterium which exists naturally either in a free-living planktonic state or as a symbiont of certain luminescent fish or squid. It is a heterotrophic and free-living form lives in decaying organic matter. Considering these characteristics, this test can be used as a first step to evaluate the toxic impact of water-soluble compounds. But *Vibrio fischeri* cannot be relevant to assess the risk of biodegradable polymers used in agriculture, all the more when it is the only ecotoxicological test used.

The difficulty to sum up the former studies comes from the variety of methodologies: sample preparation (polymer in small pieces or particles), dilution substrate (ratio and type of substrate: fresh biowaste, mature compost, soil), sampling period (during the biodegradation process or at the end). Most of the time, the composting process are not linked to the reality (type of waste, temperature, duration …) and so for the compost tested.

Therefore bioassay was carried out to assess the impact of polymer materials on a specific biological activity of agricultural soils. A new test, not yet standardised, has been set up using the bacterial nitrification as a bioindicator. Soils sampled from field were sieved at 4mm and the fraction of plastics fragments above 4 mm were cut up and incorporated again to the soil. This fraction was the reference dose used to calculate the quantity of non-biodegradable material to be added to the soil. Soils were incubated in a microcosm conditions (20°C, 50% of soil moisture). Three treatments were tested: soil/soil + biodegradable mulch material/soil + conventional mulch material.

The results showed that microbial nitrification is an efficient bioindicator that could be used to evaluate the impact of polymers. The effect of polymers was more discriminating when the initial level of soil nitrification was important. Reduction of nitrification activity was observed for the two biodegradable polymers. With the non-biodegradable material, no effect was observed after exposure time of 7 days.

The first results of this bioassay have to be confirmed by further tests with various types of biodegradable polymers and various exposure conditions: mulching film incorporated to soil and amended soil with compost made with biodegradable polymers.

The relevance of this bioindicator will be evaluated in comparison with others in the BIOPLASTOX project. The objective of this project is to draw a parallel between the results of the degradation of two plastics (biodegradable packaging) during the composting and the evaluation of the ecotoxicity of the final compost containing these plastics.

This study was granted by the French Ministry for Agriculture, Food, Fisheries, Rural affairs and Spatial planning.

1. INTRODUCTION

This work was carried out as part of an agreement with the Ministry of Food, Agriculture, Fisheries, Rural Affairs and Regional Planning. The terms of this agreement are briefly recalled below.

An action plan for sustainable production and industrial policy is being developed at European level. The proposed new framework "Waste" has been adopted by the Council in October 2008. The project includes the evaluation of the impact of waste by "Life Cycle Assessment". In parallel the work of the "Grenelle de l'Environement" are being translated into French legislation. Individual and professional consumers are the target of these two policies: the act of purchase should be motivated towards the best performing products on the environment, which implies to have available a "sincere,, objective and complete environmental information on products ".

In this context, the communication on the end of life products and / or their composition becomes a selling point. Today, products like packaging, bag, disposable equipment ... are mixed on the market with information on their end of life by biodegradation or composting or anaerobic digestion. It is the same trend for professional products for agricultural use as mulch film, the mounting hardware for horticulture and market gardening, and resins for coating granular fertilizers ... Many of these products are made from synthetic polymers. They come from diverse geographical places and fit into a global market leaded by Asian and Anglo-Saxon. Trials on the accumulation in the soil of "biopolymers" used in agriculture have been made in a study realized by CEMAGREF-ADEME-AGRICE-2006. For each tested material, this study shows a phenomenon of accumulation of their residues. It is therefore essential to know the chemical sort of the residues and their ecotoxicity.

The ecotoxicity of synthetic organic polymer materials can directly result from the impact of these materials on organisms living in ecosystems where these materials will be introduced or the indirect impact of these materials through their incomplete degradation. Indeed, upon degradation of synthetic organic polymers can release into the environment of monomeric or oligomeric molecules having a specific toxicity. In addition, they can release elements such as antioxidants or dyes or other components, introduced into the polymer to give it a particular property. These elements may also have certain toxicities when they are released.

Therefore, as part of a study on the impact of synthetic organic polymeric materials on the environment and particularly agricultural soils, it should not only consider the polymers themselves, but also products that can be released through their degradation.

The study proposed here aims to:

- Explore the bibliographic ecotoxicity plastic polymers and their degradation products.
- Initiate an experimental study to develop an appropriate analytical method for measuring degradability and ecotoxicity can be used as a reference for scientists and for possibly a prescriptive approach.

2. METHODOLOGY

2.1 Critical analysis of the methods and ecotoxicological results described in the literature

To identify publications relevant to the scope of our study, two databases were consulted for an initial assessment: "Current Contents" and "ScienceDirect". The second base, more accessible and widely used in universities today was then widely consulted. Thus, 60 references were identified (Table 1). Reading some publications has allowed us to find new relevant documents. Thus, 11 references are directly within the scope of the study.

TABLE 1 **Results of research published in two databases**

Database	**Keywords used**	**Number of references found**
Current Contents	plastic* AND ecotox*	41
	tox* AND bioplastic*	4
ScienceDirect	TITLE -ABSTR-KEY (plastic*) AND TITLE-ABSTR-KEY (ecotox*)	12
	TITLE-ABSTR-KEY (tox*) and TITLE-ABSTR-KEY(bioplastic*)	3

Publications, reports and thesis considered in our study were analyzed according to three criteria:

- The laboratory of belonging is that indicated in the publication by the author.
- Sources of funding scientific articles were identified through the thanks given by the authors in their publication (details of the acknowledgments and references are given at the end of this report, in the "references").
- The journal impact factor or JIF.

TABLE 2 **References, sources of funding and impact factors of publications, reports and thesis analyzed in this study (np: no provided)**

Author (year) *Journal*	**Laboratory - Compagny**	**Source of founding**	**Impact factor**
Bellia *et al.* (1999) *Polymer Degradation and Stability*	Novamont S.p.A., Italy	European Commission	2,154
Degli-Innocenti *et al.* (2001) *Polymer degradation and stability*	Novamont S.p.A., Italy	European Commission	2,154
Feuilloley *et al.* (2002) Commission européenne	CEMAGREF, France	European Commission	
Fritz *et al.* (2003) *Macromolecular Symposia*	IFA-Tulln, Austria	np	0,913
Grima, S (2002) Thesis	Laboratoire de Chimie Agro-Industrielle, INRA INPT ENSIACET & Information et Technologie pour les Agro-Procédés - UMR CEMAGREF-ENSAM-CIRAD, France	ADEME - CEMAGREF	
Jayasekara *et al.* (2003) *International Biodeterioration & Biodegradation*	Centre for Applied Colloid and Biocolloid Science, School of Engineering and Science, Swinburne University of Technology, Australia	np	2,252
Kapanen *et al.* (2008) *Journal of Polymers Environment*	VTT Technical Research Centre of Finland, Finland	European Commission	1,571
Rasal *et al.* (2010) *Progress in Polymer Science*	Department of Chemical and Biomolecular Engineering & Center for Advanced Engineering Fibers and Films, Clemson University, United States	National Science Foundation	23,753
Rudnik *et al.* (2007) *Chemosphere p107*	Industrial Chemistry Research Institute, Poland	np	3,253
Silvestre *et al.* (2006) Final report ADEME	Laboratoire de Chimie Agro-Industrielle, INRA INPT ENSIACET, France	ADEME (French Agency for Environment)	
Tosin *et al.* (1998) *Journal of Environmental Polymer Degradation*	Novamont S.p.A., Italy	np	1,129
Tuominen *et al.* (2002) *Biomacromolecules*	Department of Chemical Technology, Polymer Technology, Helsinki University of Technology, Finland	National Technology Agency of Finland	4,502

2.2 Experimental study

Based on a survey of Alsatian farmers, three gardeners were contacted again to take samples of soil and agricultural plastics in their fields. Both used biodegradable plastic mulch and conventional plastic mulch. The samples were taken.

Samples have been made in land parcels mulched this year or in previous years. In order to have a ground reference for each focused plot, samples were also carried out along it or close. The upper horizon of soils was sampled 15 cm deep; the second horizon, named lower horizon sample was between 15 and 30 cm deep.

The usual precautions were taken to avoid contamination between samples (gloves, washing tools samples). In the laboratory, samples containing plastics were cleaned of foreign bodies (stones, herbs, roots ...), and sieved to 4 mm. Pieces of plastic found were recovered, weighed and ground (<2 mm). The reference soil samples were also sieved to 4 mm. Parameters holding capacity and pH were determined.

The mulching films tested come from soil samples. Three different mulching films were tested during this study: two biodegradable plastics: Bionov A and B of Barbier and Aglex of Agripolyane.

Site 1: soils with the biodegradable mulch material incorporated into the soil each year between 2007 and 2009 (S1.1) or once in 2010 (S1.2)

Site 2: soil with another biodegradable material incorporated to the soil at the end of crop season (on 2010)

Site 3: soil with a conventional non-biodegradable mulching film.

Test in microcosm: followed by a specific biological activity: measurement of nitrification activity

The test protocol is based on the recommendations of the AFNOR 14238. Test principle: Nitrification is the provision of forms of nitrogen assimilated by plants, often limiting factors for crops. The study is based on the response of the activity of bacterial populations implicated in soil nitrification activity between a soil without plastic mulch and the same soil mixed with plastic mulch.

Nitrification is an oxidation of ammonium to nitrite followed by oxidation of these nitrites into nitrates. In soils, this process is mainly carried out by two groups of microorganisms: the ammonia-oxidzing bacteria (AOB) perform the first step (NH4 + ◊ NO2-) and nitrate-OXIDIZING bacteria (NOB) perform the second step (NO2 - ◊ NO3-).

The addition of ammonium stimulates the microflora involved in the nitrification process and test the effectiveness of this microflora in performing this function.

The kinetics of nitrification activity was monitored for 21 days in mesocosms set up with soil sites 1, 2 and 3 with or without plastic. The nitrification activity is evaluated by extracting nitrogen ions (NH4 +, NO2-, NO3-) by a solution of potassium chloride (KCl) in 1M during incubation with a substrate of soil nitrogen (NH4 +).

To evaluate the effectiveness of the microflora in the nitrification process, the addition of ammonium at 100 mg N / kg dry soil is made; it helps stimulate the microflora involved. Terms with and without added ammonium are implemented.

3. RESULTS AND DISCUSSION

3.1 Critical analysis of the methods and ecotoxicological results described in the literature

Much of the work in studying agricultural context (Grima, 2002, 2005 and Silvestre 2006 INERIS, Feuilloley 2002) do not relate to the assessment of ecotoxicity of the materials tested but their biodegradation. The safety tests are often lacking in detail, and despite numerous requests, we have not been able to obtain descriptive or more complete data. In the thesis of the study and Sophie Grima AGRICE, the tests were carried out by INERIS (National Institute of Industrial Environment and Risks) and VTT (Finnish research center) in the study published by Feuilloley. It appears that some of the results are not usable as a result of problems encountered in the manner as a control (negative control) or with reference substances (positive control). Statistical analysis is rarely made and the lack of hard data does not allow us to realize when it is missing.

In these three studies, tests on plants or earthworms and tests on aquatic organisms have been made. When tests are made with terrestrial bioindicators, the studied material is mixed with soil or compost. When tests are carried out with aquatic bioindicators, the tests are conducted on an aqueous extract of the studied material mixed with soil or compost. It is therefore important to take into account for evaluating the results of these studies, preparation of the material, the substrate to which it is made and used bioindicators.

The methods used on plants are relatively similar: ISO 11269-2 or OCDE208 with barley, lettuce and radish. The tests on the leachate used different bioindicators: an alga, a bacterium and a bioluminescent microcrustacean.

Particular, we noted the following biases on the substrates and bioindicators:

- The results highlight the importance of environment in which tests are performed (agricultural soil, artificial substrate, vermiculite, pozzolan, compost) that do not lead to similar conclusions about the safety of the materials tested. While, overall, the tests show no significant ecotoxicological effect of materials studied, it should be noted that these results are not always usable because of environmental contamination or references giving themselves bad results.

- In addition, the relevance of certain bioindicators can be discussed. Thus, methods ISO 11348, ISO 21338 (commonly known as "Microtox") and the "flash test", using a bacteria "Vibrio fischeri" to assess the ecotoxicity of aqueous extracts of soil or compost. This bacterium lives in the marine environment which gives a low ecological representativeness of freshwater aquatic ecosystems. Yet it was these freshwater systems (groundwater, river, lake, and pond) that are the target of aquatic ecotoxicity studies conducted on biodegradable polymers. On the other hand, the presence of polymer particles or material may mask some of the light produced by bacteria and thus lead to an erroneous conclusion on the toxicity of the product ("false positives").

Studies specifically on ecotoxicity have been found in scientific papers from international journals ones we have analyzed relating ecotoxicity testing of plastic materials called "biodegradable" are all made with biobased materials already marketed or specifically synthesized for the study.

In the latter case, we can underlines a study of the safety of additives (Tuominen, 2002), which is very interesting because, in all other studies we analyzed, the raw material is often specified in the literature (based on starch, wheat gluten, etc. ..) but no additives. But as we discussed earlier, they may be involved in ecotoxicological reactions.

In two publications, the authors indicate the conditions of use of the materials tested by specifying that they are used as mulch film (Kapanen, 2008 - Jayasekara, 2003). The publication of Kapanen (2008) is to our knowledge, the only study that tests a material under conditions of use, in situ, that is to say a mulch film used for a culture of Strawberry and plowed into the soil with crop residues. And the quantities of material tested on the ground are made consistent with the uses. For all other studies, the products were reduced in size before being added to a soil, a bed called "mineral" (pozzolan), in waste costs, a growing medium or in compost. In these studies, the protocols in place lead to a bias in the interpretation of results, the tests were not conducted under conditions similar to the use of materials it n ' is not possible to conclude the risk posed by the use of these materials. One example is the use of mature compost to introduce the material tested before placing the mixture in contact with the bioindicators studied. In this case, the material will not be degraded as it would be mixed with organic waste and then be subjected to an industrial composting before being added to the soil.

Preparing materials (prior to incorporation in a soil or compost) is variable and some are "aged" before their ecotoxicity is assessed. Thus, depending on the state of the material before an aging phase, it will sooner or later (bio) degrade, and depending on when the ecotoxicity test will be performed during the aging of the material, degradation products may be present and be bioavailable to the bioindicator used. For example, studies show that different responses are obtained depending on the progress of material degradation (Tuominen 2002, Jayasekara 2003, Kapanen 2006, and Silvestre 2006) and they all highlight that the answers vary over time. Thus, depending on the materials, the peaks of toxicity are identified during the degradation or end thereof. Now, according to the standard methods for this type of material, safety is assessed while the material is biodegraded (EN 13432:2000 - Packaging) or after a fixed period of time, whatever the expected life of the test material (NFU 52-001:2005 - Movies mulching). Thus, for EN 13432:2000, ecotoxicity testing will not reveal the potential toxicity associated with intermediate steps in the degradation of materials, steps in which sub-degradation products could be released. As part of NF U52-001: 20005, states of degradation of the films (and therefore the consideration of the degradation products) will be different depending on the lifetimes of the films studied.

The analysis of these studies shows the importance of work remaining to be done in the field of ecotoxic impact of plastics called "biodegradable" in agricultural soil. Products on the market are many and varied, and the methods used today to evaluate their ecotoxic potential, whether derived from standards or scientific literature, not to verify their safety. It therefore appears necessary to conduct tests during the degradation of the material studied in the conditions of use and developing a battery of ecotoxicology tests (and thus bioindicators) to take into account the risks towards the aquatic and terrestrial ecosystem of organisms and trophic levels of different sensitivity.

The terms "biodegradable", "bioplastic", "biopolymer", "biobased" should also be specified so that there is confusion or not.

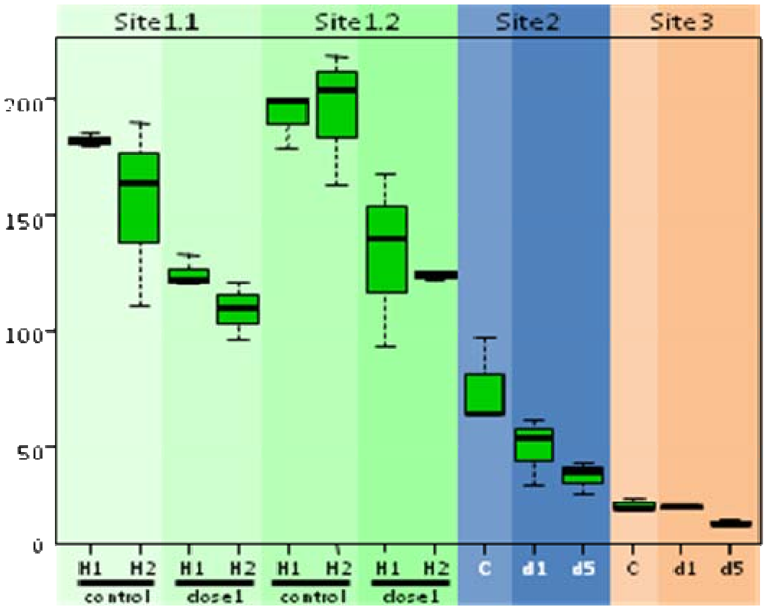
3.2 Experimental study

The results of these tests demonstrate an inhibitory effect of the addition of biodegradable plastics on the nitrification activity of soil. It also appears that the effects of biodegradable plastics on the nitrification activity of soil depend on the dose given.

No clear difference was observed between the two horizons tested; also preincubation of day-10 does not seem to have any impact on the observed effects. The work conducted at RITTMO has thus demonstrated the feasibility of a bioassay "nitrification activity" to assess the impact of plastic mulch on soil microflora. The first results obtained with this bioindicator is satisfactory, however, the relevance of this bioindicator must be validated with other polymers by considering the repeatability of results within the same trial but also a test to another.

Moreover, the results of initial physical and chemical analysis initiated in this study were not very conclusive. This is partly due to the low intake polymer sample to be analyzed, since this part was not anticipated in the project. Furthermore, methods of physicochemical analysis of polymers require a preliminary step to adapt these techniques to the polymer will be studied.

FIGURE 1 **Nitrate concentration after 7days (mg/kg dry soil) (Control (C) Soils sampled from field, sieved at 4mm ; Dose 1 (D1) : Sieved soil + fraction of plastics fragments above 4 mm were cut up and incorporated again to the soil. D5 = Dose 1 x 5 ; H = sample depth H1 = 0-15 cm H2 = 15-30cm)**



4. CONCLUSIONS

The literature has shown the existence of a wide variety of methods, products, and approaches that make it particularly difficult interpretation and synthesis of all experiments performed on synthetic organic polymer materials placed in end of life in agricultural soils. To this difficulty is compounded by the use of terms (biodegradable bioplastics...) that do not all have the same definition according to the authors.

The results of experimental studies have shown an inhibitory effect of the addition of biodegradable plastics in soil nitrification activity, an effect that would depend on the dose given. The work conducted at RITTMO AgroEnvironnement has thus demonstrated the feasibility of a bioassay "nitrification activity" to assess the impact of plastic mulch on soil microflora under conditions of use of materials.

At the end of this work, it appears important to conduct trials to test a set of terrestrial and aquatic indicators. Indeed, the literature review points out that" the bioindicators used today are not diversified and often the same. In addition, the first experimental tests we conducted showed interesting results that demonstrate that the bioindicator used is sensitive to the presence of biodegradable mulching films.

In a future study, it will be important to validate the relevance of this bioindicator by improving tests on other soils in which biodegradable mulching films were used. On the other hand, it will also compare its sensitivity to other ecotoxicity tests in connection with the establishment of a more comprehensive battery (tests with terrestrial and aquatic bioindicators from different trophic levels). This battery will be applied to soil in the presence of mulching films at different times of incubation; this will take into account the evolution and the kinetics of (bio) degradation of these films.

Now, a battery of bioindicators will be implemented in a new project, "BioPlasTox" on packaging. This project is jointly conducted by RITTMO AgroEnvironnement and CRITT Matériaux Alsace for a period of three years (2011-2014). This is the assessment of ecotoxicity and degradation of plastic packaging materials called "biodegradable" composted. The objective of this study is to parallel the results of monitoring the degradation of selected materials during composting WITH those of ecotoxicological assessment of compost containing materials.

5. ACKNOWLEDGEMENTS

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Session 15

(47) GREENHOUSE GAS MONITORING FOR OPTIMIZATION OF PROCESS EFFICIENCY OF BIOGAS PLANTS

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EXECUTIVE SUMMARY

Within the research project "KLIMONEFF" modern optical remote sensing (ORS) technology is applied as an innovative approach to detect methane losses of diverse parts of biogas facilities in order to reduce their greenhouse gas (GHG) losses and optimize their energy efficiency. An Austrian biogas plant has, therefore, been selected to quantify the GHG emissions of the entire plant but also methane losses (methane loads) from various plant components for the representative period of one year. Within this period, meteorological variations as well as plant-specific and operation-specific changes (from daily to seasonal) will be considered. In parallel a monitoring-tool to quantify particularly the methane losses will be developed. Based on the actually measured data recommendations for future technological development and improvements of biogas plants will be given.

The emission rates are quantified using the Lagrangian particle dispersion model LASAT (Lagrangian Simulation of Aerosol-Transport). Continuous meteorological measurements with a 3D ultrasonic anemometer provide the necessary wind and turbulence parameters for the dispersion model. Controlled tracer gas tests at five representative biogas plants will be used to constrain the dispersion model.

The outcome of the research project "KLIMONEFF" will provide a monitoring and (quality) management tool for biogas plants as well as a better decision basis for the optimization of facilities and operation as well as political-strategical developments within the framework of the Austrian energy and climate policy.

First results have indicated varying CH4 emissions (based on path-integrated concentration data) from open digestate storage tanks depending on the filling level, plant process efficiency as well as weather conditions. Also gas releases from the overpressure safety device installed at the dry gas storage tank have been detected. Since most of the emissions will vary strongly over time it is important to conduct long-term measurements in order to get representative estimates of the average emissions.

1 INTRODUCTION

Biogas plants can contribute to the reduction of greenhouse gasses (GHG), and, therefore, play an important role in meeting national as well as international environmental targets. However, the net GHG reduction will heavily depend on the process efficiency. During the last years, the numbers of biogas plants has considerably increased. In Austria, 362 biogas plants are installed corresponding to an electric capacity of 104.1 MW (E-Control, 2011), while in Germany close to 6,000 plants are operating with an installed capacity greater than 2,000 MW (FNR, 2011). Greenhouse gas emissions and methane losses (= loss of resources) can arise from diverse parts of biogas facilities along the entire process chain of biogas generation and utilization. Certain plant components (e.g. open digestate storage tank, membrane, safety valves) are suspected as potential emitters. An improvement and optimization of various plant components and, therefore, an increase in efficiency of the entire biogas plant cannot be achieved until the losses are detected and quantified. However, up to now, there are no reliable data records available in order to evaluate the relevance of these losses and emissions demonstrating the actual (energy) plant efficiency.

In recent years, numerous international studies have demonstrated the huge potential of Optical Remote Sensing (ORS) technologies supported by meteorological data and dispersion models. Beside the use of Open-Path Fourier Transform Infrared (OP-FTIR) and Ultraviolet Differential Absorption Spectroscopy (UV-DOAS), also Open-Path Tunable-Diode-Laser-Spectroscopy (OP-TDLS) has been applied to overcome the limitations of conventional methods.

These technologies have been used to characterize fugitive emissions from large area sources including landfills (Galle et al., 2001; Scheutz et al., 2011), agricultural operations (McGinn et al., 2006) and biodigesters (Flesch et al., 2011). In comparison to traditional point monitoring approaches, ORS-based measurements provide much better spatial and temporal resolution and concentration data, allowing for characterization of emission plumes and the calculation of emission fluxes.

The research project “KLIMONEFF” aims to create a representative data pool and to simultaneously develop a simply applicable monitoring and quantification tool for emission control and quality management of biogas plants. Methane and carbon dioxide emissions from an Austrian biogas plant will be quantified by the use of an OP-TDLS system in combination with meteorological measurements and a dispersion model. Thereby not only the GHG emissions of the entire plant will be quantified but also methane losses (methane loads) from various plant components. Emissions will be measured for a representative period of more than one year, taking into account meteorological variations as well as plant-specific and operation-specific changes (daily to seasonal variations, start October 2011). The choice of an appropriate dispersion model (Lagrangian, Eulerian or Gaussian model) depends, among others, on the site conditions (e.g. complexity of buildings and topography) and, therefore, is an important prerequisite for the determination of emission fluxes from the area of interest. In this case, the back-calculation will be done with the Lagrangian particle dispersion model LASAT (Lagrangian Simulation of Aerosol-Transport; Janicke, 1985, 2007). Continuous meteorological measurements with a 3D ultrasonic anemometer over a period of one year as well as controlled tracer gas tests (using C2H2 as a tracer) at five representative biogas plants will be used to constrain the dispersion model.

Subsequently, a practice-oriented catalogue of measures based on the gained data pool will be developed indicating optimization potentials and solution approaches (technical, operational, biotechnological) concerning an increase in efficiency of gas yield and minimization potentials of gas losses.

2 MATERIALS AND METHODS

2.1 Study site

The biogas plant, selected for the measurement campaign of one year, processes energy crops and pig manure for co-digestion (see Figure 2). The plant was built in 2003 and is located on a pig breeding farm, where liquid manure (20 m^3/day) is used as a co-substrate (30 % share) in order to homogenize the solid energy crop feedstock (dilution of the substrates to less than 10 % dry matter). About 11,000 t of energy crops, which mainly consist of maize silage and small amounts of by-products from vegetable processing, together with 7,300 t of liquid pig manure are processed annually in two parallel digesters and a covered final digestate storage tank. The biogas storage is integrated in one of the digesters and in the final digestate storage tank. Additional storage capacity is provided by an external dry gas storage tank. Since the application of digestate on farmland is not allowed during winter, the fermentation residues are stored in open storage tanks once the capacity of the covered digestate storage tank is exceeded.

The generated biogas (about 4,020,000 m^3/year) is utilized in two combined heat and power units with an installed capacity of 1 MW electric energy and 1,034 MW thermal energy. The electricity is supplied into the grid and the thermal energy is integrated into a local district heating network.

2.2 Emission measurement technique

In general, the OP-TDLS instrument is a fast, interference-free and sensitive technique for making continuous gas concentration measurements over an open path (up to 1,000 m) between a laser emitting transceiver and a retro reflector. The retro-reflector will initially be targeted by using an aiming scope and an on-board visible aiming laser. The laser light emitted from the transceiver unit propagates in the atmosphere to the remote retro reflector and is reflected back to the transceiver unit. By analyzing the laser absorption on the wavelength of a particular gas of interest, average gas concentration data along the optical path can be determined. A schematic representation of the used tunable diode laser (GasFinder® 2.0, Boreal Laser Inc.) is shown in Figure 1.

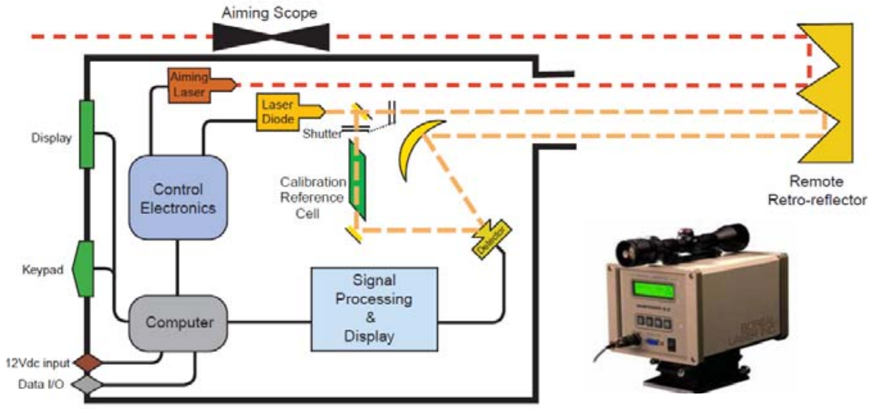

FIGURE 1 **Schematic representation of GasFinder® 2.0 System**

Since only path-integrated concentrations of single pollutant species can be determined by this method, three diode lasers detecting CO2, CH4 and C2H2 as a tracer gas are applied in this research project. During the measurement campaign of one year, CH4 and CO2 emissions are measured simultaneously. The CH4 laser is automatically aligned to multiple reflectors in order to collect path-integrated gas concentration data along multiple beam paths. The open-path laser is, therefore, mounted on a digital scanning motor (PTU D300, Directed Perception Inc.) which is remotely controlled using available hardware and software (Boreal Laser Inc.). The path-integrated CH4 concentrations are recorded almost every second for 60 s along each path using the digital scanning motor. CO2 concentrations are measured in parallel using a manual scanning mount. Every 5 minutes the CO2 laser is manually aligned with the next retro reflectors. The concentration data will be averaged over 10-min, 15-min and 30-min periods, respectively.

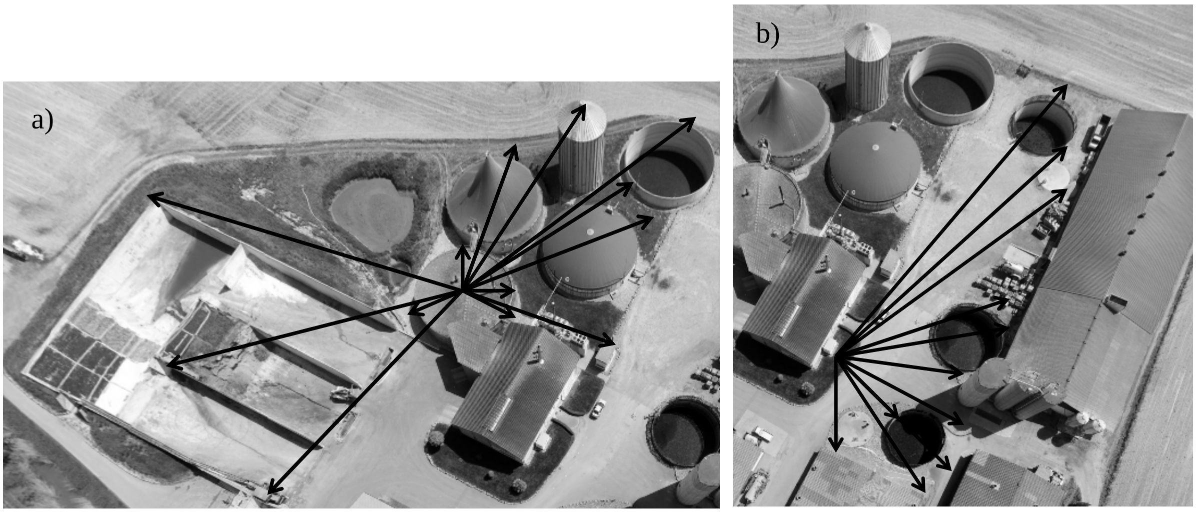

FIGURE 2 **Schematic view of the laser paths (arrows) over the study site (a: 13 laser lines; b: 11 laser lines)**

In general, a biogas plant represents a multi-source problem, and therefore a careful reflector configuration has to be chosen with respect to the suggested emission sources and the prevailing wind directions. The retro reflectors are mounted on tripods and high-tripod-systems (up to 10 m height), respectively, and positioned according to Figure 2 on the study site. Path lengths range between 7 to 90 m. Figure 3 depicts the laser paths (approx. 350 m) used for measuring the entire emissions downwind from the biogas plant. Depending on the prevailing wind direction, the lines are switched manually. The measurements are conducted at two different heights (1.8 m and 10.6 m). In order to determine the background concentrations of CH4 and CO2, the lasers are also positioned upwind of the biogas plant at regular intervals.

Each set-up arrangement (Figures 2a and 2b as well as Figure 3) is measured twice a month for several hours in order to evaluate the variability in the short-term emissions. In addition, measurement campaigns over a period of 24 h are planned.

FIGURE 3 **Location of the laser lines used for measuring the totality of emissions from the biogas plant depending on the prevailing wind directions (a: NE and E-winds, b: SW and W-winds)**

2.3 Dispersion model

The choice of an appropriate dispersion model (Lagrangian, Eulerian or Gaussian model) depends, among others, on the site conditions (e.g. complexity of buildings and topography) and, therefore, is an important prerequisite for the determination of emission fluxes from the area of interest. In this case, the back-calculation will be done with the Lagrangian particle dispersion model LASAT (Lagrangian Simulation of Aerosol-Transport; Janicke, 1985, 2007) which computes the transport of trace substances in the atmosphere for all wind speeds down to 0.5 m/s. It simulates the dispersion and the transport of a representative sample of tracer particles utilizing a random walk process (Lagrangian simulation). LASAT can use the complete meteorological and turbulence information of a 3D ultrasonic anemometer, including a vertical profile. Especially atmospheric stability is directly deduced from the measured turbulence (Monin-Obukhov Length) instead of discrete stability classes. Methane/carbon dioxide emissions are re-constructed from the measured concentrations by an inverse dispersion technique (Schauberger et al., 2011; Flesch et al., 2005). The emission rate Q (mg s^{-1}) is calculated from the measured gas concentration C (mg m^{-3}), the unity emission Q_0 and the corresponding concentration C_0 according to the following equation:

$$Q = C * \frac{Q_0}{C_0}$$

Continuous meteorological measurements with a 3D ultrasonic anemometer provide the wind field and turbulence data base for the dispersion model. At the study site, the 3-D sonic anemometer “uSonic-3 Scientific” manufactured by Metek company, Germany,is located at the north-east edge of the substrate (energy crops) storage during the project. The sonic is mounted on top of a 10 m high mast above the soil surface to measure key wind and turbulence parameters needed for the dispersion model. The components of the wind vector and the sonic temperature are measured at 10 Hz. Controlled tracer gas tests at five representative biogas plants will be used to constrain the dispersion model.

3 FIRST RESULTS

3.1 Time series of fugitive emissions (concentrations)

As an example, Figure 4 shows the diurnal cycle of two laser lines at the project site on three and four different days, respectively. Figure 4a represents the line-averaged CH4 concentration from the dry gas storage tank, whereas Figure 4b depicts the CH4 concentration development from an open digestate tank. Field observations have been prepared in time series of 10-min averages. It has to be noted that the concentration scale of Figures 4a and 4b differs by a factor of 10. The results show that the overpressure safety device installed at the gas tank releases very high concentrations of methane (up to 110 ppm) once the pressure in the headspace reaches a critical level. However, the gas releases represent only a very short period of time. Concentration measurements at an open digestate tank also show varying CH4 concentrations over time but with a lower level compared to the safety device. Since the application of digestate on farmland is not allowed during winter, the storage of fermentation residues in open tanks started at the beginning of January 2012. Consequently, CH4 concentrations on 13-12-2011 represent - more or less - the background concentration within the study site (approx. 2 ppm). Higher CH4 concentrations were observed, in particular, on 08-02-2012 between 1 and 3 pm which indicates higher methane emissions due to changing weather conditions. It can be concluded that CH4 emissions from the open digestate tank will fluctuate depending on the filling level, plant process efficiency, temperature, wind, atmospheric pressure, etc. In order to gain a better insight into the plant process efficiency also the remaining gas potential in the digestate is determined on a monthly basis (data in progress).

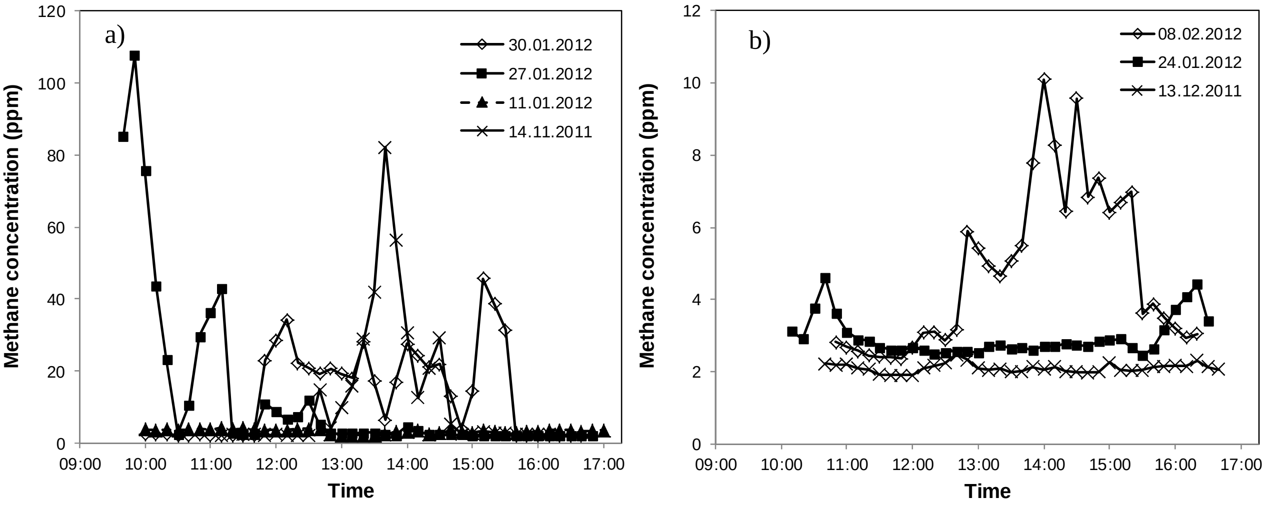

FIGURE 4 **Time series of fugitive methane emissions from the dry gas storage tank (a) and an open storage tank for fermentation residues**

3.2 Model application details

Biogas plants usually consist of a complex building structure which might be challenging for the determination of emission fluxes from plant components, such as open digestate storage tanks. The simulation of a three-dimensional wind field modified by complex buildings is, therefore, a prerequisite for modelling the dispersion of contaminants. The dispersion model LASAT computes the flow around buildings, including the recirculation and increased turbulence in the lee. The wind field will be simulated from diverse meteorological conditions using different wind directions and atmospheric stability conditions. In general, the atmospheric stability plays the most important role in transporting and dispersing air pollutants (Zoras et al., 2006). It is defined as the atmospheric tendency to resist or intensify vertical motion or alternatively, to suppress or augment existing turbulence (Zoras et al., 2006). In addition, it is also related to the temperature change with height as well as wind speed. Figure 5 shows an example of the wind field model of the project site.

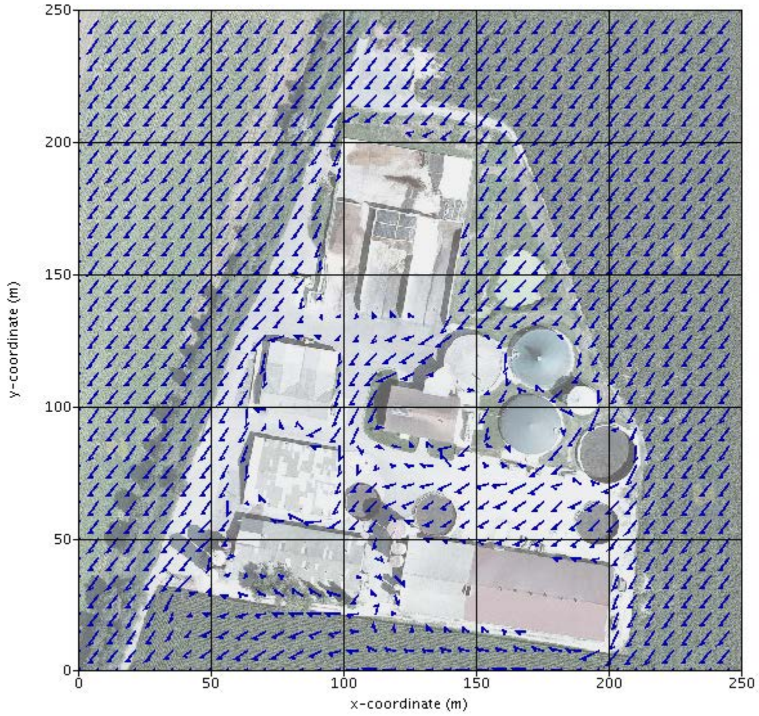

FIGURE 5 **Example of the wind field simulation with LASAT influenced by the building array (neutral atmospheric stability, wind direction 40°)**

4 CONCLUSIONS

First results indicate that the emissions will vary strongly over time and it is important to conduct long-term measurements in order to get representative estimates of the average methane losses. Traditional point monitoring techniques (e.g. floating chamber, portable methane detector), therefore, represent only a random sample and are generally associated with spatial and temporal limitations. Especially, open-path measurements at an open digestate have shown that CH4 emissions fluctuate depending on the filling level, plant process efficiency and weather conditions (temperature, wind, atmospheric pressure, etc). However, the influencing parameters still need to be investigated in more detail. In order to gain a better insight into the plant process efficiency also the remaining gas potential in the digestate is determined on a monthly basis.

The outcome of the research project "KLIMONEFF" will provide a monitoring and (quality) management tool for biogas plants as well as a better decision basis for the optimization of facilities and operation as well as political-strategical developments within the framework of the Austrian energy and climate policy.

5 ACKNOWLEDGEMENTS

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(76) LCA OF A COLLECTIVE BIOGAS PLANT TO MANAGE MANURE IN A FRENCH INTENSIVE FARMING AND AGROINDUSTRY AREA

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EXECUTIVE SUMMARY

This work has been carried out to evaluate the environmental impact through a Life Cycle Assessment (LCA) of a collective biogas plant incorporating pig slurry, cattle manure and waste from food processing industry. This collective biogas plant is actually a project located in an intensive farming and agro-industry area close to Rennes (France). Water from surface resources (river) located within this area is used to supply the towns located around for tap water. However, the excess of nitrogen locally applied on agricultural soils led to an increase of nitrate concentration in the water of the area and consequently, the water plant was recently closed. A collective manure management including biogas plant and post-treatment of digestate is seen as a possible solution to reduce the local pollution (mainly nitrate) through export of nitrogen. In this context, the aim of this study was to provide scientific elements on such solutions and to take into account the global environmental impact. So, a LCA has been realised to compare different scenarios of manure management according to an environmental point of view.

Three scenarios of manure management are evaluated including a reference scenario representing the common practice and two other scenarios with collective biogas plant. The difference between the scenarios with collective biogas plant concerns the post-treatment of digestate: the post-treatment is composting in one scenario while it is evaporation process with concentration of digestate in the second one. These three scenarios make it possible to focus (1) on the impact of transport which was precisely modelled for each scenario using Geographic Information System (GIS), (2) on uses of energy from biogas and (3) on treatment techniques and agronomical management of the digestate. In order to compare these three scenarios, a functional unit describes scenarios functions related to the practices of the area. The boundaries of this study begin to the storage until the export and application of digestate. The scenarios were modelled thanks to GaBi 4 software.The CML 2001 method was used to calculate environmental impacts. The scenario with biogas plant and evaporation seems to be the least impacting even if the differences between the two scenarios with biogas plant are small. Results analysis only focuses on three impacts categories: acidification potential, eutrophication potential and global warming potential. These three impact categories are judged relevant to provide answers to nitrogen excess, manure management and transportation importance. These results are examined through foreground and background boundaries and through generated and avoided impacts. For climate change, at the foreground, biogas plant by itself does not contribute to the reduction of greenhouse gases but the manure management plays a major role, through the reduction of storage time in farm. The LCA results clearly show, especially for global warming, the interest of the utilization of biogas thanks to energetic recovery through electricity and heat in terms of avoided impacts to the background. For the impact of acidification, the evaporation and concentration scenario presents the lowest impact mainly due to the reduction of ammonia emissions during land spreading (because of the acidification of the digestate during post-treatment). On the contrary, the composting scenario does not present a decrease of the impact compared to the reference because the decrease of ammonia emissions during storage are compensated by an increase of ammonia emissions during composting and land spreading. For eutrophication, we do not find a global trend. The scenario with evaporation shows a lowest eutrophication impact within the local area resulting from the nitrogen export but firstly the reduction of the impact is low in comparison with the quantity exported due to the increase of ammonia emissions and secondly it is just a potential impact transfer to another place. To identify potential impact transfers especially for local impact like eutrophication, it would seem wise to more precisely locate nitrogen releases.

This study provides interesting results but also highlights several limitations as regards the assumptions and results. Certain assumptions are questionable especially the allocation rules for mineral fertilizer and the modelling of nitrate leaching. The complexity of a rural area, the plurality of stakeholders and the waste diversity show that the implantation of a collective biogas plant should result from a consultation process. Such a project must combine interest of each stakeholder and take into account features of area and environmental issues. The realization of LCA may bring some elements to the collective debate and insert environmental considerations in decision making support.

1 INTRODUCTION

These last decades were marked by a frantic search for renewable and clean energetic resources. It results from two significant environmental phenomena: the awareness of climate change generated by the increase in the greenhouse gases emissions and an energetic resources exhaustion. Socio-economic and political phenomena come in addition. In parallel, the production of organic waste increases in France and in the most developed countries because of the increase in the production sources and the establishment of organic waste collection. The management of these waste flows is a real challenge as regards to the nuisances and pollution which can be generated but also and in a paradoxical way the potential energetic and renewable resource. It should take advantage of these potential energetic resources for the double aim: the anticipation of regulatory changes and the economic benefits.

1.1 Background

It is in this context that the process of anaerobic digestion emerged since 2000. The anaerobic digestion had known a rise in the 70s after the two oil shocks with a hundred plants in France but the French regulatory has led to the decline of this technology. The anaerobic digestion aims to produce from organic waste a biogas (rich in CH_4 and CO_2) and a digestate (Baks and al., 2009). The biogas can be recovered in electricity and heat thanks to cogeneration. Then it is possible to substitute biogas for fossil fuel and to reduce greenhouse gases emissions. The digestate can be spread in agricultural field and can lead to nutrients recovery. Additionally, anaerobic digestion of livestock waste makes it possible to reduce methane emissions occurring naturally during the storage of this wastes. Despite these potential benefits, the environmental relevance of the different developments of the process and its implementation within territories highly depends on territory characteristics in which it fits and especially the issues of the existence of opportunities to recover biogas and digestate. This relevance remains difficult to assess and must be examined case by case which limits its social acceptance. That is why the positioning of local decision makers is often difficult.

1.2 Research objectives

In order to study and understand the environmental relevance of an anaerobic digestion process within a territory, it clearly appears that the territory in question must present some characteristics and constraints. The region of Brittany in France is typical for its intensive farming, the bad quality of drinking water and its energetic dependence. To model the implementation of an anaerobic digestion plant and its impacts, the scale of watershed is desirable. The Coglais is a rural area in Brittany with about 5000 inhabitants located fifty kilometers northeast of Rennes. Agriculture occupies 15% of the workforce (well above the national average). It is a ground water supply of the city of Rennes and in this area of intensive farming (dairy farming but also dominant pig), the issue of protection of water quality arises. Besides, this watershed “Les Echelles” is in dispute with the European Commission due to the non-compliance with the nitrates directive because of the spreading of an important quantity of nitrogen in view of the nitrogen excess in soil. So as to answer this set of issues, the implementation of a collective biogas plant with a step of post-treatment of digestate is considered. The post-treatment should make it possible to export a certain amount of nitrogen outside the territory. To highlight the environmental benefits and impacts of the potential energetic recovery and the interest of post-treatment of this type of scenario, an environmental assessment, a Life Cycle Assessment (LCA), is carried out. This evaluation uses a detailed modeling of different scenarios for the implementation of anaerobic digestion within this territory especially thanks to Geographical Information System (GIS). This detailed modeling focuses on the identification of energetic waste and the transport of organic waste and digestate.

2 METHODOLOGY

To carry out an environmental assessment of anaerobic digestion within a territory, the most suitable tool has been chosen according to the context and the aim of the study, the nature and the complexity of the system. Taking into account these various criteria, it appears that the most relevant tool is Life Cycle Assessment (LCA) for this eco-design approach.

2.1 The choice of Life Cycle Assessment

LCA is a recently standardized tool by the International Standard of Organization between 1997 and 2006 (ISO, 2006). It makes it possible to identify and quantify environmental aspects and impacts, throughout the life cycle of systems. Thus, for each life cycle stage, associated emissions and environmental impacts are quantified. The existence of pollution transfers into space and/or in time and the significant flows or stages are identified. The results of LCA are used in a decision-making process. LCA is an iterative method which is composed of four following stages:

- goal and scope definition: definition of the aim of the study, the system boundaries, the functional unit (the quantification of the system function, basis of the comparison), allocation rules, assumptions…;
- inventory: data collection;
- impact assessment: environmental impacts quantification;
- interpretation: conclusions from LCA outcomes.

In order to have an overview of research studies about environmental evaluation of anaerobic digestion, a bibliographic review about LCA of manure treatment has been realized. The aim of this review is to identify methodological characteristics of LCA of a biogas plant within a territory. The first observation is the low number of publications. For LCA of waste management in general, the functional unit focuses on the inputs. Certain authors explain clearly the functional unit and the functional unit most frequently chosen is “the treatment of a certain quantity of manure” (Hamelin and al. 2010, Lopez-Ridaura and al., 2008, Lopez-Ridaura and al., 2009, Prapaspongsaa and al., 2010). But other authors are interested by the outputs: electricity production (Hartmann, 2006) and quantity of exported nitrogen (Rehl and al., 2007). Most papers take into account the biogas and digestate recovery. All these information are useful to model the anaerobic digestion scenarios and to focus on three previous environmental issues.

2.2 Goal and scope definition

Goal and scoping definition is an essential stage for the implementation of a LCA. It makes it possible to determine the goal, the nature of the system, the functional unit and the system boundaries.

The aim of this study is to assess the environmental performances of different scenarios of manure management and especially a collective biogas plant scenarios and a reference scenario (current situation). A collective biogas plant is able to treat waste produced within a territory such as agricultural waste but also agro-industrial waste. For the studied territory, the power of the plant is estimated at 1 MW. The heat production has to be sufficient to meet the need of an important step of digestate post-treatment such as the evapo-concentration. According to local stakeholders, farms and agro-industries considered are within the watershed with a perimeter additional of 3 km in order to have a sufficient quantity of organic waste to reach the desirable power. In the watershed, the average nitrogen pressure is about 180 kgN/ha per year of agricultural area. One of the limits is the lack of accurate data about the quantity for export to reach an agronomic balance and to comply with regulations. Otherwise, a target of maximum export is set at 50% of manure raised on farms in order to be below 140 kg Norg/ha. So different scenarios are possible (cf. Figure 1).

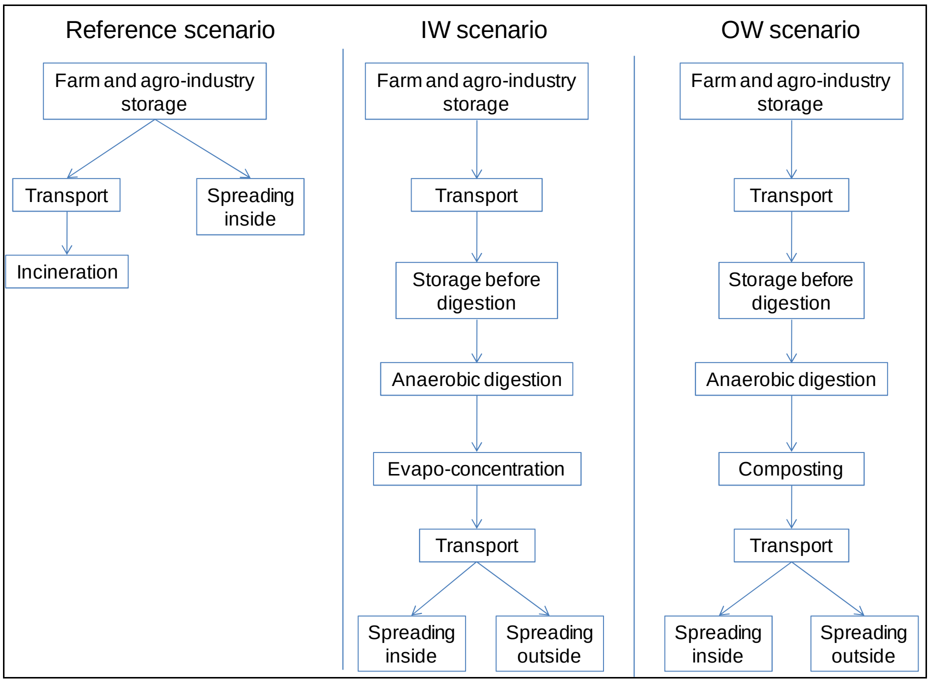

FIGURE 1 **Flow chart of the three scenarios**

So three scenarios are assessed:

- a reference scenario with manure storage and spreading within the watershed (without anaerobic digestion process);
- a collective biogas plant scenario with a step of composting for digestate post-treatment. The heat produced from biogas is used by a dairy in Saint Brice en Cogles located out of the watershed. This scenario makes it possible to optimize the use of thermal energy leads to more transport. It is called OW (out watershed).
- a collective biogas plant scenario with a step of evapo-concentration for digestate post-treatment. This post-treatment consumes substantially all of the energy. The plant is within the watershed and near the farms in order to limit the transports. This scenario is called IW (in watershed).

The functional unit is defined according to the input: the management of the annual production of cattle and pig manure and agro-industrial waste produces in watershed and within 3 km that is, 24375 t of agro-industrial waste and 8046 t of cattle manure and 47016 t of liquid cattle manure and 9652 t pig manure. The boundaries of the studied systems start from the storage of these organic wastes until the spreading and the export of the digestate, that is, a “gate to cradle” analysis. The scenarios present some characteristics and especially some co-functions which are not described in the functional unit. These co-functions are subject to allocation rule:

- heat and electricity recovery after the digestion (for IW and OW scenarios);
- heat and electricity recovery after the incineration (in reference scenario);
- nutrients recovery (N, P, K) after the digestate spreading (for the three scenarios).

For these three co-functions, the rule of allocation is the substitution by subtraction. This rule makes it possible to subtract from environmental impact of a scenario the avoided impacts thanks to the substitution of the additional functions:

- for the biogas recovery the electricity is substituted to the same quantity of electricity produced in a classic way, that is, the country mix; and the heat is substituted to the same quantity of heat produced in a classic way for industry, that is, natural gas furnace.
- for the recovery of heat and electricity produced by the incineration: the electricity is substituted to the same quantity of electricity produced in a classic way, that is, the country mix; and the heat is substituted to the same quantity of heat produced in a classic way for district heating, that is, wood furnace.
- for the recovery of nutrients thanks to digestate spreading: due to the lack of data, only N was considered and N is substituted to mineral fertilizer according to an assumed rate 50% of total nitrogen.

Besides for scenario with composting, the incorporation of green waste and a structuring agent is necessary in order to make compost from digestate. As these products are not covered in other scenarios; gaseous emissions from these wastes are not considered. It does therefore not create a bias in the boundaries of the three scenarios.

2.3 Inventory

This step consists in obtaining data for all life cycle stages. To carry on the inventory of these three scenarios, different sources of data are used including bibliography, field measurements and expert consensus. To precisely model the deposit of waste, the distance of transport and the localisation of the collective biogas plant, the GIS are used (Bioteau et al., 2012). Each stage of life cycle is modelled with parameters in order to link operating conditions to matter and energy balance (especially balance of nitrogen and carbon) and to atmospheric emissions.

2.4 Impacts assessment

This quantification step has been implemented thanks to a LCA software, Gabi 4. This software is a tool which allows us, after the construction of the flow chart (cf. Figure 1), to carry out impacts evaluation according to available characterization methods in this software. To do the third step of this LCA, a method of impacts characterisation has been chosen: the CML 2001. This method is the most used and converts emissions and resources consumption into potential environmental impacts. Indeed, CML 2001 is a classic and midpoint method trying to model impacts effects.

3 Results

This last step of LCA consists of analysis results and suggestion of some improvement means for stages which present important impacts. The CML 2001 method assesses ten environmental impacts but only three will be thoroughly analyzed: acidification (kg SO_2 eq.), eutrophication (kg PO_3^{4-} eq.) and climate change (100 years) (kg CO_2 eq.) (cf. Figure 2).

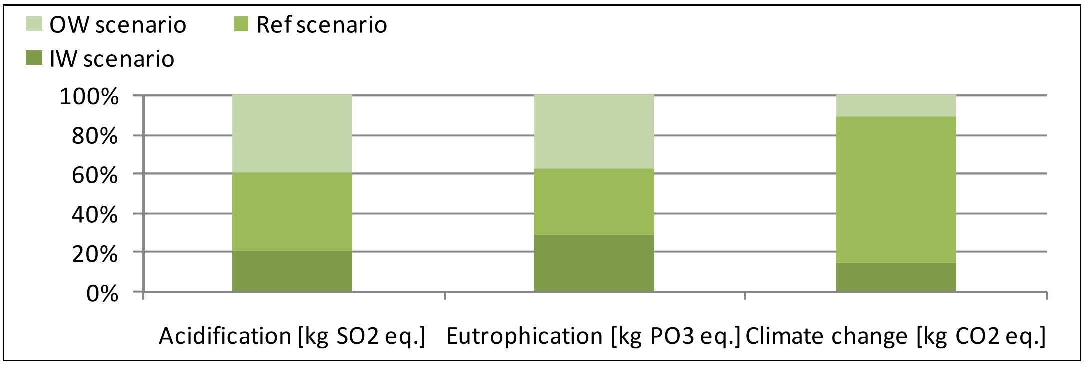

FIGURE 2 **Comparison in relative between the three scenarios for three impact categories**

These three impact categories represent three of the main environmental issues in the management of livestock manure. In addition, two of these impact categories (climate change and acidification) can highlight the possible impact of transport. Overall the implementation of a collective biogas plant improves the environmental situation compared with the reference scenario for acidification and climate change thanks to the reduction of the time of storage in the farms and for the climate change, avoided impacts thanks to biogas recovery in electricity and heat. However this benefit is highly mitigated by the leaks of biogas. For eutrophication, there is no improvement between the three scenarios because of a pollution transfer with the export of nitrogen (cf. Figure 2). The life cycle stage which is responsible for the eutrophication and acidification impacts is the spreading of treated digestate with the emission of ammonia for both and nitrate leaching for eutrophication (cf. Figures 3 and 4). To calculate gaseous emission and nitrate leaching, a model (INRA) is used to simulate nitrates leaching according to the amount applied.

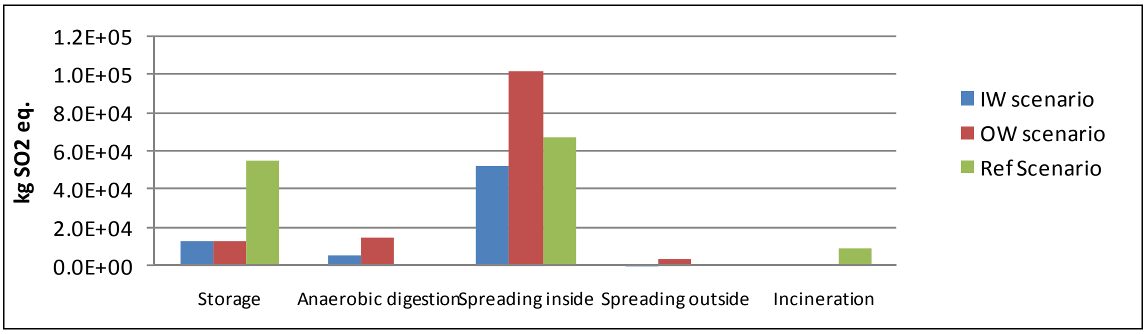

FIGURE 3 **Comparison between the three scenarios for acidification**

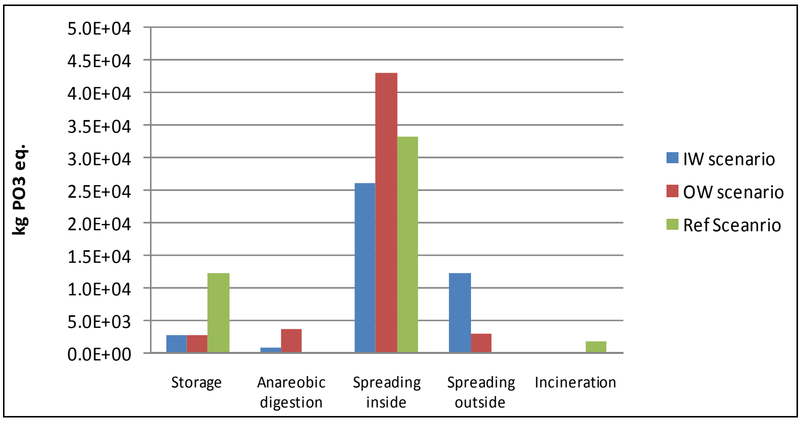

FIGURE 4 **Comparison between the three scenarios for eutrophication**

The life cycle stages which are responsible for climate change are the anaerobic digestion with methane leaks and the composting with emission of N_2O (cf. Figures 5 and 6). These N_2O emissions during composting are due to the mineralization of nitrogen.

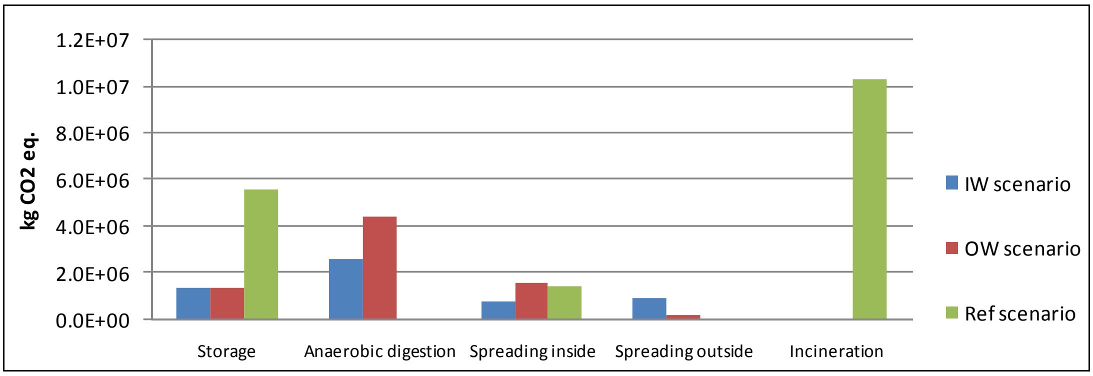

FIGURE 5 **Comparison between the three scenarios for climate change**

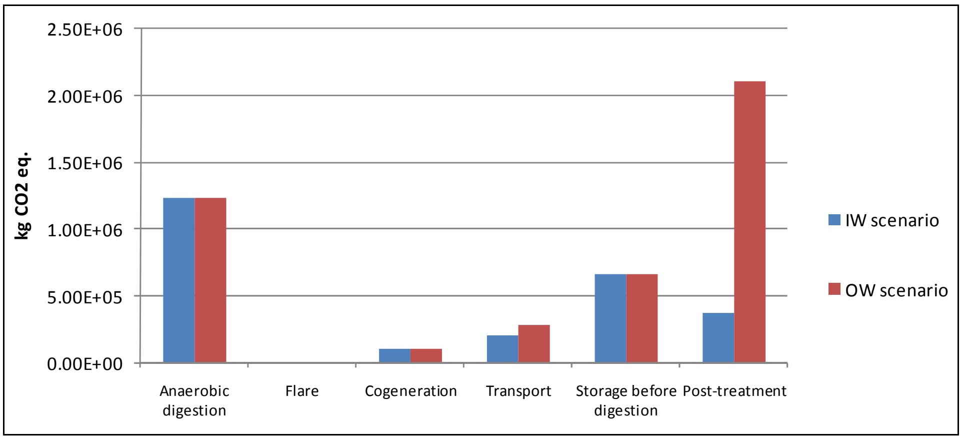

FIGURE 6 **Focus on collective biogas plant for IW and OW scenarios for climate change**

For avoided impacts, the results are contrasted only for climate change because of the difference for energetic recovery between the two collective biogas plant scenarios (cf. Figure 7). The scenario with composting shows a higher recovery because the composting process does not need heat unlike the evapo-concentration process.

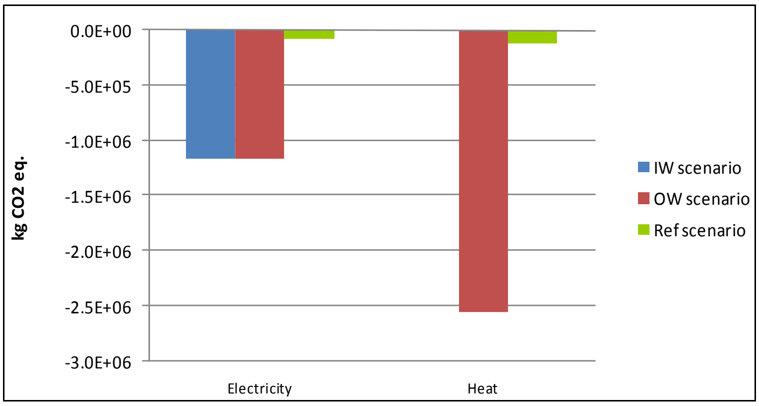

FIGURE 7 **Focus on collective biogas plant for IW and OW scenarios for climate change**

4 DISCUSSION

The expected results should have allowed us to determine the environmental relevance of a collective biogas plant within a territory with certain constraints. The obtained results are more nuanced and show rather the importance of the decision-making as regards to the environmental issues of the territory and the priority of the stakeholders. The LCA methodology presents some limits to evaluate the environmental relevance of the nitrogen export and shows only a pollution transfer because of the non consideration of sensitive area in terms of eutrophication.

4.1 Original objectives and results

The goal of the study was to model alternative scenarios for the assessment of environmental performances of anaerobic digestion and the interest of energetic and matter recovery. The analysis of results brings out the interest of energetic recovery especially when the biogas plant is near the demand (in this case, the dairy in Saint Brice en Cogles). For the digestion performances it clearly appears that the leaks of biogas contribute highly to the climate change (21% of the total impact for the evapo-concentration scenario and 16% for the composting scenario).

To reach the objective of nitrogen export for a part of waste and thereby to contribute to the improvement of the quality of drinking water, it is necessary to choose the evapo-concentration as post-treatment. This process makes it possible to export twice more digestate than composting while concentrating the nitrogen. Thus 200 t of nitrogen are exported against 39 t after composting. However this process needs a lot of thermal energy.

Some means of improving can be identified:

- for acidification, the reduction of ammonia emissions (about 90% of the total impact for the three scenarios) during spreading and during the storage in the farm;
- for eutrophication, the spreading and the storage are also the most impacting stages of life cycle about 90% of the total impact for composting and reference scenarios and 70% of the total impact for the evapo-concentration scenarios.

For these two impact categories, the improvement of the sealing could limit the leaks and the decrease of pH may block the ammonia volatilization.

The transport does not contribute highly to the three impact categories relative to other stages of life cycle. These outcomes are probably due to the fact that the farms are concentrated within the territory and transport distances are not important even for the OW scenario. For the OW scenario including composting, the distances are more important but this impact is mitigated by the environmental benefit permitted by the heat recovery in the dairy.

4.2 Major trends and the role of stakeholders in the decision-making

To meet decision-makers will for the nitrogen export to limit the excess situation, the scenario with evapo-concentration seems to be the most adapted. It brings a local answer for eutrophication even if this improvement is due to pollution transfer in space, to another location. Nevertheless it probably makes it possible to maintain the level of concentration of farms in this area and the pollution transfer can not be a sustainable solution.

If the will of decision-makers is rather a project with multi stakeholders within the territory favoring the collaboration between farmers and industrial, the scenario with heat recovery by an industrial of the territory should be preferred. However this option excludes the use of a post-treatment which consume a lot of heat. Contrariwise, the aim of export should be lowered. This type of project has the advantage to consider all the stakeholders.

4.3 The need of spatial differentiation

The results of this LCA show the importance of the geographic scale according to the aim of the study. In this study, the main aim is to see the environmental interest of the nitrogen export thanks to digestate post-treatment. The export of nitrogen outside the watershed shows a decrease of eutrophication in the studied watershed. However the eutrophication impact is just displaced to another watershed because of the non-consideration of the sensibility of the impacted environment. In order to more precisely assess the impact of the nitrogen export according to the sensibility of the media, it seems relevant to take into account the sensibility to eutrophication throughout the characteristics of the watershed such as quantity of nitrogen in soil, intensive farming, and presence of watercourse. This impact assessment with the account of local characteristics is called spatial differentiation and the consideration of these characteristics should make it possible the calculation of a local impact of eutrophication.

5 CONCLUSIONS

In the context of energetic crisis and water pollution, the management of livestock manure is a challenge. The anaerobic digestion coupling with digestate post-treatment is hopeful to solve some environmental issues. An environmental evaluation throughout a Life Cycle Assessment is needed to check the environmental relevance of this collective biogas plant within a rural territory.

This study in the territory of Coglais shows the environmental interest of a collective biogas project, especially in terms of avoided impacts to climate change linked to the use of heat and electricity. It also showed that the biogas plant, in itself, is not involved in the reduction of climate change but that is the decrease in time effluent storage, permitted by the biogas plant, which plays the main role.

The two variants made up of digestate post-treatment direct to two different issues: the export of an important quantity of nitrogen outside the watershed thanks to the process of evapo-concentration, meeting the objectives of export, and the heat recovery for the dairy industry which does not answer the nitrogen excess in the territory of Coglais (case of composting). With the process of evapo-concentration, the environmental performances of the study area for eutrophication are improved only thanks to a potential impact transfer. However for acidification, the results of evapo-concentration process show a real decrease for the study area while the results of scenario including composting show an increase because of the ammonia volatilization during spreading after composting. This aspect is highlighted by the life cycle thinking. In the case of composting, the heat recovery avoids consequent impacts related to climate change. Finally, the transport weakly contributes to the three studied impacts (climate change, acidification and eutrophication), compared to livestock manure storage or leaks related to the biogas plant.

Some limits were raised during this study and helped to highlight the need for spatial differentiation and for accurate scientific data and adapted to the LCA. Recall that this method is iterative and very time-consuming, which is also one of the limitations of the study. Besides this study brings together a number of research works carried out under the whole research project called BIODECOL2. It shows the need for partnership between researchers, public stakeholders, industrial and farmers.

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(189) LEAKAGE CONTROL OF BIOGAS PLANTS

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EXECUTIVE SUMMARY

Anaerobic digestion has become a very important technology to treat organic waste and to generate renewable energy. During construction and operation leaks at biogas plants may occur and methane is emitted. This is undesired because methane is a greenhouse gas and contributes to global warming. Additionally leakages may be a significant safety issue. Finally methane emissions decrease the economic efficiency of the plant as the emitted methane cannot be used as renewable energy. In the context of the Industrial Emission Directive, emissions from anaerobic digestion (AD) plants are re-evaluated and leakages may be of importance.

Different strategies for leakage detection were evaluated on their efficiency. A combined strategy using a methane sensitive camera combined with gas analysis instruments seems to be the best approach to detect leakages. The advantages of the camera are the documentation of leakages even at remote spots of the plant.

Based on the experience of leakage detections on more than 600 AD plants, an evaluation tool was developed that takes safety, environment and economy into account.

1 INTRODUCTION

Leakages at AD plants emit methane. This can be a safety problem because of the increased danger of explosion. Additionally, CH_4 is a potent greenhouse gas that affects our climate. And finally emitted gas cannot be used as renewable energy source and may influence the economic balance of an AD plant significantly.

Leakage detection can be performed with gas sensors and leakage sprays (aqueous solution with detergents sprayed on potential leaking spots or areas). These methods are very time consuming and many parts of the plant cannot be accessed by the staff. In contrast the use of mid-range IR cameras for methane visualisation offer an efficient method to analyse the whole AD plant on leakages- including parts of the plant that are difficult to access. If this technology is used professionally, this is the most recommendable detection method.

We describe different methods for leakage detection and introduce an evaluation scheme for leakage reporting at AD plants.

2 LEAKAGE DETECTION METHOS

2.1 Pressure

AD fermenter can be pressurized before the start up. Then the decrease of pressure is monitored. A fast decrease hints on leakages. Sometimes smoke is produced in the fermenters and it is observed if the smoke is visible at the outside of the fermenter. The major disadvantage is that AD plants have a rather low pressure inside, usually only 1-3 mbar higher compared to the ambient pressure. This makes a reliable pressure measurement hard in such a small pressure interval, especially if temperature is changing. Usually, smoke emission shows only big leakages. Sometimes the pressure decrease is analysed during plant operation but then pumping and agitating processes have to be considered as they influence the pressure as well.

2.2 Leakage spray or foaming agents

If a leakage is expected on the plant, the area is sprayed with a leakage spray or a foaming agent. This method is very cheap but it is very time consuming. Additionally not all parts of the plant can be accessed with this method.

2.3 Gas sensors

Methane concentrations can be analysed with different methods:

- Flame ionisation detector (FID)
- Semiconductors

- Catalytical combustion (reclaescence)
- Heat conductivity
- Electro-chemical sensors
- Infrared-absorption (cuvettes)

Based on the analysed concentration the size of a leakage can be estimated. Some tools such a measuring rod increase the accessibility of the devices. The concentration measurement is performed at a single spot only. As a consequence the whole plant has to be checked and this makes the method very time consuming. According to our experiences, CH_4 clouds from leakages may lead to a signal in a gas sensor even there is no leakage at the point measured. This may lead to wrong conclusions at the AD plant.

2.4 Non-Destructive Testing

From a distance CH_4 can be detected as well. Molecules of different atoms absorb radiation according to their molecular structure. For CH_4 this is the case in the infrared. This characteristic is used in laser and infrared cameras.

- Laser

Lasers do emit a light beam in the infrared. Methane absorbs some of the signal in the reflected beam. The CH_4 concentration between the laser and the reflected surface is analysed. The unit is ppm m. Especially gas membranes don´t reflect enough energy for an appropriate analysis. This makes it rather time consuming to analyse the whole plant. Again, clouds of CH_4 from nearby leakages may lead to high CH_4 signals and may result in wrong leakage detection.

- Gas cameras

Gas cameras don´t have an own emission source but they are “classical” infrared cameras that use a narrow band pass filter. This filter covers typical ranges for the molecule to be analysed in the infrared. For example, methane absorbs radiation around 3.3 µm and around 7.8 µm. If CH_4 is emitted, it can be displayed by the camera and can be documented as a movie or picture.

Gas leakages can be determined reliably out of a certain distance (bigger leakages e.g. open over pressure valves out of a distance of more than 100 meters). The whole AD plant can be examined rather easy. Misinterpretation of CH_4 clouds are avoided, because the camera can identify the actual leakage point. The documentation of leakages is in form of pictures and/or movies. This makes documentation and maintenance rather easy. Although this method requires a high investment and highly qualified staff to operate the camera it is the only possibility for a fast and reliable leakage control, at the moment.

3 QUALITY MANAGEMENT

According to the experience of more than 600 controlled AD plants, the leakage control with an infrared camera alone is not enough for an appropriate result. A group of four leakage service providers initiated a working group on quality control in Germany in 2011. The working group is called “Quality Assurance for Methane Emission Detection at Biogas Plants”.

3.1 Goal of the working group

The goal is to develop a uniform approach for leakage detection service at a high quality standard. Additionally, objective and transparent criteria for the evaluation of leakages are developed. This is necessary to take all environmental, economical, safety and legislative issues into account. Additionally the operator needs information if the leakage has to be maintained immediately or whether it can be fixed during the next revision of the plant.

A successful control needs certain standards for the applied analysis, the staff and the reporting.

3.2 Minimum requirements for leakage detection

The minimum requirements are necessary to visualize the leakages and to evaluate their significance. Additionally boundary parameters need to be detected:

- Methane sensitive IR-camera (cooled to < -150°C) with a narrow band pass filter for hydrocarbons, sensitivity < 25mK (figure 1)
- Methane sensitive analytical device combined with a pump for an active gas supply to the detector
- Devices for the documentation of pressure, temperature and wind speed
- Devices to determine the air flow (e.g. anemometer)

FIGURE 1 **Example of two gas sensitive IR-cameras**

3.3 Documentation

The documentation is to report clear defined leakages and it contains:

- Customer and leakage service company
- Meteorological parameter: weather, pressure, temperature, wind speed
- Description of the plant in form of a figure or photo
- Current operating conditions (if possible: gas pressure, gas level in the gas storage)
- Plan of the detected leakages
- Every leakage is documented in as photo and movie
- Every leakage is evaluated according to an evaluation matrix

3.4 Evaluation matrix

The matrix considers safety issues, environmental and economic concerns. For each criterion 0-2 points are allocated (table 1). The sum of the points classifies the leakage.

TABLE 1 **Evaluation matrix**

	Category 2 (0 points)	Category 1 (1 point)	Category 0 (2 points)
CH_4 (Vol.-%)	< 0,5	2,5	> 2,5
Accessibility of the leakage	Under normal operation no access	Access only with additional equipment (e.g. ladder)	Easy access
Distance to the next ignition source	No ignition source	< 2m	< 1m
Potential increase of the leakage	no	-	yes (e.g. cracks in membranes)
Point of emission	Exposed (roof of the fermenter)	Partly exposed (platform)	Enclosed space (CHP)
Emission potential (l/h)	< 100	< 1.000	> 1.000

3.5 Leakage classification

In general every leakage is unwanted and should be fixed. However it needs to be considered how fast a leakage should be maintained (table 2). A small and not safety relevant leakage needs not to fixed immediately, especially if the maintenance induces high emissions, e.g. when a gas roof is replaced. In such a case the potential emission by the maintenance may be higher compared to the emission savings from the fixed leakage. Safety relevant leakages require a fast maintenance. If possible leakages are fixed immediately if fixing requires simples measures, e.g. sealing a flange.

TABLE 2 **Leakage classification**

Category	Points	Maintenance
3	< 3	Maintenance at the next revision of the plant
2	3-4	Within 6 months
1	5	As soon as possible*

*: if there is an increased safety danger for persons or an increased explosion danger, the leakage hast tob e fixed immediately.

3.6 Qualification of the staff

Experienced and well trained staff is necessary to perform a successful leakage control. In addition service companies should be independent. The requirements are suggested as follows:

- Service companies or independent consultants should be experienced in the biogas sector
- Service companies or independent consultants should be independent and should not plan or construct biogas plants
- The leakage detection staff successfully takes part in a course on visualisation of gas leakages
- The companies/persons take part regularly in seminars on gas leakage

3.7 Experiences on leakages

The members of the working group have analysed more than 600 AD plants so far. Around 80-90% of the AD plants showed some CH_4 leakage. As operators tend to order a leakage analysis only if they assume some gas losses it is assumed that the overall leakage rate at AD plants is lower.

According to the leakage definition:

- around 30% showed smaller leakages (category 3)
- around 45% showed medium leakages (category 2)
- around 25% showed big leakages (category 1)

Frequent leakages were the connection of fermenter walls and membranes and non-sealed holes for wires (for submersed agitators). More over about 10% over the over pressure devices were open.

4 RECOMMENDATIONS

During operation leakages may occur at any time. Leakage control should consist of two parts: self-control and external control.

Self-control means to control the AD on a regular base. Daily the performance of the plant and over pressure devices are controlled. On a monthly base non sealed holes for wires (for submersed agitators), water seals, portholes and the exhaust gas from double foil roofs should be controlled. For this, a gas sensor is necessary.

External control means a yearly control from experts for a professional leakage control. After start-up operation, the first leakage control should take place. Additional controls may be useful e.g. after roof removal, if the plant does not produce the expected amount of biogas but the biological process works fine, if odour occurs and the emission spot cannot be detected by the operator.

5 EXAMPLES

5.1 Non sealed screw

	category 2 (0 points)	category 1 (1 point)	category 0 (2 points)	Evaluation
CH_4 (Vol.-%)			X	2
Accessibility of the leakage			X	2
Distance to the next ignition source			X	2
Potential increase of the leakage	X			0
Point of emission		X		1
Emission potential (l/h)		X		1
			Sum	8

Category 3: < 2 points:	maintenance at the next revision of the plant
Category 2: 3-4 points:	within 6 months
Category 1: > 4 points:	**as soon as possible**

Note:in this case the screw was fixed within seconds

5.2 Wire that fixes a submerged agitator

	category 2 (0 points)	category 1 (1 point)	category 0 (2 points)	Evaluation
CH_4 (Vol.-%)			X	2
Accessibility of the leakage		X		1
Distance to the next ignition source	X			0
Potential increase of the leakage	X			0
Point of emission	X			0
Emission potential (l/h)		X		1
			Sum	4

Category 3: < 2 points:	maintenance at the next revision of the plant
Category 2: 3-4 points:	**within 6 months**
Category 1: > 4 points:	as soon as possible

5.3 Connection of membrane an fermenter wall

	category 2 (0 points)	category 1 (1 point)	category 0 (2 points)	Evaluation
CH_4 (Vol.-%)			X	2
Accessibility of the leakage	X			
Distance to the next ignition source	X			0
Potential increase of the leakage	X			0
Point of emission	X			0
Emission potential (l/h)		X		1
			Sum	3

Category 3: < 2 points:	maintenance at the next revision of the plant
Category 2: 3-4 points:	**within 6 months**
Category 1: > 4 points:	as soon as possible

5.4 Hole in the gas holder membrane

	category 2 (0 points)	category 1 (1 point)	category 0 (2 points)	Evaluation
CH_4 (Vol.-%)	X			0
Accessibility of the leakage	X			0
Distance to the next ignition source	X			0
Potential increase of the leakage			X	2
Point of emission	X			0
Emission potential (l/h)		X		1
			Sum	3

Category 3: < 2 points:	maintenance at the next revision of the plant
Category 2: 3-4 points:	**within 6 months**
Category 1: > 4 points:	as soon as possible

(207) CHINA BIOGAS POTENTIAL AND ITS ESTIMATED CONTRIBUTION TO CLIMATE CHANGE MITIGATION

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EXECUTIVE SUMMARY

China comes first on the world list as to coal and nitrogen-fertilizer consumption, solid waste production and CO_2 and CH_4 emissions. At the same time, the country has to cope with one of the most rapid periods of urbanization in history. China is currently making many efforts to reduce and even to cope with these problems in order to ensure its energy supply and a sustainable social, economic and environmental development. Anaerobic digestion technology could have a great impact on all the above-mentioned problems and contribute positively to the renewable energy sector. An assessment of biomass resources was performed to evaluate the theoretical biogas potential from urban wastes (household waste, municipal sewage sludge) and agricultural wastes (crop residues, animal manure). This study calculates only the biogas potential of feedstock, which is used in middle- and large- scale biogas plants. Landfill gas, decentralized wastewater treatment systems and small household digesters are not included. A theoretical current annual biogas potential of 290 billion m^3 was estimated, which could account for 6.9% of the total energy demand. Furthermore, the annual potential could increase to 439.4 billion m^3 by 2030 by including the feedstock energy crops from marginal land. Biogas plants with an initial capacity potential of 71.4 GW could, therefore, be installed and could contribute more to the renewable energy targets as planned so far. Digestate, a by-product of the process, is a sustainable green fertilizer. By treating the whole assessed feedstock, the digestate generated would replace nearly 30% of inorganic fertilizer and exceed China's current demand for bio-fertilizer. By using the anaerobic digestion process as the main treatment option for the selected feedstock, the current total reduction potential of CO_2 equivalents is 4.75 billion tons. By 2030, even 7 billion tons of CO_2 equivalents could be saved. The current and future forecasts of the biomass availability say that the resources are far from being fully exploited, but they also show that the policy targets to develop sustainable biogas energy (6.6% by 2010 and 10% by 2020) could be easily reached and much more ambitious.

1. Introduction

In the last decades, renewable energy such as biomass, solar and wind energy as well as hydroelectricity has become a very important factor for energy supply and for making the climate change less severe. (Tang et al., 2010)
Bioenergy uses bio-organic materials, i.e. biomass for energy generation. Therefore, it includes materials of vegetable and animal origin as well as their waste products. However, before using them as renewable energy source, they have to be treated. Transportation, mechanical treatment and deposit must be done in such a way as to be able to coordinate the produced biomass with the energy demand. (FNR, 2006) The following bio-energy technologies are applied in China: biogas, syn-gases from straw and stalk gasification, electricity from biomass power generation, and various solid and liquid bio-fuels, such as bio-ethanol. (Wu et al., 2009) This study deals with the technology of anaerobic digestion focusing on the main feedstock of China which is suitable for biogas generation, such as agricultural waste, i.e. crop residues and animal manure, urban waste, i.e. the native organic fraction of household waste and municipal sewage sludge, as well as energy crops from marginal land.

1.1 Research objectives

This paper is divided into four parts, which are listed below:

Part 1 assesses, based on present data, the amount, availability and the status-quo of the biogas energy potential of the PRC´s abundant biomass resources. **Part 2** assesses, based on present arising, the energy potential in 2030. **Part 3** gives insight into the extent of China's total energy demand, which could be met by the estimated biogas energy potential and could thus replace primary energy sources. In addition, present and future policies are seen in the light of biogas potential in order to demonstrate the current standing and resource utilization of biomass. **Part 4** deals with the contribution of biogas energy to climate change mitigation. Calculations of saved amounts of Greenhouse gas (GHG) emissions are made using the total available biomass feedstock for biogas generation.

2. METHODOLOGY

International papers and Chinese governmental reports were the basis for collecting data. Furthermore, interviews and discussions with experts were made. Individual methods of feedstock estimation as well as biogas potential calculation had to be chosen for each kind of feedstock. In addition to the collecting and selecting of data, basic statistical analyses were made.

The following tables show the data, which were used to calculate levels as well as the biogas potentials of each kind of feedstock.

2.1 Current Feedstock Levels and Biogas Potential

Table 1: Urban Household Waste

Data	Source
Organic Fraction within MSW: 80%	*Raninger et al., 2009*
Biogas Potential: 100 m^3/t FM	
Total Solid (TS) content: 35%	
Volatile Solid (VS) content: 80%	*Steffen et al., 1998*
Waste in Cities	
Urban Population China: 621,860,000	*NBSC, 2010a*
Generation of MSW: 0.9kg/c/d	*Raninger et al., 2009*
MSW Generation: 200 million tons	*Raninger, 2011*
Waste in Counties	
Population in Counties: 626,223,092	*NBSC, 2010a*
Generation of MSW: 0.7kg/c/d	*Raninger, 2011*
MSW Generation : 160 million tons	

Table 2: Municipal Sewage Sludge

Data	Source
Population in cities and counties: 1,248,083,092	*NBSC, 2010a*
Total amount: 133 million t/a	*Zhao et al, 2006*
Biogas potential: 0.027m^3/d.c	*Lindeburg, 2011*
TS content: 20%	*Raninger, 2011*

Table 3: Productivity of Crop Straw in 2009

Crops	Yield of Crops (10^4 t) *(1)*	Index straw:crop *(2)*	Yield of residues (10^4 t)	Proportion (%)	Biogas potential (10^9 m^3)
Rice	19510.3	0.623	12155	16.3	36.5
Wheat	11511.5	1.366	15725	21.1	47
Maize	16397.4	2	32795	44.1	98
Beans	1930.3	1.5	2895.5	3.9	8.7
Tubers	2995.5	0.5	1497.8	2	4.5
Oil bearing crops	3154.3	2	6308.6	8.5	19
Cotton	637.7	3	1913.1	2.6	5.7
Sugarcane	11558.7	0.1*	1155.9	1.6	3.5
Total	67695.7	--	74445.9	100	222.9

Source: (1) NBSC, 2010b, (2) Wang, 1994

Table 4: Crop Residues

Data	Source
Biogas Potential: 300m^3/t FM	*Raninger, 2011*

Current Utilization and Availability of Energy Usage:
However, according to *Shi (2011)*, approximately 60% of the total crops of straw could be used for energy generation.

Table 5: Production of animal manure and biogas in 2009

Animals	Population on hand (10^4) (1)	COD (kg/d/c) (2)	Biogas potential (10^9 m^3 /a)
Pig, total	46996	0.6	23.7
Cow, total	10726	5	45
Poultry, total	533000	0.02*	8.9
Total	590722	--	77.6

*Note: * Layer chicken;* 1kg COD = 0.23 m^3 biogas. (ADB, 2009)
(1) NBSC, 2010c, (2) ADB, 2009

Utilization and Availability for Energy Usage:
According to *Cui et al. (2005)* approximately 35-40% remains untreated, 50% is returned to the field directly, 5% are composted and only 3% is going to methane fermentation. (MOA, 2011) Besides the 3%, which is already in use, a utilization factor of 43% is taken for the calculations, assuming the huge untreated amount can be fully made use of.

2.2 Future Feedstock Levels and Biogas Potential

The year 2030 was chosen to show the further increase in amount and biogas energy potential of each feedstock in the future.

2.2.1 Urban Waste

As a result of the rapid urbanization, 1 billion people will live in urban areas by 2030. (McKinsey&Company, 2008) Therefore, only 50% of the counties` population are still remaining in the counties, which results in 313,111,546 people. This study assumes that the organic fraction of MSW will remain nearly the same.

2.2.2 Crop Residues

Liu et al. (2007) calculated the crop residues increase rate since the 1970s whereby the average rate has been about 1.2% in the last twenty years. This seems to be a constant index and is also used for predictions until 2030.

2.2.3 Animal Manure

The following figure shows the continuous increase in arising of livestock for the last 10 years. It is assumed that the average rate of 12% from 2000 until 2009 will stay the same.

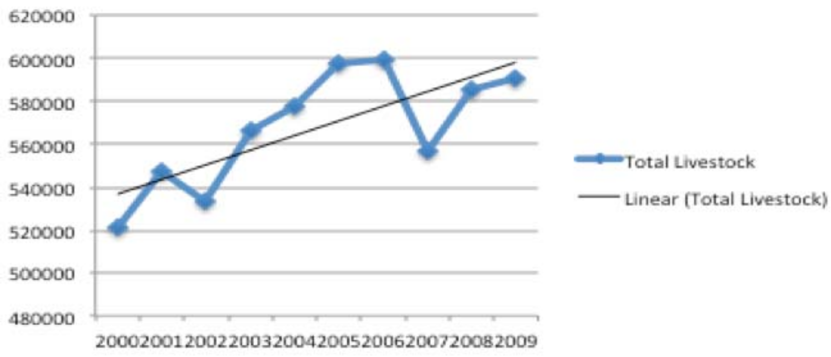

Figure 1: Total arising of livestock from 2000 to 2009 (unit 10^4)
Source: NBSC 2003, NBSC 2005, NBSC 2007, NBSC 2009, NBSC 2010c

2.2.4 Energy Crops from Marginal Land

Definition of 'Marginal Land'
In order to estimate the available size of marginal land, one must first define what is meant by marginal land. However, no reliable source concerning the definition of marginal land has been found as data vary very much, and researchers choose different kinds of species for their evaluation.

'Marginal land' is used in this study as 'the land which has poor natural conditions, has not been used for agriculture currently but has production potential and development value to produce a certain amount of biomass. Such land is not fit for farmland temporarily, but can grow some kinds of plants with high adaptability'. (Hu, 2010a)

Assumptions and Data Recalculation:

Evaluation of biogas potential is performed in the following steps:

Step 1: Marginal land appropriate for growing energy plants is analysed. The crucial results are the size and composition of marginal land in China.

Step 2: Exploration of energy plants that are suitable for biogas production is made and what amount and species of plants can be grown per hectare are taken account of in the calculations in order to get data about how many tons of plants are available for the entire generation of energy.

Step 3: The Biogas potential of the amount of different kinds of plants is estimated.

Step 4: A utilization factor of 40% by the year 2030 is estimated in this study.

Table 6: Potential energy output of biomass plants of marginal land

Type of marginal land	Area/ 10^4 hectares (1)	Suitable energy plants and area percentage (1)	Energy Output (tce/t.ha) (1)	Unit output (t/hectares FM) (1)	Biogas energy potential (10^9 m^3 VS)
Suitable for farmland	2 787	Sweet sorghum, 35%; Sweet potato, 15% (north);	2-3	60-80 (stalks) 15-20	205 25
Current marginal land	2 000	Cassava, 35% (south) Sorghum, 15% (south)	4-6	20-30 60-80 (stalks)	53 63
Barren mountain	5 004	Energy 60%	3.3	6.5	59
		Oil plant forest 20%	1.8	4.0 (seedlings)	12
		Energy crop 20%	3-5	6.5	20
Desert	700	Xerophyte shrub	2.6	4.0*	8.4
Firewood forest	175	Firewood forest	4.2	4.0**	2.1
Oil plant forest	343	Oil plant forest	1.8	4.0 (seedlings)	4.1
Shrub wood	5 365	Shrub wood	2.6	4.0	64
Total	16 374	--		--	515.6

Source: (1) Shi, 2011

Table 7: Energy crops

Data	Source
Biogas potential: 300m^3/t VS	*Braun et al., 2010*
Reference Plant: Miscanthus TS content: 30% (m/m FM) VS content: 67% (m/m DM)	*Jewell et al., 2003*

i. GHG Savings – Contribution of Biogas to Climate Change Mitigation

The following table has been used to estimate the saved amounts of emissions in CO_2 equivalents.

Table 8: Global Warming Potential$_{100}$ of Gases

Gas	Value (in kg CO_2 equivalents)	Source
CO_2	1	*IPCC, s.a.*
CH_4	23	
N_2O	296	
NH_4 NO_3 as N	7.2	*Williams et al., 2006*

2.3.1 Gaining Electricity out of a Renewable Energy Source; Replacing Coal

This calculation assumes that the heat of the CHP process is not used. Therefore, the calculation, which is stated below, slightly underestimates the amounts of saved CO_2 emissions.

- **Methane (m^3) x Calorific Value of Methane (MJ/N m^3) x Efficiency of CHP**

Table 9: GHG Savings – Calculation No.1

Data	Source
CV of CH_4: 36MJ/Nm^3	*FNR, 2011a*
CHP Efficiency (only electricity): 35%	*FNR, 2005*
Emission intensity of conventional coal: 1.186 CO_2 /MWh	*Wang et al., 2009*

2.3.2 Choosing Anaerobic Digestion instead of Landfilling as a Waste Treatment Option

In this section, only two kinds of feedstock, namely urban and agricultural biomass waste, are looked at. Energy crops are planted with the purpose of generating energy from them. Therefore, landfill is no option for this resource.

This calculation assumes that not all the landfill sites are controlled. Rather than collecting methane and using it as a renewable energy source, the gas is venting into the atmosphere. Furthermore, it has to be mentioned that methane emissions of landfill sites will take longer to emit into the atmosphere than during an AD process, even Chinese easily decayable bioorganic waste fraction converts very fast into leachate and some biogas. (Raninger et al., 2010)

For these calculations the following equations are used:

- **Emissions of Landfill = 2.46 t CO_2equivalent/t of waste** (The World Bank, 2005)
- **Emissions of Anaerobic Digestion = Methane (m^3) (CO_2/ CH_4) x (kg CO_2/Nm^3 CO_2) x GWP of CO_2**

Table 10: GHG Savings – Calculation No.2

Data	Source
Density of CO_2 : 1.977kg/m^3	*O'Leary, 2000*

2.3.3 Replacing Inorganic Fertilizer

In 2009, China's consumption of nitrogen (N) fertilizer resulted in a total of 36.9 million tons, being 35% of the total world´s use. (FAO STAT, 2011) and the per capita consumption is about 3 three times bigger than the world's population average.

In order to ensure the same amount of plant yield, only 25-30% of organic fertilizer can be added to the inorganic fertilizer (11 million tons). (Roelcke, 2011), but the plant availability of the total nitrogen in AD slurry (mainly Ammonia) is 60%. (Wendland. 2011) The total N content in the total feedstock even overtops this demand (see Table 11), which exceed China's demand.

Assumptions:

The process of generating inorganic fertilizer, mainly Ammonium Nitrate, is highly energy consuming. The nitrogen (N) of the e.g. food waste, if landfilled, would then be completely lost. This is not true of the crop residues as the alternative can be left on the ground so that they still supply N for the next cultivation. But as farmers mainly burn it on the open fields, it can be assumed to be mainly lost.

The emissions, which are generated while the fertilizer is produced, are the so-called embedded emissions i.e. 'indirect emissions' of the inorganic fertilizer. These are measured by the amount of active compounds they contain (kg of N). From each kg of N contained in the fertilizer, it takes 5,0395 kg CO_2 equivalents, as a lot of gas has to be burnt.

In this study it is assumed that the bio-fertilizer from AD is as well absorbed by the crops as the inorganic fertilizer and that, therefore, they are no insignificant N losses of the organic fertilizer.

For these calculations the following equation is used:

- **N/Feedstock (t) x GWP of NH_4NO_3**

The following table lists the data, which are used for the calculations.

Table 11: Total Solid (TS) and typical values of Nitrogen (N) content of the feedstock

Feedstock	TS content in %	N content in %	N content in 10^6 t	N content in 10^6 t	Source
			Current Levels	Future Levels	
Food Waste	35	4.14	4.2	4.7	*Dong et al., 2011*
Crop Residues	40	2.79	6.5	6.6	
Animal Manure	4	1.36*	0.36	0.4	
Energy crops	30	1.2**	--	2.4	
Urban Sludge	20	11kgN/t	0.3	0.31	*Defra, 2010*
Total	--	--	11.36	14.41	--

*Notes: *Pig Manure, **Sugar beet*

2.3.4 Using Crop Residues as Feedstock rather than Burning Them on Fields

Assumptions:

According to *Liu et al.*, (2007), 37% of crop residues are burnt directly on open fields. Instead, by treating this amount in a clean, sustainable way in an AD plant, huge amounts of GHG could be avoided.

Calculations:

The amount of GHG gases generated by burning straw is calculated, using the methodology published by the Intergovernmental Panel on Climate Change (IPCC) in '2006 IPCC Guidelines for National Greenhouse Gas Inventories'. The formula used by IPCC was slightly changed for the purpose of this paper. It should also be mentioned that indirect green house gases like carbon monoxide (CO) and nitrogen oxide (NOx) are neglected. Therefore, the result is slightly conservative as CO as well as NOx have negative impacts on the climate and lead to atmospheric and surface effects. (ITS, 2005)

Estimation of GHG emissions from fire:

- $\mathbf{L_{Fire} = B \times C_f \times G_{ef} \times 10^{-3}}$

Where:

L_{Fire} = Amount of greenhouse gas emissions from fire, tons of each GHG e.g., CH_4, nitrous oxide (N_2O), etc.
B = Biomass available for combustion, tons
C_f = Combustion factor, dimensionless, for maize residues: 0.80
G_{ef} = Emission factor, g/kg dry matter burnt, for
CO_2 : 1515
CH_4 : 2.7
N_2O : 0.07

By converting the GHG into tons of CO_2 equivalents and adding them up, the amount of tons of CO_2 equivalents can be calculated.

3. RESULTS

3.1 Total Current and Future Biogas Energy Potential until the Year 2030

The total biogas potential, including urban and agricultural biomass waste sum up to 290 billion m^3. Figure 2 shows the different types of feedstock and their biogas energy potential.

Due to the predicted increase in urbanization, the highest increase in the occurrence of waste will be in cities. It will reach 263 million tons of organic fraction in major cities and 64 million tons of biodegradable fraction in counties; Resulting in 327 million tons in total, i.e. 33 billion m^3. The resource of wastewater sewage sludge will also undergo an enormous increase of about 6%, resulting in a biogas potential of 13 billion m^3. As the increase in crops is limited by factors like land use changes and the regional and climatic situation, this fact will not change a lot in the future; only 1.2% is assumed. Therefore, a biogas yield of 218 billion m^3 is predicted. On account of the rapid economic development and the increased living standards, the demand for meat as well as for dairy products is rising. This has led to a steady grow in the number of commercial livestock farms from about 2.4 million in 2002 to about 4.3 million in

2006, with an improved quality and increased productivity. (ADB, 2009) Assuming the average rate of 12% during the period from 2000 to 2009 will stay the same, a biogas yield of approximately 37.4 billion m^3 would be the result by the year 2030. Therefore, due to an increase in urbanization and change in people's lifestyle, the highest rise will concern the resources of household waste as well as animal manure.

3.2 Energy Crops from Marginal Land

A fifth kind of feedstock, the energy crops from marginal land, is also included in the forecast calculations of biogas potential for the year 2030. Using energy crops for energy production is a relatively new field, especially for the biogas generation. Bioenergy produced from energy plants will play an increasingly important role in future energy supply. In contrast to other countries, China has a large population but limited arable land. (Hu, 2010a) As bioenergy industries should not compete for land with the production of food, feed and fiber, the plantation of energy crops will be restricted to marginal land. (Tang et al., 2010) In addition to the energy production, economic development in remote and depressed rural area can be improved, too. Research studies also prove that besides greenhouse gas emission abatement, significant eco-environmental benefits can be achieved. By planting bio-energy plants on marginal lands, the eco-function of soil/water conservation and wind erosion protection can be improved. (Li et al., 2009)

Shi (2011) estimates that around 163.74 million hectares of China´s total marginal land might be suitable for growing energy crops. 84.91 million ha, which account for 51.9% of the total area, are still not used. 70.8% of the total marginal land is well suited for forestry, 29.2% for agriculture. (Shi, 2011)

The total energy potential of energy crops planted on the total available area of 163.74 million hectares comes to nearly 345 billion m^3 biogas. As this study assumes a utilization factor of 40%, 138 billion m^3 will be used by the year 2030.

3.3 Total Future Biogas Energy Potential

Figure 2 summarizes the current biogas potential and shows also the expected changes up to the year 2030. Any kind of feedstock will increase in the future. The ranking will remain the same, i.e. crop residues are on the top of the list, followed by the new but highly valued feedstock of energy crops and animal manure. Urban waste represents the smallest amount and, therefore, also in energy potential. Nevertheless, it is more likely to be used than, for example, energy crops.

In summary it may be said, the future biogas potential will increase to 439.4 billion m^3 by 2030 with a total increase of 52%, respectively.

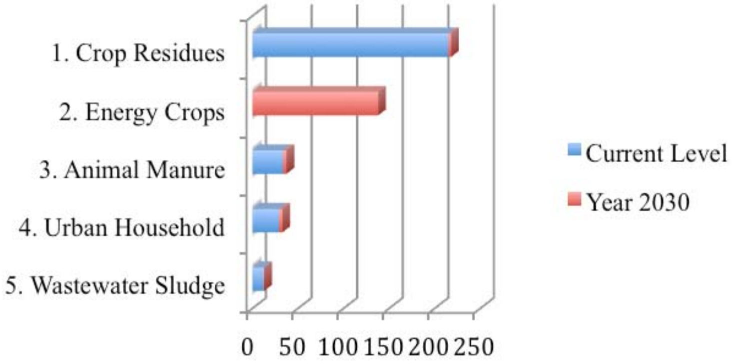

Figure 2: Current and future biogas energy potential (in billion m^3 biogas)

3.4 Contribution of Biogas Energy to China's Energy Demand

China being the most populous country is the largest energy consumer in the world. It is ranked first in global coal production as well as in coal consumption amounting to 3.5 billion tons in 2009, which makes nearly half the world's consumption with a 180% increase since 2000. Since 2009, the country has not exported but rather imported coal. (EIA, 2011) According to the 'State Grid Energy Research Institute', in 2010, China's energy demand summed up to 91,000 petajoule (PJ), and will further increase to 189,000PJ by 2030. (Hu, 2010b)

Assuming all the available biomass feedstock is used as an energy source, a total biogas energy potential of about 6,246 PJ/a could contribute to 6.9% the overall energy demand. Due to an increase in energy demand by the year 2030, 5%, which are 9.463PJ of the total energy demand, could be met. The following figures represent the respective current and future contribution of each feedstock.

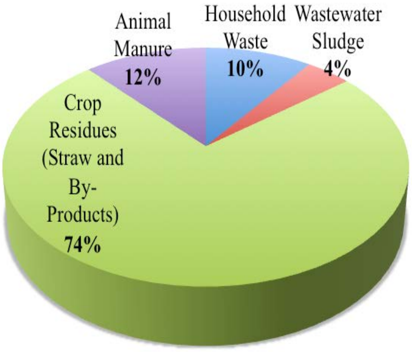

Figure 3: Contribution of the Current Potential of Biogas Energy to the Total Energy Demand

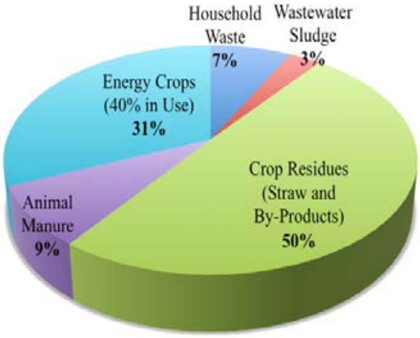

Figure 4: Contribution of the Future Potential of Biogas Energy to the Total Energy Demand

3.5 Potential versus Production; including Policy Targets

Given the amount of biomass waste available, the use of biogas is still relatively low although China has made efforts to support the production of biogas by setting appropriate goals until 2020. The current performance of biogas-plants and grid-connection will not even allow to reach these goals.

Policy Targets:

The biogas energy sector counted for 19 billion m^3 (including the weak household digesters) in 2010. The PRC intents to raise the energy production to 44 billion m^3 by 2020.

Part 1 of this study estimates a current potential of up to 290 billion m^3 of municipal sewage sludge and agricultural waste, the two kinds of feedstock, which are already mainly in use in China. However, according to the renewable energy targets achieved by the NDRC in 2010, only 6.6% of the available feedstock was used for energy supply on the market. The use of feedstock will increase to 10% until 2020.

Production:

The PRC has already invested large sums in the construction of biogas plants in the last years, especially in those treating agricultural waste. The numbers of small, medium and large sized plants treating agricultural waste feedstock rose rapidly from 26,586 in 2007 to 39,510 in 2008 and to 56,534 in 2009, which results to a total biogas yield of 764.92 million m^3 out of agricultural biomass waste. (GIZ, 2011a) However, this is less than 1% of the total agricultural resources of about 248.4 billion m^3.

As animal manure has a biogas potential of 33.4 billion m^3 resulting in 60 GWh electricity, it would not have been a problem to contribute about 14% to the demand for electricity in households of China (440 GWh) by transforming the amount of animal manure from industrial livestock farms into biogas. (NBSC, 2011)

Another example of a lost opportunity in terms of biomass resources in China is the sewage sludge.

Less than 1%, which is 100 million m^3 of the total amount of municipal wastewater sludge (12.3 billion m^3), was used in biogas energy systems in 2010.

3.6 GHG Emissions and Savings

According to the ‘United Nations Framework Convention on Climate Change’ (UNFCCC) - secretariat (1994), over 74% of the total GHG emissions are generated by China´s energy sector, which made up to 7.5 billion tons of CO_2 equivalents in 2009 (IEA, 2009), followed by 15% emitted from the agricultural, 7% from the industrial and 4% from the waste sector (UNFCCC – secretariat, 1994), resulting in nearly 10 billion tons of GHG emissions in 2009.

Therefore, replacing fossil fuel sources by energy generated from clean, renewable resources turns out to be a big opportunity to contribute to climate change mitigation. In addition, avoiding CO_2 emissions also reduces airborne pollutants, such as toxic heavy metals, ozone-forming chemicals and sulphur dioxide, which is responsible for acid rain (Li et al., 2009).

3.6.1 Contribution of Biogas to Climate Change Mitigation

This part of the study aims at estimating the amount of saved tons of carbon equivalent of CO_2 emissions due to the production of biogas energy and organic fertilizer gained from the feedstock of urban and agricultural biomass waste as well as of energy crops from marginal land whose potential was carefully estimated in Part 1 of this study. In addition, it shows which role biogas technology could play in order to achieve the ambitious targets of the national policies concerning reductions in GHG emissions.

Calculations and Results:

There are three ways to avoid GHG emissions by generating biogas from an AD process.

First, energy is produced out of renewable resources.

Secondly, the waste is used in biogas plants rather than going to landfill or open lagoons. Thirdly, the second highly valuable end-product of the process of anaerobic digestion, namely the produced digestate replaces inorganic fertilizer, whose production requires energy. Additionally, CO_2 emissions can also be saved, if a certain amount of crop residues are no longer burnt on open fields.

The current total reduction potential of CO_2 equivalents is 4.75 billion tons and it will rise to nearly 7 billion tons until the year 2030.

4. CONCLUSIONS

With regard to the specific aims of the present study, the following conclusion can be drawn and recommendations are made:

- Due to its large and diverse areas and the huge population, the country has abundant biomass resources available. This study estimates a current annual biogas potential of 290 billion m^3, which could cover 6.9% of the total energy demand. Furthermore, the resources will constantly grow and there will be an amount of 439.4 billion m^3 by 2030.
- The agricultural sector has the greatest potential, such as crop residues, followed by energy crops from marginal land and animal manure. Animal manure currently used as the main feedstock for biogas production should be co-digested with diversified feedstock, especially with straw.
- As to the highly valuable feedstock of energy crops gained from marginal land, the country is, besides of a vivid bio-ethanol production to be blended with petrol (10% bio-ethanol in many provinces), still at the starting point of doing research in this very promising field. So far, there is neither an agreed definition of the term ‘marginal land’ nor have assessment standards been implemented concerning suitable land resources, energy crop varieties, feedstock availability and related energy potential. Therefore, research in this highly valuable resource should be supported.
- Concerning the future of the biogas potential, the ranking will still remain the same, i.e. crop residues are on the top of the list followed by energy crops and animal manure. Urban waste represents a smaller amount. But it should be mentioned that due to the rapid urbanization, urban waste will increase the most, namely over 12% by 2030. It is not only a great challenge for China to gain energy from this waste but also to cope with these huge amounts of waste in the cities in terms of environmental pollution. Therefore, municipal solid waste management needs to be enhanced.
- Biomass resources offer a realistic chance to ensure energy security and sustainable social and economic development. However, the resources are still not used in a sustainable and efficient way and instead, they are wasted. High quantities (35-40%) of animal manure are untreated and huge amounts of crop residues are still burnt on open fields, which leads to pollution (Liu et al., 2007). A more environmentally-friendly management in this sector would also be required.
- Although the country is rich in biogas feedstock, the current and future forecasts concerning the availability of biomass not only mean that the resources are far from being fully exploited but they also show that the policy targets to develop sustainable biogas energy are not ambitious enough; 6.6% of it was used by 2010 and 10% will be used for 2020.
- Notwithstanding this, an enormous increase in both biogas production and utilization has been recognized during the last years. From 2007 to 2009, the numbers of plants was increased by 53%. However, only three of them have been connected to the electrical supply network so far and only seven units are so-called CDM-Projects, whereby only two can generate some CDM carbon credits based on certified emission reduction (CER).
- In spite of the discernible trend of a further increase in biogas energy production in the future, the necessary know-how and technology concerning design, construction and performance of the plant as well as pre-treatment of the feedstock and maintaining of the whole process are often still missing. The limited capacity of technical service for project operation creates a bottleneck for the rapid development of biogas projects at present. It is, therefore, important not only to enhance the utilization of biogas feedstock and to extend energy supply but also to encourage the competent authorities to invest into maintenance and control of the process of existing plants. A further step would be promoting the industrialization of biogas energy development.

- Besides methane, which is used to generate energy from the biogas, the second product, the digestate, is also of great potential. By treating the whole feedstock, the generated amount would exceed China's demand for bio-fertilizer. Therefore, not only money could be saved but also a huge amount of GHG emissions could be avoided by replacing inorganic fertilizer.
- The study estimates an amount of 4.75 billion tons of CO_2 equivalents, which could be saved by using anaerobic digestion as the main treatment option and it could rise to nearly 7 billion tons until the year 2030. Energy could be produced at lower costs - provided low-priced carbon is sold on the world market - which would entail an increase in the bio-energy development. (Li et al., 2009)

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Session 16

(28) COMBINAISON OF THREE NEW MICROBIAL INDICATORS TO MONITOR COMPOSTING BIOAEROSOLS

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EXECUTIVE SUMMARY

Bioaerosols generated at composting plants are released during processes that involve vigorous movement of material: fresh waste delivery, shredding, compost pile turning, screening. Bioaerosols are a cause of concern because of their potential impact on the health of workers and residents living close to such facilities. The main goal of this research was to look for phylotypes which would represent a microbial signature of compost aerosols. These phylotypes could then be used as microbial indicators to evaluate bioaerosol dispersal around composting plants.

The microbial diversity of bioaerosols collected at five open industrial composting plants treating different types of waste was characterized by a culture-independent approach. Bioaerosols were collected during the turning of compost piles in thermophilic phase. Bacteria and Eukaryota rDNA libraries were built for each of the five bioaerosols samples and more than 800 sequences were analysed. Core species of the composting bioaerosols were identified. Among them, three phylotypes, affiliated to *Saccharopolyspora rectivirgula*, to a *Thermoactinomycetaceae* sp. and to *Thermomyces lanuginosus,* were validated as indicators of compost aerosols in the air collected during piles turning. They were then quantified by qPCR in air collected outdoor in natural environments and on ten composting plants together with 4 other microbial markers: cultivable bacteria and fungi, total bacteria (epifluorescence microscopy) and viable bacteria (solid-phase cytometry). The increase of concentration due to the turning of composting piles compared to the background concentration obtained in natural environments was determined for each marker. Based on these results, a combination of three microbial indicators relevant to monitor composting bioaerosols was defined: one general indicator of bioaerosol emission, the viable bacteria, and two bacterial phylotypes specific to composting bioaerosol, NA07, affiliated to *Saccharopolyspora* sp. and NC38, affiliated to the *Thermoactinomycetaceae*.

In order to study bioaerosols dispersal, these three indicators were then applied on site (11 plants) at increasing distances downwind during the turning of compost windrows in thermophilic phase. The specificity of the indicators is at present further investigated, as well as their relevancy for monitoring bioaerosols emitted during other activities (shredding, screening…).

In conclusion, defining the microbial signature of composting aerosols gave us access to indicators that could be used for analyzing bioaerosol dispersal outside composting platforms.

1 INTRODUCTION

Bioaerosols are emitted at composting plants during such operational activities shredding, compost pile turning or compost screening. At open-air composting plants, bioaerosols are released directly into the surroundings without any pretreatment such as biofilters or bioscrubbers (Sanchez-Monedero et al., 2005). Bioaerosols are a cause of concern because of their potential impact on the health of workers and residents living in close proximity (Herr et al., 2003; Bünger et al., 2007). Assessing exposure relies on estimating downwind concentrations at a distance from the plant and comparing them with data measured upwind or at background locations (Taha et al., 2006). However, analyzing the dispersal of bioaerosols emitted by composting platforms is not an easy task due to the fact that, notably no indicators specific to composting bioaerosols have been defined. It is difficult therefore to consider that measurements taken at a distance from composting activity relate to this activity as opposed to other non-compost sources of bioaerosols (Taha et al., 2005). Previous studies on composting bioaerosol quantified broad microbial groups (bacteria, fungi) or microbial groups associated with the compost biotope but which are also widespread in many of other environments (actinomycetes, *Aspergillus fumigatus*) (Albrecht et al., 2007, Grisoli et al., 2009, Hryhorczuk et al., 2001, Kämpfer et al., 2002, Millner et al., 1980, Sánchez-Monedero and Stentiford, 2003, Taha et al., 2006). Furthermore, these microorganisms were quantified using cultural methods, which are known to underestimate the number of microorganisms present in air (Peccia and Hernandez, 2006; Albrecht et al., 2007).

The aim of this study was: firstly to determine core species in bioaerosols emitted during the mechanical turning of compost piles in thermophilic phase; secondly, to design and to valid qPCR indicators based on these core species; finally, to use a relevant set of indicators (molecular and non-molecular) to analyze the dispersal of bioaerosols into the surrounding area of composting plants.

2 CORE SPECIES IN COMPOSTING BIOAEROSOLS

The five bioaerosols studied were collected from five different composting platforms treating different types of waste (Le Goff et al., 2010). The comparison of the five bioaerosols showed similarities at the phyla level, with *Ascomycota* largely dominant for fungi and *Firmicutes* and *Actinobacteria* for bacteria (fig 1). Diversity indices were also in the same range, high for bacteria, lower for fungi. Furthermore, some bacterial and fungal species were recovered in several or even in all bioaerosols and belonged to the dominant components of the bioaerosol. At the genus level, big differences in the distribution of the dominant species were observed from one site to another. However, several phylotypes common to at least four sites were identified. Only one phylotype was present in the five bioaerosols; it represented 7% of total sequences and had 97% similarity with *Saccharopolyspora rectivirgula*. The six phylotypes found in four libraries were close to *Planifilum yunnanesis*, *Geobacillus thermodenitrificans*, *Thermactinomyces intermedius*, a *Thermoactinomycetaceae sp.*, *Thermobifida fusca* and *Saccharomonospora glauca*. Fungi common to different bioaerosols could also been identified: three phylotypes were common to two sites, two phylotypes to four sites and only one phylotype was present in the five libraries. It was affiliated to *Thermomyces lanuginosus*, gathered 49% of fungal sequences and was differently represented in the five bioaerosols. The two phylotypes common to four libraries were close to a *Penicillium sp.* (11% of fungal sequences) and to *Aspergillus fumigatus* (11% of fungal sequences).

3 EXPERIMENTAL VALIDATION OF THE MICROBIAL INDICATORS OF COMPOSTING BIOAEROSOLS

Three phylotypes were selected as potential indicators of composting bioaerosols among core species identified in aerosols (Le Goff et al, 2011). The two bacterial phylotypes selected,‘NA07’ and ‘NC38’, belonged respectively to the genus *Saccharopolyspora*, and to the *Thermoactinomycetaceae*. The fungal phylotype, ‘EQ05’, was related to *Thermomyces lanuginosus*. The criterion used for the validation was that the concentration in air of the indicator tested should increase during bioaerosol emission on composting platforms. In a first step, each potential indicator was quantified by qPCR in outdoor air samples collected in various natural environments (marine coasts, grassland, moor, forest, private gardens in suburb areas…) not impacted by an industrial composting activity. Then, these background concentrations were compared to those obtained in air samples collected on composting platforms during the turning of composting piles, one operational activity known as emitting bioaerosols. Figure 2 presents results for NA07 (*Saccharopolyspora*).

Air sampling on composting platforms was performed at two locations: few meters downwind of a composting pile turning, and at an upwind point located outside the composting plant and used as a control. During the turning activity, all three phylotypes were quantified. The concentration ranges for ‘NA07’ and ‘NC38’ were $2x10^6$ to $4x10^7$ copies of 16S rDNA.m^{-3} and $9x10^3$ to $2x10^6$ copies of 16S rDNA.m^{-3} respectively. The concentration of the ‘EQ05’ varied from $5x10^6$ to $4x10^8$ copies of 18S rDNA.m^{-3}. For the two bacterial indicators, the concentrations observed in the control samples collected upwind were in the range of those obtained in natural environments, and were included between $5x10^2$ and $5x10^3$ copies of 16S rDNA.m^{-3} (Fig 2). The ‘EQ05’phylotype was not detected in the upwind samples. Overall, for the three phylotypes, concentrations measured outside the composting plants in the upwind samples were always lower than concentrations measured downwind during the turning activities. An increase of 2 to 4 orders of magnitude (3,2 logarithmic units in average) was observed between the upwind and the downwind samples for ‘NA07’. A lower increase was observed for ‘NC38’, compared to ‘NA07’: 2,7 orders of magnitude in average. This significant increase of the concentrations indicates that the turning activity on the composting plants induced an important emission of the three microbial phylotypes in air. The concentrations measured downwind the turning activity on composting platforms were also systematically above those observed in natural environments. Overall, the compost turning on industrial composting platforms induced an increase of the concentration of the three phylotypes of at least two orders of magnitude, compared to the concentration measured in control samples collected upwind, and of at least one order of magnitude compared to the background concentration measured in natural environments not impacted by industrial activities. However, for ‘EQ05’ indicator the difference between upwind and natural environment concentrations was considered as too low. By this way, ‘EQ05’ was not used for further analysis.

4 MEASUREMENT OF DISPERSION AROUND COMPOSTING SITE

The dispersal of composting aerosols emitted during the turning of composting piles were evaluated using an association of three measurements : the viable bacteria, ‘NA07’ (*Saccharopolyspora* sp.) and ‘NC38’ (*Thermoactinomycetaceae* sp.) from 11 different composting plants. Each value corresponds to the difference between the measured concentration and the average background concentration. Results presented on figure 3 are gathered in box plots with 4 ranges of distances: 0-50m (n=18), 50-100m (n=9), 100-200m (n=9) and 200-550m (n=4). Gathering values obtained with different meteorological conditions and in different contexts of bioaerosol emission (type of waste, technology used for the turning…) produced variability in the set of data. Nevertheless, a decrease in the concentration with the greater distance was observed for the three indicators. At distances be far 100m, the impact of bioaerosol emission in concentration of microorganisms was demonstrated in all cases, with an average increase of the concentrations above 1.5 Log_{10}. At distances between 100 and 200m, the impact was still systematically observed for NA07 and the viable bacteria (increase above 1 Log_{10} in average), whereas for NC38, the concentration were close to the background level in some cases. At greater distances, discrepancies between the three indicators increased (impact observed for the four samples analyzed for NA07, frequent impact for the viable bacteria, concentration like to the background for NC38). This analysis of three indicators in parallel showed that if they gave the same trend at low distances (below 100m), their dispersal at greater distances did not seem to follow the same lines.

5 CONCLUSION

The comparison of five different bioaerosols has shown variation from one composting site to another in phylotype distribution but also close similarities: the dominance of *Ascomycota*, *Firmicutes* and *Actinobacteria* in all bioaerosols; bacterial and fungal phylotypes present in all or in most bioaerosols. Two specific microbial indicators, one affiliated to *Saccharopolyspora rectivirgula*, and one belonging to the *Thermoactinomycetaceae*, were demonstrated as able to sign composting bioaerosols emitted during turning activities on composting plants. These indicators could be particularly relevant for air dispersal studies and to reveal an exposure to composting bioaerosols.

A general indicator of bioaerosol emission, the viable bacteria, was added with the monitoring by qPCR of two bacterial specific indicators for analyzing the dispersal of composting bioaerosols during their mechanical turning on 11 different composting plants.

6 ACKNOWLEDGMENTS

We are grateful to Veolia Environment and to the employees at the different composting sites for their help during the sampling campaigns. This work was supported financially by the Languedoc-Roussillon Regional Government Council (France) and the French Agency for Environmental and Energy Management (ADEME) (ADEME, Contract no. 0375C0033).

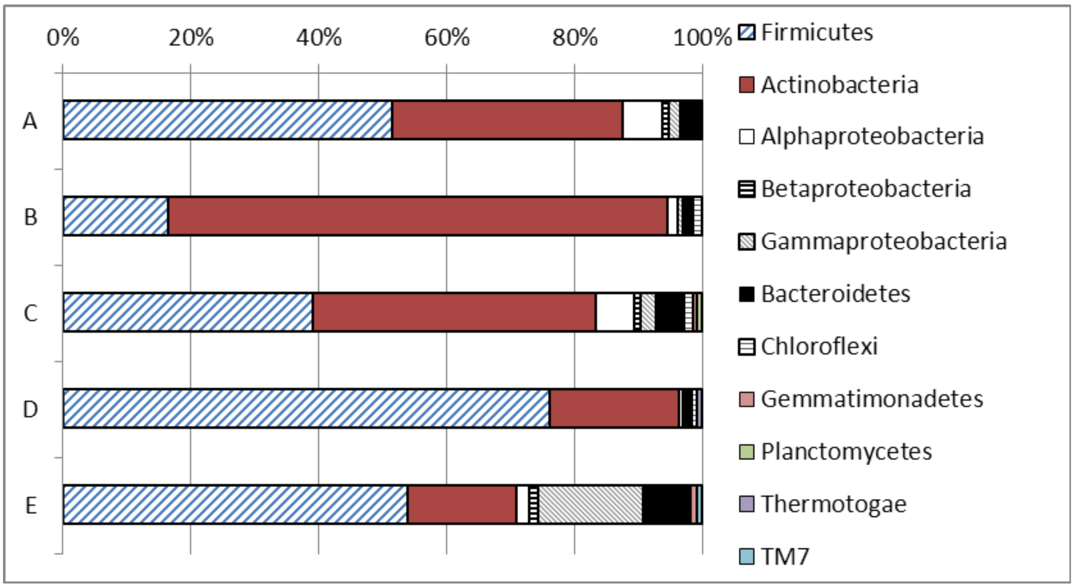

FIGURE 1: percentage of different phyla within bioaerosols collected within different composting platforms treating different types of waste (A to E)

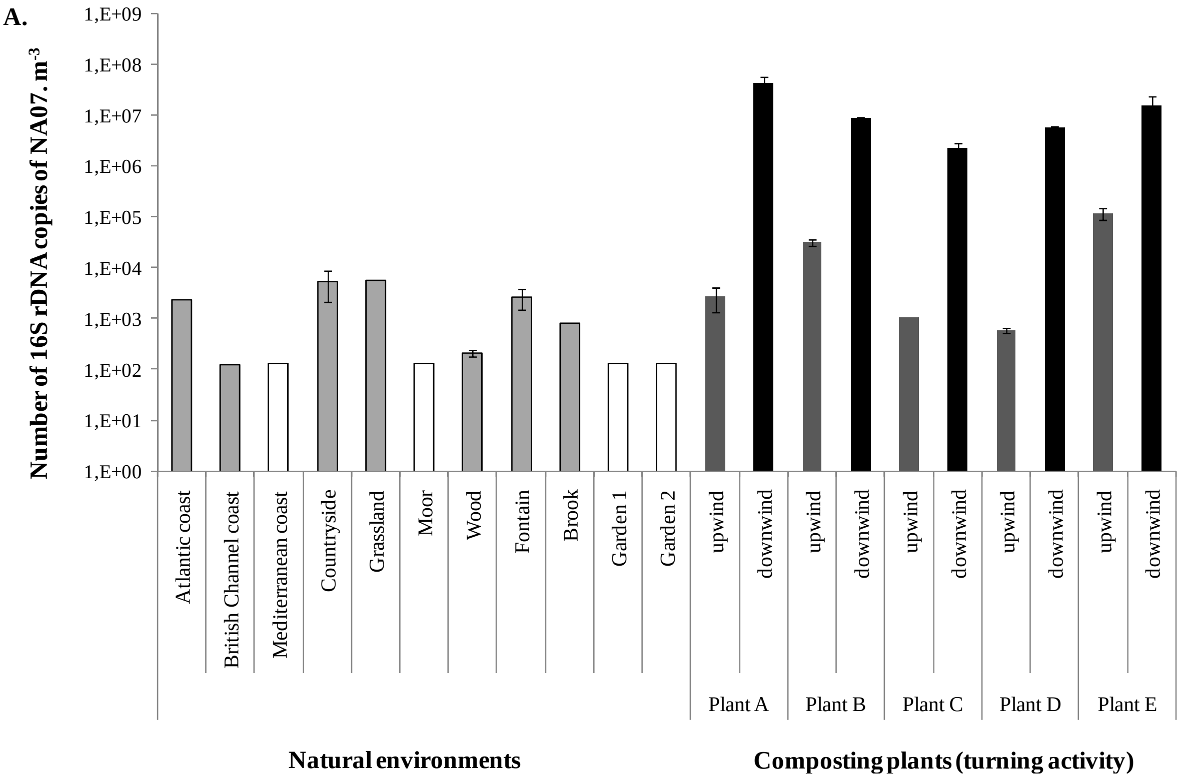

FIGURE 2: Concentration of NA07 in air samples collected in natural environments and on five composting platforms during the turning of composting piles in thermophilic phase (upwind and downwind). When the phylotype was not detected, the quantification limit was indicated using white bars. Arithmetic means and standard deviation were calculated from qPCR replicates.

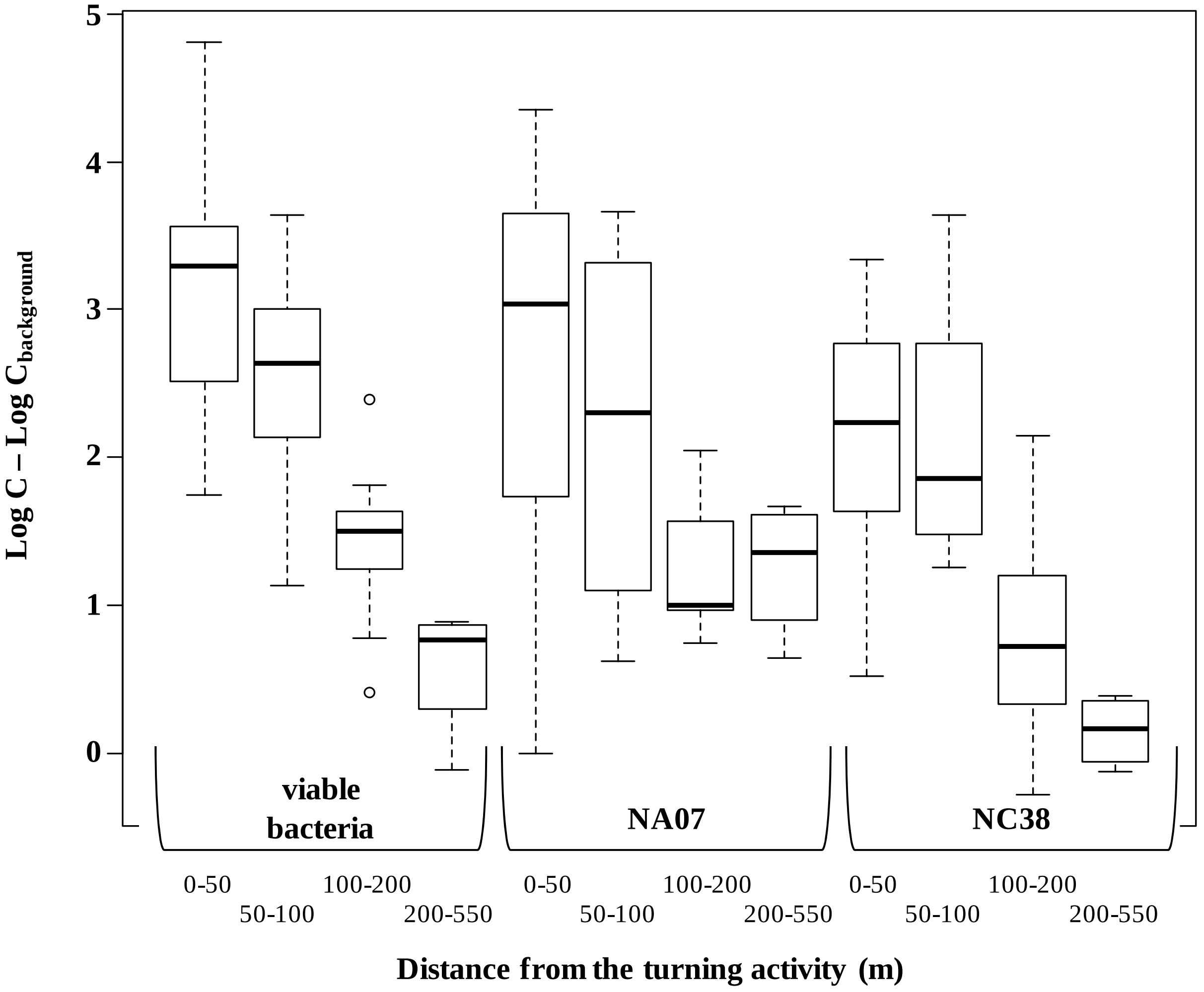

FIGURE 3: Increase in the concentration (Log_{10} units) compared to background concentrations in air of viable bacteria, of NA07 phylotype (*Saccharopolyspora* sp.) and of NC38 phylotype (*Thermoactinomycetaceae* sp.) at various distances downwind from an undergoing turning activity on an industrial plant (11 composting plants)

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(101) ASSESSING THE TEMPERATURE-CONTACT TIME CRITERIA AND TURNING EFFECT IN COMPOST SANITATION

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EXECUTIVE SUMMARY

For a finished compost to be safe for market, it should have reached the necessary sanitation conditions. This means that the number of known pathogenic microorganisms in compost should comply with the levels specified in the national and provincial guidelines. North American guidelines, such as those published by the USEPA in the United States and the CCME in Canada, specify the levels of some pathogenic and indicator microorganisms as <3 MPN·4 g^{-1} dry solids for *Salmonella*, <1000 MPN·g^{-1} dry solids for fecal coliforms and < 1 PFU·4 g^{-1} dry solids for enteric viruses. These levels are generally assumed to be achieved by operating the composting process such that every particle is exposed to a temperature of 55°C or higher for at least 3 consecutive days. For in-vessel and static pile systems, this means that the whole pile should be maintained at or above 55°C for at least 3 days, while in turned windrows the high-temperature period should last for at least 15 days with the pile being turned at least 5 times during this period. A review of the literature illustrated that, regardless of maintaining high temperatures and frequently turning piles, there may still be pathogenic microorganisms, which survive composting. This survival may be linked to spatial and temporal temperature variation in large composting piles, or it could be due to the fact that recommended temperature-contact time conditions are not adequate (the contact time is shorter or the target temperature lower than it actually needs to be for industrial scale operations).

The overall objective of the current study was to assess the efficacy of existing sanitation requirements imposed by Federal and Provincial regulators. Specific objectives were to investigate: 1) the likelihood that a random compost particle would actually meet the required temperature (≥55°C) for three consecutive days in a full-scale operation; and 2) how effective the time-temperature requirement actually is in reducing the number of pathogenic microorganisms to compliance levels. To the knowledge of the authors, no similar study aimed at validating the time-temperature criteria from a random particle perspective has been undertaken to-date.

To satisfy the objectives of this study, a temperature probe was developed consisting of temperature recording circuit and a compartment for a cryovial (to hold microorganisms) enclosed in a cylindrical casing made of anodized 6061 grade aluminium. Two field trials confirmed that the temperature probe behaved like a random particle in compost. Twenty-four temperature probes were used to assess the compliance of an aerated static pile with national regulations and to correlate observed temperature patterns with inactivation of pathogens. Seventeen temperature probes containing a mixture of *Salmonella meleagridis*, *Escherichia coli* K12, and phi-S1 bacteriophage (all at levels of ~1×10^6 CFU (or PFU)·ml^{-1}) were randomly introduced into a covered aerated static pile, along with seven probes monitoring temperature only. After 8 weeks of composting, with one pile turning, they were recovered. Organism levels were determined via culturing methods, and half of the organism mixture from each probe was stored for future molecular analysis.

Before pile turning, 80% of probes satisfied the time-temperature criteria. After turning, this number increased to 87%, demonstrating that turning was useful for sanitation. One of the probes with cryovials reached only 42°C, and survival of *S. meleagridis* (2.5×10^6 CFU·ml^{-1}) was observed. The remaining probes with cryovials exceeded 55°C and were pathogen free. It appears that the specified time-temperature conditions are likely adequate. However, more observations are needed before a firm conclusion can be made.

1 INTRODUCTION

1.1 Background

Composting is the degradation of organic matter by microorganisms. The indigenous microorganisms found in compost feedstocks transform biodegradable organic materials into a biologically stable state, and the stable product can be used as a soil conditioner (Haug, 1993). The raw feedstock for composting oftentimes can be contaminated with pathogenic microorganisms and, therefore, in order to ensure that the finished compost product is hygiene, direct enumeration of pathogenic and indicator microorganisms is typically carried out. In addition, it must be verified that minimum process criteria (time-temperature) have been met (NRC, 2002; USEPA, 2003). Indicator organisms typically used to measure treatment efficiency include: 1) helminth ova and enteric viruses, due to their hardiness and resistance (NRC, 2002); and 2) total and fecal coliforms and fecal streptococci, as these may be affected by environmental stresses similarly to most bacterial pathogens and therefore can serve as useful indicator of predominantly pathogenic *Escherichia coli*, as well as *Shigella* and *Salmonella* sp. (e.g. Yanko 1988; NRC, 2002). Federal and provincial regulations in North America require that, for unrestricted compost use, the level of fecal coliforms should not exceed 1,000 MPN·g^{-1} dry solids and that of *Salmonella* should be less than 3 MPN·4 g^{-1} dry solids (Ge et al., 2006; BNQ, 2005; CCME, 2005; USEPA, 2003). In terms of process criteria, the North American guidelines require every particle of compost to be exposed to ≥55°C for at least 3 consecutive days. For in-vessel systems and static piles, it is assumed that the preceding requirement can be achieved by ensuring that ≥55°C is maintained for three days throughout the pile, while for windrows thermophilic temperatures should be maintained for at least 15 consecutive days with five pile turnings during that period (BNQ, 2005; CCME, 2005; USEPA, 2003). This combination of process and end-product criteria has become common practice for the past two decades to ensure hygienic compost products are produced (Morales et al., 2005). Similar regulations exist in Europe, Australia, and Asia (Hogg et al., 2002).

It is possible that existing temperature monitoring practices may give the false impression that the time-temperature criterion has been met, whereas in reality it hasn't. A literature review by Wichuk and McCartney (2007) revealed that viable pathogenic organisms have sometimes been detected in compost that appeared to comply with the North American time-temperature requirements. The most commonly cited factors relating to pathogen survival were: (i) failure to detect low temperature zones where microorganisms could have remained intact, which occur as a result of spatial and temporal temperature variability; and/or (ii) the inadequacy of existing time-temperature criteria (Barrena et al. 2009; Brinton Jr. et al. 2009; Inglis et al. 2010; Pourcher et al. 2005; Rao et al. 2007; Wéry et al. 2008; Xu et al. 2009). With existing technological capacity there has, to-date, been no endeavour to confirm that existing composting methods ensure the exposure of every particle to 55°C for 3 consecutive days and to correlate these findings with the survival of key pathogen microorganisms all on a compost particle level. Existing regulations, such as those of the USEPA (2003) and CCME (2005), do not provide guidelines on how to ensure that the contact time-temperature criteria have been actually satisfied in any particular technology.

To cover this gap, Wichuk and McCartney (2008) hypothesized that the best approach to monitor the temperature experience of a random compost particle in compost would be to introduce directly into a process a device with which would: a) be self-contained; b) behave like a random particle during pile agitation and settling; c) be sturdy enough to withstand the physical and chemical stresses of composting; and d) have the capability to record real-time temperature data for an extended period of composting. A commercial temperature data logger (probe) was modified so that it would be better adapted to a composting environment, as per Wichuk and McCartney (2008). The probes had a length of 110 mm, a diameter of 25.4 mm, and a density of 1600 kg·m^{-3} Some probes also had a further modification to include a cryovial holder for pathogen inactivation studies. These probes, as a result of the cryovial compartment, had a length of 141 mm, a diameter of 25.4 mm, and a density of 1630 kg·m^{-3}. Two field trials confirmed that both probes behaved like a compost particle when placed in a full-scale pile, thus providing an opportunity to assess the compliance to time temperature criteria from a random particle perspective.

1.2 Research objectives

The overall objective of the current study was to assess the efficacy of existing sanitation requirements imposed by Federal and Provincial regulators using the proposed temperature probes. Specific objectives were to investigate: 1) the likelihood that a random compost particle would actually meet the required temperature of ≥55°C for three consecutive days in a full-scale operation; and 2) how effective the time-temperature requirement actually is in reducing the number of pathogenic microorganisms to compliance levels. To the knowledge of the authors, no similar study aimed at validating the time-temperature criteria from a random particle perspective has been undertaken to-date.

2 METHODOLOGY

2.1 Compost pile

The study was conducted during the period from July to September 2011 at the Edmonton Waste Management Centre's biosolids composting facility, which uses covered aerated static pile technology. The experimental pile had a trapezoidal profile with the following approximate dimensions: a length of 50 m; a height of 3 m; and a base width of 6 m. About 264 tonnes of feedstock material comprised of biosolids mixed with woodchips at a ratio of 50:50 (weight basis) were composted in the experimental pile. Once the pile was built, it was covered with a selective membrane cover to prevent moisture escape from the system.

2.2 Temperature probes

In total, 24 temperature probes, which behave like random compost particles and are designed to withstand conditions within a compost pile, were used in the current study. Seven of these probes were designed to monitor temperature only, and seventeen contained an additional compartment for holding a cryovial, in order that microbial inactivation in response to temperature could be studied. Prior to the experiment, the probes were calibrated according to the manufacturer's instructions. After successful calibration, the devices were batch programmed to read and record temperature data with a 15-minute frequency starting on the day the experiment was slated to begin. Lastly, a 20 m length of fishing line was attached to the end of each probe; this length was greatly in excess of the maximum distance that a probe could be buried in a pile in order to not affect its random placement. The fishing line was used to improve the recovery efficiency of the probes; after placement in the pile, the end of the line was placed outside of the pile. At the end of experiment, the line was used to locate and recover the probes during pile deconstruction.

2.3 Microbial inoculum

Pure cultures of *E. coli* K-12 (ATCC # 10798), *Pseudomonas fluorescens* (ATCC # 13525), and *Pseudomonas fluorescens* phage phi-s1 (ATCC # 27663-B1) were obtained from Cedarlane. A strain of *Salmonella meleagridis*, previously isolated from compost materials, was supplied by the Provincial Laboratory of Public Health (ProvLab, Alberta, Canada). Stock solutions of these microorganisms were prepared 3 days before the experiment. The final solution ("inoculum") was prepared one day before the experiment start date, after the concentration of microorganisms in the stock solutions was defined. Calculations were done to determine the volume of each stock solution required to obtain a final solution containing a mixture of organisms in the following concentrations: *E. coli* K-12 at 10^6 colony forming units (CFU)$\cdot$ml^{-1}; *S. meleagridis* at 10^6 CFU$\cdot$ml^{-1}; and phi-S1 phage at 10^6 plaque-forming units (PFU)$\cdot$ml^{-1}. One ml of the inoculum was then transferred into each cryovial, all of which were sealed and stored at 4°C until being inserted into the temperature probes.

2.4 Experiment setup

The night before the experiment, 17 cryovials were inserted into the 17 temperature probes containing cryovial compartments. The cryovial compartment of each probe was sealed to prevent any leakage into the environment. Two additional cryovials were designated as controls – one was stored at 4°C and another at room temperature (~22°C). All 24 probes (17 with cryovials and 7 without cryovials) were transferred to the composting facility and introduced into the experimental compost pile. All probes were introduced randomly into the pile: at randomly selected times, they were thrown onto the discharge belt of the mixing truck used to construct the pile. The composting process involved two back-to-back stages. In stage 1, the pile was covered and aerated periodically for 30 days. After stage 1, the cover was removed from the pile and the entire mass turned with a front-end loader. Then the pile was covered again and left to compost for another 26 days (stage 2).

At the end of stage 2 composting, the temperature probes were recovered, as the pile was being moved to the curing site, by pulling the end of the fishing line. The fishing line attached to some of the probes was broken during pile building or turning; these probes were recovered from the compost by screening them out with a trommel screen. The recovered probes were brought to the laboratory for microbial survival and temperature analysis.

2.5 Determination of microbial survival

Analysis of microbial viability after exposure to elevated temperatures was begun within 24 hours after the probes were removed from the compost pile. When not in use, samples were stored either in a 4°C fridge or on ice on the laboratory bench. All serial dilutions were done in triplicates. *Pseudomonas* phi-s1 phage viability was determined using a soft Pseudomonas agar overlay plaque forming dilution assay. *E. coli* concentrations in the cryovials were determined using Colilert® in the QuantiTray™ format and lysine deoxycholate (XLD) agar selective media.

For quality control, some QuantiTrays™ were also inoculated with *S. meleagridis* to ensure that this organism would not show positive results that may interfere with *E. coli* enumeration. *S. meleagridis* was detected and quantified by culture methods using XLD agar.

2.6 Temperature data analysis

The temperature records from each probe were downloaded using the manufacturer's software and then imported into a spreadsheet. The data corresponding to the time before the compost pile was covered and after the cover was removed after stage 2 composting were deleted from the dataset. Graphical analysis was done using the graphical software SigmaPlot 12.2.

3 RESULTS AND DISCUSSION

3.1 Temperature data analysis

The data from randomly introduced probes showed that during stage 1 the temperature within the compost pile started to rise immediately. As can be seen in Figure 1, the rising pattern was very steep. The mean temperature was in the thermophilic range (>45°C) until the end of stage 1. The maximum temperature observed was 79°C, and it was recorded on the 2nd day of composting. As the temperature in the pile rapidly increased, so did its spatial gradient. Large differences between recorded maximum and minimum at any particular time started to be observed on day 2, and peaked at 64°C on day 3. Thereafter, the spatial temperature gradient varied between 20 and 40°C.

The end of stage 1/beginning of stage 2 was characterized by an obvious transition of the pile from the thermophilic to mesophilic domain due to pile turning. When the pile was rebuilt after turning, the temperature started to rise again. The mean temperature profile throughout stage 2 was lower than that in stage 1, whereas the temperature fluctuation was higher. As Figures 1 demonstrates, the mean temperature profile was around 45°C for much of stage 2, which corresponds with the lower thermophilic boundary. The maximum temperature detected during stage 2 composting was 60°C.

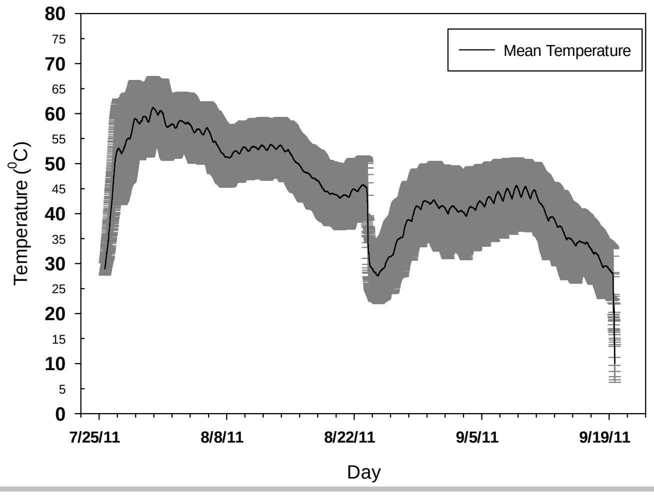

FIGURE 1 **The temperature profile of randomly introduced temperature probes. The black line in the centre indicates the mean temperature at that particular time, while the gray area around the mean temperature shows the 99% confidence interval of temperature distribution from all probes at that particular time.**

3.2 Temperature contact time criteria analysis

The novelty of the temperature probes used in this study lies in their ability to mimic random compost particles (Wichuk and McCartney, 2008). Moreover, if the entire compost pile is assumed to be a population and each compost particle a member comprising that population, then randomly introduced probes should represent randomly sampled particles of compost. Thus, the conditions monitored by the probes should shed light on how long a random particle would be likely to experience thermophilic conditions. Approximately 58% of the probes within the pile reached 55°C during the first 3 days of composting.

By the end of stage 1, the temperature-contact time criteria was met in 80% of the introduced temperature probes. When 55°C or higher temperatures were reached within the pile, they generally persisted for 4 to 20 consecutive days. The remaining 20% of probes did not reach 55°C in stage 1. Based on these numbers, it can be stated that in the experimental pile, given the composting conditions, the likelihood for any random particle to be exposed to 55°C for three consecutive days was 80%. One of the objectives of pile turning is to increase the chances for every particle to comply with the temperature-contact time criteria. In fact, during stage 2, two additional randomly introduced probes met the temperature-contact time criteria. Thus, after stage 2 the probability that every compost particle would comply with the process criteria was increased to 87%.

The finding that some particles did not achieve the necessary temperature-contact time was not a significant concern for this particular facility. After composting in the covered aerated static piles, the material is cured in mass beds for approximately 5 months more (stage 3). Thermophilic temperatures are maintained within the mass beds for much of this time, and the beds are turned on an approximately monthly basis (Wichuk et al., 2011). These conditions should be sufficient to eradicate most known pathogens of concern. It should also be noted that the mass bed material is tested for pathogen indicator organisms prior to being accepted as finished compost.

3.3 Microbiological analysis

3.3.1 Survival of Salmonella meleagridis strain

According to the culture-based method, after composting *S. meleagridis* was alive and culturable in one cryovial at a concentration of 2.5×10^6 CFU·ml^{-1}, which was twice its initial concentration. In order to confirm that this was not an experimental error, the culture test was repeated and the finding was confirmed. *S. meleagridis* is a strain typical to birds with body temperatures around 38-40°C. The temperature profile of the probe in which *Salmonella* survived is shown in Figure 2. It can be seen that the maximum temperature reached in this probe was 42°C, which is near optimum for the proliferation of *S. meleagridis*. This observation supports the results showing that the concentration of *S. meleagridis* increased during composting in this probe. Culturable *Salmonella* was absent from the remaining cryovials.

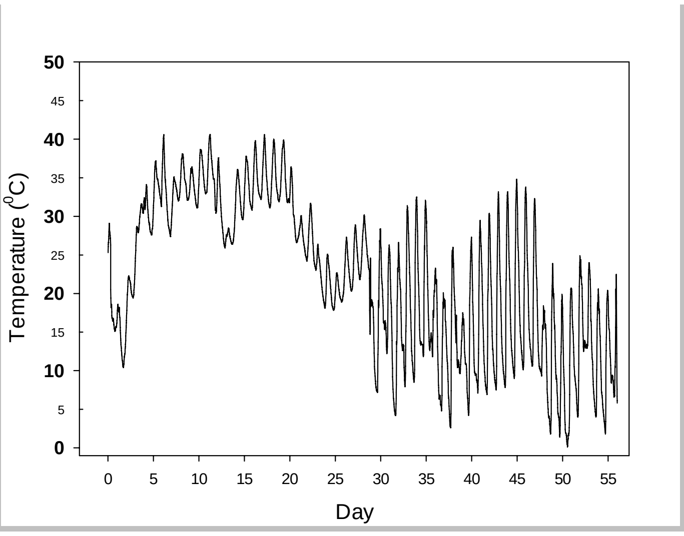

FIGURE 2 **The temperature profile of a temperature probe in which *S. meleagridis* survived**

3.3.2 Survival of Pseudomonas phage phi-s1

There were no plaque formations in any cryovial samples following aerated static pile composting. However, it was not clear if the compost temperature affected the concentration of phi-s1 phage or if natural die-off occurred, as the concentration of phi-s1 phage also significantly decreased in both controls. At the end of the study, the concentration of phi-s1 phage in the control stored at the room temperature was 2×10^3 PFU ·ml^{-1}, compared to the starting value of 1×10^6 PFU·ml^{-1}. The concentration in the vial stored at 4°C was 3.5×10^4 PFU·ml^{-1}. It was concluded that the lack of resilience to environmental changes in Pseudomonas phi-s1 phage makes it a poor environmental indicator.

3.3.3 Survival of E. coli strain

According to the results from the high QuantiTray™ and XLD plates, the temperatures reached during composting were sufficient to inactivate *E. coli* K-12 in all cryovials. This was a surprising result because it was expected that the cryovial containing *S. meleagridis* would also contain viable and culturable *E. coli* K-12 cells. The survival characteristics of *E. coli* K-12 and *S. meleagridis* in the two controls stored at 22°C and 4°C provided some insight into this unexpected result. At 22°C, the concentration of culturable Salmonella rose to 4×10^6 CFU·ml^{-1} from the initial stock concentration of 1×10^6 CFU·ml^{-1}, whereas at 4°C the concentration declined to approximately 1.5×10^5 CFU·ml^{-1}. On the other hand, the concentration of *E. coli* K-12 at 4°C exceeded 10^7 CFU·ml^{-1} and at 22°C declined to nearly 5×10^4 CFU·ml^{-1}. Based on this result, it was concluded that when *E. coli* K-12 and *S. meleagridis* are present together in one medium, *E. coli* K-12 outcompetes *S. meleagridis* under psychotropic conditions, while in mesophilic and thermophilic conditions *S. meleagridis* outcompetes *E. coli*. Thus, it is difficult to determine if the absence of *E. coli* K-12 in the cryovial with culturable *Salmonella* was due to the temperature or due to the competition for nutrients with a stronger microbial rival. In future trials, it may be advantageous to include these two organisms in separate cryovials instead of in a mixture and/or to select different strains of *Salmonella* and *E. coli* which will not compete with each other in the inoculum mixture.

4 SUMMARY OF RESULTS AND RECOMMENDATIONS

In this experiment, 24 temperature probes were used to investigate the temperatures within a full-scale covered aerated static pile and to answer two questions: 1) how likely is it that a random compost particle would experience 55°C for at least three consecutive days; and 2) how effective is this temperature-contact time criteria in eradicating pathogenic microorganisms commonly present in compost feedstock. *S. meleagridis*, *E. coli* K-12, and *Pseudomonas* phage phi-s1 were used as microbial surrogates to assess the effectiveness of the temperature-contact time criteria.

Results demonstrated that composting in a covered aerated static pile, which takes place in two back-to-back stages, allowed 87% of particles within the compost pile to experience thermophilic temperatures ≥55°C for at least three consecutive days. It was also found that pile turning increased the likelihood of exposure, and therefore was confirmed as a practical measure to meet temperature-contact time compliance. Since, at the studied facility, several turning events are completed as part of the curing process using a mass bed turner, this (along with indicator organism testing) should be sufficient to ensure that all particles are meeting the temperature-contact time criterion.

With regard to pathogen inactivation, there is still much to be understood about the behaviour of the microorganisms used for the study. Although it was found that all probes meeting the temperature-contact time criteria contained no culturable microorganisms, firm conclusions on the efficiency of the criteria specified in North American guidelines could not be made. Additional tests need to be conducted, and it is in the scope of the current study to conduct molecular tests to gather further information about the fate of the microorganisms in the probes. In addition, it was not clear if the absence of culturable *E. coli* K-12 was due to the temperature or to the presence of *S. meleagridis,* which possibly suppressed its growth. Likewise, it was not explicit why the control inoculum, which was stored at room temperature for 56 days, had a three-log decline in the concentration of phi-s1 phage. These facts must be considered prior to repeating this experiment. For example, it might be recommended that different strains of *E. coli* and/or *Salmonella* be used together as indicator microorganisms and that their relationship be studied prior to carrying out a composting experiment, or that these organisms be placed in separate vials instead of in a mixture. Also, it is recommended that more thermotolerant phages and bacteria strains be selected and that the spectrum of microorganisms be expanded to include parasites in the study.

5 ACKNOWLEDGEMENTS

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(151) COMPOSTING OF PIG FAECES WITH CORN STALKS IN CHINA – MICROBIOLOGICAL EXAMINATIONS; HYGIENIC ASPECTS AND SANITATION CAPACITY

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EXECUTIVE SUMMARY

Background: With the increasing demand for meat (pork) the pig production in China increased over the last years, especially in the Northern East of China around mega cities like Beijing. Increasing pig production in large scale pig farms head to enormous amounts of organic wastes (such as pig faeces), over-fertilization of agricultural areas and environmental pollution in regions with high pig production and density. Pig manure can contain pathogens with risks for animal health, public human health and the environment.

Objectives: Composting is biotechnological treatment and a possibility to produce a fertilizer in consideration of hygienic safety which could be transported to agricultural areas in regions far away from these mega cities. In order to protect soil and plant quality the agronomic efficiency and hygienic safety of such fertilizer must be demonstrated prior to their application on cultivated fields. The challenge was to determine the sanitation capacity of different variants of composting processes, especially under winter conditions in Beijing (China), and to estimate the microbiological risk for human health, animal health and the environment.

Material and methods: Pig manure and corn stalks were used for composting (ratio 1:7) carried out in small rotting boxes ($1m^3$). The six variants of composting differed in turning interval (no turning, turning once and turning twice a week) and cover. Two boxes were the same in turning interval, one covered and one uncovered. The composting trial ran for ten weeks without forced aeration. As test organism heat resistant strain *Salmonella* Senftenberg W775 (H_2S negative) (called “W775”) and as indicator organism faecal streptococci were used for microbiological examinations in the experiment. Mixed input materials were inoculated with a suspension of test organism “W775” and put in sterile sacks. These served as test carriers and were inserted in the rotting boxes in three different positions, representative for parts of the rotting boxes with different risks for hygienic safety. During experiment, qualitative and quantitative examinations were carried out to determine the presence of “W775” and faecal streptococci in control and test samples on day 0, 14, 49 and 70. Data logger were used for temperature measurements on each position of test samples. Temperature, dry matter content and exhaust gas composition were regularly monitored.

Main result, brief discussion and conclusion drawn: The numbers of “W775” in the control and starting sample in the beginning of experiment were 10^7 cfu/g. The “W775” was not detectable after 14, 49 and 70 days of composting in all of the tested samples. In the positive control samples, stored at room temperature, “W775” was still detectable more or less without any decrease by orders of magnitude over the whole composting period. In regard to the sanitation capacity the temperature and other influences of composting procedure at each position in every box seem to be sufficient to inactivate the heat resistant strain “W775” in our test samples in all six variants of composting. In conclusion composting in this way is a suitable treatment for organic wastes to produce a fertilizer in consideration of hygienic safety. These fertilizer can be used to export nutrients from areas with high animal density to other regions, far away, with a minimized microbiological risk.

1 INTRODUCTION

1.1 Background

The use of organic matter (wastes) for sustainable reuse (recycling) of nutrients is a very important issue all over the World, especially in countries with distinct agriculture and livestock production. China developed very fast over the last years with an increasing luxury. Over the last years, the raising demand for meat (pork) head to an increased pig production in China, especially in the Northern East of China around mega cities like Beijing. Increasing pig production in large scale pig farms head to enormous amounts of organic wastes (e.g. pig faeces, manure, slurry), over fertilization of agricultural areas and environmental pollution in regions with high pig production and density. In 2008 pig production worldwide achieve around 100 million tonnes of slaughtered pigs and China is leading with 48,5 million tonnes, followed by European Union with 22 million tonnes and the U.S.A. with 10,45 million tonnes ("Das meiste Schwein wird in China produziert", Agrarheute.com 2009). Intensified pig production around mega cities head to a decoupling from regions with intensive livestock production and areas of agricultural landscapes. Recycling of organic wastes in agriculture after appropriate biological treatment can produce valuable organic matter and be of great interest in countries where soils are depleted (Hassen 2001). A variety of heavy metals and pathogens can be found in raw bio-wastes (Strauch 1996). Animal manure is a well-documented source of zoonotic pathogens, such as Escherichia coli O157:H7, *Salmonella* spp., and *Campylobacter* spp. (Erickson 2009). Pig manure can contain pathogenic micro-organisms creating the risk of spreading animal diseases and zoonotic agents. Pathogens can be spread from farm into and through the environment into the food chain via plants, food of animal origin, and into drinking water and may cause human infections. (Böhm, R., Martens, 2009). Handling and use of organic wastes of animal origin are connected with risks for animal health, public (human) health and risks for the environment. New transmission routes of pathogens between rural and urban areas can be created through the use of organic waste products, it might be occur through aerosols, run-off from arable land to adjacent watercourse, contamination of groundwater, contamination of food and feed as well as because of vector animals such as birds and rodents. (Elving, J. 2009). Sanitation of organic waste before application to soil is recommended because the pathogens may survive for extended periods in the soil environment (Nicholson et al. 2005). Safety use of organic wastes as fertilizer demands a biotechnological treatment which guarantees a sufficient sanitation capacity. The sanitation capacity of composting process and the hygienic safety of the end product should be monitored and validated using measurement of temperature, insertion of test carriers with specific test organism (e.g. heat resistant *Salmonella* Senftenberg W 775) and following microbial examination of test carriers and end products, e.g. in accordance to the German Bio-Waste Ordinance, which is under novelling at the moment.

1.2 Research objectives

Aerobic, thermophilic composting is a common method to treat organic wastes. Composting is a possibility to produce a fertilizer in consideration of hygienic safety which could be transported to agricultural areas in regions far away from these mega cities. In order to protect soil and plant quality the agronomic efficiency and hygienic safety of such fertilizer must be demonstrated prior to their application on cultivated fields. The challenge was to determine the sanitation capacity of different variants (turning interval and covering) of composting processes, especially in cold season in Beijing (China), and to estimate the microbiological risk for human health, animal health and the environment. These hygienic risks should be considered under winter conditions because winter is the season with the highest expected difficulties for a thermal treatment process like composting procedure.

2 METHODOLOGY

2.1 Composition of composting trial

Pig manure and corn stalks (straw chaff) were used for composting in a ratio 1:7 (wet weight / wet weight) and carried out in small rotting Boxes ($1m^3$) as an half technical experiment in a farm building under roof. The six variants of composting differed in turning interval (no turning (Box 1 and 2), turning once (Box 2 and 3) and turning twice (Box 5 and 6) a week) and cover. Two boxes were the same in turning interval, one covered and one uncovered. The composting experiment ran over ten weeks, from end of November 2009 until beginning of February 2010, without forced aeration. The composition of our experiment and the bacteriological investigations were designed in accordance to the German Bio-Waste Ordinance (2011). As test organism *Salmonella* Senftenberg W775 (H_2S negative) (called "W775") and as indicator organism faecal streptococci were used for microbial examinations in the experiment.

"W775" is a thermally stable and not pathogen *Salmonella* strain for human health (Garibaldi 1969). Mixed input materials were inoculated with a suspension of test organism "W775" and put in sterile sacks. These served as test carriers and were prepared immediately before starting the composting experiment by inoculation with test organism. The test carriers were inserted in the rotting boxes in three different positions, representative for parts of the rotting boxes with different risks for hygienic safety (sanitation capacity). The first location was the base (10-20cm over the bottom, in the corner 10-20cm far from the box wall), the centre and the margin of the box (10-20cm under surface of pile, in the corner 10-20cm far from the wall of composting box). In the trial the sampling plan included a sampling after 14, 49 and 70 days. The sampling after 14 and 49 days occurred to see the progress of sanitation after a shortened duration of composting procedure. For this purpose three test samples (for sampling after two, seven and ten weeks) was positioned on each position. During turning of composting boxes the test samples were taken out and inserted again after turning on the same position in the composting boxes. This should guarantee that the conditions are representative for the basis, the centre and the margin over the whole time of experiment.

During the experiment qualitative and quantitative examinations were carried out to determine the presence of "W775" and faecal streptococci in control and test samples on day 0, 14, 49 and 70. Data logger were used for temperature measurements every hour on each position of test samples during the whole time of composting experiment. After termination of composting trial it was possible to compare the results of microbiological examinations of test samples with the temperature process (values and a curve about the development of temperature during the whole composting procedure in every box on similar position). The temperature, dry matter content and exhaust gas composition were regularly monitored.

2.2 Methods and materials

Samples were stored at 4°C until microbial examination in the laboratory. For qualitative detection of Salmonella there was a pre-enrichment of 50g of each sample in 450ml buffered peptone water (with novobiocin). For quantitative detection 20g of each sample were weight in into 180ml of sterile sodium chloride solution (0.9%), shaked and ten fold diluted. For each dilution three parallel tubes with buffered peptone water were inoculated and incubated for 24h (48h) at 37°C. From pre-enrichment 0.1ml was inoculated in selective enrichment broth Rappaport-Vassiliadis (RV-broth) and incubated for 24h at 42°C. After incubation the RV-broth was streaked out onto XLD (xylose-lysin-desoxycholat)- and BPLS (brilliantgreen-phenolred-lactose-saccharose)-agar and incubated for 24h at 37°C. Suspicious colonies were streaked out onto standard-I-agar, incubated for 24h at 37°C, to receive pure colonies. These colonies were confirmed as "W775" by object slide agglutination with Salmonella specific test-sera. The (quantitative count) number of "W775" was quantified using the most probable number-method (mpn-method).

For quantitative detection of faecal streptococci the same ten fold dilution series in sodium chloride solution (0.9%), as described before for detection of *Salmonella*, was used. Each dilution was used for inoculation of three parallel tubes containing acid dextrose broth (AD-broth) and incubated for 48h at 37°C. After incubation, the AD-broth was streaked out onto kanamycin-äsculin-acid-agar (KAA-agar) and incubated for 48h at 37°C. The number for faecal streptococci was quantified using the most probable number-method (mpn-method). Positive colonies appear as grey to black colonies with a black background colour of the KAA-agar.

3. RESULTS AND DISCUSSION

The numbers of "W775" in the control and starting sample in the beginning of experiment were 1.5 – 4.3 x 10^7 cfu/g. The results in Figure 1 show that "W775" was not detectable after 14, 49 and 70 days of composting in all of the tested test samples in three different positions, representative for areas in the composting boxes with different hygienic risks. In the positive control samples, autoclaved and inoculated silica sand stored at room temperature, "W775" was still detectable more or less without any decrease by orders of magnitude over the whole composting period. The numbers of faecal streptococci ranged from 10^5–10^6 cfu/g in the beginning of the experiment. A reduction of faecal streptococci to numbers ranged from 10^0–10^2 cfu/g, but no complete elimination was observed on day 14 of composting (Data are not shown). In regard to the hygienic safety (sanitation capacity) the temperature and other influences of composting procedure seem to be sufficient to inactivate the *Salmonella* Senftenberg W775 (H2S negative) strain at each position in every box in our test samples in all six variants of composting. Temperature is the most important factor to eradicate "W775" and when temperatures in the composting heap are 60°C, test organism "W775" will be eliminated within 10h of composting (Ceustermans 2006). Exemplarily, the temperature profiles for one rotting box (variant turning twice a week, covered) in our composting experiment are shown in figure 2. Temperatures of 55°C and higher were achieved during first week of composting and last for at least one up to three weeks.

They correspond to the recommendations in national or international standards (e.g. German Bio-waste Ordinance). These results should be transferred into full technical composting plant. The results of our experiment show that composting of pig faeces mixed with corn stalks (straw chaff) is a possibility to inactivate pathogenic micro-organism such as Enterobacteriaceae and to reduce the hygienic risk of organic wastes used as fertilizer. The hygienic risk can be minimized and the contamination of the environment can be avoided or reduced. It seems to be possible to produce a fertilizer with high quality in consideration of hygienic aspects by composting of organic wastes using tested variants. These fertilizer could be transported into other regions, to use and export nutrients.

4. CONCLUSION

The enormous amount of organic wastes, such as pig faeces, from intensive animal husbandry and meat production in China need a biotechnological treatment which guarantee the microbiological (hygienic) safety of produced fertilizer. Land application of raw manure also results in contamination of agricultural runoff and water supplies. Contaminated drinking water has the potential to cause extensive outbreaks. Salmonella is relatively persistent in soil compared to other pathogens. (Holley 2003) He noticed that proper composting of manure can yield safe fertilizer and is obviously important for its use on food crops, but may also be important for forage crops to reduce levels of pathogens (E.coli O157:H7) in livestock. The results of our experiments show that composting of pig faeces with corn stalks is a possible biotechnological treatment to recycle organic wastes, in consideration of hygienic safety, and to use and export nutrients to other regions. It should be possible to eliminate the major bacterial pathogens from pig faeces by composting in these variants. Our results for detection of test organism “W775” and the temperature profiles in different positions of rotting boxes show that there is a elimination of pathogens and therefore a sanitation by composting. Temperatures in rotting boxes achieve high values (over 55°C) for a duration of 1 to 3 weeks and correspond to temperatures recommended in national or international standards, like the German Bio-waste Ordinance (2011). Our results need to be transferred into full technical composting plant. Our study demonstrates that all six variants of composting processes tested in our experiments were sufficient to eliminate “W775” providing temperatures as recommended. It seems to be a suitable treatment for organic wastes in order to produce a fertilizer with good hygienic quality and to minimize the risk for contamination of environment, therefore for safe feed, food and animal production. These fertilizer can be used to export nutrients from areas with high animal density to other regions, far away, with a minimized microbiological risk. The production of safe fertilizer is the basis (fundament) to avoid contamination of soil and groundwater with pathogenic micro-organism, especially zoonotic bacteria such as Salmonella. This is the precondition for safe forage and for avoidance of input pathways of pathogens into the food chain. Therefore it is part of animal and public health. Another focus should be on the avoidance of contamination of water and groundwater. Water resources are very limited worldwide and it is very important to protect these water resources, for the moment and especially for the future. In conclusion we notice that treatment of organic wastes (e.g. pig faeces) in consideration of hygienic aspects is very important for the recycling and use of nutrients and especially for the protection of soil and water (groundwater).

FIGURE 1: Detection of test organism Salmonella Senftenberg W775 (H2S negative) “W775” in control and test samples on day 0, 14, 42 and 70

Values < 0,1 cfu / g:	Detection limit - no detection of test organism “W775”
Values of 1 cfu / g:	Qualitativ detection of “W775” / no quantitative detection

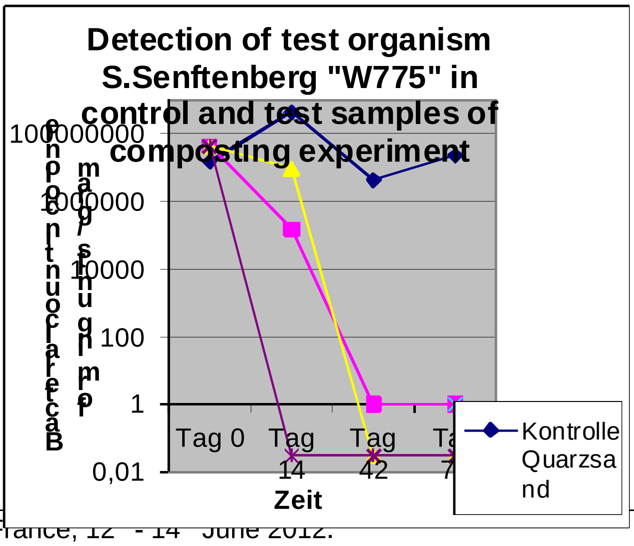
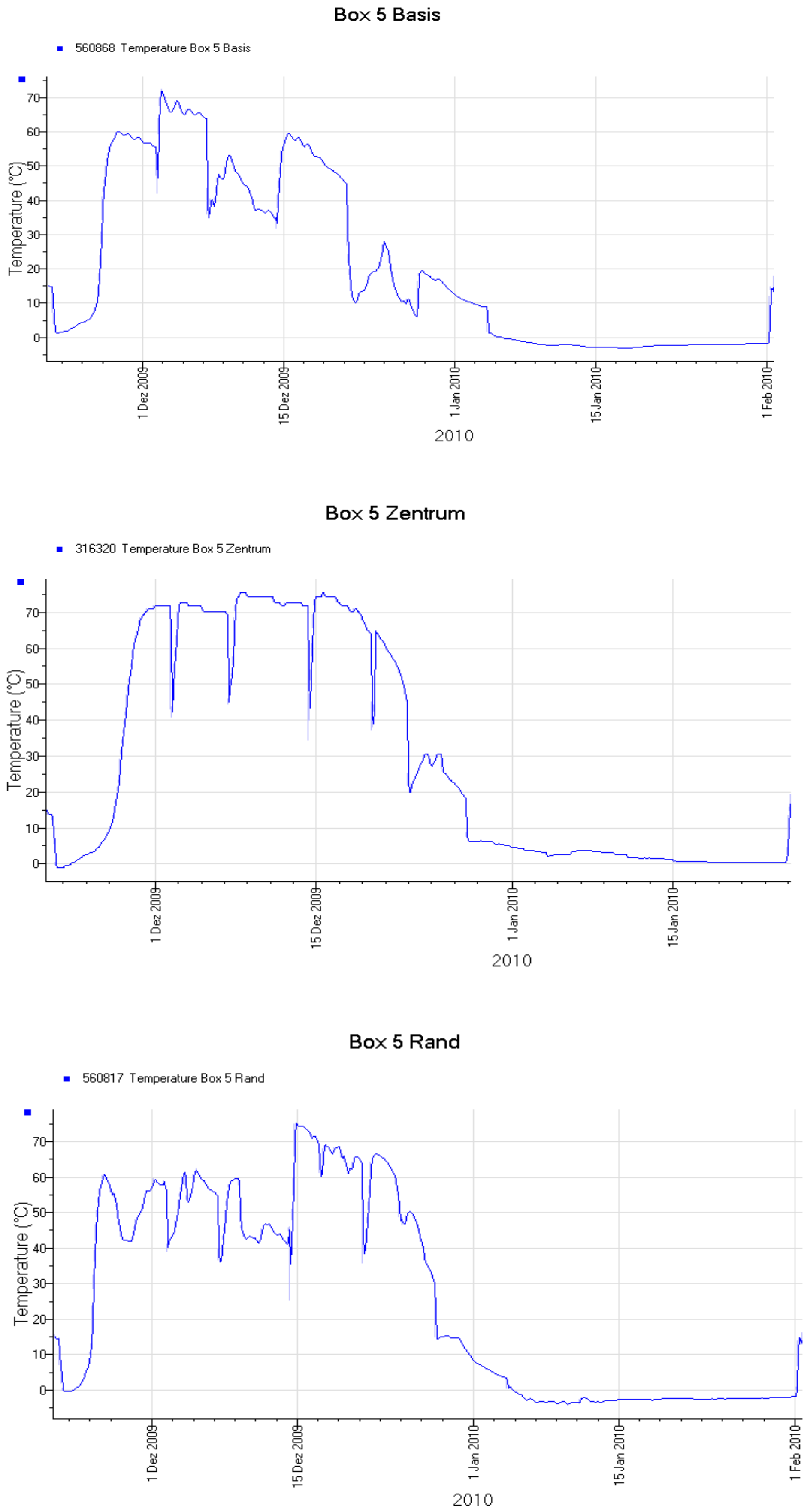


FIGURE 2: Temperature profiles composting box 5 (turning twice, covered)



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(164) ISOLATION AND SCREENING OF THERMOPHILIC AND PSYCHOTROPHS BACTERIAL STRAINS USED IN THE COMPOSTING PROCESS

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EXECUTIVE SUMMARY

Psychrotrophs and thermophilic are two categories of microbes who can survive in extreme low and high temperature environments. Theoretically, the introduction of such micro-organisms can increase the initial temperature at the early mesophilic phase or maintain the high temperature at the thermophilic phase of aerobic composting. And the key is to find the strains growing fast and decomposing efficiently of protein, starch, cellulose and other organic substances in different temperature environments.

10 samples from different organic sources, as sewage water, soil and animal manure were collected in Heilongjiang province, China, and from which we isolated four psychrotrophs bacterial strains, as B6-38，B9-13，B10-40 and B10-44. They all showed the characteristics of fast growing and rapid degrading of organic substances at low temperature (15 ℃). Determined with 16S rDNA gene sequencing and growth characteristics, B6-38 and B10-40 are associated with the genera *Aeromonasa*, B9-13 with the genera *Enterobacter* and B10-44 with the genera *Paenibacillus.* A composting experiment with chicken manure and sawdust was carried out in a small-sized container to study the effects of microbial inoculation（B6-38，B9-13，B10-40 and B10-44, individual strain）on the composting process. The results showed that the rate of temperature growth of the treatment with B6-38 was the highest, 61.79% higher than control, among those 4 treatments.

Meanwhile, two cellulose decomposing thermophilic bacterial strains, HNS39 and HNC41, were screened from 13 thermoplic compost samples (at high temperature phase, about 80°C) from Hebei, Hainan and Shandong provinces, China. Both are associated with the genera *Geobacillus* determined by using 16S rDNA gene sequencing and growth characteristics. The similar composting experiment was done to study the effects of microbial inoculation（HNS39, HNC41 and the mixture）. The results showed that the use of the HNS39 and HNC41 were more effective in terms of the increasing of initial temperature, maintaining of high temperature at thermophilic phase and slowing down of the temperature decline at maturation phase. However, there was no significant difference between the effect of individual strain and their mixture (1:1 v/v).

Those two laboratory studies explained that external introduction of functional microbial strains could give potential to improve the composting process in a biological way and further studies might focus on the exploration of the related mechanisms and the development of effective inoculants used in the composting process.

1 INTRODUCTION

Composting technology is a common treatment method for the selection of recyclable and compostable materials (Deportes, 1995), which can be inoculated with preselected microorganisms (Wang, 2011). Zeng et al. (Zeng, 2009) found that inoculation with lignocellulolytic microorganism during the second fermentation phase improved compost maturity.

Temperature is a limited factor in Compost process. On one hand, in low temperature stage, the rise of composting temperature is the key that decided how quickly compost comes into high temperature phase (Bolta, 2003). On the other hand, high temperatures inhibit microbial growth and slow down the organic matter biodegradation (Diaz, 2007). Cellulose is the most abundant component in organic wastes and its decomposition by cellulose-degrading bacteria is often the limiting step for composting process. Therefore, we attempted to isolate and screen efficient bacteria strains from composts and soils adapting to high and low temperature, respectively.

1.1 Background

Composting is a common treatment method for organic wastes. The researches (He, 2007) have shown that inoculation of psychrotrophs can accelerate composting temperature from low temperature phase to high temperature phase quickly. However, high temperature and lack of oxygen supply can lead to inactivation of cellulose-degrading bacteria, and subsequently a slow biodegradation and a prolonged time in composting. Therefore screening and using of psychrotrophs and thermophilic strains could accelerate the progress of composting theoretically.

1.2 Research objectives

Psychrotrophic bacteria and thermophilic bacteria were screened by separated experiments respectively. These bacteria will be further tested in the composting process.

2 METHODOLOGY

2.1 Psychrotrophic Bacteria

2.1.1 Sample collection

10 samples were collected from sewage sludge, poultry manure and paddy soil from Harbin, Heilongjiang province and Shijiazhuang, Hebei province in spring of 2008.

2.1.2 Screening

10 g of each sample was added into 90 mL aseptic water, shaking 30min (200r min^{-1}). 10 ml supernatant was added into LB liguid medium to proceed temperature-gradient training (5℃~7℃~10℃) and diluted to 10^{-7}. The diluents spread on the solid medium, which were then incubated at 4°C until the isolates formed colonies. Then the colonies were inoculated in the selective medium to observe the size of transparent circle and colony diameter of bacteria.

The liquid medium contained (per liter) peptone 10g, beef extract 5g, NaCl 5g. The solid medium contained (per liter) peptone 10g, beef extract 5g, NaCl 5g, agar 15g. The isolated medium of starch-degrading bacteria contained (per liter) peptone 10g, beef extract 5g, NaCl 5g, soluble starch 2g, agar 15g. The isolated medium of protein-degrading bacteria contained (per liter) peptone 10g, casein 5g, glucose 1g, NaCl 5g, Tyrosine 0.1g, $CaCl_2$ 0.1g, agar 15g, during sterilization adding 1% of Tween-80. The isolated medium of cellulose-degrading bacteria contained (per liter) K_2HPO_4 0.5g, $MgSO_4$ 0.25g, CMC-Na 1.88g, Congo red 0.2g, gelatine 2g, agar 20g.

2.1.3 Enzymes measurement

The DNS method (Ghose, 1987; Wang, 2001) was used to measure the cellulase activity of the bacteria. The Yoo method (Shi, 1996) was used to measure the amylase activity of the bacteria. The Branford method (Wang, 2009) was used to measure the protease activity of the bacteria. The cellulase fermentation medium contained (per liter) NaCl 5g, peptone 10g, yeast extract powder 5g, CMC-Na 5g, KH_2PO_4 1g. The amylase fermentation medium contained (per liter) peptone 10g, beef extract 5g, NaCl 5g, soluble starch 2g. The protease fermentation medium contained (per liter) peptone 15g, yeast cream 3g, NaCl 5g, glucose 5g.

2.2 Thermophilic Bacteria

2.2.1 Sample collection

13 compost samples were taken from composting piles when the composting temperature rose higher than 70℃, with the compost raw materials of sludge, cow muck, chicken manure and sugar refinery residues from Kangdi Eco-fertilizer Co. Ltd., Mckinnon Biological Technology Co. Ltd., Jintudi Organic Fertilizer Co. Ltd., and Yuwo Bio-technology Co. Ltd., respectively. Muck soil was also collected from ditches in the tropical rain forest of the Five-finger Mountain in Hainan Province, and then mixed into one soil sample as bacteria source.

2.2.2 Enrichment and isolation of thermophilic cellulose-degrading bacteria(CDB)

Weigh 5 g sample with 50 ml aseptic water into a triangular flask and shake at 160 r/min for 30 min, then staying at 80 for 3h. 1 ml of the mixture was transferred to Hutchinson enriched medium and incubated at 80°C for 30 days. During incubation, the medium was maintained at 80-100 ml in volume by adding the enriched medium regularly. Hutchinson enriched medium (pH 7.2) contained (per liter): K_2HPO_4 1.0 g, NaCl 0.1 g, $NaNO_3$ 2.5 g, $MgSO_4$ 0.3 g,

$FeCl_3$ 0.01 g, $CaCl_2$ 0.1 g. ml of the above enriched culture solution was transferred to cellulose-congo red medium using the spread plate method and incubation for 2 days.

Large colonies with distinct red circles in the Congo red agar medium were chosen, and the streak plate method was used to isolate pure HCDB strains. Congo red agar medium (natural pH) contained (per liter): K_2HPO_4 0.5 g, $MgSO_4$ 0.25 g, CMC-Na 1.88 g, Congo red 0.2 g, gelatin 2 g, agar powder 15 g, phytagel 10 g, and deionized water 1000 ml.

In order to obtain microorganisms with high cellulose-degrading activities, pure HCDB strains were screened by cellulose hydrolyzation studies, growth mass comparison, FPase activity measuring and filter paper-degrading abilities studies.

(1) Cellulose Hydrolyzation Experiment

Activated strains were inoculated into cellulose-Congo red medium, 3 points each plate. After incubation at 80 for 48 h, colony diameter (d) and circle diameter (D) around which produced were measured, calculated the Up value according to formula: Up $=(D/d)^2$. A higher Up value indicates a higher cellulose-degrading capacity.

(2) Growth Comparison

Activated strains screened above were inoculated into triangular flask (250 ml) with 50 ml broth which contained: 1% peptone, 0.5% beef extract and NaCl. Control with no bacteria inoculated was also set up. After 48 h of incubation at 80 , we compared the turbidity of the culture medium between bacteria-inoculated and control. Higher turbidity in the inoculated ones indicates the strains inoculated can grow well in micro-aerobic environments.

(3) Assay of FPase activity

Activated strains screened from (2) were incubated in broth for 48 hours at 80 to obtain seed culture. The fermenting condition cellulose production was: 80 , no revolution, inoculation rate 5%, 100 ml medium in a 250 ml triangle flask, fermenting time 7 days. Fermentation medium (natural pH) contained (per 1 L deionized water): K_2HPO_4 3 g, $(NH_4)_2SO_4$ 2 g, $MgSO_4 \bullet 7H_2O$ 0.5 g, $FeSO_4 \bullet 7H_2O$ 0.01 g, corn meal 2 g, wheat bran 0.5 g, peptone 5 g, and $CaCO_3$ 0.5 g. During fermentation, certain amount of the fermentation medium was drawn and centrifuged at 5000 r/min for 20 min. The supernatant was used for FPase activity meauring.

Dinitrosalicyclic acid (DNS) method was used to measure cellulase activity. Filter paper cellulase activity in the cell-control was determined using the DNS (3,5-dinitrosalicylic acid) method. One unit (U) of enzyme activity was equal to 1 μg glucose produced per minute. Measurement of cellulase activity was performed in an assay mixture (2 ml) that contained a piece of XinHua filter paper, 1 ml of 100 mM phosphate buffer (pH 7.0), and 1-ml of supernatant. The mixture was incubated at 80 for 30 min for cellulase activity assay and reaction was stopped by adding 3,5-dinitrosalicylic acid (DNSA) solution, boiling for 5 min, and cooling in water for color to stabilize. The optical absorbance was measured at 540 nm and the amount of reducing sugars (glucose equivalents) produced was estimated against a glucose standard curve. Appropriate enzyme and substrate controls were included in all assays.

(4) Filter paper-degrading ability test:

5 mL seed culture of CDB screened from 2.2.2 was inoculated into 100 mL Hutchinson medium in a 250 mL flask sealed with aluminum foil. After 14 d of static incubation at 80 , filter paper strips was washed several times first with a mixture of dilute hydrochloric acid and dilute nitric acid, then with deionized water to remove residual acid, dried to constant weights at 80 , and weighed. Filter paper degradation rate was calculated as follows:

degradation rate (%) = (weight before incubation - weight after incubation) / weight before incubation×100

2.2.3 Identification of CDB

DNA extraction was performed using the classic method (Wilson, 2001). The 16S rDNA was amplified with polymerase chain reaction (PCR) using the two universal primers of 27f (5′-AGAGTTTGATCMTGGCTCTCAG-3′) and 1492r (5′-TACGGTTACCTTGTTACGACTT-3′) (Wang, 2009). PCR was performed with Taq DNA polymerase (TaKaRa, Dalian, China). PCR was run for 30 cycles in a DNA thermal cycler. The following thermal profile was used for the PCR: initial denaturation for 10 min at 94℃, denaturation at 94 ℃ for 40 s, primer annealing at 60 ℃ for 40 s and extension at 72 ℃ for 1.5 min. The final cycle included extension for 10 min at 72 ℃ to ensure full extension of the products. The amplified PCR products were analyzed in 1.0 % (w/v) agarose gel to ensure the product was what we wanted. The obtained 16S rDNA sequences were compared to sequences in nucleotide databases using the nucleotide-nucleotide BLAST program.

2.3 Application in Composting

For the low-temperature degrading bacteria, the chicken manure and the saw dust was used as composting material and the box of foamed plastic was used as composting container. The initial parameters of composting were adjusted to 28 of C/N, 60% of moisture, 5kg of weight.

Then the experiment, including one blank group, two control groups and four treatments, was started in a cold weather about 13 -15 and determined with the temperature changes of the composting pile for each 4 hours. The blank experiment was not introduced microbial inoculums, the two control experiments were respectively added with 2‰ of VT and 2‰ of P1, the other treatment groups were respectively introduced with 2‰ of B6-38, B10-13, B10-40, B10-44.

For thermophilic bacteria, 3 portions of chicken manure and 1 portion of returned compost by the addition of 2% cornmeal and 0.5% wheat bran by weight were mixed, showed in Table 1. After the temperature reached 50 , the static pile was subdivided into four containers. According to Table 2, 7.5% (wt) of CDB seed culture solution adsorbed by equivalent mass of cornmeal were inoculated into each container for further composting. Compost temperature was determined with alcohol thermometer from 3 points in per container at 10:00 and 16:00 everyday and compost temperature of the day was the average of the two readings.

3 RESULTS AND DISCUSSION

3.1 Isolation and Screening Psychrotrophic Bacteria

During screening experiments, we obtain following different degrading strains: 11 strains degrade starch, 13 strains degrade protein, 8 strains degrade cellulose. In Table 3 to table 5, the results of hydrolysis transparent circle and colony diameter measurement were shown. And these strains were psychrotrophs to degrade organic substances. The screened strains were used for amylase activity, protease activity and cellulase activity measurement after cultured at 15 . The experiment results were shown in Figure 1(A, B). From the figures, the strain B10-40 had the highest amylase activity about 63.64U/mL, the strain B6-38 had the highest protease activity about 46.08U/mL, the strain B6-15 had the highest cellulase activity about 24.94U/ml.

3.2 Isolation and Screening Thermophilic Bacteria

Fifty thermophilic cellulose-degrading bacteria were isolated and purified from the enriched culture with cellulose-Congo red medium. CDB with Up values (Table 6) of the top 50% (44.98±5.69~13.75±0.44, Table 6) were chosen for further screening. 50 CDB above whose grow mass in the broth after 48 h were chosen and the flasks that were inoculated with Strains of No.3, No.5, No.27, No.32, No.33, No.37, No.39, No.41, No.46 and No.51 were more cloudy than CK, which had a competitive advantage to adapt at the thermophilic phase of composting. Then the active strains were transferred into culture medium for producing cellulose. It was found that the filter paper enzyme activity reached the maximum on the third day except for strain No.3 and No.32. The maximum filter paper activity of these 10 CDB strains was arranged from high to low, and CDB having the maximum of more than 8 U/ml were used for testing their ability of degrading filter paper in Hutchinson mediums. At the end of incubation (the 14th day), the degradation rate of CDB No.27, No.37, No.39, No.41 was in the order of: 9.0%、10.9%、15.9% and 16.1%, respectively. The ability of No.39 and No.41 to degrade filter paper was obviously higher than that of No.27 and No.37. So CDB No.39 and No.41 were the targeted strains (Figure 1. C, D).

Colonies of strain No.39 and No.41 were pale yellow, circular when grown on NR medium for 2 days at 80 . Cells are straight rods. Growth occurs at 50~80 . The results from nucleotide-nucleotide BLAST program showed that the sequence of the 16S rRNA gene of strain No.39 had 100%, 99% and 99% similarity to the 16S rRNA gene sequence of *Geobacillus* sp.TERI NSM, *Geobacillus* sp.N60 and *Geobacillus* kaustophilus HTA426. As to strain No.41, with 99% sequence similarities to *Geobacillus* sp.TERI NSM, *Geobacillus* sp.N60 and *Geobacillus kaustophilus* HTA426, respectively. Based on these results, we came to conclusion that both strain No.39 and No.41 belong to *Geobacillus* sp., we named strain No.39 *Geobacillus* sp.HNC39 and strain No.41 *Geobacillus* sp.HNS41. *Geobacillus* sp.HNC39 was isolated from a high-temperature sugar refinery residues compost, *Geobacillus* sp.HNC39 from a muck soil in tropical rain forest.

3.3 Bacteria used in composting

In low-temperature experiment, groups added with microbial inoculums showed higher temperature than that of the control about 23.0 , the raise speed of temperature from the two experiments with B6-38, B10-40 was faster than the experiment with P1. In 28 hours, the temperature of composting with B6-38 and B10-40 could up to 39.6 and 38.3 , arounding 16.6 and 15.3 higher than the control experiment. The other two cold-adapted bacteria, B10-44 and B9-13, could also rise the composting temperature, but the effect was less effective than the control experiment (Figure 2, A).

For thermophilic bacteria, the composts with different treatments had gone through three phases (growing, thermophilic and cooling phases) and temperatures from different points were monitored in the composts (Figure 2, B). T-1 and T-2 showed two days earlier than other treatments to reach the thermophilic phase, and also two days longer at this phase. Besides, the temperatures of T-1 and T-2 were significantly higher than those of T-3 and CK on the second and third day (p=0.05), and there was no differences in temperature between T-1 and T-2 (p=0.05) as between T-3 and CK. There was no significant differences among T-2, T-3 and CK for the increasing temperature but still lower than T-1 significantly. Therefore, HNS39 was more efficient than HNS41 in maintaining high-temperature during composting. The temperature of the composts decreased to below 50 and entered the cooling phase on the fifth day. The temperature of T-2 was significantly lower than that of T-1 on the sixth day due to rapid decrease in T-2, but still higher than that of T-3 and CK whose temperatures changes little. At the end of composting, there were no differences in temperature between treatments. HNS39 was more effective than HNC41 in slowing down the temperature at the maturation phase. However, there were no significant differences in temperature changes between treatments with mixture of HNS39 and HNC41 (at the mixing ratio of 1 to 1) and the control.

4 CONCLUSIONS

Psychrotrophs and thermophilic microbes were isolated and screened. During the separation experiment, there are three cold-adapted bacteria can be effectively degrade organic substances. The three strains (B10-40, B6-38, B6-15) can rapidly reproduce and effectively degrade organic compounds with highest enzyme activity in low temperature. During composting experiment, the result was shown that cold-adapted bacteria had a better effect on the raise of composting temperature, especially B6-38 and B10-40. Therefore, the experiment result was fully shown that isolated cold-adapted bacteria had high potential of application in the composting in cold environment.

Meanwhile, two cellulose-effectively-decomposing thermophilic bacteria, HNS39 and HNC41, were screened from 13 thermophilic compost samples and one soil sample from tropical rain forest. It was found that they both belong to the genus *Geobacillus* according to 16S rDNA gene sequencing and their growth characteristics. Addition of microbial agents with single HNS39 or HNC41 was more effective in improving the progress of composing. For application purpose, HNS39 was better than HNC41. However, there were no significant differences in temperature changes between treatments with the mixture of HNS39 and HNC41 (at the mixing ratio of 1 to 1) and the control.

FIGURE 1 **Characterization of bacteria. A: Amylase activity and protease activity of low-temperature degrading bacteria; B: Celluase activity of low-temperature degrading bacteria; C, D: cellulase activity of the tested thermophilic strains**

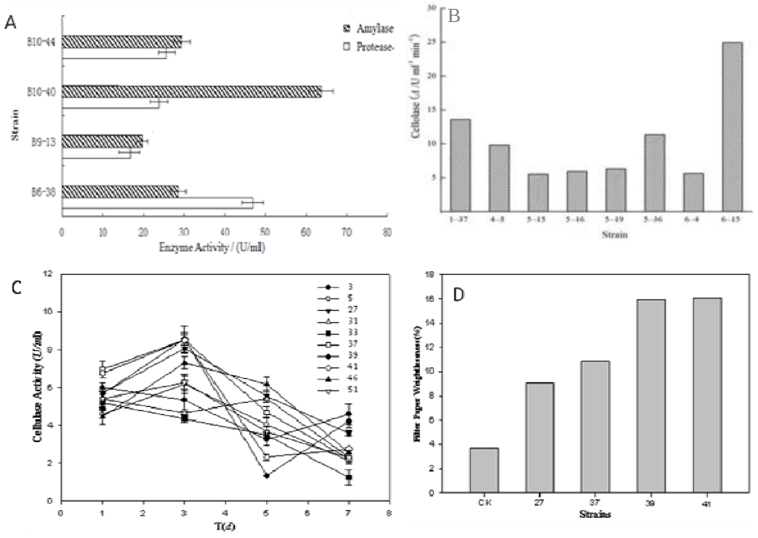

FIGURE 2 **The change of composting temperature when inserted different microbial inoculums. A: low-temperature degrading bacteria; B: thermophilic strains**

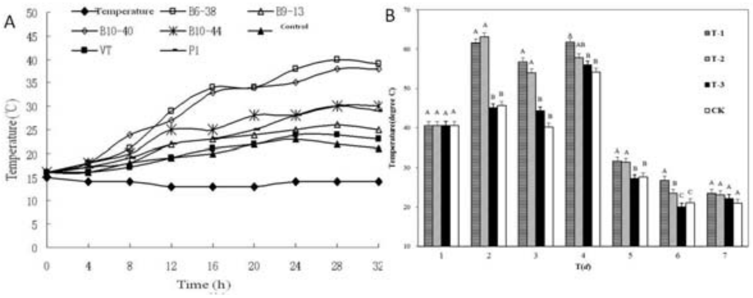

TABLE 1 **Characteristics of raw materials for composting with thermophilic strains**

Raw materials	MC(%)	TOC(%)	TN(%)	C/N
chicken manure	72.99	32.48	2.61	12.4
returned compost	43.11	38.68	1.69	22.9

TABLE 2 **Experimental design of inoculations of thermophilic strains**

Treatments	T-1	T-2	T-3	CK
Inoculations	HNS39	HNC41	CA *	SW**

*CA, compound agent mixing with seed liquid of HNS39 and HNC41 by 1:1 (v/v)

**SW, sterile water

TABLE 3 **The size of transparent circle and colony diameter of starch-degrading bacteria in screening at low temperature**

Strain	Transparent circle diameter (D/mm)	Colony diameter (d/mm)
B1-6	10.62	3.30
B1-18	13.22	3.46
B1-24	10.02	4.02
B1-32	12.24	5.04
B4-4	8.06	3.18
B5-15	10.64	3.46
B5-19	10.72	3.58
B6-38	14.32	3.86
B9-13	16.42	4.68
B10-40	21.62	6.24
B10-44	20.42	6.16

TABLE 4 **The size of transparent circle and colony diameter of protein-degrading bacteria in screening at low temperature**

Strain	Transparent circle diameter (D/mm)	Colony diameter (d/mm)
B2-7	8.20	4.62
B2-16	7.56	4.22
B2-30	3.64	2.48
B5-15	6.26	4.62
B5-19	7.42	4.58
B6-12	8.20	4.32
B6-17	7.28	4.16
B6-38	6.26	3.98
B7-8	6.92	4.58
B9-13	7.76	5.14

B10-27	7.54	4.16
B10-40	7.12	5.02
B10-44	7.96	5.58

TABLE 5 **The size of transparent circle and colony diameter of cellulose-degrading bacteria in screening at low temperature**

Strain	**Transparent circle diameter (D/cm)**	**Colony diameter (d/cm)**
B1-37	0.80	0.15
B4-5	0.80	0.10
B5-15	0.80	0.20
B5-16	0.90	0.30
B5-19	0.70	0.20
B5-36	0.60	0.15
B6-4	0.65	0.20
B6-15	1.00	0.25

TABLE 6 **The results of Screening by the cellulose-Congo red medium of thermophilic strains**

Strain No	**Up**	**SD**	**Strain No**	**Up**	**SD**
30	44.98	5.69	47	11.41	1.19
33	43.07	5.13	44	10.84	0.39
37	40.19	4.22	7	10.18	0.53
20	40.11	0.00	24	9.81	0.78
32	36.00	0.00	12	9.70	1.22
27	32.11	0.00	8	9.62	0.88
51	30.89	2.12	43	9.00	0.00
23	30.50	7.78	22	8.58	0.59
56	28.47	2.51	13	8.42	0.82
52	27.63	3.71	10	8.40	0.53
16	27.30	1.99	17	8.28	1.02
11	26.81	5.34	1	8.28	1.02
14	25.56	0.98	35	8.20	0.73
46	24.02	1.39	19	7.55	0.55
45	19.78	1.73	9	5.85	0.69
28	18.78	0.00	18	5.55	1.00
39	18.71	0.92	4	5.42	1.17
6	18.13	3.01	42	5.35	0.47
15	17.39	1.96	26	5.08	0.20
3	16.04	2.00	31	4.95	0.16
21	16.00	0.00	25	4.35	0.49
36	14.06	0.00	34	3.81	0.28
41	14.01	0.80	49	3.81	0.28
29	13.90	1.92	50	3.62	0.54
5	13.75	0.44	2	2.38	0.28

SD, standard deviation.

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(203) COMPOSTING USED AS A LOW COST METHOD FOR PATHOGEN ELIMINATION IN SEWAGE SLUDGE

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EXECUTIVE SUMMARY

Spreading sewage sludge from municipal wastewater (MWW) treatment on land is still a common practice in developing countries. However, it is well known that sewage sludge without special treatment contains various pollutants, which are (re)introduced into the environment by sludge landspreading and which might in turn have harmful effects to the environment and human health. This is more dangerous in places like Merida, Mexico, where soil is calcareous with fractures along the ground and thin layers of humus (Duch 1988). Consequently, any liquid and semisolid wastes has the potential of percolating to the subsurface and contaminate the aquifer (Magaña 2002).

Management of residual sludge has become an important problem and, in some cases, with expensive solutions. One of the solutions implies the reduction of pathogen microorganisms before applying it to soils as a conditioner agent. Mexican legislation suggests addition of calcium hydroxide to a septic sludge to obtain a safe product. After that, disposal is carried out by putting all waste in ditches previously constructed (Comisión Nacional del Agua, 1997). In Merida, most householders use a septic tank as a sewage system. Once the septic tank is full, its content is collected and discharged into an artificial oxidation pound system where sludge and water are separated by decantation. Separated sludge is put away on the ground where it is sun dried. Endemic birds and insects like flies and worms have continuous contact with this waste. For flies, it becomes an ideal place for laying its eggs due to considerable amount of nutrients. As a consequence, they become a way of transporting viruses and bacteria with potential of spreading human diseases. The most common pollutants contained in sewage sludge from MWW are human pathogens like fecal coliforms, salmonella and helminth eggs.

The main aim of this work was using composting as a low cost process to eliminate pathogens contained in sewage sludge from MWW treatment in order to use the final product for land spreading in a safe way for both environment and human health.

Two piles for composting process at real scale were settled using a mixture of sewage sludge from municipal waste water and green waste. Composting was carried out by windrow process and it was monitored during four weeks. Concentration of helminth eggs, salmonella and faecal coliforms were measured twice a week to observe its behaviour and, as a control process, Temperature, Moisture Content (MC), and pH were also measured.

After 30 days of composting sludge from municipal waste water system, salmonella was eliminated by 99%, faecal coliforms by 96% and helminth eggs by 81%. The results showed that a composting process is an economical and efficient method for pathogens elimination in sewage sludge from municipal waste water.

1. INTRODUCTION

The present work was developed in the city of Mérida, located on the Yucatán Península which is formed by a karst surface. Due to karstic conditions, there are a lot of fractures in the subsoil, the aquifer receives water from rainfall and with pollution. The aquifer is also the unique source of water in the region. The main sources of pollution of the aquifer are the wastewater and sludge from the municipal wastewater system.

The main goal of this study was to test the compost process as an economical and efficient method for pathogen elimination in sludge from a municipal waste water system.

In the case of the city of Merida, Mexico, this process turns out to be important because at present the sludge from the municipal wastewater system does not receive any treatment before being deposited on soils, so it might represent a potential source to spread pathogens.

The composting process has shown to be an economic method, capable of eliminating or reducing the pathogenic microorganisms present in the material being treated by composting.

According to Epstein E. (1997), composting is very effective in destroying pathogens, as a result of temperature – time relationship.

1.1 Background

Most houses in Merida have septic tanks as a sewage system. Once the septic tank is full, its content is collected and discharged into an oxidation pond system (fig. 1) where sludge and water are separated by decantation. Separated sludge is put away on the ground where it is sun dried. Endemic birds and insects like flies and worms have continuous contact with this waste. For flies, it becomes an ideal place for laying its eggs because of the amount of nutrients it has. As a consequence, they become a way of transporting pathogenic microorganisms with potential of spreading human diseases.

As expected, municipal solid waste, sludge from municipal waste water (biosolids), food waste and yard waste, contain human pathogenic organisms. Composting, if carried out properly, is an effective process to destroy pathogens like faecal coliforms and Salmonella pp. The latter is the result of temperature – time relationships (Epstein, 1997).

During the thermophilic stage of composting, an increment of temperature can occur through the sludge mass as a result of the microorganisms' activity. Thus pathogens content within the sludge could be destroyed. Temperatures above 45 ºC characterizes this period of composting (Haug 1993; Diaz et al, 1993; Epstein 1997). During the next stage temperature descends, causing a decrease of degradation speed. At the end of the composting process, resulting material looks similar to humus, and is free of easy available organic matter, weed seeds and pathogens.

Composting is an aerobic process, thus oxygen is essential element for the microbial activity. Sludge from waste water treatments usually contains large amounts of moisture which inhibits the free flow of air. Wood or bark chips, sawdust, yard trimmings and pruning trees have been tested like bulking agents providing free spaces for an adequate oxygenation (Das et al 2003,Manios et al., 1997). For the current work yard trimmings were used.

Composting by windrow has shown a good performance for sludge mixed with yard trimmings. According to Velazquez *et al*.(2006), with this process, fecal coliforms were eliminated by 90% after a month.

The windrow composting method needs a lot of space and some mechanism for agitating the material (adding oxygen). For the city of Merida, the land is very cheap and the addition of oxygen has been proposed for manual turning, ie a couple of people turn their compost heap. Consequently, resources are very low cost.

1.2 Research objectives

To test the windrow composting as a low cost process to eliminate pathogens contained in sewage sludge from municipal wastewater treatment.

To observe the behaviour of helminth eggs, salmonella and faecal coliforms during the windrow composting process.

To obtain a final product that can be deposited on the soil safe to environment and human health.

2. METHODOLOGY

2.1 Composting process

- Two real size-composting piles nearby the municipal waste water system in Merida, Mexico were built for this work. In this city, the municipal wastewater system is an oxidation pound (Fig. 2).
- Each pile was constructed by a mixture of sludge from the municipal waste water system (40%) and green waste or yard trimmings (60%).
- Composting was made by windrow system, using Temperature, Moisture Content and pH as control parameters.
- Composting piles were monitored during four weeks for the active stage by analysing, twice a week the concentration of Helminth eggs, Salmonella and faecal Coliforms.
- After the active stage, the composting material was tested twice a month the following two months for the same pathogens and checking the germination index.

2.2. Laboratory Techniques

- Salmonella and Faecal Coliforms

For salmonella and faecal coliforms tests, the procedures described by the NOM-004-SEMARNAT-2002 were used.

The above methodology is based on the procedures described in the "Standard Methods for the examination of Water and Wastewater", where the techniques of Most Probable Number (MPN) were used. These procedures have been established by the Mexican environmental legislation specifically for the identification of pathogens in sludge and also to establish the maximum permissible limits for pathogens in sludge to be deposited in soils safely.

- Helminth Eggs

For determining helminth eggs concentration, the method described by Moodley *et a*l., (2008) was used. The method is based on three fundamental processes: washing, filtering one or more times, and then floating and sedimenting of the retrieved parasites. A flotation step is used for the isolation of helminth ova using density gradient centrifugation and a chemical solution that is saturated at a specific gravity of 1.3 so that all helminth ova having relative densities that range from 1.13 (e.g. *Ascaris*) to 1.27 (e.g. *Taenia*) are able to float in that solution. The deposit is transferred to one or more microscope slides and examined under the microscope to enumerate each species of Helminth ova using the 10x objective and the 40x objective to confirm any uncertainties.

- Germination Index

Germination index was performed by a phytotoxicity bioassay, using a liquid extract prepared from 30 grams of composts and distilled water for watering radish seeds (instead of cress seeds), as described by the Methodology used by the Lab Staff at University of Leeds. Results were calculated based on the following formula:

GI = (Total Length of roots in test place / Total Length of roots in control plate) x 100

- Control parameters

Values of potential of Hydrogen (pH) and Moisture Content were obtained according with the "Standard Laboratory Procedures for the Analysis of Compost" from Leeds University, U.K. and Federal Compost Quality Assurance Organization Manual, Germany.

3. RESULTS AND DISCUSSION

After 30 days of composting sludge from municipal waste water system, Salmonella was eliminated by 99%, Faecal coliforms by 96% and Helminth Eggs by 81% (Table 1).

The potential risk of the presence of faecal coliforms in sewage sludge is because they are extremely resistant to certain conditions and their persistence for long periods of time. Therefore, faecal coliforms are used as indicators of the effectiveness of treatment processes in the destruction of bacteria, and regulate the quality of sewage sludge that can be used safely. They are also indicative of the concentration of *Salmonella spp*., bacteria that are usually associated with gastrointestinal diseases in humans and thus reducing faecal coliform ideally reflecting a decrease in *Salmonella spp*. (Barrios *et al* , 2000).

Although early in the process the sludge have high values in the content of salmonella (higher than faecal coliforms), with increasing temperature during the composting process, the content of salmonella decreased to values lower than fecal coliforms, this coincides with the nature of coliform group, which have higher survival times that of enteric pathogens.

The concentration of Salmonella was decreased up to by 99%, but failed to reach the maximum levels of 300 NMP/g reported by the NOM-004-SEMARNAT-2002, for classification in Class C (For forest, soil improvement and agricultural uses). This may be due to the nature of the sludge. Faecal coliform levels were below 2 000, 000 MPN / g corresponding to the class C according to NOM-004-SEMARNAT-2002. This indicates that the sludge can be disposed of safely in regard to fecal coliforms.

Helminth eggs, particularly Ascari, are very resistant to changes in environmental factors, which are stressful for other microorganisms, thus present an additional challenge for sludge treatment processes. The number of helminth eggs found after the composting process is a Class C according to NOM-004-SEMARNAT-2002. Several authors suggest that conventional processes, only those who apply high temperatures of about 50 ° C or more, reduce the density and the viability of different Helminth eggs, this coincides with the results, since the temperatures reached in the process of composting were higher at 60 ° C.

For the germination index was only possible to sample every month, the samples were performed in duplicate and showed positive results (Table 2). The first month the germination rate was 65% on average, which is consistent with results reported by Silva sludge et al (2006). The last test performed strong in the third month and reported a GI =

160%. According to Manser and Keeling (1996) values above 100 % are indicative of positive influence of compost to the germination process.

However, these values as unique criteria are not enough to make a real assessment and it is important to clarify that this procedure is only focused in the initial germination phase and it cannot predict the behavior during the whole plant growth.

4. CONCLUSIONS

- After 30 days of composting sludge from municipal waste water system, Salmonella was eliminated by 99%, Faecal coliforms by 96% and Helminth eggs by 81%.
- The fecal coliform concentration decreased to levels below the maximum permissible limits
- The sludge can be disposed directly on the ground safely with respect to the concentration of fecal coliforms.
- Better control is required for the elimination of Salmonella to achieve maximum permissible.
- Germination Index values indicate that the material can be used as a soil improvement. But it is necessary more testing.
- The results showed that a composting process is an efficient method for pathogens elimination in sewage sludge from municipal waste water.

5. ACKNOWLEDGEMENTS

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FIGURE 1 **Oxidation ponds system in Mérida, Mexico.**

FIGURE 2 **Composting piles at real scale**

TABLE 1 **Results**

Composting Time (days)	**** Salmonella NMP/g lodo**	**** Faecal Col NMP/ g lodo**	**** Helminth Eggs / g de lodo**	****Control parameters**		
				T °C	MC %	pH
0	240000000	2400000	146	27	52.77	7.27
4	46000000	1100000	131	56	68.57	7.7
9	2100000	460000	87	63.5	58.61	7.87
14	1500000	240000	38	63.0	67.23	7.73
18	750000	210000	32	53.6	56.21	7.71
23	39000	150000	29	49.1	50.14	7.69
28	11000	120000	29	37.5	48.86	7.67
31	3000	93000	27	37.2	46.72	7.64
Max. Permissible limits For sludge "C" class * NOM-004-SEMARNAT-2002	**300**	**2000000**	**35**			

**Sludge "C" class are allowed for forest, soil improvement and agricultural use*

*** All values are the average of 3 replicates*

TABLE 2 **Germination Index**

Composting time (months)	**Germination Index (%)****
1	65
2	98
3	160

*** All values are the average of 3 replicates*

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Session 17

(55) MBT SCRUBBER EFFLUENT: wastewater or fertiliser?

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EXECUTIVE SUMMARY

The design of waste air cleaning in Mechanical Biological Treatment (MBT) plants frequently includes acid scrubbing followed by biofiltration. The main purpose of the former process is to remove ammonia from the exhaust air, which produces odours and is toxic for the biofilter microorganisms. The ammonia is then solubilised as ammonium sulphate or ammonium nitrate, according to the acid used in the scrubber (sulphuric or nitric acid, respectively). When the washing solution reaches a preset conductivity, it is partially discharged and replaced by fresh water. As a consequence, an effluent with high ammonia concentration is produced, which is difficult and expensive to treat in a waste water treatment plant. Yet, if we see it in the agronomical perspective, an ammonia rich solution may not be a pollutant but a fertiliser.

The objective of the present study was to evaluate the potential of the scrubber effluent from Amarsul MBT plant in Setúbal, Portugal, to be used as a nitrogen liquid fertiliser. The experiments were carried out with wheat and grass. These species were selected so that land application of the product could be performed all around the year. In the wheat experiment, nitrogen fertilisation supplied by the scrubber effluent was compared to the conventional fertilisation with ammonium sulphate. Results showed that the obtained yields, considering grain and straw fresh and dry weight, were not significantly different, independently from the nitrogen source. No significant differences were also observed in the grass experiment, in which three nitrogen applications were carried out: one at the beginning of the experiment and the other two following the first and second cuts. Grass yield was determined after each cut. In both experiments, soil analysis indicated a more expressive drop in pH (relative to the control) for the treatment with mineral fertilisation, comparing to that receiving scrubber effluent. The composition of the scrubber effluent was determined in order to detect eventual pollutants such as heavy metals, organic compounds and pathogens. The results show that it fulfils the quality required in Portuguese legislation for irrigation water (DL 236/98) and for sewage sludge used in agriculture (DL 276/2009).

Besides evaluating the agronomic and environmental quality of the potential fertiliser, it is important to optimise the product logistics, considering storage in the MBT plant and transport to the client site. Hence, different scrubber operation regimes were tested with the objective of maximising the ammonium sulphate concentration of the effluent, without causing damage to the equipment or affecting the odour removal performance. Results demonstrated that it is possible to reach compatibility between air treatment operation requests and fertiliser logistics.

This study demonstrates that the scrubber effluent from exhaust air treatment in Amarsul MBT plant could be an environmentally safe good quality ammonia fertiliser, with high potential under an agronomic point of view.

1. INTRODUCTION

AMARSUL Mechanical Biological Treatment plant has an input of 48 000 t/a of municipal solid waste plus 2 000 t/a of garden waste, producing about 4 500 t/a of compost mostly used in vineyards. The biological process is carried out inside an enclosed hall subject to negative mode aeration. An airflow of 30 000 m^3/h is sucked from the composting bed and delivered to a scrubber where it is washed with a dilute sulphuric acid solution for ammonia removal. After this step, the air is blown through a biofilter together with 60 000 m^3/h of less polluted exhaust air (previously humidified) coming from the reception hall and from the top of the composting hall.

In the scrubber the ammonia is solubilised as ammonium sulphate. When the washing solution reaches a preset conductivity, it is partially discharged and replaced by fresh water. As a consequence, an effluent with high ammonia concentration is produced, which is difficult and expensive to treat in the waste water treatment plant. The objective of the present study was to evaluate the potential of this effluent to be used as a nitrogen liquid fertiliser, considering both agronomic benefit and environmental safety.

2. METHODOLOGY

2.1 Agronomic experiments

The agronomic experiments were carried out with wheat and grass. These species were selected so that land application of the product could be performed all around the year. The composition of the scrubber effluent used in the experiments is presented in Table 1 and results from operating at a conductivity set-point of 15 mS/cm, corresponding to 1% ammonium sulphate.

TABLE 1 **Scrubber effluent composition.**

pH	Conductivity (mS/cm)	NH_4 (g/l)	SO_4 (g/l)	Ca (mg/l)	Mg (mg/l)	Na (mg/l)	Cl (mg/l)
6.9	14.4	2.67	7.50	22.5	5.00	21.5	92.3

2.1.1 Wheat experiment

The objective of this experiment was to evaluate the effect of replacing conventional fertilisation with ammonium sulphate by nitrogen fertilisation supplied by the scrubber effluent. It took place in pots containing 9.5 kg of a Vertisol top soil which composition is described in Table 2.

TABLE 2 **Composition of the top soil Vertisol used in the wheat experiment.**

pH (H_2O)	Organic matter (%)	Extractable* (mg/kg)		Exchangeable bases** (cmol(+)/kg)			
		P_2O_5	K_2O	Ca	Mg	K	Na
6.97	1.71	545	248	27.23	6.57	0.6	0.48

* Extractable by ammonium lactate solution (AL).
** Exchangeable by ammonium acetate at pH 7.0.

Treatments (replicated three times) were as follows:

A – PK (control)
B – NPK
C – N'PK

All received a base PK fertiliser application – 0.1 g P_2O_5 and 0.15 g K_2O/kg soil – in the form of potassium dihydrogen phosphate and potassium chloride, respectively. Nitrogen – 0.15 g N/kg soil – was applied in three steps: 0.075 g N/kg soil as base fertiliser and 2 x 0.0375 g/kg soil in the following applications. N-nitrogen was supplied as ammonium sulphate (20.5% N) while N'-nitrogen was provided by the scrubber effluent.

The experiment started on November 23 and the wheat seeding was carried out on November 26, 2009. After ten days, the number of plants per pot was set to twenty. During the experiment soil moisture content was kept at 60% saturation capacity. The 2nd and 3rd fertilisations were performed on February 21 and March 22, 2010, respectively.

On May 3, 2010, the plants were harvested for yield measurement. Grain and straw fresh and dry weight were determined. Crop and soil samples were collected for chemical analysis.

2.1.2 Grass experiment

The trial took place in an existing lawn at AMARSUL composting plant. A lawn section was arranged in twelve 1 m^2 plots, as described in Figure 1. A security margin separated control from fertilised plots.

Before starting the experiment soil samples were collected and analysed. The results are shown in Table 3.

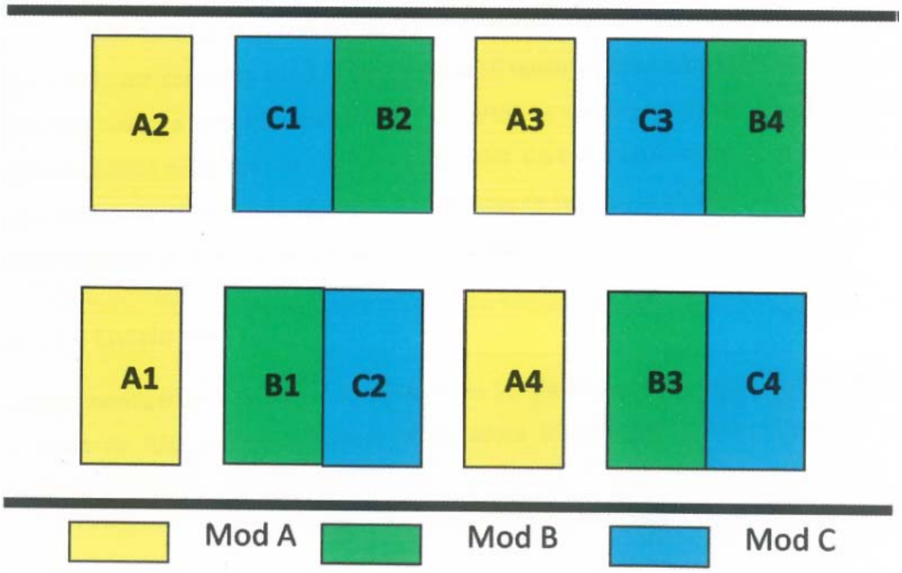

FIGURE 1 **Plot arrangement for grass experiment.**

TABLE 3 **Composition of the soil used in the grass experiment.**

pH (H_2O)	Organic matter (%)	Extractable* (mg/kg)		Exchangeable bases** (cmol(+)/kg)			
		P_2O_5	K_2O	Ca	Mg	K	Na
6.1	0.32	346	81	4.12	0.50	0.23	0.16

* Extractable by ammonium lactate solution (AL).
** Exchangeable bases by ammonium acetate at pH 7.0.

Treatments (replicated four times) were as follows:

A – Control
B – 90 kg N/ha applied as ammonium sulphate
C - 90 kg N/ha applied as scrubber effluent

Nitrogen fertilisation – 30 kg N/ha – was performed in three steps: December 18, 2009 (beginning of trial), March 16 and April 30, 2010 (after first and second mowing). The experiment finished on June 1, 2010, after the third mowing. A 0.34 x 0.34 m section in each plot was cut for yield measurement. Fresh and dry weight were determined. In the end of the experiment grass and soil samples were collected for chemical analysis.

2.2 Pollutant analysis

To assess the environmental safety of the scrubber effluent used in the agronomic experiments, the following parameters were analysed:

- Heavy metals: Cd, Cu, Ni, Pb, Zn, Cr, Hg [ICP – ASTM D 1976 (2007)];
- Organic pollutants: AOX (DIN EN 1485, 8.2.2), PAH (DIN 38407-18), NPE (ISO/FDIS 18857-1), DEHP (GC-MS), LAS (GC-MS), PCB (DIN 38407-2), PCDD/F (ISO 18073);
- Pathogens: *E. coli* [L25-PT-MA-01 (2005-04-11), Membrane Filtration] *Salmonella sp.*(ISO 6340:1995, Membrane Filtration).

2.3 Scrubber performance

Various scrubber operation regimes were tested with the objective of maximising the ammonium sulphate concentration of the effluent, in order to improve the product logistics. Conductivity set-point was progressively increased from 15 mS/cm up to 250 mS/cm, according to the following steps: 15, 100, 150, 200, 240 and 250 mS/cm. Ammonium sulphate concentration was determined (method ISO 5664:1984 rev.03, 2008, for NH_4 and NP 413:1966 rev.03, 2008, for SO_4) for each operation mode.

Data collected included scrubber operation temperature, recirculation pump pressure and visual inspection for salt deposition in piping and nozzles. Ammonia was measured in the scrubber input and output airstreams. The samples were collected in Tedlar bags and the ammonia concentration was determined using Dräger-Tube-Measurement-System.

3. RESULTS AND DISCUSSION

3.1 Effect of scrubber effluent on wheat crop

Table 4 shows the wheat yields per pot. No significant differences were observed between treatments **B** (mineral fertiliser) and **C** (scrubber effluent), which yields were in turn considerably higher than that obtained in treatment **A** (control). The quality of grain and straw was not affected by the nitrogen source, as shown in Table 5.

TABLE 4 Effect of scrubber effluent fertilisation on wheat yield.

Treatment	Total fresh substance (g/pot)	Total dry substance (g/pot)	Grain (g/pot)
A	83.70 b	44.17 b	13.23 b
B	125.50 a	87.03 a	37.87 a
C	116.63 a	80.60 a	32.75 a

Note: In each column, means with different letters are statistically different (p<95%).

Table 6 presents the soil composition in the end of the experiment. The results indicate a significant drop in pH (H_2O) relative to the control for the treatment with mineral fertiliser (**B**), which was not observed for scrubber effluent (**C**). No further significant differences were detected.

TABLE 5 Wheat composition (grain and straw) in the different treatments.

Treatment	N (%)	P (%)	K (%)
Grain			
A	1.80 b	0.40 a	0.52 a
B	2.33 a	0.36 a	0.45 b
C	2.09 a	0.36 a	0.43 b
Straw			
A	0.24 b	0.09 a	1.74 a
B	0.42 a	0.03 b	2.28 b
C	0.36 a	0.03 b	2.09 b

Notes: In each column, means with different letters are statistically different (p<95%).
Values expressed in dry substance.

TABLE 6 Soil composition in the end of the wheat experiment.

Treatment	pH (H_2O)	Organic matter (%)	Extractable (mg/kg)		Exchangeable bases (cmol(+)/kg)			
			P_2O_5	K_2O	Ca	Mg	K	Na
A	7.59 a	1.57 a	553 a	167 a	30.73 a	6.93 a	0.43 a	0.73 a
B	7.17 b	1.69 a	546 a	132 b	29.58 a	6.84 a	0.38 ab	1.00 a
C	7.41 a	1.62 a	562 a	137 b	29.81 a	6.65 a	0.35 b	0.78 a

Note: In each column, means with different letters are statistically different (p<95%).

3.2 Scrubber effluent as N-liquid fertiliser for lawn application

The grass yield per plot, taking a 0.34 x 0.34 m section, is shown in Table 7.

TABLE 7 Effect of scrubber effluent fertilisation on grass yield.

Treatment	Fresh substance (g/plot)	Dry substance (g/plot)
First cut		
A	26.64 b	6.66 b
B	41.42 a	10.35 a
C	53.36 a	13.34 a
Second cut		
A	39.68 b	9.92 b
B	183.28 a	45.82 a
C	138.15 a	34.54 a
Third cut		
A	88.48 b	22.12 b
B	168.4 a	42.10 a
C	175.7 a	43.93 a
TOTAL yield		
A	154.19 b	32.63 b
B	393.09 a	65.68 a
C	367.21 a	66.58 a

Note: In each column, means with different letters are statistically different (p<95%).

The addition of scrubber effluent at 3 x 30 kg N/ha (treatment **C**) resulted in yields that were equivalent to 3 x 30 kg/ha of nitrogen applied as ammonium sulphate (treatment **B**). Both treatments with N increased grass yield compared with the control (**A**). As observed in the wheat experiment, the nitrogen source did not affect grass quality (Table 8).

TABLE 8 Grass composition in the different treatments.

Treatment	N (%)	P (%)	K (%)
First cut			
A	1.59 a	0.27 a	1.62 a
B	1.94 a	0.35 a	1.95 a
C	1.89 a	0.31 a	1.83 a
Second cut			
A	2.26 a	0.40 a	2.43 a
B	2.65 a	0.45 a	2.58 a
C	2.33 a	0.40 a	2.33 a
Third cut			
A	2.85 a	0.43 a	2.75 a
B	2.96 a	0.47 a	2.96 a
C	2.99 a	0.44 a	2.69 a

Notes: In each column, means with different letters are statistically different (p<95%).
Values expressed in dry substance.

The results of the soil analysis (Table 9) suggest a drop in pH (H_2O) as a consequence of ammonium sulphate application (**B**), which is not significant when nitrogen is supplied by the scrubber effluent (**C**). No further significant differences were detected between treatments **B** and **C**.

TABLE 9 Soil composition in the end of the grass experiment.

Treatment	pH (H_2O)	Organic matter (%)	Extractable (mg/kg)		Exchangeable bases (cmol(+)/kg)			
			P_2O_5	K_2O	Ca	Mg	K	Na
A	6.69 a	3.76 a	196 a	91.75 a	4.89 a	0.83 a	0.14 a	0.23 a
B	5.57 b	3.72 a	148 a	62.50 b	4.32 a	0.83 a	0.14 a	0.20 a
C	6.06 ab	4.54 a	174 a	50.75 b	4.41 a	0.71 a	0.13 a	0.20 a

Note: In each column, means with different letters are statistically different (p<95%).

3.3 Evaluation of environmental safety

The concentration of heavy metals, organic pollutants and pathogens in the scrubber effluent used as nitrogen liquid fertiliser is summarised in Table 10.

The scrubber effluent fulfils the limits for trace elements in irrigation water recommended by FAO, Ayers and Westcot (1994), as well as those indicated in Portuguese legislation, DL 236/98 (Table 10). The pathogens analytic results demonstrate good sanitation.

The amount of organic pollutants added to the soil as a consequence of scrubber effluent treatment was calculated based on the application rate used in the grass experiment. The values obtained were compared with those resulting from the application of 1 t/ha (dry substance) of a sewage sludge presenting the limit values for organic pollutants defined in the

Portuguese legislation, DL 276/2009. As shown in Table 11, the organic pollution caused by the scrubber effluent is negligible.

TABLE 10 **Scrubber effluent quality comparing with the requirements for irrigation water.**

Parameter	Scrubber effluent (1% ammonium sulphate)	Recommended maximum concentration in irrigation water	
		DL 236/98	FAO, 1994
Heavy metals			
Cd (mg/l)	< 0.05	0.01	0.01
Cu (mg/l)	< 0.05	0.2	0.2
Ni (mg/l)	< 0.05	0.5	0.2
Pb (mg/l)	< 0.05	5.0	5.0
Zn (mg/l)	0.11	2.0	2.0
Cr (mg/l)	< 0.05	0.1	0.1
Hg (mg/l)	0.003	-	-
Organic pollutants			
AOX (mg/l)	< 0.10	-	-
PAH (µg/l)	< 0.01	-	-
NPE (µg/l)	1.3	-	-
DEHP (µg/l)	3	-	-
LAS (µg/l)	< 10	-	-
PCB (µg/l)	0.11	-	-
PCDD/F (ng I-TEQ/l)	0.0098	-	-
Pathogens			
E. coli (CFU/100ml)	2	-	-
Salmonella sp.(/1000ml)	Absent	-	-

TABLE 11 **Soil organic pollution resulting from the application of scrubber effluent versus sewage sludge.**

Parameter	Scrubber effluent*	Sewage sludge**
PAH (g/ha)	$< 4.3\times10^{-4}$	6
NPE (g/ha)	5.6×10^{-2}	450
LAS (kg/ha)	$< 4.3\times10^{-4}$	5
PCB (g/ha)	4.8×10^{-3}	0.8
PCDD/F (µg I-TEQ/ha)	0.425	100

* As used in grass experiment.

** Presenting the limit values for organic pollutants defined in the Portuguese legislation, DL 276/2009, applied at 1t/ha (dry substance).

3.4 Effect of increasing ammonium sulphate concentration

By increasing the scrubber conductivity set-point from 15 mS/cm to 250 mS/cm it was possible to obtain an effluent 40 times more concentrated in ammonium sulphate, as shown in Figure 2. According to these results, the 43.4 m^3 needed to apply treatment **C** to 1 hectare lawn could be reduced to 1.1 m^3, being diluted on site.

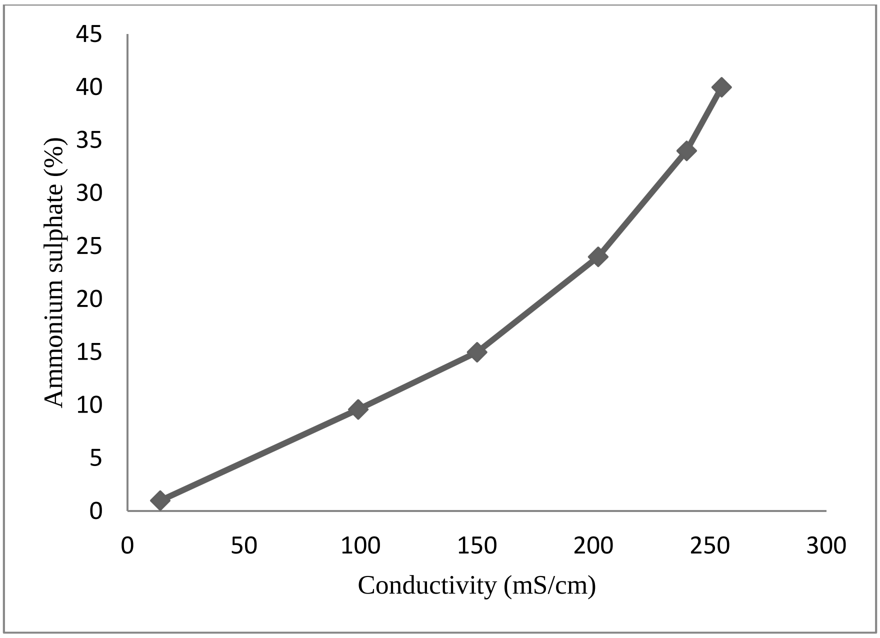

FIGURE 2 **Effect of increasing conductivity set-point on the ammonium sulphate concentration of the scrubber effluent.**

The new operation mode did not seem to affect scrubber parameters like temperature (50 – 60 ºC) or recirculation pump pressure (1.5 – 1.7 bar). Even at 40% ammonium sulphate, no salt deposition in piping or nozzles was observed. Additionally, the efficiency of ammonia removal from exhaust air was not affected. In fact, the ammonia concentration measured in the output airstream was always under 5 ppm.

4. CONCLUSION

This study indicates that the scrubber effluent from Amarsul MBT plant can effectively replace conventional nitrogen fertilisation on wheat crop and lawn maintenance without negative effects on yield, plant or soil quality. Furthermore, it complies with the Portuguese legal requirements for environmentally safe agriculture use. The liquid fertiliser can be supplied as a concentrated solution – 40% ammonium sulphate – to be diluted on site, reducing substantially the storage and transport costs.

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(58) PRODUCTION OF ORGANIC FERTILIZERS FROM RESIDUES OF THE OLIVE OIL PROCESSING INDUSTRY

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EXECUTIVE SUMMARY

The olive oil processing industry generates large amounts of liquid and solid residues (olive-mill wastewater, OMWW and olive mill solid waste, OMSW, respectively). OMWW is mainly collected in storage ponds and evaporated during summer leading to serious environmental problems. OMSW is further processed in central pomace oil plants to recover the residual oil by chemical extraction. After this extraction a final residue, deoiled solid cake (DOC), is produced. Currently, DOC is composted, used directly as soil improver or sold as fuel. In previous studies, it has been demonstrated that anaerobic digestion can be a promising alternative to treat these residues (Bilbao et al. 2010 and Sterr et al. 2011). This process has the advantage that the residues are converted to energy in form of methane and that the digestate can be dried and used as a stabilized organic soil improver. In fact, land application of stabilized digestate is an excellent practice to increase soil fertility (Stoll et al. 2012). However, in regions with intensive livestock production, land application is not always possible because of the already high nutrient content of the soil. In those regions, digestate must be either transported to other areas with nutrient demand or stored for long periods. Drying and pelletizing digestate could (1) stabilize the digestate, (2) reduce the transportation costs and storage requirements, and (3) facilitate handling and commercialization of the product. The objective of this study was to convert digested OMWW and DOC to a compact organic soil improver to create a commercial product, which could be stored, transported and spread with common fertilizer spreaders used in agriculture.

Two types of digestate: (A) from the co-digestion of OMWW and dairy wastewater, and (B) from the co-digestion of DOC and manure, were collected from two anaerobic reactors (100 L) at our laboratories. A solid-liquid separation was performed and the solid fraction was dried at 100°C until the sample had a total solid content (TS) of 85%. The dried solid was then milled and pelletized. The water content and plant nutrients concentration (TN, NH_4, TP, K, Mg, Ca, S and Fe) were measured before and after each process to compute a complete water and nutrient balance of the whole process. To determine whether the obtained product could be used as an organic fertilizer relevant plant nutrients and potential pollutants (Tl, As, Pb, Cd, Cr, Ni, Hg, Cu, Zn) were measured.

During centrifugation 90% of the mass was separated as liquid fraction. This liquid fraction is rich in nutrients and can be used for fertigation purposes. Alternative, these nutrients can be precipitated as phosphate and/or ammonium salts to be used as inorganic fertilizers. The mass of the wet solid fraction (TS = 28-30%) was then further reduced by the drying process by 75%, leading to a total mass reduction of 98% after centrifugation and drying. In terms of nutrient balance, more than 85% of the TP (total phosphorous) remained in the solid fraction after centrifugation. Thus, most of the P contained in the digestate is from organic nature or precipitated as inorganic salts. In contrary, most of the NH_4 (>70%) was found in the liquid fraction after centrifugation. Moreover, 26 to 50% of the total N in the digestate was volatilized as NH_3 during the drying and pelletizing process. This N could be easily recovered as ammonium sulfate using an acid scrubber. According to the experiments performed, the optimal TS value for pelletizing the dried digestate was 85%. Thus, the digestate should be dried until this TS value to achieve a high mechanical durability and to avoid the percentage of fines and fungal degradation. The N, P and K content (as N, P_2O_5, K_2O in dry matter) from the pellets produced with digestate A (OMWW and dairy wastewater) was 2.3%, 5.3%, and 0.85%, respectively and from digestate B (DOC and manure) 3.1%, 3.5% and 1.1%, respectively. These results showed that both products were rich on plant nutrients and are comparable with other organic fertilizer currently on the market. Moreover, the potential pollutants were below the maximum limits set by the German Fertilizer Ordinance (2008), exposing no threat for further usage in the agriculture.

This study proves that it is possible to convert digested residues from the olive oil processing industry into a compact organic soil improver. Moreover, it was demonstrated that drying and pelletizing digestate are excellent processes to stabilize and reduce the mass of the product (98%) creating a competitive commercial fertilizer.

1 INTRODUCTION

1.1 Background

The olive oil processing industry generates large amounts of liquid (olive-mill wastewater, OMWW) and solid (olive mill solid waste, OMSW) residues. OMWW is currently collected in storage ponds and evaporated during summer leading to serious environmental problems. OMSW is further processed in central pomace oil plants to recover the residual oil by chemical extraction. After this extraction a final residue, deoiled solid cake (DOC) is produced. Currently, DOC is composted, used directly as soil improver or sold as fuel. In previous studies, it was demonstrated that anaerobic digestion can be a promising alternative to treat these residues (Bilbao et al. 2010 and Sterr et al. 2011). This process has the advantage that the residues are converted into energy in form of methane and that the digestate can be used as a stabilized organic soil improver. In fact, land application of digestate is an excellent practice to increase soil fertility (Stoll et al. 2012). However, in regions with intensive livestock production, land application is not always possible because of the already high nutrient content of the soil. For instance, in a conventional agricultural biogas plant with 500 kW_{el}, approximately 100 metric tons of nitrogen (N) per year are generated with the digestate. If a fertilization of 170 kg N pro Ha is used, then 588 Ha are needed for that quantity of N (Fuchs and Drogs 2010). Thus, in regions with intensive livestock, this digestate must be either transported to other areas with nutrient demand or stored for long periods. Drying and pelletizing digestate could (1) stabilize the digestate, (2) reduce the transportation costs and storage requirements (Stoll et al. 2010), and (3) facilitate handling and commercialization of the product.

1.2 Research objectives

The aim of the present study was to convert digested residues of the olive oil mill industry (OMWW and DOC) to a compact organic soil improver to create a commercial product, which could be stored, transported and spread with common fertilizer spreaders used in agriculture.

2 METHODOLOGY

2.1 Raw material and sample preparation

For the production of a compact soil improver two types of digestates: A) from the co-digestion of OMWW and dairy wastewater, and B) from the co-digestion of DOC and cattle manure, were collected from two anaerobic digestion reactors (100 L) at our laboratories. Both olive mill residues originated from Spanish olive mill factories. The total solid (TS) content of digestate A and B was 1% and 2.9%, respectively. Both digestates were first centrifuged using the Beckman Coulter rotor, model JLA 8.1 at 7000 rpm during 25 minutes. After centrifugation, the solid fraction was dried using a Binder ED 53 Dryer at 100°C until they reached the desired total solid content (TS). Subsequently, the dried sample was milled and homogenized using the shredder Retsch SM100 to eliminate the large particles. The output from the shredder machine was a fined grained material which was optimum for further usage in the pelletizer. The material was then pelletized with the pelletizer Model Helo 7.5 KW (**Erreur ! Source du renvoi introuvable.**).

2.2 General mass and nutrient balance for the process

The water content and plant nutrient concentration were measured before and after each process to compute a complete water and nutrient balance of the whole process. The water content was measured indirectly as TS according to the DIN standard method EN 12880. The plant nutrients were measured according to the methods indicated in TABLE 1.

2.3 Study of the quality of the pellets as fertilizer

The effect of the TS of the material at the input of the pelletizer on the quality of the pellets was studied. For this, pellets were pressed with three dried and milled materials with different TS values. The TS value of the dried and milled digestate was 70%, 80%, 85%. After pelletizing, the pellets were stored at room temperature for 24 weeks to observe their stability against fungi growth. For this, only visual observations were performed.

Moreover, the mechanical stability of the pellets from digestate A und B (initial TS = 85%) was analyzed. Hence, the bulk density (D_{ar}), mechanical durability (DU) and the percentage of fines of the pellets was measured according to the standard method CEN / TS 15149:2006. Additionally, relevant plant nutrients (TN, NH_4, TP, K, Mg, Ca, S, total Fe, B, Na, Mn, Mo, Co, Se), potential plant pollutants (Tl, As, Pb, Cd, Cr, Ni, Hg, Cu, Zn), as well as other parameters (Cu, Zn, Cr) were measured to determine whether the obtained pellets could be admitted as organic fertilizer under the German Fertilizer Ordinance (2008).

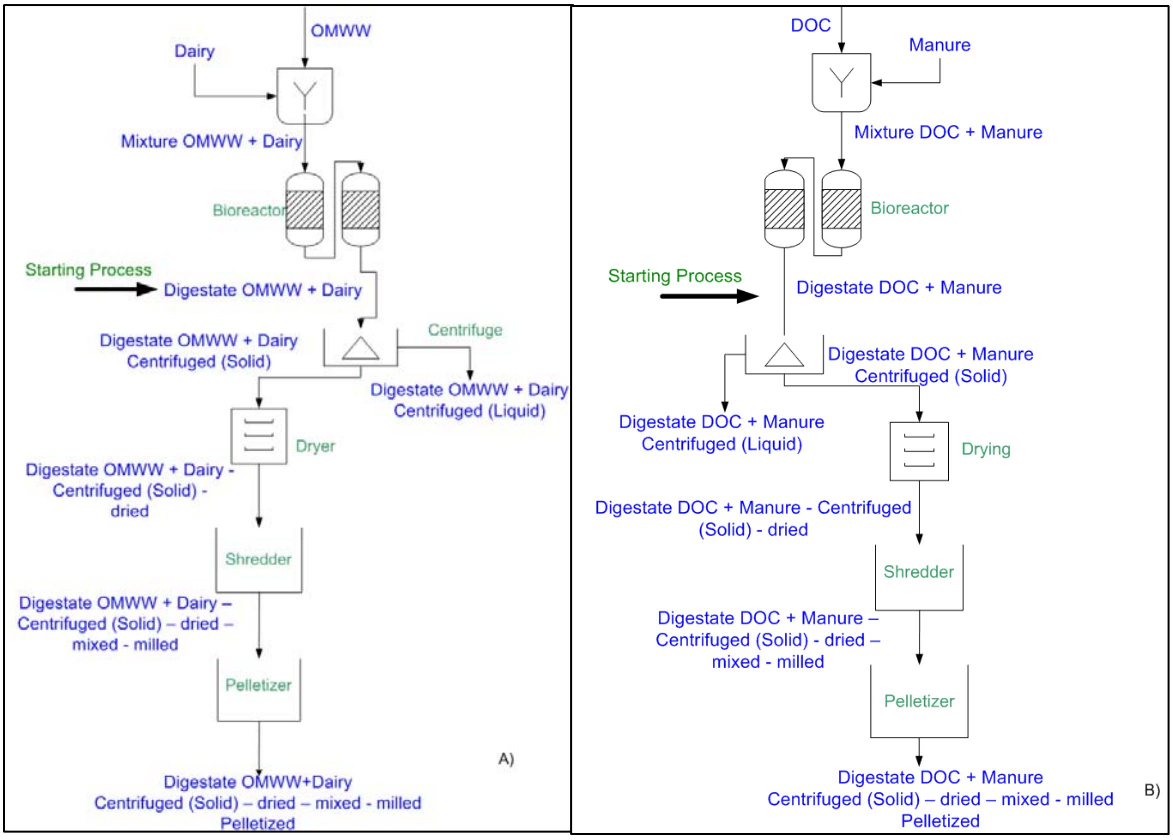

FIGURE 1 **Flow diagram of the process A) Digestate OMWW + Dairy wastewater, B) Digested DOC + Manure**

TABLE 1 **Analytical methods**

Parameters	**Method**
Total Nitrogen (TN), Mercury (Hg)	Kehres and Thelen-Jüngling (2006)
Ammonium Nitrogen (NH_4-N)	DIN 38406-E5-2
Total Phosphorus (P), Total Potassium(K), Magnesium (Mg), Calcium (Ca), Total Sulfur (S), Total Iron (Fe), Arsenic (As), Lead (Pb), Cadmium (Cd), Nickel (Ni), Copper (Cu), Zink (Zn), Cromium (Cr)	DIN EN ISO 11885
Selenium (Se), Thallium (Tl)	DIN EN ISO 17294-2 (E29)

3 RESULTS AND DISCUSSION

3.1 General mass and nutrient balance for the process

The mass balance of the process was similar for digestate A and digestate B (FIGURE 2). During centrifugation approximately 90% of the mass was separated as liquid fraction. The liquid fraction can be used for irrigation or fertigation purposes in agricultural land closed to the biogas plant. The mass of the wet solid fraction (TS = 28-30%) was then further reduced by the drying process to approximately 2 to 3%. Only a very small amount of mass was lost during milling and pelletizing (0.5% of the total digestate mass). Thus, with the process proposed in this paper, it was possible to reduce the mass of the digestates in approximately 98%. This facilitates the transport, storage and application of the product, improving its competitiveness as stable marketable fertilizer.

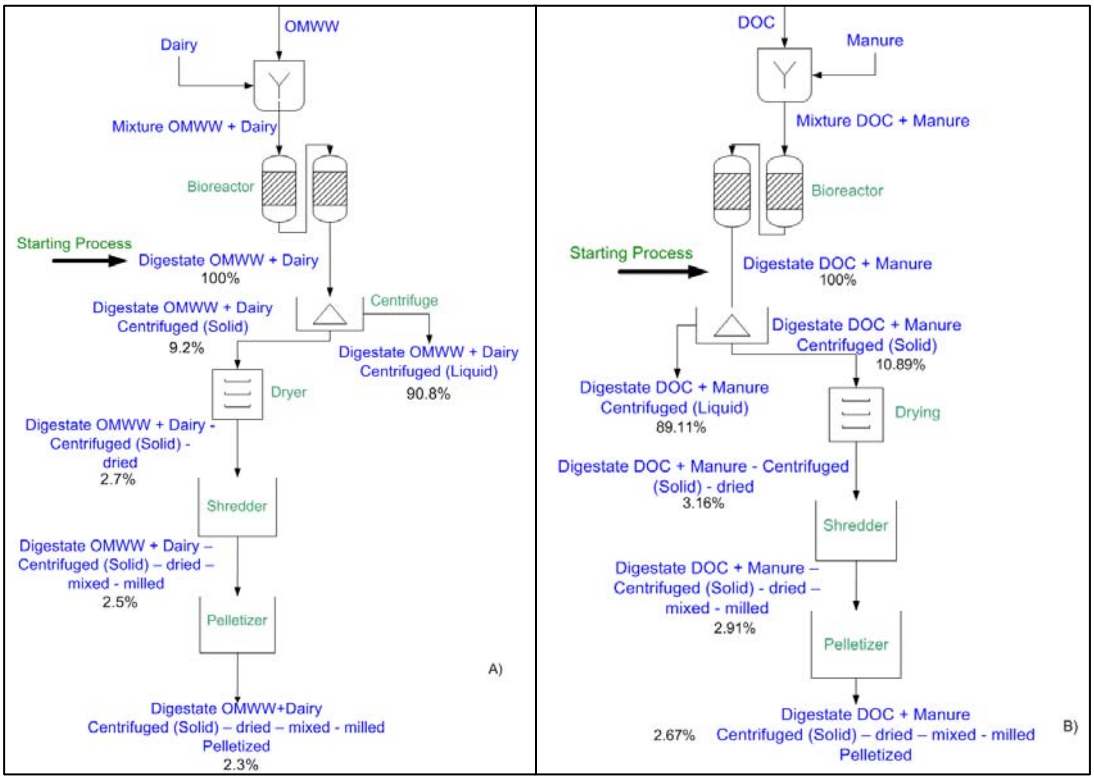

FIGURE 2 **Mass balance of the process A) OMWW + Dairy wastewater, B) DOC + Manure**

The nutrient mass balance for digestate A is presented in FIGURE 3. During centrifugation, 59% of the TN remained in the liquid fraction, while 41% was found in the solid fraction. 83% of the TN found in the liquid fraction was present as NH_4-N and the remaining 17% N was found in the solid fraction. Thus, 75% of the TN in the solid fraction was of organic nature. Moreover, 50% of the TN from the digestate was volatized during drying and pelletizing. In the case of P, 95% of the total P in the digestate remained in the solid fraction after centrifugation without any further change trough the whole process. This means that most of the P contained in the digestate is from organic nature or has precipitated as an inorganic salt. In contrast, the K content remained mostly in the liquid fraction (73%) after centrifugation, while the other 27% of the solid fraction remained the same throughout the process. In addition, after centrifugation, Ca, Mg, S and Fe remained mostly in the solid fraction (92%, 95, 97% and 99.5% respectively). During drying and pelletizing, 29% of S in the solid fraction was volatilized. The other compounds Ca, Mg and Fe remained unchanged throughout the process.

Unlike digestate A, during the centrifugation of digestate B, more than 71% of the TN remained in the solid fraction. 76% of the TN found in the liquid fraction was NH_4-N and the remaining 24% N went to the solid fraction. Hence, more than 88% of the TN in the solid fraction was of organic nature. A large content of organic N in the material is beneficial, since NH_3 loses during the process are minimized. Thus, in contrast to digestate A, only 26% of the TN of digestate B was lost during drying and pelletizing. The P content after centrifugation was 86% in the solid fraction without any further change trough the whole process. This means that most of the P contained in the digestate is from organic nature or precipitated as an inorganic salt. In the case of K, 57% remained in the solid fraction after centrifugation with no further change throughout the process. Most of the Ca, Mg, S and Fe remained in the solid fraction after centrifugation (78%, 75%, 80% and 95% respectively). In addition, during the drying process 23% of S was volatilized. The other compounds Ca, Mg and Fe remained unchanged throughout the process (FIGURE 4).

In order to minimize NH_3 and S loses during the process, NH_3 can easily be recovered from the dryer as ammonium sulfate using a state-of-the-art acid scrubber, and the temperature of the pellet machine could be maintained under 40°C. Moreover, the liquid fraction, which contains a large quantity of nutrients, can be used for fertigation purposes. Alternative, these nutrients can be precipitated as inorganic salts, such as struvite (magnesium ammonium phosphate), K-struvite (potassium magnesium phosphate) or calcium phosphate. Ammonium sulfate and the recovered inorganic salts are also excellent fertilizer for the agriculture and can be applied directly to the soil. In this way, these salts can be easily stabilized, transported, and commercialized as inorganic fertilizers.

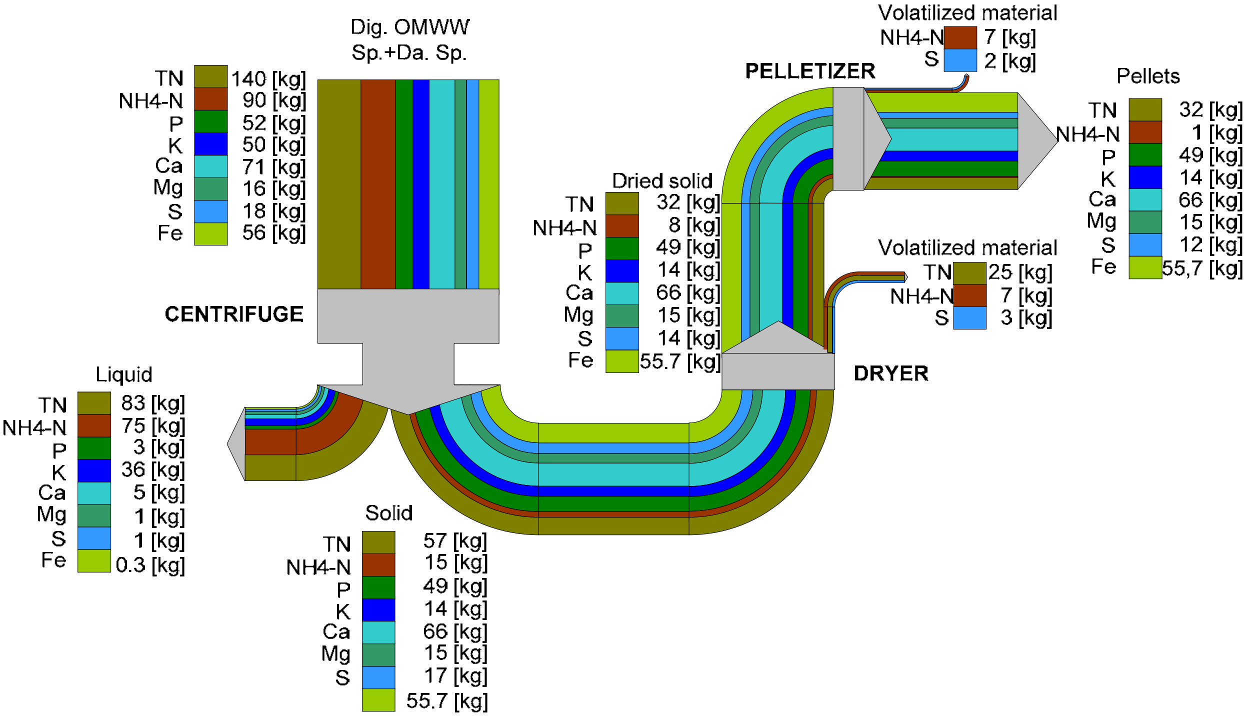

FIGURE 3 **Nutrient mass balance of the process with digestate A**

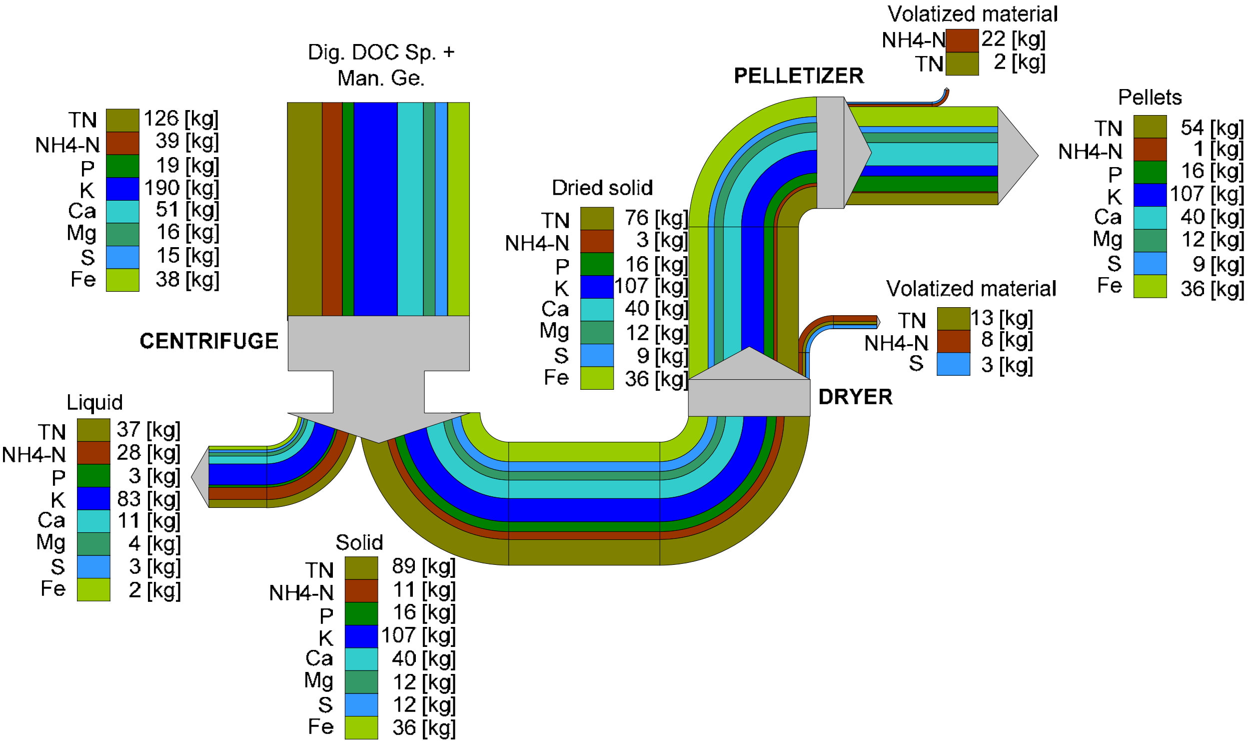

FIGURE 4 **Nutrient mass balance of the process with digestate B**

3.2 Study of the quality of pellets as fertilizer

The effect of the TS of the material at the input of the pelletizer on the quality of the pellets was studied. In the first trial with an initial TS content of the material of 70%, the pellets formed had a brittle structure and low mechanical durability (FIGURE 5A). The TS content of the formed pellets was 76%, which means a TS increase of 4% during the pelletizing process. After 2 days of storage at room temperature, fungal growth was detected in the pellets (FIGURE 5B). Therefore, the initial TS of the material before pelletizing must be higher than 70% to avoid the biological degradation of the material. The second trial was done by increasing the TS content of the sample by 10%. The pellets performance was improved in terms of brightness and mechanical durability. After completion, the TS content of the pellets was 83%. There was a 3% increase of the TS during pelletizing and no fungal degradation was observed even after 24 weeks (FIGURE 5C). However, the best performance was achieved in the last trial. The sample tested had an initial TS content of 85% at the start of the experiment. The pellets presented much more brightness, mechanical durability and the percentage of fine was lower than with the other samples. The final TS of the sample increased in 5% resulting in a TS content of 90% and no fungal growth was observed even after 24 weeks (FIGURE 5D).

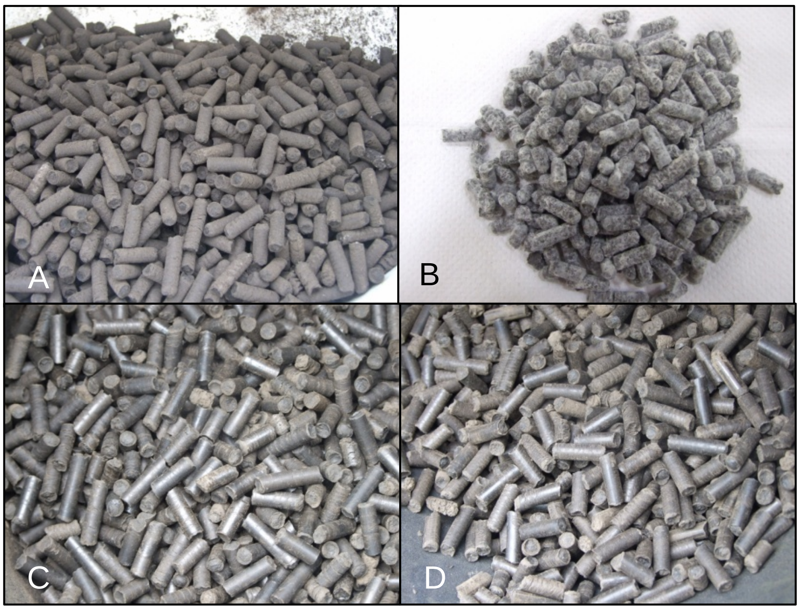

FIGURE 5 **A) Pellets (initial TS = 70%) immediately after the experiment, B) pellets (initial TS = 70%) two days after the experiment, C) pellets (initial TS = 80%) and D) pellets initial TS = 85%**

Since the mechanical properties for digestate pellets are not regulated, the requirements of European Standard EN 14961 for wood pellets were compared to the values obtained with the pellets from digestate A and B with an initial TS content of 85% (TABLE 2). The percentage of fines for both types of pellets is low (2.5-2.6%). Therefore, dusting potential during transportation and application will be minimized. The bulk density was extremely high (≥ 880) in both pellet types, which denotes, that the pellets were very compact. This will spare volume during transportation and storage. The mechanical durability from digestate A und B was 85.1% and 87.8% respectively. Thus, the pellets will not break easily during packing, transport and application.

TABLE 2 **Mechanical properties of the digestate**

Parameter	**Unit**	**Digestate A**	**Digestate B**	**Requirements for wood pellets in EN 14961-2**
Percentage of Fines	%	2.6	2.5	≤1
Bulk Density	kg/m^3	1000	880	≥600
Mechanical Durability DU	%	85.1	87.8	≥97.5

Additionally, relevant plant nutrients, and potential plant pollutants were measured to determine whether the obtained pellets could be admitted as organic fertilizer under the German Fertilizer Ordinance (2008, TABLE 3). The total content of primary and secondary macronutrients (N, P, K, Mg, Ca, S, and Fe) from the pellets produced with digestate A and B was high and comparable with other soil improvers found in the market. Thus, their application might contribute to plant growth. On the other hand, the potential pollutants Tl, As, Pb, Cd, Ni and Hg were below the limit values according to the German Fertilizer Ordinance (2008). Therefore, the produced pellets have a potential value as an organic soil improver and no threat is exposed to the soil or the plant.

TABLE 3 **Mechanical properties of the digestate**

	Unit (Dried Solid)	**Digestate A**	**Digestate B**	**German Fertilizer Ordinance (2008)**	
Plant Nutrients				**Declaration from**	
Total N	% weight	2.3	2.09	0.1	
Total P	% weight	2.3	1.54	0.04	
Total K	% weight	0.71	0.93	0.08	
Total Mg	% weight	1.02	0.81	0.06	
Calcium	% weight	2.99	2.43	3.57	
Sulfur	% weight	0.70	0.61	0.10	
Total Iron (Fe)	% weight	4.31	2.88	0.50	
Total Boro (B)	% weight	0.00280	0.00520	0.01000	
Sodium (Na)	% weight	0.05620	0.03650	-	
Manganese (Mn)	% weight	0.05500	0.04400	0.10000	
Molybdenum (Mo)	% weight	0.00114	0.00058	0.00100	
Cobalt (Co)	% weight	0.00085	0.00061	0.00400	
Selenium (Se)	% weight	0.00023	0.00013	-	
Potential Pollutants				**Limit Value**	**Declaration from**
Thallium (Tl)	mg/Kg	0.3	0.2	1	0.5
Arsenic (As)	mg/Kg	13	7.1	40	20
Lead (Pb)	mg/Kg	59.4	35.4	150	100
Cadmium(Cd)	mg/Kg	1.03	0.64	1.5	1.0
Nickel (Ni)	mg/Kg	79.9	53.4	80	40
Mercury (Hg)	mg/Kg	0.39	0.26	1	0.5
Other Parameters				**Declaration from**	
Copper (Cu)	mg/kg	557	321	500	
Zinc (Zn)	mg/kg	917	551	1000	
Chromium (Cr)	mg/kg	544	335	300	

4 CONCLUSIONS

This study proves that it is possible to convert digested residues from the olive oil processing industry into a compact organic soil improver. Moreover, it was demonstrated that drying and pelletizing digestate are excellent processes to stabilize and reduce the mass of the product (98%) creating a competitive commercial fertilizer. During the nutrient recovery process, especially during drying and pelletizing about 25 to 50% of the N was lost to the atmosphere as NH_3. This N can be recovered as ammonium sulfate during drying using a state-of-the-art acid scrubber. Moreover, the liquid fraction, which contains large quantity of nutrients, can be used for fertigation purposes. Alternative, these nutrients can be precipitated as inorganic salts, such as struvite (magnesium ammonium phosphate), K-struvite (potassium magnesium phosphate) or calcium phosphate. Ammonium sulfate and these inorganic salts are also excellent fertilizer for the agriculture and can be applied directly to the soil. In this way, these salts can be easily stabilized, transported and commercialized as inorganic fertilizers.

According to the experiments performed, the optimal TS value for pelletizing the dried digestate was 85%. Thus, the digestate should be dried only until this TS value in order to achieve a high mechanical durability and to avoid the percentage of fines and fungal degradation. The macro- and micronutrient content of the pellets is high enough to use them as soil improver. Moreover, the potential pollutants were below the maximum limits set by the German Fertilizer Ordinance (2008), exposing no threat for further usage.

5 ACKNOWLEDGEMENTS

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(96) EVALUATION OF TREATED MANURE AS FERTILIZER

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EXECUTIVE SUMMARY

The livestock production has experienced significant changes in the last decades such as intensification and regional conglomeration leading to significant amounts of surplus manure in regions where it cannot be efficiently used. Thus, in those regions the surplus manure has to be treated and disposed losing its benefit as fertilizer and soil conditioner. For this case, alternative technologies are being investigated to use the manure as a valuable resource instead of regarding it as an expensive problem.

The EcoBug Project aims to support the organic agricultural sector by producing a cost-effective, smell-free, solid organic fertilizer made out of digested manure and an organic insect repellent agent against the cabbage root fly that can be applied together with the organic fertilizer as pellets. The objective of this research was to examine the performance of six different EcoBug pellets (Eco 1 – Eco 6) as plant fertilizer in comparison to a standard mineral fertilizer mixture.

The pot experiments were performed with a sub-soil with low content of nutrients. The dry subsoil was fertilized in four replicates with the six different samples. Additionally, a 0-variant without nutrient application and a variant with mineral fertilizers (200 mg N as calcium ammonium nitrate, 40 mg P as triple superphosphate and 110 mg as potassium chloride, accordingly a fertilization of 100 kg N, 20 kg P and 110 kg P per hectare) were investigated as reference samples.

Before starting the pot experiments the nutrient content and pH value from each sample were measured. After one week in every pot 150 mg of German ryegrass (Lolium perenne) was sown (according to 40 kg per hectare). Then, the pots were randomly arranged in a greenhouse with controlled temperature (24 °C by day and 18 ° C by night). The pots were irrigated during the experiment by a Tropf Blumat irrigation system with a weekly control of the water content. At three times after sowing the ryegrass was cut about 3 cm up to the soil surface. The harvested biomass was weighed, dried at 80 °C and weighed again to determine the fresh and dry matter. Then, the plant material was milled up to < 500 µm for the determination of nutrient content.

At the first harvest the plant yield of all variants was low due to the early stage of plant growth. In this stage, a statistic significant difference between the variants could not be observed. At the second harvest the materials Eco 1 – Eco 3 did not show an effect on plant yield, whereas for the materials Eco 4 – Eco 6 an increase of yield was measured. But a statistic significant increase could only be observed for Eco 6. In this harvest, Eco 4 and Eco 6 were comparable with the mineral fertilizer (MF) concerning their effect on plant yield increase. At the third harvest a significant increase of plant yield was noted only for Eco 6 and the MF. The materials Eco 2 and Eco 3 resulted only in a slight increase of plant yield whereas the materials Eco 1, Eco 4 and Eco 5 double the yield.

In comparison to the control, Eco 1 and Eco 3 showed only a slight increase of N uptake at the second harvest while Eco 2 was within the same range as the control. The materials Eco 4, Eco 5, and Eco 6 had a better effect on nutrient uptake. However, a significant increase was only observed for Eco 6 and the MF. These samples increased N-uptake twice to four times, compared to the control. At the third harvest the observations were similar. Eco 4, Eco 5, and Eco 6 showed a considerable increase of N-uptake but again only Eco 6 and the MF increase N-uptake significantly. The effect on N-uptake of Eco 2 and Eco 3 was low. Concerning the pH value, only small variations between the variant could be observed. All of the tested samples increased the pH value of the soil slightly but not significantly. The response of yield and of N-uptake on fertilization varied depending on the sample used. The application of the samples Eco 1 to 5 did not lead to an increase in plant biomass and N-uptake. Only Eco 6 and the MF increased the plant biomass yield and the N-uptake significantly. The sample Eco 6 had a good nutrient availability and was as efficient as the MF.

Due to their nutrient concentration all tested samples conformed to the requirements of the German fertilizer ordinance. Nevertheless, an evaluation of these digested manure samples as organic fertilizers has to be investigated where additional data like trace elements and organic contaminants can be determined.

1 INTRODUCTION

The worldwide sector of organic agriculture includes 37 million hectares of certified land for organic production representing only 0.9% of the global agricultural land (Willer and Killcher, 2012). Organic farming practices have very strict limits on chemical synthetic pesticide and synthetic fertilizer use (European Commission). Therefore, the supply of enough nutrients to ecological crops in a competitive manner is one of the principal challenges that organic farming face at the moment.

Animal manure and digestate can be used as fertilizer in organic farming. However, the use of manure and digestate is principally restricted to locations where they are produced. Due to the high water content of these materials, it is very costly to transport them to other sites with demand for organic fertilizers. In addition, application of the wet manure is a labour intensive and costly process and is normally applied separately from organic insecticides. Such method of separate fertilizing and insecticide application is labour and fuel consuming, resulting in higher costs for ecological farmers.

In order to support the expansion of the organic farming sector and at the same time to make a more efficient use of the manure as valuable resource, alternative technologies must be developed and implemented in this sector.

1.1 Background

The EcoBug Project aims to support the organic agricultural sector by producing a cost-effective smell-free and solid organic fertilizer made out of manure and an organic insect repellent agent against the cabbage root fly that can be applied together with the fertilizer. The overall project objective was to develop all necessary technologies for the production of a combined product in form of pellets ("EcoBug pellets"), including the validation of the insect-repellent activity and the fertilizer effect of "EcoBug pellets" (EcoBug project). The final EcoBug product is an organic product for the farming of cabbage and other Brassica crops. Brassica vegetables (cabbage, savoy cabbage, cauliflowers, broccoli, brussels sprouts, kale) are important crops in Europe. The total amount of area used for the growth of these crops is 452.237 hectares with a total production of 11.5 million tonnes per year, which is 16.5 % of world's total production (FAOSTAT, 2008 data).

1.2 Research objectives

The aim of the present study was to examine six different organic EcoBug pellets (Eco 1 – Eco 6) as for their effect as fertilizer in comparison to a standard mineral fertilizer mixture. Therefore, the effect of the pellets on plant yield, plant nutrient-uptake (nitrogen, phosphorus, and potassium, N/P/K), soil parameters such as pH value, plant available P and K, and total N were determined.

2 METHODOLOGY

2.1 Test materials

Cattle manure from an organic farm (Bioskiva), located in Trondheim, Norway, was collected and anaerobically digested. The digested manure (digestate) is well suited for the production of an organic fertilizer. The digestate was then centrifuged and dried (Stoll et al. 2010). The dried material was then used as a base for the production of the six different EcoBug pellets to be tested as fertilizer. Other ingredients, such as organic binder and plasticisers were added in different concentrations to increase the stability of the pellets. Besides, an additional organic nutrient source was used in two samples (Eco 4 and Eco 6) to increase the amount of N in the pellets. A general characterization of the produced EcoBug pellets was carried out according to the analytical methods described in section 2.3.

2.2 Pot experiments

The effect of the products on plant yield and nutrient-uptake, as well as the effect on soil parameters (pH value, plant available P and K, and total N) were determined in pot experiments with a subsoil with low content of nutrients (Weinfurtner et al., 2009). The subsoil was sampled from the Bv-horizon of the German RefeSol 01-A, originated in Hagen, Lower Saxony. Before the start of the pot experiments the nutrient content and pH value from the soil was determined (TABLE 1). Six kg of a dry soil were fertilized with 200 mg N in form of the six materials in four replicates for every product and additionally for a 0-variant without nutrient application and a variant with mineral fertilizers (200 mg N as calcium ammonium nitrate, 40 mg P as triple superphosphate and 110 mg as potassium chloride, according to a fertilization of 100 kg N, 20 kg P and 110 kg P per hectare).

TABLE 1 **Characteristics of test soil**

Parameter	**Unit**	**Hagen, subsoil**
Sand	g/kg	810
Silt	g/kg	170
Clay	g/kg	20
pH (0,01 M $CaCl_2$)	-	5.24
C (total)	g/kg	3.24
C (organic)	g/kg	3.21
N (total)	mg/kg	260
K_{CAL}	mg/kg	87
P_{CAL}	mg/kg	21

After fertilization the soils were adjusted up to 70 % of water holding capacity (WHC). After one week in every pot 150 mg of German ryegrass (Lolium perenne) was sown according to 40 kg per hectare (FIGURE 1). After sowing, the pots were randomly arranged in a greenhouse (FIGURE 2) with controlled temperature (24°C by day and 18°C by night). The pots were irrigated during the experiment by a Tropf Blumat irrigation system with a weekly control of the water content.

FIGURE 1 **Sowing of German ryegrass**

FIGURE 2 **Pot experiment**

At three times after sowing, the ryegrass was cut about 3 cm up to the soil surface. TABLE 2 summarizes the corresponding time schedule of the pot experiment. The harvested biomass was weighed, dried at 80°C and weighed again to determine the fresh and dry matter. After that the plant material was milled up to < 500 µm for the determination of nutrient content.

TABLE 2 **Time schedule of pot experiments**

Cultivation measures	**Date**
Fertilization of subsoil	May 25th, 2011
Sowing of German ryegrass	June 1st, 2011
First harvest	June 30th, 2011
Second harvest	July 13rd, 2011
Third harvest	August 4th, 2011

2.3 Analytical methods

Total nitrogen in soil and plant material was determined according to “VDLUFA Methodenbuch” with a Kjeldahl extraction by using a Büchi K 370 Auto Unit. Total carbon and organic carbon was detected according to DIN ISO 10694 by using a Ströhlein C-MAT 5500. The plant available P and K was determined in the calcium-acetate-lactate extract (CAL) according to “VDLUFA Methodenbuch”. For the detection a Thermo Solar AAS (K) and a Varian Cary 300 (P) were used. The pH value was determined by using a WTW pH meter Series 196 according to DIN ISO 10390.

The aqua regia extraction for determining the total content of P, Fe, Ca, K and Mg was performed according to DIN ISO 11466. For detection of the elements a Thermo Intrepid II XSP ICP-OES and a Thermo Solaar AAS (for K determination) were used. For the plant analysis 0.5 g dried plant material was weighed in ceramic pots and incinerated at 500°C for at least 2 hours. After cooling the ash was moistened with few drops deionised water. 10 ml of 2 M HCl were added carefully and then the pots were slowly heated up to the boiling point. After cooling the content of the pots was given in 100 ml flasks, filled up with deionised water and filtered over a paper filter. The remaining filtrate was used for determination of P and K.

3 RESULTS AND DISCUSSION

3.1 Test materials

The main characteristics of the test pellets are summarized in TABLE 3. The pH value varied only within a small range of 5.96 to 6.25. Carbon (C) was present principally as organic C with small variations between the tested materials. A larger range of values is observed for the main nutrients N, P and K. The material Eco 4 showed only a slightly higher N-concentration while Eco 6 has an N-concentration two times higher than the other test materials. The total P concentration was extremely high by Eco 6; however, their P-availability for the plant (P_{CAL}) was the lowest. In terms of P-available (P_{CAL}) for the plant, only Eco 1 showed a very high P-availability (almost 100% of the total P), whereas for the other materials the plant availability ranged between 8% of total P for Eco 6 and 65% for Eco 5. This high availability by Eco 5 was probably due to the small particle size of the ground manure (1 mm). The measurement of K-available (K_{CAL}) showed that about 60% of the total K content is plant available in all samples. The concentration of Ca, Fe and Mg was very similar in all samples, denoting that none of the additives had a significant effect on the 6 samples.

TABLE 3 **Characteristics of test materials**

Parameter	Unit	Eco 1	Eco 2	Eco 3*	Eco 4	Eco 5**	Eco 6
pH (0,01 M $CaCl_2$)	-	6.25	6.36	6.25	6.23	5.96	6.08
C (total)	g/kg	397.8	409.4	402.2	401.3	397.4	441.5
C (organic)	g/kg	397.8	409.0	402.2	400.9	397.4	441.5
N (total)	g/kg	22.1	23.3	23.1	26.2	22.8	55.0
P (total)	mg/kg	5,252	10,870	8,636	10,674	8,807	61,007
P_{CAL}	mg/kg	5,232	5,671	5,212	5,376	5,730	4,820
K (total)	mg/kg	14,723	17,859	17,510	14,531	14,431	12,427
K_{CAL}	mg/kg	10,898	10,968	10,712	10,466	9,004	7,078
Ca (total)	mg/kg	14,996	17,669	16,513	14,879	16,899	13,865
Fe (total)	mg/kg	2,773	3,658	3,131	3,004	3,726	2,705
Mg (total)	mg/kg	9,223	13,061	12,603	11,946	12,937	10,394

* coated pellets

** ground manure < 1 mm

3.2 Plant yield

The FIGURE 3 represents the average of four replicates (columns) and the standard deviation (bars) of the three different harvest results. In addition, a difference in significance compared to 0-control is remarked ("*"). At the first harvest the yield of all variants was low due to the early stage of plant growth. At the second harvest the materials Eco 1 – Eco 3 did not show an effect on yield whereas for the materials Eco 4 – Eco 6 an increase of yield was noted. But a statistic significant increase could only be observed for Eco 6. Eco 6 was comparable with the mineral fertilizer (MF) in their effect on yield increase. A significant increase of yield is noted for Eco 6 and the MF at the third harvest. The materials Eco 2 and Eco 3 resulted only in a slight increase of yield whereas the materials Eco 1, Eco 4 and Eco 5 double the yield.

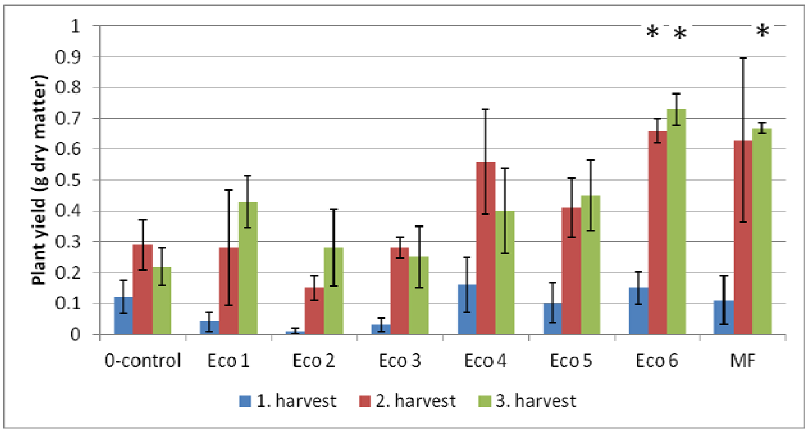

FIGURE 3 **Biomass yield of first, second and third harvest**

3.3 Plant nutrient-uptake

Due to the low yield of the first harvest it was not possible to measure the nutrient content of the single replicates so that the four replicates of every variant are pooled to one sample. But this does not allow a statistic analysis. Thus, the nutrient uptake is presented only for the second and third harvest in the following sections.

3.3.1 Nitrogen

FIGURE 4 shows the results concerning N-uptake. In comparison to the 0-control, Eco 1 and Eco 3 showed only a slight increase of N uptake at the second harvest. Eco 2 was within the same range as the 0-control. The materials Eco 4 to Eco 6 have a better effect on N-uptake even if a significant increase is only observed for Eco 6 and the MF. These materials increased the N-uptake twice to four times, compared to the 0-control. At the third harvest the observations are similar. Eco 4 to 6 and also Eco 1 showed a considerable increase of N-uptake but again only Eco 6 and the MF increase significantly the N-uptake. The effect on N-uptake of Eco 2 and Eco 3 was low.

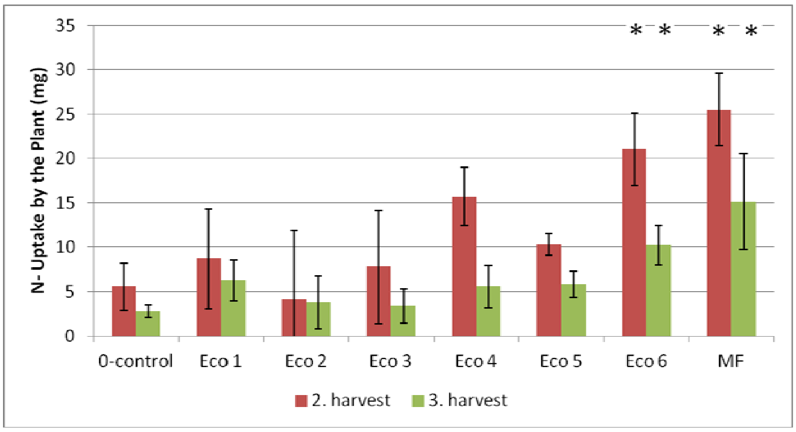

FIGURE 4 **N-uptake at second and third harvest**

3.3.2 Phosphorus

FIGURE 5 summarizes the results concerning P-uptake. At the second harvest a slight but not significant increase of P-uptake was observed only for the MF. At the third harvest the MF, Eco 1 and Eco 3 to 6 showed a slight increase of P-uptake but a significant increase is only observed by Eco 6 and the MF.

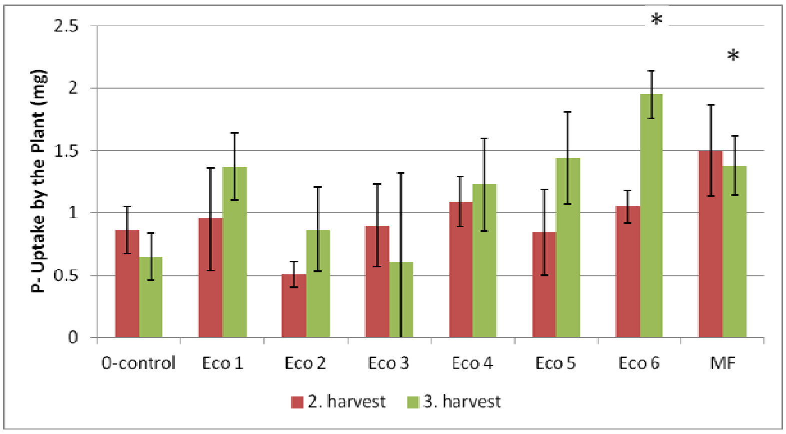

FIGURE 5 **P-uptake at the second and third harvest**

3.3.3 Potassium

FIGURE 6 shows the results concerning K-uptake. Similar to P-uptake the K-uptake by the plant was not influenced by Eco 1 to 3 at the second harvest. Eco 4 to 6 and the MF result in a slight but not significant increase of K-uptake. At the third harvest the MF, Eco 1 and Eco 3 to 6 caused a slight increase of K-uptake but a significant increase is only observed by Eco 6 and the MF.

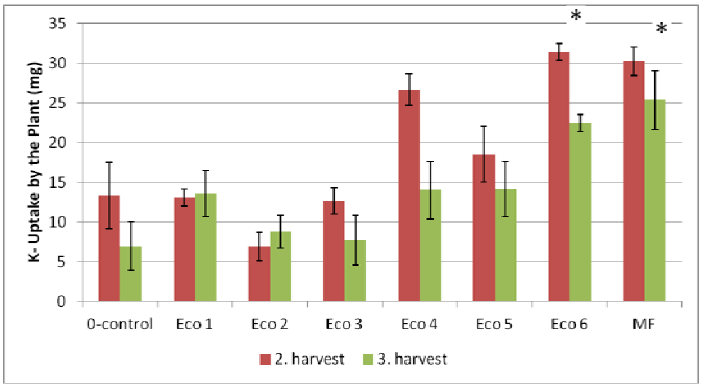

FIGURE 6 **K-uptake at the second and third harvest**

3.4 Soil parameters

After the third harvest soil samples were taken from every replicate and the pH value and the nutrient status were determined and are summarized in TABLE 4.

For the pH value only small variations between the variants was measured. The pH value of the soil increased slightly but not significantly in all tested materials.

Concerning the nutrient concentrations, the soil N concentration is not influenced by the fertilization. But for the plant available P and K an effect of the tested materials can be noted. All materials induced an increase of P_{CAL} up to 50 % of the unfertilized control. This increase was significant only for Eco 5. Even though Eco 6 had the highest P concentration the effect on soil P is low, perhaps due to the low content of available P in this material.

The variants Eco 1 to Eco 5 caused an increase in plant available K in the soil. With exception of Eco 4 the increase was significant compared to the 0-control. A reduction of plant available K was observed for Eco 6.

TABLE 4 **Average of total N (g kg-1 dry matter). P_{CAL} and K_{CAL} (mg kg-1 dry matter) and pH in soil after third harvest**

Sample-Id	**pH**	**Std_{dev}**	**N-conc.**	**Std_{dev}**	**P_{CAL}**	**Std_{dev}**	**K_{CAL}**	**Std_{dev}**
	-	**-**	**g kg^{-1}**	**g kg^{-1}**	**mg kg^{-1}**	**mg kg^{-1}**	**mg kg^{-1}**	**mg kg^{-1}**
0-control	5.17	0.08	0.26	0.02	8.3	0.45	67.5	4.52
Eco 1	5.32	0.03	0.25	0.01	11.7	0.45	85.2	10.33
Eco 2	5.29	0.08	0.27	0.00	10.9	0.19	88.0	1.88
Eco 3	5.31	0.03	0.27	0.00	11.9	0.56	90.3	4.53
Eco 4	5.21	0.09	0.27	0.04	11.0	0.25	76.6	4.36
Eco 5	5.25	0.07	0.26	0.01	12.6	1.41	83.8	1.30
Eco 6	5.25	0.06	0.26	0.02	9.2	0.20	61.8	1.15
MF	5.15	0.08	0.26	0.02	9.3	0.67	89.5	5.62

4 CONCLUSIONS

In this research, six different produced organic EcoBug pellets (Eco 1 to Eco 6) were characterized and evaluated as for their effect as fertilizer in comparison to a standard mineral fertilizer mixture. The N, P, K concentration of the tested pellets was similar, except for the N and P concentration of Eco 6. Eco 6 had a N concentration, which was twice as high as for the other materials and a very high total P content (6 to 12 times larger than the other materials). However, the P-available in Eco 6 is similar to the other materials. Due to their nutrient concentration the tested materials conformed to requirements of the German fertilizer ordinance. Nevertheless, a complete evaluation of the tested materials as organic fertilizers was not possible because additional data as for the content of trace elements and organic contaminants must be determined.

The response of yield and of nutrient uptake on fertilization varied depending on the material used. The application of the material Eco 1 to 5 did not lead to a significant increase in plant biomass and nutrient uptake. Only Eco 6 and the mineral fertilizer (MF) increased significantly the plant biomass yield and the nutrient uptake. This means, that the material Eco 6 has a good nutrient availability and is as efficient as the MF. Therefore, it is possible to obtain an organic EcoBug fertilizer with high nutrient content, producing high plant yield. The lower content of plant P-available of Eco 6, in addition to the higher plant extraction of P and K from soils explains why the soil contents of P_{CAL} and K_{CAL} are lower in the experiments with Eco 6 than in the experiments with the other materials.

5 ACKNOWLEDGEMENTS

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(145) COMPOSTED AND VERMICOMPOSTED CATTLE MANURE EFFECT NUTRIENT SUPPLY AND LETTUCE GROWTH

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EXECUTIVE SUMMARY

The nutrient supplying potential of vermicompost and compost of the same substrates was tested using lettuce in a greenhouse experiment. Fifteen treatments were compared. Four treatments were vermicompost and compost prepared from beef manure and dairy manure. Four additional treatments consisted of vermicompost from a 75:25 (vol.) ratio of shredded cardboard and beef manure, 50:50 vermicompost:compost blend for each manure source, and an unamended control. Each amendment was applied at rates of 1.25% and 2.5% (w/w) to either a low fertility sandy soil or a moderately fertile loamy soil. Lettuce yields and nutrient uptake were measured for the 2.5% application rate (only). Soil nutrient supply rates were measured during a 75day period using ion exchange membranes in a separate set of pots using the 2.5% application rate. Vermicompost produced larger lettuce yields and supplied more N, P, K, S, and Mg than compost. There was no added benefit from the mix of composted and vermicomposted manure. Vermicompost produced from the cardboard and manure mix was the poorest amendment used, reducing lettuce productivity in the low fertility soil. The source of manure was found to have a significant influence on N supply and availability and lettuce growth.

1 INTRODUCTION

Vermicomposting may provide benefits in comparison to conventional composting, including increased mineralization of nutrients, reduced capital costs, and an ability to digest materials that are difficult to compost thermophilically (Edwards 1995). However, few direct comparisons between vermicomposted and composted manure have been conducted in relation to nutrient availability and plant growth (Lazcano et al. 2008).

The ratio of C to N has been used as an indicator of maturity and the quality of composts. It is generally recognized that a C:N ratio of 25 to 35 is optimal for aerobic microbial degradation of organic materials. Ndegwa and Thompson (2000) reported that a C:N ratio of 25 resulted in the most stable end product, best value as a fertilizer, and lowest potential for environmental pollution of vermicomposted biosolids. When earthworms were added to organic ‘wastes’, decomposition was enhanced, as indicated by the decrease in the C:N ratio (Atiyeh et al. 2000; Sudha & Kapoor 2000; Pattnaik & Vikram Reddy 2010); however, Warman and AngLopez (2002) wrote that C:N, C:P and pH of vermicompost could not be used as indicators of maturity. Regarding available P, earthworms can boost the mineralization rate of P from organic wastes allowing larger amounts of readily available forms of P to be maintained (Ghosh et al. 1999). Warman and AngLopez (2002) also found the amounts of water soluble P in vermicomposts varied with the source of feedstocks and duration of vermicomposting.

The potential for using earthworms to decompose paper and cardboard waste has been previously reported (Warman & AngLopez 2002; Gajalakshmi et al. 2001; Edwards 1995). When vermicomposting was used for the stabilization of waste paper sludge, the final product resulted in substantially more water soluble nutrients, such as nitrate, compared to a product obtained with traditional thermophylic composting (Short et al. 1999). Hammermeister et al. (2006) evaluated vermicasts (VC) produced by vermicomposting sheep manure, peat moss and cardboard and other organic amendments for lettuce and orchardgrass production . They found lettuce yields were greater using the vermicasts compared to compost, feather meal or alfalfa meal, but yields from VC were less for orchardgrass when comparable rates of the other amendments were used.

Studies have determined there is an optimum combination of vermicasts and sand, growth media or compost for plant growth. Using radishes, seed germination and plant development were significantly affected by different vermicompost and sand mixture contents (Buckerfield et al., 1999), increasing vermicompost concentrations resulted in reduction of seed germination but increased plant growth. These results are very similar to those reported by Warman and Anglopez (2010) using radish, marigold and upland cress. Ali et al. (2007) showed that lettuce biomass production was optimal with a 20/80 (v/v) worm casts and green waste-based compost blend, whilst pure worm cast and green waste compost yielded poor growth. Loncaric et al. (2005) also extensively analyzed vermicompost produced from cattle manure using *Eisenia* fetida and found lettuce growth was comparable to commercial potting media.

There has been little work directed at nutrient release kinetics from the use of vermicomposts; Hammermeister et al. (2006) and McGinnis et al. (2010) are the only two publications we have found. The primary objective of the following research was to evaluate the nutrient supply rates of composts and vermicomposts using ion exchange membranes, and determine the fertilizing potential of the amendments for lettuce. Specifically, the research was designed to a) compare conventionally-produced compost from beef and dairy manure with vermicompost made from the same feedstocks, and vermicompost made with cardboard fibre and beef manure, b) determine if vermicomposts accelerate early plant growth and/or development, and c) determine if there is any benefit associated with combining conventional compost with vermicompost from the same feedstocks.

2 METHODOLOGY

The experimental units (pots) were arranged in a randomized complete block design with four blocks. Two sources of manure were used for conventional thermophylic composting and vermicomposting (Table 1). The first source was a one-year-old pile of beef manure (straw-faeces), which was composted conventionally (CM1), vermicomposted (VCM1), or combined with shredded cardboard in a 75:25 mix of cardboard to manure (fresh volume basis) (VCM1+F). A 50:50 mix of CM1 and VCM1 produced the Blend1 treatment. The second source was fresh dairy manure composted conventionally (CM2) or vermicomposted (VCM2). A 50:50 mix of the CM2 and VCM2 produced the Blend2 treatment. For reference purposes, additional nonreplicated pots were amended with inorganic fertilizer, a commercially available vermicompost, and thermophylic poultry manure compost. The beef manure came from and was composted conventionally at the NSAC farm; the dairy manure came from and was composted conventionally at Kipawa Holsteins. The Good Earth Organic Resources Group Ltd produced the vermicompost at their facility. The manures for vermicomposting were placed in separate bins about 2.4 m by 1.2 m and approximately 30 cm in depth. Worms (*Eisenia fatida)* were added to the top of the bins and allowed to migrate. For the VCM2, manure was added periodically and a layer of straw was placed on the surface of the bins but not incorporated. Samples were taken every month and pH, moisture content, conductivity, and worm biomass was measured and recorded. Vermicomposting continued for approximately four months.

The amendments were applied at rates of 1.25% or 2.5% of the growth medium on a dry weight basis; each pot contained 3.92 kg of air-dried soil. A control treatment with no organic amendments was also included. Eight replicated treatments were combined with either a Brookside loamy soil of medium fertility (140 P, 123 K, 514 Ca, 101 Mg, 21 S) or a Saywood sandy soil of extremely low fertility (7 P, 6 K, 118 Ca, 4 Mg, 11 S)(all units as mg kg^{-1}). Four replicates were used for lettuce growth trials, the remainder were left unplanted for nutrient supply rate trials as described below. Lime was applied to the Saywood soil several weeks before applying amendments to bring the soil pH to 6.5. The soil moisture content of all pots was maintained at 75% of field capacity, the growth room temperature was maintained at 22^{o}C±2/16^{o}C±2 (day/night), the day length was 14 hours, and the light intensity at the pot surface was 120±20 µE $s^{-1}m^{-2}$.

Lettuce (cv. Grand Rapids) was seeded directly into the pots two weeks after amendment applications. Seedlings were allowed to establish for approximately 14 days and subsequently thinned to 28 plants/pot. Lettuce plants were sampled at three, one-week intervals, and finally at two months after planting by cutting seven plants at the soil surface. Fresh and oven dry weights (48 hours at 57^{o}C) were recorded for each sampling period.

A soil sample was collected from each pot at planting and stored at 4^{o}C for later mineral N determination by 2.0 M KCl extraction; extracts were frozen until analyzed by automated colorimetry (Maynard & Kalra 1993). After harvest, soil samples were collected from the pots, a subsample was air-dried and the pH measured (1:2 soil:water). Lettuce tissue samples from all sampling periods (2.5% rate only) were combined and ground to <1mm. The total N content of the lettuce and amendments was determined using a LECO® CNS Analyzer; the total content of other nutrients was determined by nitric acid digestion (Zheljazkov & Warman, 2002), and analyzed using Inductively Coupled Argon Plasma Emission Spectroscopy (ICAP). Plant nutrient uptake was calculated by multiplying dry weight yield by nutrient concentration.

Ion exchange membranes (Plant Root Simulator (PRS)™ probes,Western Ag Innovations, Saskatoon, SK, Canada) were used in unplanted pots to assess nutrient availability or supply *in-situ* with minimal disturbance (Qian & Schoenau 2002; Hammermeister et al. 2006). Each unvegetated pot had a cation and an anion probe inserted immediately after soil and amendments were mixed. Soil moisture was brought to 75% of FC and maintained at this level throughout the duration of the experiment. Probes were removed and replaced in exactly the same location after 4 days and then at one-week intervals for 10 measurement periods (a total of 75 days). After removal, the PRS ™ probes were cleaned with deionized water and eluted for one hour using 0.5 M HCl. Western Ag Innovations analyzed the eluate for mineral N using automated colorimetry, other nutrients were analyzed using ICAP.

Statistical Analysis: Data was analyzed as a randomized block design using SPSS™ software (Norusis, 1993). The two soils were analyzed separately. Lettuce dry and fresh weights were analyzed as a two-way factorial (treatments, rates). Significant treatment x rate interactions was used for mean comparisons among all treatment combinations, including the control. Plant nutrient uptake and soil nutrient supply rate data was collected for the 2.5% application rate, only; therefore, analysis was a one-way Block design. Missing nutrient uptake data (for one sample) was calculated as per Steel and Torrie (1980). Tests for significant differences among treatments used Fisher's Protected LSD when the ANOVA indicated significant difference at the 5% level. Pearson correlation coefficients between nutrient supply rates, lettuce yield and nutrient uptake were calculated using SPSS™ (Norusis, 1993).

3 RESULTS AND DISCUSSION

3.1 Amendment Characteristics

There was a larger difference in the %C and %N of the CM1 and VCM1 amendments than between the CM2 and VCM2 amendments, which were nearly the same (Table 1). The vermicompost, therefore, did not have a lower C:N ratio than conventionally composted manure, as reported by Sudha and Kapoor (2000) and Pattnaik and Vikram Reddy (2010) As a result, the total amount of N added to each pot of the VCM1 treatment was twice as high as that of the CM1 treatment and 1.6x higher than the Blend1 treatment. The VCM1+F treatment had the greatest C:N ratio, due to the cardboard, which resulted in a substantially lower rate of N application than the other treatments. The dairy manure-based treatments were very similar in total N content and total N application rates. Relative to the conventionally composted manures, the vermicomposts contained less total K and Na, slightly more P and S, similar Ca content, while the Mg content varied depending on the manure source. These results are in contrast with Sudha and Kapoor (2000) who found no significant difference in nutrient content between vermicompost and compost but are more similar to Pattnaik and Vikram Reddy (2010) who found the nutrient content of vermicomposts from three earthworm species were more than that of green waste compost and their respective substrates.

TABLE 1 Description of treatment amendments and their elemental analyses

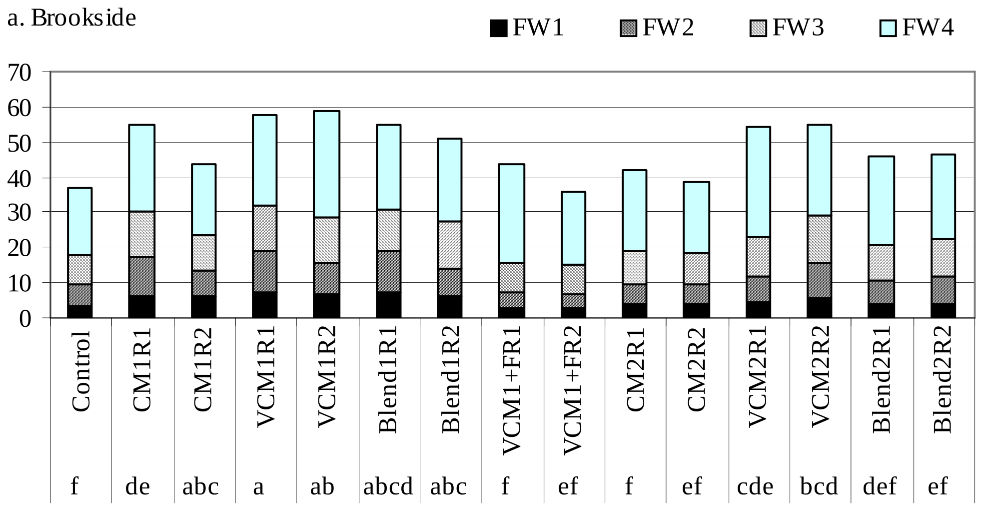
Treatment	Composting Method	C	N	P	K	S	Ca	Mg	Na
		%	%	g kg^{-1}					
CM1	Composted BM	15.5	1.2	5.3	18.0	2.5	20.0	6.6	1.0
VCM1	Vermicomposted BM	34.6	2.4	6.5	9.8	5.7	19.0	8.8	0.5
Blend1	50% CM1:50%VCM1	20.5	1.5	4.7	12.4	2.9	14.5	6.4	0.7
VCM1+F	25% BM:75% cardboard	24.8	1.0	2.4	5.3	2.8	18.9	5.7	0.5
CM2	Composted DM	37.0	2.0	7.6	35.4	5.9	27.0	7.9	4.0
VCM2	Vermicomposted DM	37.4	2.1	8.2	26.8	6.0	22.1	6.5	2.6
Blend2	50% CM2:50% VCM2	36.0	2.0	8.6	35.2	6.5	26.4	8.0	3.8

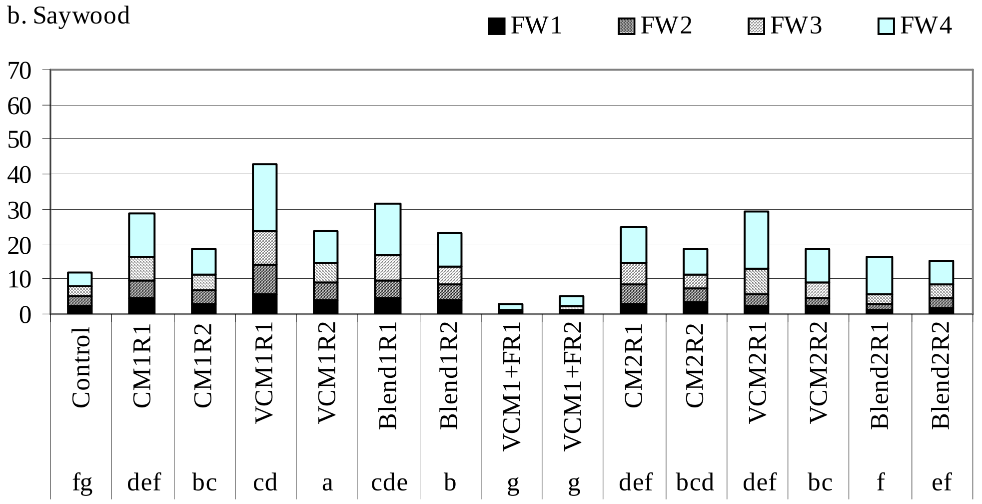
[a] CM1 – composted beef manure (BM); CM2 - composted dairy manure (DM)
[b] VCM1 – vermicomposted BM; VCM2 – vermicomposted DM
[c] Blend 1 or 2 – 50:50 mix of CM and VCM treatments of a manure source
[d] VCM1+F - Beef manure and shredded cardboard fibre at a 25:75 mix on a volume basis

3.2 Lettuce Yield

Fresh weights from the VCM treatments were higher than from the CM treatments of the same manure source except for the dairy manure applications to the Saywood soil (Figure1). Mixing vermicomposted (VCM) and composted (CM) manures provided no significant yield benefit; however, in some cases, depending on the treatment rate, yields increased compared to the CM or VCM treatment. The VCM1+F treatment did not increase yields above those of the Control for either soil, and was significantly lower than that of other treatments. In other field trials, that used a more mature fibre-based vermicomposts, we observed increased yields in response to treatments (unpublished data). Treatments with the composted beef manure resulted in generally higher yield than the same treatments using the dairy manure, although differences were not always statistically significant. Doubling the amendment rate from 1.25 to 2.5% usually decreased yields in the Saywood soil (Figure 1), while yield differences between the two rates were slight for the Brookside soil (Figure 1). The lettuce sampled at the earliest growth stages showed the same trends as the total fresh weights (Figure 1); consequently, the VCM treatments provided no early growth advantages compared to the CM treatments.

Figure 1 **Cumulative fresh weight (g pot^{-1}) of lettuce for four sampling periods (FW1 – FW4) for treatments applied at 1.25% (R1) and 2.5% (R2) to Brookside (a) and Saywood (b) soils**



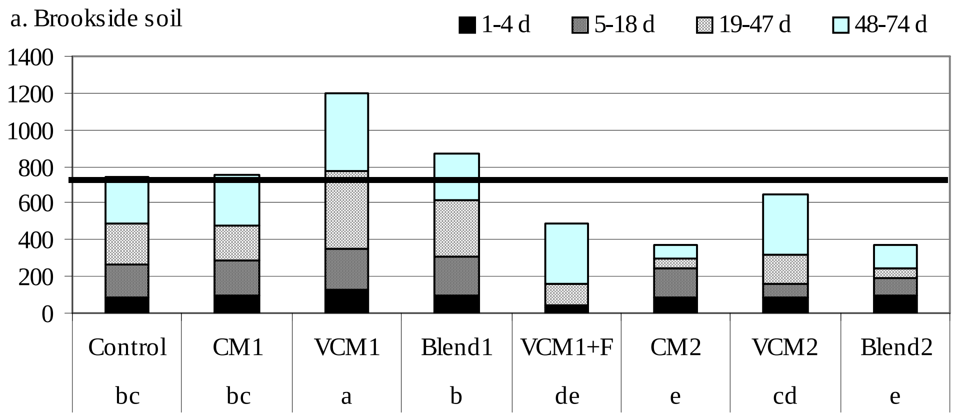


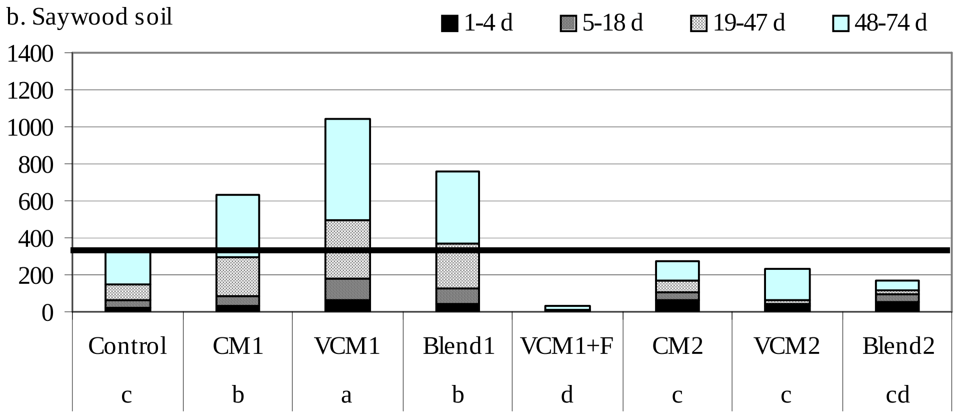
*Fresh weights for each soil followed by the same letter are not significantly different ($P<0.05$)

3.3 Nutrient supply and uptake by the lettuce

Cumulative NO_3^--N supply (over 74 d) in the Control treatment was twice as high in the Brookside soil as in the Saywood soil (Figure 2), which resulted in higher cumulative NO_3^--N supply in all of the amended treatments of the Brookside soil. After subtracting the NO_3^--N contribution of the Control soil, however, the net supply of NO_3^--N of the amended treatments was generally higher in the Saywood soil. This may be related to higher mass flow of the NO_3^--N in the soil water of the sandier Saywood soil than the Brookside soil and the lower initial available N in the Saywood soil. The NO_3^--N supply rate measured the first four days (1-4 d) ranged from 5 to 24% of the total NO_3^--N supply depending upon treatment (Figure 2). This NO_3^--N was most likely available in the soil and/or amendments in mineral form when the amendments were applied. For the 74 d sampling period, NO_3^--N was immobilized in the CM2, VCM2, Blend2 and VCM1+F treatments (Figure 2). There was no significant N mineralization in the CM1 treatment to the Brookside soil, however, there was significant N mineralization in the VCM1 Brookside soil and the VCM1 and CM1 treatments to the Saywood soil.

Figure 2. Cumulative NO_3^--N supply (ug/10 cm^2) for four measurement periods for treatments applied at the 2.5% rate to Brookside (a) and Saywood (b) soils. The black bar in each graph shows the value of the Control.





*Cumulatve NO_3^--N supply for each soil followed by the same letter are not significantly different ($P<0.05$)

In a comparison of composting methods, the NO_3^--N supply was significantly higher in the VCM1 treatment than the CM1 treatment in both soils, whereas only VCM2 was significantly higher in nitrate supply than CM2 in the Brookside soil (Figure 2). The difference between the manures was somewhat expected since the C:N ratios were lower in the CM1 and VCM1 treatments. Our results indicate the VCM1+F treatment would make a poor fertility source unless it was supplemented. For example, Arancon et al. (2004) supplemented vermicompost derived from recycled paper and supermarket food waste with inorganic fertilizers to balance the initial unavailability of macronutrients, especially N. Yet, the VCM1+F treatment supplied proportionally the same amount of N as the VCM1 treatment to the Saywood soil, but not to the Brookside soil (data not shown). Furthermore, Short et al. (1999) reported increased N availability with vermicomposting of papermill sludge.

No significant differences due to treatments were measured in total lettuce N uptake in the Brookside soil, however, all amended treatments had higher numerical uptake than the Control (Table 2). The lettuce showed the classic pattern of the dilution effect, that is lower tissue N contents for the VCM1 and Blend 1 (data not shown) with higher yields. With the Saywood soil, total N uptake generally followed the fresh weight trend (Table 2), as there were few differences between treatments in tissue N concentration.

TABLE 2 Lettuce dry weight yield and total nutrient uptake (mg pot^{-1}) amended at the 2.5% rate[a]

	Yield	N	P	K	S	Ca	Mg
Brookside							
Control	3216c	27.7a	6.8c	141d	14.8c	24.9d	7.3c
CM1	4649b	39.6a	11.8ab	234ab	21.3b	32.5abc	9.9b
VCM1	5618a	40.5a	13.7a	253a	22.2ab	36.8a	11.7a
Blend1	5026ab	38.0a	12.5a	240ab	20.6b	34.7b	10.7ab
VCM1+F	3497c	37.9a	9.1bc	181cd	13.7c	28.6bcd	7.1c
CM2	3398c	34.8a	10.3bc	192cd	20.0b	28.1cd	8.1c
VCM2	4608b	39.5a	13.4a	255a	25.0a	34.3abc	10.3ab
Blend2	3661c	33.4a	11.9ab	206bc	21.9ab	29.3bcd	8.3c
Saywood							
Control	1333de	7.7e	2.3d	44c	3.8c	8.8c	2.2a
CM1	2306bc	17.3bc	7.3bc	113b	10.4bc	20.9abc	4.8a
VCM1	3598a	28.0a	11.9a	174a	13.7ab	29.8a	7.5a
Blend1	2942ab	21.5b	11.1ab	113b	19.2a	34.5a	8.7a
VCM1+F	391f	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
CM2	2160c	16.1cd	6.2cd	105b	10.6abc	22.6abc	4.9a
VCM2	2026cd	21.2bc	7.7bc	127b	12.8ab	22.8ab	5.1a
Blend2	1305e	11.7de	4.6cd	71c	8.9bc	14.3bc	3.4a

[a] Within a soil, treatment means for a nutrient followed by the same letter are not significantly different (P<0.05)
n.a. = Data not available due to shortage of sufficient sample

There was a significant positive correlation between lettuce fresh weight and both the supply of NO_3^--N and the total amount of N applied (data not presented) for both soils. There was no significant correlation between NO_3^--N supply and the amount of N applied, which indicated differences in the availability of N from the different amendments. In the Saywood soil, N uptake by the shoot was significantly and positively correlated with NO_3^--N supply and N applied, however, there was no similar positive correlations in the Brookside soil. This indicated that N was not limiting in the Brookside soil but was limiting in the Saywood soil. There was a good indication that lettuce had the potential to take up more N; in pots containing a poultry manure compost treatment (unreplicated), PRS™ NO_3^{-}N, lettuce N concentration and yield were higher than any of the main experimental treatments. Using the same type of PRS^{TM} probes, Hammermeister et al. (2006) reported that available N from a VC treatment was 10% of the total N applied, which was too low to meet the minimum N required for orchardgrass.

The soil $H_2PO_4^-$-P supply rate was greater in the VCM treatments than the composted CM treatments but differences were only significant in the Brookside soil; the VCM1+F treatment was not different from the VCM1 treatment in the Brookside soil, but significantly lower than the VCM1 treatment in the Saywood soil (Table 3). Lettuce P uptake was numerically greater in the VCM than CM treatments (Table 2), and significantly greater in the Saywood soil. The VCM treatments increased soil P supply rates (Table 3) and plant uptake (Table 2) relative to the Control; CM treatments were higher in soil P supply and uptake than the Control but differences were not always significant. The supply rate of soil P was significantly correlated with both lettuce fresh weight and total uptake (data not shown), which may indicate that P was also a limiting factor in plant growth.

Table 3 Soil nutrient supply rate (μg 10cm^{-2} 28 d^{-1}) from day 19 to 46 amended at the 2.5% rate[a]

	Phosphate	K^+	Sulfate	Ca^{2+}	Mg^{2+}
Brookside					
Control	0.8c	414d	14f	758b	320b
CM1	1.4c	1652c	44cd	864b	358b
VCM1	4.2b	1742c	52bcd	2144a	896a
Blend1	2.2bc	1328c	42de	1058b	446b
VCM1+F	2.4bc	682d	22ef	1094b	338b
CM2	1.8bc	2622b	68b	996b	382b
VCM2	12.2a	3932a	96a	1792a	698a
Blend2	1.8bc	2936b	64bc	862b	362b
Saywood					
Control	1.2c	250f	44e	2666	258d
CM1	7.0abc	1250cd	136ab	3168	484abc
VCM1	10.8a	896de	62de	2678	592ab
Blend1	8.8ab	820def	92cd	2490	412bcd
VCM1+F	4.2bc	350ef	36e	2624	300d
CM2	2.8bc	1720bc	104bc	2390	394cd
VCM2	8.4ab	2528a	140a	2952	598a
Blend2	7.8ab	2236ab	132ab	2220	448abcd

[a] Within a soil, treatment means for a nutrient followed by the same letter are not significantly different($P<0.05$)

The K^+ content (Table 1) of the dairy manure was substantially higher than for the beef manure; this result was reflected in the soil K^+ supply rate (Table 3). Relative to the Control, the soil K^+ supply rate was increased significantly by all treatments except the VCM1+F treatments and the Blend1 treatments to the Saywood soil. The VCM2 supplied significantly higher K^+ than CM2 but there was no significant difference between the CM1 and VCM1 treatments. Plant uptake of K^+ was significantly greater in the VCM1 treatment than the other treatments but only in the Saywood soil (Table 2); this difference is generally attributed to differences in lettuce yield. No significant correlation was found between fresh weight and soil K^+ supply or the amount of K applied for either soil, which suggests that K^+ supply was not a yield-limiting factor. A significant correlation between K^+ applied and soil K^+ supply rate was measured. The supply rate of soil SO_4^{-2}-S was significantly higher in the VCM2 treatment compared to the CM2 treatment in both soils; SO_4^{-2}-S supply rate of the VCM1 treatment was either not different from or significantly lower than the CM1 treatment (Table 3). Although plant S uptake due to the VCM treatments was always greater than for the equivalent CM treatments, only the VCM2 and CM2 treatments to the Brookside soil were significantly different (Table 2). Lettuce S uptake and soil SO_4^{-2}-S supply rate were significantly correlated with the amount of SO_4^{-2}-S applied to the Brookside soil but not to the Saywood soil (data not shown). The supply rate of soil Ca^{2+} from the VCM treatments was significantly higher than from the CM treatments for both manure sources for the Brookside soil; however, there were no differences among treatments for the Saywood soil (Table 3). Ca uptake from all amendments was greatest with the VM1 or the Blend1 treatment. For both manure sources, the supply rate of soil Mg^{2+} and Mg uptake (Brookside soil) were significantly higher in the VCM treatments than in the CM treatments (Tables 2, 3). Lettuce fresh weight was significantly correlated with Mg^{2+} supply rate for both soils. Lastly, the VCM treatments produced mathematically higher values than the CM treatments in the ratio of PRS™ $H_2PO_4^{-}$P, K, Ca, and Mg supply to the total P, K, Ca, Mg applied (data not shown). This indicated that vermicomposting generally increased the availability of P, K, Ca or Mg, but not S.

4 CONCLUSIONS

On a comparative dry weight basis, vermicomposted manure had higher N availability than thermophylically composted manure but vermicomposting did not increase N availability relative to the total N of the amendments when applied at the 2.5% (w/w) rate. The PRS™ supply rates of $H_2PO_4^-$-P, K^+, SO_4^--S and Mg^{2+} were all increased by vermicomposting compared with conventional composting, indicating higher plant availability of these nutrients. There was no added growth or nutrient benefit to mixing conventionally composted manure with vermicomposted manure. Vermicompost produced from a 75:25 cardboard:manure mix did not improve lettuce productivity compared to the Control in either soil, and actually immobilized N. Manure source had a significant influence on N availability and lettuce growth; the composted or verrmicomposted beef manure had lower C:N ratios which affected the above factors.

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(243) FERTILIZATION PRACTICES ON PERIURBAN FARMING SYSTEM AND RECYCLAGE ORGANIC RESIDUAL PRODUCT AVAILABLE IN MAHAJANGA

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EXECUTIVE SUMMARY

In Mahajanga, Madagascar's provincial city with around 250.000 inhabitants, the urban population growth around 3% per year and concomitantly the need of food It is an opportunity for the development of a periurban agriculture to meet this growing demand. Soil fertility management is determinant in plant production. Market gardening currently suffers from a lack of organic fertilizers and minerals fertilizers remain inaccessible for a significant proportion of producers. One option to response to this organic matter needs is to mobilize all organic resources that could be recycled. We carried out a cross-analyze between the present fertilizers' practices in market gardening and the potential resources or organic residual products in and around the city. Agronomic value of the identified organic residual products was asses through chemical and biochemical analysis. The results showed that combined use of intensive animal organic matter and mineral nitrogen (urea) were the main fertilizers used in the leafy vegetables cropping systems. Twenty organic waste products were listed in the town; ten were selected for characterization through chemical and biochemical analysis. The organic residual products contained significant amounts of major nutrients for plant production. However, as the role of organic product and mineral fertilizers inputs were more complex than a simply question of chemical balance (mulch, quality products, fertilizers and amendment effects), it was necessary to test innovate organic fertilizers with an assessment of economic and social acceptation. Marginally of these work, long term consequence of current farmers' practices of fertilization showed the important risk of pollution by nitrate in Mahajanga. More studies would have to be done to better assess the environmental impacts of the conventional and also new fertilization practices.

Keywords: Market gardening, periurban, Madagascar, organic wastes, organic residual products.

1. INTRODUCTION

The demographic studies showed that over 60% of the population will be urban by 2030 (United Nation, 2003). In 1950, urban population accounted for less than 25% of world population; and it was over the rural population since 2007. Urban growth is especially fast in the cities of developing countries. However, the increasing urban food insecurity stimulates the development of urban agriculture in developing country (Veron, 2007). The agriculture inside or around the urbanized area is essential as it could provide foods, employment and richness for this increased population (Moustier, 2007). Investment in urban agriculture in Mahajanga, Madagascar, is one of the strategies for increasing household income and improving food security. The development of leafy vegetables production, the main vegetables consumed by Malagasy people and livestock keeping in and around urban center contribute largely to supply food and generating income for urban population. In Mahajanga, agriculture is practiced in around lakes and on lowlands after the three-month wet season from December to February. Most of annual rainfall (1000-1500mm) falls in this period. Lowlands are devoted to rice production on wet season and to market gardening on dry season. The fringes of the lakes are cultivated gradually as they become dry after the end of the rainy season.

The main constraints to further development of these agricultural systems are the land tenure system, the deterioration of natural resources, the weak access to organic and mineral fertilizers and to money. As it was generally the case in tropical area, the organic matter inputs (OI) are necessary to insure a sustainable production. Traditionally, OI came from urban livestock's production but the available manures become more and more limited to respond to the increased farmers demand.

The town offers a diversity of organic resources that could be used in cropping systems. However, before proposing and testing new organic matters to improve the crop production it is necessary to increase knowledge's of soil fertility management and of agronomic value of organic resources.

Researches were carried out in Mahajanga, Madagascar to analyze the fertilization farmers' practices on traditional market garden production of leafy vegetables and to inventory available organic resources which could be used in cropping systems.

2. MATERIALS AND METHODS

2.1. Analysis of fertilization practices

Conceptual model of technical decisions relating to fertilizers practices were established according to the methodology described by Aubry (2007). On a sample of farms (n=12) the crop management sequences practices during the cropping season (6 months) were weekly recorded during two years. The results obtained were then verified by surveys on a larger sample of farmers on the three main sites of Mahajanga to validate a model of fertilization practices.

2.2. Inventory and characterization of available organic resources

We conducted a survey to identify the organic resources: livestock, household, market waste and industries which produced organic waste such as fishery, soap factory and oil mill.

Uses and available quantities of each type of organic resources in Mahajanga were assessed. Samples of organic resources were collected for chemical characterization: nitrogen, carbon, phosphorus and potassium contents, and biochemical composition to assess the biodegradability of the organic components (Swift et al., 1979).

3. RESULTS AND DISCUSSION

3.1. Characteristics of the farming systems

In Mahajanga, the areas of the market garden productions were determined by the availability of water, a prerequisite for the practice of market gardening. Depressions were devoted to rice production during the rainy season and used for market gardening during the dry season. Market gardening was also located around permanent or seasonal lakes (Dumont, 2006). The growing season of the leafy vegetables began after the last rain and run to eight or nine months. After the rainy season, the leafy vegetables' plots were settled progressively as the withdrawal of flood waters.

Farmers had only a small plot of land and practice intensive crop rotation to maximize the use of land. The technical and commercial unit of crop management was a micro plot of 12 to 14m² called beds. The number of beds of culture increased during the dry season to reach 120 cropping beds per producer. On lowland cultivated areas ranged from 400-1200 m². The average number of cultivated bed was 48 per gardeners,. The average farm size for the lowlands of different cropping areas of Mahajanga were 474 m² in Amborovy,506 m² in Ambondrona and 676 m² in Belobaka (Mawois, 2009). Crop areas were smaller on lowlands, where land pressure was more important due to urbanization and low water availability.

Working people were often composed exclusively of members of the family, both parents and children during holidays. Most of the farmers cultivated only leafy vegetables that constituted their main source of income. The main cultivated leafy vegetables were firstly the short cycle plants, 21 to 30 days, dominated by the greens of Brassicaceae's family, with known varieties such as *Brassica campestris peruviridis*, *Brassica campestris pekinensis*. For these plants, harvesting was done by uprooting the whole plant. The second main crop was the long cycle plants as *Solanum nigrum* (Solanaceae) and *Acmella oleracea* **(**Asteraceae*)* which were cut every 3 or 4 weeks during 3 to 6 months.

The leafy vegetables production was almost entirely sale in municipal markets. Usually, the products were bought by intermediate traders generally women.

The transaction was directly done on the field based on a global evaluation of the entire leafy vegetables cropping beds. So, the global appearance of the plant, particularly the green color was an important criterion to set prices. Green leaves and free of holes had the best prices (Audois, 2007).

3.2. Fertilization practices: organic and mineral

There was one application of manure and at least one application of urea (mineral N) each crop cycle. Generally, chemical fertilizers were applied twice but the second input was optional. The organic or mineral supplies were renewed at each plant cycle or each successive plant yield for perennial leafy vegetables. Organic inputs as chemical fertilizers were always spread on the soil surface after seedlings transplanting. The first application of manure and urea was 4 to7 days after transplanting. Organic materials used by farmers were mainly, in the order of preference, cattle, pig, poultry, sheep or goat manure coming from local livestock. The amount of fresh manure applied was about 18-24 kg per bed equivalent to 1.7 kg.m-². Farmers applied almost 100 kg N ha-^{1} of urea per application.

According to farmers, the role of manure's addition was also to create mulch that conserved moisture at the level of the roots system. This observation would indicate that the organic matter supply could have a function of mulch for the current plant cycle but would become a fertilizer or an amendment for the following plant cycle.

According also to farmers, the first supply of urea aimed to grow vegetables in height and the addition of manure provides the growth in width. The second application of urea, 5 to 7 days before harvesting, aimed to insure the green color of leaves as intermediate traders desired.

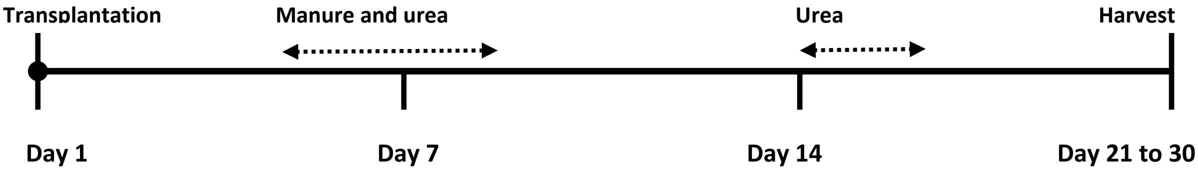

Figure 1: Conceptual framework of organic and mineral fertilization of short cycle leafy vegetable in Mahajanga.

3.3. Town available organic resources

Twenty organic products (OP) were identified in the city of Mahajanga As we have seen previously, manure was absorbed by market gardening. There would be 28t of fresh manure produced per day by livestock located in urban and periurban. However, there were frequent supply problems on dry season because there were important demands on this period. The supply of market gardeners was guaranteed by intermediary carters whom were in charge of finding manure and delivering it near crop fields.

Concerning industrial wastes, they were used as raw materials for chicken, cattle and pig feed. Soap and oil factories' wastes were essentially peanut, copra and cotton cakes.

Zebu blood was recuperated in slaughterhouses to feed domestic animals. Zebu horns were also available and were tested by a private company as amendment.

In fishery industries, actually fishing boats were equipped with facilities that allow them to process immediately seafood on board. Only products of marine small-scale fisherfolk are treated on land. The waste is either dumped or shared to employees or sold to resellers or animal feed (Ramahefarison, 2011).

As for market and household wastes, the municipality of Mahajanga was in charge of the collect. From 2001 to 2011, a local association support by a NGO, developed urban waste composting However, the high price of compost, four times more expensive than manure was a barrier to its use by local farmers. So that compost was only accessible for landscape gardeners or for high value production as medicinal plants and, for instance some plant production for biofuel. In addition, not enough advice was given to local farmers to use this compost, and studies were needed to adapt the compost quality, to the cropping systems of vegetables producers.

3.4. Chemical and biochemical characterization of OP

Chemical analysis of organic product showed that there were some OP that contented high rate of nitrogen and carbon such as dried blood and fishery waste. However, these products were absorbed by livestock chain or rejected in the sea. Other organic matter may be used as amendment according to their content in carbon. Regarding other major chemical elements such as phosphorus and potassium, we noticed significant levels of phosphorus for poultry manure and fishery waste.

Manures and compost from municipal wastes are some of organic product which contented important rate of potassium.

Table 1: Chemical compositions of organics products

Organic products	C (mg.g-1)	N (mg.g-1)	P total (mg kg-1)	K (mg kg-1)
Urban waste compost	200	12,9	5663	12201
Loam	99,4	5	3349	3064
Horn powder	236,1	73,7	784	4687
Poultry manure	354,2	26,4	6000	9182
Fishery waste	228,6	39,5	8412	3843
Sheep manure	404	21,5	2448	10132
Pig manure ABD	201,1	11,5	6513	4884
Dried blood	403,3	99,2	2481	3044
Cattle manure	365,1	17	2190	16272
Poultry manure without litter	313,5	33,4	15299	9497

Organic products collected in Mahajanga were divided into three classes:

- OP with a high rate of soluble fraction and a large nitrogen content, which could be used as fertilizer: for instance dried blood and fishery waste.
- OP which were rich in soluble and hemicellulose fraction (> 45%) considered as amendment
- OP which presented an important percentage of cellulose and lignin (> 65%), which could be considered as amendments: for instance powder zebu horn and most of the manures used by market gardeners.

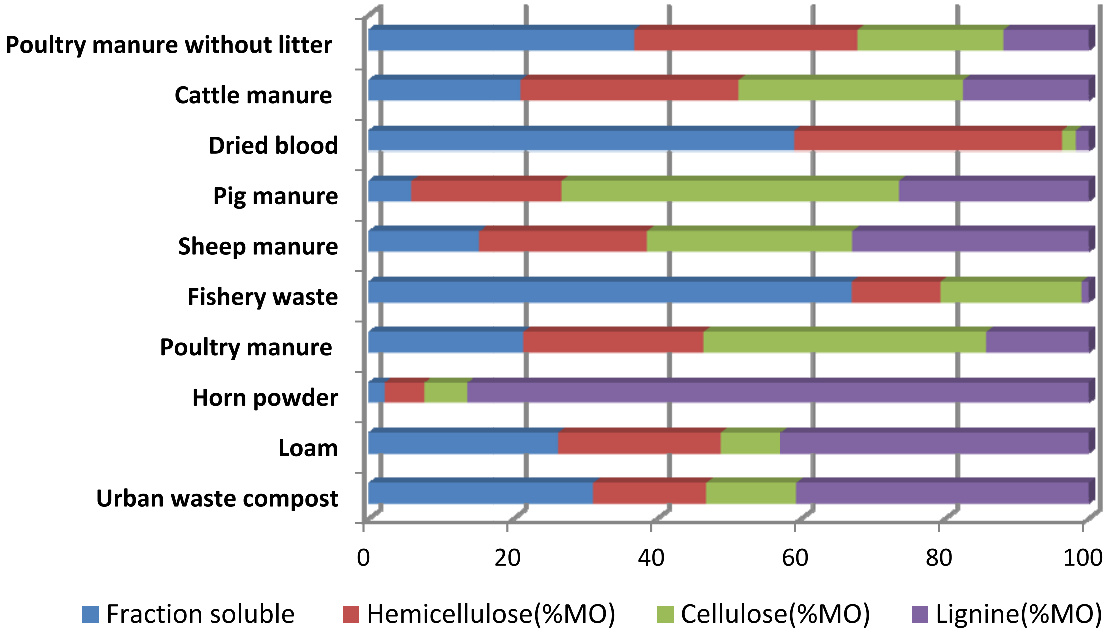

Figure 2: Biochemical composition of organic products in Mahajanga

4. CONCLUSION

To support the rapid development of the periurban agriculture in most cities of the South, nutrients and organic inputs are necessary. The enhancement of the recycling of organic product in the market gardening cropping system could be a win-win approach; farmers could reduce their dependence to external and expensive mineral fertilizers, and municipality could resolve a part of the environmental problem of wastes that have to be managed.

Livestock and agricultural industries were major sources of organic material currently used for urban agriculture. The analysis of the organic and mineral fertilization of leafy vegetables production in Mahajanga highlighted different functions of the organic products or mineral fertilizers that they were applied. The organic products had a role of fertilizer but could also favor soil water retention. However, the interactions between the both mineral and organic products have to be assessed along the cropping sequence of leafy vegetables. It is the condition to find appropriate alternatives of urban origin organic inputs coming from the urban activities. Moreover, alongside this study, problems of water and soil pollutions certainly due to over-fertilization were noticed. It will be necessary to explore this issue. And finally, social and economic studies are needed to insure the implementation of innovate practices of soil fertility management.

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Session 18

(20) PRODUCTION OF LIPASES BY SOLID STATE FERMENTATION USING VEGETABLE OIL - REFINING WASTES

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EXECUTIVE SUMMARY

SSF holds tremendous potential for the production of enzymes when compared to stirred/submerged fermentation, due to its ability to use various substrates including solid waste. Different studies of lipase production have been carried out using few grams of substrate, mesophilic temperatures and known microorganisms (Godoy et al., 2009). The main objective of the current study was to develop a scalable SSF process for lipase production simulating real adiabatic conditions in full-scale processes and evaluate the use of fermented solids as biocatalysts.

The solid matrix for SSF experiments is a mixture of winterization residue (WR) and raw sludge (RS). The RS/WR mixture had in total 20% of fats (dry basis) (Gea et al., 2007). Experiments were performed in 4.5 L Dewar vessels, during the time period of 35 days, and samples were withdrawn periodically. The extraction was carried out as described by Santis-Navarro et al. (2011). Lipase activity in solid samples (wet and air-dried) was determined as described by Hernández-Rodríguez et al. (2009), whereas the activity in liquid extracts was measured using a lipase colorimetric assay (Resina et al., 2004). The effects of pH and temperature on lipolytic activity were analyzed applying a full experimental design.

The extracted lipases were most active at 61–65 ºC and at pH 7.7–9. For the solid samples, the lipolytic activity reached up to 120,000 AU/g of dry matter. These values are considerably high and highlight the possibility to work with the fermented solid as effective biocatalysts. During the study it was observed that in the raw solid sample more enzymatic activity can be achieved than that in the buffer solution containing surfactant. The levels of lipolytic activity obtained from fermented solids are among the highest reported on SSF to the authors' knowledge. Considering other published works, usually the enzyme activity is obtained using a pure strain.

The study on scalable SSF with vegetable oil refining industry waste had shown that this waste is a good source for lipolytic enzymes production with promising properties. The use of fermented solids as biocatalysts has a great potential in terms of low-cost production processes obtaining high yields. Current experiments are undertaken to analyze potential application of the obtained catalyst to environmental bioprocesses. Fermented solids show a promising capacity to enhance fats, oils and grease metanization, reducing lag phase and total process time.

1 INTRODUCTION

1.1 Background

Solid-state fermentation (SSF) is defined as the fermentation process on moist solid substrate in the absence or near absence of free water (Pandey, 2003). SSF can be used for the production of enzymes utilizing various substrates including solid wastes. Lipase production by SSF under different process conditions, with different microorganisms and substrates has been reported (Godoy et al., 2009; Hernández-Rodríguez et al., 2009; Sun et al., 2009). However, most studies were carried out using a few grams of substrate, mesophilic temperatures and pure cultures of known microorganisms.

Only a few studies have been carried out at pilot or industrial scales (Kumar et al., 2009; Edwinoliver et al., 2010). SSF encounters problems related to mass and heat transfer phenomena associated with solid substrates (Pandey et al., 2008), and the use of natural solid substrates can hinder downstream processes (Rodríguez-Couto and Sanromán, 2006), especially when extracting lipophilic enzymes such as lipases (Mala et al., 2007).

Fermented solids have been used as naturally immobilized biocatalysts for synthesis reactions in lyophilized (Hernández-Rodríguez et al., 2009) or dried form (Hellner et al., 2010). This approach can lead to lower costs of enzyme preparations since no extraction and purification steps are carried out.

1.2 Research objectives

The main objective of the current study was to develop a scalable SSF process for lipase production simulating real adiabatic conditions in full-scale processes, to optimize the extraction procedure of the lipases and to evaluate the use of fermented solids as biocatalysts. Waste derived from the vegetable oils refining industry was used as substrate and sewage sludge served as source of microorganisms. The thermostability of the enzyme extract was proved to be effective in commercial tests used for determining the hydrolysis activity. Moist and air-dried fermented solids were tested as biocatalysts for the hydrolysis of olive oil, selected as standard hydrolysis reaction.

2 METHODOLOGY

2.1 SSF materials

A mixture of winterization residue (WR) and raw sludge (RS) was used as solid matrix for SSF experiments. WR (53.1% fat content on dry basis) was provided by the LIPSA (Lípidos Santiga S.A, Barcelona) oil-refining facility. WR is obtained by submitting vegetable oil to rapid cooling to 5°C over 24 h and removal of waxes by filtering with diatomaceous earth. This waste was selected as a source of fats because of its stable and homogeneous composition according to the manufacturer's information.

RS was added to WR as inoculum and co-substrate to provide moisture and nutrients (Gea et al., 2007). RS was collected after centrifugation from the Metrofang wastewater treatment plant of Besòs (Barcelona, Spain), a very big facility treating wastewater of 1.5 millions inhabitants. The RS/WR mixture had 19.7% of total fats (dry basis) (Gea et al., 2007). Wood chips were added as a bulking agent to the mixture of RS and WR at a ratio of 1:1 (v:v) to provide proper porosity to maintain aerobic conditions. The water content of the mixture was adjusted to 50% by adding tap water before SSF.

2.2 SSF experiments

Experiments were undertaken in 4.5 L Dewar® vessels containing 2.5 kg of mixture. The vessels set-up (described in Sayara et al., 2010) allowed a continuous air supply, temperature monitoring, separate leachate collection and oxygen monitoring to ensure aerobic conditions (oxygen content around 10-12%). Due to the thermal isolation of the vessels, these reactors work under near-adiabatic conditions, to simulate real SSF processes where a non-constant temperature evolution is produced due to the limitations of heat transfer in organic matrices (Barrena et al., 2006). The SSF experiments were undertaken for 35 days, and samples were collected after 6, 14, 27 and 35 days. The two replicate reactors were opened and the content was mixed well to obtain homogenous and representative samples.

2.3 Enzyme extraction optimization

A full factorial experimental design was used for the determination of the best conditions for lipase extraction. The optimal values for extraction were 500 mL/g of buffer dose and 2% of surfactant. Under these conditions, extracted activity accounted only for 5% of total extractable activity. However, it was decided not to extend the range of study because doses higher than 500 mL/g would not be economically viable at industrial scale. Other strategies such as a multistep extraction procedure (Mala et al., 2007) should be considered in the future for the scale-up of the extraction process.

2.4 Lipolytic activity

Lipase activity in solid samples (wet and air-dried) was determined as described by Hernández-Rodríguez et al. (2009). This protocol is based on using solid samples as biocatalysts for the hydrolysis of olive oil, selected as standard hydrolysis reaction. The activity in liquid extracts was measured using a lipase colorimetric assay (kit 1821792, Roche diagnostics, Basel, Switzerland) (Resina et al., 2004).

For both methods, one lipolytic activity unit (AU) was defined as the amount of lipase necessary to hydrolyze 1 µmol of ester bond per minute under assay conditions (temperature 30ºC, pH 8) and it was referred to the amount of solid substrate used for obtaining the extract sample, both wet (AU/g) or dry (AU/g DM).

2.5 Effect of pH and temperature on lipolytic activity

The effects of pH and temperature (T) on lipolytic activity were analyzed by a full factorial experimental design consisting of 15 experiments (12 experiments and three replications at central point for statistical validation). The temperatures were fixed at 30, 45, 60 and 75 ºC and the pH at 5.0, 7.0 and 9.0. Residual lipolytic activity (RA, referred to the initial activity of the extracts) after one hour of incubation was selected as the objective function and as a measure of lipase stability. Buffers used for the incubation at the selected pH were: Tris-HCl 1M, pH 9.0; Tris-HCl 1M, pH 7.0; acetic acid-sodium acetate 1M, pH 5.0.

2.6 Analytical methods

Moisture content and organic matter were determined according the standard procedures (Gea et al., 2007). The fat content was measured using a standard Soxhlet method with n-heptane as organic solvent (The U.S. Environmental Protection Agency, Method 9071B).

3 RESULTS AND DISCUSION

3.1 SSF experiments

The fermentation process was under thermophilic conditions for 20 days. An important reduction in fat content was observed in the first 15 days (Table 1) but no significant fat degradation occurred after that moment, resulting in a final fat content around 5%, which may correspond to fats difficult to biodegrade (Gea et al., 2007).

TABLE 1. **Fat content and Lipase activity measured in extracts and fermented solids and total extractable activity**

Sample	**Total fat content**	**Total extractable activity**	**Enzymatic activity in solid samples**	
	(%, dry basis)	AU_{total}/g DM	Wet samples (AU/g DM)	Air-dried samples (AU/g DM)
Day 6	16	1752	106517	13938
Day 14	5	49113	120731	20925
Day 27	6	1371	87906*	88000*
Day 35	5	2478	44928	85251

* Samples with enzymatic activity that is not statistically different.

3.2 Enzyme extraction and lipolytic activity in extracts

Values of extracted activity were around 50 AU/g at optimized conditions, and thus in the lower range of values reported in the literature (Hernández-Rodríguez et al., 2009). Despite to optimized conditions, only small percentages of activity were recovered in extracted samples, being these values around 5-7 % of total extractable activity. The lipolytic activity showed a maximum at day 14, coinciding with the period of maximum fat degradation (Table 1). After 13 days the activity considerably decreased (day 27), which can be attributed to the complete consumption of biodegradable fats (Gea et al., 2007).

3.3 Lipolytic activity in fermented solids

A statistically significant difference in the lipolytic activity in wet and air-dried samples was observed except for the sample of day 27 (Table 1). Wet samples obtained at the beginning of the process showed higher activity than wet samples taken at later time points, whereas the opposite was noted for the dry samples.

The activity in the solid samples was higher than that of aqueous extracts. The lipase activity in the solids was very high, and it would be of interest to identify the different enzymes that contributed to this high level of activity. A possible explanation for this difference may be related to the low water activity that most lipases require (Hasan et al., 2009).

The levels of lipolytic activity obtained from fermented solids are the highest reported on SSF to the authors' knowledge, two orders of magnitude over any value published. Although lipase activity units reported on literature are obtained with very diverse methods, and often they are not directly comparable, the findings reported here highlight an extraordinary potential for the use of fermented solids as biocatalyst.

3.4 Characteristics of lipases

The effect of temperature (T) and pH on stability of the extracted enzymes was studied by means of a factorial experimental design. The best fitting for experimental residual activity that was selected as objective function was obtained for a second-order polynomial model for the four samples analyzed. The equations obtained in this case were:

$$RA_6 = 0.9940 + 0.2700T + 0.0735pH - 0.2357T^2 - 0.0695pH^2 - 0.0339TpH$$

$$RA_{14} = 1.0015 + 0.3408T + 0.0965pH - 0.3299\ T^2 - 0.0942\ pH^2 - 0.0554TpH$$

$$RA_{27} = 0.9070 + 0.3063T + 0.0925pH - 0.2709\ T^2 - 0.0189\ pH^2 - 0.0518TpH$$

$$RA_{35} = 0.9579 + 0.2078T + 0.0968pH - 0.2657\ T^2 - 0.0838\ pH^2 + 0.0192TpH$$

Lipase activity was more sensitive to temperature (T) than pH (the values of coefficients for T are higher than those of pH). The coefficients indicated that high values of both T and pH have a positive effect on residual activity. Lipolytic activity was markedly stimulated by temperature in the thermophilic range that indicated a lipase reactivation. For the four samples analyzed, optimal stability was observed at temperatures in the thermophilic range (61-65ºC) and alkaline pH (7.7-9.0).

3.5 Potential applications of fermented solids

The potential applications of fermented solids are currently investigated in the hydrolysis and metanization of fats, oils and grease from waste water treatment plants. Preliminar results show that the addition of fermented solids as catalyst improve the metanization process of this substrate by significantly reducing the lag phase.

4 CONCLUSION

The study on scalable SSF with vegetable oil refining industry waste has shown that this waste is a good source for lipolytic enzymes production with promising properties. The use of fermented solids as biocatalysts is also promising in terms of low-cost production process with high yield potential. Further research should explore the application of the obtained lipases in novel synthetic routes and their identification. Another point that needs attention is the reproducibility of the source of microorganism used since sludge and, in general, organic solid wastes are inherently variable in chemical composition and in the characterization of the existing microbial communities.

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(166) REDUCING THE ENVIRONMENTAL IMPACT OF BIOGENIC PHAs PRODUCTION USING AN ENZYMATIC RECOVERY STRATEGY

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EXECUTIVE SUMMARY

Bioplastics, in particular polyhydroxyalcanoates (PHAs), are widely studied as biodegradable materials for substitution of oil derived polymers. However, their production at an industrial scale is still limited due to high production costs. In addition, industrial PHAs recovery needs the use of large volumes of organic solvent like chloroform, which is not convenient for environmental safety (Byrom, 1994). Bosco and Chiampo (2010) reported four ways to reduce these costs including new cheap substrates, new fermentative strategies, new recovery and purification steps, and new microorganisms for accumulation of high PHA concentrations. In this context, sewage sludge can be considered as a source of renewable organic carbon and of diversified microorganisms for biogenic PHA production. In addition, sewage sludge may also provide lytic micro-organisms that are of a great interest for PHAs recovery using new and safe extraction procedure..According to this objective, a new strategy for the recovery of intracellular PHAs using multiple hydrolytic activities secreted by thermophilic strains isolated from composting sludge is presented in this study.

Sewage sludge was sampled in a municipal composting plant (Muret, France), during the thermophilic stage of the process. Bacterial strains were isolated by cultivating these samples at 65°C on plate count agar and 7 different strains could be isolated. Two of them, named a and B, were selected for their ability to grow on anaerobic digested sludge. Further characterization using API ZYM® (Biomérieux; France) of the a and B exocellular activities showed the presence of phosphatase, lipase and osidase activities, but no protease activity was detected.

The lytic potential of the a and B exocellular enzymes was then tested on pure cultures of *Cupriavidus necator (*Aragao et al., 1996). After PHA accumulation due to nutrients limitation, these producers were submitted to an osmotic shock (successive incubation in a hypertonic and hypotonic solution) and incubated with the hydrolytic supernatants of strains A and B selected from compost (37 and 65°C, 30 min, 10 ml of supernatant/g of dry matter). The released PHAs were quantified by GC method after their mild extraction with chloroform. When using a and B supernatants, 81±2% and 99±2% of PHAs were recovered at 37°C, respectively. Increasing either the temperature or the supernatant/ culture medium ratio did not significantly improve the percentage of PHA recovery.

Similar extraction conditions were applied to a multi-species culture incubated with B supernatant, but only 65±3% of PHAs could be extracted because of the presence of flocs which limit the access of the hydrolytic enzymes to the cells. A physical pre-treatment has thus to be considered to dissociate flocs and improve the cellular lysis.

In conclusion, this study demonstrates that enzymatic recovery of biogenic PHAs from various producers can be performed using lytic bacteria selected from sewage sludge, providing thus interesting reduction of the environmental impacts of PHAs bioproduction. In addition, the selected lytic strains are also of great interest for general improvement of carbon bio-availability in various organic wastes and their valorisation as renewable carbon source.

1 INTRODUCTION

1.1 Background

In a context of petroleum depletion, bioplastics are considered as biodegradable and renewable materials for substitution of oil derived polymers. Polyhydroxyalkanoates (PHAs) are a family of such biodegradable polymers that offer a broad range of industrial applications (Philip et al., 2007). However, their production at an industrial scale is limited due to high production costs and difficulties during PHA recovery and purification steps. Several bacteria accumulate PHA as carbon and energy reserves under certain nutrient depletion conditions. Some gram-negative species such as *Azobacter sp., Methylobacterium sp.* or *Cupriavidus necator* have presented this ability while it has never been demonstrated for gram-positive bacteria (Byrom, 1987). As an example, *Cupriavidus necator* can accumulate high PHA content up to 0,8 g of PHA per g of dry cells when cultured under phosphate limitation in a high cell density fed-batch reactor (Ryu et al., 1997). Due to the intracellular location and hydrophobic character of PHAs, their extraction was generally performed using large volume of organic solvent like chloroform (10 ml of solvent/g of biomass) that generates chlorine-containing effluent which is not convenient for environmental safety (Byrom, 1994). PHB extraction with chloroform allows to recover 55% of PHB with a purity of 94% when performed at 61°C during 30 min. At room temperature and, after 12 h incubation with chloroform, only 27% of PHB are extracted with the same purity (Ramsay, 1994). Although leading to significant increase in PHA yield, one disadvantage of high temperature extraction is molecular weight reduction of biopolymer (Ramsay, 1994). Other studies using chemical methods such as anionic SDS detergent or hypochlorite were investigated. However, in addition to their unfavorable environmental impact, they were shown to affect the polymer quality and industrial performances (Kim et al., 2003; Hahn et al, 1993). Due to these problems, enzymatic digestions based on bacterial cell-wall hydrolysis and involving milder extractive conditions have been proposed. Commercial enzymes such as lysozyme, alcalase, bromolein, or trypsin were tested leading to various extraction yield and PHA purity. Further extraction step with solvent was needed to improve final PHA purity that was general low (under 80%) after the enzymatic treatment due to the presence of cellular components. This purity can be improved with combined enzymatic treatments as described by Kaprotchkoff et al., 2006 that obtained a solution with 87% of PHB after sequential digestion with trypsin and bromelain (Kaprotchkoff et al., 2006). Alternatively, exoenzymes secreted by bacteria can also be efficient for cell lysis since the incubation of *Microbispora* culture filtrate with *Sinorhizobium meliloti* cells followed with chloroform extraction of PHB at room temperature allowed to recover 94% of the intracellular PHA with a good purity level of 92% (Yasotha et al., 2006; Lakshman and Shamala, 2006).

1.2 Research objectives

The aim of the present work was to propose an environmental friendly procedure for PHA extraction from bacteria. The proposed method involved a mixture of exoenzymes obtained from the culture supernatant of two thermophilic strains isolated from composting sludge. The lytic activity of the secreted enzymes was first evaluated on the solid and soluble fractions of anaerobically digested sludge before application to PHA bacterial producers. The exoenzymes were tested on the gram-negative bacteria *Cupriavidus necator* containing high PHA content and on a multi-species culture of PHA producers. To select these microorganisms, a sludge microbial consortium was cultured under limited phosphorus conditions in order to promote the development of bacteria that can produce PHB. Cell lysis and PHA release were evaluated for both cellular suspensions and a global extraction strategy was proposed including enzymatic digestion and mechanical treatment allowing PHA recovery from selected producers with low cost and low environmental impact.

2 METHODOLOGY

2.1. Selection of A and B strains for production of lytic activities

- Isolation of strains:

Strains have been isolated from sewage and containing 50% of sludge that comes from Ginestous plateform of composting (Toulouse) and 50% of husk. After four weeks under aeration at 65°C and 8 weeks more at room temperature, the final mixture was collected.

From this final mixture, strains were isolated on plate counting agar (PCA) at 20.5 g/l: after dilution of bacteria culture in 0.9 % NaCl solution, 100 µl of the diluted bacterial solution were spread on PCA and incubated at 60°C.

Seven different strains could be isolated.

- Culture of strains:

10 ml of sterile trypticase-soja (TS) solution containing tryptone (17 g/l), peptone papaïnic of soya (3 g/l), glucose (2.5 g/l), K_2HPO_4 (2.5 g/l) and NaCl (5 g/l) were inoculated from these strains during 48 h, at 60°C. The isolated strains were kept on agar slant.

- Selection of strains:

Selection for lytic activities was performed after incubation, during 24 h, at 60°C, on agar (20 g/l) containing refractory matter. This last one is low biodegradable waste water sludge obtained after anaerobic digestion.

- Production of supernatants:

The previously selected strains, named a and B, were inoculated on TS solution during 24 h, at 60°C. After centrifugation (10000 g during 20 min at 4°C), the supernatants were aliquoted, then concentrated by vacuum-drying at 40°C during 24 h. The protein concentration of final concentrated supernatants was determinate by BCA assay (Smith et al., 1985)

2.2. Enzymatic activities evaluation of strains isolated from compost supernatants

- Qualitative evaluation:

The API ZYM® system (Biomérieux) allows to detect 19 enzymatic activities (phosphatases, lipases, glucosidases, protéases) without purification. 5 ml of ultrapure water were deposed in the bottom of the incubation box. 65 µl of supernatant were added in each little cup. After 4 h of incubation at 60°C, one drop of ZYM A and ZYM B solutions are added in each little cup. The reading was performed 5 min later with a colorimetric scale from 0 to 5.

- α et β glucosidase activities:

400 µl of 0.5% p-nitrophényl-α-D glucopyrannoside or p-nitrophényl-β-D glucopyrannoside were mixed with 500 µL of buffer (20 mM Tris-HCl, pH 7.2). This substrate and 1.2 ml of supernatant were separately incubated at 30 or 60°C, during 10 min. The kinetic was begun by adding 1 ml of supernatant in the substrate solution. The temperature was kept at 30 or 60°C. The absorbance at 410 nm was measured all 2 min, during 20 min.

- Protease activity:

800 µL of 0.5% azocaseine solution and 200 µL of supernatant were mixed and incubated at 30°C or à 60°C during 1 h. 500 µL of 15% trichloracétic acid were added and the incubation went on 30 min more at room temperature. Each sample was centrifuged (14500 g, 6 min). 800 µL of supernatant were mixed with 200 µl of 2 M NaOH solution. After vortex stirring, the absorbance was read at 440 nm.

- Lipase activity:

A work solution was prepared with palmitate p-nitrophenol at 20 mM in propan-2-ol. 1 volume of stock solution was diluted with 19 volumes of 20 mM Tris-HCl, pH 8, containing 0.1% arabic gum and 0.4% Triton X-100. This mixture was centrifuged during 6 min, at 14500 g and the supernatant, named work solution, was placed at 30°C. 0.1 ml of sample was added to 0.9 ml of work solution and the absorbance variations were followed at 410 nm, during 20 min, at 30 and 60°C.

- Lysozyme activity:

The substrate used was a *Micrococcus lysodeikticus* in 0.1 M phosphate sodium buffer (pH 6.2). This solution was incubated 10 min at 30 or 60°C, then 50 µl of sample were added to 1.5 ml of substrat solution. The absorbance variation at 450 nm was followed every minute, during 20 min. One unit will produce a ΔA450nm of 0.001 per minute at pH 6.24 at 25°C using a suspension of *Micrococcus lysodeikticus* as substrate, in a 2.6 ml reaction mixture.

2.3. Obtention of *Cupriavidus necator* and multi-species culture suspensions

The *C. necator* suspensions and the multi-species culture inoculated from biological wastewater sludge were cultivated to favour the PHA production by LISBP (Aragao et al., 1996; Grousseau, 2012). The bacterial suspensions obtained were centrifuged at 4500 g during 15 min at 4°C. Physiological water was added to the pellets to have a final concentration of 25 mg of dried matter/l, before being stocked at -20°C.

Dried matter (DM) and suspended matter (SM) were determined by Afnor NF VO3-706 and NF EN-872 standard, respectively.

2.4. Total PHA extraction

The total PHA amount of bacterial cultures was obtained by Braunegg et al. method and modified by Brandl et al. (Braunegg et al., 1978; Brandl et al., 1988). 100 µl of bacterial solution were introduced in a glass tube then dried at 100°C, during 24 h. After cooling, 1 ml of chloroform and 1 ml of acid methanol solution (85/15, v/v, methanol/H_2SO_4 85%) containing 0.7 g/l of benzoic acid were introduced in the tube containing the dried cells. The tube was closed with a stipper in polytetrafluoroethylene and heated at 100°C during 2h15. In the same time, a standard range of PHB was treated with the same procedure. After cooling of tubes, 1 ml of distilled water was introduced. The tubes were shaken few seconds to extract H_2SO_4 from organic phase. The chloroform phase was analyzed by CPG.

The chromatographe (Hewlett Packard 5890) was equipped with a dimethylpolysiloxane column (0.53 mm x 30 m, CP-sil 5 CB) and flame ionization detector. The nitrogen flow was 20 ml/min and the injector and detector temperature were 250 and 270°C, respectively. The temperature program of the oven used was: 115°C during 5 min; a ramp of 5°C per min rise up to 155°C; a ramp of 20°C per min rise up to 230°C and hold at 230°C for 10 min.

2.5. Enzymatic lysis of cells

The 2 ml of bacterial suspension (25g/l) were centrifuged at 4500 g during 15 min. The supernatant was eliminated and 2 ml of hypertonic solution (sucrose at 20 g/l, 0,3 M Tris-HCl and 10^{-4} M EDTA, pH 7,4) were added at the centrifugation pellet before being shaken with vortex to homogenise the suspension. Then, the bacterial suspension was centrifuged at 4500 g during 15 min. The supernatant was eliminated and 2 ml of distilled water were added at the centrifugation pellet before being shaken with vortex to homogenise the suspension. Then, the bacterial suspension was again centrifuged at 4500 g during 15 min and the supernatant was eliminated. A 2 ml buffer solution (5.10^{-2} M Tris-HCl and 10^{-3} M EDTA, pH 8.0) and lysozyme or supernatant a or B was added. The mixture was shaken and incubated at 37 or 60°C. At the end of the incubation, the lysed bacterial suspension was centrifuged at 4500 g during 15 min and the supernatant was eliminated to stop the lysis. The pellet was suspended in distilled water for the following solvent PHB extraction step.

2.6. Mechanical and chemical pre-treatments

Two types of mechanical treatments were applied on bacterial suspension: vortex stirring and/or ultrasonic treatment. Two different ultrasonic apparatus were tested at 20 KHz and 4°C: ultrasonic bath with a power of 20W/g DM during 60 min and ultrasonic probe with a power of 80 W/g DM, during 15 min. The stirring vortex was performed at room temperature during 60 min. A combined mechanical pre-treatment was performed: ultrasonic bath during 30 min followed vortex stirring during 60 min.

Two surfactants were tested for chemical pre-treatment: a neutral natural surfactant, the tween 20 and a anionic petrochimic surfactant, the sodium dodecylsulfate (SDS). SDS (2%, w/v) or tween 20 (2%, v/v) was added to the microbial suspension. The mixture was kept at room temperature during 30 min. Then the mixture was centrifugated and the lysis process was performed.

2.7. Quantification of exocellular PHAs

The exocellular PHAs had to be extracted from the bacterial solution without damaging the intact cells. So, the lysed bacterial solution previously obtained (paragraph 2.4) was introduced in a glass tube.

A same volume of chloroform was added. This mixture was shaken with tambour rotate agitator during 2 h, at room temperature. This extraction allowed to extract the totality of exocellular PHAs without breaking intact cells (results not shown). Then, the tube was centrifuged 10 min at 4500 g. 1 ml of chloroform phase was sampled and introduced in glass tube. 1 ml of acid methanol solution was added like previously.

The ratio of the exocellular PHA concentration from lysed cells on the total PHA concentration allowed to evaluate the PHA recovery.

$$PHA\ recovery\ (\%) = \frac{[exocellular\ PHA]}{[total\ PHA]} \times 100$$

3. SELECTION OF HYDROLYTIC STRAINS AND STUDY OF LYTIC POTENTIALITIES OF THEIR EXOENZYMES

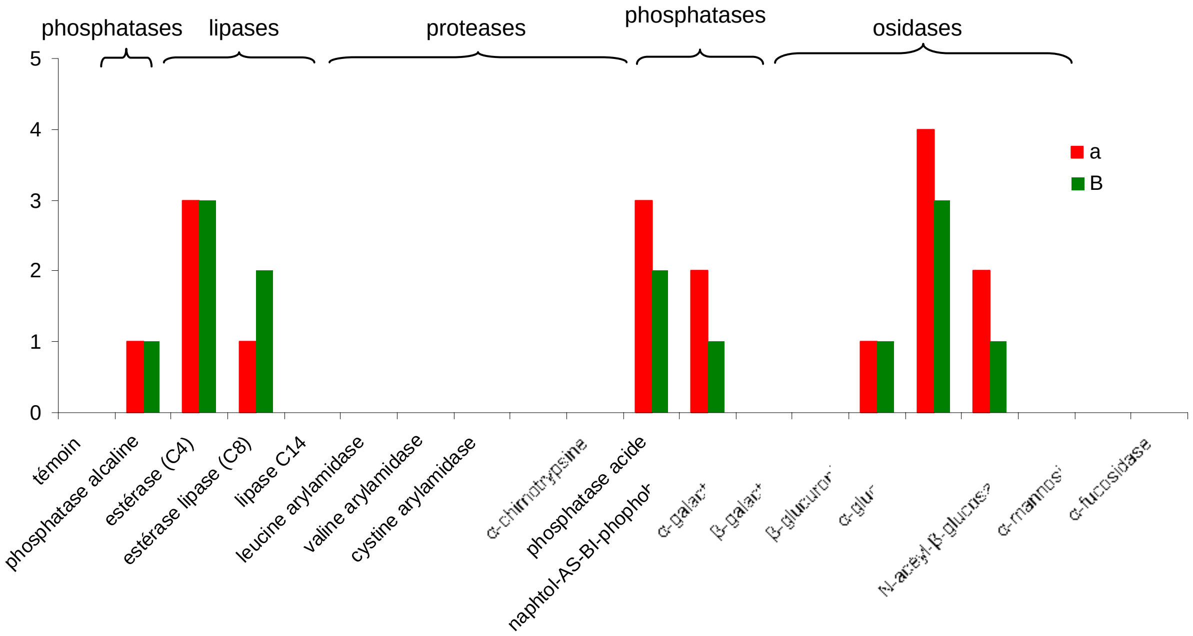
Microoragnisms isolated from compost are of great interest for industrial bioprocess since the selected bacteria are able to live in thermophilic conditions and to digest others microorganisms. Seven strains were isolated from compost and incubated at 60°C and 35°C on agar supplemented with anaerobically digested sludge. Two strains have presented colonies after incubation during 24 h at 60°C, confirming the thermophilic character of these microorganisms and their ability to use refractory biological material for growth (results not shown). Table 2 presents some microbiological characteristics of these two strains, named a and B.

Table 1 : Macroscopic and microscopic characteristics of hydrolytic bacterial strains a and B isolated from compost.

souche	colony characteristics	microscopic observations	Gram
a	big yellow, round, plate and smooth outline	long and large bacillus	positive
B	white, in nappe	very long and thin bacillus with spores	positive

The isolated a and B strains were cultivated for 24h at 60°C on TS liquid medium and the supernatants were recovered by centrifugation for further analysis of the released exoenzymes. Qualitative evaluation of the enzymatic activities was performed with an API ZYM® system. As shown in Figure 1, the two supernatants have a quite similar profile showing the presence of osidase, phosphatase and lipase activities while protease activities were not detected.

Figure 1: Test API ZYM® of supernatants a and B



Enzymatic assays using the specific chromogenic substrates, p-nitrophényl-α and β–D glucopyrannoside, confirmed the presence of α-glucosidase activity in both supernatants while β-glycosidase could be only detected in B (Table 3). No lipase activity could be detected using palmitate p-nitrophenol as a substrate (C16). This data confirms the specificity of the lipase activities towards their substrate. Indeed, previous APIZYM profile indicated that in both supernatants, C4 and C8 esters can be used as substrate but not C14. Enzymatic assays performed with the chromogenic protein azocasein confirmed the absence of protease activity. Tests performed with *Micrococcus lysodeikticus* for the detection of lysozyme -related activities were not successful.

Table 2 : Enzymatic activities of supernatants from a and B at 30 and 60°C.

souche	α-glucosidase ($\mu mol.ml^{-1}min^{-1}$)		β-glucosidase ($\mu mol.ml^{-1}min^{-1}$)		protéase ($\mu mol.ml^{-1}min^{-1}$)		lipase ($\mu mol.ml^{-1}min^{-1}$)		lysozyme (U)	
	30°C	60°C	30°C	60°C	30°C	60°C	30°C	60°C	30°C	60°C
a	0.0685	1.028	0	0.068	0	0	0.0068	nd	0.12	nd
B	0.0685	2.733	0.0308	0.0171	0	0	0	nd	0	nd

4. STUDY OF LYTIC POTENTIALS OF EXOENZYMES PRODUCED BY STRAIN A AND B IN PURE CULTURE

The ability of the selected a and B strains for efficient lysis of PHA producers was first evaluated on cellular suspensions of the Gram negative bacillus, *Cupriavidus necator* previously cultivated under phosphorus limitation in a batch reactor fed with acetic acid as carbon substrate. Such conditions allowed high PHB accumulation since extraction of PHB using the drastic chloroform/methanol method at 100°C indicated that the amount of intracellular PHA reached 80% of total dry matter.

After an osmotic shock to damage the external membrane, *C. necator* suspensions were incubated during 1h at 37°C or 65°C with supernatant a or supernatant B previously concentrated by speed-vacuum and added at two different Enzyme / Substrate ratios of 10 ml or 25 ml per g of DM. Quantification of the proteins by the bicinchonic acid method indicated that the concentrated supernatants a and B contained respectively 1,7 and 0.82 g of proteins per liter. Incubation of the *C. necator* suspension with lyzozyme at a final concentration of 3000 U/mg was also performed as a positive control. Figure 2 shows the lytic efficiency obtained in each condition.

Considering that the PHA content was homogeneous in *C. necator* cells, the percentage of cell lysis was evaluated after comparison of the amount of PHAs released after cell digestion with the amount of total PHAs (paragraph 2.7). Whatever the temperature or the Enzyme / Substrate ratios, supernatant B has lysed more efficiently *C. necator* cells than supernatant a. With the addition of 10 ml of B per g of DM, 99±2% of the cells were lysed at 37°C, and 92±2% at 65°C showing that the temperature increase did not enhance the performances. Similarly, the 2.5 times increase of the Enzyme/substrate ratio did not improve cell lysis efficiency.

Thus, supernatant B incubated for 1h at 37°C and used at 10 ml/ g DM (i.e 8.2 mg protein /g of DM) has presented excellent lytic efficiency on *C. necator* cells accumulating PHAs, with a percentage of cell lysis of 99% which is similar to that of the commercial lysozyme.

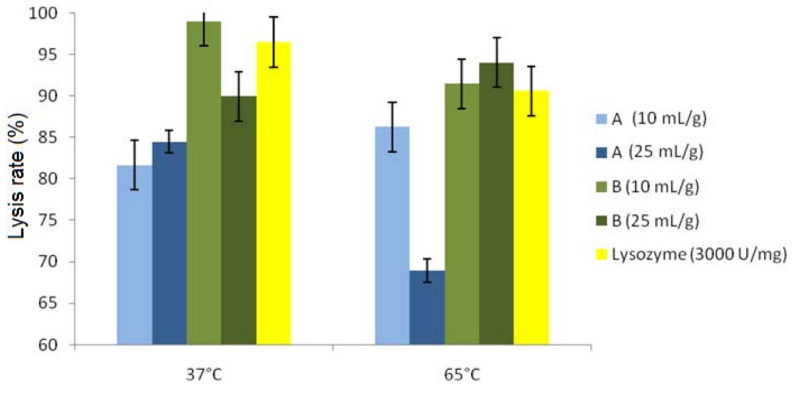


Figure 2: Lysis efficiency of *C. necator* after incubation for 1h with supernatant a, supernatant B or lysozyme at 37 or 65°C.

5. LYSIS OF MULTI-SPECIES CULTURES BY EXOENZYMES SECRETED BY THE STRAIN B

Supernatant B was thus selected for further lysis of a mixed-culture of unknown PHA producers cultivated under phosphorus limitation in a batch reactor fed with acetic acid as carbon substrate. The drastic chloroform/methanol method performed at 100°C indicated that the amount of intracellular PHA reached 59% of the total dry matter.

The lysis procedure previously performed on *C. necator* suspensions was applied on the PHA producers and the PHA recovery was 68±5 % with the supernatant B (20.5 mg of protein/g of DM) and 65±5 % with the lysozyme (3000 U/mg of MS). The lower extraction yield obtained on multi-species cultures compared to *C.necator* cultures can be explained by either the microbial heterogeneity of the PHA producers or by possible limitation of the cellular walls accessibility to the hydrolytic enzymes because of bacteria aggregation. Preliminary destructive treatments were thus performed before the lysis procedure in order to dissociate the bacterial flocs. The effect of various pretreatments including sonication , mechanical stirring or addition of detergents was compared. As shown on figure 3, all these treatments except the addition of non-ionic tween 20 significantly increased the percentage of PHB recovery.

Addition of SDS and ultrasonication have both greatly enhanced cell-wall digestion by lysozyme and B exoenzymes (up to 89% of PHB recovery) while vortex stirring appeared somewhat less efficient for cell digestion with lysozyme.

The use of Triton or Tween 20 for the extraction of extracellular polymeric substances in biological aggregates was previously described by Ras et al. (2008). Indeed, these natural non-ionic detergents allow disruption of the biological matrix and have interesting biodegradable properties.

The use of anionic SDS was also described in literature but very high SDS concentrations were necessary (20% w/v) for total PHB extraction from *C. necator* as a unique cell lysis process (Kim et al., 2003). Here, SDS chemical pre-treatment combined with enzymatic treatment allowed to obtain similar PHB extraction yield and 10 times less surfactant was necessary which represents a significant decrease of environmental impact of the global process.

In consequence, preliminar ultrasonication during 15 min with an ultrasonic probe delivering about 80W/g of DM can be included in the global procedure. This step allowed the best ratio of PHB recovery/pre-treatment time and about 89% PHB can be then extracted after osmotic choc and cell digestion by B exoenzymes at 37°C.

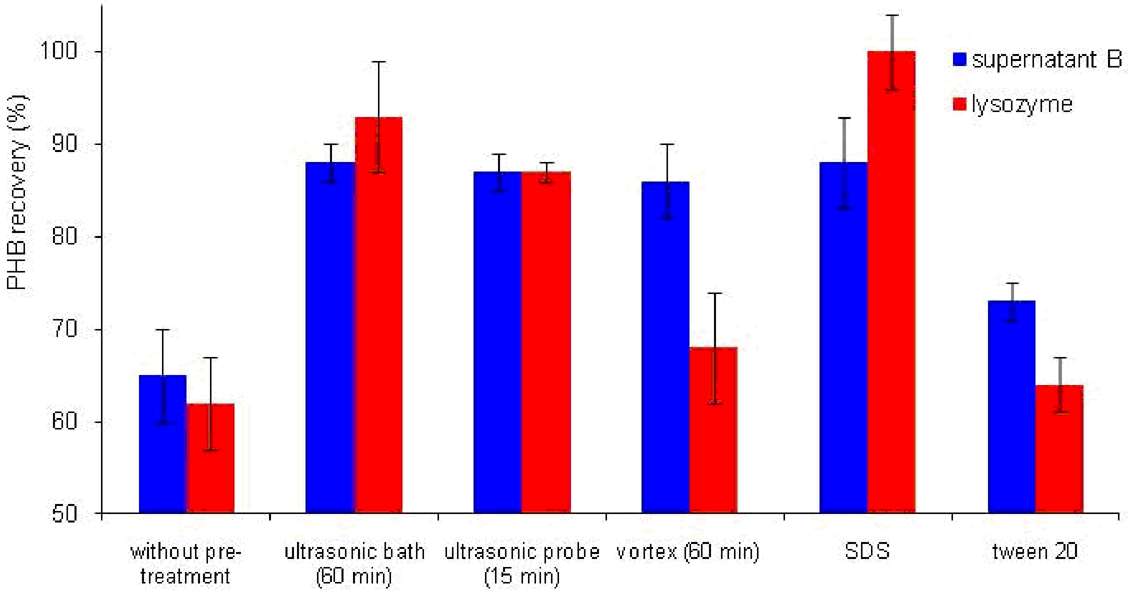


FIGURE 3 : **PHB recovery from multi-species culture of PHA producers obtained without pre-treatment (negative control) or mechanical pretreatment – vortex stirring (1 h at 20°C), ultrasonic bath (1 h, at 20°C), ultrasonic probe (15 min, 4°C) or with chemical pre-treatment – incubation in 2%SDS or 2% tween solution, 30 min at 20°C – followed of enzymatical treatment (lysozyme at 3000 U/mg of DM and supernatant B at 20.5 mg of protein/g of DM).**

6. CONCLUSION

This study demonstrates that enzymatic recovery of biogenic PHAs from various producers can be performed using lytic bacteria selected from compost, providing thus interesting reduction of the environmental impacts for PHAs bioproduction.

Two selected a and B bacteria were able to grow on anaerobic digested sludge and to secret several specific hydrolytic enzymes showing glucosidase, phosphatase and lipase activities. They can be thus of great interest for global reduction of various organic wastes and their valorization as renewable carbon source. The a and B exoenzymes were also efficient for cell lysis and PHA recovery from cellular suspensions of various PHA producers. Using B exoenzymes, all the intracellular PHAs can be recovered from *Cupriavidus necator* cultures.In mixed cultures of PHA producers organized under aggregated structures, a preliminary disruptive treatment must be however performed in order to improve cell accessibility for the hydrolytic enzymes.
A global PHAs extraction strategy based on enzymatic digestion and mechanical disruption of the PHAs producers was thus proposed. These results confirm the accuracy of B exoenzymes, instead of commercial lysozyme, for enzymatic lysis of bacterial PHA producers and underline the interest of these exoenzymes for general recovery of biogenic compounds using low cost and low environmental impact extraction.

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(178) FIRST STEPS IN THE DEVELOPMENT OF A COMPOSTABLE SHOE

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EXECUTIVE SUMMARY

The use of organic material as feedstock in the production of clothes, utensils and then in the industry has been a constant in human society. The exponential exploit of these natural resources generally does not return to the environment, because when the life cycle of these products are exhausted, often they are considered waste materials and find a final fate in landfills or industrial incinerators, with a high environmental cost.

According to consumable goods, like shoes, organizations estimate a worldwide consumption of 23 billion (USA) pairs by 2015. A very small percentage of them are considered biodegradable, mainly due to the use of vegetal plastics used in their manufacture. However, the combination of traditional materials in the shoe manufacture, matching metals, plastics and leathers (usually tanned with Cr) prevents its biodegradation and utilization.

The aim of this work is to take the first steps to develop a shoe, which, at the end of their lifespan can be treated by composting. Even during composting process or remaining in soil, the material should not pose an environmental damage. This way, the matter cycle will close, minimizing the environmental impact, making recycling possible and avoiding waste status.

The experiment was conducted with the (supposedly) different biodegradable materials that compose a shoe, like biodegradable thermoplastic soles, titanium-tanned leather, felt and latex insoles, cotton thread and natural glues.

Some preliminary composting trials were executed in an integrated system with 12 aerated static bioreactors (30 litres). Each bioreactor has two sensors for temperature control and a pipe system for conduct intake air and to evacuate the outlet gas from bioreactor to oxygen sensor. In these trials we tested the effect of the presence/absence of these materials in three organic wastes composting: sewage sludge (SLD); Organic Fraction of Municipal Solid Waste (OFM) and cow manure (MAN), using like bulking agent remains of pruning and gardening (TRV), with a 1:3 volume ratio.

The pilot scale composting trial was conducted in a static aerated bioreactor (1000 litres). The bioreactor has a forced air system, an oxygen sensor and three temperature sensors to monitor temperature at three different depths. An industrial PLC monitored, recorded and controlled temperature and oxygen levels continuously throughout the composting process. In this trial we used the mixture (waste-bulking-shoe) that had better results in the preliminary test.

After three weeks of intense biodegradative process in the bioreactor and two months of maturing phase in turning pile, the material was sifted to obtain compost. The compost was evaluated, among other parameters, for nutrient and heavy metals content, biological stability and phytotoxicity. Part of that compost is being used in a fertility test with pots in a greenhouse at different doses, different plant species and comparing the results with other chemical and organic fertilizers.

It seems like the presence of the shoes did not create a negative effect on the wastes composting. At the same time, shoes contribution to physical and chemical parameters of the composting process was evaluated.

In three weeks of composting, the total solids degradation of the shoes achieved a loss of 42% of their initial dry mass. Also, initials tests on the compost obtained in pilot scale reflected a moderate fertilizing capacity and a negative phytotoxic effect.

1 INTRODUCTION

The use of organic material as feedstock in the production of clothes, utensils and then in the industry has been a constant in human society. The exponential exploit of these natural resources generally does not return to the environment, because when the life cycle of these products are exhausted, often they are considered waste materials so they find a final fate in landfills or industrial incinerators, increasing even more the environmental costs. Consumers throw away 30 kg·person^{-1}·year^{-1} of clothing and textiles (University of Cambridge, 2006), while the average for US citizens is a little higher, 30,8 kg·person^{-1}·year^{-1} (Claudio, 2007).

The European Commision developed a *Thematic Strategy on the Sustainable Use of Natural Resources* with the overall objective of decoupling economic growth from environmental degradation through improved resource efficiency and reduced environmental impact of resource use (Bathes, 2008). According to consumable goods, like shoes, organizations estimate a worldwide consumption of 23 billion (USA) pairs by 2015 (BLC, 2011). A very small percentage of them are considered biodegradable, mainly due to the use of vegetal plastics used in their manufacture. However, the combination of traditional materials in the shoe manufacture, matching metals, plastics and leathers (usually tanned with Cr) prevents its biodegradation and utilization.

The concept of use organic materials in clothes, footwear and accessories to return them to the nature when the lifecycle is over, already exists (Gam *et al*, 2010) but should be supported. Even when the concept of “cradle to cradle” was introduced by McDonough and Braungart (2002), classifying each material as a biological nutrient that can be easily biodegrade without any negative impact for the environment, in 1994 the Snipe brand made a biodegradable shoe named *Snipe Nature.* All the materials of that shoe were organics and without any harmful substance for the environment, and it received the prize to the innovation from the government of Valencia (Spain) in 1994. This work follows that initiative.

1.1 Research objectives

The aim of this work is to take the first steps to develop a shoe, which, at the end of their lifespan, can be treated by composting and finally return to soil like nutrients. Even during composting process or remaining in soil, the material should not pose any environmental damage. This way, the matter cycle will close, minimizing the environmental impact, making recycling possible and avoiding waste status for this shoe. Objectives were divided in two points:

- Assuming that most of the materials that would be part of the shoe were biodegradable, investigate how would they be degraded under composting conditions.
- Determinate if there was any kind of effect, positive or negative, to the process and to the final compost if those materials were added to the composting matrix.

2 METHODOLOGY

As regards to main substrate studied (shoes), the experiments were conducted with the (supposedly) different biodegradable materials that compose a shoe, like biodegradable thermoplastic soles, titanium-tanned leather, felt and latex insoles, cotton thread and natural glues. Their manufacturers had analyzed leather, soles and glues for biodegradability, but there were not data about the rest of materials. Another co-substrates used in this experiment were sewage sludge (SLD) from nearby SSTP municipality, cow manure (MAN) from local family farm, kitchen waste organic fraction (OFM) from university canteen, and remains of pruning and gardening (TRV) like bulking agent. The main substrate (SHO) required a previous conditioning process, because the shoes were provided totally dissembled, that is, each material (soles, leather, etc...) were provided separately. All the components of the shoes were hand cut with stainless steel scissors to obtain pieces of 3 – 5 cm and be placed in the mixes with the same proportions (mass %) that in original assembled shoe. In these preliminary trials we tested the effect of the presence/absence of shoes (SHO) in three organic wastes composting (SLD, MAN, OFM) and TRV.

For the first preliminary trial we tested four different composition mixes. One of them contained only TRV and was considering like the “blank” treatment, while the second was a mixture of TRV and SHO with 3:1 volume ratio. Another one was a mixture of shoes and sewage sludge with 3:1 volume ratio. The next mixture was composed by remains of pruning and gardening -previously sieved by 1 cm mesh- and sewage sludge with 3:1 volume ratio, and the last one was a mixture of remains of pruning and gardening, sewage sludge and shoes with 2:1:1 volume ratio. For the second preliminary trial we performed four new mixes. A mixture was formed with TRV and MAN with 3:1 volume ratio. The next one was realized with TRV, MAN and SHO with 2:1:1 volume ratio. Another mix contained a mixture of TRV and OFM with 3:1 volume ratio. The last mixture was TRV, OFM and SHO with 2:1:1 volume ratio.

These preliminary composting trials were executed in an aerated static bioreactors integrated system. The system is constituted by 12 individual bioreactors, 30 litres capacity each, designed as stated in Pérez *et al* (2008). Each bioreactor is a cylinder made of PVC, thermally isolated from the external conditions by a screw top lid and a case made off polyurethane, cork and wood. The cylinders show at the bottom a "plenum chamber"; which function is collecting the leachates that might occur and let a homogeneous distribution of injected air trough the mass.

Each bioreactor has three internal thermal probes of two different lengths (20 and 40 centimetres long), and the air for the composting system is supplied by an air compressor motor equipped with a 100 litres reservoir.

There are two system pipes to carry the air trough the composting system, the inlet air pipe (carry the fresh air injected to reactors) and the outlet air pipe (carry the air from reactors). At the end of the compressor motor, it is placed a flow-meter, equipped with a built-in analogical eletrovalve that keep a steady air flow in both inlet and outlet pipes. Similarly at the end of the outlet pipe is placed a gas analyzer (electrochemical oxygen detector) that analyzed only one sample from single bioreactor at time.

All temperature and oxygen signals are transmitted to a PLC (Programmable Logic Controller), which controls and register these parameters, in the same way, specific software allow us see the data and control the process in real time. The parameters control is realized quickly by the "PLC system alarms". For these trials we pre-established a 5% of minimum oxygen concentration, 55º C of maximum temperature value and an air flow rate of 5 $L \cdot min^{-1}$ (normal status) or 25 $L \cdot min^{-1}$ (alarm status) to ensure an optimum oxygen level inside the material. As a result of working with an integrated system, we could maintain optimum composting conditions anytime, so the system operate automatically against any alarm status (temperature over 55ª C or oxygen concentration below 5%) activating the air compressor motor and injecting air. Knowing the flow of air ($L \cdot min^{-1}$) and the O_2 concentration in the outlet air from each reactor, the Oxygen Uptake Rate (OUR) has been calculated during the process ($g\ O_2 \cdot h^{-1}$) (Puyuelo *et al*, 2010).

All preliminary trials mixes were three replicated and were conditioned to achieve a 55 – 60% of water content. Also, the volume and the weight of the load material of each reactor were registered at the beginning and at the end of the two experiments; even the leachates that might occur were collected. Both trials had duration of 15 days, where the sampling schedule included a previous analysis of each initial substrate, and a starting/final sample analysis of each reactor mixture.

After analyzing the behaviour of all mixtures in the preliminary experiment, we did not detect adverse effects by the use of shoes in composting process, so decided to design a larger scale composting trial to study shoes behaviour under a whole composting process (including maturation phase) and obtain a mature compost that could be analyzed. Once again, all the components of the shoes were hand cut with stainless steel scissors to obtain pieces of 15/20 cm and placed in the mixes with the same proportions (mass %) that in original assembled shoe. However remains of pruning and gardening (TRV) used like bulking agent, did not be previously sieved, since the reactor dimensions allow us working with industrial composting facilities particle size. For this trial we chose one of the mixtures that had better results in the preliminary tests. MAN, TRV and SHO formed the mix selected, with a volume ratio of 1:1:1.

The pilot scale composting trial was conducted in a static aerated bioreactor. The bioreactor is an isothermal structure of 1000 litres capacity with 4 temperature probes (3 wires PT-100); three of them are used to measure the material temperature at different depths (20, 40 and 60 cm), and the forth measure the external temperature. Also there is an air probe that sends an atmosphere sample from the inside of reactor to an electrochemical oxygen sensor. The bioreactor has two load gates (front and top); at the bottom there is a "plenum chamber" that let a homogeneous distribution of injected air trough the mass, and to collect the leachates (if any). The structure shows two ventilation holes, one at the top and another at the bottom. Thanks to the system pipes and valves configuration the fresh air injected by a powerful fan can be introduced upwards o downwards depending on the requirements of the process. All sensor signals are transmitted to a Graphic DAQ Recorder (Eurotherm 5100V), which controls and register temperature and oxygen parameters. This device provides a real time control of the composting process; when the temperature or the oxygen values exceed the limits of a pre-established working range, the fan turn on and inject fresh air to restore the conditions. In this trial the working conditions were 60ºC of maximum temperature and a 5% minimal concentration of oxygen to avoid the appearance of anaerobic conditions.

The final mix was conditioned to achieve a 55 – 60% of water content. Also, the volume and the weight of the load material were registered at the beginning and at the end of the experiment. The sampling schedule included a previous analysis of each initial substrate, and a starting/final sample analysis of the load mix, while the liquid effluents that might occur were collected and analyzed each two days once the trial was started.

The enhancement biodegradative phase, carried out inside of the reactor, had duration of 20 days. Once finished this phase, the material was removed from the reactor and a biopile of 1 meter high was built up. Throughout the maturation phase, parameters, such as oxygen and temperature, were monitored at least 3 times per week, and measured at three different points of the pile. As significative differences between external and pile temperature were found, three turnovers (one each two weeks) were made in order to detect a temperature rise. Also a pile sample was taken and analyzed the of 36th day maturation phase, to check the material status. After 60 days of maturation phase, and 80 days of composting process, the final material was sieved by 1 cm mesh, where bulking agent, compost and pieces of shoes were collected and analyzed separately to evaluate the physico-chemicals changes that would have occur.

2.1 Analytical methods

Samples were analyzed for total solids (105ºC, 24h); volatile solids (550ºC, 4h); pH (1:10, MicropH 2000); conductivity (1:10, Crison CDTM523); NH_4-N and N-NO_3 (Büchi distiller); N Kjeldahl, Organic Carbon (modified Walkey & Black method), total nitrogen and carbon (elemental analyzer Leco CN-2000) and heavy metals (ICP-OES Perkin Elmer Optima 4300 DV).

3 RESULTS AND DISCUSSION

3.1 Preliminary trials (reactors of 30 litres)

During the process temperature, O_2 concentration and OUR have been monitored for each reactor. All the mixtures experimented the rise of temperature and O_2 consumption expected in composting, with more or less intensity depending of the co-substrate used. Differences between the treatments with or without shoes were only found in the second trial, for MAN and OFM, where the reactors with SHO had more biological activity till the end of the experiment (figure 1).

Significative differences were only found in OFR treatment with and without SHO for: time to reach maximum temperature, time to reach thermophilic phase and time in thermophilic phase. The mass balance (table 1) did not show big differences related to the SHO presence or not. Only in the treatment with MAN there was a higher degradation of dry and volatile matter in the reactors with SHO.

TABLE 1 **Percentage balance of losses of fresh, dry and volatile mass for each treatment, with the average volume reduction**

Treatment	Fresh matter (%)	Dry matter (%)	Volatile matter (%)	Volume (%)
TRV	2,0 ± 0,1	15,8 ± 3	18,8 ± 3	0,6 ± 0,8
TRV+SHO	1,9 ± 0,04	15,5 ± 3,0	12,6 ± 2,9	0,3 ± 0,3
TRV+SLD	4,4 ± 1,4	17,1 ± 1,4	17,4 ± 3,4	18,5 ± 1,6
TRV+SHO+SLD	4,5 ± 0,0	14,5 ± 1,9	14,1 ± 3,2	14,2 ± 0,7
TRV+MAN	5,1 ± 3,2	10,4 ± 6,4	13,1 ± 6,1	2,6 ± 1
TRV+SHO+MAN	3,1 ± 0,3	24,4 ± 0,3	24,6 ± 0,5	2,7 ± 3,3
TRV+OFR	17,4 ± 1,9	8,4 ± 1,3	17 ± 2,1	5,9 ± 1,6
TRV+SHO+OFR	14,6 ± 2,0	11,0 ± 4,5	16,1 ± 4,5	9,0 ± 1,6

In all the treatments the final pH was higher than the initial. Conductivity was influenced by the presence of SHO, of such form that the final values were always higher than at the beginning (table 2). Temperatures and pH in the reactors increased the NH_3-N volatilization (Pagans *et al*, 2006), but SHO materials have an important water retention capacity, above the TRV, so there is a tendency that the soluble compounds related to the conductivity were also retained.

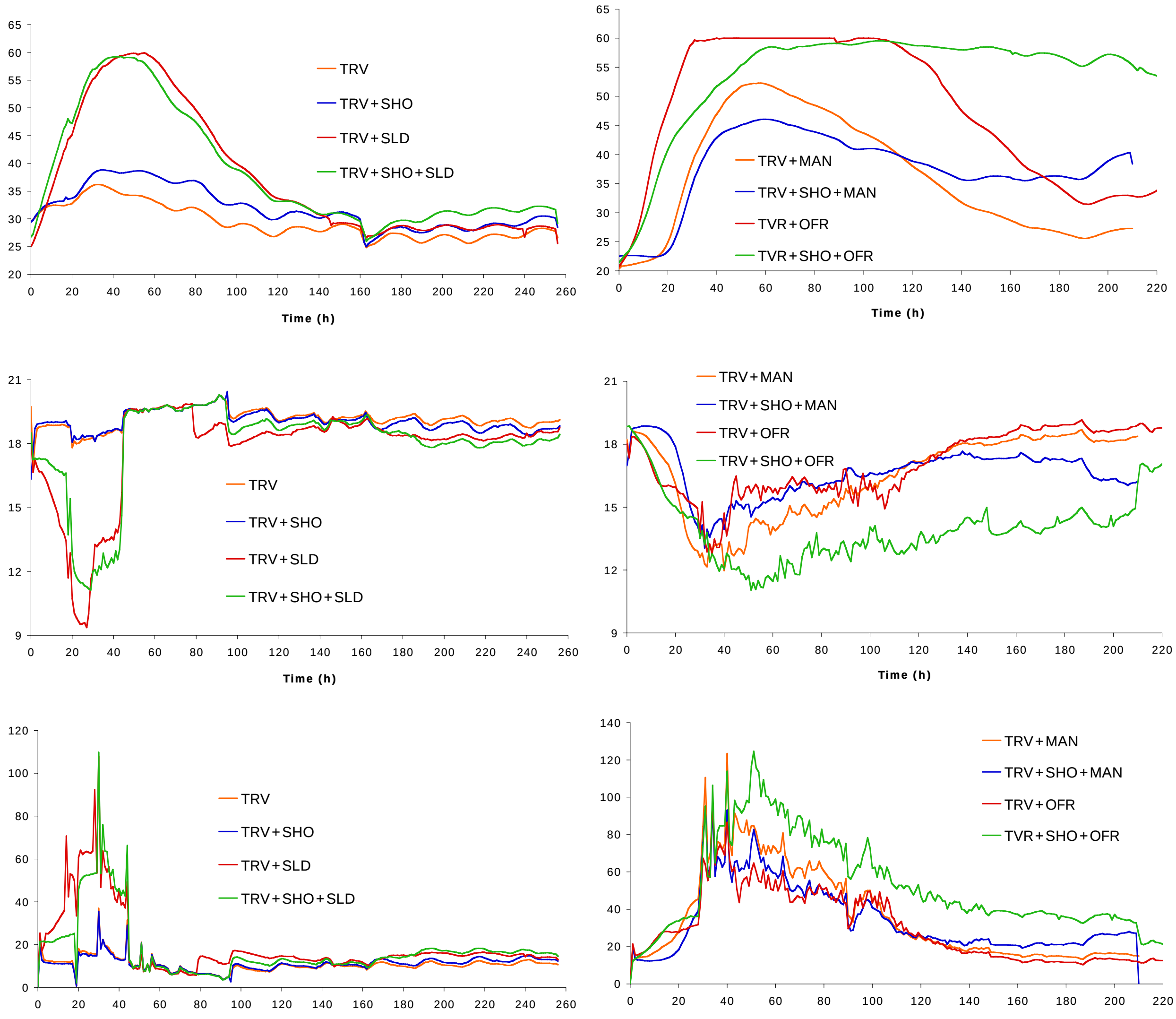

FIGURE 1 **Upper left, temperatures (ºC) of TRV and SLD treatments. Upper right, temperatures (ºC) of MAN and OFR treatments. Centre left, O_2 levels (%) of TRV and SLD treatments. Centre right, O_2 levels (%) of MAN and OFR treatments. Bottom left, OUR (g $O_2 \cdot h^{-1}$) of TRV and SLD treatments. Bottom right, OUR (g $O_2 \cdot h^{-1}$) for MAN and OFR treatments. Average values are represented.**

The losses of organic carbon have been different for the presence or absence of SHO depending of the co-substrates. For the treatments with only TRV the margin error of the analysis seems to be not enough to appreciate the differences between the initial and final organic carbon content. SLD treatments had more organic carbon losses in absence of SHO, but for the MAN and OFR treatments, the presence of SHO increased the degradation of organic carbon (table 2).

TABLE 2 **Initial and final pH and conductivity for each treatment. The final balance of organic carbon (%) was calculated over dry matter.**

Treatment	pH		Conductivity (µS ·cm^{-1})		Balance Organic Carbon (%)
	Initial	Final	Initial	Final	
TRV	6,9 ± 0,06	7,3 ± 0,02	148 ± 16,7	131,2 ± 16,4	2,1 ± 1,5
TRV+SHO	6,3 ± 0,04	6,7 ± 0,03	176,3 ± 26,9	201,4 ± 8,3	7,6 ± 4,4
TRV+SLD	7,0 ± 0,03	8,7 ± 0,09	490 ± 28,8	1.419,9 ± 50,5	-14,6 ± 3,0
TRV+SHO+SLD	6,9 ± 0,03	8,7 ± 0,04	466 ± 24,5	1.366,7 ± 69,0	-1,1 ± 0,4
TRV+MAN	6,5 ± 0,11	8,7 ± 0,06	2.116,7 ± 20,8	1.230,9 ± 28,7	-10,7 ± 12,6
TRV+SHO+MAN	6,5 ± 0,4	7,9 ± 0,25	998,3 ± 54	1.134,4 ± 191,9	-33,8 ± 1,3
TRV+OFR	4,9 ± 0,1	8,2 ± 0,37	1.694,3 ± 420,7	1.496,3 ± 169,5	-14,3 ± 1,7
TRV+SHO+OFR	5,5 ± 0,27	7,0 ± 0,93	1.824,3 ± 144,1	2.010,6 ± 311,6	-23,3 ± 7,0

Attending to the reduction of the oxidizable carbon in the SHO materials after the composting with the four co-substrates (table 3) the biological degradation was more efficient for the treatments with OFR and MAN. Its high C/N relation limits the composting of wood wastes, and the sludges were already partially stabilized after the depuration treatment, so it would explain the lower reduction of oxidisable carbon

TABLE 3 **Mass reduction (total solids) of SHO materials in each treatment and degradation of oxidizable carbon for the SHO materials after the composting with the four co-substrates.**

	TRV	SLD	MAN	OFR
Mass reduction of SHO (total solids)	39,7%	41,9%	37,9%	29,6%
Oxidizable carbon reducton	51,0%	58,5%	96,2%	93,8%

3.2 Pilot scale trial (reactor of 1000 litres and maturation pile)

In the preliminary trials the treatments with MAN and OFR had the better results attending to the degradation of oxidizable carbon. It was very easy to obtain more manure from the same cattle farm so this co-substrate was selected for the pilot scale trial. This trial was performed in two stages. The first 20 days of process happened in the closed system, with forced aeration. In the reactor temperature and inner O_2 and CO_2 concentrations were monitored and registered continuously, but in the maturation stage data (temperature and O_2) were measured every two days.

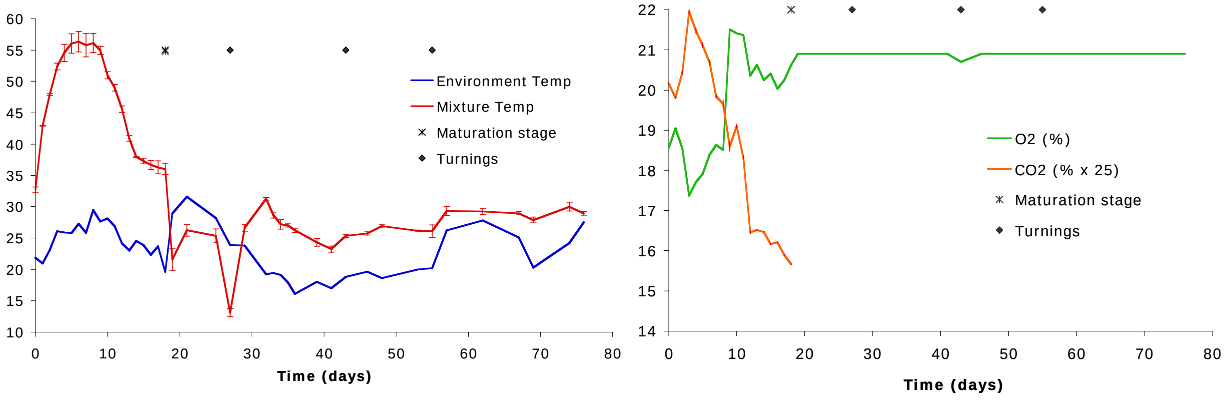

FIGURE 2 **Left, average temperature evolution in the pilot scale trial. Right, O_2 and CO_2 concentrations evolution. The maturation stage began when the reactor was unloaded and the pile was created. Turnings of the pile are signalled.**

Evolution of temperature in the reactor followed a pattern very similar to the previous trial (figure 3). The thermophilic phase lasted for ten days, followed for a fast cooling of the mass, even when the cycles of functioning of the fan were changed (data not show). Since the creation of the pile of maturation till the first turning, the mass temperature was cooler than the environment temperature, because the rate surface/volume was not adequate. At the first turning the pile was reconstituted in a conic form, so the losses of temperature were reduced. Moisture levels were periodically analyzed to prevent that could affect to the biological process, but watering was not needed during the maturation stage.

Oxygen levels were kept at atmospheric levels during all the maturation phase, and only were affected by the second turning, indicating that the biological process was not intense. After 70 days of process samples were taken for a *Rottegrade* test that indicated that the material was mature (grade V).

At 80th day the pile was sieved and the different fractions weighted and the compost was analyzed. In table 4 are show the analyses of the initial material (MAN, TRV and SHO) mixed and introduced in the reactor, as well as the final compost after 80 days.

TABLE 4 **Analyses of the different materials used in the pilot scale trial to constitute the initial mixture and the final compost. Only total solids expressed in fresh matter, the other parameters are expressed in dry matter.**

	MAN	TRV	SHO	Initial mixture	Compost
Total Solids (%)	15,0 ± 4,6	82,8 ± 1,8	95,5 ± 0,7	39,9 ± 2,1	26,4
Volatile Solids (%)	86,5 ± 4,9	95,3 ± 1,4	96,07 ± 0,2	95,5 ± 0,4	84,4
pH	6,8 ± 0,1	6,48 ± 0,2	4,1 ± 0,1	7,38 ± 0,3	6,5
Conductivity (mS·cm^{-1})	2,5 ± 0,1	1,0 ± 0,2	0,3 ± 0,1	1,2 ± 0,7	0,96
NH_4^+ (mg·kg^{-1})	6.144,8 ± 158,7	67,3 ± 9,8	48,5 ± 5,9	482,9 ± 59,3	140,42
NO_3^- (mg·kg^{-1})	184,3 ± 47,6	21,35 ± 8,1	14,33 ± 2,1	59,9 ± 7,2	25,07
N Kjedhal (g·kg^{-1})	22,3 ± 0,4	14,5 ± 0,4	49,5 ± 15,3	19,8 ± 0,9	20,7
N Total (%)	2,2	1,2	3,2	1,9	2,77
C org (%)	35,9 ± 0,5	36,0 ± 0,4	9,3 ± 0,7	34,9 ± 0,4	31,9
C Total (%)	41,4	45,7	59,9	43,5	40,7
C/N	19,2	37,5	18,6	23,4	14,7
Cd (mg·kg^{-1})	< 1,25	< 1,25	< 1,25	< 1,25	< 1,2
Cr (mg·kg^{-1})	2,97	6,71	61	11,1	9,99
Cu (mg·kg^{-1})	18,62	50,73	17,71	60,12	50,28
Ni (mg·kg^{-1})	2,77	5,07	22,06	4,91	6,04
Pb (mg·kg^{-1})	3,11	7,28	13,60	13,40	9,41
Zn (mg·kg^{-1})	101,00	90,40	5.270,00	181,00	408,58
Hg (mg·kg^{-1})	0,05	0,05	0,03	0,07	0,08
Ti (mg·kg^{-1})	149,40	102,60	985,50	383,80	510,73

The compost had high moisture content, and the volatile solids were still high, so maybe the biological degradation was not so intense as expected. Nevertheless there was an important loss of ammonia, but not of nitrogen due to the low temperatures and pH. Other nutrients in the compost that are not reflected in table 3 were K_2O (1,19 %), P_2O_5 (1,24 %) and MgO (0,71 %), at levels similar to other composts made from cow manure (Gil *et al*, 2008).

Titanium (Ti) was analyzed because of the tanned leathers. Even when it is an element with known benefits for the physiology of vegetals (chlorophyll contents, development of biomass,…) it could be toxic at high doses (Kuzel *et al*, 2003). So it could be a limiting factor to cropfields application. Fertilization trials have been made with this compost at the Public University of Navarra (Spain) to determinate the fertilization potential and the application doses. Results will be published soon.

Heavy metals concentrations were low, excepted for Zn, obtaining class B compost attending to Spanish legislation (RD 824/2005). The origin of Zn was in the SHO materials and later analyses demonstrated that it came from the latex used in the insoles. This specific material was dismissed and substituted for wool in the next steps of the development of the final shoe. Without this material in the SHO a class A compost would be obtained.

Germination percent (101,12%) and phytotoxicity action (-1,64) were also made to this compost (Zucconi & De Bertoldi, 1981, 1987). The negative value of the phytotoxic action and the high germination percentage regarding to the blank control indicate that this compost has a fertilizer value, with higher results than composts obtained from urban biowastes. Germination index (101,64) is correlated to conductivity and compost maturity, pointing out also that this was a mature compost.

4 NEXT STEPS

Experiments at composting facilities are being realised to investigate the biodegradation of these materials at real scale and their possible role as bulking material. Other experiments at home composters are also been made, to know the time and better conditions for the composting of these materials.

5 CONCLUSIONS

- All the preliminary trials demonstrate in the measured parameters that the presence of SHO in the composting mixture is not negative for the composting process. Significative differences regarding to benefits in the composting process because of the presence of SHO were only found in the treatment with kitchen waste.
- The structure of the SHO materials and the capacity of absorption the excess of moisture in the composting mixture allow them to act like a bulking material for other biowastes like cattle manure, sludges or municipal organic waste source selected.
- The degradation of organic carbon in the SHO materials was higher with OFR and MAN (93,8 and 96,2%).
- The compost obtained from cattle manure and SHO materials had a fertilizer value and do not evidence any phytotoxic effects.
- Latex in the insoles must me substituted to avoid levels of Zn in compost that would reduce its quality. With that change class A compost can be obtained in presence of SHO materials.

6 ACKNOWLEDGEMENTS

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(194) STRUVITE PRECIPITATION AS A TECHNOLOGY TO BE INTEGRATED IN A FARM SCALE ANAEROBIC DIGESTION-COMPOSTING TREATMENT PLANT

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EXECUTIVE SUMMARY

Manures which are potentially fertilizers or soil conditioners must be considered as resources that should be managed adequately. The farm SAT Sant Mer decided to include a biogas/composting plant to manage the excess of manure produced. In order to improve nutrient management and to reduce cropland requirements, the inclusion of a struvite precipitation process was assessed in lab-scale and pilot-scale experiments.

A serial of batch and continuous experiments were conducted to assess the influence of operational parameters on struvite crystallization (pH, temperature and alkalinity). A first group of sixteen batch assays were performed using synthetic liquor. The effect of variation in reactor mixing velocity and the effect of the introduction of CO_2 stripping were investigated in a second group of three experiments, carried out with the same synthetic liquor in a continuous reactor. Finally, a third group of three assays were carried out in the continuous reactor with digested swine manure, to investigate the effect of total solids and organic matter content

It has been observed that as the pH of the medium increased, the percentage of ion removal in the supernatant gradually increased. An optimum, in terms of process efficiency, could be set around pH 9 (99’86%±0’01 for phosphate, 95’49%±0’88 for magnesium and 89’43%±0’60 for ammonium removal). The effect of temperature on ammonia, magnesium and phosphate recovery as struvite was assessed also at 36ºC (simulating the outflow conditions from the anaerobic digestion process). No differences were observed in terms of process efficiency between 25ºC and 36ºC. The effect of the system buffer capacity and presence of other cations was assessed at equivalents alkalinities of 5.125, 10.25 and 20.50 $gCaCO_3/L$ with $CaCO_3$ or Na_2CO_3. When sodium carbonate (Na_2CO_3) was used, the percents of elimination were similar to the assays without added buffer. When calcium carbonate was used ($CaCO_3$), the percent of magnesium and ammonium removed decreased, while the percent of phosphate removed was not affected. This effect can be explained by the formation of other salts as dolomite ($CaMg(CO_3)_2$), hidroxyapatite ($Ca_5(PO_4)_3OH$) and calcite ($CaCO_3$).

Continuous experiments with the synthetic solution, performed in a lab-scale reactor of 20 L with a mixing velocity of 100 rpm, showed an ammonium removal efficiency of 66.03%±7.69. The removal efficiency was improved with a mixing velocity of 200 rpm (78.26%±0.14), although the size of the formed crystals was reduced. When the reactor was equipped with an aeration system for CO_2 stripping, despite a slightly lower ammonium removal efficiencies reported (71.93%±9.67) a reduction in reagents (NaOH) was obtained (15 – 28%). Continuous experiments with the digested pig slurry, performed with a mixing velocity of 100 rpm showed an ammonium removal efficiency of 85.18%±0.75. The removal efficiency was of 84.63%±0.91 when the mixing velocity was raised to 200 rpm. As expected, higher organic matter resulted in poor quality of the struvite in terms of organic matter contamination, and crystals size.

These results pointed struvite precipitation as a good alternative to improve nutrients management. Laboratory preliminary results were confirmed at farm-scale pilot plant. Nevertheless, results should be contrasted in order to establish/adjust operation parameters, operational cost, and quality of produced struvite.

1. INTRODUCTION

Manures which are potentially fertilizers or soil conditioners must be considered as resources that need to be managed adequately. According to this simple concept, manure must be handled as a by-product of livestock production and, when required, processed, for fitting the objective of an optimal management plan. Such objective must be determined for every farm or group of farms in a given area, based on nutrients mass balance in the influence area and considering local constrains and opportunities, such as incentives for renewable energy production (Flotats et al., 2009). Anaerobic digestion (AD) is a well-know and robust technology which converts organic materials into an energy-rich biogas. Currently, AD technology is well established in terms of performance and is technically and economically feasible.

The AD process displays a good performance when treating high strength organic wastes under conditions of controlled temperature. Nevertheless, it still has some drawbacks when applied to livestock manure. The role of nutrients is often central when dealing with manure management. The AD process does not affect the total nitrogen content, and thus it needs to be coupled with other processes for nutrient removal or recovery. The combination of the AD process with the ammonia stripping and its subsequent absorption in an acid solution (Bonmatí and Flotats, 2003a), or thermal concentration of the digestate (Bonmatí and Flotats, 2003b, 2003c) have previously been studied by our group. Although, their technical feasibility has been contrasted, their implementation is dependent on local conditions.

Struvite precipitation process consists in the crystallization of the nitrogen (ammonia, NH_4^+-N) and phosphorous (phosphate, PO_4^{3-}) present in the inflow in the form of struvite ($MgNH_4PO_4 \cdot 6H_2O$), also called MAP, which is considered as a slow releasing and valuable fertilizer (Yetilmezsoy and Sapci-Zengin, 2009). The struvite precipitation is produced when the concentration of Mg^{2+}, NH_4^+ and PO_4^{3-}exceed the solubility product. The main factors affecting the process efficiency are: pH, reactor design (agitation and sedimentation properties), process temperature, and presence of competitive cations, as calcium (Nathan et al., 2003). The recover efficiency as well as its quality are factors affecting its implementation in a certain country/area.

Pla de l'Estany is a county of the Girona province (Catalonia, Spain) designed as vulnerable zone, according to Decree 283/98 (1998) of the Catalan Government. In this context of nitrogen surplus, the farm SAT Sant Mer decided to build a biogas/composting plant to process and to manage the produced manure. Although the benefits of biogas production (incentive for energy production) and composting of the digestate solid fraction (stabilized and easier to handle-export product), is still needed large amount of croplands to absorb the nutrients (nitrogen and phosphorus present in the liquid fraction). In order to improve nutrient management and to reduce cropland requirements, the inclusion of struvite precipitation process to treat the liquid phase was assessed.

In the present study, struvite precipitation from anaerobically digested manure is investigated in batch and continuous lab-scale assays. Batch assays were conducted to estimate the reagents requirements and the optimum operational pH, while continuous assays were conducted to estimate the process efficiency and key limiting steps. Obtained results were validated in a pilot-scale facility located in the studied farm.

2. MATERIAL AND METHODS

2.1 Substrates and reagents

The synthetic solution used in the preliminary batch assays, and also fed into the continuous lab-scale reactor, was made with ammonium chloride (NH_4Cl), ammonium dihydrogenophosphate ($NH_4H_2PO_4$) and potassium dihidrogeno-phosphate (KH_2PO_4) to obtain a solution with a concentration of 625 mg N-NH_4^+/L and 4152 mg PO_4^{3-}/L. Reagents were added to obtain a ratio 1:1 (molar base) between ammonium and phosphate. All the chemicals used were analytical grade (Sharlab).

Centrifuged anaerobically digested pig slurry used in the continuous experiments was obtained from a centralized treatment plant (Juncosa, Les Garrigues). As can be observed in Table 1, ammonia concentration was greater than phosphate and magnesium concentration. Hence, the addition of phosphate and magnesium will be necessary to remove all the ammonium present in the slurry.

2.2 Experimental set-up

A series of batch and continuous experiments were conducted to assess the influence of operational parameters on struvite formation (pH, temperature and alkalinity).

A first set of sixteen batch assays were performed using synthetic solution. A second set of three experiments were carried out with the same synthetic solution in a continuous reactor; and finally, a third group of three assays were carried out in the continuous reactor with digested pig slurry.

Table 1. Characterization of the digested swine**.**

Parameter	**Units**	**Value**
pH	-	8,39±0,06
Mg^{2+}	mg/L	120,03±9,96
PO_4^{3-}	mg/L	43,92±62,11
NH_4^+	mg/L	4.341,69±45,13

2.2.1 Batch assays

Sixteen batch experiments were carried out in 600 mL glass vials. A volume of 400 mL of synthetic solution was mixed with a magnetic stirrer while 100 mL of magnesium solution was added with a dropper (during 90 min). All the experiments were performed in triplicate. After 90 min of reaction, stirring was stopped, and samples were taken. The supernatant fraction was analyzed in order to determinate removal efficiencies of Mg^{2+}, $PO_4^{3-}$and NH_4^+, and the solid phase was dried in an oven at 36 ºC for 48 h, and conserved for further analysis to determinate struvite purity.

Removal efficiencies of magnesium, phosphate and ammonium were quantified comparing the residual concentration of the supernatant with the initial concentration. On the other hand, a portion of the dried sample was solubilized in concentrate HCl and Mg^{2+}, PO_4^{3-} and NH_4^+ were analyzed by ionic chromatography (IC). The NH_4^+ content of the salt was used to estimate the amount of struvite obtained respect the total amount of salt precipitated, assuming that Mg^{2+}, PO_4^{3-}were in equimolar relation.

Three different operational parameters were evaluated; pH, temperature, and buffer capacity. The initial pH was adjusted with NaOH (2M) and was controlled along the experiment with a pH probe. The experiments were performed at room temperature, except the mesophilic assays, in which temperature was fixed at 36 ºC, placing the vials in a incubator. The influence of alkalinity was assessed using Na_2CO_3 or $CaCO_3$ at three different concentration (5.125, 10.25 and to 20.50 g $CaCO_3$/L).

2.2.2 Continuous assays

Efficiency of the struvite formation process, with synthetic solution (3 experiments) and digested manure (3 experiments) was assessed in continuous lab reactor. The effect of reactor mixing velocity and CO_2 stripping was investigated. The design of the crystallization lab-scale reactor used was based on the work of Mangin & Klein (2004). The scheme of the reactor set-up is shown in Figure 1. Basically, the experimental set-up consists in an isotherm glass reactor, three pumps for dosing the synthetic solution or pig slurry, magnesium solution, and sodium hydroxide to maintain the pH at the desired value. The alkali pump was controlled by a pH probe (Crison 5212) connected to a PID control system (Chemitec 4238).

The reactor (20 L) was divided in two zones, the reaction and a settling zone (Figure 1). The reaction zone was equipped with four baffles to prevent the formation of vortexes effect during mixing. The settling zone was located over the reaction zone and was cone-shaped. The effluent flowed out at the top of the settling zone, though a cylindrical baffler, that limits solids wash-out. Synthetic solution or digested slurry was fed into the base of the reactor together with the magnesium solution. Alkali was dosed along the experiment to maintain the pH at the set-point fixed (pH=9).

The reactor was operated in continuous mode for the liquid phase and in batch mode for the solid phase (manual solids discharge). Periodically the reactor was stopped, letting the struvite particles settle at the bottom of the reactor. Two mixing velocities (100 and 200 rpm) were assessed with each substrate, and in a third experiment CO_2 stripping with air was assessed in order to reduce the NaOH consume. The hydraulic retention time (HRT) was set around 20 h in all the experiments carried out.

Samples of the reactor outflow were taken during the experiment to measure Mg^{2+}, PO_4^{3-} and NH_4^+ content. Besides, at the end of each experiment, samples of the solid phase were taken in order to observe the crystals at the microscope and to analyze Mg^{2+}, PO_4^{3-} and NH_4^+ content.

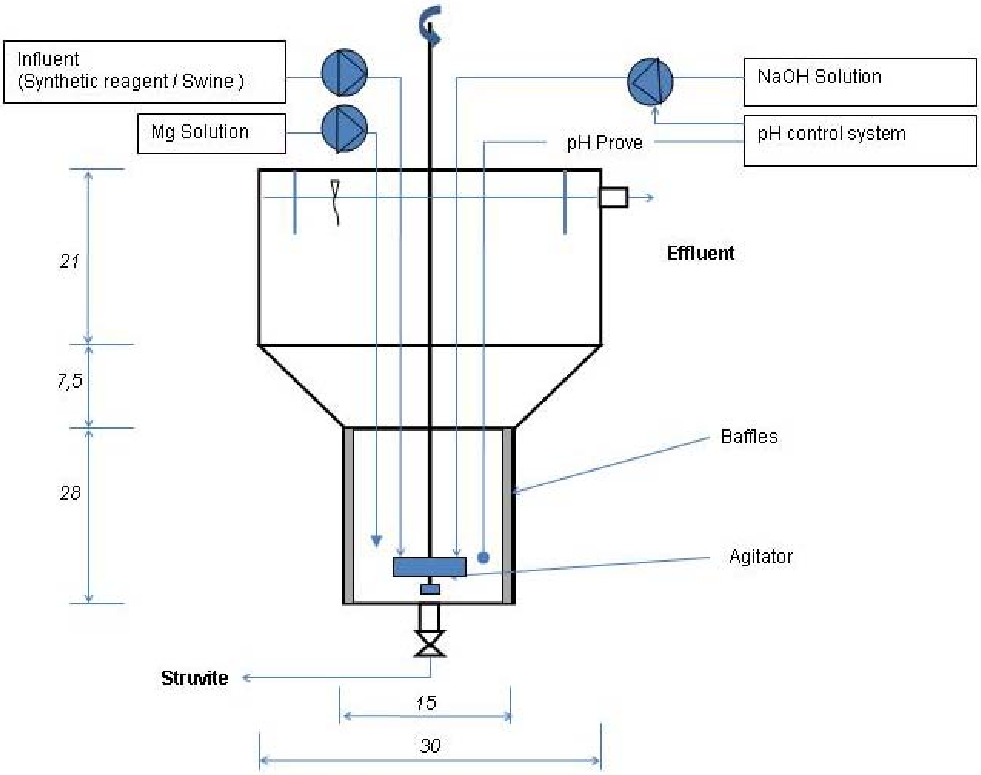

FIGURE 1 **Crystallization lab-scale reactor (size in cm)**

2.3 Analytical methods

Substrates and products characterization was performed by total solids (TS), volatile solids (VS), chemical oxygen demand (COD), total Kjeldhal nitrogen (TKN), ammonia nitrogen (NH_4^+-N), alkalinity (Alk) and pH determinations, according to Standard Methods (APHA, 1995).

Anions (Cl^-, NO^-, NO_2^- PO_4^{-2}, SO_4^{-2}) concentration were measured by ionic chromatography (861 Advanced Compact IC, Metrohm, Switzerland) using a column Metrosep A Supp 4-250 and CO_2 suppressor. Cations (Na^+, K^+, Ca^{+2}, Mg^{+2}) were also measured by ionic chromatography (790 Personal IC, Metrohm, Switzerland) and Metrosep C2 column. Samples were previously diluted and filtrated by nylon (0.45 mm) and BonElut JR C18 (Varian, USA) micro filters.

Images of the obtained crystals were observed upon light microscope IX71 (Olympus, Japan) equipped with digital camera DP70 (Olympus, Japan)

3. RESULTS AND DISCUSSION

3.1 Batch assays

The effect of different operational parameters (pH, temperature and buffer capacity) was evaluated in a series of batch assays using synthetic solution. The results of these experiments are showed below.

3.1.1 Effect of pH on the removal of NH_4^+, PO_4^{3-} and Mg^{2+}

The effect of pH on ammonia, magnesium and phosphate recovery as struvite was evaluated at pH 7, 8, 9 and 10 at room temperature (20-25 ºC). The results, in terms of supernatant elimination or reacted substrates (in % w/w) are presented in Figure 2.

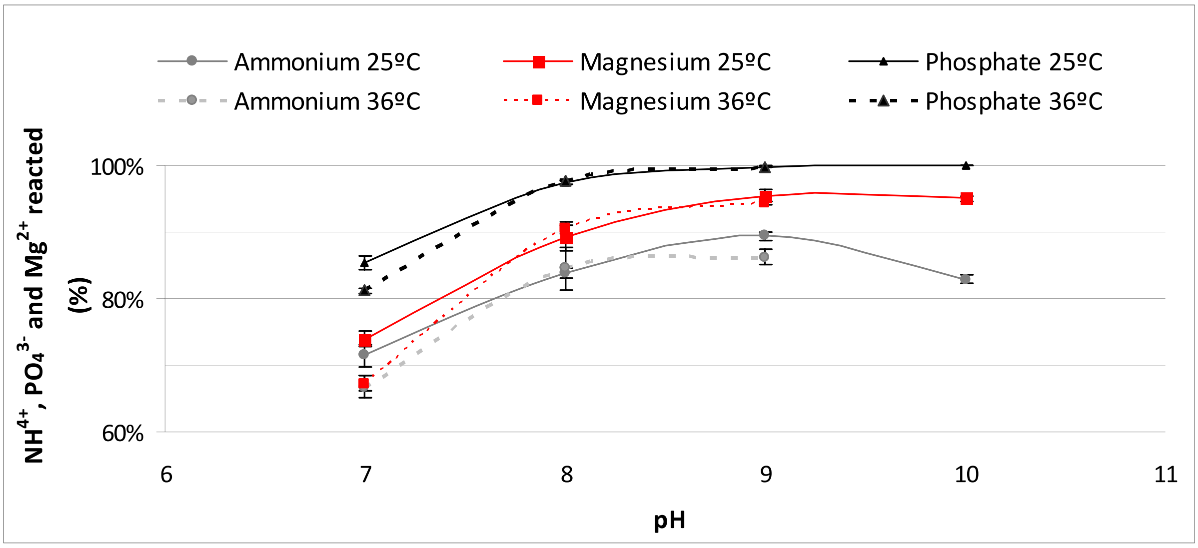

FIGURE 2 Percent of NH_4^+, PO_4^{3-} and Mg^{2+} reacted from the supernatant in batch assays

From Figure 2 it can be observed that as the pH of the medium increased, the percentage of ion removal in the supernatant gradually increased. An optimum, in terms of process efficiency, could be set around pH 9 (99’86%±0’01 for phosphate, 95’49%±0’88 for magnesium and 89’43%±0’60 for ammonium). These results are in concordance with previous studies (Doyle *et al*, 2000; Nelson *et al*, 2003; Yetilmezsoy and Sapci-Zenguin, 2009). Higher pH do not improved the percentage of ions elimination but represents higher NaOH requirements (with its consequence on operation cost).

Considering the formed salts or crystals, a higher quantity of visible precipitate is formed at the bottom of each vial when increasing the pH (Figure 3). The ammonium content of the salt was analyzed as previously described to calculate the amount of struvite produced. As can be seen, the amount of struvite increase when increasing the pH. The salts obtained at pH 10 is slightly higher than at pH 9, but probably other species than struvite are formed (Doyle et al., 2000; Çelen *et al*., 2007).

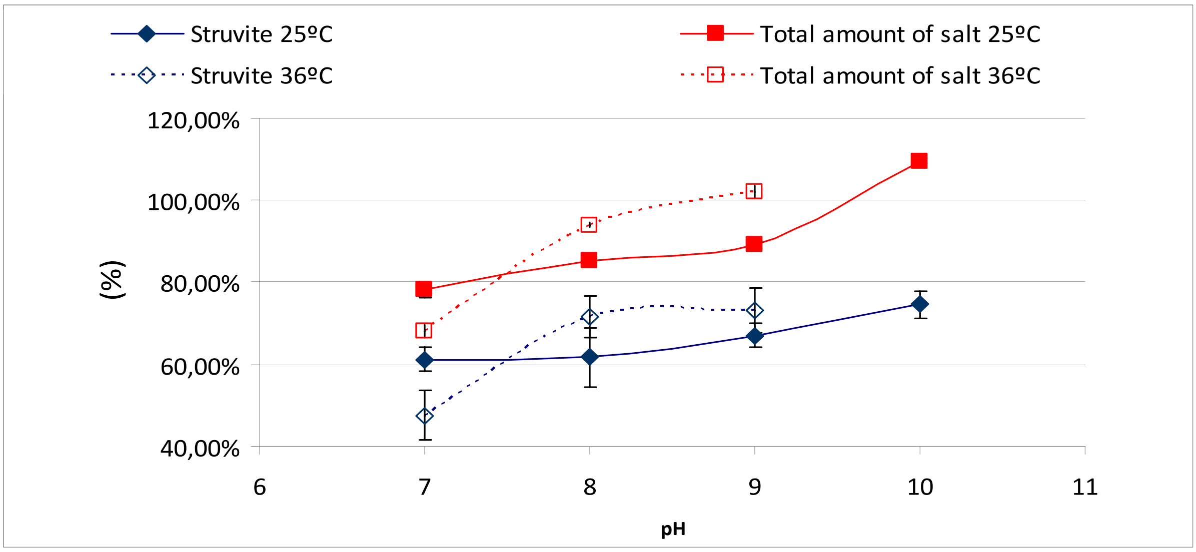

FIGURE 3 Percent (w/w) of salt and struvite obtained respect the maximum possible in batch assays

3.1.2 Effect of temperature on the removal of NH_4^+, PO_4^{3-} and Mg^{2+}

The temperature of effluent may affect the equilibrium of struvite formation, yielding on different ammonia recoveries (Doyle and Parsons, 2002). The effect of temperature on ammonia, magnesium and phosphate recovery was assessed at 36ºC (simulating the outflow conditions from the mesophilic anaerobic digestion process) and compared to previous results (Figure 2). No significant differences were observed in terms of process efficiency between 25ºC and 36ºC. Only at pH 7 the removal efficiency was slightly lower at 36ºC, compared to room temperature conditions. Similar results were obtained when analyzed the amount of obtained salts (Figure 3). This effect was also observed by Çelen and Türker (2001), when working with anaerobic digestion effluents in a range temperature between 25 and 40ºC. Consequently it can be concluded that temperature has a negligible influence on struvite formation at the range of temperature studied.

3.1.3 Effect of buffer capacity on the removal of NH_4^+, PO_4^{3-} and Mg^{2+}

The effect of buffer capacity of the liquid stream and the presence of other cations (Ca^{2+} and Na^+), over struvite formation was assessed. Three different equivalents alkalinities of 5.12, 10.25 and 20.50 g $CaCO_3$/L, using $CaCO_3$ and $NaCO_3$, simulating the mean, one half, and the double of the usual alkalinity of a digested slurry were essayed. Experimental results are presented in Figure 4. When sodium carbonate (Na_2CO_3) was used as buffer nutrient removal efficiencies were similar to the previous assays (without Na_2CO_3). When calcium carbonate ($CaCO_3$) was used, the percent of magnesium and ammonium removed decreased, while the percent of phosphate removed was not affected. According to stoichometric calculation, this fact can be explained by the formation of other salts as hidroxyapatite ($Ca_5(PO_4)_3OH$), dolomite ($CaMg(CO_3)_2$), and calcite ($CaCO_3$), as reported in Pastor *et al* (2008) and Moerman *et al* (2009).

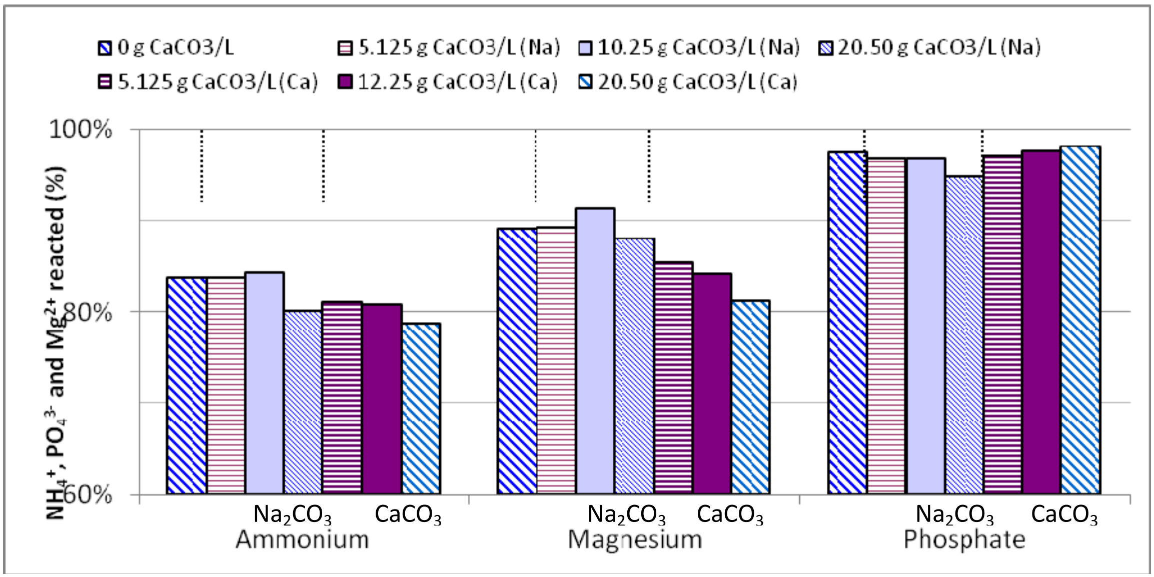

FIGURE 4 **Percent of NH_4^+, PO_4^{3-} and Mg^{2+} reacted from the supernatant in batch. Between parenthesis is shown if the assay was performed with sodium carbonate (Na) or calcium carbonate (Ca)**

3.2 Continuous assays

Mixing velocity (at 100 and 200 rpm) and stripping of CO_2 were evaluated in the continuous assays. Three assays with pure synthetic water and three assays with digested pig slurry were carried out.

3.2.1 Synthetic solution

Continuous experiments with the synthetic solution were performed in a lab-scale reactor of 20 L with a HRT of 20 h. Although struvite formation occurs at lower HRT (Pastor et al, 2008), a long HRT was set to favor crystal growth. Experiments with a mixing velocity of 100 rpm, showed an ammonium removal efficiency of 66.03%±7.69. The removal efficiency was improved when mixing velocity was increased up to 200 rpm (78.26%±0.14), although the obtained crystals are of a lower size (data not shown).

When the reactor was equipped with an aeration system for CO_2 stripping, despite a slightly lower ammonium removal efficiencies was reported (71.93%±9.67), a clear reduction in alkali requirements (NaOH) was observed (15 – 28%).

3.2.2 Liquid fraction of digested pig slurry

Continuous experiments with the digested pig slurry with a mixing velocity of 100 rpm showed an ammonium removal efficiency higher than that reported with the synthetic solution (85.18%±0.75). The removal efficiency was slightly reduced to 84.63%±0.91 when the mixing velocity was raised up to 200 rpm. Although the higher reported efficiencies obtained with the digested swine manure the size of the obtained crystals was clearly reduced respect the size of the crystals obtained with the synthetic solution, as shown in Figure 5. As expected, the presence of residual organic matter in the digestate resulted in poor quality of the struvite in terms of organic matter contamination and smaller size of crystals.

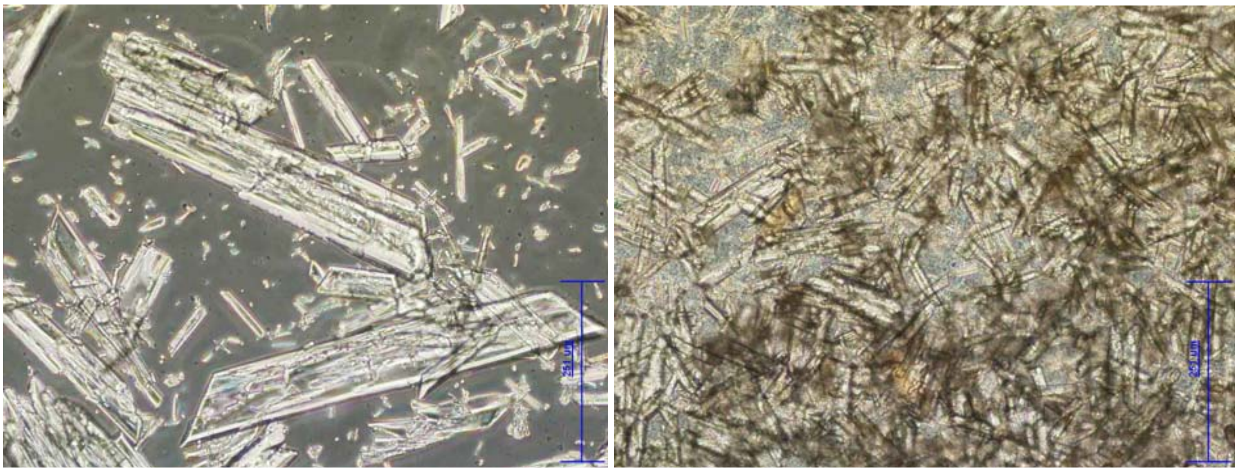

FIGURE 5 Crystals of struvite obtained in continuous assays with synthetic solution (left) and digested swine (right).

When the reactor was equipped with an aeration system for CO_2 stripping, the obtained ammonium removal efficiencies were slightly lower (71.76%±2.68) although a clear reduction in reagents (NaOH) requirements was confirmed (10%), concluding that aeration or CO_2 stripping is a reliable approach in real inflows to increase media pH and reduce operational costs (Suzuki et al., 2002)

3.3 Pilot plant assays and economic analysis

A 3.10 m^3 pilot-scale plant was built at the farm and preliminary results were obtained during 2 weeks of operation. Despite some operational problems due to a high solid content in digested slurry and deficient struvite settling, ammonia reductions of 52% were achieved.

Economical analysis were performed considering the data from the case study treatment plant, and the operational costs and the incomes of the struvite process have been estimated with data from a full-scale plant (Jaffer et al., 2002). Ammonia removal efficiency was considered to be 52%, and the production of struvite per m^3 of swine manure 16 kg. The price of struvite as fertilizer has been calculated as a function of its content of ammonium and phosphate (57 kg N/tn and 291 kg P_2O_5) and the price of fertilizers (1.36€/kg N and 1.40€/kg P_2O_5), resulting a price of 240€/tn, and the cost of reagents are shown in Table 2. The net production of energy considered was 34 kWh/m^3 that could represent a net income of 4.45 €/m^3. Taking into account all these considerations the treatment cost would be of 4 €/m^3.

TABLE 2 Evaluation of the cost of reagents

Reagent	Amount of reagent per m^3 of swine (kg)	Cost of reagent per m^3 of swine treated (€)
NaOH	10	0,75
$MgCl_2 \cdot 6H_2O$	30	3,45
KH_2PO_4	20	8,00

4. CONCLUSIONS

The following conclusions can be drawn from the presented work:

- Batch assays showed that the optimum operational pH for ammonia recovery was around 9; and the reaction temperature in the range studied (25 ºC - 36 ºC) did not have any influence on the struvite formation
- Calcium (Ca^{2+}) ions have a greater interference in struvite formation than sodium ions (Na^+).

- CO_2 stripping has been demonstrated to be a good way to reduce NaOH requirement to control the pH.
- Ammonia removal efficiencies in continuous assay with the digested pig slurry were reported to be 85%.
- Microscopic examination of the struvite crystals showed that the higher the pH, the longer the crystal, but the presence of organic matter reduces their size.
- So far, the reduction of ammonium reached in the pilot plant was 52%.
- Struvite precipitation technology in combination with anaerobic digestion is an interesting alternative for pig slurry processing. The removal of N and P reduces the area of cropland required to absorb the effluent of the treatment plant. Furthermore, the salt obtained is a valuable fertilizer that can be sold.

5. ACKNOWLEDGMENTS

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(205) MICROBIAL DEGRADATION OF PESTICIDES WASTES IN RUSTICS DEVICES TYPE BIOBEDS: THE BIOBACS

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EXECUTIVE SUMMARY

Derived from the system of biological beds proposed by Swedish researchers, called biobeds, and from the Bayer Crop Science system called Phytobac®, or the biobac, is a tank insulated from the subsoil and filled with a mixture of organic and mineral materials. Thanks to the developed biological processes in them, the biobeds can provide simple and attractive solutions for the confinement and treatment of pesticide wastes. A biobac can last for up to 8 to 10 years.

Despite the increasing interest shown for these rustic processes of bioremediation, limited data is available on their efficiency and monitoring. Biobacs specifications may vary with environmental conditions, waste volume and composition and pesticide concentration. The cost and availability of “carrier materials” and organic substrates used to support microbial activity must also be considered (biomixes). All these aspects need to be considered when improving biobeds efficiency.

The objective of this study was to confirm the efficiency of biobacs evaluating certain aspects such as: the importance of biomixes and the study of the microbial mechanisms that are developed during the treatment process (stimulation or inhibition degradation).

Numerous trials were first carried out under laboratory conditions to test the different materials which might be useful as biobed fillers. These were generally short experiments, using small samples and measuring the degradation of radioactive pesticides. Longer trials were performed in ten rigid crates containing 250 litres of substrate treated with one mixture of pesticides. In this case samples are removed periodically and the microbial activities determined from global or specific indicators such as biomass size or different catabolic capacities. The ability of soil micro-organisms from biobeds to mineralise each chemical was determined by radiorespirometry. Ecotoxicological assays risks were performed to the evaluation related to spreading the contents of biobacs in fields.

The work results have confirmed the effectiveness of the devices. It was show that less of 1% of a mixture of 14 products herbicides representing more than 100 g of active ingredients disappeared from organic or aqueous extracts of different biomixes analyzed 15 months after the last waste application. The adaptation of the microbial populations to the mineralization of compounds was shown. The presence of fungicides or insecticides, or other pollutants (motors oil, detergent) did not modify the degrading activity of the microflora. Nevertheless a small residual phytotoxicity was observed in certain biobeds.

1 INTRODUCTION

Derived from the system of biological beds proposed by Swedish researchers, called biobeds, and from the Bayer Crop Science system called Phytobac®, the biobac is a tank insulated from the subsoil and filled with a mixture of organic and mineral materials. Thanks to the developed biological processes in them, the biobacs can provide simple and attractive solutions for the confinement and treatment of pesticide wastes. A biobac can last for up to 8 to 10 years.

Despite the increasing interest shown in in several countries for these rustic processes of bioremediation, limited data is available on their efficiency and monitoring. Biobed specifications may vary with environmental conditions, waste volume and composition and pesticide concentration. The cost and availability of "carrier materials" and organic substrates used to support microbial activity must also be considered.

It is evident that a necessity exists for further knowledge about microbial behaviour in these complex ecosystems. It is necessary to develop simultaneously different experimental approaches to the problem of pesticide-wastes treatment.

The main problem in selecting suitable biobac materials is the need to cope very large volumes of waste. Another line of work in the biobed is to investigate certain bio-augmentation strategies. Inoculums from efficient degrading biobeds could probably be used as starters to initiate degradation in new installations.

These starters could also contain exogenous micro-organisms selected in different ways. Different strategies need to be tested for future research. Biobeds and related systems can be considered as solid-phase bioreactors. Therefore, as in liquid bioreactors, all parameters which favour microbial growth, such as nutrients, organic or mineral carriers and environmental conditions must be optimized to ensure the rapid pesticide degradation. This is the strategy which was chosen at the present time in Dijon. A second strategy could be to use a large ration of slowly-decomposing natural substrates such as straw manure or peat both to improve some cometabolic mechanisms but above all to trap the pesticide residues. A relatively low degradation rate can be expected for many pesticides under these latter conditions. Fungi rather than bacteria are probably involved and the risk of spreading bioavailable residues in the environment should be lower.

1.1 Research objectives

The objective of this study was to confirm the efficiency of biobacs evaluating certain aspects such as: the importance of the biomix (mix of organic substrates) and the study of the microbial mechanisms that are developed during the treatment process (stimulation or inhibition of the degradation).

2 METHODOLOGY

Numerous trials were first carried out under laboratory conditions to test the different materials which might be useful as biobed fillers. These were generally short experiments, using small samples and measuring the degradation of radioactive pesticides. Longer trials were performed in ten rigid crates containing 250 litres of substrate treated with one mixture of pesticides. The mixtures of substrates are showed in the table 2. In this case, samples are removed periodically and the microbial activities measured from global or specific indicators such as biomass size or different catabolic capacities. Chemical or physico-chemical parameters such as nutrient availability, pH or water capacity for example can more easily be measured in these conditions.

The ability of soil micro-organisms from biobeds to mineralise each chemical was determined by radiorespirometry. Methanolic solutions of radio-labelled atrazine, isoproturon, glyphosate, bentazon or diuron, 50µl per sample were added to soil samples (20 g dry weight) to supplement the samples directly with 0.1 mg or 1 mg of active ingredient and 1.55 kBq of radioactive material (one sample per treatment). Control samples received only methanol. Each sample was homogenized by handshaking and the moisture content was adjusted to 90% WHC. Sample bottles were put into sealed 1-1 glass jars and incubated in the dark at 20°C for several days to allow a significant mineralization of pesticides. Vials with 5 ml NaOH (0.2 N) were used to trap evolved CO_2 in the incubation vessels and released radioactivity was measured by liquid scintillation counting (Tricarb TR 1900, Packard) after mixing with 10 ml of a scintillation cocktail (Emulsifier Scintillator plusTM, Packard Instrumental Company).

3 RESULTS AND DISCUSSION

3.1 Incubations studies

Table 1 shows the effect of previous pesticides treatments in the soil content in the biobacs. The influence of the previous treatments pesticides was demonstrated. The maximum speed of mineralization for three of four products and for all the mixtures of substrate (biomixes) is systematically higher that the samples not treated previously. But then, the phenomenon does not appear with the glyphosate what could be related to the co-metabolic character of the degradation of this product.

TABLE 1 Influence of previous pesticides treatments in the maximum speed of mineralization (Sm in % degradation/day) of various products to the concentration of 100 mg /kg soil. (-) out, (+) in.

Biobacs substrate	***Pesticides***	***Atrazine***	***Diuron***	***Isoproturon***	***Glyphosate***
Soil	-	0,3	0,1	0,7	1,8
	+	4,9	5,6	10,7	1,9
Soil/ straw	+	2,8	6,3	5,8	2,3
Soil/compost/vermiculite	-	1,9	0,2	4,5	2,1
	+	4,0	5,3	15,0	3,9
Soil/straw/vermiculite	-	3,3	1,5	11,9	2,9
	+	5.0	9.8	15.0	2.1

Figure 1 show certain complementary studies carried out with different components of the biomixe: the compost of vegetal (14) and animal (17) origin. It is seen that the bentazon normally not degraded in the experimental soil, is quickly mineralized in the presence of the composts. On another side, during the same experimentation, we observed a significant inhibition of the mineralization of isoproturon in the presence of these same composts.

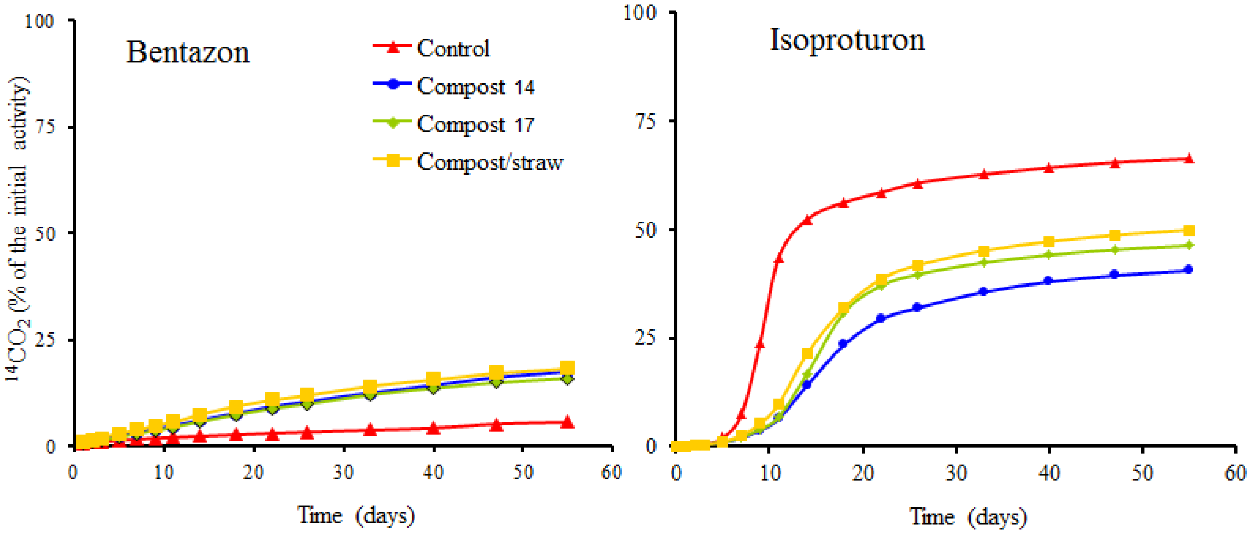

FIGURE 1 Effect of two natural composts and the straw on the mineralization of the bentazon and the isoproturon.

Farmers usually mix the soil with straw or peat to keep a good structure and aerate the medium. Peat can retain five hundred times its weight in water but the natural material is acid and may be inconvenient to most pesticide-degrading bacteria. The pH of the soil-peat mixture has to be increased in order to ensure a satisfactory degradation of atrazine in laboratory conditions. In our experiment calcareous sand was used to increase the pH. We are also testing calcium carbonate amendments or the addition of a phosphate buffer. Another problem could be related to the difficulty for micro-organisms to colonize the peat structure. We observed that adding a nutrient broth solution to the material permitted a more rapid establishment of pesticide-degrading activity.

We also tested vermiculite, an expensed clay, which is able to retain very large amounts of water but, unlike peat, does not bind the pesticide residues. Results have confirmed that atrazine can easily be degraded in a soil-vermiculite mixture but that very high water content is necessary for more efficient biodegradation. One possible problem with vermiculite could arise from the fragility of thin clay sheets. Therefore, new experiments are now being carried out using perlite, another clay material with 70-80% SiO_2, which is usually used as a carrier for bacterial inoculants.

Expanded clay beads with low water capacity have been used in certain experiments. Despite the introduction of high volumes of water, rapid degradation of the pesticide was maintained for several months.

In accordance with our initial results, the mixture of wheat straw and soil had a negative effect on atrazine and pendimethalin degradation.

This could result from the weak bioavailability of pesticides resulting from strong fixation to the organic material (similar problems were also observed in separate experiments with farm manure instead of straw). The addition of ammonium nitrate to facilitate straw decomposition again reduced the degradation rates of both pesticides. Much further research is needed on the choice of suitable carriers and substrates for biobed studies. Special attention must be paid to the relation between water potential and pesticide or microbial substrate availability in these complex material mixtures.

Recent experiments with such isolated strains have been carried out in our laboratory. We especially focussed on some of the difficulties associated with such inoculations. The herbicide degrading strain used in these trials was more efficient in a soil very similar to the one from which it had previously been isolated (pH 7.3) than in an acidic soil (pH 5.4). Complementary experiments confirmed that this was not correlated with a difference in pesticide bioavailability between the two soils. Delaying pesticide treatment in the inoculated soil resulted in a decrease in the degradation rate especially in the acidic soil. These observations could be also explained by the decrease in the organic carbon content of the soil. However, pesticide retreatment of the successfully-inoculated soil after one month incubation resulted in rapid degradation of the compound.

3.2 Analysis of pesticide residues from biobacs

Table 2 shows remaining quantities of pesticides residues and substrates loaded in biobacs 15 months after the last contribution of effluent. Results of pesticide multi-residue analysis indicate that less of 1% of the initial quantities remain (102,35 g) after 15 months of the last contribution. The effectiveness of the biobacs was confirmed; however the influence of the nature of the biomix was not clearly established in our study.

TABLE 2 Assessment of pesticides residues all analysed molecules, in each biobac 15 months after the last treatment with pesticides.

Biobac	*Substrate (biomixes)*	*Volume (litres) and percentage (%)*	*Total pesticides (g) (all the molecules)*	*Remaining amounts (mg)*	*Remaining amounts (%)*
1	Soil	200 (100%)	without pesticide		
2	Soil	200 (100%)	102,35 g	809 mg	0,79%
3	Soil/vermiculite	100/100 (50%-50%)	102,35 g	610mg	0,60%
4	Soil/peat/vermiculite	100/50/50 (50%-25%-25%)	102,35 g	343 mg	0,34%
5	Soil/compost/vermiculite	100/50/50 (50%-25%-25%)	102,35 g	247 mg	0,24%
6	Soil/compost/vermiculite	100/50/50 (50%-25%-25%)	without pesticide		
7	Soil/straw/vermiculite	100/50/50 (50%-25%-25%)	102,35 g	571 mg	0,56%
8	Soil/straw/vermiculite	100/50/50 (50%-25%-25%)	without pesticide		
9	Soil/straw	100/100 (50%-50%)	102,35 g	643 mg	0,63%
10	Soil/compost/Pearlite	100/50/50 (50%-25%-25%)	102,35 g	296 mg	0,29%

The products not detected in the pesticide multi-residue analysis in biobac were: atrazine, DEA (deethylatrazine), chlorpropham, glufosinate, isoproturon, isoxaben, mesosulfuron, dethylterbuthylazine. The products present as traces (less than 1% of the initial quantity) were: DIA (deisopropylatrazine), carbetamide, diuron, chloridazon, ethofumesate, acid 3-MPP, glyphosate, phenmedipham, and the products present at more than 1% of the initial quantity were: AMPA, terbuthylazine, metsulfuron methyl. The primary metabolite of glyphosate is aminomethylphosphonic acid (AMPA). Although the glyphosate practically disappeared, the larger quantities of AMPA persist in the different biomixe 15 month after the last contribution from pesticides effluent. In the case of the terbuthylazine of the quantities of residues present in biobacs were relatively low.

The metsulfuron represents the only example for this study of a product not easily degraded in the biobac. About 50% of the initial quantities of product in the various biobacs were detected. The great stability of this sulfonylurea in alkaline medium can be the origin of the persistence of this product under the conditions of the study.

This results show the general effectiveness of devices of the type biobacs, nevertheless they show also the necessity to know the nature of effluents to adapt the nature of the biomixes, in fact, for the metsulfuron, by choosing a substrate more acid.

3.2 Potential inhibitors essays

The pesticides effluents often contain products of different nature, that is to say herbicides, fungicides, insecticides or not degradable products as the copper, the engine oils or the detergents. The table 3 show the potential inhibitors effect of products of different nature on the effectiveness of the biobacs. The results of this study suggest that the effectiveness of the biobacs has not been modified durably by the majority of the potential inhibitors associated with the diuron or with the glyphosate only the DNOC presented an important inhibiting activity and prolonged.

TABLE 3 Potential inhibitors effect of products of different nature on the effectiveness of the biobacs. Mineralisation of diuron-^{14}Ccarbonyl and the glyphosate-^{14}C. Averages and standard deviation for 4 samples by treatments.

Potential inhibitors products (mg/kg)	***% Mineralisation (standard deviation)***			
	Diuron		***Glyphosate***	
	($^{14}C0_2$ % of the initial activity)			
	10 days	***55 days***	***10 days***	***55 days***
Control without treatment	34 (2)	76 (4)	23 (0,7)	49 (0,8)
Copper oxychlor (160 mg Cu/kg)	40 (2)	82 (4)	23 (1)	49 (2)
Copper oxychlor (1600 mg Cu/kg)	30 (2)	78 (0,9)	20 (2)	44 (3)
Copper/Manebe/Zinebe(1600/550/550 mg/kg)	17 (2)	64 (6)	23 (2)	50 (0,7)
DNOC (1600 mg/kg)	7,4 (1)	34 (4)	23 (3)	50 (5)
The mixture fungicides*	32 (3)	72 (6)	24 (0,4)	50 (1)
Detergent (1600 mg/kg)	22 (5)	72 (8)	21 (3)	47 (5)
Engine oil 16,7 ml/kg	26 (3)	73 (4)	23 (1)	51 (3)

Only the DNOC inhibited an 80% the mineralization on the diuron after 10 days and 55% after 55 days. Several other products seem to exert a reversible inhibition on the mineralization. In a decreasing order was cooper oxychlor (50% inhibition after 10 days), the detergent (35%), and the engine oil (18%).

3.3 Ecotoxicology assays

The first part of this study shows the good effectiveness of the biobacs for treatment de pesticides waste. However a limit often with the development of the biobeds concerns their contents after use. For this reason it was decided to perform some test of ecotoxicological evaluation risk to spreading the contents of biobacs in fields.

Table 4 shows the results obtained of the ecotoxicology tests in samples of biomixes (or their aqueous extracts) collected after the last analyses of residues.

The principle of test MICROTOX is the measure of the inhibition of the bioluminescence of the *Vibrio fischeri* with the addition of toxic substances in the bacterial suspension. The aqueous extracts of the biomix the substrate did not inhibit the bioluminescence, therefore these subtracts of biobacs are considered nontoxic for this assay.

TABLE 4 Ecotoxicology assays results.

Biobac	*Substrate (biomixes)*	*Microtox assay*	[+] *Toxic effect on S. obliquus*	[¥] *Germination test*		
				Oats	*Radish*	*lettuce*
1*	Soil	non toxic	+ 26%	90%	93%	97%
2	Soil	non toxic	-43%	90%	83%	80%
3	Soil/vermiculite	non toxic	-43%	90%	83%	87%
4	Soil/peat/vermiculite	non toxic	-39%	100%	57%	57%
5	Soil/compost/vermiculite	non toxic	-9%	90%	83%	83%
6*	Soil/compost/vermiculite	non toxic	+43%	87%	80%	60%
7	Soil/straw/vermiculite	non toxic	-21%	90%	76%	56%
8*	Soil/straw/vermiculite	non toxic	+29%	90%	63%	83%
9	Soil/straw	non toxic	-54%	93%	63%	80%
10	Soil/compost/Pearlite	non toxic	+6%	90%	57%	53%

* Without pesticide treatment. [+] Percentage of inhibition or stimulation of the growth of the *S. obliquus* compared to the growth of the control biobac (algae in water distilled). [¥] Germination rate of seed at 7 days.

Compared to the culture control of *Scenedesmus obliquus*, the concentrated extracts of the biobacs control determine systematically a big raise of the cellular mass estimated by optical density. This increase is thus related to the favorable effect of the extract of ground on the growth of the algae. On the other hand, appears systematically an important reduction of the growth of the algae in all the concentrated extracts of samples coming from biobac treated with the pesticide effluents. These results are expressed as the percentage of inhibition or stimulation of the growth of the algae in compared to the growth of the control algae (algae in water distilled).

The results of the germination essays does not show effects on germination rate of the seed of oats, concerning to radish and the lettuce it seems that a reduction in the rate of germination at 7 days can be observed in the majority of the treated biobacs and in particular in the biomixe: soil/straw, soil/compost/vermiculite, soil/peat/vermiculite, soil/compost/perlite.

Some of the extracts of biobacs with pesticide effluents show a reduction of the growth of the radicle and roots compared to the control extracts. These results are illustrated in the figure 1 where the development of the radicle and the roots of radish are seen clearly

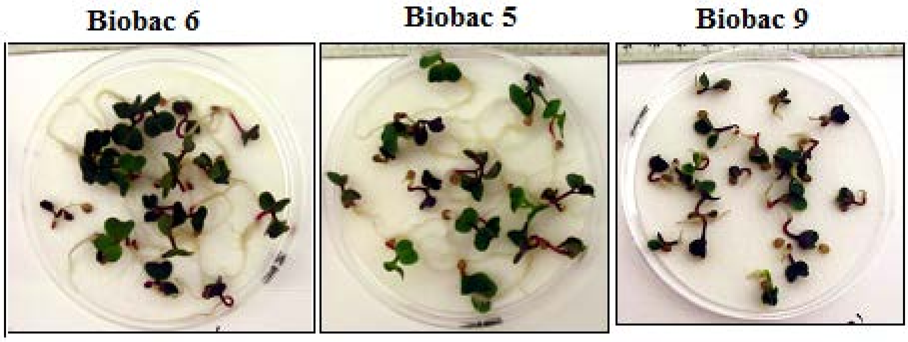

FIGURE 1 Development of the radicle and the roots of radish in the biobac 5 and 9 compared with growth observed in the control biobac 6.

4 CONCLUSIONS

The results obtained during this study confirm the effectiveness of the microbial degradation of pesticides wastes in devices type biobeds, but nonsystematic (case of the metsulfuron).

The nature of the substrate used in the biobac had not any influence in the reduction of the extractable quantities of pesticides residues after treatment, but the nature of the biomixe modified the individual dynamics of the products (example of the composts).

In the soil with frequent pesticides application, the phenomenon of the adaptation of degrading microorganisms is the origin of their strong degrading capacity for many pesticides, but this phenomenon is not systematic (case of the glyphosate)

The mixture of the different pesticides, the presence of copper or others co-pollutants as the detergent, the engine oil, etc., does not seem to influence in the system effectiveness.

The contents of biobacs 15 months after the last treatment with pesticides present an herbicide activity but this one seems reduced.

5 ACKNOWLEDGEMENTS

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Session 19

(9) COMPOST PILE MONITORING WITH GC-MS, E-NOSE AND OLFACTOMETRY: COMPARISON OF DIFFERENT APPROACHES

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EXECUTIVE SUMMARY

In many cases, odours emitted by municipal solid waste (MSW) plants are bothersome for the surrounding population. However, regulation and controls are difficult to determine. The complexity of emissions and the subjectivity of odour perception can partially explain this difficulty. Another concern is that the various techniques to measure such odours are not standardised. This paper compares complementary approaches to monitor odours. The odour source selected for this study is green waste compost at different maturity stages. The study site is a composting facility located in the south of Belgium.

A portable e-nose developed by the Environmental Monitoring Research team (Arlon, Belgium) was placed at different points of the facility to monitor odorous emissions from the compost piles. The sensor chamber consisted of humidity and temperature sensors and six Figaro™ metal oxide sensors (TGS2610, TGS822, TGS2620, TGS842, TGS2180, and TGS880). The device is battery powered and equipped with a pump that absorbs the odour through the sensor chamber with a flow of 200 ml/min. Simultaneously to the in-situ e-nose measurements, the volatile organic compounds were collected on Tenax™ cartridges for 30 min with a flow rate of 150 ml/min. Chemical analyses were performed in the laboratory using a TD-GC-MS (from Thermo).In addition, odours were also sampled simultaneously in Tedlar bags and subsequently analysed with the same e-nose in the laboratory. The aim was to compare the response of the sensors when used directly in situ or in the laboratory with odour sampled in bags. For e-nose measurements in the laboratory, odourless reference air was used to perform odour-odourless measurement cycles. The odourless air was collected in the field at about 500 metres upwind of the compost piles at the same time as the odours were sampled. For each sample, a second bag was also analysed by olfactometry according to the EN 13725 standard to determine odour concentration (ou_E/m^3) using the Odile olfactometer (Odotech). Certain parameters of the composting process such as temperature and turning date were considered to assess the maturity of the compost. The large number of data sets obtained was explored by statistical methods such as principal components analysis (PCA) and partial least squares analysis (PLS). An increase in odour concentration during the first days of the degradation process was determined by olfactometry. Most of the chemical families are terpenes, organic acids, ketones, aldehydes and alcohols like D-Limonene, Butanoic acid, Thujone, Hexanal and 2- Butanol, respectively. E-nose data are linked to the chemical composition and to the odour concentration. These preliminary results highlight the possibilities of monitoring the composting process as well as the released odours with simple measurement approaches.

1 INTRODUCTION

In recent years, odour nuisances from different industrial sources have become a serious environmental concern, especially in the case of odour emissions from municipal solid waste (MSW) plants. The principal reason for the increase in odours complaints is that industrial plants are situated nearer to urban and residential areas (Selena Sironi et al., 2007). The complexity of emissions and the subjectivity of the odour perception can partially explain the difficulties involved in determining regulation and control. Although odour legislation in the form of acts or regulations has been enacted in North American, Asian, Australasian and European jurisdictions (Bokowa A.H., 2010), the current legislation does not cover certain aspects related to the control and regulation of odour emissions.

For instance, the various techniques usually used to measure off-odours are not yet standardised. Nowadays, there is only a European olfactometry standard to measure the concentration of odour expressed in ou_E/m^3. So, there is really a need to improve the usual techniques or to develop new ones to help lawmakers. The need to develop an adequate monitoring of odour emissions allows achieving complementary approaches.While composting is one of the most efficient ways to treat different kind of organic wastes, it is always associated with off-odours and volatile organic compound (VOCs) emissions. These odours can be produced during the different stages of the process: reception, conveying, turning over, composting, and curing or storage. However, the major emissions of odour and VOCs occur from the reception step and during the turning over of the compost piles for the aerobic biological treatment (M. Delgado-Rodríguez et al., 2012).The composting process can be developed using different raw material such as municipal solid waste, poultry waste, household organic waste and green waste. The chemical composition of odour emissions depends on the waste materials, the level of decomposition and type of handling (Bidlingmaier and Müsken, 2007).In a previous paper, Romain et al. (2008) revealed the importance of odour emissions from landfill areas and composting facilities using different methods such as chemical analyses, dynamic olfactometry, sniffing teams and electronic noses.Dynamic olfactometry is a sensorial technique that allows determining the odour concentration of an air sample. A selection of panellists sniffs the sample in various dilution levels and indicates if they smell the odour of the diluted sample or not (European Norm EN 13725). This method is used in an air-controlled laboratory a few hours after sampling in the field. Many authors use this standard method to quantify odours emitted by the composting process (Paolo Littarru, 2007; P. Bruno *et al.*, 2007). The e-nose is able to monitor gas emissions in real time and in the field and to link them to the odour concentration expressed in ou (A.C. Romain et al., 2010), albeit with some limitations. Chemical analyses such as gas chromatography-mass spectrometry (GC-MS) provide information on the chemical composition of the emissions. However, the relationship between the chemical profile and the odour is not always well established (Tsai et al., 2008). All these measurement techniques are complementary and have drawbacks and advantages. Thus, the main objective of this paper is to compare these three approaches usually used to monitor odours and to highlight their utility in monitoring odour emissions as well as the process of green compost piles.

2 MATERIALS AND METHODS

2.1 Raw material and Sampling

The odour source selected for this study was green waste compost at different stages of maturity. The study site is a composting facility located in the south of Belgium. Typically, the facility had six to eight piles at different maturity stages. The final pile size was about 2.5 m high and 50 m in length. Aeration was achieved by turning the pile about twice a week. The odours released by compost vary with time and type of handling. Green waste composting is a slow process and the composition of pile compost varies so slowly that the odour emissions will depend on management. For this reason, VOCs and odour emissions are more influenced by turning activity and parameters such as temperature or humidity.

The measurement campaign was carried out over two months from 17 May to 20 July 2012. Three different batches of green waste were randomly monitored during this period. Measurements were taken in both the morning and the afternoon under different meteorological conditions.

The gaseous samples were collected in 60l-Tedlar bags and placed in a sealed barrel maintained under negative pressure by a vacuum pump. The samples of the emissions released by the different green compost piles were taken in duplicate.

One of the samples was analysed by olfactometry and the other with the electronic nose in the laboratory within a maximum of 30 hours after sampling. Moreover, an odourless sample was collected in the field about 500 metres up wind of the compost piles and analysed in the laboratory with the e-nose. The aim was to compare the sensitive range of the sensors in situ and in the laboratory. Adsorption on Tenax cartridges and e-nose measurements were performed directly in the field.

2.3 Dynamic Olfactometry

Dynamic olfactometry is a sensorial method that determines the odour concentration of an odorous air sample using selected panellists. Odour concentration is expressed in European odour unit per cubic metre (ou_E/m^3) and represents the number of dilutions with neutral air that is necessary to bring the concentration of the sample to its odour perception threshold concentration.

The analysis was performed using an Odile olfactometer (Odotech, Canada) according to EN 13725:2003 at the Olfactometric Laboratory of the Environmental Sciences and Management Department, Campus d'Arlon, University of Liége. The different dilutions of the odour were presented to a panel of 6 trained panellists by a decreasing step sequence in geometric series of factor 1.58

2.4 Electronic nose system

The odour emissions of the compost piles were monitored using a portable e-nose developed by the ULg team. The e-nose consists of a battery-powered sensor array and a PC board with a small keyboard and a display. The array contains six commercial metal oxide sensors (Figaro®) (TGS822, TGS2620, TGS2180, TGS842, TGS2610, and TGS880), a temperature sensor and a humidity sensor. The array is placed inside a thermostatic chamber and linked to a pump with a constant flow rate of 200 ml/min. Specific software controls the hardware and allows sensor signals to be obtained. The raw electrical conductance of the sensors is recorded every 15 seconds in the local memory. The data are then downloaded to be off-line processed by multivariate tools using Statistica and Matlab software. Different types of sensor arrays were used (see A.C. Romain et al., 2005 for a description).

2.5 Physico-chemical measurements

The volatile organic compounds were collected on Tenax^{TM} cartridges for 30 min with a flow rate of 150 ml/min using a calibrated air pump (**3Sc, lowflow module, constant flow** Gilair and **Gilibrator** (Gilian®)) and then desorbed using a thermal desorber (TD, Unity, Markes International) in the laboratory linked to a GC-MS (Thermo). The carrier gas was helium and a split ratio of 5 was applied on the sorbent tube. The chromatographic column is a CP-sil 8 CB MS (5% phenyl and 95% dimethylpolysiloxane) with a length of 50 m, an internal diameter of 0.25 mm, and a film thickness of 0.4 µm. The running conditions for the GC were initial temperature 38ºC, initial time 7 min, rate 5ºC/min, and final temperature 200-ºC. The compounds were identified by comparing the mass spectra obtained to the reference mass spectra of the NIST database (National Institute of Standards and Technology, United States) using XCallibur software. This software permits quick comparisons to be made and determines the degree of certainty of identification. Moreover, the retention times of the analysed compounds were compared to the retention times of the compounds previously identified by means of AMDIS in order to confirm some uncertain identifications and the qualification. External calibration was used for quantification. The concentrations of the substances were evaluated by external calibration at several calibration points (four different concentrations in the range of expected concentration). Standard samples were prepared in methanol solution and injected onto the cartridges ("spiking" method) using the Markes International™ calibration solution loading rig. External calibration was carried out using toluene following the ISO 16000-6 standard and the NIOSH d guidelines for VOC screening (Method 2549, VOC screening using multibed thermal desorption sorbent tubes, GC-MS, 1996). Hence, only semi-quantitative analyses were performed. The samples were analysed in the lab within a maximum of 4h after sampling.

2.6 Statistical analysis

The observations were analysed via processing software using statistical methods, specifically Principle Components Analysis (PCA) and Partial Least Squares (PLS) to highlight correlations between the sensors, the chemical families and odour concentration (ou_E/m^3). The data was pre-processed and treated using ad hoc software (Statistica and Matlab).

3 RESULTS AND DISCUSSION

3.1 Univariate correlation between the chemical-olfactometry-e-nose variables and composting maturity time

3.1.1 Evolution of odour concentration with time of maturity

Measurements taken over a total of 22 days were used to investigate the relationship between the e-nose data, the chemical analysis and the odour concentration during maturity time of composting piles. The results (Figure 1a) show that the odour concentration (ou_E/m^3) tends to increase during the first six days of maturity time to a value of 714 ou_E/m^3, after which an expected decrease in odours is observed. The initial odour concentration may be due to higher biological degradation in the first days of the composting process which generates volatile emissions. Odours are mainly caused by volatile organic compounds (VOCs) such as alcohols, esters, ketones, aldehydes acid and terpenes. This is in line with Schlegelmilch et al. (2005), who showed that odour concentration increases in the first stage of a composting cycle over seven weeks.

A rather low odour concentration range for the compost emissions was observed. This may be due to the composition of the green compost. Vegetable remains contain high amounts of cellulosic material which degrade slowly by microbiologic activity. Moreover, green waste releases lower emissions than household waste. In addition, it can be also an indicator of a good composting process. For these reasons, the less VOCs emitted, the lower the odour concentration.The odour concentration and VOC emissions showed different trends. Although both decreased with time of maturity, the odour concentration increased until day 6 of the composting process, while the total VOC emissions decreased with composting time. The study of each chemical family allowed determining that the concentration of some families increased the first days of composting as well as the odour concentration.

3.1.2 Evolution of chemical families with time of maturity

The chemical families studied were terpene, ketone, aldehyde, organic acid, alcohol, ester and hydrocarbon compounds. The results of the GC-MS analysis allowed detecting the highest concentration (as $\mu g/m^3$) of some volatile organic compounds at the start of the process. According to Scaglia B. et al., 2011, a total reduction of 98.6% of the VOC concentration was calculated from the start to the end of the composting process. The concentration of terpene and alcohol decreased with time of maturity as well as the total concentration ($\mu g/m^3$). On the other hand, the concentration of ketone, acid and ester compounds decreased with time of maturity, but increased on day 5, 5 and 6 of maturity time, respectively. These compounds exhibited the same behaviour as the odour concentration. The hydrocarbon compounds did not show a clear trend with time of maturity. The aldehyde compounds increased during the composting process. This increase could be due to interferences with the sorbent material used for the sampling (Tenax^{TM}). According to Raul Muñoz et al., 2010, despite recent advances in gas chromatography, several limitations remain. In particular, this analytical technique is still limited by: 1) the broad concentration range of the compounds present in odorous emissions and difficulties inherent in the preparation of multi-component gaseous standards; 2) the potential decomposition of thermally unstable volatiles compounds due to high operating temperatures in both thermal desorption units and injectors; and 3) water interferences.

3.1.3 Relationship between chemical families and odour concentration

The odours produced depend on the presence of organic volatile compounds in the exhaust air (Smet et al., 1999). A comparative study on the relationship between chemical families' concentration and odour concentration was realized. The results showed a good correlation ($r^2 = 0.83$) between the concentration of acid compounds and the odour concentration detected with dynamic olfactometry (Figure 1b). Moreover, acid compounds are one of the most odorant chemical families. The results did not show a high correlation with the other families or with the total chemical concentration.

3.2 Univariate correlation between e-nose with odour concentration, maturity time and chemical families

The results obtained in this section show a positive correlation between the conductance of the six sensors and odour concentration, except for the TGS 2180 which is very sensitive to humidity.

This sensor was used to correct the humidity, but not to develop the odour classification model or determine the chemical families (except for the aldehyde family, see previous explanation). Firstly, the linear correlation of each sensor with odour concentration was quite low, with the highest being $r^2 = 0.2$. The correlation with VOCs was about

0.5-0.6, slightly higher values than for the odour. Despite the sensors having a similar linear coefficient for total concentration and hydrocarbon compounds, each sensor has a different coefficient for each chemical family. This indicates that there was not a typical pattern for the hydrocarbon and odour concentration unlike the chemical composition emissions. In Table 1, the red values show the best correlation coefficient between each sensor and each chemical family. The results indicate that the sensors were more sensitive to alcohol and ester compounds. Finally, a negative coefficient was observed for composting time. Although this coefficient was low (about 0.3), it can be concluded that each sensor reacts as a function of maturity time. Due to the decrease in emissions according to maturity time, the sensors are able to respond to this timer.

3.3 Multivariate analysis: the e-nose data set

The previous results indicate the appropriateness of performing multivariate analysis, PLS and PCA to test the efficiency of the e-nose in monitoring the compost pile emissions.

Figure 2 shows a PLS analysis performed with the normalised variable taking into account the results of the 24 measurements made in the field and in the laboratory.

It is possible to observe the sensitivity of sensor TGS822 to alcohol compounds and the sensor TGS842 to hydrocarbon. Moreover, the sensors TGS2610 and TGS2620 have similar sensitivities. These results highlight the sensitivity of the e-nose to some odorous chemical compounds.

Principal components analysis (PCA) was also performed (Figure 3a) with the measurements obtained in the compost piles (forty observations). The odourless samples and TGS842 response (only sensitive to hydrocarbons) were not considered. The combined response of the sensors permitted obtaining PCA factors and provided global information of the chemical profile and chemical concentration of all the compounds. This PCA analysis, with the data described above, explained 93% of the total data variance.

Hence, odour concentration did not linearly correlate well to maturity time and chemical composition, with the exception of the acid compounds. Probably, more processing of data sets would be useful to develop an approach troubleshooting.

When considering the new components (factors 1 and 2) rather than the sensor responses, factor 1 versus maturity time were represented so that non-conforming measurements could be highlighted (Figure 3b). The results revealed some abnormal data which can be explained. June 14th coincided with a high value of odour concentration (the maximum value before a decrease in odour concentration). On June 24th the composting piles were turned, which may have increased the gas emissions. This activity could also have influenced the measurement taken on June 25th June. If we consider these new components rather than the sensors responses and we plot this factor as a function of time, nonconforming measurements can be highlighted.

4 CONCLUSIONS

- The odour concentration was not linearly correlated with chemical composition (except the acid compounds) and no linear correlation was observed with maturity time.
- There was no linear relation between odour concentration and maturity time as a peak of odour (714 ou_E/m^3) was observed on day 6 of the composting process, which could be due to the higher biological activity during the first days of composting.
- There was no linear correlation between chemical concentration and maturity time, with the exception of the acid compounds which is one of the most odorant families.
- Although the e-nose, using common linear multivariate approaches such PCA or PLS, was unable to accurately predict the odour concentration and maturity time. The combined responses of the sensors (the PCA factors) provided global information about the chemical concentration of all the compounds and highlighted some specific events.
- Compared to dynamic olfactometry and GC-MS, the advantage of the e-nose is that it is capable of monitoring emissions and odours in real time. The e-nose could be used as a rapid and easy detection tool to monitor the composting process in field as well as compost quality (acid compounds).
- It would be useful to develop a troubleshooting approach.

5 ACKNOWLEDGEMENTS

The authors are grateful to the Ministry of Science and Innovation for funding the PhD grant (BES-2009-012109). The authors would like to thank the Environmental Sciences and Management Department of the University of Liége for their collaboration.

Figure

FIGURE 1a **Evolution of odour concentration (ou_E/m^3) with maturity time (days)**
FIGURE 1b **Relationship between odour concentration (ou_E/m^3) and acid compounds concentration (µg/m^3)**

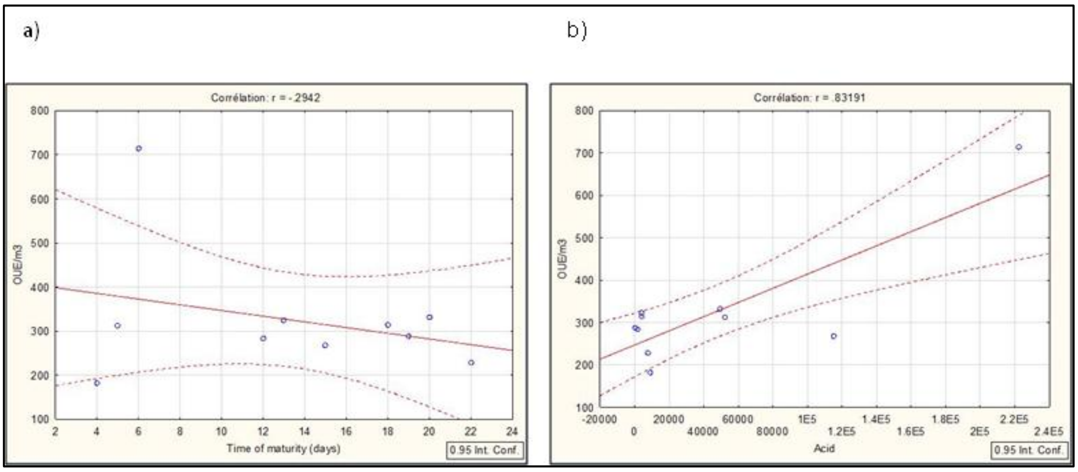


FIGURE 2 **Illustration by PLS of quantitative chemical families' concentration and sensor sensitivity**

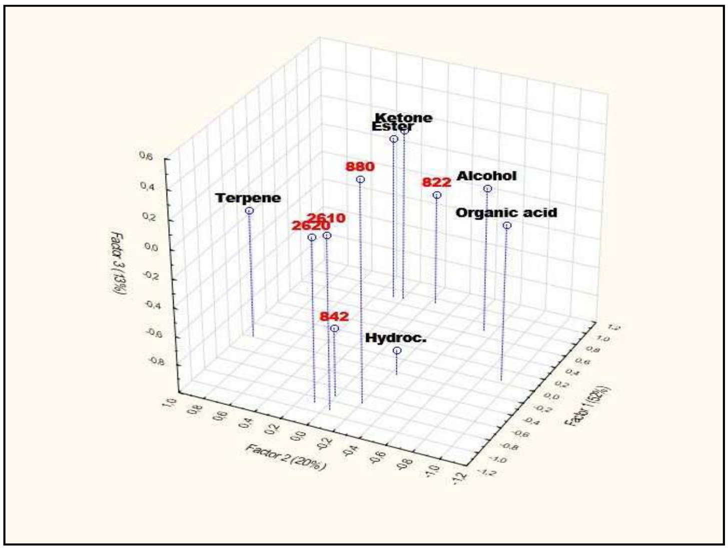
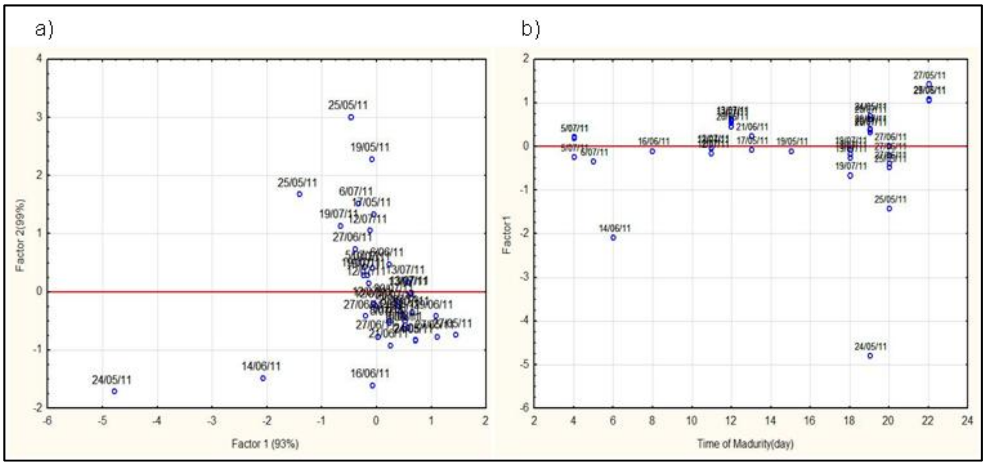


FIGURE 3 **Illustration of PCA**



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(12) COMPOSTING OF SEPARATED ANIMAL SLURRY SOLID FRACTION: EFFECT OF BULKING AGENT TYPE AND MIXING-RATIO ON GASEOUS EMISSIONS

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EXECUTIVE SUMMARY

There is increased interest in composting separated animal slurry solid fractions as a potential strategy to improve manure management and recycling. Composting, however, may result in greenhouse gas (GHG) emissions. The aim of the present study was to study the effect of solid fraction bulking agent type and mixing-ratio on gaseous emissions. Solids separated from animal slurry commonly have a wet weight moisture content of up to 70-80%. Due to this high moisture content, fibrous or bulky material such as wood chips or crop residues are commonly mixed with solid fraction to adjust the mixture moisture content, to provide structural support and maintain air spaces within the composting matrix, while enhancing the biodegradation process. Few studies, however, have been conducted to determine the effects of ratios of bulking agent to solid fraction on the emissions of CO_2, CH_4, NH_3 and N_2O during composting. Increasing concentrations of CO_2, CH_4, and N_2O in the atmosphere are agreed to lead to global warming. Nitrogen loss via NH_3 volatilization during composting may reduce the agronomic value of compost as a soil amendment, leads to eutrophication and acidification of land where it is deposited, and may also indirectly contribute to greenhouse effect. We, thus, determined the effects of different types of bulking agent: wood chips (WC), cereal residue (CR) and lupin residues (LR) addition on GHG emissions during composting of anaerobically digested, centrifugally (decanter centrifuge) dewatered solids (DEC). In order to isolate the effect of bulking agent type (different C and N availability) and mixing ratios of the compost on gaseous emissions, plastic tube (PT) as an inert bulking agent was also used for composting of DEC. Two mixing ratios (3:1 and 6:1 of solids:bulking agent on a dry weight basis), and a control with only DEC were evaluated. Experiments were performed in laboratory-scale (10 L) reactors for 28 d under condition of continuous forced aeration. Physicochemical parameters for solids, bulking agents and mixture of these were measured at the beginning and end of composting. Gaseous emissions were continuously measured using a photoacoustic field gas-monitor (INNOVA 1412) in conjunction with a multipoint sampler (INNOVA 1309). Results demonstrate that most physicochemical parameters exhibited a significant change during composting. Regardless of the differences in bulking agent types and ratios of bulking agent to solids, composting mass in all treatments reached thermophilic conditions. For all treatments, emissions of CO_2, CH_4 and N_2O were found to be the highest at the thermophilic stages, while NH_3 emissions exhibited a great variation during composting. During 28 d of composting, however, variation in CO_2, CH_4, NH_3 and N_2O emissions due to differences in the mixing ratios across all treatments was observed. This research helps to quantify GHG emissions from separated animal slurry solid fraction composting, and should lead to an insight towards better control of composting for reduced emissions.

1 INTRODUCTION

1.1 Background

Increasing environmental concerns associated with intensive livestock production and generated large volume of animal manure has lead farmers to seek alternative manure management techniques. In recent years, a wide range of commercially available solid-liquid separation as alternative technologies have been integrated into the animal slurry handling systems of many livestock operations.

The solid fractions produced during the mechanical and/or chemical separation process have a relatively small volume but usually contain high moisture content. Composting could be an alternative option to treat the solids fraction of animal slurry to stabilize the nutrient content in the final compost. Because of the high initial moisture content in solid fractions, high carbon substrate (bulking agent) such as woodchips or cereal residue are often mixed to provide adequate aeration within the matrix. Although composting process has been considered as a potential strategy to reduce greenhouse gas emissions, few studies have been conducted to quantify the gaseous emission during composting. Quantitative information about gaseous emission during composting could lead better management of solid fractions of animal slurry.

1.2 Research objectives

The objective of the present study was to quantify the effect of selected bulking agent type and mixing ratio on greenhouse gas (GHG) and NH_3 emissions during composting of anaerobically digested solids under laboratory-scale experimental conditions. The evaluation was based on observe change in physicochemical parameters, estimate of C and N transformation during composting and quantifies CO_2, CH_4, NH_3 and N_2O emissions.

2 METHODOLOGY

Anaerobically digested solids (DEC) (separated using a decanter centrifuge) was collected from a biogas plant in Denmark. Woodchips, cereal residue, lupine residue and plastic tube as bulking agents (particle size range: 25 to 30 mm) were obtained from locally available sources. DEC were mixed with woodchips in a 3 to 1 and 6 to 1 ratios (DEC:WC); with plastic tube in a 3 to 1 and 6 to 1 ratios (DEC:PT); with cereal residue in a 3 to 1 ratio (DEC:CR); and with lupine residue in a 3 to 1 ratio (DEC:LR). All mixing ratios were based on dry weight bases. In addition, a control with only DEC was included. Water was added to obtain a moisture goal of the compost mix prior to run a composting experiment (Table 1). Composting experiments were conducted in laboratory-scale (10 L) reactors for 28 d under condition of continuous forced aeration. Temperature and inlet air flow rate were recorded every 15 min during the composting experiments Gaseous emissions were continuously measured using a photoacoustic field gas-monitor (INNOVA 1412) in conjunction with a multipoint sampler (INNOVA 1309). The composting materials was analysed both before and after composting for total solids (TS), fixed solids (FS), pH, EC, organic nitrogen (Org-N), ammonium nitrogen (NH_4-N) and total carbon (TC).

TABLE 1. Composting treatments (db: dry weight basis; wb: wet basis; DEC: digested solids; WC: woodchips; PT: plastic tube: CR: cereal residue; LR: lupine residue).

Materials	Treatment	Mixing ratio of digested solids to bulking agent (db)	Moisture content (%, wb)	Abbreviation for treatment
DEC+WC	1	3:1	60	T1
	2	6:1	67	T2
DEC+PT	5	3:1	63	T3
	6	6:1	66	T4
DEC+CR	7	3:1	65	T5
DEC+LR	8	3:1	65	T6
DEC only	9	-	69	T7

3 RESULTS AND DISCUSSION

Results demonstrate that most physicochemical parameters exhibited a significant change during composting. After 28 d of composting, loss of moisture contents of all treatments was very small. This may be due to substrate confinement in laboratory reactors. With closed reactors, the amount of water that evaporated during composting could not easily exit the reactors. Instead, much of the water vapor became condensed on the inner walls, top of the lids and outlet tubes of the reactors before falling some amount back on to the composting mass, which could easily account for a small decreased in moisture contents.

We observed clearly this phenomenon when the reactors were opened for sampling. Relatively small decreased in moisture contents from all treatments may have substantial influences on gaseous emissions. After a 28 d of composting, changes in pH, EC, volatile solids were observed, and decreased in TN, TC and C/N ratios were recorded. Although there were differences in bulking agent types and ratios of bulking agent to solids, composting mass in all treatments reached thermophilic conditions (Fig. 1). However, temperatures produced from all treatments varied in terms of their obtainable maximum temperatures and the time required reaching the maximum temperature. This variability in the temperature rise between the treatments could be attributed to the availability of the readily biodegradable organic matter and nitrogen compounds for microbial growth.

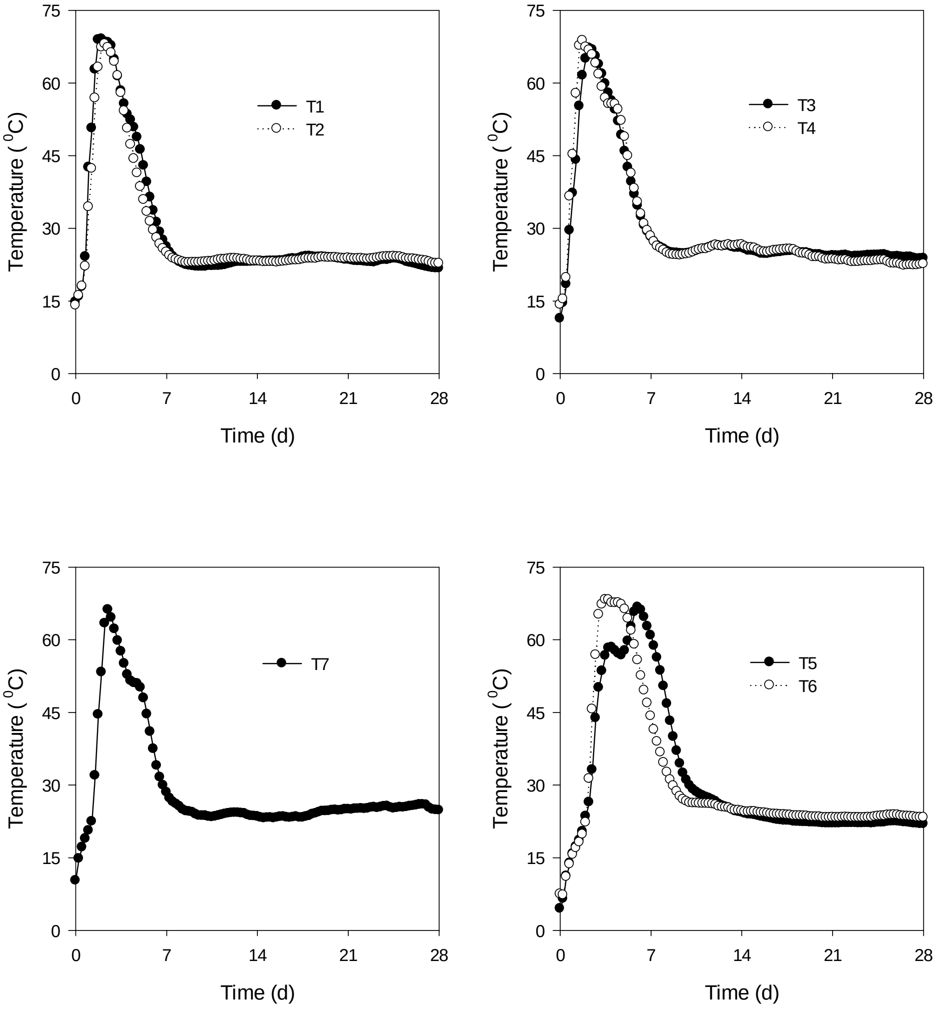

FIGURE 1: Temperature profiles during composting (T1-DEC:WC=3:1; T2-DEC:WC=6:1; T3-DEC:PT=3:1; T4 DEC:PT=6:1; T5-DEC:CR=3:1; T6-DEC:LR=3:1; T7-control). Error bars are removed for clarity. See Table 1 for abbreviations.

For all treatments, emissions of CO_2, CH_4 and N_2O were found to be the highest at the thermophilic stages, while NH_3 emissions were found to be variable during composting. During 28 d of composting, however, variation in CO_2, CH_4, NH_3 and N_2O emissions due to differences in the mixing ratios across all treatments was observed. This may be due to the effect of different C-rich bulking agents and their degradability during composting. Compared to N_2O, low emissions of CH_4 across the treatments were recorded. However, CH_4 emissions could be a result of the formation of anaerobic sites within the compost matrix, while N_2O emissions may be a due to nitrification and denitrification process during composting.

Using the global warming potential for N_2O and CH_4 being 310 and 21 times greater than CO_2, the total estimate of N_2O was found higher than CH_4 emission expressed as CO_2 equivalent, across the treatments.

4 CONCLUSIONS

The emissions of CO_2, CH_4, NH_3, and N_2O during composting of anaerobically digested solids mixed with different bulking agents and their mixing ratios were recorded. NH_3 emissions across the treatments were found variable. A relatively higher emission of N_2O compared to CH_4 was observed.

To gain better understanding and quantitative data of how composting solid fractions of animal slurry influence CO_2, CH_4, NH_3, and N_2O emissions, more research is needed in future.

(162) ON-SITE ASSESSMENT OF METHODS TO MEASURE GASEOUS EMISSIONS FROM BIOLOGICAL TREATMENT OF WASTE

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EXECUTIVE SUMMARY

Landfilling of biodegradable waste must decrease to fulfil the Council Directive 99/31/EC on landfills, in order to reduce the emission of gaseous and liquid pollutants during the landfill lifetime. Therefore, pre-treatment of the organic fraction of municipal waste prior to landfilling is being developed in several countries. In France, the organic fraction is either separated and treated through selective collection of biowaste, or through mechanical sorting in the plant followed by biological treatments (anaerobic or aerobic), the refuses only being landfilled. Or the mixed waste is stabilized by an aerobic process before landfilling. These different processes emit gases which may be harmful for health or the environment (toxic, explosive, odorants, greenhouse gases...). Some of the emissions can be collected and treated through biofilters, while other gases are emitted by surfaces (typically, compost windrows) and cannot be collected unless they are enclosed. Also, the efficiency of the biofilters must be assessed. IRSTEA and INERIS have been working together for several years on the use, comparison and improvement of surface emission measurement methods, applied to biological treatment plants of solid waste.

Gaseous emissions were studied on: composting process of pre-sorted organic matter from mixed waste, with a small or larger mesh and porosity, in either turned or aerated windrows, on biofilters, and on landfills which are located beside the composting plants. Depending on the ventilation air flux, different measurement methods were used: static (accumulation), dynamic or chimney type chambers, and a total cover of a biofilter with a plastic tarb. Several of these measurements were undertaken in order to evaluate the global gaseous emissions from those sites, to provide data to an environmental technology validation exercise (ETV).

Measurement campaigns presented here comprise: comparison of fluxes measurement techniques, calculation of gas fluxes (CO_2, CH_4, NH_3 and N_2O) emitted from composting windrows and biofilters, calculation of biogas emission (methane + CO2) before and after a final cover was set on a landfill.

Comparisons of the two first chambers have been made since 2007 on several sites (composting of the organic fraction of municipal solid waste or stabilization prior to landfilling). On the first site (non aerated windrows and small mesh) the difference between the measured fluxes was a factor of 2. This factor is rather small: differences between flux measurements using different devices can lead to differences as large as a factor of 100. More recent tests, presented here, show a better agreement: the difference between the two techniques lies within the measurement uncertainty.

Comparison of surface air speed measured by two different chimney chambers lead to comparable results. During one experiment, the global air flow interpolated from chamber data was underestimated compared to input flow measurement, because of preferred pathways of the air flow along the wall of the biofilter. When the border effect is correctly taken into account, the total gas flow measured with the chimney chamber and the one measured by a total cover of the biofilter show a good agreement.

Biogas surface emissions were measured with the static chamber, on a landfill which receives biologically stabilized waste. This landfill was partly uncovered, so only a part of the biogas was collected and flared. After the final cover was installed, the total biogas flow which was collected and flared was comparable to the sum of (the surface emissions + the collected biogas) without the total cover.

The results presented here show that on different sites, different emission measurement methods were used, and that generally there is a good agreement between the methods, providing the care of use are respected.

Advantages and care of use for the different methods, depending on the aeration conditions, have been established and some recommendations are given.

1. INTRODUCTION

Landfilling of biodegradable waste must decrease to fulfil the Council Directive 99/31/EC on landfills, in order to reduce the emission of gaseous and liquid pollutants during the landfill lifetime. Therefore, pre-treatment (sorting or stabilization) of the organic fraction of municipal solid waste prior to landfilling is being developed in several countries, named mechanical-biological treatment (MBT). In several locations in France, the organic fraction is separated and treated either through selective collection of biowaste, or through mechanical sorting in the plant followed by biological treatments (anaerobic or aerobic), in order to produce compost. In the latter case only the sorting refuses are landfilled. In some plants, mixed waste containing organic matter is stabilized by an aerobic process before landfilling. These different processes emit gases which may be harmful for health or the environment (toxic, explosive, odorants, greenhouse gases...). Some of the emissions may be collected and treated through biofilters, while other gases are emitted by surfaces (typically, compost windrows) and cannot be collected unless they are enclosed. Also, the efficiency of the biofilters must be assessed.

1.1 Background

The environmental assessment of the biological treatment of biodegradable waste takes into account all the emissions of the treatment plants and the outputs when they are valorised or landfilled. Among them, surface gaseous emissions may be important and need to be evaluated. Standard methods exist for the precise determination of exhaust or flare emissions, but surface emissions are more difficult to measure quantitatively. IRSTEA and INERIS have been working together for several years on the use, comparison and improvement of surface emission measurement methods, applied to biological treatment plants of solid waste

1.2 Research objectives

The research project, conducted in the partnership aimed to compare different surface emission measurement chambers that were available, and, depending on the process or area to be monitored, to give recommendation for the selection and use of the appropriated chamber(s). Gaseous emissions were established on different MBT plants and associated landfills in order to provide data for an environmental technology validation exercise.

2. METHODOLOGY

Gaseous emissions were studied on different plants, comprising: composting of pre-sorted organic matter from mixed waste, with a small or larger mesh and porosity, in either turned or aerated windrows, on biofilters, and on emissions from the treatment buildings or devices such as rotating drum bioreactors. Surface emissions of landfills which receive either stabilised waste or sorting refuses, associated to the biological treatment plants, were also investigated.

2.1 Measurement of surface flux gases

Depending on the ventilation air flow, different measurement methods were used:

- a static flux chamber developed by INERIS allows short time measurements on rather small or medium fluxes (typically less than 5 L/m^2/min). In this chamber, gas emitted by the soil or compost surface accumulates This method, applied to a great number of measurement points, gives a set of local fluxes which can be interpolated by kriging, in order to cartography a zone and calculate the total flux over the area. It has been used for the emission measurement on landfills surface, and on non-aerated or aerated windrows with low aeration speeds. 2 analyzers measure methane and CO_2 simultaneously;
- a dynamic flux chamber developed by IRSTEA takes longer time for one measurement, but allows measurements on higher fluxes and can be used for monitoring the fluxes variations over a period of time. In this chamber, there is an admission and an extraction of the outside air, with a control of the pressure equilibrium between the inlet, outlet and outside of the chamber. The measurement can be done without effect of overpressure (under-estimation of gases emissions) or depression (over-estimation of gases emissions) in the chamber. It was designed to capture convective emissions and has been also used on non-aerated and aerated windrows. Gaseous concentrations are measured by an INNOVA analyzer, measuring on the same time CO_2, CH_4, NH_3, and N_2O (possibly SF_6) in the ppm range;
- for larger emitted air flows (typically, on biofilters) a chimney chamber is more adapted. The chamber outlet is constituted of a reduced tubing, increasing the gas speed, which is measured more accurately. Both INERIS and IRSTEA have such a chamber, designed in different geometries and materials. Different types of analyzers can be connected to these devices (the first measurement is the air speed).

2.2 Measurement campaigns

Measurement campaigns were undertaken on several waste treatment plants. They are shortly described thereafter.

2.2.1 Site A

This site is a composting plant which processes municipal solid waste. Raw waste is pre-treated in a rotating bioreactor (drum) for 4 days. During this time, waste bags are opened, and paper and cardboards are somewhat decomposed in small pieces. After this pre-treatment, waste is carefully sorted through different devices in order to obtain a clean and fine organic fraction. Before 2010, this fraction was mixed with a bulking agent (shredded green waste) and disposed in windrows which were turned twice a week with an automatic windrow turner (A1). Since 2010, the mixture is disposed in successively 2 aerated cells, with a discontinuous aeration, for 3-4 weeks each. After that, the compost is stored outside without any turning until it is taken by local farmers to be used on fields (A2). Polluted air from the composting boxes and the rotating drum is treated by biofilters (A3 and A4 respectively). The refuses of sorting are stored in a landfill which sits on the same site (A5).

2.2.2 Site B

Site B is an anaerobic digestion (AD) plant which also processes raw MSW. The pre-treatment and sorting of the organic matter is quite similar to the one of site A, but after the sorting the organic matter is hydrolyzed (dry conditions, around 30 % dry matter) and sent to large digesters (volume approximately 1500 m^3) for 1 month. Feeding of the reactor is semi –continuous and temperature is kept at 37 °C (mesophilic conditions). The digestate is then dried by pressing, mixed with crushed green waste, and composted in aerated boxes for 14 days: air from this process is treated through an acid washing tower followed by a biofilter (B1). The compost is then stored for maturation without turning (B2, before 2012), until it is used by local farmers, between 1 and 3 months after. Since our measurements, the site manager has decided to turn the maturating compost every week or so, in order to limit the methane emissions to the atmosphere. Efficiency of this new procedure will be evaluated. Refuses of sorting are sent to an outside landfill or incinerated: no emission measurement was possible on this fraction.

2.2.3 Site C

Site C processes MSW, but do not prepare a compost for agricultural purpose. The waste is biologically stabilized before landfilling. The larger fraction of waste (> 50 and 70 mm typically) is not stabilized. A part of the plastics (fraction between 70 and 450 mm) is sorted and recycled. The larger fraction, and the refuses of the intermediary fraction, are landfilled without treatment. The smaller fraction, which is rich in organic matter, is treated in aerated boxes for 2.5 months (C1). Aeration is discontinuous, with a 3 minutes-3 minutes cycle. Treated matter is moved from one box to another one, every 2 weeks. Stabilized matter is used as a temporary cover, every week or so, on the landfill cells (open cell: C2, closed cells: C3)

2.4 Surface emission measurement on non aerated windrows

2.4.1 Emissions from composting and maturation windrows, composting site

The first measurements were performed in 2007 on site A. Methane and carbon dioxide were measured with both a static chamber and a dynamic chamber on several windrows of different ages (A1, A2) and on the landfill cells (A5): "EMISITE", 2007, and Zdanevitch *et al*, 2009) Individual measurements taken with the static chamber were used for emission cartography by kriging, using Surfer 8. Figures 4 and 5 show methane and CO_2 emissions on the same windrow. Table 1 reports the total methane and CO_2 fluxes for windrows of different ages.

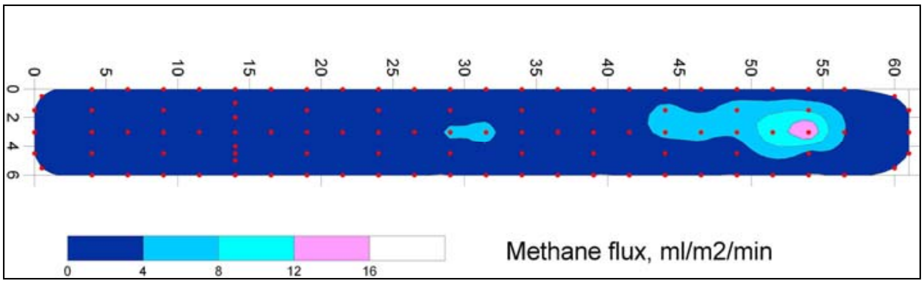

FIGURE 1 **Methane flux determined with a static chamber, non aerated windrow (windrow size in meters)**

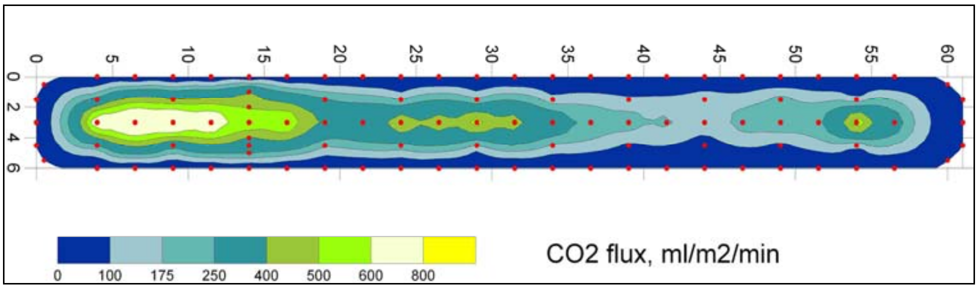

FIGURE 2 **CO_2 flux determined with a static chamber, non aerated windrow (windrow size in meters)**

TABLE 1 **Methane and CO2 emissions from non aerated windrows, depending on the age of the compost**

Windrow	Age	Surface, m^2	CH_4 flux, m^3/h	CO_2 flux, m^3/h
7	15 days	366	0.04	4.5
6	1 month	294	0.34	4.6
Mat	3 months	432	0.82	4.3

Discussion: CO_2 emissions from composting at different ages were quite similar. The maturation windrow was larger but also higher than the composting one (6 and 7), so CO_2 emission, related to the quantity of matter, is a little lower, but in the same order of magnitude. This suggests that the first stage of composting is not sufficiently efficient. Methane emissions are more scattered: they are very low on young compost (less than 2 weeks). They increase with the age of the compost, and also on the maturation windrow, which is not turned.

Comparison of results between fluxes measured by the static chamber and the dynamic chamber led to differences between 1 and 5 (mean value around 2: "Emisite", 2007). These differences could not be completely understood, so comparison was repeated during other campaigns (see chapter 4).

2.4.2 Emissions from maturation windrows

As we noticed in 2007, methane emissions can be large on undisturbed windrows. Site A, after a modification of the composting conditions, and site B, store compost in large windrows, without turning, for 1 to 3 months before its use on agriculture soils. We measured methane and CO_2 emissions on maturation windrows on these two sites (A2, B2) with the same method, the static flux chamber. Results are presented in table 2.

TABLE 2 **Methane and CO_2 emissions measured on maturation windrows**

		Surface, m^2	CH_4 flux, m^3/h	CO_2 flux, m^3/h	Relative CH_4, $m^3/h/m^2$	Relative CO_2, $m^3/h/m^2$
Site A (MBT, composting alone)	**Windrow 1**	272	7.2	26	0.03	0.10
	Windrow 2	380	9.4	40.2	0.02	0.11
Site B (MBT, AD and composting	**2 windrows (Oct. 2010)**	500	13.7	22.4	0.03	0.04
	1 windrow (May 2011)	300	6.1	11.5	0.02	0.04

Discussion: methane emissions are rather important, showing that anaerobic conditions prevail inside the composting windrows, and that the compost is not stabilized despite its age. Emissions are very similar between the two sites, e.g. there is no influence of the anaerobic digestion prior to composting on the methane emission potential of the compost. Nevertheless, CO_2 emissions are similar between windrows of the same site, but 2 to 3 times lower on site B (with AD) than on site A (composting alone). This shows that 1 month of AD followed by two weeks of aerated composting probably biodegrades more the organic matter than the 2 months of aerated composting. In order to compare the results, emissions have been reported to the surface. They should be reported to the mass of compost, but this information is not yet available.

2.5 Surface emission measurement on aerated windrows

Parallel measurements using static and the dynamic chambers, were undertaken on the composting boxes of site C (C1). The measures where done on three boxes, which contained the fine waste fraction of different ages. The aeration cycle has been lengthened from the standard one: 3/3 minutes with/without aeration, to 15/15 minutes, to allow the measurement with the dynamic chamber and INNOVA analyzer. Measurements with the static chamber being faster, several measurements can be made successively during the aeration or non-aeration stage. Figures 6 and 7 report measurements made with the 2 chambers on the same windrow, the same day, during non aerated and aerated cycles.

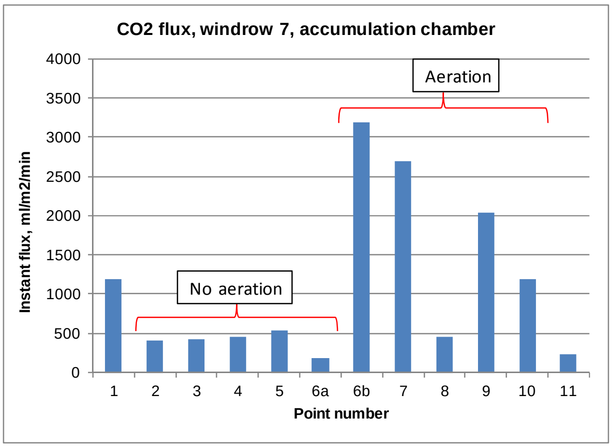

FIGURE 3 **CO_2 flux measured with the static chamber**

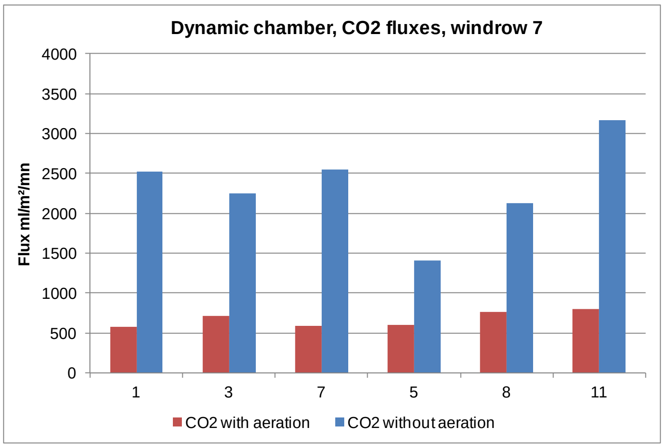

FIGURE 4 **CO_2 flux measured with the dynamic chamber**

TABLE 3 **Comparison of CO_2 surface flux on windrow 7, established with the static and the dynamic flux chambers**

Mean CO_2 flux, $m^3/m^2/day$		
Static chamber	Dynamic chamber	Difference, %
1.97	2.26	13

Despite different measuring times (2 minutes for the static chamber, 10-20 minutes for the dynamic chamber), it is interesting to notice that the results of the two chambers are similar. The static flux chamber is able to differentiate emissions with and without aeration, although this chamber was not initially designed for aerated surface sources The difference between the mean flux (sum of the aerated and non-aerated periods) is as low as 13 %. It should be noticed that the overall uncertainty of the static flux chamber measurement technique (assuming that measurements are made over several days periods) has been evaluated around 20 %. Knowing the total surface of the windrows (corresponding to a given mass of compost) allowed us to calculate the total CO_2 emissions for the whole composting plant, and to compare it with the emissions from the landfilled stabilized waste. These results will be used for an environmental technology validation exercise (ETV) and will be published shortly.

2.6 Surface emission measurements on biofilters

2.6.1 Biofilter treating the air from the composting boxes on site A (A3)

Gaseous fluxes cartographies (kriging interpolations) were established on this biofilter using a chimney chamber. During a previous experiment, the global air flow interpolated from chamber data was underestimated comparing to input flow measurement, because of preferred pathways of the air flow along the wall of the biofilter. On this site, care has been taken on this point. Two different analysers were connected to the chamber: an ECOPROBE 5 (infrared), measuring CO_2 (and hydrocarbons but at rather high concentrations, useless here), and an INNOVA (photacoustic) which measures simultaneously 4 gases (CO_2, CH_4, NH_3, N_2O) in the ppm range. Along with the cartography of the biofilter, the results gave a comparison of CO_2 fluxes measured on the same device with the two analyzers: see table 4.

TABLE 4 **Emission measurements (CO_2, methane, NH_3, N_2O) on the composting boxes biofilter on site A**

Biofilter (Air from compost. boxes, S = 216 m^2)	CO_2 (ECOPROBE)	CO_2 (INNOVA)	CH_4 (INNOVA)	NH_3 (INNOVA)	N_2O (INNOVA)
Flux, m^3/h	9,1	10,6	0,59	0,009	0,003

The difference between CO_2 fluxes measured by the two analyzers is 16 %. It is similar to the difference that was measured on the aerated windrows with two types of chambers. It means that the difference between flux measurements is probably due to analyzers uncertainties, as well as the difference between techniques. CO_2 flux is 16 times larger than CH_4 flux, a thousand times larger than NH_3 flux, and 3 thousand times larger than N_2O flux. This allows the calculation of warming effect due to greenhouses gases emissions from this biofilter (NH_3 is not a GHG but has also environmental impacts). Once again, these results will soon be used in an ETV exercise.

2.6.2 Biofilter treating the air from the rotating drum on site A (A4)

On the same site, surface CO_2 emissions were measured on a biofilter treating the air from a rotating drum. Different methods were tested to follow the gas emission evolution. First, a cartography of the biofilter CO_2 emissions was established using the chimney chamber (see figure 8). The other method consisted in covering the whole biofilter surface with a plastic tarp, in aim to measure all the gas emissions.

With these two methods, it was possible both to cartography the biofilter emissions, and to follow the total emissions during time and characterize the treatment efficiency of the biofilter.

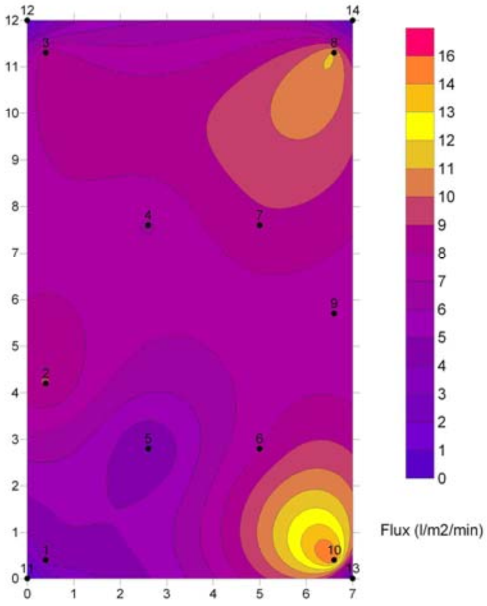

FIGURE 5 **CO_2 fluxes determined with a chimney chamber**

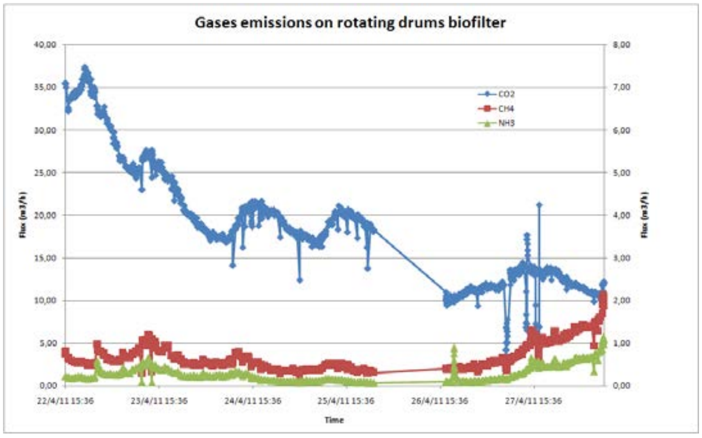

FIGURE 6 **Gases emissions monitoring on the rotating drums biofilter**

Results on figure 6 show that we obtain similar values for the first 2 two days (April 21-22) but the following days, the plastic sheeting method shows a trend of decrease. The decrease can be explained for the three following days by the week-end (from the 23 to 26th of April), and the fact that there is no new matter entering in the rotating drum. It is more complicated to explain why this decrease continued on the following days, when the site activities came back.

TABLE 5 **Emissions measurements (CO_2, CH_4, NH_3, N_2O) on the rotating drums biofilter on site A**

Biofilter (Air rotating drums, S = 84 m^2)	CO_2 (SURFER) 2011/04/21	CO_2 (INNOVA) 2011/04/22	CH_4 (INNOVA) 2011/04/22	NH_3 (INNOVA) 2011/04/22	N_2O (INNOVA) 2011/04/22
Flux, m^3/h	39	34,82	0,58	0,19	0,01

Comparing to the gaseous emissions of the two biofilters of site A shows that the rotating drum's biofilter presents higher gases emissions. It can be explained for a part by the fact that, at this time, the composting boxes where open, due to a functioning problem. So, the composting biofilter treated only a part of the gaseous emissions. New measurement should be done on composting biofilter with closed boxes.

2.7 Surface emission measurement on landfill cells vs flared biogas

On site C, "stabilized" waste is landfilled. In fact, even after 2.5 months of active composting, the organic fraction of MSW is not completely stabilized, and when it is landfilled under anaerobic conditions, biogas builts up. This biogas contains approximately 55-60 % v/v methane, 35-40 % v/v CO_2, and trace gases like H_2S up to 800 ppm. Initially on this site, biogas was not burned as the total flux was supposed to be low. But neighbours reported odour problems due to H_2S, therefore a flare has been installed. When we measured the surface gaseous emissions of the landfill (July 2011), the flare had been used for 4 months and biogas was pumped only on closed cells (C3). The static chamber was used to measure biogas (methane + CO_2) emissions on the surface of the landfill (C2 and C3). Figure 8 represents the methane emissions. Different areas of the landfill have been identified:

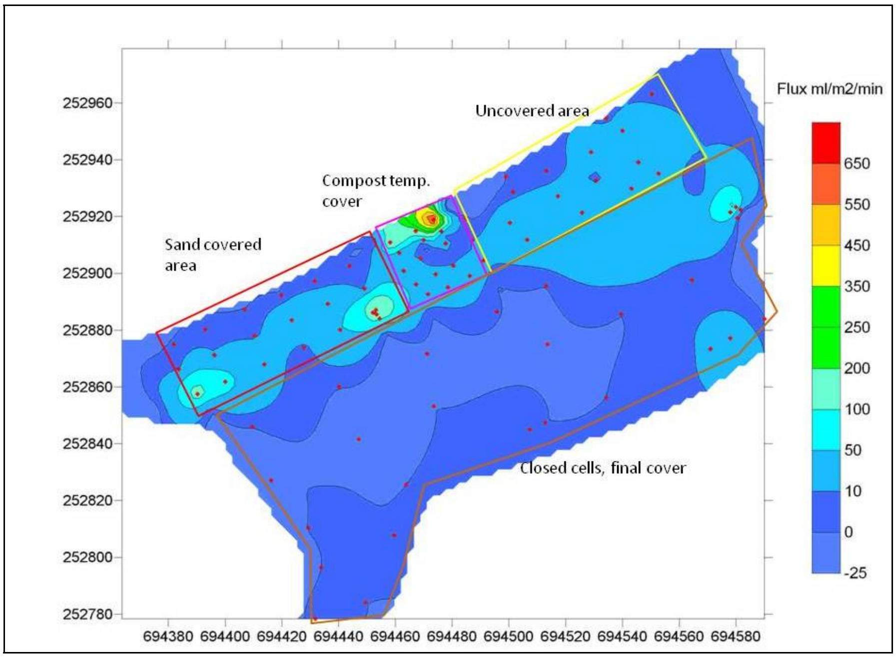

FIGURE 7 **Methane surface emissions on site C (landfill cells)**

TABLE 5 **Calculation of total biogas flux from the whole landfill, site C (July 2011)**

	Mean flux, m^3/h	
Methane, surface, static ch.	17 (a)	Total biogas flux (surface + flared) : **85**
CO_2, surface, static ch.	43 (a)	
Flare, biogas from 6 wells (data from the plant manager)	25	

(a) Though biogas is richer in methane than in CO_2, CO_2 surface fluxes are higher than methane ones. It implies that a fraction of methane is oxidised within the surface layers (constituted of mixed sorting refuses and compost) before it is emitted to the atmosphere.

4 months after our measurement campaign, the exploited zone of July had its final cover and biogas was pumped through 14 biogas wells distributed all over the landfill area. The mean biogas flux burned by the flare was then 70-80 m^3/h. This represents a difference with the estimated total flux of July, of about 6 to 17 %. Once again, this difference lies within the normal uncertainty of the static chamber measurements.

3. RECOMMANDATIONS

The choice of the measurement technique (type of chamber, gas analyzer) depends on the application. First of all, it is important to know what are the aim and the uses of the measures.

If the aim is to cartography an area, choices are static chamber for a compost windrow or chimney chamber for a biofilter. There must be a careful consideration on the measurement strategy to place the points of measurements (on a windrow, at least one row on the top and one, or better, two rows on the edges). The number of points depends on the dimensions of the area and on the variation of the fluxes between points. Even if the area is small, a minimum of points is needed in order to get a good kriging. Typically, a minimum of 20 to 30 points give good results when fluxes are rather stable over the area. Border effects must also be taken into account, especially on biofilters, depending on the type of filtering material. If it is rather coarse, preferred pathways can exist along the wall of the filter. The chamber must be placed in contact to the wall in order to take this phenomenon into account.

Measurements with a static chamber are fast and allow a large number of measures in a short time; but if there are quick changes in the flux, this technique can lead to wrong results. On the other hand, it is possible to repeat measurements and to calculate the mean value.

Measurements with a chimney type chambers take longer. These chambers are very useful for flux measurements on biofilters, but there is little information on this technique in the literature. The following points are important: first, to measure the gas concentration correctly, the volume of the chamber must have been replaced at least 6 times; this depends on the volume of the chamber and on the gas flux. The air speed may have changed between the start and the end of the measurement: it must be controlled, and if the difference is too high, the measurement should not be kept.

To measure fluxes along time, the dynamic chamber is well suited. It allows long time measurements, which is useful to integrate changes in emissions along the changes in process (as example aerated windrow). The measure depends on the pressure inside the chamber which must be controlled and measured.

On a biofilter, covering the whole surface with a plastic tarb is a good alternative to follow emissions of a process along time, but the installation is tricky and takes a long time.

4. CONCLUSIONS

They are several available techniques for gas fluxes measurements and the choice will depend on the application. The strategy of measurement is important and must be thought before the on-site campaign. All the campaigns run together by INERIS and IRSTEA allowed us to raise some recommendation about gas measurements.

When there is high variability in the gaseous emissions in a process, measures will be more representative if the emissions can be followed along time. Depending on the process, short measurement times can give random results which can introduce a misunderstanding of the process gases emissions, or mistakes in the calculation of gaseous emissions.

The static chamber is a good method to cartography large surfaces (like landfills) because of its quickness. It can also be used on compost windrows. It allows the measurement on a large number of points over a short period of time, which allows precise determination of highly emissive points (“hot spots”).

The dynamic chamber is better used for a continuous monitoring over a long period of time. Chimney chamber is useful on processes with high aeration speeds as biofilters.

Comparison of methods, between static chamber and dynamic chamber, gave recently good results on some sites, but more tests should be necessary to get a better comprehension of the differences observed in the past.

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(188) INTEGRATED WASTE MANAGEMENT COMBINING ANAEROBIC AND AEROBIC TREATMENT: A CASE STUDY

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EXECUTIVE SUMMARY

The generation of wastes has undergone a notable increase due to some factors, such as the concentration of the population in urban areas, the intensification and concentration of livestock production, and the rapid growth and development of the agroindustrial sector. The intensification of the livestock systems has resulted in a high density of animals in relatively small geographical areas, where large quantities of manure are produced. Intensive livestock production is associated to several detrimental environmental side effects, such as uncontrolled discharges to surface waters, leachate to groundwater (e.g., nitrogen, phosphorus), contamination of soils (e.g. heavy metals) and gaseous and odour emissions to the atmosphere. Therefore, there is a need for more environmentally sound methods for the treatment and utilisation of animal manure. Anaerobic digestion is a viable alternative to the energetic valorisation of these wastes, because this process not only produces a renewable energy, but also reduces the greenhouse emissions, and improves the fertilising value of the treated materials. This treatment transforms organic wastes into two products: a source of renewable energy (biogas) and a potential fertiliser, the digested material (digestate), which could be used as soil fertiliser. However, some characteristics of the digestate (phytotoxicity, odour, etc.) can limit its direct application to agricultural soils in certain circumstances. Hence, a post-treatment to anaerobic digestate is advisable to enhance digestate properties prior to its agricultural use. This post-treatment can consist on a solid-liquid separation of the digestate and the later composting of the obtained solid fraction. Composting, a treatment based on the aerobic biological decomposition and stabilisation of organic substrates, can constitute a suitable method to improve the properties of the solid fraction of digestate and thus, enhancing its fertilising value, since reduces the moisture content, odour emission, the potential phytotoxicity and also favours the elimination of pathogens.

The aim of this work was to study the feasibility of composting, as a post-treatment option, to manage the solid fraction of anaerobically digested pig slurry (SFP) produced in an industrial waste treatment plant, together with other wastes processed in the facility (sewage sludge (SS), paper mill sludge (PS) and pig hair waste (PW), and two bulking agents (maize straw (MS) and wheat straw (WS)). Six composting mixtures were prepared: P1 (100% SFP), P2 (80% SFP + 20% MS), P3 (60% SFP + 40% MS), P4 (35% SFP + 35% SS + 30% mixture of MS and WS), P5 (35% SFP + 35% PS + 30% mixture of MS and WS) and P6 (35% SFP + 35% PW + 30% mixture of MS and WS).

Throughout the composting process, temperature evolution was monitored and physico-chemical and chemical parameters were determined. Furthermore, evolution of the microbial community structure was assessed. The relative richness of 16SE rRNA gene (eubacteria), mcrA gene (archaea) and ITSqrRNA gene (fungi) were analyzed by means of the real time PCR technique (qPCR).

The use of the bulking agents influenced positively the development of the composting process, especially in the mixtures P2 and P5. In general, all the composts showed a good degree of maturity and absence of phytotoxicity, but the use and proportions of determined raw materials (PS, SS and SFP) produced composts with several limiting aspects (high salinity and/or notable contents in Cu and Zn) for their agricultural use. The increase of the ratio fungi/eubacteria by the end of the process was consistent with the maturity of the produced compost and the good performance of the process. Pile P1, where no bulking agent was added shows the lowest ratio (<4%) and pile P5, one’s of better process performance, the highest (> 30%). Despite archaea were detected in all the samples, the ratio archaea/eubacteria was maintained below 0.4%, as indicator of no anaerobic proliferation or good aerobic condition of the piles.

1 INTRODUCTION

1.1 Background

The constant increase in the generation of organic wastes, especially from the livestock sector, and the concern for environmental issues associated to their management, such as soil pollution and/or gaseous emissions, has led to an increasing demand for eco-friendly technologies that require waste management options including energy recovery. In this scenario, anaerobic digestion constitutes as a technology that allows the production of renewable energy (biogas) being one of the most efficient biological methods to reduce GHG emissions (Holm-Nielsen et al., 2009; Poeschl et al., 2012). The effluent from the anaerobic process, the digested substrate (digestate), presents better agronomic properties than the raw animal slurry, such as higher proportion of mineral N and less decomposable organic matter (Sørensen and Møller, 2009). However, some undesirable characteristics of the digestate, such as; odour, viscosity, high moisture content, presence of volatile fatty acids, which can be phytotoxic and the possible presence of seeds of weeds and pathogens (Walker et al., 2009), may restrict its application to agricultural soils without an adequate post-treatment (Abdullahi et al., 2008 and 2009). One possible option is a phase separation in a liquid and a solid fraction, and the latter being composted in order to obtain valuable and marketable end-products for agriculture (Holm-Nielsen et al., 2009).

The composting of the solid fraction of the digestate can constitute a feasible method to manage these materials, as it improves the quality of the end-product. Odour abatement, reduction of moisture content, removal of phytotoxic substances, such as ammonia and VFA (Smet et al., 1999), as well as of weeds and pathogens (Tchobanoglous et al., 2002), are the main benefits of the composting process.

1.2 Research objectives

The main objectives of this work were: i) to study the feasibility of composting process, as a post-treatment, to manage the solid fraction of anaerobically digested pig slurry (SFP) produced in a centralized treatment plant, together with other industrial organic wastes, and two different bulking agents; and ii) to evaluate the effect of the type and proportion of the co-composting agents used in the mixtures, on the final chemical, agronomic and microbiological characteristics of the obtained composts.

2 METHODOLOGY

2.1 Experimental design

Six different composting mixtures were prepared using the solid fraction of digested pig slurry (SFP), and three (3) different wastes available in the local industrial facilities, such as sewage sludge (SS), paper mill sludge (PS) and pig hair waste (PW). Two different bulking agents were used (maize straw -MS-, and wheat straw -WS-) to ensure aerobic conditions during composting process. The main characteristics of these initial materials are shown in Table 1.

TABLE 1 **Characteristics of the raw materials used in the mixtures.**

	Solid fraction of digested pig slurry	Sewage sludge	Paper mill sludge	Pig hair waste	Maize straw	Wheat straw
pH	6.83	5.94	7.81	5.33	6.31	7.47
EC (dS/m)	3.76	2.92	0.72	1.90	4.81	3.98
OM (%)	69.9	78.2	37.2	99.4	91.7	93.1
TOC (%)	38.0	47.0	24.7	53.3	46.2	47.1
TN (%)	2.62	3.50	0.61	14.6	0.74	0.78
C/N ratio	14.5	13.4	40.5	3.65	62.4	60.4

EC: electrical conductivity; OM: organic matter; TOC: total organic carbon; TN: total nitrogen.

Six different mixtures (on fresh weight basis) were used to form six different composting heaps

- P1: 100% SFP
- P2: 80% SFP + 20% MS

- P3: 60% SFP + 40% MS
- P4: 35% SFP + 35% SS + 30% mixture of MS and WS
- P5: 35% SFP + 35% PS + 30% mixture of MS and WS
- P6: 35% SFP + 35% PW + 30% mixture of MS and WS.

The proportions used in the different composting mixtures were mainly established for recycling the maximum proportion of SFP, taking into account the porosity, the C/N ratio of the mixtures, as well as the residues available in the composting plant. These mixtures were prepared in trapezoidal piles (1.5 m high with a 2 x 2 m base) and composted using the windrow composting system in a concrete platform located in an composting plant (Gestcompost S.L., Pina de Ebro, Zaragoza, Spain). The piles were turned five times, when the temperature started to decrease in order to improve both the homogeneity of the materials and the composting process. The moisture of the piles was controlled weekly by adding the necessary amount of water to keep the moisture at > 40 %, and the evolution of the temperature was monitored. The bio-oxidative phase of composting was considered finished when the temperature was close to the ambient and re-heating did not occur after turning. Then, the composts were left to mature over a period of one month. The samples were taken by mixing seven subsamples from seven representative sites of the pile, from the whole profile (from the top to bottom of the pile). Each sample was divided into two parts, one of which was air-dried and ground to 0.5 mm for chemical analysis and the other was immediately frozen and kept for later determinations.

2.2 Analytical methods

Raw materials and compost evolution were analysed according to standard chemical parameters. Electrical conductivity (EC) and pH were analysed in a 1:10 (w/v) water-soluble extract. Organic matter (OM) was assessed by determining the loss-on ignition at 500 °C for 24 h. Total organic C (TOC) and total N (TN) were determined by automatic microanalysis (EuroVector elemental analyzer); water-soluble organic carbon (Cw) and water-soluble nitrogen (Nw) were determined in a 1:20 (w/v) water extracts by using an automatic analyser (TOC-V CSN Analyzer, Shimadzu). The germination index (GI) was calculated using seeds of *Lepidium sativum* L. as described in Zucconi et al. (1981).

In the mature composts, after $HNO_3/HClO_4$ digestion, P was analysed colorimetrically as molybdovanadate phosphoric acid. Na was determined by flame photometry and Ca, Mg, Fe, Cu, Mn, Zn and heavy metals (Cd, Co, Cr, Pb and Ni) by inductively coupled plasma-optical emission spectrometry (ICP-OES). Mature compost was also evaluated by batch cultivation. All the analyses were made in triplicate.

The ratio of eubacterias, archeaea and fungi were assessed by means of real-time PCR (qPCR) following the methodology described in Prenafeta-Boldu et al. (2012). Total DNA was extracted from lyophilized compost samples with PowerSoil DNA Isolation kit (MoBio Laboratories, Inc., Carlbad, CA), according to manufacturer instructions. Three primers sets (forward-reverse) were used to selectively amplify total bacterial, archaeal and fungal rDNA fragment (F341GC-R907, ME1-ME3R and ITS5-ITS2, respectively) with a Mastercycler (Eppendorf, Hamburg, Germany) as described in Prenafeta-Boldu et al. (2012). Gene copy numbers of eubacterial 16S rRNA, archaeal mcrA rRNA and fungal ITS1 rRNA fragments were quantified with the quantitative real-time PCR (qPCR). The analysis was carried out using Brilliant II SYBR Green qPCR Master Mix (Stratagene, La Jolla, CA) in a Real Time PCR System MX3000-P (Stratagene). The standard curves were performed with reference genes and quantified by Quant-iTTM PicoGreen® dsDNA Reagent using MX3000P (Stratagene) as a detector system. The qPCR efficiencies of amplification were greater than 95%. All the results were processed by means of MxProTM QPCR Software.

3 RESULTS AND DISCUSSION

3.1 Temperature evolution

The evolution of the temperature showed the typical composting temperature trend, all the composting piles reaching thermophilic temperature values (> 40ºC) during the first two weeks of composting, except for P1, elaborated only using the solid fraction of digested pig slurry (SFP) (Fig. 1). On the contrary, P5, constituted by SFP, paper mill sludge (PS) and the mixture of maize straw (MS) and wheat straw (WS), reached the thermophilic temperature values faster and showed the highest temperature values than the rest of piles, possibly due to the addition of PS, since the temperature profiles reflected the labile nature of the C forms present in the wastes and its thermal inertia (Sundberg et al., 2004). Pile turning reactivated the composting process in all the mixtures, especially after the third whirl, which led to a new increase of the temperature values. After fourth whirl, the temperature decreased in all the composting piles to values close to the ambient values.

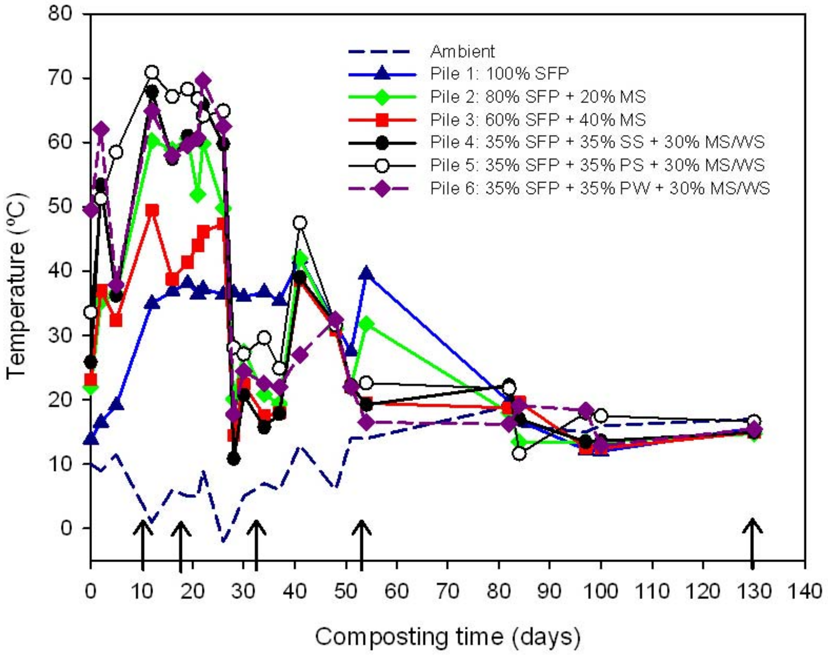

FIGURE 1 **Temperature evolution of the piles throughout the composting process. Arrows indicate turnings.**

3.2 Physico-chemical properties

At the beginning of the composting process, all the mixtures showed pH values between 6.5-7.5 (Fig. 2a), within the range suggested as suitable for composting (pH = 6-8) (Bustamante et al., 2008). In general, the pH values in the mixtures slightly decreased throughout the composting process, except for P1, P4 and P6, which showed a slight initial increase that may be attributed to the degradation of acid-type compounds, such as carboxylic and phenolic groups from the SFP, and to the mineralisation of proteins, amino acids and peptides to ammonia (Bustamante et al., 2008). Also, the decrease of pH observed in all the piles at the end of the composting process could be associated to the nitrification process (Bustamante et al., 2007).

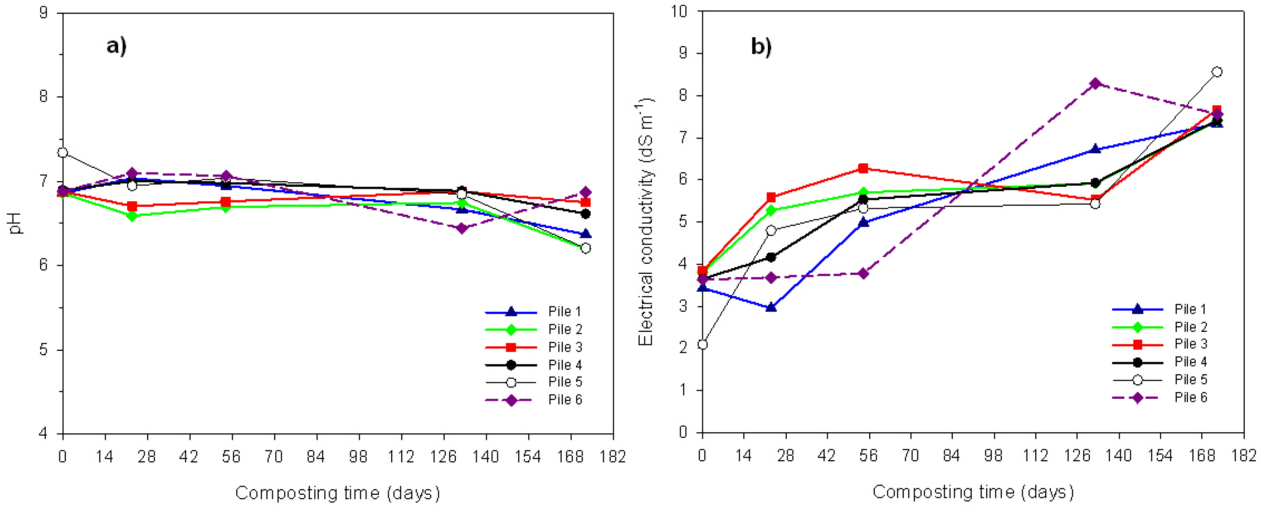

FIGURE 2 **Evolution of pH (a) and electrical conductivity (b) of piles 1 [100% SFP], 2 [80% SFP + 20% MS], 3 [60% SFP + 40% MS], 5 [35% SFP + 35% SS + 30% MS/WS], 6 [35% SFP + 35% PS + 30% MS/WS] and 7 [35% SFP + 35% PW + 30% MS/WS] during composting.**

The electrical conductivity tended to increase in all the piles (Fig. 2b), due to the production of inorganic compounds and their increased relative concentration due to the mass loss of piles (Bustamante et al., 2008). However, only P1 showed an initial decrease, probably due to the volatilisation of ammonia and the precipitation of mineral salts, as it was reported by Huang et al. (2004) in an experiment of composting of pig manure with sawdust.

3.3 Chemical and biological properties

Initially, all the piles showed organic matter concentrations between 60-70%, except for P5 elaborated with SFP, PS and the mixture of MS and WS, which showed the lowest concentrations (51.0%) (Fig. 3a). In general, the values of this parameter decreased during the process in all the piles, showing the organic matter degradation process, especially in the piles elaborated using other wastes as co-composting agents. At the end of the process, all the piles showed organic matter concentrations between 47.3% for P5 and 57.5% for P8.

In general, the C/N ratio tended to decrease in all the mixtures, especially in P5, elaborated with SFP, PS and the mixture of MS and WS. This phenomena was also observed in the piles that only used MS as bulking agent (P2 and P3). Nevertheless, at the end of the composting process all the piles or mixtures reached a similar value (<10; Fig. 3b). This decrease has been observed by other authors in other composting experiments (Bustamante et al., 2007, 2008). The final values of the C/N ratio suggest that all the composts had reached an acceptable degree of maturation, since they were <10 (Bernal et al., 2009).

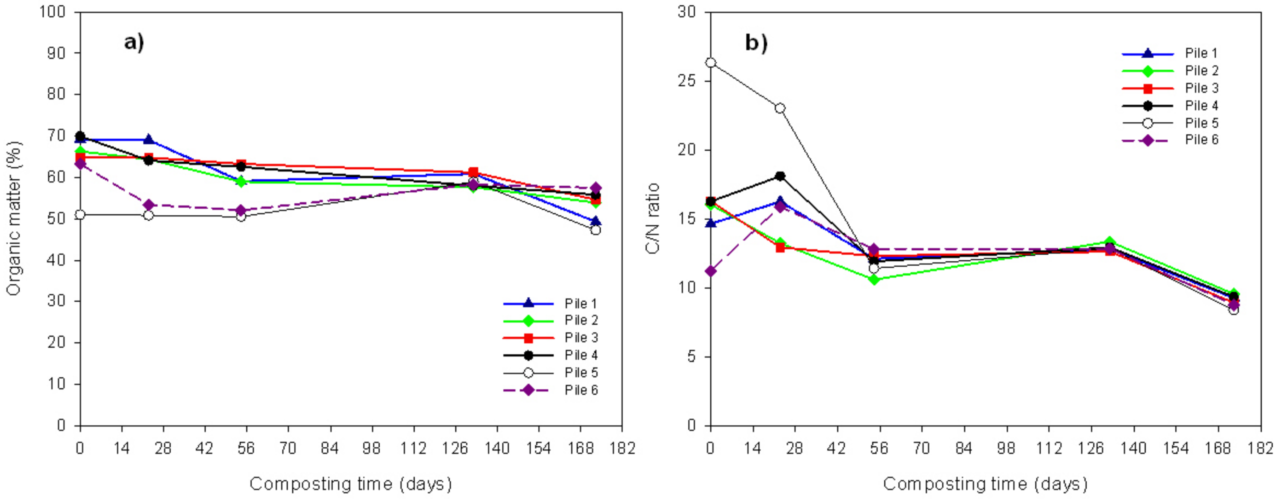

FIGURE 3 **Evolution of organic matter concentrations (a) and C/N ratio (b) in the piles 1 [100% SFP], 2 [80% SFP + 20% MS], 3 [60% SFP + 40% MS], 5 [35% SFP + 35% SS + 30% MS/WS], 6 [35% SFP + 35% PS + 30% MS/WS] and 7 [35% SFP + 35% PW + 30% MS/WS] during composting.**

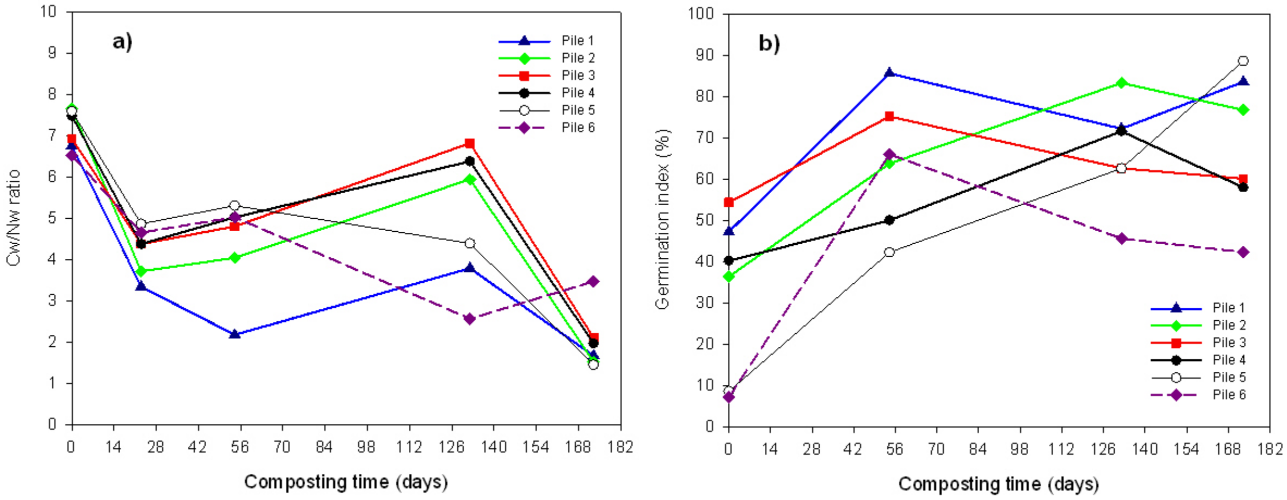

FIGURE 4 **Evolution of water-soluble C/water-soluble N (a) and of the germination index (GI) (b) in the piles 1 [100% SFP], 2 [80% SFP + 20% MS], 3 [60% SFP + 40% MS], 5 [35% SFP + 35% SS + 30% MS/WS], 6 [35% SFP + 35% PS + 30% MS/WS] and 7 [35% SFP + 35% PW + 30% MS/WS] during composting.**

In general, the water-soluble organic C to water-soluble nitrogen ratio (Cw/Nw) decreased throughout the composting process in all the mixtures, mainly during the bio-oxidative phase (Fig. 4a), as a consequence of the degradation of simple water-soluble organic compounds, such as sugars, amino acids and peptides.

The germination index (GI) values increased notably in all the composting piles throughout the composting process (Fig. 4b), from low values initially, especially in P5 and P6, to values higher than 50% at the end of the process (except for P6 that shows GI values lower that 50%). According to Zucconi et al. (1981), GI values higher than 50% indicates absence of phytotoxic compounds; therefore, the final composts obtained, except for P6, showed an acceptable level of germination.

3.4 Evolution of the microbial communities

The bacterial, archaeal and fungal biomass concentration in the original substrates were estimated from the number of ribosomal gene copies per gram of sample (data not shown). Bacterial was the most abundant domain ($9\cdot10^{9}$-2 10^{10}) in all the materials, while highest quantity of archaeal copies ($6\cdot10^{7}$) were detected in digested sludges samples (SFP and SS), indicating its anaerobic nature. Opposite, the highest quantity of fungal copies ($8\cdot10^{6}$) was detected in the paper mill sludge (PS), indicating its cellulosic-hemicellosic composition. The evolution of bacterial-archaeal-fungal biomass along composting time (samples taken at time 0, 24 and 56 days) were expressed as concentration ratios (archaeal/bacterial or fungal/bacterial), as plotted in Figure 5. Pile P1, P2 and P3, mostly composed by SFP, presented a stable archaeal ratio along sampling time, indicating the presence of anaerobic zones (Figure 5a), and presents the lowest ratios of fungal species (<5%, Figure 5b), indicating possible limitations during maduration process. Contrary, piles where SPF was reduced up to 35% of the mixture, with 30% of bulking material and several co-substrates as SS, PS and PW (Piles P4, P5 and P6) presents a clear improvement in fungal/bacterial ratios (up to 25%) at the end of thermophilic period, indicating the possible formation of humic and fulvic substances. This fact was especially remarkable in P5, the pile that reaches the highest temperature, although the initial fungal composition in PS substrate was also the highest. The evolution of ratio archeal/bacterial on those mixtures it is not so clear. Despite archaea were detected in all the samples, the ratio archaea/eubacteria was maintained below 0.4%, indicating good aerobic condition of the piles. It must be also considered that the detection of microbial DNA does not indicate the presence of alive or intact microorganism due to the persistence of DNA within or released from cells that once lived in the investigated site, and that fact must be kept in mind when DNA-based techniques are used in analysis microbial numbers in environment (Hultman et al., 2010).

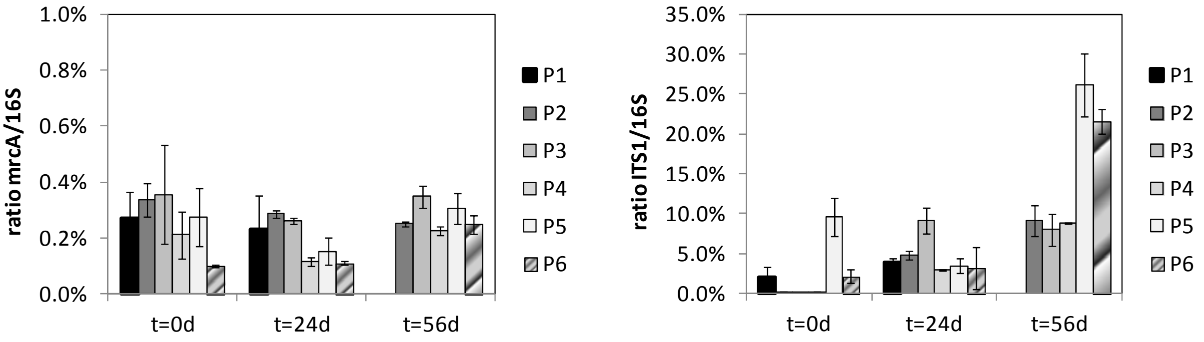

FIGURE 5 **Average archaeal/bacterial (Figure 5a) and fungal/bacterial (Figure 5b) ratios from three independent DNA extracts in samples taken from compost piles at time 0, 24 and 56 days. Standard deviations are presented as error bars.**

3.5 Evaluation of compost quality

Table 2 shows the main physico-chemical, chemical and biological properties of the mature composts obtained. All the composts showed pH values near to neutrality, suitable for their agricultural use. However, the electrical conductivity values were slightly high, especially in the compost 5, probably due to the use of paper mill sludge, co-substrate that presents a high mineral content (Table 1). The values of the C/N ratio ranged between 9.6 and 8.4, but in all cases being lower than 12, the limit established by the Spanish legislation (BOE, 2011) and by different authors as the limit for mature compost (Bernal et al., 2009). In addition, all the composts showed notable concentrations of total organic C and macronutrients, with final contents of total N higher than 2%, and similar concentrations of P and K to those found in other materials frequently used as organic amendments, such as composts from agroindustrial wastes and manures (Bustamante et al., 2010) or those elaborated using sewage sludge or municipal solid waste (Farrell and Jones, 2009).

TABLE 2 **Physico-chemical, chemical and biological characteristics of the mature composts obtained.**

	Compost 1	Compost 2	Compost 3	Compost 4	Compost 5	Compost 6
pH	6.37	6.20	6.75	6.62	6.21	6.87
EC (dS m^{-1})	7.34	7.41	7.67	7.42	8.67	7.56
TOC (%)	24.2	24.7	29.3	27.6	24.0	27.6
TN (%)	2.63	2.60	3.29	2.95	2.87	3.09
C/N ratio	9.21	9.54	8.97	9.34	8.38	8.94
P (g/kg)	16.2	19.4	14.7	16.5	12.7	15.5
Na (g/kg)	3.93	4.28	4.20	5.85	7.45	7.45
K (g/kg)	7.50	7.67	9.34	8.36	7.16	9.43
Ca (g/kg)	101	88.0	89.6	84.0	129	82.8
Mg (g/kg)	17.7	21.0	16.3	18.4	13.5	16.5
Fe (mg/kg)	4969	5394	4779	5208	4059	5894
Cu (mg/kg)	396	389	293	456	392	663
Mn (mg/kg)	664	817	634	711	514	618
Zn (mg/kg)	3012	3799	2671	3297	2217	2877
Cd (mg/kg)	0.23	0.31	0.18	0.24	0.24	0.22
Pb (mg/kg)	20.4	13.0	12.9	17.4	22.8	29.5
Ni (mg/kg)	18.5	20.5	16.9	37.3	32.4	57.8
Co (mg/kg)	1.29	2.83	1.89	2.44	1.17	1.23
Cr (mg/kg)	20.2	23.3	18.2	20.3	18.5	29.0
GI (%)	83.5	76.7	60.0	57.9	88.5	42.2

EC: electrical conductivity; TOC: total organic carbon; TN: total nitrogen; GI: germination index.

In general, micronutrient and heavy metal concentrations were similar to those reported in agroindustrial wastes and lower than those observed in compost from municipal wastes, both previously mentioned. Heavy metal contents were lower than the limits established for compost by the Spanish legislation (BOE, 2011) and the European guidelines (EC, 2001), except for Cu in compost 4 and compost 6, and Zn in all the composts (probably coming from the anaerobically digested pig slurry). In relation to maturity parameters, all the composts fulfilled the criterion related to the absence of phytotoxicity, as indicated by the germination index and also verify the C/N ratio, as it has been previously commented.

4 CONCLUSIONS

Composting of the solid fraction of digestate from the anaerobic digestion of pig slurry with different wastes as co-composting agents constitutes an efficient method to recycle these wastes and to obtain composts with, in general, a good degree of maturity and suitable physico-chemical and chemical properties, except for the notable salinity contents and the concentrations of Cu and Zn in some of the mature composts coming from anaerobically digested pig slurry that can limit their agricultural use. The use of the bulking and co-composting agents in the mixtures allowed the recycling of these materials and improved the composting process, especially maize straw in the proportion of 20% and paper mill sludge at 35%, showing these piles (P2 and P5) a better development of the thermophilic phase than the pile elaborated only with SFP (P1). However, the type of waste used as co-composting agent strongly influenced the final properties of the compost obtained, since the use of SS and PW increased the Cu concentrations, while PS increased the salinity in the end-product. Microbial culture independent techniques have been shown as a good tool that can help to understand process evolution.

5 ACKNOWLEDGEMENTS

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Session 20

(190) SUCCESSFUL PILOT PROJECT – COMPOSTABLE ECOVIO® BIO-WASTE BAGS FOR SEPARATE ORGANIC WASTE COLLECTION

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EXECUTIVE SUMMARY

BASF, Veolia Environmental Services, GML Abfallwirtschaftsgesellschaft, and the waste management service of the district of Bad Dürkheim, Germany conducted a three-month pilot project to ascertain whether ecovio® FS organic waste bags are suitable for residents' everyday use for organic waste collection. Furthermore the behavior of the bags was examined on an industrial scale in the composting plant Grünstadt - operated by the plant manager Veolia on behalf of the owner GML.

Almost 65.000 households in the municipality Bad Dürkheim were included in this trial. IBK-solutions, a consultant engineer office, was responsible for the project supervision. At the households the compostable waste bags were used for convenient, more hygienic separate organic waste collection. The filled bags were discarded in the regular organic waste bins and transported to the composting plant. These bags do not have to be separated out at the composting plant and together with the organic waste they can be further processed into compost. The degradation behavior of ecovio® organic waste bags was studied under realistic technical conditions.

For this purpose, two test heaps with ecovio® organic waste bags were compared to a reference heap without films made of this biodegradable material. The experiment was carried out at the organic composting plant in Grünstadt, which is authorized for a capacity of 35,000 Mg of organic waste per year and 5,000 Mg of plant cuttings per year.

The retention time in the compost pile was 3-4 weeks (3 weeks for the organic waste added last to the respective heap) for both test and the reference heaps.

A visual inspection during the emptying procedure ascertained that about 70% to 80% of the ecovio® waste bags had already been torn open when they came out of the collection vehicles because of the high mechanical stress to which they were subjected there (pressing and rolling).

The ecovio® organic waste bags did not cause any clogging or tangling in the low-speed grinder. The bags were torn open and shredded into flat films.

The decomposition process was not affected by the ecovio® organic waste bags. The temperature curve and the moisture content in the test heaps containing the bags made of the biodegradable material matched the values of the reference heap.

No remnants of ecovio® film were found during manual sorting of the material and visual inspection after 21 days, corresponding to the end of the decomposition period.

Following air separation and fine screening, only a few remnants of organic waste bags made of ecovio® were found in the reject. These were negligible in relation to the total volume of compostable ecovio® film material in the heap corresponding to a virtually 100% degradation.

An increased use of bags made of standard plastics was not observed after the pilot study.

Additionally the long time stability of the organic waste bags was tested. After 2 weeks beyond the normal storage time in the household, no changes occurred in the strength of the organic waste bags. The tear strength of these bags was comparable to that of the original bags.

1. INTRODUCTION

BASF, Veolia Environmental Services, GML Abfallwirtschaftsgesellschaft, and the waste management service of the district of Bad Dürkheim, Germany conducted a three-month pilot project to ascertain whether the organic waste bags made of BASF’s compostable plastic ecovio® FS Film were suitable for the residents’ everyday use for organic waste collection. Furthermore the behavior/processibility of the bags was examined on an industrial scale in the composting plant Grünstadt.

Almost 65.000 households in the municipality Bad Dürkheim were included in this trial. At the households the compostable waste bags were used for convenient, more hygienic separate organic waste collection. The filled bags were discarded in the regular organic waste bins and transported to the composting plant. These bags do not have to be separated out at the composting plant and together with the organic waste they can be further processed into compost.

In this manner, the organic waste bags made of ecovio® can contribute to reducing the amount of organic waste that ends up in the residual waste and thus lowering the costs associated with its disposal. An additional value in using ecovio® bags instead of conventional plastic bags is the reduction of plastic residues/fragments in the compost product used in agriculture.

If biodegradable materials are being processed in composting facilities, it is absolutely necessary to achieve a virtually 100% degradation during the technical retention time in the compost pile.

The degradation behavior of ecovio® organic waste bags was studied under the realistic technical conditions of the organic composting plant in Grünstadt (district of Bad Dürkheim, Germany).

Two test heaps containing ecovio® organic waste bags were compared to a reference heap without films made of this biodegradable material.

The objective was to assess the material behavior of the ecovio® organic waste bags during the individual process steps, ranging from the collection of the organic waste to the composting operation and all the way to the compost as the final product.

The other objective was to monitor the improper disposal of conventional plastic films and bags together with the organic waste before and after the experiment, and subsequently to make a comparison in order to evaluate the compost quality after the use of the ecovio® organic waste bags.

2. METHOD TEST SET-UP AND TIMELINE

The experiment was carried out at the organic composting plant in Grünstadt, which is authorized for a capacity of 35,000 Mg of organic waste per year and 5,000 Mg of plant cuttings per year. This composting plant processes organic waste collected by the waste management company GML from the districts of Bad Dürkheim and Rhine-Palatinate, the city of Ludwigshafen as well as the towns of Frankenthal, Speyer and Worms.

The bags were distributed to the rougly 65,000 households (each receiving a 10-pack, totaling roughly 650,000 bags) of the district Bad Dürkheim starting on April 6, 2011. The organic waste collected from this district was handled separately from the other delivered garbage.

The volume of foreign matter was monitored starting with the zero measurement before the distribution of the ecovio® organic waste bags to the households on September 23, 2010 and September 25, 2010. Again from March 31, 2011 to April 12, 2011 as well as four weeks after completion of the study from June 21, 2011 to July 1, 2011.

The composting process in a reference heap without organic waste bags made of ecovio® as well as in two test heaps containing ecovio® organic waste bags was monitored within the scope of the pilot project.

The retention time in the windrows was 3-4 weeks (3 weeks for the organic waste added last to the respective windrow).

The process sequence at the Grünstadt composting plant is descriped below:

- Receiving:

The organic waste delivered by the collection vehicles is mixed in the receiving hall by means of wheel loaders, subsequently freed of coarse foreign matter and conveyed through a flat bunker to the processing line.

- Coarse treatment:

The organic waste is immediately shredded by means of two low-speed shredders operating alternately. Before and after the shredding, metallic impurities are removed by means of magnetic separators.

Intensive-composting:

The organic waste is piled to table windrows by wheel loaders. The heaps are aerated by means of an individual pressurized aeration systems with oxygen regulation. The exhaust air passes through an humidifier and a biofilter, where it is purified.

At this stage complete hygenization of the organic matter is achieved.

- Fine treatment:

After the intensive composting, the raw compost is removed and sieved through a coarse screen deck (adjustable from 0 mm to 60 mm) and a fine drum-screen (perforations of 12 mm to 15 mm) to yield a coarse fraction (screen overflow), a fine fraction (vintner's compost) and a medium fraction (circulation material). An air separator picks up the lightweight materials of the fine fraction and blows them into a film tower. Dust is removed from the exhausted air by means of a Herding filter.

- Product:

ALVAHUM-quality compost bearing the RAL quality assurance seal is used primarily in agriculture as well as for specialized crops (wine, fruit and asparagus), but also for soil improvement in gardening and landscaping.

3. RESULTS AND DISCUSSION

3.1. Estimates of the number of bags that had already been torn open in the collection vehicles

At the times when the organic waste loads from the district of Bad Dürkheim were delivered, the emptying of the collection vehicles was observed on two days of the week. A visual inspection served to estimate the number of ecovio® organic waste bags that were already torn open and of those that were still intact.

The visual inspection during the emptying procedure ascertained that about 70% to 80% of the ecovio® waste bags had already been torn open when they came out of the trucks because of the high mechanical stress to which they were subjected (pressing and rolling). Consequently, the aerobization and biodegradation of the organic waste start immediately when the product is placed onto the windrow. A noticeable aspect was that most of the torn organic waste bags were located in the rear of the collection vehicle near the pressure plates, and in the front of the truck near the rotary drums.

3.2 Storage strength of ecovio® organic waste bags that are filled with organic waste but that have not been torn open, over the course of 1 or 2 weeks (long-term behavior)

The goal was to test whether the filled organic waste bags retain their strength during prolonged use in the households. For this purpose, still unopened organic waste bags that had been collected from the households were stored for two additional weeks and their strength was checked sensorially as well as visually.

No significant changes occurred in the strength of the organic waste bags after the additional time of storage; the tear strength of these bags was comparable to that of the original bags.

3.3 Behavior of ecovio® organic waste bags during the shredding step (low-speed roll-grinder) in the composting plant

The bags made of the biodegradable material underwent a visual and sensorial evaluation after being processed in the shredder (roll-grinder); the shredding behavior as well as the tendency towards clogging were also observed.

In low-speed grinders, ecovio® organic waste bags do not cause clogging or tangling. They were torn open and shredded into flat films.

3.4 Monitoring the conditions in the windrows and the degradation behavior of the organic waste bags

The process-related boundary conditions of the composting process were monitored by continuous recording of the temperature in the reference heap and in the two test heaps containing ecovio® organic waste bags. Additionally the temperature in each windrow was controlled by two manual measurements per week with an insertion thermometer. Moreover, the moisture content in the windrows was determined twice a week at a depth of about 0.5 meters.

The online measured temperature curves exhibit a similar course for the reference as well as for the test windrows which is typical for microbial degradation processes during composting.

The accompanying temperature measurements obtained by means of the insertion thermometer clearly substantiate this conclusion.

The moisture content of the material samples from a depth of about 50 cm from the surface was at a low level in all three heaps:

Moisture content of the reference heap: approximately 45% to 38%
Moisture content of test heap 1: approximately 45% to 40%

Moisture content of test heap 2: approximately 49% to 38%
The decomposition process was not affected in the least by the ecovio® organic waste bags. The temperature curve and the moisture content in the test heaps containing the bags made of the biodegradable material matched the values of the reference heap.

3.5 Degradation behavior of ecovio® organic waste bags after 1, 2 and 3 weeks of composting

At one-week intervals after the windrow was completed, which means that the open-top box was entirely filled, a partial removal was carried out in order to determine the quantities of remnants of ecovio® films that were found. The total decomposition time of the heaps, starting at the time when the wheel loader deposited the first load, was about 4 weeks for all of the heaps being compared. The decomposition time for the material that was stacked last onto the heap was only 3 weeks. This material was sampled. Remnants of ecovio® films that could still be recognized were subjected to a visual and sensorial inspection.
The quantity of remnants found as well as their characteristics after a decomposition time of 1, 2 and 3 weeks are compiled in the table below.
No remnants of ecovio® films were found during manual sorting of the material and visual inspection after 21 days, corresponding to the end of the intensive composting period.
The temporary breakdown (not because of the project) of the charging bunker involving the metering and conveyance to the shredder had no noticeable effect on the degradability of the ecovio® organic waste bags. During the breakdown, the bags were transferred to the test heaps without being shredded, where they nevertheless degraded as readily as the bags that had previously been shredded.

Table 1 Quantity and properties of ecovio remnants found

Screened amount in test heap 1:	Quantity and properties of remnants found		
2.5 m³ of organic material	after 7 days	after 14 days	after 21 days
	fragments of 14 bags: like parchment; slightly frayed; still tear-resistant	fragments of 13 bags: material frayed; strips; no mechanical strength	no remnants found
Screened amount in test heap 2:	Quantity and properties of remnants found		
2.5 m³ of organic material	after 7 days	after 14 days	after 21 days
	fragments of 18 bags: some only slightly degraded; severe loss of mechanical properties for fragments found in moister zones	no remnants found	no remnants found

3.6 Recovery rate for ecovio® remnants after mechanical post-treatment of the compost

The quantity of remnants found was studied by evaluating the total surface area parameter according to the quality and testing stipulations of the German federal quality association for compost, Bundesgütegemeinschaft Kompost e.V.
- after 20 mm screening
- after 15 mm screening
- after 12 mm screening

The total surface area parameter only evaluates flat components (essentially only film) in the compost. Limit values of 0.5% by weight in the dry substance apply to hard plastics, glass, metal, etc.
The test heaps were treated by air separation and screening immediately after the end of the technical retention time at the composting plant in Grünstadt. All fractions from the treatment were examined once again for remnants of ecovio®.

Very few residues of the organic waste bags made of ecovio® were found in the screen overflow of the two test heaps. These were negligible in relation to the total volume of film material in the heap. These values correspond to virtually 100% degradation.

In order to ensure statistical reliability, the evaluations of the compost quality were conducted with 4 repetitions. The 20-mm screening (coarse compost) that would yield a larger deviation from the standard was chosen for the comparison of the total surface area parameter.

Table 2 Total surface area as examined by the plancotec laboratory.

	Total surface area				
	1st sample	2nd sample	3rd sample	4th sample	**mean value**
reference heap	2 cm²	13 cm²	18 cm²	3 cm²	**9 cm²**
test heap 1	5 cm²	2.1 cm²	0.6 cm²	3.4 cm²	**2.7 cm²**
test heap 2	12 cm²	9 cm²	2 cm²	8 cm²	**7.8 cm²**

It can be concluded that the use of organic waste bags made of ecovio® does not affect the total surface area of the films in the compost.

3.7 Evaluation of user behavior by ascertaining the volumes of film in the organic waste delivered prior to the pilot project and after its conclusion

At the time of the introduction of the organic waste bins, the residents of the district of Bad Dürkheim were instructed to properly wrap moist kitchen waste in tissue paper or in newspaper; after that, it was found that bags made of standard plastics were used to a greater extent.

In this pilot project, the organic waste loads were examined before the ecovio® organic waste bags were distributed as well as four weeks after the end of the project. The amounts of plastic films (films and plastic bags) were recorded each time.

Greater use of bags made of standard plastics for collecting moist organic waste was not observed after the end of the pilot project. Moreover the evaluation indicated that ecovio® organic waste bags had not yet all been used up in the six weeks that followed their distribution and that considerable numbers of them could still be found in the collected garbage.

The individual values are compiled in the table below.

Table 3 Total surface areas of the films in the organic waste at the time of delivery.

			A	B	C	D	E	F	G	H
date of recording	recorded quantity in m^3	recorded quantity in Mg	in m^2	in m^2	in m^2	in kg	in kg	in kg	**in m^2**	**in kg**
23.09.2010	5 m^3	2.75 Mg	59.35			4.75			**21.58**	**1.73**
25.09.2010	5 m^3	2.75 Mg	60.73			4.86			**22.08**	**1.77**
31.03.2011	5 m^3	2.75 Mg	45.14	8.96		3.61	0.72		**19.67**	**1.57**
05.04.2011	5 m^3	2.75 Mg	30.41	2.43		2.43	0.17		**11.94**	**0.95**
07.04.2011	2.5 m^3	1.38 Mg	35.18	2.56	0.48	2.81	0.20	0.04	**27.35**	**2.18**
12.04.2011	2.5 m^3	1.38 Mg	42.75	2.40	4.48	3.42	0.19	0.36	**32.72**	**2.62**
20.04.2011	0.54 m^3	0.3 Mg	8.87	0.64	8.32	0.71	0.05	0.67	**31.70**	**2.54**
28.04.2011	2.5 m^3	1.38 Mg	14.42	0.16	6.56	1.15	0.01	0.52	**10.57**	**0.84**
21.06.2011	5 m^3	2.75 Mg	54.89	3.52	9.28	4.39	0.28	0.74	**21.24**	**1.70**
24.06.2011	4.6 m^3	2.53 Mg	42.63	2.24	7.04	3.41	0.18	0.56	**17.74**	**1.42**
28.06.2011	6.5 m^3	3.55 Mg	52.20	4.80	9.28	4.18	0.38	0.74	**16.06**	**1.28**
01.07.2011	5 m^3	2.75 Mg	57.23	4.48	7.68	4.58	0.36	0.61	**22.44**	**1.80**

A = total surface area of films made of standard plastics
B = total surface area of films made of biodegradable materials other than ecovio®
C = total surface area of ecovio® films
D = total weight of films made of standard plastics
E = total weight of films made of biodegradable materials other than ecovio®
F = total weight of ecovio® films
G = **total surface area of all films except ecovio® films, per Mg of input**
H = **total weight of all films except ecovio® films, per Mg of input**

specific weight of the organic waste at the time of delivery:
550 kg/m^3 on the average

🟨 before the test
🟦 after the test

4. CONCLUSION

The pilot project showed that the organic waste bags made of BASF's compostable plastic ecovio® FS Film completely meet the actual-practice composting requirements. Virtually 100% of the material degraded during the technical retention times of 3 to 4 weeks. No impairment of the compost quality was observed. No increased disposal of standard plastics was observed during the period under study.

Consequently, ecovio® organic waste bags can be used without problems for collecting organic waste in the district of Bad Dürkheim. A survey conducted subsequent to the project showed that the majority (~ 90%) of citizens who participated in the survey were satisfied with the product performance in their household's organic waste collection. Around 45% stated that they collect more organic waste because of improved hygiene, reduced bad smell and less cleaning of the waste bin. In conclusion the virtually quantitative decomposition of the material in the industrial composting process and the positive feedback from the citizens led to the approval of the ecovio® FS organic waste bags for collection of organic waste in the district of Bad Dürkheim.

The Grünstadt composting plant, with its current method and process conditions, particularly the short retention times, constitutes a facility that demands very short degradation times of biodegradable plastics. It can be concluded that the results of the pilot project in this composting plant can be transferred to other composting processes, provided that not only the temperature, but also the moisture content of the decomposing material are kept within the usual values of good practice. Depending on the configuration of the installation in question, it is advisable to conduct individual assessments on a case-to-case basis.

When it comes to the methods involving the usual pre-shredding of the organic waste or coarse screening with downstream shredding of the screen overflow, it can be assumed that the collection and delivery of organic waste in ecovio® organic waste bags will not have any detrimental impact on the composting.

In the case of installations that make use of visual inspection and manual removal of foreign matter such as films, there might be restrictions to the use of ecovio® organic waste bags.

(223) THE ADVANTAGES OF COMPOSTABLE BAGS FOR FOOD WASTE COLLECTION

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EXECUTIVE SUMMARY

After an introduction about biowaste legislation in Europe, this paper explains the benefits of using compostable bags, in this case Mater-Bi® bags, for residential food waste collection schemes. Mater-Bi® identifies a family of compostable resins produced by Novamont that contain native starch and other raw materials of agricultural origin. All Mater-Bi® grades are certified according to all main international compostability standards and some Mater-Bi® grades are specifically designed for producing food waste collection bags.

Two prominent features of this type of bags made for food waste collection are then described: compostability in various environments and water vapour permeability (breathability). In the last part, the advantages of breathability are presented through the results of a comparative study between vented vs. non vented kitchen containers tested in combination with Mater-Bi® breathable bags containing food waste. Weight loss, moisture and odour reduction have been investigated on a quali- quantitative basis. The conclusions show that vented collection systems can ensure higher comfort in the kitchen increasing the acceptance of food waste collections. Furthermore, vented kitchen containers in combination with Mater-Bi® breathable bags make it possible to collect less water and therefore a more energy rich feedstock for subsequent processing with a concurrent optimisation in the collection phase and reduction of overall impacts.

1 BACKGROUND TO BIOWASTE LEGISLATION AND MANAGEMENT IN EUROPE

In the European Union all Member States are bound by the principles and obligations introduced by the EU waste legislation as shown in Fig. 1 below (ETC/SCP, 2010).

FIGURE 1 **EU waste management legislation**

The revised EU Waste Framework Directive 2008/98/EC (rWFD) defines biowaste as “biodegradable garden and park waste, food and kitchen waste from households, restaurants, caterers and retail premises and comparable waste from food processing plants”. The total yearly production of biowaste in the EU ranges between 118 and 138 Mt., of which 30 to 50 Mt come from industrial sources (e.g. from the food processing industry) and about 88 Mt are municipal. 30% to 40% of MSW in the EU is biowaste (around 170 kilograms per inhabitant) and constituted by two main fractions: green waste from gardens and parks and food waste from households and commercial premises. 36 Mt (40%) of municipal biowaste is still landfilled and in some Member States this amount exceeds 90% (Arcadis et Al., 2010).

The Landfill Directive 1999/31/EC has provided long term and intermediate targets to reduce biodegradable municipal waste going to landfill, leaving Member States the freedom to implement alternative policies at country level. Diversion policies usually include a mix of source separation and material recovery, incineration and/or mechanical-biological treatment (EC, 1999).

In 2008 the revised EU Waste Framework Directive reinforced the waste hierarchy approach and introduced recycling targets for specific fractions. A 70% target by 2020 for construction and demolition waste was set whereas for household waste a target of 50% by 2020 was introduced along with a mandatory requirement for the collection of glass, paper, metals and plastics .

Whilst the rWFD does not contain specific targets or obligations for collecting and treating biowaste, in Article 22 it does state that Member States shall take measures to encourage the separate collection of bio-waste for composting and digestion whilst ensuring a high level of environmental protection for the production of environmentally safe materials from bio-waste. Furthermore, the Commission shall carry out an assessment on the management of bio-waste examining the opportunity of setting minimum requirements for bio-waste management and quality criteria for compost and digestate from bio-waste (EP/EC, 2008).

In response to this, in 2008 the Commission published a Green Paper on the Management of Bio-waste in the EU (EC, 2008) as well as preparing an Impact Assessment of a potential legislative proposal. This assessment (Arcadis et Al., 2010) has identified significant environmental and economic benefits from improved management of biowaste in the EU: “The market for quality compost could be increased by a factor of 2.6 to reach about 28 Mt. This could help to improve the quality of 3% to 7% of depleted agricultural soils in the EU and to address the problem of degrading soil quality in Europe. Maximizing composting could also substitute 10% of phosphate fertilizers, 9% of potassium fertilizers and 8% of lime fertilizers. About one-third of the 2020 EU target to use renewable energy in transport could be met by using the biogas produced from bio-waste as vehicle fuel. If all the bio-waste produced in the EU in 2020 was treated by anaerobic digestion and the gas used by public vehicle fleets, the potential gain would be around 13 Mt CO2-equivalent. In terms of the achievement of the 10% biofuel target this would represent an average contribution ranging from 6% in Luxembourg up to 85% in Romania. Emissions of between 10 Mt to a maximum of 50 Mt CO2-equivalent could be avoided by more prevention and biological treatment, representing between 0.4 % and 2.3 % of the EU 2005 GHG emissions (not covered by ETS) in 2005. This could represent between 4% to a maximum of 23% of the 2020 EU target (10% reduction compared to 2005 emissions) for the non ETS GHG emissions.” (EC, 2010b)

The assessment was followed in May 2010 by the “Communication on future steps in bio-waste management in the European Union” (EC, 2010a). In this document the European Commission claims that according to its analysis it doesn’t see any policy gaps that justify the need for a specific Biowaste Directive. On the other hand the Communication also states that: “Aligning the management of bio-waste better with the waste hierarchy and other provisions of the rWFD could result in environmental and financial benefits of €1.5 billion (moderate increases of recycling) to €7 billion (ambitious recycling and prevention policies). Furthermore: “Composting and anaerobic digestion offer the most promising environmental and economic results for bio-waste that cannot be prevented. An important pre-condition is a good quality of the input to these processes. This would in the majority of cases be best achieved by separate collection. Member States should make strong efforts for introducing separate collection in order to meet high quality recycling and anaerobic digestion. Highly efficient systems based on source separation of various streams of biowaste exist already in Austria, Germany, Luxembourg, Sweden, Belgium, the Netherlands, Catalonia (Spain) and certain regions in Italy. Systems of separate collection can differ significantly depending on, e.g. the types of waste collected (food waste, garden waste etc.) and the availability of treatment options. The key for success lies in adaptation to local conditions and user-friendly design.” (EC, 2010a).

In 2009 the European Environment Agency published the report “Diverting waste from landfill “ which shows that biowaste, along with paper and cardboard, represents the largest share of per capita recycling of MSW in the EU, and the differences between Member States in the amount of biowaste being recycled has a significant impact on the total recycling rates. It also showed that since the introduction of the Landfill Directive composting and anaerobic digestion capacity has grown significantly e.g. in Italy it has tripled since 1999 (EEA, 2009).

European countries with highest total proportion of recycled biowaste, i.e. Denmark and Luxembourg, also have the highest amount of recycled garden waste per capita (80 to 120 kilograms). However, these two countries have a much lower level of recycled residential food waste (18 to 42 kilograms) (ETC/SCP, 2009). Typically, residential food waste still represents a significant part of the total MSW and needs to be tackled for effective diversion.

2 THE CONTRIBUTION OF COMPOSTABLE PLASTIC BAGS TO SOURCE SEPARATION OF BIOWASTE AND DIVERSION FROM LANDFILL

Since the very first pilot projects in 1992 with the Agricultural Institute “Istituto Agrario San Michele all’Adige” in Italy and the German Local Authority of Fürstenfeldbruck, near Munich (C. Bastioli and F. Degli Innocenti, 1996), compostable plastic bags for the separate collection of municipal organic waste have come a long way and have gained popularity in European and North American source separation programs. Today, thousands of municipalities across the world are allowing and promoting the use of compostable plastic bags in their schemes for collecting yard trimmings and most importantly residential and commercial food scraps.

2.1 Europe

Biodegradable and compostable plastic bags complying with the EU standard EN 13432 have already proven to be a useful tool in optimising kerbside collection of biowaste (C. Saintmard, 2005).

In general, for source separation of food waste from households, the use of compostable plastic bags is more frequently associated to those schemes that have chosen to split the collection of garden waste from food waste. In the UK in 2011, 95% of the local authorities who operated separate residential food waste collection and 60% of those operating co-mingled schemes either provided for free or promoted the retail purchase of compostable liners. The English WRAP, Waste and Resources Action Programme, has performed extensive research on separate food waste collections (WRAP, 2009) with a specific focus on compostable plastic bags (WRAP, 2008). Waste authorities in the Spanish region of Catalonia are actively supporting the use of certified compostable plastic bags for kerbside food waste collections (I. Puig Ventosa et al., 2008).

Also in countries where co-mingled collection of garden and food waste is the rule, biobags can be quite popular: in Norway, a survey in 2005 showed that 46% of the municipalities were using biobags (H. Lystad, 2011). Further examples can be found in Austria and in the region of Bayern in Germany (F. Schneider et al, 2005). In Switzerland, compostable plastic bags are actively promoted along with vented kitchen containers by the main national energy and anaerobic digestion company (Axpo AG, 2009), (A. Brechtbuehl, 2010). A household survey performed in 2011 has shown highest captures in those municipalities collecting on a weekly basis and where vented kitchen containers and compostable bags were promoted (F. Ruoss, 2012).

Italy is the country with the most extensive use of compostable bags worldwide. In 2010, over 20 million people were connected to a food waste collection scheme (M. Giavini et al., 2012). After the introduction of a non-biodegradable shopping bag ban in 2011 where compostable shopping bags are exempted, these have become the most popular tool to collect residential food scraps with significant beneficial effects on the quality of the collected feedstocks going to composting. Italy’s kerbside collection schemes are source segregating food separately from yard waste and well established curbside collection programs capture around 200 Kg/hh*y food scraps. Recent research from the UK confirms that schemes with combined food and garden waste collection achieve a much lower yield per household, compared to weekly food waste only collections, and hence a lower level of diversion of food waste from the residual waste (WRAP, 2010). This system is characterized by high frequency collections of food waste in order to keep the organic fraction low in the residual waste stream (less than 10%). Food waste collection typically ranges from one to three times a week, depending on season and local climate conditions. As a consequence, residual waste can be collected on a weekly or biweekly (fortnightly) basis or even less frequently because of its reduced content of putrescible materials. This is balancing the cost of higher collection frequencies for food waste.

A key feature of the system for keeping high participation and food waste captures is the use of vented kitchen containers (8 to 10 litre) and breathable and compostable plastic bags, certified according to the European standard EN 13432 (C. Garaffa et al., 2010). Fig. 9 showcases examples of European communities that have adopted this system which is described under §5.

BRUN DUNK
Bruksanvisning og nyttig information

Bruksanvisning på 1-2-3
① Sett bioposen i kjøkkenspannet
② Kast matavfallet i bioposen
③ Legg bioposen i den brune utedunken ved behov

Weekly Food Waste Collections Are Coming!

Improving your wheeled bin service
Instructions Inside

Starting After October 10th

Food waste can go in your green wheeled bin along with your compostable garden waste

recycle for Cardiff
ailgylchu dros Gaerdydd

C2C 2087 2087

Free with this leaflet you will have received:

A Kitchen Caddy

Biodegradable Caddy Liners*

*Only use the FREE Council provided liners.
If you have not received these, please call C2C on 2087 2087

How do I use my kitchen caddy?

It's as easy as 1, 2, 3

1 Line your kitchen caddy with the biodegradable bags provided and fill it with food waste. Please remove all food waste packaging and wrappings.

2 When the bag starts to get full, tie it and put it in your green wheeled bin along with your compostable garden waste.

3 Place your green wheeled bin on the kerbside weekly along with your black wheeled bin.

Please start using your caddy from October 10th – see enclosed leaflet for important collection information. Your new collection service starts the week commencing October 13th.

Einfach – sauber – ökologisch

Private Haushalte sammeln Bioabfälle

Von der Abfall- zur Ressourcenwirtschaft: Die Grünabfuhr macht's möglich

Kraft der Vergärung zu einer nachhaltigen Gesellschaft

Wussten Sie, dass

Axpo Kompogas AG
Ihr Partner für eine nachhaltige Zukunft

WIR in STAINACH

Aus der Gemeindestube

Neuartige Bioabfalleimer erhältlich!

bioMat

Luftig, frisch und einfach Bioabfall sammeln!

Gemeinde Stainach übernimmt die Kosten des Fahrtechnikkurses für die Stainacher Jugend!

Impressum

Nächster Redaktionsschluss: 24.10.2005 - Bitte Termin vormerken!

Seite 04

Modalità di raccolta

Raccolta porta a porta
2 volte alla settimana

Guia pràctica per a la recollida **porta a porta** en municipis de fins a 5.000 habitants

4.1.4 Metodologia de lliurament de les diferents fraccions.

No hi ha una única forma de lliurament en els sistemes de recollida porta a porta, tanmateix el quadre següent enumera les formes més freqüents de lliurament de les diferents fraccions:

FIGURE 2 **Examples of communication by European local authorities and waste management companies about vented kitchen containers and compostable plastic bags. From top left to bottom right: Norway, UK, Switzerland, Austria, Italy, Catalonia (Spain).**

2.2 North America

In the United States, the number of communities with residential food waste collection service has grown by more than 50 percent since 2009. There are now over 150 communities with SSO (source separated organics) programs spreading across a total of 16 states. Most communities provide kitchen bins to each household, and many encourage use of certified compostable plastic bags (R. Yepsen, 2012). The majority of the SSO programs targeting residential food scraps are based on the west coast in California and Washington State. Most of them are accepting and actively supporting the use of certified compostable plastic bags in the collection, e.g. the cities of San Francisco and Seattle (Fig. 3).

FIGURE 3 **San Francisco, communication about food scraps collection with compostable bags (left). Seattle, three bin system with green bin accepting compostable packaging and food service ware (centre). King County (Washington State), on-line promotion for compostable bags starter kit (right).**

King County in Washington State, conducted an investigation to determine specific concerns of people about food waste collection and to analyze which methods, policies and/or programs of encouraging food scrap recycling might help people overcome their concerns (S. Belcher, 2008). Survey results suggest the major concerns happen when people actually start to recycle food scraps. A variety of incentives exist to engage current non participants in food scrap recycling. They include, but are not limited to, outreach and education, particularly on how to make it easier to recycle food scraps, financial incentives like lowering garbage bills in response to recycling food scraps, weekly pickup of food scraps, and low cost products to make it easier to recycle food scraps. One question of the survey addressed specifically the tools to make the collection easier: “I would recycle food scraps if the city provided products cheaply or at cost to make food scrap recycling easier and less messy, like…”. There was very little difference between non participants and participants the the (voluntary) SSO program, who responded positively to this statement. Both options, kitchen containers for food scraps and compostable plastic bags, received a very strong positive response (Fig. 4).

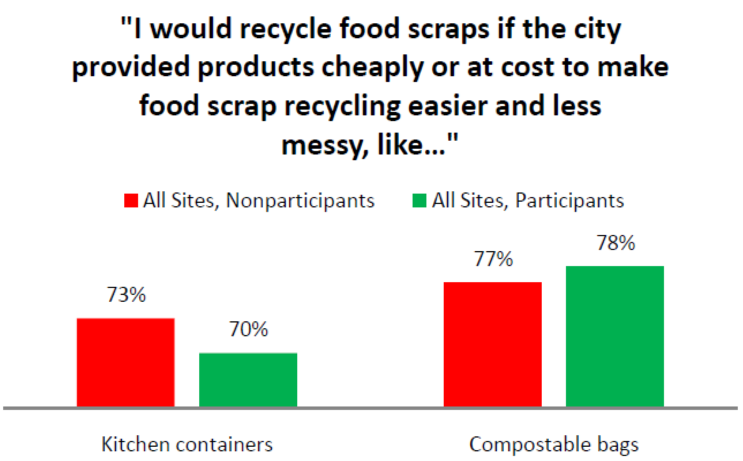


FIGURE 4 **Types of information requested by those who responded “yes” to the question “I would recycle food scraps if the city provided products cheaply or at cost to make food scrap recycling easier and less messy, like…”. (S. Belcher, 2008)**

In North America, the largest place with consolidated collection programs for residential food scraps is probably the Greater Toronto Area (GTA), which is the largest metropolitan area in Canada, with over 6 million people. The GTA is made of the city of Toronto, along with four confining Regions: York, Halton, Durham, and Peel. The Regions of Durham, Peel and Halton banned plastic bags from the beginning to control contamination issues. York Region and the City of Toronto chose to allow (non compostable) plastic bags as a way to encourage public acceptance and participation in the SSO program. When compared with Durham, Peel and Toronto, York Region’s Green Bin program collects more SSO material per household (299 kg/hh*y). Convenience for residents has always been a key focus for York Region’s SSO program. The more convenient a program, the more likely residents will participate: in turn this drives increased waste diversion. To improve the quality of the collected materials, since may 2011 non compostable plastic bags in the SSO program are banned and the use of certified compostable bags in the York Region is mandatory (L. McDowell, 2010). Durham, Peel and Halton (and now also York) have education programs in place to prevent non-compostable bags from entering the processing system (Fig. 5).

SORRY...

Your **GreenCart** was not collected because it contains an unacceptable liner bag.

Green recycling bags, oxo-biodegradable bags, plastic shopping bags, and garbage bags are not accepted as they are made of regular plastic and do not compost. Not all liners are accepted for use in the GreenCart.

Acceptable liners include:

- Paper towels
- Cardboard
- Paper food waste bags
- Newspaper/flyers
- Paper bags
- Certified compostable bags with this logo:

For a list of GreenCart acceptable bags and where they can be purchased, visit **www.halton.ca/waste.**

PWES-09093

Halton Region
Dial 311 or 905-825-6000
Toll free 1-866-4HALTON (1-866-442-5866)
TTY 905-827-9833
www.halton.ca

FIGURE 5 **York Region, communication about compostable bags (left). Halton Region, non compliance sticker placed on the green bin in case of contamination from non BPI certified compostable bags (right).**

Among its routinely household surveys regarding the SSO program, Halton Region has also investigated the set out preferences of households i.e. what type of bag each resident is using, if any, for collecting the food scraps. The first study where Halton Region started looking at bags in the green bin was in summer 2009 (N. Mantel, 2010). The number of residents using BPI certified compostable plastic bags was much higher than any other liner. Also, within the study, any resident using unacceptable liner bags had their green bins tagged (Fig. 5, right). This tag let residents know they were using the wrong bag, and their GreenCart (green bin) would not be picked up that week. Those residents that were tagged were given a written letter on the differences between plastic bags and BPI certified bags and were given samples of proper BPI liner bags. The number of people using improper plastic bags decreased during the 4 week investigation (Tab. 1).

Set out preference	Week 1	Week 2	Week 3	Week 4	Average
Compostable plastic bags	72.99	79.77	78.80	78.82	77.60
Paper bags	7.05	7.77	10.40	11.84	9.26
Nothing	11.35	5.83	6.00	5.61	7.20
Non compostable plastic bags	8.61	6.63	4.80	3.74	5.95

TABLE 1 **Shares of householders (percentage values) using different types of bags for collecting their food scraps in the Halton Region during a survey conducted in 2009.**

The Halton Region performed further participation set-out studies twice in 2010 and once in 2011. Tab. 2 below summarizes the 2009, 2010 and 2011 average percentages. The use of certified compostable plastic bags has gone up over the years, and the use of unacceptable plastic bags has gone down. Halton Region's program success definitely relies on the use of BPI certified compostable plastic bags (N. Watt, 2012).

Set out preference	2009	2010	2011
Compostable plastic bags	77.60	82.68	86.54
Paper bags	9.26	7.88	6.39
Nothing	7.20	5.27	4.99
Non compostable plastic bags	5.95	4.19	2.07

TABLE 2 **Shares of householders (average percentage values) using different types of bags for collecting their food scraps in the Halton Region during the surveys conducted in 2009, 2010 and 2011.**

2.3 The benefits of using compostable plastic bags in SSO programs

Since the very first pilot projects in 1992 with the Agricultural Institute "Istituto Agrario San Michele all'Adige" in Today, among those communities promoting the use of compostable plastic bags, the value of this tools for collecting organic waste by keeping high participation, high capture rates and high quality inputs to treatment facilities is very well recognized, both in areas of single-family houses and areas with denser population and multi-family buildings.

The advantages of compostable plastic bags (liners) are summarized below:

- Compostable plastic bags combine the strengths of conventional plastics (watertight, flexible and mechanically strong) with those of paper (compostable);
- Make the source separation of food scraps cleaner in the household (North Americans would say they reduce the "yuck factor");
- Reduce pests (e.g. flies);
- Favour higher participation rates. Especially where collection programs are voluntary, the use of liners can be a critical factor for active participation by property managers and householders;
- Favour higher capture rates. User friendly systems help in diverting more food waste from residual waste especially by allowing the collection of meat and fish scraps along with vegetables;

- Without liners the householders tend not to put wetter food waste into the kitchen containers;
- In multi-family buildings, liners make it possible to drop the food scraps on the way out (e.g. going to work) without the need to return the kitchen container;
- Less frequent washing of kitchen containers and outdoor bins needed;
- Liners can also extend the life of kitchen containers and outdoor bins;
- Liners are preferred by collection crews as food scraps don’t stick to the bins and all food waste is tipped easily into the collection vehicle;
- Collection vehicles are kept cleaner and spills are reduced;
- Transport and intermediate storage of residential food waste are easier to manage;
- Compostable bags made with specific bioplastics containing native starch have high water vapor transmission rates and are therefore breathable. In combination with vented kitchen containers they allow for water evaporation and drying of the food waste, hence reducing fermentation processes and the production of unpleasant odors in the household;
- By loosing up to 15% water and weight during storage in the vented kitchen container, breathable liners act as a waste reduction tool, minimizing the organic waste to be transported;
- Compostable plastics can act as a communication tool during outreach and education campaigns positively influencing the sorting behaviour of householders and consumers;

Considerations about the added value given by compostable plastics can be further extended from bags to other items e.g. food service ware. Many communities (e.g. San Francisco and Seattle on the US west coast) have implemented legislation mandating the use of food service ware items either recyclable in their systems or compostable and are accepting this products in their collection programs. The reason behind this choice is that the use of disposable non recyclable food service ware in the commercial sector usually generates a mixed waste containing food scraps and non compostable plastic items. This waste is hard to recycle and is usually sent to landfill or incineration. By using compostable items, an alternative waste management scenario is possible where a homogeneous waste fraction (food scraps with compostable items) can be sent to organic recovery facilities. Under an LCA perspective the environmental advantages of this choice have been described by F. Razza et Al., 2009.

3 THE MATER-BI® RESIN AND NOVAMONT

3.1 What is Mater-Bi®

Mater-Bi® is a family of bioplastics that uses substances obtained from plants, such as starch, and biodegradable and compostable polymers obtained both from renewable raw materials and fossil raw materials. Mater-Bi® was the first family of industrial biopolymers using starch by preserving the chemical structure generated by photosynthesis. For developing Mater-Bi® Novamont was awarded by the European Patent Office and the EU “Inventor of the year 2007” for the 1992 – 2001 patents on bioplastics and industrial achievements.

Mater-Bi® resin is produced in granular form and can be converted into products using the most common processing techniques for plastics. It can be used to manufacture products having similar characteristics to those of traditional plastics, but featuring biodegradability and compostability according to the main international standards like the EN13432 and EN14995 (Europe), ASTM D6400 and ASTM D6868 (USA), BNQ 9011-911/2007 (Canada) and the international standard ISO 17088.

Usually plastic materials are not or only partially water soluble and the long polymer chains cannot be transported directly into the microbial cells (Fig. 6). Therefore microorganisms excrete enzymes breaking the polymer chains down to water soluble fragments which are incorporated into the microbial cells and metabolized (this way of assimilation is normally used for natural polymers). For the general understanding of the term biodegradable in connection with plastics it has been widely agreed, that the microbial induced attack of a polymer, (e.g., determined as deterioration or weight loss), is not sufficient but ideally a complete transformation of the entire plastics components into naturally occurring materials is required (R.J. Müller, 2005).

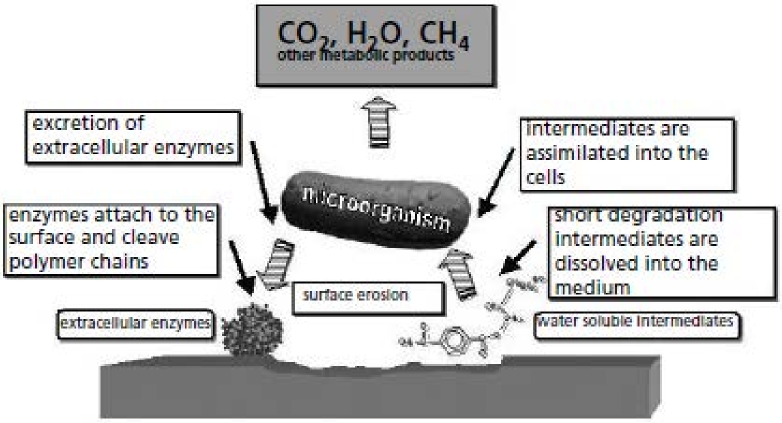


FIGURE 6 **General scheme of microbial polymer degradation (R.J. Müller, Aliphatic-Aromatic Polyesters, Handbook of Biodegradable Polymers, RAPRA, 2005, 309).**

Today, under the Mater-Bi® trademark, Novamont produces a wide range of biodegradable and compostable materials for various industrial applications e.g. for organic waste collection, agriculture, packaging, food service ware. In the grades containing starch, Novamont's Mater-Bi® technology almost completely destroys the crystallinity of amylose and amylopectin, in the presence of macromolecules which are able to form a complex with amylose such as specific polyesters. They can be of natural or synthetic origin, and are biodegradable. The ratio between amylose and amylopectin, the nature of the additives, the processing conditions and the nature of the complexing agents enables the engineering of various supramolecular structures with very different properties. Blending starch with aliphatic polyesters improves their processability and biodegradability. Particularly suitable polyesters are PCL and its copolymers, or polymers of higher melting point formed by the reaction of 1,4 butandiol with succinic acid or with sebacic acid, azelaic acid or poly(lactic acid), polyhydroxyalkanoates and aliphatic-aromatic polyesters. These types of materials are characterised by excellent compostability, mechanical properties and reduced sensitivity to water (C. Bastioli, 2005).

Different grades of Mater-Bi® are available for extruding films for specific applications. They can be processed in the same way as traditional plastics using standard polyethylene processing equipment. Some Mater-Bi® grades are specifically designed for producing bags for biowaste collection, be it garden (yard) waste or food waste from commercial or residential sources. Depending on the specific strength, compostability and breathability requirements, compostable films made of Mater-Bi® can have different properties within a wide range. Some of these properties are shown in Table 3.

Test	Standards Method	Unit	Mater-Bi	LDPE
Melt Flow Index	ASTM D1238	g/10 min	2-8	1-6
Breaking Strain	ASTM D882	MPa	20-40	20-40
Elongation at Break	ASTM D882	%	200-600	150-600
Young's Modulus	ASTM D638	MPa	100-600	100-300
Start of Tearing	ASTM D1938	N/mm	20-120	70
Start of Propagation	ASTM D1938	N/mm	20-120	70
Water Vapour Transmission Rate	ASTM E96	g*30μm/m² day 38°C 90%ΔRH	200-1100	15

TABLE 3 **Some Mater-Bi® Vs. LDPE (low density polyethylene) film properties.**

Two key features of compostable waste bags made of Mater-Bi® resin, namely compostability and water vapor permeability (breathability) will be discussed in §4 and §5 respectively.

3.2 Novamont's third-generation biorefinery

Starting as a research centre, which was demerged from Montedison in 1996 and bought by the merchant banking unit of Intesa - San Paolo Bank and by other institutional investors, today Novamont is an industrial company with more than 200 employees of which 25% are working in R&D. In 2010 Novamont reinvested over 6% of its turnover in R&D and is steadily growing. The company holds about 1.000 patents and has published more than 120 articles.

The continuous effort in research and development and recent industrial developments will make it possible to produce bio-products from non food crops grown in marginal and non irrigated land areas, and to use waste and residues to produce the energy needed in the processing phase, with the goal to progressively replace the remaining fossil based components of the Mater-Bi® polymer with renewable ones. Novamont's vision aims to develop a mature bioeconomy in Europe, by working closely with farmers, local communities and research institutions, and by moving the focus from a product-based economy to a more comprehensive system-based economy. This also means efficiently integrating the optimal end of life management of e.g. every day life disposable products by using biodegradable and compostable materials. This model of bioeconomy is based on the imitation and the closing of natural cycles, on the promotion of local production chains (from the raw materials down to the finished goods), on sound waste management practices and the recovery of resources to be put back into the production chain.

The mean to achieve these goals is represented by the concept of a third-generation biorefinery locally integrated in the territory. In June 2011 Eni, through its subsidiary Polimeri Europa, signed a cooperation agreement with Novamont SpA to convert Eni's Porto Torres chemical plant into an innovative bio-based chemical complex to produce bio-plastics and other bio-based products (bio-lubricants and bio-additives) for which significant growth is expected in the medium/long-term. The project will be supported by an integrated supply chain and raw materials of vegetable origin. Novamont will contribute with its technologies and skills in the bio-plastics and bio-based chemical sector. Eni will contribute to the joint entity with its Porto Torres plant, infrastructure and professional staff as well as its industry, technical engineering and commercial know-how in the petrochemical sector (ENI, 2011). The joint venture has been named "Matrìca" and this project aims to start a positive path based on technological innovation and sustainability with relevant social and occupational implications. More generally, this model of locally integrated biorefinery can potentially be replicated in different territories and an essential part of this innovation will be played by the upstream integration with local agriculture. Given the widely expected reduction in agriculture subsidies during the next decade in the EU, the project is a unique opportunity for the farming sector. Downstream, the loop will be closed with the application of compost produced by local treatment facilities on the land of the farmers producing the crops and supplying the raw materials for the biorefinery. Source separation of organics is therefore an integral part of this vision.

Besides the Matrìca project, another company Mater-Biotech S.p.A., 100 percent controlled by Novamont in joint venture with Genomatica Inc., is converting a dismissed industrial plant in Adria (near Venice, Italy), to use Genomatica's patented process for the synthesis of biobased 1,4 butandiol (Bio-BDO). Genomatica Inc. is headquartered in San Diego, California, and delivers manufacturing processes to produce intermediate and basic chemicals from renewable feedstocks. The company's proprietary biotechnology platform allows to create fermentation-based manufacturing processes of renewable feedstocks into intermediate and basic chemicals, including butandiol. Novamont is financing the plant conversion in Adria and will operate the facility, which is expected to have a production capacity of approximately 20.000 tpa with a first start up in 2013. This will be the first industrial plant in Europe producing Bio-BDO from renewable feedstocks. The Bio-BDO will be completely used by Novamont to meet the increasing demand for its biopolymer products that incorporate BDO as a key monomer.

4 COMPOSTABILITY IN INDUSTRIAL, HOME AND ON-SITE COMPOSTING: FLEXIBILITY ON THE WHOLE RANGE OF COMPOSTING ENVIRONMENTS

Currently, the compostability criteria for packaging and plastics are very well regulated at European and worldwide level. Mater-Bi® resins are certified 'biodegradable and compostable' under the EN13432 and EN14995 standards for Europe and under the ASTM D-6400 standard for the U.S. Some products made of Mater-Bi® are certified according to similar standards in Canada (BNQ 9011-911/2007), Australia (AS 4736) and Japan (GreenPLA). In Europe, the certifying bodies operating in the biodegradable and compostable plastics sector are CIC (Italian Composting Council) in collaboration with Certiquality (Italy), AfOR (U.K.), DIN CERTCO (Germany) and VINÇOTTE (Belgium). In North America BPI (Biodegradable Products Institute) in collaboration with NSF is operating in the U.S. and BNQ in Canada. Each program is managing an identification logo for product labeling and to assure compostability in industrial composting facilities (Fig. 7).

FIGURE 7 **Compostability logos managed by CIC-Certiquality (Italy), DIN CERTCO (Germany), VINÇOTTE (Belgium), BPI (U.S.) and BNQ (Canada).**

The above mentioned standards and certification programs are referred to industrial compostability only. Products certified accordingly do not necessarily break down in a home or back yard composting environment mainly because of the lower temperatures involved in the process. Although no European standard exists for home compostability, Vinçotte runs its own certification program and logo for home compostability (Vinçotte, 2012) by setting different temperature regimes and biodegradation and disintegration rate requirements for treating bioplastics in small scale systems. Moreover, the Association for Organics Recycling (AfOR) has established a 'home compostable' certification scheme and logo for the U.K., under a partnership arrangement with Vinçotte. The two certification logos are shown in Fig. 8.

FIGURE 8 **Home compostability logos managed by VINÇOTTE (Belgium) and AfOR (U.K.).**

Specific Mater-Bi® grades for the production of waste bags are certified according to these programs. A significant study has been conducted by the Bauhaus-Universität Weimar in Germany during the Kassel Project in 2002 (M. Klauss and W. Bidlingmaier, 2003). In this case, home compostability of Mater-Bi® bags has been successfully tested also in field trials. More recently, Mater-Bi® bags certified for home compostability have been tested in small scale on-site composting machines. In 2011 a full scale, 2 month long test has been carried out in Brighton (U.K.), in a housing area where a "Big Hanna" composter is treating the food scraps of 172 flats. During the test period approximately 200 Mater-Bi® bags filled with kitchen waste have been loaded into the hopper inlet. Within the first quarter of Big Hanna's cylinder only small residues of Mater-Bi® were still visible whilst either in the rest of the cylinder or in the finished compost further fragments could be found (Susteco AB, 2011).

5 BREATHABILITY: AN ADDED VALUE FOR THE COLLECTION OF FOOD WASTE

5.1 Breathable Mater-Bi® bags and vented kitchen containers

Depending on the required functionality, Mater-Bi® films can be highly resistant or highly permeable to water vapour. ASTM E96 is a standard test method for water vapour transmission rate (WVTR) of materials by which films can be compared for their ability to conduct moisture. Table 2 shows water permeability values of Mater-Bi® films at a temperature of 38°C and 90% relative humidity, ranging from 200 (fairly resistant to water vapour permeation) to > 1000 (highly permeable to water vapour). Mater-Bi® grades with high WVTRs are used for the production of highly breathable compostable bags. This feature is of particular advantage for the collection and storage of food scraps in the household when used in combination with vented kitchen containers (Fig. 9).

In recent years, many European programs have been switching from traditional non vented (solid sided) kitchen containers to vented models. This is because highly breathable Mater-Bi® resins enable wet food waste to transpire providing a weight and volume reduction (less waste to be collected and treated), improved storage (less odours), higher acceptance and participation by the householders. Fig. 2 shows examples of communication about vented kitchen containers and breathable and compostable bags promoted by local authorities and waste management companies in Europe.

In ventilated kitchen containers, the side walls, base and lid contain vents and open areas for moisture to escape through the highly breathable Mater-Bi® film. This in turn allows for the food scraps to dry reducing the build up of anaerobic conditions in the container. As a consequence, there is a reduction in the development of moulds, leachate and unpleasant odours. The moisture reduction is clearly shown by the significant weight loss measured during the storage of the food waste in the bag. Various investigations have been performed in different European countries e.g. Austria (H. Insam and S. Klammer, 2002), Norway (Aasen R., 2004). and Catalonia, Spain (Martin P., 2010).

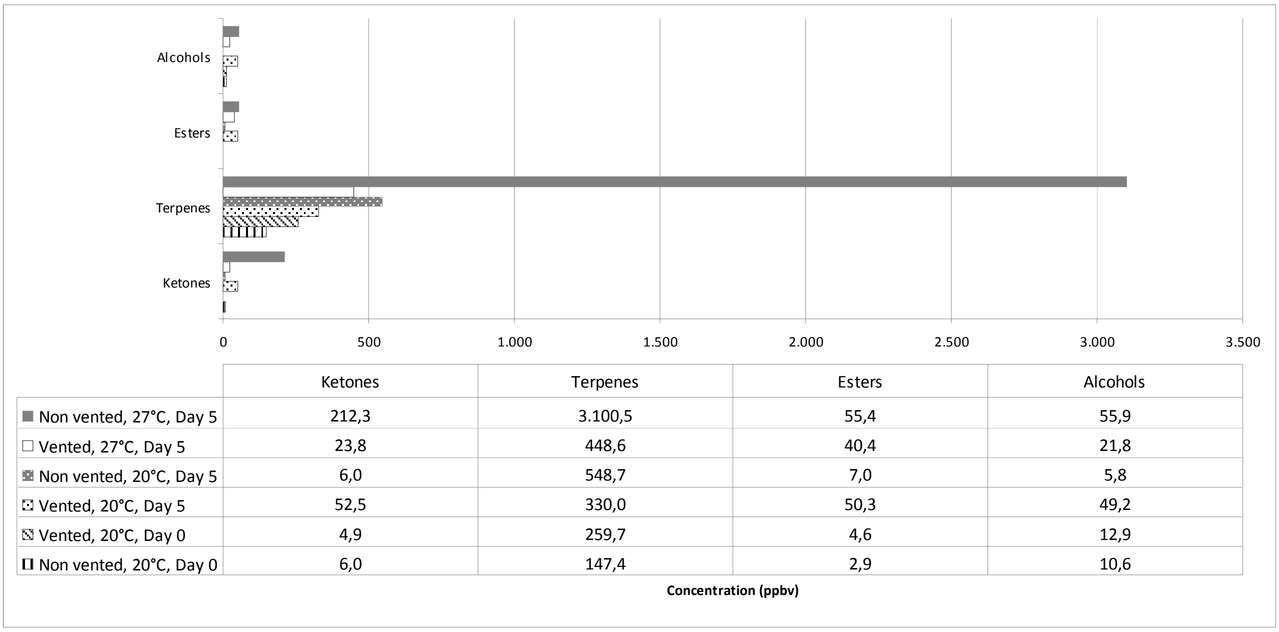
FIGURE 9 **Examples of vented kitchen containers and compostable bags made with highly breathable Mater-Bi® film.**

5.2 Odour generation by food waste stored in the household

The reason why odour is generated by food waste is that it rapidly begins to decompose, typically under partially anaerobic conditions, releasing various types of volatile organic compounds (VOCs) which have a low odour threshold (the concentration at which they are detected by the nose) and a negative "hedonic tone" i.e. give an unpleasant sensation. Terpenes, are usually the most abundant class of VOCs found in organic waste and are typically related to the perceived odour intensity (Mazza et al., 2000).

FIGURE 10 **Air sampling with a surface emission isolation flux chamber and Nalophan® bags. (M. Giavini et al., 2011).**

Odour emissions from vented Vs. non vented containers with Mater-Bi bags have been investigated (M. Giavini et al., 2011). The containers were filled with food waste and stored for 5 days under two different environmental conditions: 20°C, 55% RH and 27°C, 70% RH. Air samples were taken using a surface emission isolation flux chamber and then drawn into 5 litre Nalophan® bags using a sampler working with the lung principle (Fig. 10).
Fig. 11 shows the total concentrations of VOCs detected by Gas Chromatography - Mass Spectrometry (GC-MS) analysis. Terpenes appear to be dominant, whereas oxygenated compounds like alcohols, esters and ketones contribute much less to the odour because, despite their detection threshold similar than that of terpenes, the concentrations measured in relatively fresh food waste emissions are significantly lower. Compounds like di- and trimethylamines, dimethyl sulfides and disulfides, volatile acids like butyric acid, which have a very low odour threshold, weren't detected at all.



	Ketones	Terpenes	Esters	Alcohols
■ Non vented, 27°C, Day 5	212,3	3.100,5	55,4	55,9
□ Vented, 27°C, Day 5	23,8	448,6	40,4	21,8
▩ Non vented, 20°C, Day 5	6,0	548,7	7,0	5,8
⛶ Vented, 20°C, Day 5	52,5	330,0	50,3	49,2
▧ Vented, 20°C, Day 0	4,9	259,7	4,6	12,9
▥ Non vented, 20°C, Day 0	6,0	147,4	2,9	10,6

FIGURE 11 **Total concentrations of VOCs detected by GC-MS of air samples taken from vented and non vented kitchen containers and breathable Mater-Bi® bags after 5 days of food waste storage. (M. Giavini et al., 2011).**

GC-MS analysis has shown significant differences in terpene concentration values from vented Vs. non vented containers with breathable Mater-Bi® bags: at 20°C and 55% RH, 549 ppb_v (non vented) Vs. 330 ppb_v (vented) were detected, whereas shifting to summer-like conditions at 27°C and 70% RH, the difference increased to 3.100 ppb_v (non vented) Vs. 449 ppb_v (vented). Fig. 12 shows the main terpenes identified by GC-MS. The five main peaks are associated to Limonene (the most abundant), β-Pinene, α-Pinene, 3-Carene, β-Phellandrene.

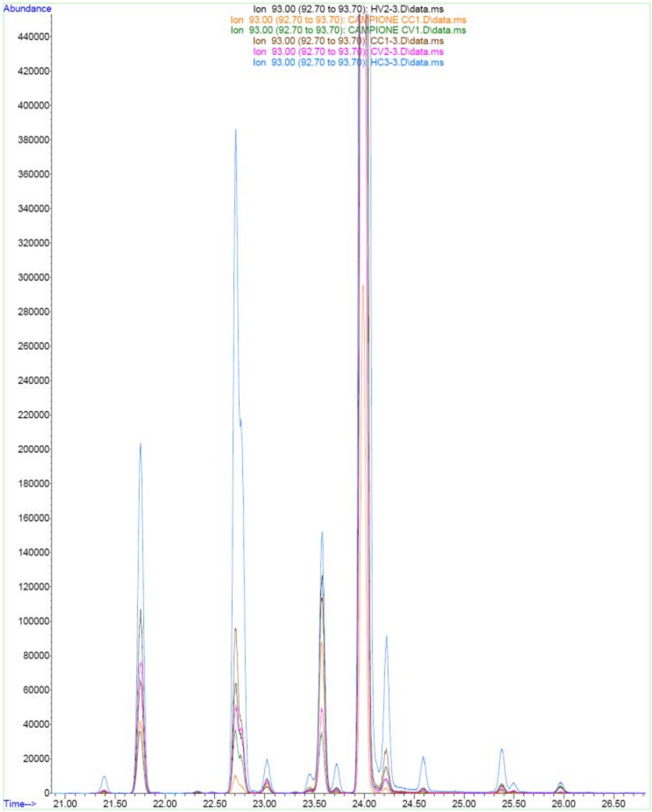


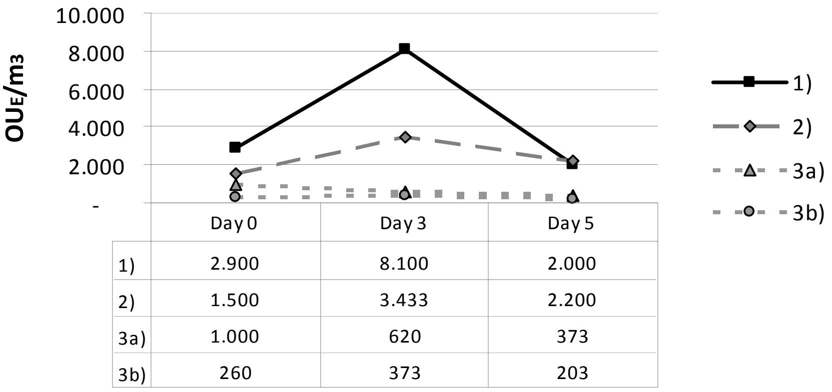
FIGURE 12 **GC-MS spectra of terpenes detected in the air samples taken from vented and non vented kitchen containers and breathable Mater-Bi® bags after 5 days of food waste storage. (M. Giavini et al., 2011).**

5.3 Dynamic olfactometry testing of vented Vs. non vented systems

Human nose responses may be based on the detection of compounds not included in GC-MS quantification analysis, so olfactometric assessments using human panelists have been also performed to quantify odour emissions in vented Vs. non vented containers (M. Giavini et al., 2011). Dynamic olfactometry for odour intensity and hedonic tone determination were used according to standards EN 13725:2003 and VDI 3882-2.
In this case four different containers with breathable Mater-Bi® bags were compared:

System 1: Non vented side walls + non vented lid
System 2: Non vented side walls + vented lid
System 3a: Vented side walls + vented lid, model A
System 3b: Vented side walls + vented lid, model B

Three replicas of each system were set up by filling them with 1,8 Kg of a mix of shredded food (cut fruit, vegetables, bread, cooked noodles, rice, meat and cheese). Air samples were drawn from the inner space of the containers after 3 and 5 days of storage at 25°C and 50% RH. For the quantitative determination according to the standard EN 13725:2003, the odour concentration was measured by presenting the air samples, at a range of dilutions, to a group of six panelists to find the dilution factor at the 50% detection threshold. The resulting concentration was expressed with European Odour Units per cubic metre (OU_E/m^3).
The results are shown in Fig. 13. After three days, system 1 (totally non vented) showed an odour concentration of 8.100 OU_E/m^3, compared to the 620 OU_E/m^3 and 373 OU_E/m^3 of the completely vented systems (3a and 3b). System 2 (vented lid only) showed 3433 OU_E/m^3. The average of the odour concentrations detected for the two totally vented systems was 14% of the system with vented lid only and 6% of the totally non vented system values.

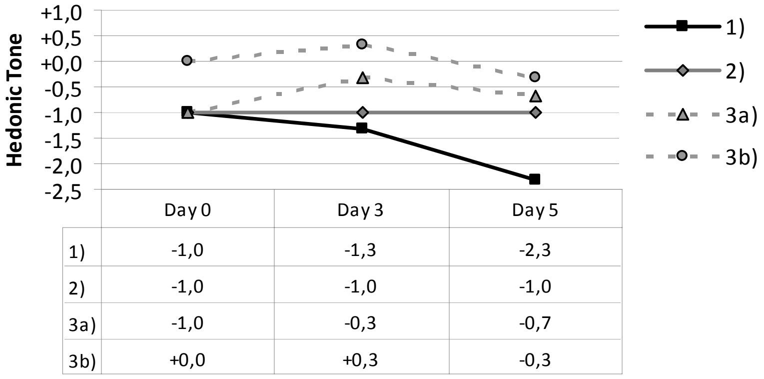


	Day 0	Day 3	Day 5
1)	2.900	8.100	2.000
2)	1.500	3.433	2.200
3a)	1.000	620	373
3b)	260	373	203

FIGURE 13 **Odour concentration determination on four different containers with breathable Mater-Bi® bags compared at 25°C and 50%RH with samples taken after 3 and 5 days of food storage. 1) non vented side walls + non vented lid, 2) non vented side walls + vented lid, 3a) vented side walls + vented lid, model A, 3b) vented side walls + vented lid, model B.**

After five days, odour concentrations declined significantly with system 1 and system 2 reaching values of 2000 OU_E/m^3 and 2.200 OU_E/m^3 respectively. In this case the average of the odour concentrations detected for the two totally vented systems was 13% of the values of system 2 (vented lid only) and 14% of system 1 (totally non vented).
The reason of this significant decline in odour concentration can probably be attributed to the consumption of the VOCs as a consequence of the fact that from day 0 to day 5 no more fresh food waste was added during the experiment.

Besides odour concentration, also the hedonic tone (relative pleasantness / unpleasantness) of the odour was determined according to VDI 3882. This standard guideline calls for a group of at least 15 assessors to be used and 6 presentations of the odour sample. To measure the hedonic tone a 9 point scale is used: +4 extremely pleasant, 0 neither pleasant nor unpleasant, -4 extremely unpleasant.



	Day 0	Day 3	Day 5
1)	-1,0	-1,3	-2,3
2)	-1,0	-1,0	-1,0
3a)	-1,0	-0,3	-0,7
3b)	+0,0	+0,3	-0,3

FIGURE 14 **Hedonic tone determination on four different containers with breathable Mater-Bi® bags compared at 25°C and 50%RH with samples taken after 3 and 5 days of food storage. 1) non vented side walls + non vented lid, 2) non vented side walls + vented lid, 3a) vented side walls + vented lid - model A, 3b) vented side walls + vented lid - model B.**

Fig. 14 shows how the perceived hedonic tone at day 0 was about -1 (slightly unpleasant). The vented systems 3a and 3b showed an improvement moving towards neutrality on day 3 and showed a slight deterioration on day 5. System 2 (vented lid only) remained stable on the value of -1. System 1 (totally non vented) showed a constant deterioration dropping below -2 at day 5.

As suggested by the standard VDI 3882, it should be noted that samples showing the same odour concentration can be perceived as more unpleasant by the human nose when being associated to a more negative value of hedonic tone. The totally vented systems 3a and 3b showed the best combination of low odour concentrations and less unpleasant hedonic tone both at day 3 and day 5.

5.4 Moisture reduction and weight loss

The weights of the four systems were measured at day 0, 3 and 5. The percentage weight loss for each system was calculated. Systems 1 and 2 showed a similar weight decrease of less than 5% in 5 days. The totally vented systems 3a and 3b showed a significantly higher weight reduction of more than 9% on day 3 and 14% on day 5. Conditions were the same as for the olfactometric analysis i.e. 25°C and 50% RH (Fig. 15)

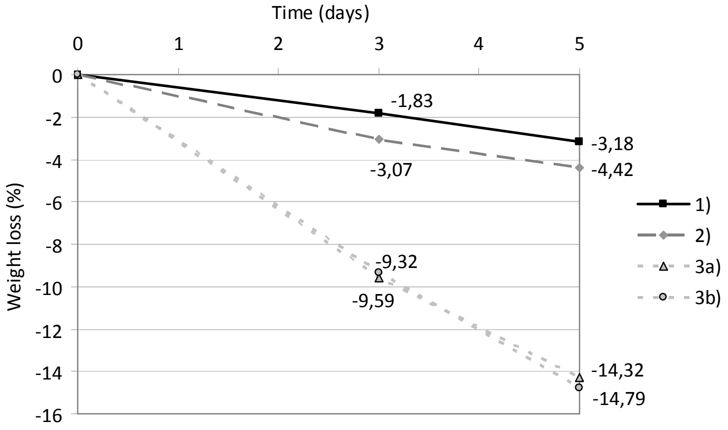


FIGURE 15 **Weight loss (%) on day 3 and day 5. 1) non vented side walls + non vented lid, 2) non vented side walls + vented lid, 3a) vented side walls + vented lid, model A, 3b) vented side walls + vented lid, model B.**

The loss in weight can almost completely be attributed to moisture evaporation and consequent water loss in the food waste. Content of moisture (water), total volatile solids (TVS) and ash were determined in the food waste on day 0 and on day 5 in of the systems 1, 2, 3a and 3b (Fig. 16). Whilst the ash and TVS remained relatively constant in all systems, a significant moisture reduction could be observed in the completely vented systems 3a and 3b only. This confirms that the weight and odour reduction in the stored food waste can certainly be linked to the water evaporation, which was significantly higher in the completely vented kitchen containers with breathable Mater-Bi® bags.

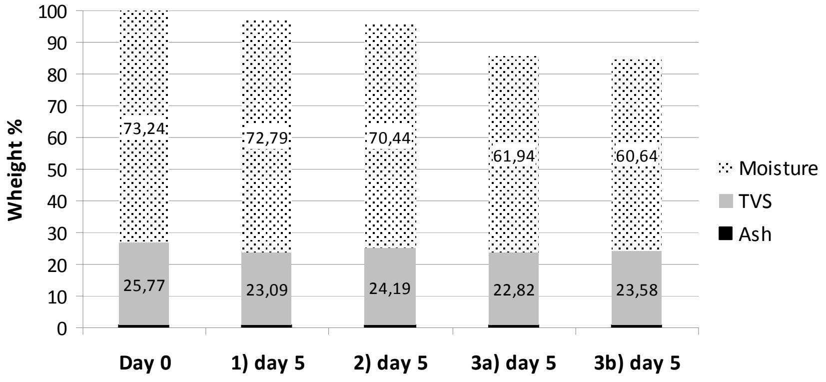


FIGURE 16 **Weight loss (%) between day 0 and day 5 of single food waste components: moisture, total volatile solids (TVS), ash. 1) non vented side walls + non vented lid, 2) non vented side walls + vented lid, 3a) vented side walls + vented lid, model A, 3b) vented side walls + vented lid, model B.**

6 CONCLUSIONS

With a progressively growing need to capture significant quantities of food waste from residential and commercial sources to achieve more ambitious targets of landfill diversion and increase the recovery efficiency of both material and renewable energy resources, today several best practice cases show how certified compostable bags are an extremely valuable tool in maximising participation and capture rates along with keeping high quality standards of the collected feedstocks. Mater-Bi® compostable resins give a further added value through specific home compostable grades (giving more flexibility with regard to specific small scale composting environments) and highly breathable grades, which combined with vented kitchen containers, represent a valuable tool for maximising comfort and acceptance by the public and achieving significant moisture reduction i.e. weight loss of the food waste to be transported.

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(235) BIO-BASED POLYMER PRODUCTS – OPPERTUNITIES AND LIMITATIONS FROM A DISPOSAL AND RECYCLING PERSPECTIVE

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EXECUTIVE SUMMARY

In the context of the discussion about limited fossil resources and climate change products made of bio-based polymers have been developed to an alternative to conventional polymers. Nowadays, products from bio-based polymers are used in several different fields, e. g. in form of films, bags or bottles as packaging materials, for catering products, everyday objects/commodities, compostable garbage bags as well as mulch films for agriculture application.

In Germany, the recycling and disposal of products from bio-based polymers follows the traditional ways together with all the other waste. However, some bio-based polymer products may cause difficulties in these established paths, pointed out the following.

The term bio-based polymers comprises a variety of polymeric materials. Therefore, a differentiated consideration is necessary regarding to the polymer species respectively types in the product as well as recycling and disposal paths.

Against the background of supporting the implementation of bio-based polymers into the market the Agency for Renewable Resources (Fachagentur Nachwachsende Rohstoffe e. V. – FNR) assigned the KNOTEN WEIMAR International Transfer Centre Environmental Technology GmbH identifying difficulties in the recycling and/or disposal process of products made of bio-based polymers and creating recommendations to support these products. The Institute of Lightweight Structures at the Chemnitz University of Technology and the law office AWKD Werner & Kollegen support the project as subcontractors on technical and legal aspects.

This paper is based on a literature review, colloquia and dialogues with different involved parties, e. g. disposers and recyclers.

Bio-based polymer products are more and more entering our daily life products – durable or non-durable. Finally, these bio-based polymer products become waste and end up in established waste streams. The biopolymer products may cause difficulties in the different waste streams being partially unsuitable for biopolymer products. At this point, a differentiated consideration of the biopolymer product variety only leads to useful waste disposal options. So far, material recycling of post-consumer products made of bio-based polymers is not known. The current missing critical mass for a material recycling is one reason. Today, most biopolymer products end up in the energetic recycling. The biological treatment as disposal option for biodegradable polymer products is controversially discussed (e. g. aspects of impurity, fertiliser effects and energy balance) and seems to be useful for niche products only. Considering the future sustainable disposal of biopolymer products a more intensive discussion between producer, disposer and recycler is preferable.

1. INTRODUCTION

1.1 Background

In the context of the discussion about limited fossil resources and climate change products made of bio-based polymers have been developed to an alternative to conventional polymers. Nowadays, products from bio-based polymers are used in several different fields, e. g. in form of films, bags or bottles as packaging materials, for catering products, everyday objects/commodities, compostable garbage bags as well as mulch films for agriculture application.

In Germany, the recycling and disposal of products from bio-based polymers follows the traditional ways together with all the other waste. However, some bio-based polymer products may cause difficulties in these established paths, pointed out the following.

The term bio-based polymers comprises a variety of polymeric materials. Therefore, a differentiated consideration is necessary regarding to the polymer species respectively types in the product as well as recycling and disposal paths.

1.2 Research objectives

Against the background of supporting the implementation of bio-based polymers into the market the Agency for Renewable Resources (Fachagentur Nachwachsende Rohstoffe e. V. – FNR) assigned the KNOTEN WEIMAR International Transfer Centre Environmental Technology GmbH identifying difficulties in the recycling and/or disposal process of products made of bio-based polymers and creating recommendations to support these products. The Institute of Lightweight Structures at the Chemnitz University of Technology and the law office AWKD Werner & Kollegen support the project as subcontractors on technical and legal aspects.

This paper is based on a literature review, colloquia and dialogues with different involved parties, e. g. disposers and recyclers.

2. GENERAL ASPECTS OF BIO-BASED POLYMERS

2.1 Definition and raw material

In the present paper the focus is on bio-based polymers and resulting products. Bio-based polymers belong to the biopolymer materials. The term biopolymer is a broader term and currently biopolymer as a material - bioplastic - is generally characterised by at least one of the following properties [DIN SPEC 2010, Endres and Siebert-Raths 2009]:

- it composes entirely or partly of renewable raw material and/or
- it is biodegradable

Thus, biopolymer materials can be subdivided in following groups:

- non-biodegradable biopolymers, which compose entirely or partly of renewable raw material (blends, compounds),
- biodegradable biopolymers, which compose entirely or partly of renewable raw material (blends, compounds)
- biodegradable biopolymers, which are produced from petroleum sources

The term biopolymer and therewith its connected products is not precisely defined up to today. According to DIN-Technical report [DIN SPEC 2010] there is a further need of standardisation, because the current definition may lead to misleading information and confusion by the end consumers.

Natural-fibre reinforced plastic composites – a material formed by a polymer matrix (resin) and a reinforcement of natural fibres – are an important group of (non-biodegradable) bio-based polymers. Wood-plastic-composites (WPC) and composites of hemp or flax fibre and polymers as polyethylene are ranked to this group.

FIGURE 1 classifies the different biopolymer groups according to their origin and biodegradability on the basis of the present definitions. Thereby, this paper focuses on the groups (I) and (II) and associated products. The so-called biodegradable materials are described by the groups (II) and (IV), but the ones in group (II) are considered only. Subsequently the used term bio-based polymer refers to the groups (I) and (II).

Nowadays, sugar as well as starch and cellulose as polymer from sugar are the primary feedstock for bio-based polymers. Corn, wheat, sugar beet, potatoes, plant oil and wood for cellulose extraction are the main suppliers for these raw materials. Against the background of competition with food the utilisation of lignocellulose is introduced, whereas lignocellulose is converted into sugar [Carus and Raschka 2011].

The considered bio-based polymers can be differentiated into non-biodegradable and biodegradable ones, see Figure 1. In case of the non-biodegradable bio-based polymers, e. g. Bio-PE, the exchange of the resource base is in the foreground. Renewable raw materials should substitute petrochemical depletable resources and maintain the established synthesis paths. The properties of these bio-based polymers are comparable with the conventional polymers.

This approach is known as drop-in-solution and focuses on the safe long-term availability of resources. [Endres and Siebert-Raths 2009]

Regarding the biodegradable bio-based biopolymers, new biopolymers, like polylactide (PLA), polyhydroxyalkanoates, cellulose and starch based biopolymers have been established. [Beier, 2009]

Additionally, there are several copolymers based on the above mentioned biopolymer groups. These biopolymers are mixed up to blends and compounds. Furthermore, a wide range of biopolymers contains additives and auxiliary materials to achieve specific properties. Consequently, the biodegradability and the amount of bio-based material in a product decrease through the usage of non-biodegradable and/or non-bio-based polymers as additive or material component. In the context of the bio-based raw material proportion, there is no regulation in Europe. So, it may happen that the bio-based raw material proportion in a product is below the petrochemical one and supposed biodegradable biopolymer-blends are non-biodegradable. [Endres and Siebert-Raths 2009; DIN SPEC 2010]

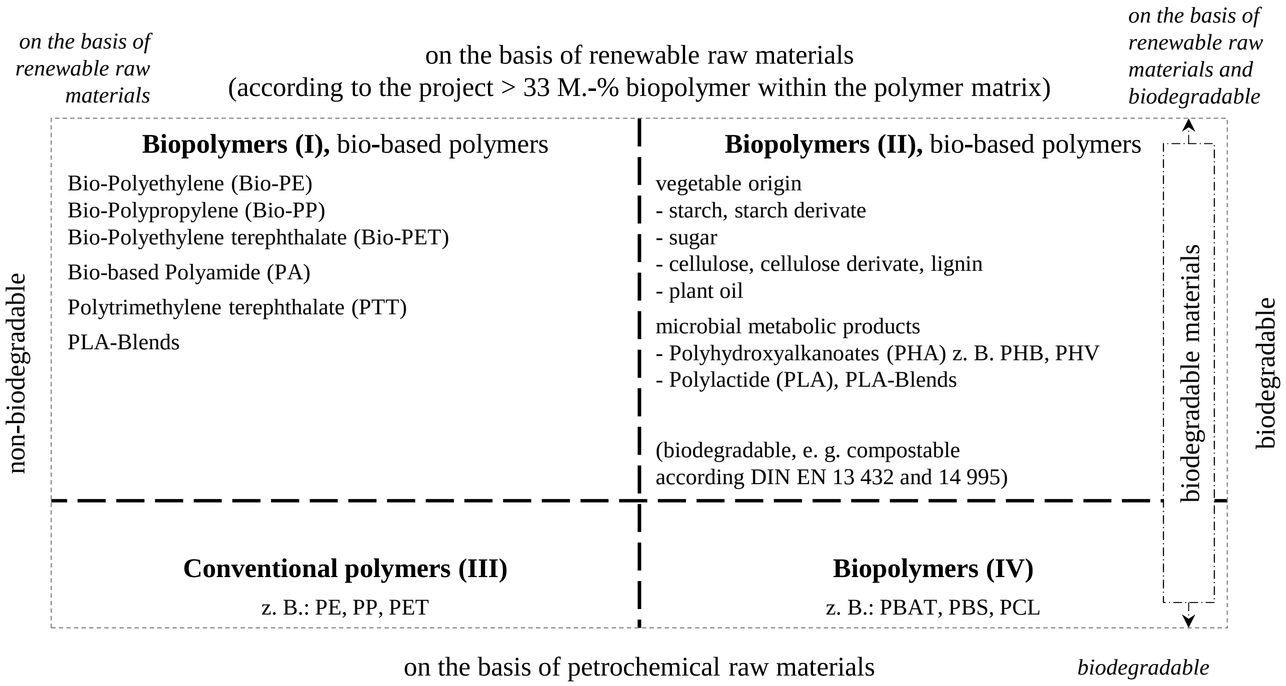

FIGURE 1 **Classification of the different biopolymer groups and conventional polymers [modified according Endres and Siebert-Raths 2009, european bioplastics 2012]**

2.2 Quantities and application

Subsequent TABLE 1 contains data about the estimated world-wide production capacities of different biopolymers for the year 2011 and as forecast for the year 2015. In the group of the bio-based non-biodegradable polymers, Bio-PE followed by Bio-PET are the biopolymers with the highest capacity. On side of the bio-based biodegradable polymers, biodegradable starch blends, polylactide and polyhydroxyalkanoates are the leading exponents. The world-wide production capacity in 2011 was estimated to 1.13 Mio Mg and is forecasted to 2.56 Mio Mg in 2015 considering bio-based polymers only.

Nevertheless, it is hard to estimate the quantity of products and as a result the waste quantity at a certain time period from recycling and disposal perspective, due to the variety of previous mentioned possibilities of composition and forms of application.

TABLE 1 **Estimated world-wide production capacity of different biopolymers [Bengs 2011]**

Biopolymer material	**Production capacity in 2011**		**Production capacity in 2015**	
	Quantity [Mg]	**Per cent [%]**	**Quantity [Mg]**	**Per cent [%]**
Bio-PUR	1,404	0.10	2,870	0.10
Bio-TPE	2,808	0.20	2,870	0.10
Bio-PC	281	0.02	20,087	0.70
Bio-PP	0	0	28,695	1.00
Bio-PA	36,499	2.60	74,607	2.60
Bio-PVC	119,323	8.50	120,519	4.20
Bio-PET	0	0	200,865	7.00
Bio-PE	199,340	14.20	1,195,434	41.66
PLA	231,627	16.50	295,559	10.30
PHA	102,478	7.30	152,084	5.30
PLA-Blends	2,808	0.20	2,087	0.70
Durable Starch Blends	14,038	1.00	14,348	0.50
Biodegradable Starch Blends	378,746	26.98	378,774	13.20
Regenerated Cellulose	36,499	2.60	37,304	1.30
Cellulose-Derivatives	8,423	0.60	11,478	0.40
PVAL	145,996	10.40	169,301	5.90
Biodegradable Polyesters	122,131	8.70	143,475	5.00
PCL	1,404	0.10	1,148	0.04
Total	**1,403,803**	**100.00**	**2,869,500**	**100.00**

In 2010, the world-wide plastic production was approx. 265 million Mg. The estimated world-wide production capacity of all biopolymers together was around 0.5% of it using the numbers from 2011. It would increase to approx. 1.0% in 2015, if the total plastic production remains constant. A large portion of the world's plastic production ends up in packaging – as short-life products - with about 40%. In Germany, this portion is around 35%. [PlasticEurope 2010, 2011]

The application of bio-based polymers is versatile and overlaps with conventional polymers. Bio-based polymers - considering also natural-fibre reinforced biopolymer composites – are used in following applications [Beier 2009, Schmitz 2010]:

- biodegradable bio-waste bags
- mulch films for agriculture application
- textiles
- packaging, like:
  - vegetable, fruit, meet and fast-food bowls
  - drinking cups and soft drink, juice and water bottles
  - plastic bags
  - trays
  - loose-fill-chips
- everyday objects, commodities and durables, like:
  - PC-cases, Pens and mobile phone cases
  - construction material
  - furniture and vehicles components

An overview of product possibilities and applied products from biopolymers is given in the biopolymer database through *www.materialdatacenter.com/bo/main*, which is the result of a research project between M-Base and the University of Applied Science Hannover financed by the Agency for Renewable Resources (FNR). The database gives information among others to the branch of applied biopolymers and biopolymer material.

Familiar examples of a non-biodegradable biopolymer product are the PET-bottle of a soft drink producer, which should be based in future of Bio-PET and yogurt cups or cosmetic bottle from polylactide.

Biodegradable plastic bags on the basis of polylactide or starch, e. g. for bio-waste are exponents for products made of biodegradable bio-based polymers. As mentioned above, the portion of bio-based raw material can vary and the products are not necessarily a hundred per cent bio-based one.

3. RECYCLING AND DISPOSAL OPTIONS

The variety of applications shows that bio-based polymers products are available in a lot of daily concerns. Thus, these products end up also in all different disposal and waste streams, e. g. packing waste, bio-waste, end-of-life vehicles and electronic waste, bulk waste and residues waste, as indicated in Figure 2. In the present paper, post-consumer biopolymer waste is of interest and products made of non-biodegradable and biodegradable bio-based polymer are considered separately.

3.1 Packaging waste and material recycling

In 2009 packaging waste represents with 60.9% the largest portion of the post-consumer plastic waste. It is followed by construction industry with 8.1% and automobile industry with 4.9 % [PlasticEurope 2010]. Consequently, products made of bio-based polymers will be found primarily in the packaging waste stream.

The disposal of packaging waste is regulated in the German packaging ordinance. The ordinance sets limits among others regarding the material recycling. The material recycling of plastic waste should be 22.5 M.-% considering total waste amount and 36 M.-% for end-consumer waste amount [VerpackV 2010]. In Germany, these targets are covered primarily by the material recycling of the standard-polymers in packaging PE, PP and PET. These polymers form the largest mass portion in the packaging sector. Biopolymer products are sorted to the mixed plastics and are feedstock or energetic recycled. The sorting process starts generally at the disposer level with collection and dry sorting, see 2nd broken line box in Figure 2. The standard-polymers are sorted positively and sold to the recycling industry, where further dry- and wet-mechanical preparation steps take place to get a single-origin polymer – recyclate – of high quality for material recycling, see 3rd broken line box in Figure 3.

The recyclate forms a secondary resource. 41% of plastic waste undergone the material recycling in Germany in 2009. The feedstock recycling plays a minor role in plastic recycling with 1% of plastic waste. 55% of plastic waste was utilised energetically and 3% were disposed and landfilled. [PlasticEurope 2010]

3.2 Bio-waste and biological treatment

Biodegradable biopolymer products as part of biodegradable materials have an exceptional. These biopolymer products can be treated aerobic in a technical composting plant, if they are certified according DIN EN 13432 and/or 14995. It this case a feedstock recycling would be done, see Figure 2. However, the bio-waste bin is not established area-wide and thus, a disposal via the so-called bio-bin is not everywhere ensured. According to Beier (2009) organisations like the Bundesgütegemeinschaft Kompost e. V. consider biodegradable polymer products as impurity and refuse its treatment via the bio-bin. A lot of communities followed this position and ban biodegradable polymer products in their statutes as input material for the bio-bin. But shouldn’t there be a distinction between niche products like biodegradable bio-waste bags and packaging?

Currently, many composting plants have installed digestions plants and thus, an upstream anaerobic process step to utilize the energy of the bio-waste, see 3rd broken line box in Figure 2. The driven force is the subsidy of renewable energy. However, a certification doesn’t exist yet for the treatment of biodegradable polymers in digestion plants.

3.3 End-of-life vehicles and electronic, bulk and residues waste

The legislations of end-of-life vehicles and electronic waste don’t include quotas regarding the material recycling of specific materials regarding the treatment of these fractions. Thus and due to the small portion of bio-based polymer products, these are not in focus. The quotas for reutilisation, feedstock and energetic recycling are covered by the other materials so far. An increasing application of biopolymers may change this focus.

Bulk and residues waste undergoes a mechanical-biological and thermal treatment before disposal in Germany since 2005. Materials with a high calorific value are sorted out depending on the plant and used for energy recovery.

4. OPERTUNITIES AND LIMITATIONS OF BIO-BASED POLYMER RECYCLING

4.1 Material recycling

The different bio-based polymers have to be considered separately in case of material recycling, e. g. Bio-PE and PLA. Whereas Bio-PE is sorted out with PE in the plastic fraction for material recycling, PLA is put into the mixed plastics.

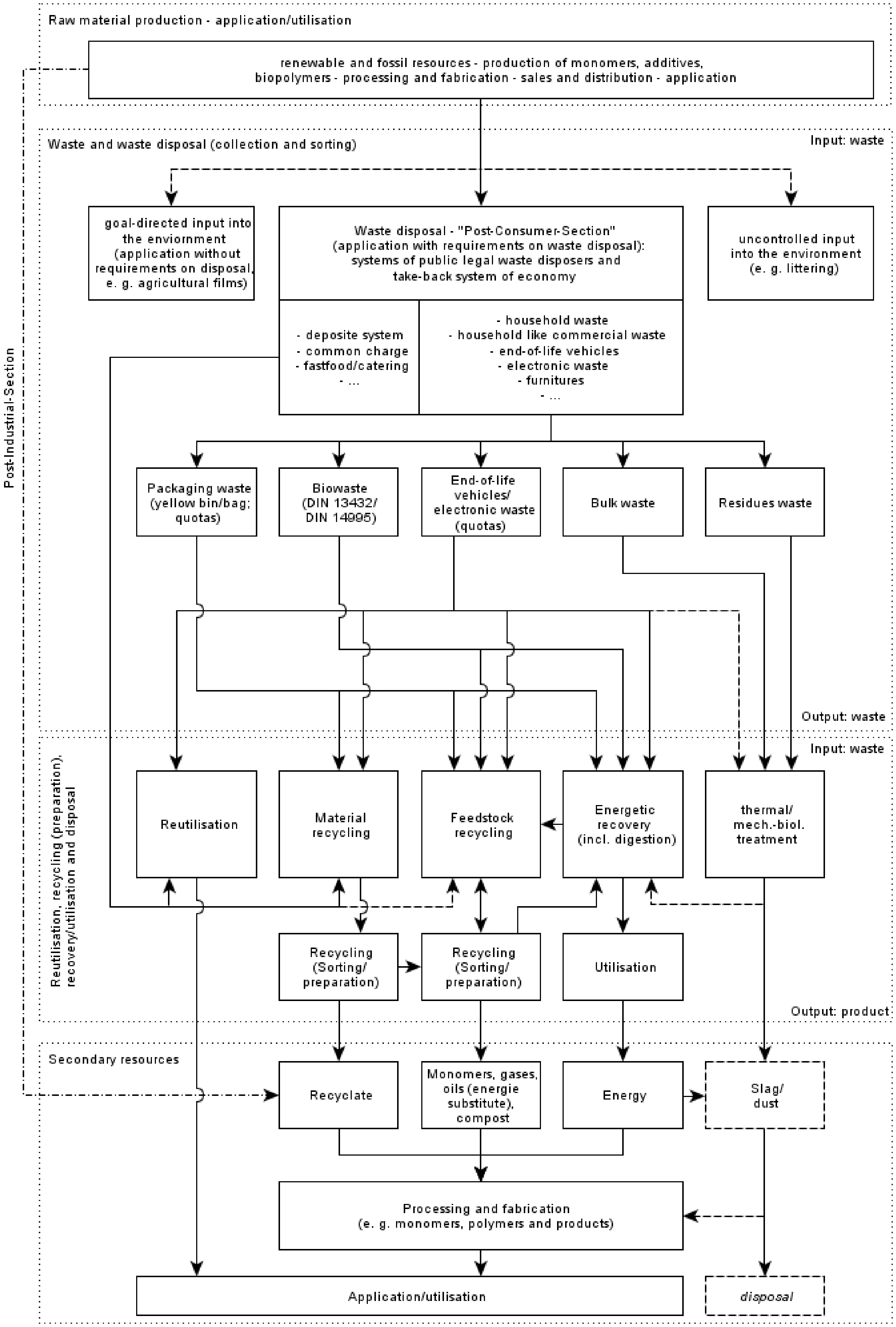

FIGURE 2 **Current recycling and disposal options of waste products made of bio-based polymers**

So far, critical quantities of biopolymer products are not available for a material recycling of biopolymers like PLA due to the geographical distribution for instance. According to Cornell (2007) a post-consumer plastic recycling facility needs to process at least 5,000 Mg/a, better 20,000 Mg/a. In case of PLA, experts recommend one type of one origin, if a material recycling of PLA is intended. The above mentioned throughput rates are estimations, because they depends on several factors like price of the raw material or recycling options. According to Probst [2012] there is no material recycling of post-consumer PLA-waste for instance at present time.

Another aspect is, expert mention that some biopolymers may harm the material recycling process of standard-polymers and reduce the quality of the recyclates, because of its physical properties and chemical composition. Especially PLA, starch and cellulose-based biopolymers are ranked to these. Bio-PE indicates no advantages in the material recycling process of conventional PE up to know. However, these aspects have to be evaluated. [Probst, 2012]

4.2 Biological treatment

Biodegradable bio-based polymer products should be considered separately as well. One is open-minded about niche products as mulch films for agriculture application, biodegradable bio-waste bags or catering products if there is an one recycling or disposal path. [Beier 2009, Probst 2012]

The application of biodegradable bio-based polymers in the packaging sector and its disposal with the bio-waste is considered critically and non-sustainable. The degradation under aerobic conditions – in composting plants – doesn't result in a fertiliser effect. On the other side, additional energy has to be put into the disposal process to treat the material within the system. [Niedersächsiches Umweltministerium 1999; Schmitz 2010]

The disposal together with the bio-waste may result in an increasing miss-sorting on consumer level. Thus, the sorting demand but also impurity amount in the compost increases. Beier (2009) mentions, that the German Federal Environment Agency (Umweltbundesamt) sees perceptively the composting of biodegradable polymers as no useful form of waste disposal.

The treatment of biodegradable polymers in digestions plants is subject to the youngest research. First results show that polylactide as one of the most often biopolymer is hardly metabolisable. Kitzler (2012) reports biogas yields of PLA between 6 and 11 l/kg oDM. The biogas yield of energy crops is in a range of 500-600 l/kg oDM. Thus, the biogas yield of PLA is between 1% and 2% compared to energy crops. Starch-based biopolymers and starch-blends reach higher biogas yield of ca. 200 l/kg oDM, one-third compared to energy crops. Nevertheless, the benefit has to be examined in detail and further research is necessary. It becomes obviously also, that a differentiated consideration of the different biodegradable biopolymers is necessary. Currently, the Professorship of Biotechnology in Resources Management at the Bauhaus-University Weimar carries out research in this field among others.

4.3 Energy recovery

The energy recovery of products made of bio-based polymers is from interest, because currently it can be supposed that the largest portion of these products goes with the packaging waste stream and the sorted out stream of biological treatment options towards an energy recovery or thermal treatment.

The heating value of bio-based polymers is similar to polymers on basis of petrochemical raw materials, because it depends on the composition of the material and not on its origin. The emissions of the combustion of biopolymers depend on the combustion temperature and composition of the fuel, thus it is similar to conventional polymers. Additives and auxiliary materials have to be considered critically against harmful substances. At this point further research is necessary. [Endres and Siebert-Raths 2009]

4.4 Life cycle assessments

In the last years life cycle assessments (LCA) where carried out to show the ecological advantage of biopolymer products, especially of biodegradable biopolymer products. The results show, that biopolymer products go easy on fossil resources and have a positive carbon dioxide balance. However, in all other impact categories biopolymers are partly worse compared to conventional polymers. [Beier 2009, Kauertz et al. 2012, nova-Institut 2012]

Also in this point a differentiated consideration of biopolymer types has to be done. Beier (2009) considers biodegradable bio-based polymers, Kauertz et al. (2012) and nova-Institut (2012) focus on bio-based polymers.

According Beier (2009) the results of the LCAs show, biopolymer products predominant to conventional polymer products if among others the raw materials are from sustainable agriculture, residues materials from agriculture and food are used as feedstock, a high-quality recycling and energy recovery takes place at the end of the product lifetime. Bio-based polymer products should be developed, which are non-biodegradable – if this isn't a product demand – durable and recyclable.

5. CONCLUSIONS

Bio-based polymer products are more and more entering our daily life products – durable or non-durable. Finally, these bio-based polymer products become waste and end up in established waste streams. The biopolymer products may cause difficulties in the different waste streams being partially unsuitable for biopolymer products. At this point, a differentiated consideration of the biopolymer product variety only leads to useful waste disposal options. So far, material recycling of post-consumer products made of bio-based polymers is not known. The current missing critical mass for a material recycling is one reason. Today, most biopolymer products end up in the energetic recycling. The biological treatment as disposal option for biodegradable polymer products is controversially discussed (e. g. aspects of impurity, fertiliser effects and energy balance) and seems to be useful for niche products only. Considering the future sustainable disposal of biopolymer products a more intensive discussion between producer, disposer and recycler is preferable.

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(241) BENEFITS AND CHALLENGES OF COMPOSTABLE PLASTICS: LESSONS FROM SAN FRANCISCO AND BEYOND IN THE USA

Contact J. Macy, City and County of San Francisco, USA

EXECTIVE SUMMARY

This paper provides an overview of benefits and challenges of compostable plastics based on lessons learned from San Francisco's composting program and elsewhere in the USA. Compostable bags and compostable food service ware can offer real benefits, including facilitating increased diversion of food scraps and reducing non-compostable plastic contamination of compost. However, compostable plastics present real challenges and uncertainty for consumers and end-of-life processors. These benefits and challenges are presented in the context of San Francisco’s food composting program and zero waste policies and other experiences in the USA.

The City and County of San Francisco has implemented the largest city food composting collection program in the USA over more than a dozen years. It has also implemented zero waste policies that have helped promote the use compostable bags and compostable food service ware to move the city toward its zero waste goal. These policies and programs have been recognized internationally as a model and this paper's author, Jack Macy, is the city’s staff lead of these efforts. The paper highlights San Francisco's policies for zero waste, including requiring compostable food service ware, and banning of non-compostable plastic bags. It describes the city's comprehensive residential and commercial composting program that provides an opportunity for beneficial use of compostable plastics.

The paper then summarizes challenges in the use of compostable plastics and responses taken to address these challenges. These include San Francisco's experience and response, such as with identification and labeling of compostable products, and subsequent compostable product labeling legislation adopted in California. The paper shares an example of the Cedar Grove Composting facility's own testing, acceptance and labeling program in the state of Washington. Finally, the paper presents broader efforts by the US Composting Council, with the involvement of the author, to address concerns through the action of a Compostable Plastics Task Force with a national dialogue of working groups on labeling and identification, consumer education, legislation and enforcement, composting operational impacts, and compostable and organics certification standards.

1. SAN FRANCISCO COMPOSTING PROGRAM BACKGROUND

The City and County of San Francisco (City) started its composting collection program in partnership with its exclusive permitted private hauler, Norcal Waste Systems (now called Recology), in 1996 as a commercial produce only program utilizing a nearby regional composting facility. Participating commercial establishments were provided training to separate vegetative food scraps into provided containers that were labeled and color coded green for door to door (or curbside) collection. No plastics were allowed. Participation was voluntary, but a financial rate discount was provided as an incentive. The commercial food collection was expanded systematically from wholesale produce to retail supermarkets to smaller retail produce and food service establishments, based on the available permitted composting capacity. Residential door to door (curbside) collection of vegetative food combined with yard trimmings started as a test in selected collection routes in 1997.

Residents were provided green kitchen pails and initially a set of cellophane lined paper bags to assist them in separate collection of vegetative food scraps, and were given the option to purchase more cellophane lined paper bags. The City chose not to provide an on-going supply of collection liner bags to program participants due to cost concerns and a perspective that using bags, while helpful, was not necessary. Subsequently by 1999, residents being added to the program were provided sample compostable plastic bags as described in the next section of this paper.

In 1998, a Recology regional composting facility (now called Jepson Prairie Organics) was permitted to compost all types of food scraps, including meat, and other compostables using the Ag Bag (plastic bag enclosed) aerated composting system. Composting collection in San Francisco then expanded to all food scraps, compostable food packaging and food soiled paper co-collected with yard trimmings. In 2000, after years of testing various container, truck and material configurations, Recology commenced a citywide rollout of a 3 stream curbside (or door to door) collection program for the residential and commercial sectors consisting of collecting: all compostables in green bins, all recyclables commingled in blue bins and remaining trash to landfill in black bins. All properties were provided the color coded bins, primarily wheeled carts (e.g., 32 gallon or 120 liter size for typical single family household) or larger bins as needed for commercial customers. Commercial customers were provided on-site assistance with program set-up, internal sorting containers, signage and multi-lingual staff training. Residents have weekly collection and pay Recology for their service based on their black cart size for the material going to landfill and commercial customers pay Recology for all bin types with a % discount based on the total volume being diverted to composting and recycling divided by total service volume of all three material streams.

After implementing citywide voluntarily composting and recycling collection, the City mandated by law in 2009 that everyone, in all residential and commercial sectors, must participate and source separate their compostables and recyclables from remaining trash. Since then the amount of compostables collected in the City, mostly of food scraps, increased by over 50% to more than 600 tons per day.

The compost produced from food scraps, plants and other compostables is found to be especially nutrient rich. It is sold in bulk to growing numbers of local farmers, vineyards, landscapers and golf courses who see increased soil fertility and healthy plant growth. Organic food and wine produced with this compost is brought back to the City and sold in farmers markets, stores and restaurants, thereby closing a nutrient loop from table to table.

2. BENEFITS OF COMPOSTABLE PLASTICS IN SAN FRANCISCO

Non-compostable plastic bags or liners are not allowed in composting collection bins in order to minimize the amount of non-compostables that have to be sorted or screened out at the composting facility to minimize plastic contamination. Compostable plastic bags or liners provided a valuable opportunity to reduce the amount non-compostable plastic contamination. Compostable plastic bags, such as the BioBag made with MaterBi resin from Novamont, were successfully tested in the late 1990’s food scraps collection in the residential and commercial sectors and for composting at Jepson Prairie Organics composting facility. These bags fit better in the kitchen pails provided to residents than did the cellophane lined paper bags used in the beginning of the residential food collection program. They also come in larger sizes for lining sorting bins used in commercial kitchens. Residents were provided a sample set of the BioBags with their new kitchen pail container when they were added to the collection program. The City spurred local retail markets to carry boxes of BioBags for kitchen pails for purchase by residents. Businesses ordered larger size bags on-line or by phone. Program participants who wanted to continue to put their food scraps into plastic bags for collection, as many were used to doing before they had composting collection, had the option to buy compostable bags. Residents and businesses reported that use of compostable liners helped boost participation by reducing odor and mess and by keeping collection containers cleaner. Bags have been especially useful for apartment building dwellers carrying their bags of post-consumer scraps down many flights to ground level collection. For those who didn't want to pay for compostable bags, acceptable alternatives included using paper bags, newspaper and paper cartons to contain food, or use no liner and washing the container. The co-collection of yard or garden trimmings with food scraps in the same composting collection bin also helped reduce the need of plastic liners.

In addition to compostable plastic bags, the City saw the benefits of using compostable plastic food ware such as cups, containers and cutlery to help facilitate the capture of left-over food that was served with disposable food ware. The sector where the City first tested and promoted the use of compostable food ware was in special events, such as outdoor street festivals and concerts. San Francisco has hundreds of these events during the year that are attended by hundreds of thousands of people who are served food and drink by a wide variety of vendors, which results in many tons of discards. These events were among the most difficult to divert waste from landfill until the City worked with them to use compostable food ware and to implement effective source separation stations for their patrons. These efforts to use compostable plastics at events were started in the early 2000's, at the time when most disposable food ware plastics, such as clear cups, containers and cutlery, were not accepted for recycling due to lack of viable markets. Getting the public to successfully remove non-compostable food ware from food scraps was a major obstacle to reducing composting contamination and in maximizing diversion of food from landfill.

By using compostable food ware and getting patrons to sort their compostables and any recyclables and remaining trash at monitored collection bin stations (using compostable bags for collecting compostables), the City found that events could achieve landfill diversion rates of over 50%, often up to 75% or more, and even over 90% in some cases. Events discovered the cache of marketing themselves as "zero waste" events. With these diversion rates, events realized significant savings in collection costs under San Francisco's incentivized variable rate structure. Those savings helped offset potential additional costs incurred when purchasing compostable food ware. After successful use by events, the use of compostable plastic food ware expanded into the restaurant take out sector and into stores selling food ware to the public. Restaurants using compostable food ware could also reduce contamination from customers sorting their compostables and increase their diversion and cost savings.

3. CHALLENGE OF COMPOSTABLE PLASTICS IN SAN FRANCISCO

The primary challenges the City - along with the public, food establishments, composting collector and processor - have addressed with compostable plastics over the last 15 years are two-fold. First, is ensuring that a product is compostable and will fully breakdown in the composting process; and second, is identifying and distinguishing compostable plastics from non-compostable plastics that may look identical without special labeling.

Over time, the City has seen an increasing number of bags and food ware products being marketed as compostable, either explicitly or implicitly, with labels such as "biodegradable", “degradable” or "back to nature". Upon investigation, the City found many of these products not to be compostable. There has been much confusion among wholesalers, retailers and users of products with often misleading or contradictory claims. For example, a distributor may claim a product is compostable based solely on biodegradation test data, yet not realize that the product didn’t pass the pass/fail threshold required or that there are two other pass/fail tests required by a complete compostability standard, such as ASTM D6400. Addressing this confusion and trying to determine whether all required tests were done appropriately and passed for a given product can be difficult and time consuming.

Given this challenge of ensuring compostability, the City chose to require that in order for a product to be considered a compostable plastic it must receive independent 3rd party certification that it has meet the ASTM D6400 Standard Specification for Compostable Plastics or a similar standard, such as EN 13234 standard. In North America it is only the Biodegradable Product Institute (BPI) that conducts an independent 3rd party certification for products that meet the ASTM D6400. If BPI verifies that a product meets the standard it then licenses the product to use the BPI logo. The experience of Jepson Prairie Organics since the late 1990’s in processing a variety of BPI certified compostable products and others not certified has confirmed the validity for the City requiring the ASTM D6400 standard for compostability.

Since the 1990’s, the City has been communicating with compostable resin producers, product manufacturers and distributors about the importance of labeling compostable plastics so they can be easily identified and distinguished from non-compostable plastics by all players in the composting program. Bags were the easiest products to label and the City required that compostable plastic bags be labeled with the word “compostable” in green color, or within a green color band around the bag, in one inch or larger easy to read sized print and display the 3rd party BPI certification logo for meeting the ASTM D6400. The City choose the green color for labeling and identifying compostable products to fit with the City’s green color coded system for its composting collection bin and associated signage.

Following compostable bags, the next compostable product type the City was able to get industry to effectively label was compostable plastic cups. The City worked with manufacturers and distributors in the early 2000’s to develop a green band design that went around the lower half of a cup to easily identify the cup as compostable. While labeling with a green band or text around the cup has common for years, the City still has to be vigilant in getting unlabeled cups out of the market. The City has had much more difficulty in getting other compostable products, such as clamshell containers or cutlery, to be effectively labeled for easy identification due to the challenges of printing in color on those types of products. While a embossing the word compostable on top of a container or cutlery may help a users, it is still difficult for collectors and composters to identify the product in a large pile or going across a sorting line.

4. SAN FRANCISCO AND STATE LAWS IMPACTING COMPOSTABLE PLASTICS

In 2002, after meeting the State of California's mandate to divert 50% of its waste from landfill, the City adopted a goal to achieve 75% diversion by 2010 and zero waste to landfill by 2020. This zero waste policy included the principles of promoting the "highest and best use" of discarded materials and requiring consumer and producer responsibility for the management of discards. This policy provided a framework that supported subsequent policies to address compostable plastics and to move the City toward its zero waste goal.

These policies included the 2007 Food Service Waste Reduction Ordinance. This Ordinance bans the use of expanded polystyrene (aka Styrofoam) food ware for serving food prepared by the City's 4500+ food establishments. It also requires any disposable food service ware used to serve prepared food be compostable or recyclable as accepted in the City's collection programs. Acceptable compostable food service ware includes not only paper and molded fiber containers but also compostable plastics meeting the ASTM D6400 standard as certified by BPI. The City lists on its website acceptable compostable or recyclable food ware products and vendors distributing those products. For acceptable compostable plastics, the City requires labeling compostable products with "Compostable" (printed or on a sticker) preferably in green color print or within a green band. Since 2007, the City visited more than 4000 food establishments and has achieved nearly 100% compliance.

The 2007 Plastic Bag Reduction Ordinance bans non-compostable single use plastic checkout bags at supermarkets and pharmacy chains. Traditional plastic bags are the primary and most costly contaminant in both composting and recycling collection. Bags are not compatible with the recycling screening technology used to separate a paper or fiber material from the glass, metal and plastic material at the recycling sorting facility. The impact of plastic bags on litter in the streets and in the regional environment, especially the Bay and ocean, were important factors in getting the ban passed.

The bag ordinance limits stores to being able to provide as checkout bags: 1) paper bags (with a minimum of 40% post-consumer recycled content) as they can be recycled in the curbside (door to door) collection or used to help collect food scraps for composting; 2) certified compostable plastic bags as those could be used for food scraps collection; or 3) reusable bags made for many uses. Nearly all stores subject to the ordinance now provided paper bags at checkout, since the cost of compostable plastic checkout bags is prohibitive.

Following San Francisco's bag ordinance, more than 40 other cities and counties in California have passed plastic bag bans, including banning compostable plastic bags.

These jurisdictions either did not have a food scraps collection or concerns over environmental impact of litter trumped perceived benefits of compostable plastics. Subsequent bag legislation in the US was improved on by requiring stores to charge, usually at least 10 cents, for each checkout bag they give out. The California legislature nearly passed statewide legislation banning single use plastic bags and requiring a charge on paper bags, but a strong lobbying effort by the plastics industry stopped it.

In February 2012, San Francisco passed legislation expanding its non-compostable plastic bag ban to all stores (in October 2012) and restaurants (in October 2013), and required them to charge 10 cents for any checkout bag they provide. The City now has the most comprehensive bag legislation by including all restaurants in addition to all stores of all size. By allowing the use of compostable plastic bags, the City was able to address concerns of the restaurant sector for carrying take-out food that may leak into a bag and also be hot. The City found that three different compostable carryout bags tested were able to hold a bag full of hot food containers and liquid at up to 180 degrees for 1/2 hour without any failure.

The City's efforts paved the way for statewide legislation. In 2009, a state law (AB 1972) passed requiring that any bag labeled "compostable" must meet the ASTM D6400 standard and that no bag can be labeled "biodegradable" or "degradable", since there is no standard for those terms. Subsequent state legislation (SB 228) passed in 2010 specifies how compostable bags must be labeled, such as printing the word compostable in green color and in one inch letter size and displaying a 3rd party certification (e.g., BPI) logo for meeting the ASTM D6400 standard. State legislation (SB 567) passed in 2011 expands compostable labeling requirement to all plastic products, so none of them can be labeled "biodegradable" or "degradable" and any plastic labeled compostable must be 3rd party certified as meeting ASTM D6400. Finally, state law (AB 2071) passed in 2011 allows a city, county or state to impose fines or penalties in enforcing the above laws. The State Attorney General has already investigated claims and taken action against a few companies for improper labeling.

5. CEDAR GROVE COMPOSTING TESTING, ACCEPTANCE AND LABELING

A different approach from that of San Francisco of accepting compostable plastics in a food composting program is that of Cedar Grove Composting in Everett, Washington serving the greater Seattle, WA area. This facility found with their composting process, utilizing the reusable GORE® Cover aerated composting system that operates up to temperatures of 170º F (~77º C) or more, that not all products that meet the ASTM D6400 standard would adequately breakdown. To ensure that products would fully compost in their system, Cedar Grove Composting instituted a formal program of technical review and field composting testing for compostable products to determine if they would accept a product as compostable. In order for a product to qualify for testing at Cedar Grove it first has to meet ASTM D6400 or EN 13432, or ASTM D6868 for poly-coated paper products. Product vendors have to submit BPI certification or adequate documentation of meeting those standards for their product to be tested. Only those products that were successfully tested are accepted in the composting collection programs served by Cedar Grove Composting. Cedar Grove lists the acceptable compostable products with their picture and vendor item number on their website. While they do not have specific labeling requirements for commercial customers, they do require that products used in residential composting collection be labeled with the Cedar Grove logo in brown color and also with brown stripes or in all brown color for cutlery.

6. NATIONAL DIALOGUE ON CHALLENGES VIA US COMPOSTING COUNCIL’S COMPOSTABLE PLASTICS TASK FORCE

he author worked with the California Organics Recycling Council (CORC) to address concerns of composters and jurisdictions implementing composting programs regarding compostable plastics. CORC organized a symposium on compostable plastics at the US Composting Council (USCC) Annual Conference in January 2011 in San Jose, CA.

The USCC then created a Compostable Plastics Task Force (CPTF) with working groups to engage a national stakeholder dialogue to address specific concerns so that compostable plastics can play a beneficial role in increasing composting. The challenges that the CPTF working groups sought to address are:

- Compostability Standards and Certification
- Labeling and Identification
- Legislation and Enforcement
- Consumer Education
- National Organic Program Standards and Operational Impacts

The CPTF task forces identified team leads, including the author for identification and labeling, and invited stakeholder participants for conference calls that were held throughout 2011. These groups presented updates at the 2012 Compostable Plastics Symposium at the USCC Annual Conference in Austin, Texas in January 2012, which included the following progress:

ASTM Standards: Some composting facilities, such as Cedar Grove Composting, find that not all products that pass current ASTM standards fully disintegrated in their composting process. As a result, there is an effort to revise standards to allow variations between composting facilities and product types. This group has collected data on operational parameters and worked with an ASTM subcommittee to recommend an additional standard be accepted for the disintegration test portion of the D6400 standard. Group members hope to come to agreement on temperature regimes and vessel sizes in order to make the standard more applicable.

Labeling and Identification: This group has focused on the challenge of needing to label and identify compostable plastics. Issues of labeling inconsistency, lack of clear definitions or use of standards, competing international standards cause confusion. Labeling standards need to be cost sensitive and include the brand owners as stakeholders. Composters as well as material processing facilities that recover recyclables need easily identifiable labeling to facilitate sorting, and consumers need easily recognizable labels for appropriate source separation. This group is developing of labeling guidelines, incorporating recommendations developed by BPI, that meet identification needs of users, collectors and composters to easily distinguish compostable from non-compostable. The group is also working to define the guidelines with language that can be used on packaging to define compostability.

Enforcement/Legislation: This group has been focused on two key objectives: 1) how to stop those "bad actor" companies making misleading claims that their products are biodegradable or compostable but don't meet any existing standards, and 2) protecting and replicating California's laws that establish compostable labeling standards. The group is looking to identify other states that can implement similar laws as in California.

National Organic Program and Operational Impacts: A recent clarification by the USDA National Organics Program (NOP) does not allow compost to be used by certified growers if the feedstock includes compostable plastics, since they are considered a synthetic input. The working group is supporting a petition by the compostable plastics industry to the National Organic Program to allow the use compostable plastics that meet ASTM D6400. The group is also evaluating operational impacts and possible best management practices for handing compostable plastics. Composters that market compost to organic growers and get compostable plastics in their feedstock, such as JPO and others those that compost San Francisco food scraps, are now having to sort out the compostable plastics and compost them in separate piles for non-organic markets.

Consumer Education: Most consumers are confused about compostable plastics. The work group has focused on several key questions including: 1) What are the primary areas of confusion that need to be better explained and articulated? 2) What educational strategy can balance stakeholder goals, be mutually beneficial, transparent, and effective? 3) How to better reach the different target audiences with appropriate education? The group recommends message that composting is local and education needs to focus on what is possible at a local level. The group is evaluating need for clearinghouse website of information on acceptable products in various municipal composting programs.

The above working groups were combined since January 2012 based on overlapping objectives and participants into the following: 1) Operational Impacts and NOP & ASTM Standards, 2) Labeling and Education, and 3) Legislation and Enforcement. These working groups are continuing their dialogue meeting at least quarterly in 2012.

7. CONCLUSION

From San Francisco's experience, maximizing food diversion toward zero waste, it is clear that compostable plastic bags and food ware can be a real benefit in increasing the diversion of food scraps for composting. Compostable plastic bags, especially, provide a big benefit in increasing participation in source separated food collection. The author does not see much potential benefit of compostable plastics for other products, such as bottles or durable products, as they don't increased diversion since recycling infrastructure exists for non-degradable plastic products.

However, while there are real potential benefits to using compostable plastics, there are many challenges to their use that have created barriers for many jurisdictions or composting facilities in allowing the use compostable plastics. This barrier in the acceptance of compostable plastics has resulted in an obstacle for the expansion of food composting programs, especially for post-consumer food scraps. The author finds cause for hope in addressing these various challenges with the increasing push for food composting and the recent national stakeholder dialogue by the US Composting Council Compostable Plastics Task Force reflecting the commitment by many stakeholders to create solutions so that compostable plastics can work for all parties involved in the important mission of increasing the beneficial use of food scraps.

(242) COMPOSTABILITY OF PLASTICS AND PACKAGING: STANDARDS ON BIODEGRADABILITY AND ECOTOXICITY

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EXECUTIVE SUMMARY

The first attempts to define a standard on requirements for plastics and packaging suitable to composting date back to the middle of 90s, when ORCA (Organic Reclamation and Composting Association) in Europe and ISR-ASTM (Institute for Standard Research of ASTM) in the USA, started fundamental preliminary works. Official standard specifications were developed shortly after. ASTM D6400 (Standard Specification for Compostable Plastics) was originally published in 1999 and the European harmonised standard EN 13432 (Packaging. Requirements for packaging recoverable through composting and biodegradation. Test scheme and evaluation criteria for the final acceptance of packaging) the year after. Since 2008 also an international standard (ISO 17088 Specifications for compostable plastics) is available. The three standard specifications basically apply the same testing approach (and requirements) based on: assessment of biodegradability (90% in 180 days), assessment of disintegrability (90% in 90 days), assessment of possible negative effects on the final compost (ecotoxicity testing and content of metals).

After 20 years from the first discussions and more than 10 years of practical application of the standards, we can now try to draw some conclusions, collect the voiced criticisms and restart some further dialogue (similarly to what done in the 90s) to see if and how the current standards can be improved.

Compostable plastics (that comply with the standards) are nowadays applied in specific applications (e.g. bio-waste bags, carrier bags, single use cutlery and tableware, etc.) that are finally delivered together with the bio-waste to composting plants. No negative reports are available to our knowledge. On the other hand, it is interesting to remark that lack of degradation has been found when testing materials that do not comply with the standards, while compostable plastics performed satisfactory.

The biodegradation level (90% in 180 days) has been considered to be too low (why 90%? What about the residual 10%?) or too high (compostable plastics are too fast and composting is a sort of “cold” incineration, fast converting the organic carbon into CO2). The biodegradation rate has been considered too slow (permissive) or too fast (conservative). The ecotoxicity testing has been criticised because carried out after 3 months composting. Somebody wanted it to be repeated at different times, following the biodegradation course. All these criticisms seem not founded enough and are discussed in this paper.

However, in spite of the successful application of the above standards, we note that further reassurance on the environmental safety of bioplastics is progressively requested due to the higher volumes that are expected to be marketed in the near future by the biodegradable plastic industry. In particular the interest is on the substances produced during biodegradation that might reach soil when compost is used as fertilizer. A first attempt to address this concern has been adopted by ISO. A recent revision of ISO 17088 requires that constituents present in amount ranging between 1% and 10% are tested individually for biodegradation. The reason of this further requirement is to show that the 90% biodegradation level (instead of “100%”) does not “hide” a 10% fraction formed by some recalcitrant minor constituents.

In order to provide a further reassurance to the concerned stakeholders, an approach currently under discussion is to focus the attention on the molecules that can be possibly generated during the first phase of degradation, when the polymer, still outside the microbial cells, is depolymerised back to the original monomers. It should be pointed out that since the original polymer must show a 90% biodegradation level, all or most intermediates must be biodegradable, otherwise this limit would not be reached. Therefore, this approach focuses on substances that can be released into the environment in traces and can also be applied to substances other than the monomers, such as additives used to enhance polymer properties and processability.

1 INTRODUCTION

1.1 Background

The first discussion on the compostability criteria for plastics and packaging were developed by the Organic Reclamation and Composting Association (ORCA). The ORCA's Guidelines for the evaluation of feedstock for source separated biowaste composting and biogasification were published in 1994, in Brussels (Lemmes, 1994). Similar discussion and test activities were carried out in the USA, at the ASTM Institute for Standards Research, within the Degradable Polymers Research Program. The purpose was to provide the basis for scientific substantiation of disposability statements for degradable polymeric materials in full scale composting systems. The goal was to determine the behaviour of degradable polymeric materials in real disposal systems, and how that correlates with ASTM and other laboratory tests in order to assure that such materials are safe for disposal and effectively degraded. The work was conducted by the IRS from 1991 through 1996 and it is summarized in the report "ASTM-ISR Degradable Polymeric Materials Program" (ASTM/ISR , 1996).

The work carried out in this period constituted the backbone of the subsequent standard specifications produced by ASTM (ASTM D6400 Standard Specification for Compostable Plastics), by CEN (EN 13432 Packaging. Requirements for packaging recoverable through composting and biodegradation. Test scheme and evaluation criteria for the final acceptance of packaging) and more recently by ISO (ISO 17088 Specifications for compostable plastics). The basic principles of the standard specification are the same and are briefly outlined in the following paragraphs.

1.2 Biodegradability

How can we determine the biodegradability of a plastic? We must apply a laboratory approach. The biodegradability of a polymer is inferred by studying a biodegradation process carried out under controlled, reproducible, feasible conditions. The biodegradation of a polymer is measured and, from the results, the conclusion that the polymer is biodegradable (namely, *it can be biodegraded*) can be drawn.

Biodegradation is the assimilation of the organic carbon present in the plastic by the microorganisms living in the compost, that feed on it. Final outcome of aerobic biodegradation is carbon dioxide and water. This closes the natural cycle of carbon. CO_2 is adsorbed by plants and come back to the atmosphere with the biodegradation. The specific standard test method is the so-called "controlled aerobic composting test" ISO 14855 "Determination of the ultimate aerobic biodegradability and disintegration of plastic materials under controlled composting conditions - Method by analysis of evolved carbon dioxide". The test method measures the evolved CO_2 under conditions similar to the composting environment (solid state, high temperature, proper oxygen and water levels, compost used as an inoculum). The test material is generally powdered and mixed with some mature compost, used as a source of micro-organisms and nutrients, brought to the correct degree of humidity and maintained at a constant temperature of 58°C. From the measurement of the CO_2 produced under these conditions, the degree of conversion (mineralization) of the organic carbon of the material is determined. In parallel, the biodegradation of the reference material, microcrystalline cellulose, is measured. The laboratory test method returns an output (biodegradation level) but itself is not sufficient to reach the conclusion of "biodegradability". This task is accomplished by the standard specifications mentioned above; the biodegradation shall be at least 90% (conversion percentage of the organic carbon into CO_2) or 90% of the level reached by the cellulose in the same time (relative biodegradation), in a maximum time of six months. Cellulose must reach at the end of the test a biodegradation of at least 70%, otherwise the test is invalid. The cellulose is here used as an internal control, to assure that the test environment is active. Being a fully biodegradable material, the mineralization level of cellulose is considered as the maximum mineralization achievable under the test conditions.

It should be remarked that the statement (unfortunately frequently spotted in products placed on the market) "it complies with ISO 14855" is clearly senseless, because no pass levels are set in this standard lab test method.

1.3 Disintegrability

A real composting process must be performed in order to verify that the test material, in its final physical form, is disintegrated within a composting cycle. Samples of the test material are mixed together with fresh biowaste and co-composted at pilot-scale level in a 200 litres bin at a concentration of 1%. At the end of the process, a screening is performed on the final compost using a 2 mm sieve. Particles of test material that are <2mm (and pass through the sieve) are assumed to be "disintegrated". Particles and pieces >2mm are counted as non disintegrated mass. The degree of disintegration is the "<2mm mass" divided by the original mass of test material.

The method is described in the standard ISO 16929 (Plastics -- Determination of the degree of disintegration of plastic materials under defined composting conditions in a pilot-scale test). The test sample can be a film, a foil, a sheet, a foam, or the packaging itself. The thickness of the specimens used in the disintegration test is important because fixes the maximum thickness at which the packaging material under study can be applied in the market. At the end of the cycle, which lasts 3 months, the disintegration is verified by sieving. The standard specifications require a minimum disintegration of 90%

1.4 Ecotoxicity and metals content

The biodegradable plastic/packaging is a “feedstock material” of composting. It, therefore, shall not affect the safety and quality of the final product, the compost. Samples of compost made with 10% biodegradable plastics are compared with samples of a reference compost, produced only with organic waste, without any plastic. The effect of the compost samples on the plant growth are assessed, using an ecotoxicity test method (a plant growth test), to show that the test material, during degradation, does not release into the compost substances toxic for the plants and the environment. The results shall not differ significantly. Other analysis are : volumetric weight (density) ; total dry solids ; volatile solids ; salt content ; pH ; levels of nutrients (total nitrogen, ammonium nitrogen, phosphorus, magnesium and potassium). In a preliminary phase, information on the packaging material is gathered.

The metals concentration in the biodegradable plastic is determined and levels shall be below given limits, as indicated in the standard specifications.

2 CONTROVERSIAL ISSUES

The standard specifications on compostable products have been tested in the field for many years now. No negative results are available in the literature, as far as we know. Negative results mean: products that although shown to comply with the standard specifications have proven to be non compostable in real plants. On the other hand, the standard specifications have been shown to be effective in recognising materials that, in real life, do not compost satisfactory (Davis, 2002; ReMaDe Essex, 2003; California State University - Chico Research Foundation, 2006).

Nevertheless, some criticisms have surfaced in the last 10 and more years of practical application, against the standard specifications on compostability. Main issues are addressed in the following paragraphs.

2.1 Biodegradability requirements

Criticisms have been voiced about the test duration (6 months) considered too long, accepting materials with a slow biodegradation rate. According to this viewpoint, there is a contradiction between the laboratory test method and real composting. The biodegradation test under composting conditions is an optimized simulation of the composting environment. Temperature, set at 58°C, is constant, humidity is controlled at optimal levels and so it is the aeration. On the other hand, the thermophilic phase of real composting processes lasts normally no more than 2-3 months. Furthermore, even during the thermophilic phase, temperature is not constantly high: after a first hot period, temperature slowly decreases tending towards ambient temperature. Not always water availability and aeration will be optimal. Therefore, the application of the laboratory scale biodegradation data to the real composting process could be misleading. Biodegradation of the material could be rather limited under real conditions and compost full of residues. According to these reviewers, the test duration should be 2-3 months maximum, similarly to the thermophilic phase in a real composting process.

For others the test duration is too short. This means that the required biodegradation rate is too fast and many potentially good materials are excluded. According to this viewpoint, the biodegradation test is a means to verify the inherent biodegradability and should have no relation with the duration of a real composting process. It is instead the pilot scale composting test (see 1.3) that provides an adequate proof that the material is sufficiently degraded under real conditions. Biodegradation can then be completed during the application phase of compost, when spread in the soils. Besides, according to these experts, the too high biodegradation level (90%) of compostable plastics is a negative traits. Composting of compostable plastics is seen as a “cold incineration” with bioplastics fast converted into CO_2, with no compost formed and with no “carbon sink”, a useful concept in the battle to the greenhouse gases.

The test duration set by the standard specifications is the result of combining these different viewpoints. It takes into account the duration of the real composting phase which, considering also the curing phase, can effectively be of 6 months. On the other hand, the test conditions are optimized in order to avoid false negative results caused by poor environmental conditions and thus verify the inherent biodegradability of the materials.

A 90% biodegradation in 6 months is considered as a good indication that the material is inherently biodegradable and with a rate compatible with the composting cycle.

The debate about the too fast biodegradation of compostable plastics (the “cold incineration”) is based on a wrong concept that confuses the laboratory approach, used to show inherent biodegradability, with behaviour on a real composting process. The ISO 14855 is carried out using mature (spent) compost as a solid matrix and as an inoculum. For technical reasons it could not be otherwise, in order to allow the correct measurement of the extra CO_2 produced by the test material. At test start, the rather starved microorganisms present in the mature compost are suddenly exposed to a biodegradable substance that switches on metabolism and competition. Soon the microbial population is forced back to a starvation phase, where biomass fast formed in the early logarithmic phase is consumed to produce maintenance energy. These conditions are recognised as not favourable for biomass formation but rather for catabolic reactions i.e. oxidation to CO_2 and water (Russell and Cook). This is a positive effect in practical terms, because the biodegradation test method is based on the determination of the evolved CO_2. On the other hand, real composting is a solid fermentation which requires the mixing of different starting materials, in a "balanced” recipe. As a matter of fact the term "compost" has the same root of compound, composite etc. Different ingredients are needed to assure proper texture, proper carbon/nitrogen ration, proper initial humidity, etc. Fast biodegradable compounds (e.g. polysaccharides) are needed together with slow biodegradable (e.g. lignin) to make the compost process happen. No composting expert would consider composting as a "cold incineration" of vegetable or fruit residues, only because they are the most biodegradable in the bio-waste mixture. Bioplastics can be considered as a constituent of the mixture, similar in behaviour to cellulose.

Another question that has surfaced over the years concerns the question of why 90% has been fixed as a pass level rather than 100%. Does this leave a residue of the remaining 10%? The answer is that experimental factors such as measuring errors and the formation of biomass make it hard to reach 100% accurately; this is why the limit of acceptability was established at 90% rather than 100%.

2.2 Disintegrability requirements

The disintegration test is carried out in a pilot plant for 3 months. This period could be seen as an indication that the compostable products will not disintegrate efficiently in a shorter composting cycle. Therefore, the compostable plastics are seen as a threat for those composting plants with cycle shorter than 3 months. However, this is a preconception that originates from the idea that the lab testing scheme adopted for the characterisation and the assessment of compostable plastics can be perfectly translated into the real composting plants. Actually the trials carried out by the IRS from 1991 through 1996 (ASTM/ISR , 1996) clearly showed that the disintegration test at pilot scale was conservative in comparison with the real composting process (“comparing the results obtained for the same material at each tier shows that for all materials compared, without exception, the degradation results obtained in a higher-level test equalled or exceeded those in a lower-level test”). Therefore, the fact that the testing period is 90 days does not imply that a compostable plastic will behave in a unsatisfactory way in a shorter cycle.

2.3 Ecotoxicity requirements

Ecotoxicity is tested at the end of a composting process. Why only at the end? Why not monitor the composting process, step by step? Ecotoxic molecules can be possibly produced and released during the composting process and the effect can be temporary or permanent, according to the lifespan of the toxic molecules. The temporary toxic effect of biodegradation on plant development is a well known phase occurring during the biodegradation process of any organic natural substrate. As a matter of fact, immature compost is not suitable for direct use in agriculture because of its phytotoxicity. Great care must be devoted in order not to mistake temporary phytotoxicity caused by the biodegradation process for a real permanent ecotoxicity. A cost-effective solution for the detection of possible ecotoxic effects is to focus on permanent activities, after the first transient phytotoxic phase. The ecotoxic activity assessed in this case is the cumulative sum of stable toxic molecules released during the different stages. It is, therefore, possible to verify with just one sampling the presence of permanent ecotoxic activities, independently from the moment of production. This is the reason why the standard specifications do only test compost at the end of composting.

3 NEXT DEVELOPMENTS

The current approaches to standardisation on composting have been considered satisfactory and suitable for the assessment of the environmental safety of compostable plastics.

The main reason lies in very high mineralisation threshold level (90%) required by the standard specifications which can be considered as a robust evidence of total biodegradation and of no remaining residues. Additionally, disintegration and ecotoxicity tests are carried out to confirm that plastic products do not affect the compost quality and safety.

However, due to the growing volumes of biodegradable polymers and plastics, interest in their environmental safety is increasing. A recurring question concerns the environmental impact of possible trace substances released during polymer biodegradation which could be spread in the soil together with the compost. This chance seems low, given the high biodegradation level required by the standards, but any concern on new products, such as the compostable plastics, must be properly addressed to gain confidence of all stakeholders.

Biodegradable polymers are solid materials not available to microbial intracellular enzymes. The surface of the product is the interface between the plastic solid phase and the aqueous liquid phase, where microorganisms are present. Microorganisms need to reduce the polymer’s molecular length before assimilation. This happens thanks to extracellular enzymes that are released by the micro-organisms, reach the plastic product and depolymerise the polymeric chain (Marten et al., 2003). Molecules such as oligomers, dimers, monomers are “peeled off” from the solid polymer, set free in the surrounding compost, assimilated by microorganisms, and ultimately converted into CO_2. (Tokiwa et al., 2009). While the original polymer can be considered as an inert material, the low molecular weight molecules resulting from its depolymerisation are of interest and need a specific characterisation, because they are mobile in the environment and can have biological effects. It is clear that a 90% biodegradation level (required by the standards) can only be reached if all or most free molecules are fast assimilated and oxidised. Still, their characterisation can be interesting when supplementary information on the environmental fate of the biodegradable plastics are requested.

Biodegradable plastic items often contain other constituents besides the biodegradable polymer such as fillers and additives which are used to obtain specific technological effects, such as plasticization and stabilization, or a change, for example, in optical, mechanical or processing properties. These additives are expected to be released from the plastic item into the surrounding environment in a similar fashion to what happens to the polymer intermediates released during the first step of biodegradation.

It should here be pointed out that recently ISO modified the standard on compostability of plastics (ISO 17088) to take this issue in consideration. The current ISO specification requires that constituents present in the plastic formulation in a concentration below 10% shall be tested individually for biodegradation. This is to make sure that also minor constituents are biodegradable, as these could not be detected by just measuring the overall biodegradation that requires a final 90% level and that could “hide” some minor recalcitrant substances.

In order to remove any doubt about the environmental safety of compostable plastics, monomers, additives and any other molecule that can be expected to be released or formed during the first stage of biodegradation (depolymerisation) can be individually assessed by using a suitable evaluation scheme. Evaluation schemes for chemicals already exist. For example in Europe, the potential impacts of chemical substances on both human health and the environment is dealt by the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) Regulation. The same approach suggested by REACH could be applied to the biodegradation intermediates, with the necessary modifications.

4 CONCLUSIONS

The current standards on biodegradability and compostability of packaging and plastics pose stringent requirements asking for fast and total biodegradation of the product, that is released in the environment. Criticisms raised in the last decade are neither proven with data nor theoretically founded. To improve confidence on these products, and erase any doubts about trace hazardous substances released in the soil we suggest to identify these potential intermediates and assess them individually by using a scheme borrowed by already established methodology, with the necessary modifications.

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(10) ENVIRONMENTAL BURDENS ASSOCIATED TO DIFFERENT TECHNOLOGIES FOR THE BIOLOGICAL TREATMENT OF OFMSW

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EXECUTIVE SUMMARY

Waste management, a complex system involving at least waste collection and waste treatment, has been analyzed from different points of view. Obviously, the economics of waste management systems are mainly required by the authorities while engineers also appreciate technical/engineering information on the system. However, in the last years, a number of authors have also been studying waste management systems by focusing on their environmental impact (mainly energy and material burdens). This impact can be added as a new parameter in the decision matrix when evaluating the suitability of different treatment technologies. Life Cycle Assessment (LCA) is a tool commonly used to calculate these impacts. Reliable results in LCA studies require the use of reliable data relating the system under study. In the case of waste management it is important to obtain data at local level since the characteristics of the municipal waste depend on different factors: eating habits, collection system, etc.

The objective of the present study is to identify and quantify the input and output flows (water, energy, treated waste, compost produced, refuse generated, gaseous emissions, etc.) of different full-scale biowaste biological treatment facilities based on different technologies in order to have representative local data for a Life Cycle Inventory. Gaseous emissions (Volatile Organic Compounds (VOC), NH_3, CH_4 and N_2O) have been determined on site following a methodology specially designed for this purpose. Gas chromatography has been used in the analysis of total VOC, CH_4 and N_2O while ammonia was determined with a specific sensor. Main families of VOC present in gaseous emissions have also been determined in one of the plants studied by Gas Chromatography-Mass Spectrometry.

Four industrial plants located in Catalonia (Spain) treating the Organic Fraction of Municipal Solid Wastes (OFMSW) have been studied over a period of two to three months each, covering a wide range of plant size (from 100 to 18000 Mg OFMSW y^{-1}): an in-vessel composting plant (CT), an aerated windrows composting plant (CCW), a turned windrows composting plant (TW) and a plant including anaerobic digestion and in-vessel composting of the digestate (ADC). A Mechanical Biological Treatment installation (MBT) has also been studied treating OFMSW (109006 Mg y^{-1}) and municipal solid waste (MSW, 130518 Mg y^{-1}) in two separate lines. The degree of technological complexity of the studied facilities has been also taken into account. Thus, low technology composting plants based on turned windrows to complex facilities with mechanical and biological (anaerobic digestion plus composting) processes have been analysed.

The Life Cycle Inventory could be summarized as: energy consumption ranging between 235 and 870 MJ t OFMSW^{-1} when computing diesel consumption and electricity. Plants including anaerobic digestion present a net electricity production due to biogas energy transformation. The ADC plant generates produces 550 MJ Mg OFMSW^{-1} while the MBT plant produces 792 MJ Mg OFMSW^{-1}. Emissions of the different contaminants considered per Mg OFMSW ranging between $3.5\cdot10^{-5}$-8.9 kg VOC, 0-8.63 kg NH_3, $1.5\cdot10^{-4}$-4.37 kg CH_4 and $1.4\cdot10^{-5}$-0.251 kg N_2O, respectively with the MBT plant presenting the lowest values. Predominant VOC families in MBT plant emissions are aromatic hydrocarbons and nitrogen compounds. The use of emission control equipment to avoid gaseous pollutants release derives in a significant increase in the energy consumption and complexity of the installation. However, when the treatment facility is located near to inhabited areas the use of emission control equipment is mandatory.

1 INTRODUCTION

Our daily activities inevitably lead to waste generation. Specifically, each person generates around 1.6 kg of waste per day, of which 500 g corresponds to organic matter (OFMSW). This organic matter (food scraps and garden waste) can be valorised using different treatment technologies, mainly anaerobic digestion and / or composting. The final product obtained, called compost, can be used as an organic amendment in agriculture. In both cases, the use of the obtained products (compost and / or biogas) generate benefits over the alternative of sending the waste to landfill. The use of compost improves the soil structure, provides organic matter and increases its water holding capacity. On the other hand, the use of compost avoids, in part, the use of chemical fertilizers (whose production generates some environmental impacts). In the case of biogas, its use in electricity production avoids the consumption (and production) of electricity from potentially more polluting and non-renewable sources. Furthermore, the use of waste heat in electricity production from biogas to maintain the temperature of anaerobic digesters can reduce even more the consumption of external energy.

However, as an industrial process, the treatment of the OFMSW generates environmental impacts that must be assessed. During the process there is energy consumption, emissions are released to the atmosphere and leachates are generated, among other impacts. Those impacts can be different depending on the technologies used for the treatment of waste. However, due to the broad number of technologies and waste collection systems it is necessary to collect real local data on each management system to generate reliable information on the environmental impacts. This information can be used for example to asses a Life Cycle Inventory or in waste management systems modelling, to compare facilities or be used in a technology decision process or in regional greenhouse gases inventories.

1.1 Background

Many of the studies related with the environmental impact of municipal solid waste treatments have been performed at laboratory scale and a few through the study of industrial-scale facilities (Komilis et al., 2004, Hellebrand et al., 2001, Pagans et al., 2006, Smet et al., 1999, Lou et al. 2009). However, little literature can be found on the global impact of a specific technology or facility by using in situ measurements (especially with biowastes). This is the case, for example, of Blengini (2008) who used Life Cycle Assessment (LCA) methodology to evaluate the environmental impacts of a composting plant in Italy. The results indicate that emissions generated during the composting process are mainly the large group of volatile organic compounds (VOCs), methane (CH_4), nitrous oxide (N_2O) and ammonia (NH_3). All these compounds can generate environmental impacts: VOCs can cause odors, as ammonia, but may also participate in photo-reactions in the atmosphere resulting in oxidizing compounds such as ozone. Methane and nitrous oxide have a high global warming potential. It is also important to determine which VOCs are emitted. In this area little work can be found, among them, Orzi et al. (2010) determined the Volatile Organic Compounds emitted during the anaerobic digestion of the OFMSW at a full scale treatment facility.

Regarding management systems modelling, some literature can be found on municipal solid wastes, for example: EASEWASTE (Kirkeby et al., 2006), ORWARE (Sonesson et al., 1997) and WASTED (Díaz et al., 2006), are simulation tools that include the environmental burdens associated to waste management. LCA has been also applied to generic waste management systems (De Feo et al., 2009) and to MSW management systems of different cities or regions such as Wales (Emery et al., 2007), Ankara (Özeler et al., 2006), Phuket (Liamsanguan et al., 2008), Corfu (Skordilis et al., 2004) or Delaware (Kaplan et al., 2009). Other authors have focused their research on the environmental impact of the different waste collection options (Iriarte et al., 2009). Some of these works include a great effort to obtain real local data to perform the study.

Clearly, the biological treatment processes also generate, directly and indirectly, CO_2 emissions. However, CO_2 emissions from biological processes are generally not taken into account in greenhouse gases inventories as they come from a biogenic source, but clearly, CO_2 emissions from energy consumption (electricity or diesel) must be determined and taken into account.

1.2 Research objectives

The objective of this paper is to present the Life Cycle Inventory (input and output flows) of five different Organic Fraction of Municipal Solid Waste treatment plants. These plants include composting facilities, a combined anaerobic digestion and composting plant and a Mechanical Biological Treatment (MBT) plant. Environmental burdens are expressed in terms of energy and water consumption, energy generation, production of compost (and quality of the product) and gaseous emissions per ton of waste treated. Besides, the composition of the Volatile Organic Compounds emitted is also assessed for the MBT plant.

2 METHODOLOGY

A combination of a questionnaire addressed to plant managers and a systematic sampling work was used to obtain the data for the Life Cycle Inventory. Data on amounts on the treated waste, refuse and compost production, electricity and water consumption were obtained through the questionnaire. Emissions of NH_3, N_2O, CH_4 and VOC were determined in situ or in the laboratory, as explained below.

A specific methodology was developed to calculate gaseous emissions from the composting process (Cadena et al., 2009; Martínez-Blanco et al., 2010) and from biofilters (Colón et al., 2009). In brief, airflow velocity and ammonia, nitrous oxide, methane and VOC concentrations on the surface of the composting pile or the biofilter were simultaneously measured in order to calculate the gas outlet emission rate (mg/s). Air velocity was determined using a thermo-anemometer (VelociCalc Plus mod. 8386, TSI Airflow Instruments, UK) and a Venturi tube (Veeken et al., 2002). The product of each pollutant concentration (mg m^{-3}) and air velocity (m s^{-1}) results in the mass flow of a given compound released per surface area unit studied (mg s^{-1} m^{-2}). The pollutant mass flow per area unit (mg s^{-1} m^{-2}) was multiplied by the entire emitting surface area resulting in the outlet mass flow emission (mg s^{-1}) at the moment of measurement for each compound.

Ammonia concentration in gaseous emissions was determined in situ using an ammonia sensor ITX T82 with a measurement range of 0 to 200 ppmv. Gaseous samples were also collected in Tedlar bags for the laboratory determination of VOC, methane and nitrous oxide. Total VOC were analyzed as stated in Colón et al. (2009). Briefly, total VOC content from gaseous samples was determined as total carbon content using a gas chromatograph equipped with a flame ionization detector (FID) and a dimethylpolysiloxane 2 m×0.53 mm×3.0 μm column (Tracsil TRB-1, Teknokroma, Barcelona, Spain). This column permits the determination of total VOC as a unique peak. The injected volume was 250 μL and the analysis time was 1 min. The gas chromatography operating conditions were as follows: oven temperature isotherm at 200ºC, injector temperature 250ºC, FID temperature 250ºC; carrier gas helium at 1.5 psi pressure. Methane was also analyzed by gas chromatography using a Flame Ionization Detector (FID) and a HP-Plot Q column (30m x 0.53 mm x 40 µm) with a detection limit of 1 ppmv. The gas chromatography operation conditions were as follows: oven temperature isothermal at 60°C, injector temperature 240°C, FID temperature 250°C; carrier gas N_2 at 4 psi pressure. Nitrous oxide was analyzed by gas chromatography using an Electron Capture Detector (ECD) and a HP-Plot Q column (30m x 0.53 mm x 40 µm) with a detection limit of 50 ppbv. The gas chromatography operation conditions were as follows: oven temperature isothermal at 60°C, injector temperature 120°C, ECD temperature 345°C; carrier gas N_2 at 4 psi pressure. Measurement data for all gases studied were acquired and quantified by the Empower® 2 software (Waters Associates Inc., Milford, USA).

Volatile organic compounds (VOC) from some air samples were analysed by SPME/GC–MS such as previously reported and tested (Davoli et al., 2003; Pierucci et al., 2005; D’Imporzano et al., 2008; Orzi et al., 2010). A manual SPME device and divinylbenzene (DVB)/Carboxen/polydimethylsiloxane (PDMS) 50–30 μm fibre Supelco, Bellefonte, PA, USA) was used. The compounds were adsorbed from the air samples by exposing the fibre, preconditioned for 1 h at 270 °C, as suggested by the supplier, in glass gas collector (250 ml) for 30 min at room temperature. A solution of deuterated p-xylene in methanol was used as internal standard (IS) for quantitative analysis. VOC analysis was performed using an Agilent 5975C Series GC/MSD. Volatiles were separated using a capillary column for VOC (Agilent Technologies DB-624) of 60 m x 0.25 mm and a film thickness of 1.40 μm. Carrier gas was helium at a flow rate of 0.8 ml min^{-1}. VOC were desorbed exposing the fibre in the GC injection port for 3 min at 250 °C. A 0.75 mm i.d. glass liner was used and the injection port was in splitless mode. The temperature program was isothermal for 2 min at 50 °C, raised to 170 °C at a rate of 3 °C/min, and after to 230ºC at a rate of 8ºC/min. The transfer line to the mass spectrometer was maintained at 235 °C. The mass spectra were obtained by electronic impact at 70 eV, a multiplier voltage of 1379 V and collecting data at a m/z range of 33–300. Compounds were tentatively identified by comparing their mass spectra with those contained in the NIST (USA) 98 library. A semi-quantitative analysis, for all the identified compounds, was performed by direct comparison with the internal standard.

3 RESULTS AND DISCUSSION

As stated above, different waste treatment installations have been studied in order to identify the potential sources of environmental impacts and quantify the input and output flows. The main characteristics of these plants are summarized in Table 1. All the plants treat OFMSW except for the MBT plant which is also treating MSW. In this case, OFMSW undergoes anaerobic digestion plus composting of the digestate and the MSW is treated by means of an in-vessel composting process. All the plants use pruning waste as bulking agent in OFMSW or digestate conditioning.

The plants studied cover a wide range of plant types and capacities from 91 to 239524 t year^{-1} representing respectively, a small composting plant receiving a high quality OFMSW (containing a very low percentage of impurities) and a large MBT plant.

The input and output flows referred to the treatment of 1 Mg of waste (OFMSW or OFMSW+MSW) determined for the five plants are presented in Table 2. Data on Table 2 have been partially published before (Cadena et al., 2010; Colón et al., 2012). The refuse produced in the treatment plants is directly related to the quality (percentage of impurities present in the input waste) of the waste treated. As can be seen in Table 2, MBT plant produces 0.58 Mg Refuse Mg input waste-1 mainly due, in this case, to the fact that refuse from the two treatment lines (OFMSW and MSW) is processed and quantified in a single value.

Electricity is mainly consumed in in-vessel and windrows aeration. The lowest value found (33.41 MJ Mg OFMSW^{-1}) corresponds to a turned windrows composting plant, a low technology plant while the highest value (770.4 MJ Mg OFMSW^{-1}) comes from a completely closed composting plant with in-vessel decomposition and aerated windrows curing. On the other hand, diesel consumption is related mainly to material handling and machinery used in pre and post treatment operations being higher in low technology plants. For the two plants with anaerobic digestion, a net production of electricity exists: 216.7 and 518.4 MJ Mg OFMSW^{-1} for ADC and MBT plants respectively balancing electricity consumed and generated. It should be noted that the efficiency in biogas conversion differs in the two plants as biogas production is very similar (98.8 and 110.2 m^3 biogas Mg OFMSW^{-1} for ADC and MBT respectively) and electricity production is clearly higher in MBT (550 in front of 792 MJ Mg OFMSW^{-1}). This fact highlights the importance of including as much treatment plants as possible in a data inventory in order to perform a reliable Life Cycle Analysis.

The influence of the gas treatment processes is clear when comparing the emissions computed for the plants studied: TW plant, completely open to the atmosphere, present the highest emissions for all the compounds studied: 8.63 kg NH_3, 5.70 kg VOC, 0.251 kg N_2O and 4.37 kg CH_4 per Mg OFMSW followed by CCW plant, also completely open. The extremely low values found in the case of the MBT plant when compared to the rest of installations in the study should be noted, especially in the case of ammonia presenting 0 emission. In fact, the location of the MBT plant in the limit of an industrial area but not far from residential buildings makes the operation of the gas treatment equipment (wet scrubbers + biofilters) essential with continuous controls and effluent concentration measurements. It is a complex and costly system consisting in different web scrubbers and a series of biofilters.

VOC emission form the MBT plant has also been characterized by GC-MS. Figure 1 represents the values obtained for the concentration of different VOC families in gaseous emissions from MBT plant after treatment in different days of measurement. As can be seen in Figure 1, aromatic hydrocarbons and nitrogen compounds are the main families found except in the fifth day of measurement when nitrogen compounds concentration was very low. Terpens, which have been described as principal compounds and responsible for odorous pollution in OFMSW treatment installations (Font et al., 2011) are present at the exit of the biofilter but in low percentage. However, the amount of total VOC emitted is very low when compared to the rest of plants studied (Table 2).

4 CONCLUSIONS

Five different MSW treatment plants have been studied and their input and output flows determined. Differences have been found within treatment technologies and plants. Plants including anaerobic digestion present a net electricity production that will have to be taken into consideration if a LCA should be performed and technologies compared from a Life Cycle perspective. Low technology plants present low electricity consumption but high gaseous emissions due to the inexistence of treatment equipment. In this sense, this equipment has demonstrate its efficiency in completely closed plants, especially in the MBT plant where gaseous emissions are the lowest.

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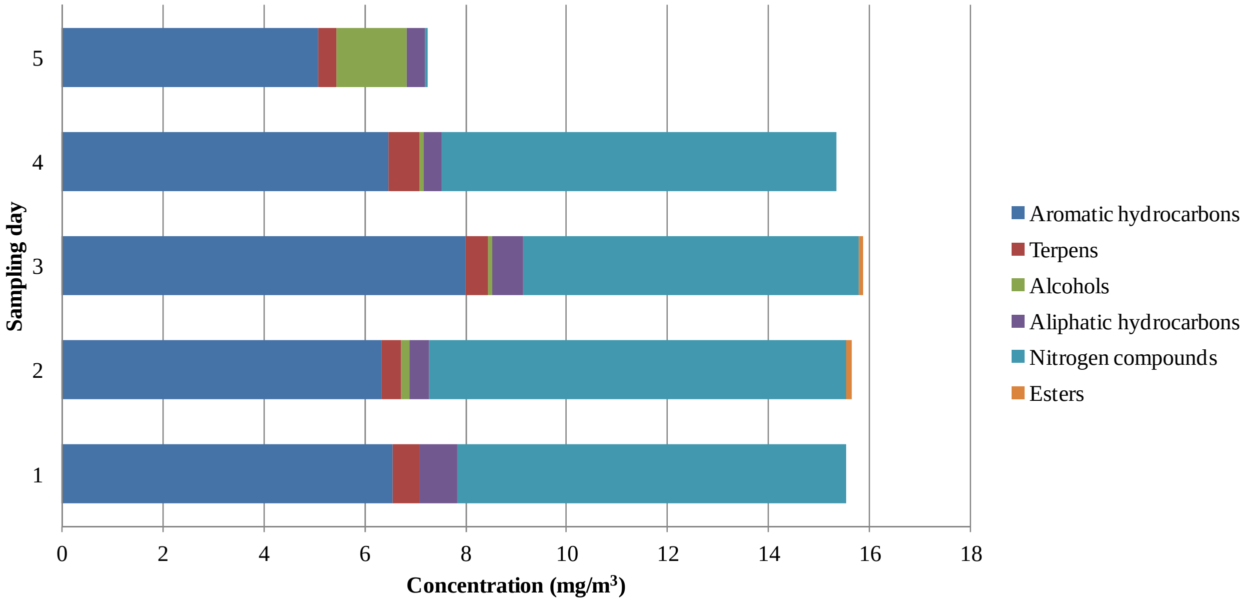

FIGURE 1 **Concentration of the different VOC families in MBT emissions (biofilter outflow)**

TABLE 1 **Main characteristics of the studied industrial facilities (CT: composting in-vessel; CCW: composting in confined windrows; ADC: anaerobic digestion plus composting, TW: turned windrow composting and MBT: mechanical biological treatment).**

Facility	**CT**	**CCW**	**ADC**	**TW**	**MBT**
Main biological process	Composting	Composting	Anaerobic digestion + composting	Composting	Anaerobic digestion + composting
Pre-treatment	Trommel* screen (80 mm)	No	Ballistic separator + Magnetic separator	Trommel* screen (80 mm)	Trommel screen (80 mm) + Ballistic separator + Magnetic separator
Decomposition phase	In-vessel composting	Aerated windrow composting	Anaerobic digestion + in-vessel composting	Turned windrow composting	Anaerobic digestion (OFMSW) aerated and turned channel composting (MSW)
Curing phase	Aerated windrow	Turned windrow	Turned windrow	Turned windrow	In-vessel composting (OFMSW)
Post-treatment	Trommel screen (10 mm) + ballistic separator	Trommel screen (10 mm)	Trommel screen (12 mm) + ballistic separator	Trommel screen (10 mm)	Trommel screen (12 mm) + ballistic separator
Type of facility	Completely closed	Completely open	Completely closed	Completely open	Completely closed
Exhaust gas treatment	Wet Scrubber + biofilter	Not present	Wet Scrubber + biofilter	Not present	Wet Scrubbers + biofilters
Waste treated (t year^{-1})	7435	91	17715	3000	239524 (109006 OFMSW + 130518 MSW)
Refuse (percentage of weight over input material)	10	1	13	11	58*

* The plant treats separately MSW and OFMSW with a single refuse flow.

TABLE 2 **Input and output flows in the studied treatment plants. All parameters are referred to 1 Mg of waste treated (CT: composting in-vessel; CCW: composting in confined windrows; ADC: anaerobic digestion plus composting, TW: turned windrow composting and MBT: mechanical biological treatment).**

Facility	**Flow**	**CT**	**CCW**	**ADC**	**TW**	**MBT**
Inputs	MJ electricity	770.40	235.80	166.32	33.41	273.60
	MJ electricity self generation	0	0	167.04	0	0
	l diesel	2.66	9.00	3.64	5.33	0.7
	Total MJ (electricity + diesel)	871.90	579.24	472.26	236.80	300.31
	m^3 water in the waste gas treatment process	0.42	n/a	0.12	n/a	n/e
	m^3 water used in the composting process	0.14	0.00	0.00	0.00	n/e
	Total m^3 water	0.56	0.02	0.12	0.00	0.32
Outputs	m^3 leachate	n/e	0.00	0.03	0.00	0.17
	m^3 biogas condensates	n/a	n/a	0.05	n/a	n/e
	kg NH_3	0.11	2.00	0.23	8.63	0
	kg VOC	0.36	6.22	0.86	5.70	$3.5\cdot10^{-5}$
	kg N_2O	0.075	0.076	0.035	0.251	$1.4\cdot10^{-5}$
	kg CH_4	0.34	1.68	2.39	4.37	$1.5\cdot10^{-4}$
	Mg Compost	0.10	0.52	0.03	0.20	0.106
	Mg Refuse	0.13	0.00	0.41	0.26	0.58
	m^3 biogas	n/a	n/a	98.90	n/a	110.2*
	Electricity MJ	n/a	n/a	550.08	n/a	792*

n/a: not applicable; n/d: not determined

* referred to 1 Mg of OFMSW (MSW does not undergo anaerobic digestion)

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(26) LIFE CYCLE ASSESSMENT OF PIG SLURRY TREATMENT TECHNOLOGIES

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EXECUTIVE SUMMARY

Animal manure is one of the major agricultural wastes and its management is associated with negative impacts on global warming, acidification and eutrophication of natural ecosystems.

These impacts are accentuated because some areas in Europe receive excessive amounts of animal manure. These so-called hotspot areas are characterized by high livestock densities and insufficient land for manure utilisation, resulting in phosphorus and nitrogen surpluses in these areas, with associated risks for losses to the environment. In other areas, the nutrients from manure are insufficient to maintain production, leading to a need to apply mineral fertilizers. This leads to the use of non-renewable natural resources for the production of the mineral fertilizer. To decrease the consumption of mineral fertilizer and avoid losses from areas with excessive availability of animal manure, the geographical distribution of nutrients in animal manure needs to improve.

To achieve this, slurry treatment technologies have been developed that focus on the separation of slurry into a solid and a liquid fraction. The liquid fraction contains most of the easily available nitrogen but less than half of the phosphorus, so is mainly valued as a nitrogen fertilizer. However, the high water content in the liquid fraction, makes this fraction less suitable for long distance transportation. Agricultural land nearby the farm often has been treated with slurry for multiple years, which implies that phosphorus concentrations in the soil are already sufficiently high to supply the crop demand. The solid fraction is more transportable, due to its relatively low water content. It has a high concentration of slowly-available nitrogen and phosphorus, so is mainly valuable as a phosphorus fertilizer. . Additional technologies that are developed to avoid environmental impacts include ammonia stripping from the liquid fraction, various technologies for energy extraction and upgrading of the solid fraction.

The objective of this study is to compare slurry treatment technologies in a Life Cycle Assessment (LCA) to see which technologies are associated with the lowest environmental impacts.

The LCA assesses the treatment of 1000 kg of slurry ex animal and provides an overview of natural resource consumption and potential impacts on the environment of five different scenarios. These scenarios are *(a)* direct land application (baseline scenario), *(b)* solid liquid separation by mechanical screw press, *(c)* screw press separation and composting of the solid fraction, *(d)* solid liquid separation by decanter centrifuge, and *(e)* decanter centrifuge separation with ammonia stripping of the liquid fraction. In the first scenario, all manure is applied to agricultural land nearby the farm. In the other scenarios, the solid fraction is transported to agricultural land 100 km away from the farm.

In general, the treatment technologies analysed in this LCA show environmental impact potential reduction. The only scenario that seems to have a higher impact potential, and only with respect to the global warming potential, is the scenario with screw press separation and composting of the solid fraction. The decanter centrifuge scenarios have equal or lower impact potentials than the screw press scenarios. The choice of the technology to implement in any given situation depends on the environmental problem in focus. If the problem is a modest phosphorus surplus, a screw press separator could be useful, but larger surpluses might require the more efficient phosphorus separation possible with a centrifuge. If there is also a problem with ammonia volatilization, ammonia stripping could be considered. Composting of the solid fraction does not appear to be an advantage in this analysis, although it may hold other advantages such as easy storage, reduced odour and lower transportation costs.

1 INTRODUCTION

1.1 Background

Large amounts of animal manure are produced in Europe and its management is associated with a number of environmental impacts. Impact categories that are mainly affected by animal manure management are *(a)* global warming potential, caused by methane and nitrous oxide emissions, *(b)* acidification potential, induced by ammonia emissions, and *(c)* eutrophication potential caused by nitrogen and phosphorus emissions.

These impacts are accentuated because an increasing share of agricultural land in Europe receives excessive amounts of animal manure. These so-called hotspot areas are characterized by high livestock densities and insufficient land for manure application. This has resulted in phosphorus surplus in these areas and associated risks for losses to the environment. In other areas, agricultural fields do not receive sufficient amounts of nutrients from manure and farmers need to apply mineral fertilizers to their fields. Non-renewable natural resources like phosphate rock, oil and natural gas, are used for the production of mineral fertilizer and there are considerable environmental emissions related to the extraction, manufacturing and use of these fertilizers. In order to decrease the consumption of mineral fertilizer and avoid losses from areas with excessive availability of animal manure, geographical redistribution of animal manure needs to improve.

To achieve this, slurry treatment technologies have been developed that focus on the separation of slurry into a solid and a liquid fraction. The liquid fraction contains most of the easily available nitrogen but less than half of the phosphorus, so is mainly valued as a nitrogen fertilizer. However, the high water content in the liquid fraction, makes this fraction less suitable for long distance transportation. Agricultural land nearby the farm often has been treated with slurry for multiple years, which implies that phosphorus concentrations in the soil are already sufficiently high to supply the crop demand. The solid fraction is more transportable, due to its relatively low water content. It has a high concentration of slowly-available nitrogen and phosphorus, so is mainly valuable as a phosphorus fertilizer. . Additional technologies that are developed to avoid environmental impacts include ammonia stripping from the liquid fraction, various technologies for energy extraction and upgrading of the solid fraction.

1.2 Research objective

The objective of this study is to compare slurry treatment technologies in a Life Cycle Assessment (LCA) to determine which are associated with the lowest environmental impacts.

2 METHODOLOGY

The LCA is based on information from literature research. Using these data, mass balances of the slurry flows from excretion by pigs for up to 100 years after field application, were carried out (Jensen et al., 2012). The LCA itself was conducted by using the GaBi software (www.pe-international.com).

2.1 Scenarios

The functional unit that forms the basis for the assessment is 1000 kg of slurry excreted by pigs. For this LCA, it is assumed that the pigs are raised in Denmark, and that they consume an average Danish feed composition. The pigs are housed in pig housing with partially-slatted flooring. All liquid manure (slurry and liquid fraction) is assumed to be stored in a tank covered with a tent. Storage of the solid fraction is covered to reduce self-heating, and compost is well aerated. Liquid manure is applied by trailing hose to fields around the farm, with an average distance of 8 km from storage. Solid fractions are transported to areas where phosphorus is needed, over an average distance of 100 km. It is assumed that by doing this, use of mineral phosphorus fertilizer is replaced on recipient farm fields. Solid fractions are applied by broadcast spreading followed by rapid incorporation.

Four treatment scenarios were compared to a baseline scenario:

- Baseline Scenario – Conventional manure management in the form of slurry
- Screw Press Scenario – Slurry separation with a mechanical screw press, with donor farm application of the liquid fraction and transportation recipient farm of the solid fraction
- Screw Press with Composting Scenario – Slurry separation with a mechanical screw press. The solid fraction is composted using windrow composting donor farm
- Decanter Centrifuge Separation Scenario – Slurry separation with a decanter centrifuge, with donor farm application of the liquid fraction and transportation recipient farm of the solid fraction

- Decanter Centrifuge with Ammonia Stripping Scenario – Slurry separation with a decanter centrifuge. Ammonia is stripped from the liquid fraction, resulting in a compact Nitrogen fertilizer

2.2 Model description

For the five scenarios, emissions from the slurry, liquid and solid fractions were analysed from the moment the slurry is excreted by pigs until 100 years after field application. Furthermore, natural resources that are needed for the treatment, transportation and field application and emissions during these processes were analysed. The environmental impact categories that are included in the analysis are the global warming potential, in CO_2-equivalents, acidification potential, in SO_2-equivalents, freshwater eutrophication potential, in P-equivalents, and marine water eutrophication potential in N-equivalents. ReCiPe2008 is used for analysis of environmental impact potentials (Goedkoop et al., 2009).

3 RESULTS AND DISCUSSION

A comparison of the treatment scenarios with the baseline scenario is done for four impact potential categories. The baseline scenario is set at 100%. For the treatment scenarios, values higher than 100% imply that the environmental impact is higher in the treatment scenario than in the baseline scenario. *Figure 1* provides an overview of the relative global warming potential. The screw press with composting scenario has an impact potential that is 25% higher than the baseline scenario due to CO_2 and CH_4 emissions during the composting process. All other treatment scenarios have a lower global warming potential than the baseline. The two decanter centrifuge scenarios show slightly lower impact potentials than the screw press scenario.

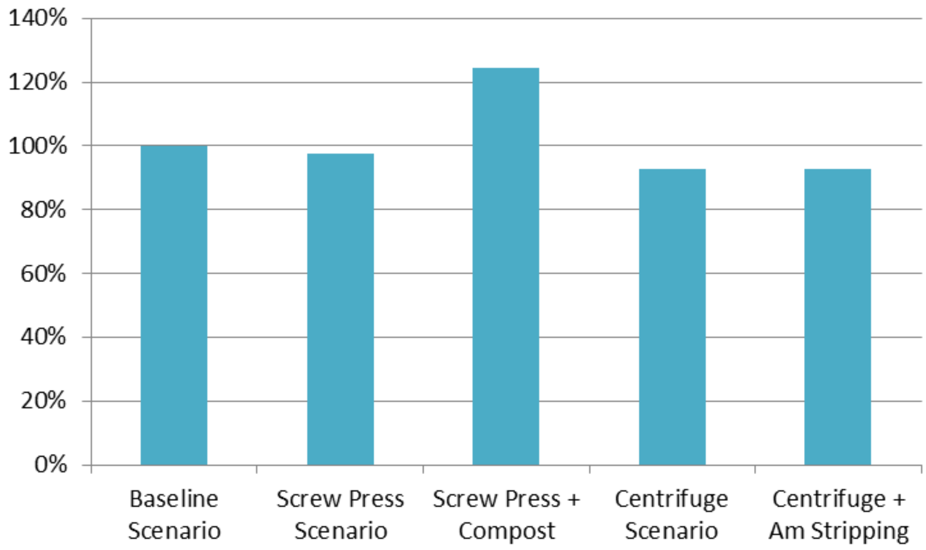

FIGURE 1 **Relative Global Warming Potential compared to Baseline Scenario (100%).**

All treatment scenarios show a lower freshwater eutrophication potential than the baseline scenario, see *Figure 2*. In the ReCiPe2008 method, fresh water eutrophication potential is calculated from the emissions of phosphorus, as freshwater bodies are assumed to be limited by phosphorus. For the two screw press scenarios, this reduced impact potential is approximately 45% smaller than in the baseline.

Phosphorus losses from the soil are calculated as a proportion of the difference between the phosphorus that is applied as fertilizer, and the average phosphorus need that plants have (assumed to be 21.5 kg P ha^{-1} yr^{-1}). If less phosphorus is applied than plants take up, a negative freshwater eutrophication potential results. This is only the case for field application of the liquid fraction after centrifuge separation. 71% of the phosphorus in slurry ends up in the solid fraction after centrifuge separation. Consequently, 29% of the initial phosphorus ends in the liquid fraction. Nitrogen separation is less efficient, resulting in a relatively larger share of nitrogen in the liquid fraction. As field application is assumed to be follow application limits for nitrogen, a smaller amount of phosphorus is spread in the centrifuge scenario. Ammonia stripping decreases the nitrogen content in the liquid fraction, and consequently decreases the nitrogen-phosphorus ratio.

Another explanation for the negative contribution to freshwater eutrophication for the two centrifuge scenarios, is that the production of mineral fertilizer contributes to freshwater eutrophication. The production of mineral phosphorus fertilizer is replaced in the scenarios where the solid fraction is applied to fields that are low in phosphorus. In the decanter centrifuge scenarios, a large share of phosphorus is in the solid fraction, 71%, and thus replacing mineral phosphorus fertilizer. After screw press separation, only 17% of initial phosphorus is in the solid fraction, implying a smaller replacement of mineral fertilizer.

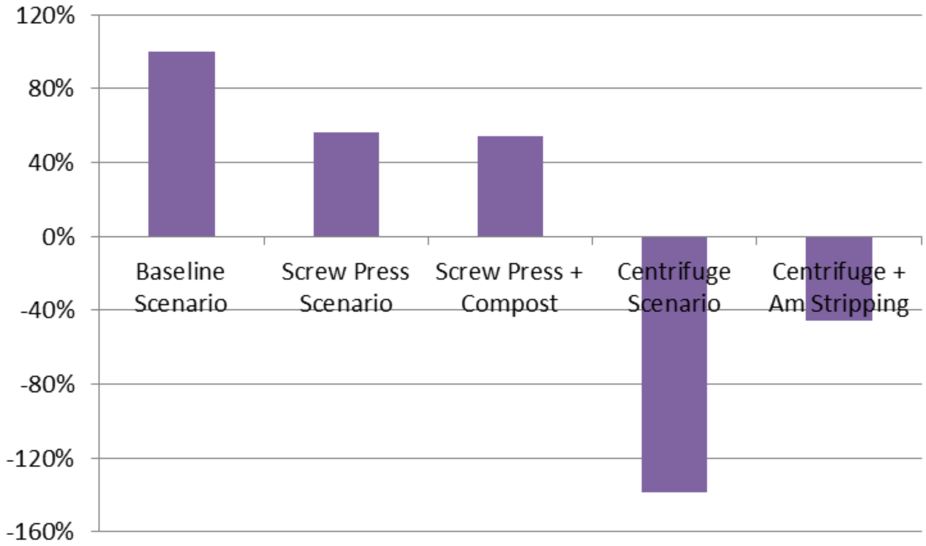

FIGURE 2 **Relative Freshwater Eutrophication Potential compared to Baseline Scenario (100%).**

Looking at *Figure 3*, it can be seen that all treatment scenarios have a lower contribution to marine eutrophication than the baseline scenario. In the ReCiPe2008 method, marine eutrophication potential is calculated from the emissions of nitrogen, because growth in saltwater bodies is assumed to be limited by nitrogen. Reductions range from 13% less for the screw press with composting scenario to 31% for the centrifuge with ammonia stripping scenario. This is mainly caused by the assumed mineral fertilizer replacement efficiency for the different slurry fraction. With 45%, the lowest replacement efficiency is assumed for the screw press with composting scenario, while the highest is assumed for the stripped ammonia, with 100%.

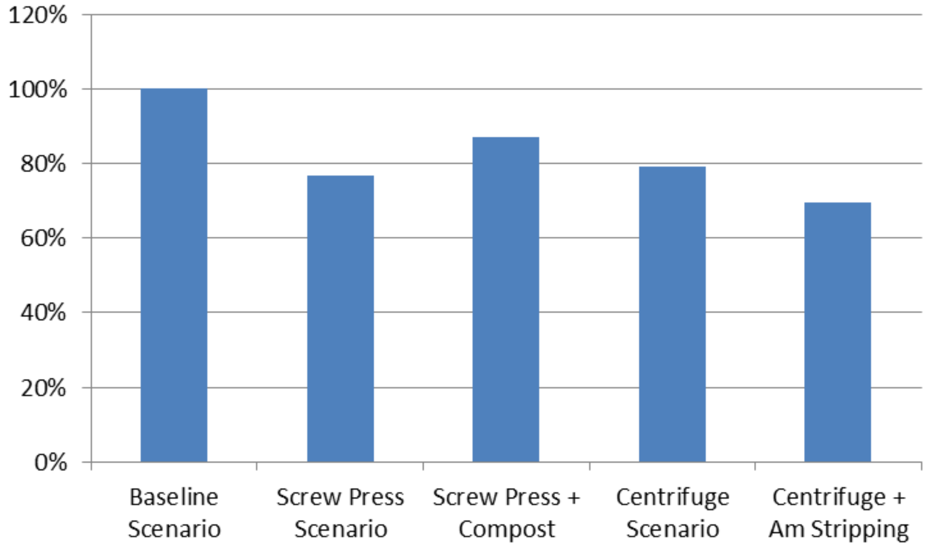

FIGURE 3 **Relative Marine Eutrophication Potential compared to Baseline Scenario (100%).**

The relative acidification potential ranges from 77% for the centrifuge with ammonia stripping scenario, to 92% for the screw press with composting scenario. The main contribution in all scenarios is caused during storage under the partly-slatted flours in the pig housing. As the storage during housing is similar in all scenarios and treatment first takes place after this stage, differences in acidification potential for the scenarios are mainly caused after field application of the slurry. For the centrifuge with ammonia stripping scenario, emissions during treatment are more than three times higher than in any other scenario. However, this increase during treatment is more than compensated for after field application, leading to a net reduction in ammonia loss from the complete stripping process.

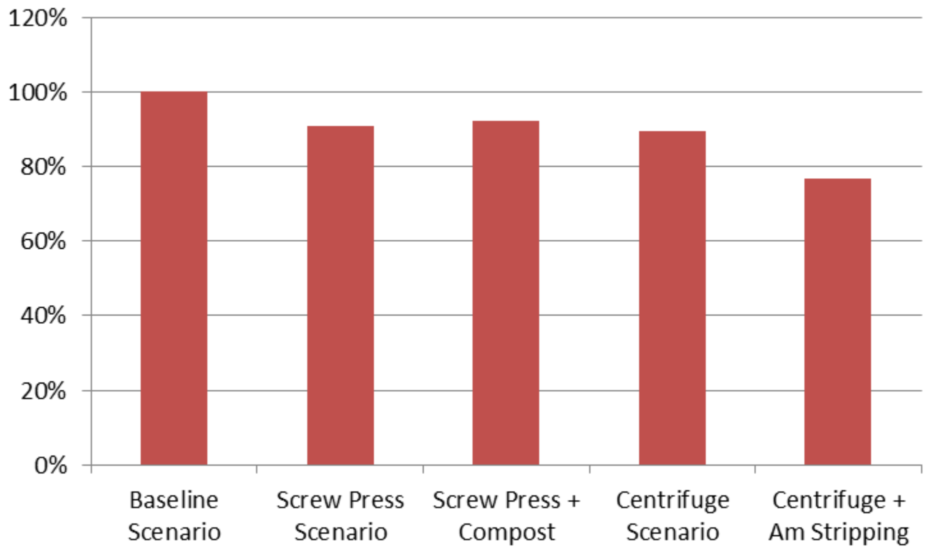

FIGURE 4 **Relative Acidification Potential compared to Baseline Scenario (100%).**

4 CONCLUSIONS

In general, the treatment technologies analysed in this LCA show environmental impact potential reduction. The only scenario that seems to have a higher impact potential, and only with respect to the global warming potential, is the scenario with screw press separation and composting of the solid fraction.

The decanter centrifuge scenarios have equal or lower impact potentials than the screw press scenarios. The choice of the technology to implement in any given situation depends on the environmental problem in focus. If the problem is a modest phosphorus surplus, a screw press separator could be useful, but larger surpluses might require the more efficient phosphorus separation possible with a centrifuge. If there is also a problem with ammonia volatilization, ammonia stripping could be considered. Composting of the solid fraction does not appear to be an advantage in this analysis, although it may hold other advantages such as easy storage, reduced odour and lower transportation costs.

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(63) LOCAL ASSESSMENT OF TOXICITY FOR MUNICIPAL SOLID WASTE THROUGH LIFE CYCLE ASSESSMENT AND USETOX

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EXECUTIVE SUMMARY

This research takes part in a PhD work aiming to develop an assessment methodology for local environmental impacts of Municipal Solid Waste (MSW) management in order to provide elements of choice to local decision-makers. MSW management is a local issue managed under the responsibility of French local authorities. These local authorities are currently threatened by a shortage risk of treatment facilities for MSW. This shortage is the result of overall treatment capacity reduction of existing facilities and, concomitantly the result of the low number of new facilities. The latter is linked to a social context rarely favourable to the construction and operation of these new plants. The damage to health and quality of life are part of the panel of arguments used by opponents.

To integrate environmental considerations, decision-makers use some environmental tools like Ecological Footprint, Carbon Footprint or Life Cycle Assessment. Because of its ability to assess global and multiple impacts, LCA is one of these tools that is used the more often. LCA enables to evaluate potential environmental impacts of a product or a system by identifying and quantifying the inputs and outputs associated for each impact category. LCA is an iterative methodology and is composed by four successive steps: goal and scope definition, inventory analysis, impact assessment and interpretation of results. However, the LCA tool does not consider the characteristics of the involved territory and consequently local assessments are not possible. Nevertheless, such a consideration appears necessary for local impacts such as toxicity and odours. Indeed, including local impacts is essential to obtain the support from people for the waste management project. To assess local impact with LCA, our work focuses on the third step of LCA: impact assessment. LCA usually assumes an impacted standard environment using a *Site Generic* approach. When local impacts are assessed, this approach is irrelevant because it does not take into account the emission characteristics, the environmental distribution, the background concentration and the sensitivity of the target (Potting and Hauschild, 1997a). To integrate these local parameters in assessment the *Site Dependent* approach can be used.

This paper focuses on a methodological development aiming to locally and quantitatively assess toxicity generated by MSW incinerator. The methodology considers the system “source/target” integrating emission conditions and parameters of impacted environment with the use of the USEtox model, a steady-state model to estimate toxicity impact in LCA. It has been adapted for local conditions and used to obtain environmental concentrations in order to build characterization factors.

The methodology described has been implemented with a case study: the atmospheric emissions of 2,3,7,8 tetrachlorodibenzo-p-dioxin (TCDD) caused by the incineration of MSW have been assessed using the USEtox steady-state model. The proposed methodology allows us to calculate an exposure concentration from background concentration and to assess the effect analysis, factor and impact characterization.

At the end of the case study, we identified the main limits of the new methodology were identified: the toxicity impact as differentiation element for decision, nested model versus meshed model, the analysis of case study results and potential rights to pollute. The impact characterization for TCDD was not relevant because the inventory data is too low related regulatory and incinerator technical improvements.

1 INTRODUCTION

In 2009, a French inhabitant produced 588 kg of Municipal Solid Waste (MSW) (ADEME, 2004). The French production of municipal waste appears to be stabilized for several years. In 2006, 50 million tons of municipal waste were produced in France. Out of these 50 million tons, 31 million tons are composed of 35,5 % of bulky and green waste and 64,5 % of household waste including residual household waste and waste selectively collected.

In France, the MSW management is a local issue which is managed under the responsibility of local authorities. Over time and changes in production, the waste management has become a national priority. This can be explained by economic, technical and environmental reasons. Firstly, France is currently threatened by a potential shortage of waste treatment facilities. It is explained by the low number of new facilities and by the current trend to reduce the overall processing capacities of existing facilities. The shortage of waste facilities is due to a late consideration of the waste production evolution. This deficiency can also be explained by the closure of some existing facilities in the event of a breach of the regulations, saturation or obsolete facilities. This shortage is also due to the local context which is rarely in favor of new facilities construction. The second reason is economic reason. In 2008, the waste management cost 14 billion Euros. Expenses related to household waste management are estimated to 8 billion Euros. More than half of these costs are borne by local authorities. Finally, waste management has become a national priority for environmental reasons so as to reduce the environmental burden and health impacts associated with non-management of these waste. While waste management aims to avoid serious pollution problems, treatment facilities generate nuisances and pollutants which may impact the environment.

1.1 Research objectives

Local decision-makers can use different environmental assessment tools for decision support like Ecological Footprint, Carbon Footprint or Life Cycle Assessment (LCA). LCA appears to be more often used with regard to its multi-criteria character. This tool is very popular and stands for an asset for environmental communication. However, despite all these advantages, this tool presents some methodological and application limitations. Many of these limitations are intrinsic to the methodology and some are related to its application to waste management.

2 LIFE CYCLE ASSESSMENT (LCA): AN ENVIRONMENTAL ASSESSMENT TOOL

LCA is a tool which assesses the potential environmental impacts caused by a system or product by identifying and quantifying the associated inputs and outputs. The use of LCA allows practitioners and stakeholders to determine improvements of environmental performances of a product or a process by considering all steps of its life cycle and to inform decision-makers of environmental consequences.

2.1 Methodology

According to the standard ISO 14040 and the Society Environmental Toxicity And Chemistry (SETAC), the LCA methodology is divided into four steps (FIGURE 1):

- Goal and scope definition. A trivial but fundamental step regarding the quality of the environmental study. The reasons for carrying out the LCA study are explained (ISO 14040, 2006, ISO 14044, 2006) and the purpose, the scope, the nature and the system function, the functional unit and the boundaries are determined.
- Inventory analysis. Mass and energy balance: identifies and quantifies the mass and energetic flows throughout the system. The inventory data must be reported to the functional unit which is the basis of the inventory quantification.
- Impact assessment. The relationship between inventory data and potential environmental impact are established. It is made up of two steps : the classification of emission in the different impact categories and the characterization which quantifies potential environmental impacts (ISO 14040, 2006). The term “potential” is used because the calculated impacts during LCA are not “real” impacts but possible. The use of potential term is due to the lack of knowledge concerning the substance environmental fate, the mechanism of impacts occurrence but also the phenomena of synergy and antagonism between substances (Potting and Hauschild, 1997a, Potting and Hauschild, 1997b).
- Interpretation of results: based on the results coming from the previous steps. It is possible to conclude and make recommendations (consistent with the objectives) to identify actions to reduce impacts of human activities on the environmental system.

The LCA methodology is an iterative methodology (FIGURE 1). At each step, it is possible and recommended to return to the previous steps adding some necessary information (details level, objectives of study, system boundaries…).

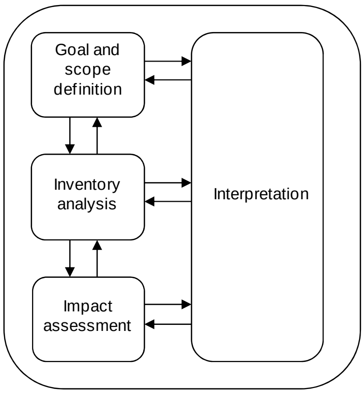

FIGURE 1 **Life Cycle Assessment framework (ISO 14040, 2006)**

The LCA methodology applied to waste management assessment differs with classical methodology LCA is some aspects.

2.2 LCA applicability for evaluation of MSW management

LCA is a popular tool used to evaluate the environmental performances of MSW management systems throughout its life cycle aims to compare waste management scenarios for a given waste (paper, glass, organic waste…). In this paper, LCA applied to MSW management is called "waste LCA".

2.2.1 A specific methodology

The methodological feature of waste LCA is the scope definition. In this study, the "life cycle" begins when the product becomes a waste which the owner wishes to dispose (FIGURE 2).

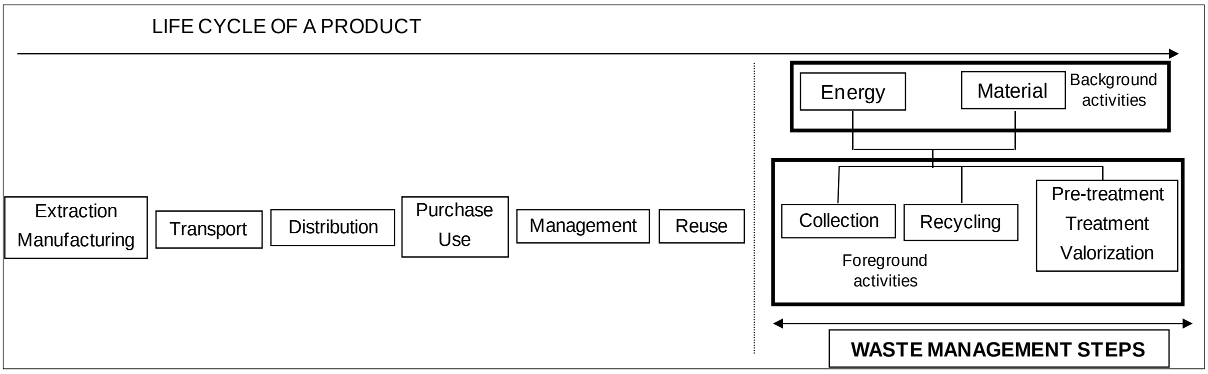

FIGURE 2 **Scope of LCA applied to MSW management**

In fact, it means that waste LCA does not include the associated emissions and consumptions to previous steps of life cycle (before product becomes a waste). Different steps of waste management are integrated (ADEME and AMORCE, 2005): collection, sorting, transfer, recycling, valorization (energy and material recovery) and disposal.

2.2.2 Limits in local issues assessment and spatial differentiation needs

The MSW management steps are decided and managed at local scale such as department, a territorial division in France. The local management is partly based on local environmental issues which are determined by local conditions. However, the issues of waste management are defined at national scale to answer national issues, but in local application, these issues are not the same. Local pressure on local decision-makers requires them to integrate local issues in their decisions.

To take into account spatiotemporal conditions, it is necessary to focus on the third step of LCA: impact assessment (FIGURE 1). To calculate a potential environmental impact in LCA, the *Site Generic* approach is used to model a standard impacted environment. According to this approach, the characteristics of the impacted environment do not influence the occurrence or intensity of the impact. This approach is relevant when global impacts are assessed (climate change, depletion of the ozone layer or depletion of fossil resources).

When local impacts are assessed, the *Site Generic* approach is irrelevant because "the location of a given source and the conditions of its surroundings strongly influence the fate of the emitted pollutants and their subsequent exposure possible sensitive receptors" (Potting and Hauschild, 2006). To integrate local impacts assessment in decision-making, it is necessary to integrate emission environmental conditions and target system characteristics. For these reasons, the need of spatial differentiation is real and can be done through a *Site Dependent* approach. The *Site Dependent* approach is built according to the theoretical environmental impact pathway which describes the impact mechanism. To improve the relevance of assessment of local impacts in LCA, integration of local parameters is essential. Concerning the toxicity impact, the main objective is to improve the reliability of its assessment.

3 A METHODOLOGY WITH SPATIAL DIFFERENTIATION FOR TOXICITY ASSESSMENT

3.1 Focus on toxicity issue in waste management

The treatment and disposal of MSW can produce the emission of certain pollutants in the natural environment and thus create a long term impact on human health. The toxicity impact is a biological consequence of the exposure to a substance which depends on molecular structure of the compound. It also depends on its interaction with the living matter. The toxic effect is considered linear and varies according to the dose. The dose/effect curve presents two thresholds: a maximum no effect concentration and the concentration levels off. Currently, there are three types of toxicity: acute toxicity (massive exposure and administration with immediate effects); subacute toxicity (related to regular absorption for several months and moderate effects which affect certain organs) and chronic toxicity (results from prolonged exposure to a variable rate and adverse effects that may occur several months to several years or even decades later).

In the case of waste management toxicity is widely taken up by opponents of incineration plant projects. Incineration is a controversial process of waste treatment. It converts waste to energy and leads to a reduction in fossil fuel consumptions thanks to energy recovery. However, waste incineration in France is associated with the case of the incinerator of Gilly-sur-Isère in 2001 where gas analysis revealed abnormally high levels of organic compounds and especially dioxins/furans.

3.2 The new methodology framework

This new methodology aims to develop a tool to more spatially assess the impact toxicity (in our case related to a waste incinerator plant). The main challenge of this new methodology is to integrate certain parameters of emission source and local affected environment. The developed methodology must follow the LCA methodology for the impact evaluation: classification and characterization of impact (FIGURE 3). The purpose is to calculate a toxicity potential impact in kg equivalent 1.4 dichlorobenzene (DCB).

This new methodology framework follows the impact pathway according to some descriptors throughout the *Site Dependent* approach. As in conventional LCA, classification and characterization steps are present, but in this new proposal their role and composition differ. The classification step is composed of emission characteristics, fate analysis, exposure analysis and effect analysis. The characterization step is only composed of the definition of factor characterization and a calculation of toxicity impact.

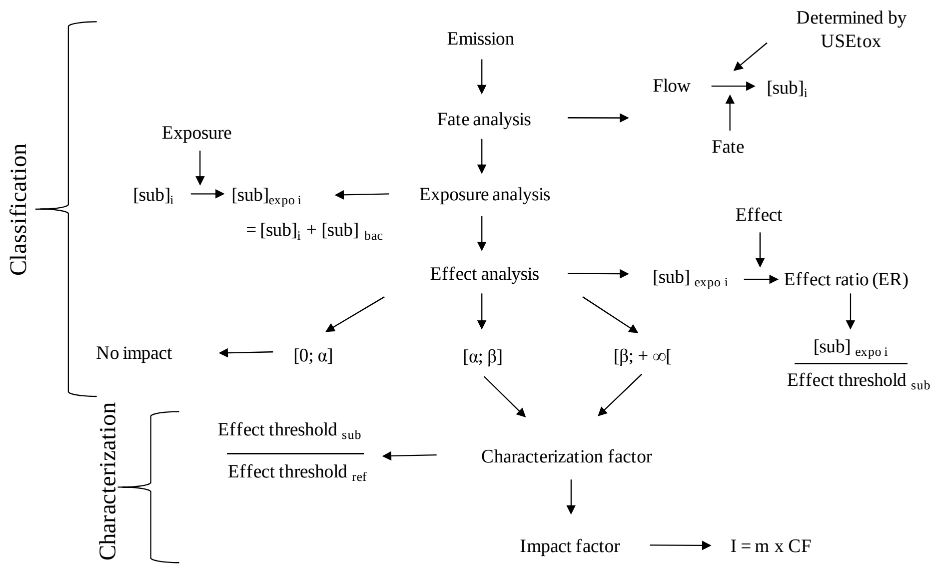

FIGURE 3 **The new methodology framework ([sub]: studied substance concentration; sub: studied substance; i: environmental compartment; bac: background concentration; ER: effect ratio; $[sub]_{expo\ i}$: exposure concentration of studied substance in environmental compartment i; α and β: classification factors; I: impact; m: mass and CF: characterization factor**

3.2.1 Emission characteristics

In the first step, the main characteristics of emission, substance and emission compartment must be defined: the type of emission (smokestack or diffuse emission), the frequency of emission (the frequency is supposed chronic), the period of emission (long or short), the emission compartment, the quantity of emission, the physical, chemical and biological characteristics of the substance and the physical, chemical and biological characteristics of emission compartment. The definition of these different points makes it possible to determine the importance of the toxicity impact which will be later calculated.

3.2.2 Fate analysis

The fate analysis is necessary so as to take into account degradation, transfer, diffusion phenomena of substance after the emission. The substance concentration that the population is exposed to can thus be determined. This fate analysis is realized thanks to the USEtox model. The USEtox model comes from a scientific consensus based on comparison of existing models and recommendations from different workshops (Rosenbaum, Bachmann, al., 2008). This model tends to become a reference multimedia model to assess toxicity in LCA through three environmental boxes (urban, continental and global) and some environmental compartments for each box (water, soil and air). It has been built from an Excel spreadsheet and is provided with a database for organic and inorganic (metals essentially) substances. This model is built according to the *Site Dependent* approach and makes it possible to calculate characterization factors for human toxicity and aquatic ecotoxicity in different environmental compartments (air, water and soil).

For local assessment of toxicity in this paper, the use of the USEtox model is restricted to the determination of the environmental concentration of the substance after emission through the fate analysis. In order to realize the fate analysis, it is necessary to adapt USEtox for a local scale. In default version, USEtox presents too macroscopic scales for local assessment of toxicity which needs microscopic scales.

3.2.3 Exposure analysis

The exposure analysis aims to include a spatial dimension to determine whether the geographic location of the emission and the presence of sensitive targets of emitted substance coincide. To integrate spatiotemporal conditions, the background concentration of this substance is added. The background concentration notion is defined as some amount of the compound which is already often present in environment, independently of the increasing concentration from the considered emission (Potting and Hauschild, 1997a). The addition of background concentration makes it possible to integrate local conditions of impacted environment and to obtain the exposure concentration.

3.2.4 Effect analysis

The effect analysis aims to determine the substance effect on a target. To determine this effect, we calculate an effect ratio according to exposure concentration (previously calculated) and effect threshold of studied substance. The threshold is determined by the "No Observable Adverse Effect Level" (NOAEL) which is determined by experiments or observations. It is defined as an exposure level at which there are no statistically or biologically significant increases in the frequency or severity of adverse effects between the exposed population and its appropriate control; some effects may be produced at this level, but they are not considered as adverse, or as precursors to adverse effects (Richards, 2008).

The effect ratio can take three possible values (FIGURE 3): comprise between 0 and alpha, between alpha and beta and between beta and plus infinity. The determination of alpha and beta parameters is initially arbitrary and pulls out of the binary classification and to define a fuzzy confidence interval. We consider no impact when the effect ratio is in first interval. For the last two intervals, we admit that there is a risk of toxicity impact and we calculate a characterization factor. Next, the toxicity impact quantification will be done by characterization step thanks to the fate and exposure analysis.

3.2.5 Factor and impact characterization

The factor characterization is calculated according to the effect threshold of studied substance and the effect threshold of a reference substance. For toxicity impact, the reference substance is commonly 1.4 dichlorobenzene (DCB). The factor characterization is the ratio between NOAEL of the studied substance and DCB. The impact characterization is calculated according to mass emitted in environment and factor characterization, the last parameter translating the potential effect of a substance on the environment. To explain more precisely the new methodology, an example is presented with TCDD emitted during incineration.

3.3 Case study with TCDD emission during MSW incineration

This case study aims to provide an example of toxic substance that can be analyzed throughout the new methodology. The substance TCDD is also known as Seveso dioxin, in reference to the accident at Seveso (Italy) in 1976 where a huge cloud of TCDD has covered the sky over a distance of up to 15 km. According to World Health Organization (WHO), it is one of the most toxic dioxins to humans, and is considered carcinogenic. The schema of the new methodology is dissected (FIGURE 3).

3.3.1 Emission characteristics

According to (Turconi, Butera, al., 2011), the Aarhus incinerator emission of TCDD is performed in the flue and its concentration is 108 ng TEQ.Mg $_{\text{waste}}$. The term TEQ means Toxic Equivalents Quantity and expresses the relative toxicity of each compound. The term dioxin groups several dioxins with a different toxicity degree. Each dioxin compound has a Toxicity Equivalent Factor (TEF) which indicates the toxicity degree compared to TCDD which is the reference value (equal to 1). From a paper of (Littarru, 2006), the repartition of the PCDD and PCDF in the emission of municipal solid waste is known in volatile phase (0.25%). It represents an emission of 0.27 ng TEQ/Mg $_{\text{waste}}$. From incinerator data (Riber, Bhander, al., 2008), it is possible to calculate an emission of TCDD of $7.9.10^{-11}$ kg/day.

The main physical, chemical and biological characteristics are already available in spreadsheet substance data in the USEtox model. The TCDD emission is assumed to be realized in French department such as Brittany. The local data of this territory are informed in landscape data spreadsheet of USEtox model. To analyze toxicity impact, an adaptation of USEtox is made through the dimension change of environmental boxes and compartments: a square of side 10 km (Fabre, Daniau, al., 2008) for the continental box (called local box) and a square of side 50 m for the urban box (called installation box). The environmental characteristics of the installation box are the same as the local box because USEtox is a nested model and the dimensions are very similar.

3.3.2 Fate analysis

The fate analysis determines the substance concentration which the population is exposed. From the inventory data ($7.9.10^{-11}$ kg/day), the adapted USEtox model calculate a steady-state atmospheric concentration in air local compartment of $1.8.10^{-24}$ kg/m^3.

3.3.3 Exposure analysis

According to data of air quality measurement, a background concentration of TCCD is used: $1.1.10^{-15}$ kg/m^3 in urban air (Comité de la Prévention et de la Précaution, 2004). So the exposure concentration is equal to $1.1.10^{-15}$ kg/m^3.

3.3.4 Effect analysis

According to the World Health Organization, the TCDD NOAEL is 1 pg/kg of body weight and the effect ratio is 0.2. Then the risk impact interval values are arbitrary:

- [0; 0.1[: we assume that when the effect ratio is in this range, there is no impact risk;
- [0.1; 1[: in this range, we assume the existence of fuzzy confidence interval where the impact risk is not clearly identified. This area overcomes the limitations associated with this new methodology (detailed in Part 4);
- [1; +∞ [: we clearly assume there is an impact risk in this zone.

For the TCDD example, the effect ratio is equal to 0,2 so according to the previous storage, we assume there is a toxic impact risk linked this TDCC emission.

3.3.5 Factor and impact characterization

To calculate the characterization factor, a reference substance for toxic impact is needed. In many characterization methods, the reference substance to assess the toxicity impact is the DCB. According to the toxicological data, the DCB NOAEL equals $5.8.10^{-4}$ kg/m^3. The characterization factor is calculated according to TCDD NOAEL (the emitted substance) and DCB NOAEL (the reference substance). The characterization factor is equal to $9.3.10^{-12}$. The impact characterization is calculated according to the characterization factor and inventory emitted mass of TCDD. So the toxic impact is equal to $7.4.10^{-22}$ kg eq DCB per day.

During this example of application, some limits were noticed in. The next part is dedicated to the explanation of the interest and limits of this new methodology.

4 DISCUSSION

This new methodology can address the problem of the localized toxicity impact assessment and in our case for MSW incineration plant, to allow us to compare toxic substances for different industrial plants and to bring environmental elements for decision support.

4.1.1 Cocktail effect of substances

Actual assessment methodologies do not take into account the cocktail effect. This effect can take three ways. Actually, the no interaction hypothesis is made. It means that the total effect is equal to the sum of the substance A effect and the substance B effect. There is a synergic phenomenon, where the total effect is greater than the sum of the substance A effect and substance B effect. The last interaction is antagonism phenomenon, it is mean the total effect is less than the sum of the substance A effect and the substance B effect. So these phenomena between substances could not be taken into account in this new methodology because of the lack of fundamental knowledge.

4.1.2 Nested model versus meshed model

To make a spatial differentiation, it is possible to use two types of models: a nested model or a meshed model. The nested model reduces the impacted environment to only three cells with different dimensions and characteristics. The meshed model divides the impacted environment into squares. The main difference between them is the purpose of the modeling. The mesh of the whole territory aims to identify the sensibility for each elementary cell whereas the nested boxes aim to present the average sensibility for each of the three types of elementary cell. In this assessment, the purpose is to realize a LCA with a focus on toxic impact assessment to bring some environmental elements into discussion during the decision-making. The toxic impact assessment needs a minimal spatial differentiation to be relevant. The nested model seems to be the most suitable model here because it can handle a particular case.

4.1.3 Rights to pollute?

The introduction of ternary classification limits the effects of the model limitations but shows a perverse effect. When the effect ratio is in the second area (between α and β), it is possible to identify rights to pollute. The first right is picture by the TCCD example. The background concentration is responsible for 99.9995% of the toxic impact. The incinerator contribution is barely measurable. But the calculation of effect ratio is made depending on environmental and background concentrations. To ensure a low effect ratio, the decision maker can prefer to set a plant in a less unpolluted area and to pollute a healthy area. The second right is disguised when the repartition of incinerator and background contributions is more delicate (ex: 50% /50%). The installation of incinerator plant in this area means a worsening of pollution level. The main goal of this new methodology is not to authorize or prohibit the installation of incinerator plant but is to determine the impact of MSW incinerator if it is installed on the territory x or y.

5 CONCLUSION

In the French context of MSW management, local decision-makers use an environmental evaluation tool called LCA, to assess environmental impacts of the different scenarios of waste management. Nevertheless, the global character of LCA leads to irrelevant assessment of local impacts because of their dependence of local characteristics of the emission source and the impacted environment. However, in waste management and decision-making, the place of local impacts such as the effects on human health is very important and their assessment should be as robust as possible.

To assess local impacts with LCA it is necessary to take into account spatiotemporal conditions of emission, environmental conditions and target system characteristics. For that, the need of spatial differentiation for the impact assessment is real and can be done thanks to the *Site Dependent* approach and the USEtox model uses. To compare classical LCA, the classification step is made more complex taking into account spatiotemporal characteristics in order to determine a fuzzy confidence interval for a potential impact risk. If a risk of impact exists, the calculation is realized through a new characterization factor.

A case study presents an example of TCDD emitted during MSW incineration. This example reveals some limits concerning the cocktail effect between substances which is not take into account and the type of model used (nested or meshed). The result analysis identifies limits concerning the TCDD emission. In first time, the TCDD concentration related incineration is negligible compared to the background concentration. In second time, the emission data of TCDD are too low because the regulatory and incinerator technical improvements have resulted in a significant decrease of dioxins and furans emissions. Among the substances being emitted by waste incinerators, there are the NO_x substances, especially NO_2. However, the application of the new method through USEtox is not possible for NO_2 because it is an inorganic substance with a short lifespan (§ 3.2.2). With the characterization method called CML2 we calculate the toxicity impact related to this substance. However, the use of this characterization method makes it possible to take into account neither the local conditions of the emission nor the impacted environment. For this example, the NO_2 impact characterization is equal to 801 kg eq DCB. The impact characterization of TCDD compared to NO_2 is order of magnitude 24. However, the spatiotemporal characteristics are not integrated in the calculation of NO_2 substance. This reinforces our idea of evaluating the inorganic substances like NO_2. The developed methodology aims to include the toxicity impact, and in general way, the local impact assessment, in the decision-making so as to integrate the assess elements in the debate.

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(140) ASSESSING GHG EMISSIONS FROM SLUDGE TREATMENT AND DISPOSAL ROUTES – THE METHOD BEHIND ^{G}ESTABOUES TOOL

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EXECUTIVE SUMMARY

In 2007, 1 100 000 tons of sewage sludge were produced in France. This figure is constantly increasing and sludges have to be eliminated. Four disposal routes are currently possible: land spreading (directly or after composting), incineration, incineration with household wastes and landfilling. These different disposal routes as well as the sludge treatments produce greenhouse gases (GHG). To help stakeholders to better understand the carbon footprint of sludge treatment and disposal options, we developed a tool called $^{G}E_{S}$TABoues.

This paper aims to present the underlying methodology used to quantify material and energy flows as well as GHG emissions all along the sludge treatment and disposal processes implemented in this tool. GHG emissions generated by our system are quantified for x tons of sludge produced by a wastewater treatment plant of x per-captia-equivalents (PCE) during one year.

The carbon footprint method we developed is adapted to sludge treatment and disposal processes and based on the "Bilan Carbone®" method. The "Bilan Carbone®" method is a general method used to quantify GHG generated from all physical processes which are necessary for any activity or human organization (ADEME, 2009). In our method, three GHG are recorded: carbon dioxide, methane and nitrous oxide. Biogenic carbon was not taken into account but its sequestration was for two types of disposal routes (land spreading and landfilling). For each process involved in the sludge treatment and disposal routes system, three types of emissions are considered: direct, indirect and avoided emissions.

(i) Direct emissions are generated by each process (storage, thickening, anaerobic digestion, composting, land spreading, incineration, incineration with household wastes, landfilling).

(ii) Indirect emissions are due to energy and chemical consumptions (combustible or electricity) to operate each process. Transport emissions (for consumables, sludges and ashes) and civil engineering emissions were taken into account. The first ones were calculated for one ton of goods transported on one kilometre (t.km) and the second ones were the toughest to implement in $^{G}E_{S}$TABoues tool. After a literature review, two main methods were identified. Renou (2006) considers that the most applicable methodology is to consider mass of all civil engineering and electrical/mechanical equipments whereas Doka (2007) considers that civil engineering emissions are defined by wastewater treatment plant for 5 classes of plants. We propose an intermediate methodology to assess these emissions : for each process, components (concrete, cast iron, steel…) of involved machineries and buildings were modelled for 3 sizes of wastewater treatment plants (<10 000 PCE, 10 000 – 100 000 PCE, >100 000 PCE).

(iii) Avoided emissions are generated when products are not used and replaced by recyclable products (heat, electricity, fertilizer…).

GHG data were collected through a literature review for each type of emissions and each process of sludge treatment and disposal routes. All collected data were implemented in $^{G}E_{S}$TABoues, developed with VBA Excel to quantify GHG emissions generated by a wastewater treatment plant of x PCE.

1 INTRODUCTION

1.1 Background

During the last decades, European institution establishes regulations which aim at protecting the environment from the adverse effects of the collection, treatment and discharge of waste water (Directive 91/271/EEC of 21 May 1991 concerning urban waste water treatment) and at maintaining and improving the aquatic environment (Directive 2000/60/EC of 23 October 2000 establishing a framework for community action in the field of water policy). These regulations were transcribed in French laws leading to important constraints on the waste water treatment plants (WWTP).

The environmental cleaning up of water in WWTP inevitably produces sludges that need to be treated and eliminated. Waste water sludge treatment can be an environmental problem as contaminants in water may land up in the sludge. The application of the above-mentioned European and French laws leads to an increase in sludge production (from 580 000 in 2000 to 1 300 000 dry matter tons in 2005; ADEME, 2001) which is eliminated according four main disposal routes: land spreading (directly or after composting), incineration, incineration with household wastes and landfilling.

A recent study (Reverdy and Pradel, 2010) shows an increase in sludge land spreading from 60 to 70% with a significant evolution of composted sludge spreading. As no sludge was composted in 2000, 23 % was spread after composting in 2007 and 46% was directly spread. Incineration slightly increases from 15 to 18% while landfilling significantly decrease from 25 to 12% in 2007.

The environmental assessment of sludge treatment and disposal routes is therefore a big concern as it can be used by stakeholders (WWTP managers, local authorities…) to choose the appropriate alternative in sludge treatments or disposal routes.

1.2 Research objectives

Several authors studied the environmental impacts of WWTP and sludge treatment processes by using Carbon footprint or Life Cycle Assessment methods in order to compare the most favourable alternatives in sludge disposal routes from an environmental or energetic point of view (Houillon and Jolliet, 2005; Lundin et al., 2004; Vandenbossche et al., 2005). These studies are highly instructive but the results greatly depend on the hypotheses and the scenarios analysed. These studies cannot be sometimes compared as the studied system and the functional unit used are different. This leads to the conclusion that the results of these studies cannot be generalized. Moreover, very few studies were conducted to assess the environmental impact regarding each process involved.

This is the underlying reason of the creation of a Carbon footprint calculation tool ($^{G}E_{S}$TABoues) designed to help stakeholders to better understand the impact of sludge treatment and disposal options on Global Warming.

This paper aims to present the underlying methodology used to quantify the material and energy flows and the related greenhouse gases (GHG) for each processes involved in sludge treatment up to those generated by each disposal routes.

The first part of this paper will present the method used (i.e. the system boundaries and the functional unit). The second part of the paper deals with the GHG data collection for each process, a special focus will be done on the transport and the infrastructure data collection. Then, the last part of the paper will show the type of results provided by the tool. Some example of the use of $^{G}E_{S}$TABoues tool will be presented in Reverdy and Pradel (2012).

2 METHODOLOGY

2.1 Carbon footprint method

Carbon footprint general framework

The Carbon footprint is defined as the total amount of greenhouse gases produced to directly and indirectly support human activities, usually expressed in equivalent tons of carbon dioxide (CO_2). It is a common method to calculate the impacts of human activities on Global Warming (IPCC, 2006). In France, the ADEME (French Environment and Energy Management Agency) developed a carbon footprint framework called “Bilan Carbone®” which allows quantifying and assessing the GHG emissions for human activities or organisations (ADEME, 2009). The fundamental principles of this method lie in equally considering the GHG emissions directly generated by the studied activity and the GHG emissions taken place outside of the studied activity but essential for it. The “Bilan Carbone®” method takes into account GHG directly emitted in the troposphere by the studied activity but not the gases produced after chemical reactions in the atmosphere.

As the quantification of these gases cannot be directly measured, they are estimated by calculating GHG emissions of each processes involved in the studied activities. The amount of each gas is then converted with an emission factor in CO_2 equivalent (CO_{2eq}) according to their Global Warming Potential (GWP), a relative measure of how much heat a GHG traps in the atmosphere over a specific time interval, commonly 20, 100 or 500 years (IPCC, 2006). The considered gases and their GWP are presented in Table 1.

TABLE 1 **Considered gases in carbon footprint and their Global Warming Potential at 100 years (GWP_{100})**

Common name	**Formula**	**Life span (year)**	**Radiative efficiency ($W.m^{-2}.ppb^{-1}$)**	**GWP at 100 years**
Carbon Dioxide	CO_2	(note)[a]	$1.4*10^{-5}$	1
Methane	CH_4	12	$3.7*10^{-4}$	25
Nitrous oxide	N_2O	114	$3.03*10^{-3}$	298
CFC compounds	$C_nCl_mF_p$	45 – 1 700	0.18 – 0.32	4 750 – 14 400
HCFC compounds	$C_nH_mCl_pF_q$	1.3 – 17.9	0.14 – 0.22	77 – 2 310
HFC compounds	$C_nH_mF_p$	1.4 - 270	0.09 – 0.28	124 – 14 800
PFC compounds	C_nF_{2n+2}	2 600 – 50 000	0.10 – 0.56	7 390 – 10 300
Sulfur hexafluoride	SF_6	3 200	0.52	22 800
Nitrogen trifluoride	NF_3	740	0.21	17 200

[a] The CO_2 response function used in this table is based on the revised version of the Bern Carbon cycle model used in Chapter 10 of 2006 IPCC report using a background CO_2 concentration value of 378 ppm.

However, “Bilan Carbone®” results can also be expressed in carbon equivalent (C_{eq}). This unit only considers the carbon molecule in the CO_2 compound, i.e. C_{eq} (kg) = 12/44 * CO_{2eq} (kg).

The CO_2 emissions can either been from fossil or biogenic origin. Biogenic CO_2 emissions are belonging to short carbon cycle. They are involved in photosynthesis or thermal or biological ways of oxidation so as the emitted biogenic CO_2 is rapidly incorporated in the carbon cycle. These biogenic emissions are not taken into account in national protocols as they are considered (by convention) as “carbon neutral” (GWP equal to zero). As fossil CO_2 emissions come from the hydrocarbon combustion, stored in the Earth surface from million years, they are belonging to the long carbon cycle. Releasing this fossil carbon by combustion increases the amount of CO_2 in the atmosphere and these emissions need to be accounted in “Bilan Carbone®” method.

Three types of emissions are considered within the “Bilan Carbone®” method: direct, indirect and avoided emissions. The direct emissions are emissions directly produced by the process or the studied activity (ex: CO_2 emissions due to fuel combustion during the activity). The indirect emissions are produced by processes needed by the activity but not directly generated by the activity (ex: CO_2 emissions during transport of goods). Avoided emissions are generated when products are not used and replaced by recyclable products (heat, electricity, fertilizer…).

Adaptation to sludge treatment and disposal routes

The « Bilan Carbone® » method is generally applied in France to assess the GHG emissions of industrial or tertiary activities. The method is not very precise concerning the way to account GHG emissions in WWTP, limiting the GHG emissions only to CH_4 emissions of the waste water released in the environment without treatment. No information is given to the assessment of sludge treatment in WWTP (ADEME, 2010a). Some methodological principles of a carbon footprint assessment in WWTP are given by ASTEE (ASTEE, 2009). The ASTEE method is based on the ADEME’s “Bilan Carbone®” but takes into account the entire WWTP, including the sludge disposal as end-of-life of WWTP wastes. No distinction is realized between the water treatment and the sludge treatment so as it is not possible to compare the environmental impact of sludge treatment and the best disposal routes.

To fill in these gaps, we proposed a method to account GHG emissions for sludge treatment and disposal routes. The first step was to propose methodological choice regarding the studied system boundaries and the functional unit used to quantify the GHG emissions.

The system boundaries are limited to the sludge treatment including all the possible disposal routes (Figure 1). Two types of analyses are done to assess GHG emissions: a detailed analysis for all the sludge treatment processes and disposal routes and a global analysis for the consumables, transport and infrastructure involved in the system. The GHG emissions are assessed for the total amount of sludge produced by a WWTP of x Per-Captia Equivalent (PCE).

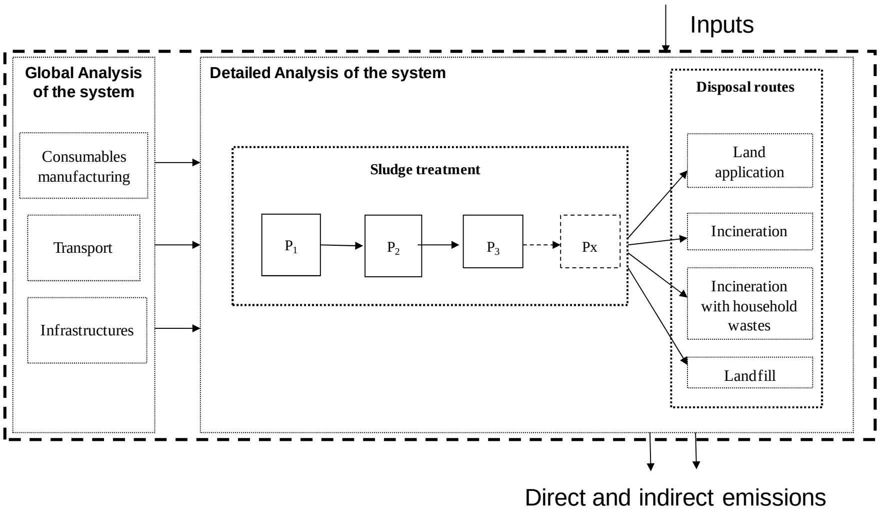


FIGURE 1 **System boundaries**

The factor x is greatly dependent on the WWTP treatment capacity and so of the PCE. We propose to assess the GHG emissions on a whole year as it is a common temporal unit for stakeholders (WWTP manager or local authorities). It can be repeatable and simplify data collection. A temporal allocation will be done if GHG emissions of sludge treatment processes are emitted on more than one year (example of reed drying beds).

The main GHG emissions accounting in WWTP system are fossil CO_2, CH_4 and N_2O. CO_2 emissions are emitted during the organic matter degradation all along the sludge treatment processes. As they are from biogenic origin, they are not taken into account in our method.

As for "Bilan Carbone®", direct, indirect and avoided emissions are taken into account in our method. Direct emissions directly originate from sludge treatment processes or disposal routes. A distinction is done between the GHG emissions due to the sludge (biological degradation…) and the other ones (emissions during fuel combustion or electricity consumption needed to run the different sludge treatment processes or occurring in sludge disposal). Indirect emissions are due to energy and chemical consumptions (combustible or electricity) to operate each process. Transport emissions (for consumables, sludges and ashes) and civil engineering emissions are taken into account. They are discussed in the following section. Avoided emissions are accounted when processes are substituted by other processes. The avoided emissions in sludge treatments and disposal routes can be generated by energy or material substitutions:

- Thermal or electric energy production from biogas: avoided emissions are due to emissions that will have take place for an equivalent non-renewable amount of energy,
- Use of sludge as fertilizers: avoided emissions are those generated by the amount of substitute mineral fertiliser production and its spreading,
- Use of sludge as a combustible or as mineral portion in cement kilns: avoided emissions are due to emissions that will have take place for an equivalent non-renewable amount of energy or the production of the substituted raw materials.

2.2 Sludge treatment processes and disposal routes

Based on OTV (OTV, 1997), 5 types of sludge can be produced in WWTP depending on the type of water treatment.

Sludges from primary treatment are classified in the A class. Sludges from secondary treatment are classified in B1 or B2 classes, the difference lies in the presence of a primary treatment (B2 class) or not (B1 class). The blending of primary and secondary sludges produces sludge that are classified in the C class while a stabilization process provides sludges classified in the D class.

These three types of sludge (primary, secondary and tertiary) are then treated to decrease their water content (by thickening, dewatering and drying processes), stabilized and sanitized before their valorisation through land application, incineration or landfilling.

The different sludge treatment processes and the possible disposal routes are presented in Figure 2. The GHG emissions for each process were quantified and implemented in ^{G}E$_{S}$TABoues tool.

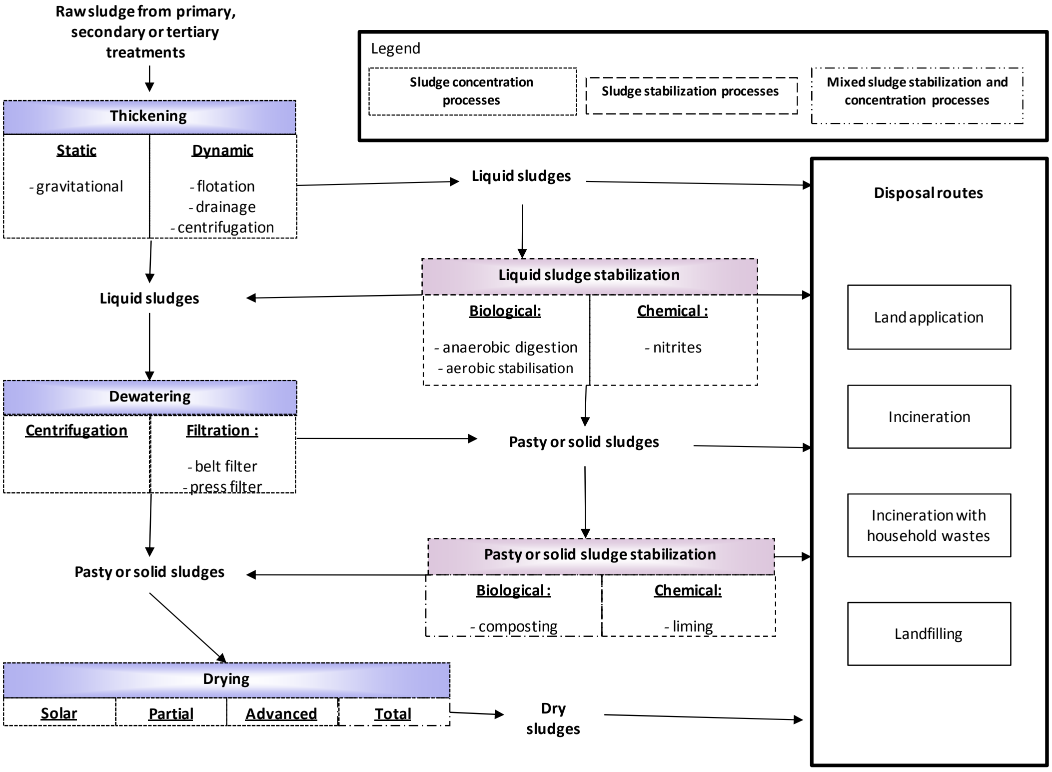


FIGURE 2 **Studied sludge treatment processes and disposal routes**

2.3 GHG data collection

A literature review was conducted to collect required information to assess the carbon footprint of the sludge treatments processes and disposal routes (Reverdy and Pradel, 2011). Data on energetic and polymer consumptions and GHG emissions were collected for each process involved in the studied system. The data analysis shows a great variability for a same type of emission (example: kg CO_2eq/ton of consumed polymer). As we implement a single value in $^{G}E_{S}$TABoues tool, we choose to calculate an average value when values collected from literature are of the same order of magnitude. When the variability is too high, extreme values were excluded and the average value calculated on the remaining data.

Data collection was done regarding two possible uses of $^{G}E_{S}$TABoues tool. On one hand, the user provides own data and collected data may be used as reference values. If the user has no specific data for his WWTP, collected data will be used to assess the carbon footprint of the selected sludge treatments and disposal routes.

GHG emissions for each studied process

Direct GHG emissions were generated for storage, reed drying beds, anaerobic digestion, composting, land application, incineration, incineration with household wastes and landfilling. These emissions are summarized in Table 2.

Indirect GHG emissions, expressed in CO_{2eq}, are generated for each process using inputs such as electricity, gas, light and heavy fuel, lime, soda, polymer, active carbon… These emissions take into account either the GHG emissions released during the input production as well as those occurring during their transport up to the WWTP. The mineral fertiliser production generates indirect GHG emissions ranging from 0.121 to 1.693 kg of CO_{2eq}/kg of nutrient (N, P or K). The variability is explained by the different technologies of manufacturing used and by the form of the produced mineral fertiliser (simple, binary or ternary). The GHG emissions generated by the production of these inputs are shown in Table 3.

TABLE 2 **Direct GHG emissions regarding the sludge treatment and disposal routes studied processes**

Processes	**Emissions**	**Unit**	**Emission factor**	**Source**
Storage	CH_4	Kg/kg BOD_5	Open silo: 0 < 2 m silo in anaerobic condition: 0.12 < 2 m silo in anaerobic condition: 0.4	Sylvis, 2009 Mallard et al, 2007 Gac et al, 2006 Record, 2008 ADEME, 2005 IPCC, 2006 Citepa, 2010 Pacaud et al, 2009 Gac et al, 2010 EPE, 2006 Shimizu et al, 2007 Doka, 2007
Reed drying beds	N_2O CH_4	Kg/PCE/an	0.0518 0.0453	
Anaerobic digestion	CH_4	Kg/ton	0.18	
Composting	CH_4 N_2O	Kg/ton	2.9 0.4	
Land application	N_2O	Kg/ton	Liquid sludge: 0.0294; Solid limed sludge: 0.05; Composted sludge: 0.05; Dry sludge: 0.2875	
	N_2O	Kg/ha	Other type of sludge and mineral fertilisers: $N_{applied}$ * [0.0157 + 0.3*0.0118 + 0.2*0.0157]	
Incineration	N_2O	Kg/ton	If combustion temperature (t°) is known : [N_{total} * (161.3 – 0.14 * t°)/100]*1.57 If t° is unknown: 1.64	
Incineration with household wastes	CO_2 N_2O	Kg/ton	390 0.092	
Landfilling	CH_4	Kg/ton	If biogas is captured: sludge C * 0.13 If biogas is released: sludge C * 0.43	

TABLE 3 **Indirect GHG emissions regarding the inputs used for each process**

Type of inputs	**Type of emissions**	**Unit**	**Emission factor**	**Source**
Electricity	CO_{2eq}	Kg/kWh	0.089	IRH, 2009 OTV, 1997 Degremont, 2005 Pradel, 2010 Hospido et al, 2005 Record, 2008 ADEME, 2010b
Gas	CO_{2eq}	Kg/kWh	0.32	
Light fuel	CO_{2eq}	Kg/kWh	0.24	
Heavy fuel	CO_{2eq}	Kg/l	2.662	
Fuel for tractors	CO_{2eq}	Kg/l	3.2	
Polymer	CO_{2eq}	Kg/kg	4.25	
$FeCl_3$	CO_{2eq}	Kg/kg	0.33	
Slaked lime	CO_{2eq}	Kg/kg	0.975	
Quicklime	CO_{2eq}	Kg/kg	1.04	
Caustic soda	CO_{2eq}	Kg/kg	1.17	
Activated carbon	CO_{2eq}	Kg/kg	6	

GHG emissions for transport

The transport process in $^{G}E_{S}$TABoues tool takes into account the transport of inputs from the suppliers storage place to the WWTP and then from the WWTP to the disposal place (either the field, the incinerator or the landfill). The GHG emissions of transport were calculated according the following hypotheses:

- CO_{2eq} emission calculation is done for the ton.km unit, i.e. the emissions generated to transport one ton of product on one kilometre.
- We assume that a single type of transport is done for one type of input. For example, the transport of polymer cannot be done with both a 2.5 ton truck and a 12 ton truck.
- Different inputs cannot be transported at the same time with the same vehicle.
- Transport of energetic consumables such as electricity, fuel or gas is not taken into account as it is already accounted in indirect GHG emissions.

The main transport modelled in $^{G}E_{S}$TABoues is a transport by truck as for "Bilan Carbone®" method (ADEME, 2010c). Sludge transport from the WWTP to the field is done according the method proposed in Pradel (2010). For liquid sludge, transport is done directly from the WWTP to the field with a tractor and a slurry tanker (4 processes are modelled). The other types of sludge are transported from the WWTP to the intermediate storage with a truck (3 processes modelled) and then to the field with a tractor and a spreader (3 processes modelled).

GHG emissions for infrastructure

After a literature review, two main methods were identified. Renou (2006) considers that the most applicable methodology is to consider mass of all civil engineering and electrical/mechanical equipments whereas Doka (2007) considers that civil engineering emissions are defined by wastewater treatment plant for 5 classes of plants. We proposed an intermediate method to take into account infrastructures in $^{G}E_{S}$TABoues tool. We estimate the amount of material needed (such as concrete, steel…) of all civil engineering and electrical/mechanical equipments involved in sludge treatment and disposal routes for 3 classes of WWTP: < 10 000 PCE (small), between 10 000 and 100 000 PCE (medium) and more than 100 000 PCE (big).

GHG emissions were calculated according to the whole life cycle of the infrastructure and the total amount of produced sludge. They are expressed in kg of CO_{2eq} /unit/ton. An example of infrastructure calculation is done in Table 4. Complete infrastructure GHG emissions can be found in Reverdy and Pradel (2011).

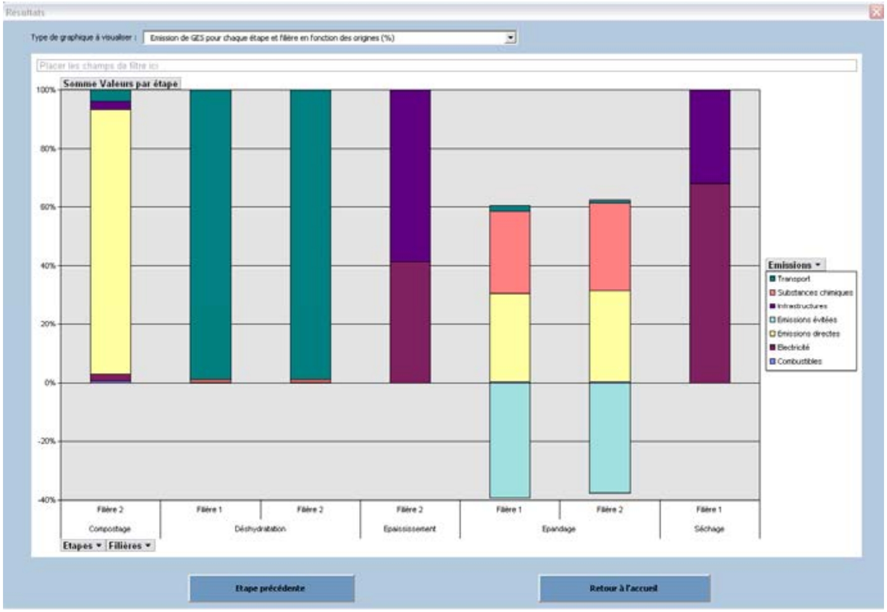
TABLE 4 **GHG emissions for sludge treatment and disposal routes infrastructures**

Infrastructure	**Capacity**	**Life span (years)**	**Description**	**Modelled processes**	**Kg CO_{2eq} /unit/ton**
Static thickening	Small	30	Thickener, diameter: 5 m, capacity: 70 m^3	Concrete, Steel, Cast iron, Stainless steel	0.0245
	Medium	30	Thickener, diameter: 12 m, capacity: 450 m^3		0.0109
	Big	30	Thickener, diameter: 20 m, capacity: 1250 m^3		0.0096
Press filter	Small	15	Press filter, 50 plates 500*500 mm, capacity: 290 l, total weight: 3 156 kg	Cast iron, Polypropylene, stainless steel	0.2674
	Medium	15	Press filter, 100 plates 1000*1000 mm, capacity: 2400 l, total weight: 12 385 kg		0.2103
	Big	15	Press filter, 150 plates 1500*2000 mm, capacity: 10000 l, total weight: 59 090 kg		0.4943
Incineration	Medium	40	Fluidized bed incinerator, total weight: 65 970 kg, height: 10 m, diameter: 3.45 m	Refractory steel, refractory fireclay, sand, concrete	0.0188

3 RESULTS PRESENTATION

All collected data presented in the previous section were implemented in $^{G}E_{S}$TABoues, a tool developed with VBA Excel to quantify GHG emissions generated by a wastewater treatment plant of x PCE. An example of results obtained with $^{G}E_{S}$TABoues tool is presented in Reverdy and Pradel (2012).

Two types of results are obtained with $^{G}E_{S}$TABoues tool. The first one is different bar charts (an example is given Figure 3) and a mass/energy balance for the entire studied sludge treatment and disposal route (Figure 4).



Type de graphique à visualiser : Emission de GES pour chaque étape et filière en fonction des origines (%)

FIGURE 3 **GHG emissions for each process and sludge treatment regarding the GHG origin (in %) ($^{G}E_{S}$TABoues tool screen shot)**

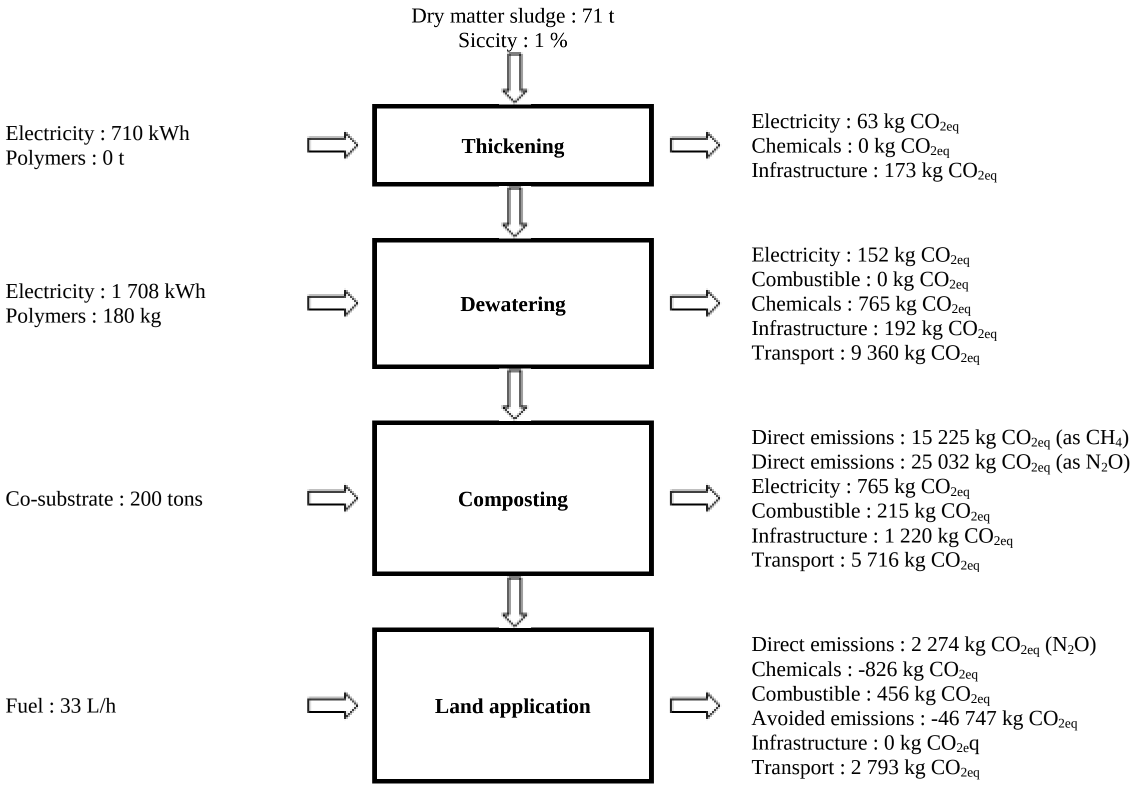


FIGURE 4 **Example of mass and energy balance obtained with $^{G}E_{S}$TABoues tool**

4 CONCLUSION

This paper proposes a method to assess GHG emissions and Global Warming impact assessment of sludge treatment and disposal routes. Its originality lies in the consideration of each process involved in the stream. However, this method needs to evolve as biogenic emissions are not taken into account while it appears from a great importance to assess the environmental impact of processes based on biological treatment. Accounting biogenic CO_2 emissions will provide a better understanding of process efficiency in sludge treatment and their inclusion in Global Warming assessment is currently a big concern in research development and for worldwide environmental agencies (EPA, 2011).

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Session 22

(30) GHG EMISSIONS DURING THE COMPOSTING PROCESS AS A FUNCTION OF THE AERATION STRATEGY

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EXECUTIVE SUMMARY

Composting is an aerobic thermophilic biodegradation process that requires oxygen to stabilize the organic wastes and optimal moisture content for the microorganisms development (Haug, 1993). Several parameters as C/N ratio, material porosity, moisture content and aeration rules for the oxygen supply must be analyzed and controlled to optimize the process development. Frequently, aeration is defined as the most important factor for the process performance. In general, the main aeration systems used are: forced aeration, physical turning and natural convection. Different aeration strategies of the first system have been proposed with the common goal of optimizing the biological activity along the process. For this reason, most aeration regulations are based on the temperature and/or oxygen content measurements. Often, the airflow supply is also regulated by means of fixed cycles. Recently, the feasibility of oxygen supply according to the biological activity evolution (measured as Oxygen Uptake Rate (OUR)) has been studied by Puyuelo et al. (2010). Briefly, it consists on finding, for every moment of the process, the airflow that increases the oxygen uptake rate through the comparison of consecutive OUR values together with the airflow applied.

In order to compare the process performance and the greenhouse gases emissions as a function of the aeration strategy, three pilot composting trials with the same organic waste but different aeration systems were undertaken. Oxygen, aeration cycles and the new OUR control were the strategies studied. Temperature control was not undertaken since the results obtained in a previous work (Puyuelo et al., 2010) showed highest energy consumption. The experiments were carried out during 20 days and temperature, oxygen content and airflow supplied were continuously measured. Daily, a sample of exhaust gases was taken to quantify by gas chromatography the N_2O, CH_4 and global Volatile Organic Compounds (VOCs) content. Different VOCs were also identified by mass chromatography. Ammonia content was measured once a day with a specific sensor. Moreover, the stability degree of initial and final samples obtained was determined by the Dynamic Respirometric Index.

Similar temperatures profiles were achieved in all experiments although the oxygen content was oscillating during the most part of the oxygen and cyclic controls. The progressive airflow evolution of the OUR control avoided this severe oxygen changes. In addition, this system achieved the highest oxygen uptake during the process and most stable end-product. Regarding the GHG emissions, the gradual airflow changes also prevented the high peaks detected under oxygen and cyclic control, when maximum airflow was applied. In Table 1 the global emissions of each contaminant are summarized. As observed, the lowest global emissions were detected by means of the OUR control. The lowest CH_4 emission showed that the systems with intermittent airflow increased the anoxic zones. Maximum VOCs measures were around 1400 mg m^{-3} for oxygen and cyclic controls and they were below 700 mg m^{-3} during the OUR control. On the contrast, ammonia and N_2O measures could not be compared since no clear evolution was observed.

All measurements permitted to demonstrate that the OUR control optimizes the biological activity, the material stabilization and reduces the gaseous emissions associated to the composting process.

Table 1. VOCs, CH_4, N_2O and NH_3 emissions for each control experiment. OFMSW: Organic Fraction of MSW.

Emissions	kg VOCs Mg^{-1} OFMSW	kg CH_4 Mg^{-1} OFMSW	kg N_2O Mg^{-1} OFMSW	kg NH_3 Mg^{-1} OFMSW
Oxygen control	0.37	0.10	> 0.0038	> 0.6
Cyclic control	0.40	0.20	0.0152	> 1.0
OUR control	0.15	0.08	0.0094	0.7

1. INTRODUCTION

The aeration rate is a crucial factor to guarantee the aerobic conditions during the composting process. The main objective of this parameter is not only associated to maintain an optimal biological activity but it can also be a critical parameter regarding the gaseous emissions of NH_3, N_2O, CH_4 and VOCs (Haug, 1993; Smet et al., 1999). In general, it is observed that the forced ventilation can be a main factor to reduce the contaminant gaseous emissions.

Today, there exist different strategies of forced aeration. One of the most implemented strategies at industrial facilities is a system based on predefined cycles of aeration. Another usual system is an oxygen feedback controller. In this case, a maximum and minimum or closed airflow are provided as a function of the interstitial oxygen content of the mass. In some cases, the airflow is also supplied as a function of temperature of the mass, although this technique does not guarantee the supply of the necessary oxygen. Some recent studies have proposed other strategies based on complex models of the process (Papadimitriou et al., 2010; Giusti et al., 2010). These can be defined as promising strategies but its implementation at industrial scale can be difficult and costly. One of the last studies about the process control showed a new control strategy based on the continuous OUR measure (Puyuelo et al., 2010). This system could be an alternative option to develop the process avoiding the disadvantages observed through the actual implemented systems.

During the biological process the aerated forced system determines the gaseous emissions, which comes from the waste or from the microbial metabolism (Pagans et al., 2006). Osada et al. (2000) in their studies about slurry composting demonstrated that a high airflow decreased the CH_4 and N_2O emissions due to the minimization of anaerobic zones (as also observed by Fukumoto et al., 2003) but increased the NH_3 emissions. This Increase was also confirmed by other authors (de Guardia et al., 2008; Kim et al., 2009; Shen et al., 2011). Again, optimum oxygen content limited the formation of anaerobic zones avoiding the generation of intermediates products of the anaerobic metabolism (Scaglia et al., 2011). Some studies about the different strategies of forced aeration have concluded that, comparing systems of continuous aeration with an intermittent aeration; the former reduces some GHG emissions (Keener et al., 2001).

In this sense, the main goal of this work was to determine and to compare the evolution and the cumulative emissions of NH_3, N_2O, CH_4 and VOCs obtained with different forced aeration strategies, which were the oxygen feedback controller, the cyclic controller and the OUR controller developed in a previous work.

2. MATERIALS AND METHODS

2.1. Composting pilot reactor

The results presented in this work were obtained from tree composting experiments in which different airflow regulation systems were applied. All of them were conducted at pilot scale using an adiabatic cylindrical reactor with an operating volume of 50 L. Approximately 25 kg of the waste selected were treated in each experiment. Two geometrically identical reactors were used in parallel. A scheme of one pilot reactor can be seen in Figure 1.

The reactor walls were thermally isolated with polyurethane foam in order to avoid heat losses. A perforated plate was fitted into the bottom of the reactor to support the material, to help leachate removal and to optimize the airflow circulation. Two orifices were situated at the bottom cover of the reactor, one to introduce air from a compressor and other for leachate removal. Two more orifices were situated at the top cover. One hole was to insert the Pt-100 sensor for temperature monitoring (Desin Instruments, Barcelona, Spain), which was placed at middle height of the material matrix. The other orifice was used to remove the exhaust gasses in order to analyze the gaseous concentration. O_2 concentration was measured continuously with an oxygen sensor (Xgard, Crown, UK) placed after a refrigeration chamber to avoid wet gasses passing through the gas analyzer. Instead, VOCs, N_2O and CH_4 concentrations were determined in a gas chromatograph after collecting the gas sample in a Tedlar® bag. NH_3 concentration was measured with a specific sensor.

The data acquisition and control system was composed by an acquisition chassis (cDAQ-9172, National Instruments, USA) connected to a PC and using LabView 8.6 software (National Instruments, USA). Temperature, exhaust oxygen gas concentration, and inlet airflow were the parameters monitored during the experimental trials. Temperature probe and oxygen sensor were connected to the data acquisition chassis. Instead, the input and output electrical signals of the flow meter were directly connected to the PC through an RS-232 serial port. All the data were recorded and shown in a graph or in the program interface from which different control systems could be programmed.

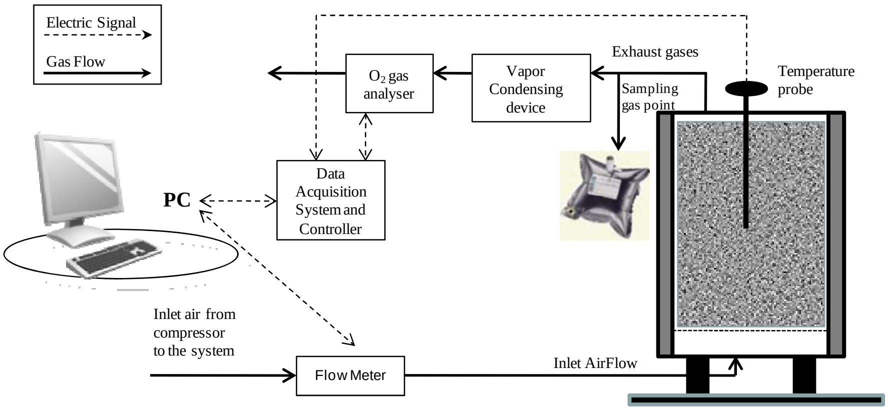

FIGURE 1. Experimental set up of the composting pilot reactor.

2.2. Composting material

The waste used in all these experiments was source-selected Organic Fraction of Municipal Solid Waste (OFMSW) mixed with pruning waste as a bulking agent. The mixture (with a volumetric ratio 1 to 1) was collected in a composting plant located in Manresa (Barcelona, Spain). A total of 200 kg were collected in order to carry out the experiments with the same material. After collection, a homogeneous sample was used for waste characterization and all the remaining waste was frozen at -18 ºC. Before starting-up each composting experiment, the material was removed from the freezer and thawed in the laboratory at room temperature for 24 hours. In this period, it was considered that the biological activity of the waste as it was confirmed by a recent study (Pognani et al., 2011).

2.3. Composting controllers

Three different control strategies were studied and described below. In all cases, airflow was used as the manipulated variable. Two different closed-loop controllers and a third system based on a timed ON-OFF control configuration were tested. The lowest airflow applied at the reactor was never below 0.2 L min^{-1} ($8\cdot10^{-3}$ L min^{-1} kg^{-1} TM) to overcome an excessive pressure drop of the reactor and to obtain a constant gas flow for oxygen monitoring purposes. Experimentally, each control strategy was applied in a 20 days composting experiment.

2.3.1. Oxygen feedback control

The controller was based on the airflow manipulation by means of the oxygen content measured in the exhaust gas. It was necessary to establish an O_2 set point to maintain the system in favorable conditions. From the literature found, the oxygen set point was fixed between 11.5 and 12.5 % (v/v). Emulating the controllers used at industrial level, the controller applied a high flow for oxygen levels below 11.5% and a low flow for measures over 12.5%, whereas the controller did not take action when the measure was within this range. The highest and lowest flows predetermined were 3 and 0.2 L min^{-1}, respectively.

2.3.2. Airflow regulation by predetermined cycles

This is the most extended aeration system in aerated windrow facilities. In this case, inlet airflow was regulated automatically by predetermined timed cycles. Basing on the study presented by Ruggieri et al. (2008), the airflow regulation was provided in cycles of 5 min at 5 L min^{-1} and 25 min at 0.2 L min^{-1}.

2.3.3. Oxygen Uptake Rate feedback control (OUR controller)

This new control strategy has been developed by Puyuelo et al. (2010). The main objective of this controller is to obtain an automatic airflow regulation in order to achieve the maximum measure of the biological activity (OUR). The system worked in cycles of 1 hour. The designed OUR control loop compares the variations reached among the successive cycles OUR measures according to the airflow applied. Firstly, after completing the n cycle, the oxygen level is revised to avoid percentages below 5% (v/v). If this level is below this limit, airflow will be increased by 50 %. If an adequate oxygen level is measured, the next step will be the control loop based on the OUR measure and applied flow comparison among two consecutive cycles. For both parameters, three situations are possible, i.e. the system determines if the current value is lower than, higher than or equal to the previous. It is important to note that different absolute thresholds were established to define the superior and inferior limit in which the variation of OUR and airflow can be considered negligible. The limit to detect an OUR variation was defined as 0.5 % of the maximum OUR achieved in previous experiments (about 15 g O_2 h^{-1}). Instead, the range considered for the airflow measures was 0.05 L min^{-1}. Considering this, the controller checks the OUR variation. Next, it compares if the OUR variation obtained is linked with an increase, decrease or a constant airflow. From this algorithm proposed, the system regulates the necessary inlet airflow to optimize the OUR achieved along the process.

To start up the control strategy, two initial parameters have to be fixed, i.e. OUR_{n-1} and F_{n-1}. Flow and OUR set to time zero are the initial values with which the first cycle results are compared. To minimize the energy consumption, the initial airflow fixed was the lowest, i.e. 0.2 L min^{-1}. The initial OUR fixed was 0.1 to force an increase airflow.

2.4. Parameters evaluated

A visual comparison among the controllers performance was undertaken from the profiles of temperature, oxygen composition in the exhaust gases, Oxygen Uptake Rate (OUR) and airflow.

In order to do a quantitative evaluation of the control systems different biological, economical and environmental variables were determined. Maximum oxygen uptake rate (OUR_{max}) achieved, total cumulative oxygen consumption (AT_u), total energy consumption and total gaseous emissions were the variables calculated during the experiments. Additionally the initial and final material stability degree was evaluated for each composting experiment by determining the corresponding DRI (Dynamic Respiration Index) (Ponsá et al., 2010). This measure was undertaken from a representative sample after vigorously mixing the final material of the reactor.

2.5. Oxygen Uptake Rate and total cumulative oxygen consumption

The experimental measure of OUR was indirectly determined on-line with empirical data on airflow and oxygen using equation (1) deduced from the mass balance in a pseudo steady-state.

$$OUR = F \cdot (0.209 - y_{O2(ml)}) \cdot \frac{P \cdot 32 \cdot 60}{R \cdot T} \qquad \text{(Equation 1)}$$

where: *OUR* is the Oxygen Uptake Rate (g O_2 h^{-1}); *F*, airflow into the reactor (L min^{-1}); $y_{O2(ML)}$, is the logarithmic mean between the oxygen molar fraction in the exhaust gases and the inlet air (mol O_2 mol^{-1}); *P*, pressure of the system assumed constant at 101325 Pa; 32, oxygen molecular weight (g O_2 mol O_2^{-1}); *60*, conversion factor from minute to hour; *R*, ideal gas constant (8310 Pa L K^{-1} mol^{-1}); *T*, temperature at which F is measured (K).

Total cumulative consumption (AT_u) was determined through the continuous OUR data obtained along experiments. It was calculated according the equation (2):

$$AT_u = \int_0^t OUR_t \cdot dt \qquad \text{(Equation 2)}$$

where: AT_u, is the total oxygen consumed between time 0 to t (g O_2); *t*, the final experimental time (h); *OUR*, the oxygen uptake rate measured in the steady state (g O_2 h^{-1}) for each cycle. It is important to note that, except for the OUR controller, the OUR measure could not be determined during all the process because the cycle time programmed was insufficient to reach the steady conditions. Despite this, the AT_u value could be estimated for the oxygen controller from the difference between total oxygen in and total oxygen out the reactor considering the total experimental time.

2.6. Stability degree

Based in the methodology proposed by Adani et al. (2006) to assess biological stability degree, the Dynamic Respiration Index was measured in the respirometer built and start-up by Ponsá et al. (2010). Briefly, it consists on placing 150 g of sample in an Erlenmeyer flask of 500 mL of total capacity and incubating in a bath water at 37 ºC. Next, a constant airflow is supplied through the sample and the oxygen content in the outgoing gases is measured. From this assay, the Dynamic Respiration Index (DRI) was determined as the maximum average value of respiration activity measured during 24 hours. It is expressed in mg O_2 g^{-1} OM h^{-1}.

2.7. Energy consumption

The total energy consumption for each experiment was estimated from the total air supplied. It was determined in kJ applying a conversion factor (396 KJ m^{-3}) to transform the total m^{3} of air supplied to the total energy consumed by the compressor.

2.8. Chromatographic Methods

The gaseous emission evaluated were Volatile Organic Compounds (VOCs), methane (CH_4), nitrous oxide (N_2O) and ammonia (NH_3). Ammonia concentration was measured in situ introducing an ammonia sensor (ITX T82) with a measurement range of 0 to 1200 ppm$_v$ inside a hermetic recipient with an inlet and outlet hole that allowed the gas circulation. This measure was taken when the value was stabilized during a period of constant flow. VOCs, CH_4 and N_2O analysis were undertaken by means of gas chromatography (Agilent Technologies 6890N Network GC system, Madrid, Spain). Gaseous samples were directly collected in a 1L Tedlar® bag. Total VOCs, CH_4 and N_2O were analyzed as stated by Colón et al. (2012).

2.8.1. Global emissions

From each emission concentration and the airflow associated at the measure time, the emission rate of each component was evaluated as follows in the equations 3 and 4:

$$E_x\left[\frac{mg}{s}\right] = C_x\left[\frac{mL}{m^3}\right]\cdot\frac{P}{R\cdot T}\cdot PM_x\cdot F \qquad \text{for } x = CH_4, N_2O \text{ and } NH_3 \qquad \text{(Equation 3)}$$

$$E_x\left[\frac{mg}{s}\right] = C_{VOCs}\left[\frac{mg}{m^3}\right]\cdot F \qquad \text{for } x = VOCs \qquad \text{(Equation 4)}$$

where: E_x is for a contaminant *x* the emission rate expressed as mg s^{-1}; C_x and C_{VOC} are the concentrations of each contaminant analytically determined as mL m^{-3} and and mg m^{-3}, respectively; P, is the pressure in atm; R, is the ideal gas constant, 0.082 atm L K^{-1} mol^{-1}; T, is the system temperature in K; PM_x, is the molecular weight of the contaminant analyzed, g mol^{-1} and F is the gas flow expressed in m^{3} s^{-1}.

In order to transform the emissions rates in units of total mass of a contaminant generated per weight of waste treated the equation 5 was applied:

$$E_T = \frac{\int_0^t E_x(t)\cdot dt}{M} \qquad \text{(Equation 5)}$$

where: E_T, is the total contaminant mass emitted per mass unit of the waste treated kg Mg^{-1}; $E_x(t)$, is the emission rate determined in a time *t* in kg s^{-1}; dt, is the time interval considered and M, is the total mass of the waste treated in Mg.

3. RESULTS AND DISCUSSIONS

For each control system compared, the evolution of oxygen, temperature, OUR and airflow supplied are shown in the Figure 2. It is important to note that due to the continuous airflow oscillation the OUR evolution during the cyclic controller cannot be determined and under the oxygen controller this parameter can only be calculated during the high-rate stage.

In general, the most relevant difference was observed in the airflow evolution. Only with the OUR controller a softened evolution was achieved. Instead, the other controllers caused continuous fluctuations that could hamper the biomass acclimatization. The main parameters evaluated are reported in the Table 1. Energy consumption, OUR_{max} and AT_u were calculated to compare the performance of each system. For the cycles aeration only energy consumption is presented. Results of energy consumption demonstrated that the cyclic and the OUR controller required less energy than the oxygen controller, although the differences were not significant. Oxygen controller results showed the highest OUR_{max} when applying a constant airflow of 3 L min^{-1} but again non-significant differences were observed. In a previous work, it was confirmed that during the high-rate phase the biological activity is maximum and the oxygen concentration in the biofilm is negligible (Puyuelo et al., 2010). Finally, AT_u and final DRI_{24h} values obtained confirmed that with the OUR controller it exists a major oxygen consumption and the final product was more stable.

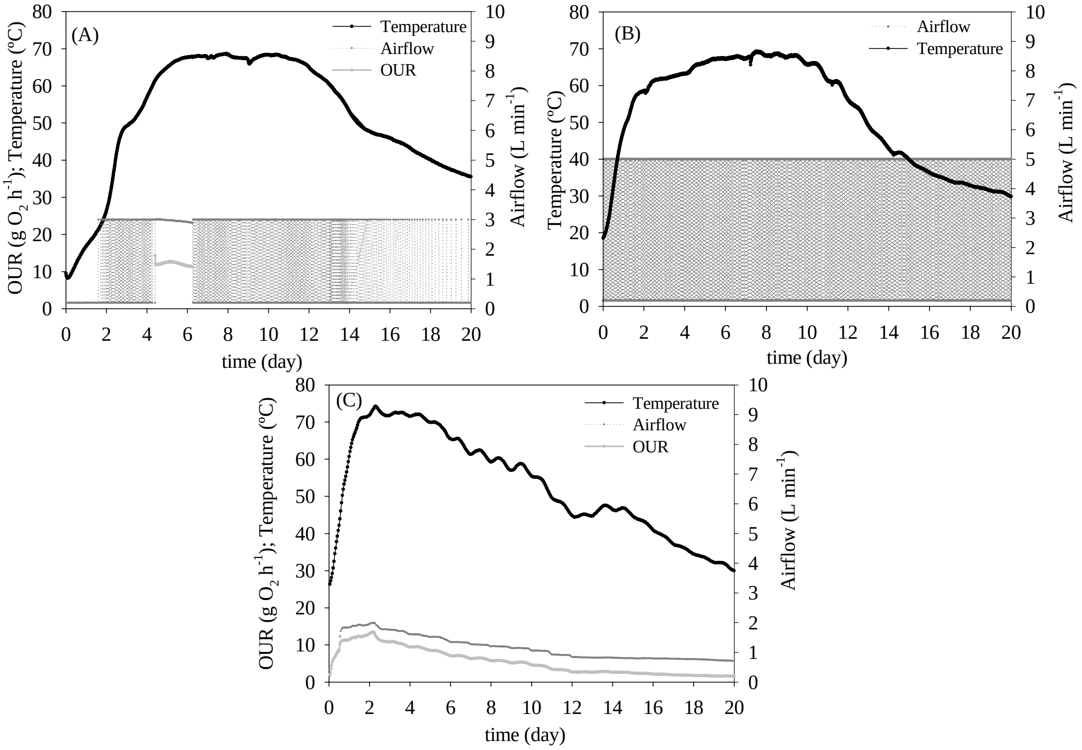

FIGURE 2. Evolution of temperature and airflow applied for the three aeration strategies studied: oxygen feedback controller (A), cyclic controller (B) and OUR controller (C). The OUR profile is also showed in the experiment C and during the high-rate stage of the case A.

TABLE 1. Principal parameters evaluated for each aerated system.

Aeration system	Energy consumption (kJ)	OUR_{max} ($g\ O_2\ h^{-1}$)	AT_u ($g\ O_2$)	Final DRI ($mg\ O_2\ g^{-1}\ OM\ h^{-1}$)
Oxygen control	$13.4 \cdot 10^3$	14.2	2133	1.99 ± 0.06
Cyclic control	$12.0 \cdot 10^3$	-	-	1.94 ± 0.01
OUR control	$13.1 \cdot 10^3$	13.5	2286	1.8 ± 0.1

OUR_{max}: maximum oxygen uptake rate achieved; AT_u: total cumulative oxygen uptake; DRI: Dynamic Respiration Index average during 24 h of maximum activity.

The global emissions of VOCs, CH_4, N_2O and NH_3 generated during each experiment are shown in the Table 2. All results demonstrated that the OUR controller reduced the GHG emissions and thus, the environmental impact associated to the composting process. Therefore, these results confirmed that the progressive airflow evolution achieved by means of the OUR controller also minimized the emissions produced. On the contrary, the cyclic controller presented always the highest emissions. In addition to this, the systems with airflow fluctuations showed higher emissions when the maximum airflow was applied.

TABLE 2. Total emissions of VOCs, CH_4, N_2O and NH_3 for the tree systems considered.

E_T	kg VOCs Mg^{-1} OFMSW	kg CH_4 Mg^{-1} OFMSW	kg N_2O Mg^{-1} OFMSW	kg NH_3 Mg^{-1} OFMSW
Oxygen control	0.37	0.10	> 0.0038	> 0.6
Cyclic control	0.40	0.20	0.0152	> 1.0
OUR control	0.15	0.08	0.0094	0.7

OFMSW: Organic Fraction of Municipal Solid Waste.

In general, the VOCs emission is related with an low aeration or with an incomplete aerobic degradation (Homans and Fisher, 1992). In all aeration strategies, the major VOCs generation was detected during the first experimental week between the mesophilic to thermophilic transition as was described by Komilis et al. (2004). After the seventh day these emissions were negligible. Other works observed that before starting the composting process the VOCs emissions have already started (Eitzer, 1995; Smet et al., 1999). The global values obtained were below than the data reported in the literature. The most similar value was cited by Smet et al. (1999) and was 0.59 kg VOC Mg^{-1} OFMSW.

During the composting process the CH_4 emission appears as consequence of oxygen deficit conditions where the anaerobic biologic activity predominates over the aerobic activity. Normally, it is due to excessive moisture or inappropriate ventilation system strategy. After the third experimental day the presence of CH_4 in the exhaust gases was not detected. As expected, the highest values were obtained during the high-rate stage. Similar global emissions have been detected by other authors.

Temperature, nitrogen content and aeration rate are parameters related with the N_2O generation (Hellebrand and Kalk, 2001). Many authors have described that the highest N_2O emissions are detected during the initial step (He et al., 2001; El Kader et al., 2007; Jiang et al., 2011). Other works, as Fukumoto et al. (2003), did not identify N_2O after starting the process. Instead, our results showed a constant emission of N_2O along the experimental time. This measure was in the range of 4 to 6 ppb$_V$. Again, the global emissions reported in other studies were higher (Amlinger et al., 2008).

The NH_3 emission is dependent on the C/N ratio, the temperature, the pH and the airflow. All profiles follow a similar trend to the temperature curve. In this case, the emission was higher than the general ranges reported in the literature but in general, these values could be included inside the range described by Clemens and Culhs (2003) in MBT facilities ranging from 0.02 to 1.15 kg NH_3 mg^{-1}OFMSW. According to a recent study (Puyuelo et al., 2011), these high emissions could be due a wrong C/N adjust, since it was determined considering all the organic carbon content instead of using only the biodegradable fraction.

4. CONCLUSION

Total airflow supplied together with the biological parameters and the global gaseous emissions allowed to demonstrate that the OUR controller minimizes the GHG emissions with a low energy consumption. In addition, the results also showed that under this aeration system the final product was more stable.

5. ACKNOWLEDGMENTS

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(44) INFLUENCE OF AERATION RATE ON COMPOSTING GASEOUS EMISSIONS WITH PIG SLAUGHTERHOUSE SLUDGE

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ABSTRACT

This study investigated the influence of composting aeration rate on emission concentrations in terms of ammonia, hydrogen sulphide, mercaptan, and Volatile Organic Compounds (VOCs). The influence of the aeration on the gaseous emission was the first process conditions investigated.

Organic waste composting generates emissions resulting in a significant level of nuisance often leading to conflicts closing or preventing the installation of treatment plants. Ammonia, hydrogen sulphide and VOCs are the main contributors to compost odours. Conventional solutions are designed to confine the odours or apply a biological or chemical treatment. By establishing the relationship between aeration rate and compost gaseous emission characteristics, preventive control techniques can be introduced.

The project investigated the influence of the aeration rate on compost emissions from pig slaughterhouse sludge, using 300 L pilots reactors under forced aeration. Five different rates were tested: 1.68, 4.03, 6.22, 9.80 and 13.04 L/h/kg of wet waste. The aeration treatment was applied to the same waste mixture consisting of Pig Slaughter house Sludge (PSS) and Wood Chips (WC), mixed at a dry mass ratio of 1.2:1(PSS:WC). The Wood Chips (WC) came from the same plot of calibrated 20-40 mm particle size. All experimental materials were analyzed and weighed before starting the experiment. Composting treatments were aerated during 30 to 60 days, and the material was turned every 10 days. The aeration rate was maintained constant while monitoring the compost temperature. The incoming and outgoing ventilation air streams were monitored for O_2, CO_2 and CH_4. A portion of the outgoing ventilation air stream was also bubbled into chemical traps for NH_3, H_2S and mercaptan quantification. Every two days, another portion of the outgoing ventilation air stream was collected using Tedlar bag and its gases trapped on activated carbon trap to analyze its VOC by Gas Chromatography coupled with a Mass Spectrometry (GC/MS); qualification and quantification of VOCs were obtained by chromatogram processing. At the end of the experiment, all reactors were emptied, to weigh and sample their content for a mass balance analysis.

In terms of composting emissions, over 45 VOCs were identified and classified based on their concentration: major constituents (nitrides, sulphides, ketones and alcohols); average constituents (nitride and sulphide compounds, aromatic hydrocarbons and aliphatic hydrocarbons), and minor constituents (terpenes, alkanes, and acids). The concentration of these compounds was based on the concentration over time per kg organic matter of initial wet PSS waste within the air flow rate.

The analysis of emission concentrations indicated that higher aeration rates increased emissions, but had no influence on the final waste stability simply because low rates resulted in a longer treatment period and more turning operations. Thus, higher aeration rate enhanced the stripping of gases from the compost matrix. Nevertheless, increasing the aeration rate changed the emission profile with higher rate producing emission peaks rather than an even profile associated with the lower aeration rates. As a result, an aeration strategy must be considered to optimize the treatment of gaseous emissions from pig slaughterhouse sludge composting plant.

1 INTRODUCTION

Composting is promoted in Europe as a means of recycling organic wastes as soil amendment. However composting is responsible for gaseous emissions which may impact human health and environment. Carbon dioxide, methane and nitrous oxide are all greenhouse gases contributing to the earth warming trend. Ammonia emissions are responsible for cid rains and eutrophication. Ammonia and hydrogen sulfide may also affect the health of composting plant workers. Finally, ammonia, hydrogen sulfide and volatile organic compounds (VOCs) contribute to odours which may lead to the closure of the composting plants or the prevention of new installations. To minimize the impacts of these gaseous emissions and especially to reduce odours, conventional solutions confine, mask or treat gaseous emissions through biofiltration and chemical cleaning (Muller et al., 2004). A better understanding of mechanisms and process conditions responsible for gaseous and odour emissions could generate preventive methods for the control of emissions.

Most research projects have focused mainly on the emissions of inorganic compounds during composting (Chen et al., 2010), or on a single VOC family (Wu et al., 2010), or finally all VOCs without inorganic compounds (Delgado-Rodriguez et al., 2011, , Pagans et al., 2006, Scaglia et al., 2011). In terms of odours, most approaches are incomplete. Noble et al. (2001) evaluated the influence of H_2S on odour emissions during composting of mushroom. Smet et al. (1999) underlined that, in many studies, the synergy effects of various gases on odour concentration were often ignored. According to Defoer et al. (2002), this synergy prevents the correlation between VOCs and odor concentrations, and can only be achieved by conducting an exhaustive characterization of the gaseous emissions.

The influence of process conditions on gaseous emissions was investigated by Delgado-Rodriguez et al. (2011) who found that aeration rate as opposed to compost moisture and C/N ratio had the most influence on VOCs emissions. Pagans et al. (2006) observed that, during the composting of Organic Fraction of Municipal Solid Wastes (OFMSW), emissions VOCs increased when the volumetric ratio of bulking agent to OFMSV was increased. D'imporzano et al. (2008), and Scaglia et al. (2011) correlated VOCs and odor emissions to the biological stability during the composting of municipal solid wastes. However, Pagans et al. (2006) did not observe such correlation when investigating several wastes including Municipal Solid Waste (MSW).

Accordingly, there is a need to research the influence of all composting process conditions, including the nature of the waste, to better understand the mechanisms responsible for the characteristics of the gaseous emissions. The objective of the present study was therefore to investigate the impact of aeration rate on NH_3, H_2S and VOCs emissions during the composting of one single organic waste, namely pig slaughterhouse sludge (PSS). The experiment was conducted in the laboratory using 300L pilot reactors. Five different aeration rates were tested, namely 1.68, 4.03, 6.22, 9.80 and 13.04 L/h/kg of wet PSS waste.

2 METHODOLOGY

2.1 Experimental set-up

The experimental set-up consisted of three similar pilot reactors housed in a laboratory kept at ambient temperature. The reactors consisted of a stainless steel cylindrical chamber of 300 L, with a height of 0.8m and a diameter of 0.7m, insulation by 100 mm layer of polyurethane. Compost turning consisted in emptying the reactor, mixing and refilling. The compost was suspended over a bottom plenum by a stainless steel 8 mm grid mesh, allowing for the entry of ventilation air. A bottom tap allowed for the drainage and collection of leachate. The upper cone shape stainless-steel hood was equipped with a gutter to collect condensate. The outlet ventilation air was dewatered by a bottle condenser before being analyzed.

The airflow rate was adjusted using a flow meter (FL-821-V, OMEGA Engineering Inc., Stamford, USA) and monitored using a volumetric gas meter (Gallus 2000, Actaris, Liberty Lake, USA). Pressure (PTX510, DRUCK,n Labège FRANCE), moisture and temperature (dew point and temperature transmitter, VAISALA, Birmingham, United Kingdom) of the in-coming air were measured continuously. Dry-air molar flow was presumed nominally constant throughout the system. The out-going water vapour molar flow was calculated by assuming saturated air conditions. Concentrations of oxygen, carbon dioxide, methane, ammonia, mercaptan, hydrogen sulphide and VOCs were measured in both the in-coming and out-going airflows. Oxygen concentration was measured with a paramagnetic analyzer (MAGNOS 206, ABB, Zurich, Switzerland). Carbon dioxide and methane were measured with an IR spectrometric analyzer (URAS 26, ABB, Zurich, Switzerland). These gases were measured during 15 minutes every hour, in sequence for each reactor outlet and for the incoming air.

The composting material temperature was monitored using four Pt100 temperature probes located in the low, middle and upper part of the compost, and under the reactor hood. The mass of waste in each reactor was monitored using a scale constituting of three weight sensors (X201-B, PRECIA MOLEN, Paris, France). The condensate and leachate were collected and weighed every day to complete the dry matter mass balance.

2.2 Experimental waste

The pig slaughterhouse sludge (PSS) was collected from the wastewater treatment plant of a slaughterhouse located in Locminé (Brittany, FRANCE). At this plant, the sludge was produced by 1844 m^3/day of wastewater subjected to a primary flocculation treatment followed by decantation, centrifugation and liming. Once collected, the PSS was stored at -18°C, and then thawed at 4°C for one week, before being used to minimize its degradation. The bulking agent (BA) consisted of wood chips (WC) with a particle size ranging from 20 to 40 mm. All experimental materials were sampled three times and analyzed before being mixed together. Two batches of compost mixture were prepared, requiring two sampling and analytical sessions (Table 1).

2.3 Method

The experimental compost mixture consisted of 50 % PSS, 40 % WC and 10 % water, on a wet mass basis (Table 2), for a moisture content of 57%. Mixing was carried out in a concrete mixer (150 L). The low coefficient of variance (CV) of less than 5 %, among mixtures and in terms of initial characteristics, indicated insignificant differences. After loading, a low aeration rate of 80-120 L/h to all reactors for 96 h until the material temperature reached 50 °C. Then, the aeration rate was adjusted to the specified treatment value and maintained constant thereafter, at 150, 350, 550, 850 and 1150 L/h. The highest aeration flow reflects that applied in industrial facilities. The low aeration rates of 150 and 350 L/h, were selected to limit O_2 supply and therefore aerobic biodegradation, while the aeration rates of 550 and 850 L/h were intermediate values. The material was turned every 1.5 weeks and the composting trial was stopped when the O_2 consumption rate appeared constant and under 0.005 mol O_2/h/kg MO_0. The experiments lasted between 36 to 58 days, the span being inversely proportional to the aeration rates.

2.4 Analytical method

All samples were sampled three times and analyzed according to standard methods. All WC and compost mixture samples were grinded to a particle size of 0.5 mm before analysis. Each pilot reactor was considered as an independent sample. No significant differences were found between the three samples analyzed for each material.

Dry matter was measured by drying 2 kg samples at 80 for 23 h and then 110°C for 1 h. Organic matter (OM) was measured by calcination at 550 °C according to the standard method NF U 44- 160 (AFNOR, 1985). According to standard NF-EN-13137 (AFNOR, 2001), total carbon (TC) was quantified by flash combustion and oxidizing to CO_2 at 1800°C, and measuring CO_2 by gas chromatography (FLASH 2000 Thermo scientific, Delft, Netherlands). The chemical oxygen demand (COD) was determined by dichromate oxidation as described by NFT 90-101 (AFNOR, 1971; AFNOR, 2001). The NH^+_4/NH_3 was determined by steam distillation using MgO, trapping in boric acid (H_3BO_3, 40 g/L) and back-titrating with sulfuric acid (H_2SO_4, 0.2 N). Total Kjeldahl nitrogen (TKN) was quantified by mineralization using 18 M sulfuric acid followed by steam distillation and then titrimetric determination of NH^+_4 /NH_3 as described above (AFNOR, 1995).

2.5 Gaseous sampling and characterization

The gaseous constituents found in the outgoing ventilation airflow required several different methods of quantification, depending on the chemical composition.

Ammonia, hydrogen sulphure and mercaptan were collected using chemical trap (Gallus 2000, Actaris, Liberty Lake, USA) changed every 48 h: NH_3 required 1 M sulfuric acid 1N; H_2S was collected using a trap with $PbNO_3$ (10 mg/L) in 0.1 M buffer acid formic/formate; H_2S and mercaptan were collected using a trap with $Hgcl_2$ (10 mg/L) in acid medium (0.1N). Trap saturation was never reached during the 48 h collection period. In the traps, H_2S and mercaptan formed a precipitate of respectively PbS and Hg (SR_2), which were recovered and quantified by filtration, drying and weighing.

Outlet aeration streams were sampled for VOC emissions were sampled every 24 hours around O2 consumption peak and turning operation, then every 48 hours thereafter. Sampling consisted in filling a 5 L Tedlar bag using a silicone septum and then absorbing the VOC on a carbon sorbent (Carbotrap 349, PerkinElmer, Waltham, USA) within 1 h of collection and at an airflow rate of 50 ml/min (Ribes et al., 2007) using a vacuum pump (Model Gilair3, Gilair, Clearwater, USA) equipped with a constant low flow module monitored by a flow meter. The carbotraps were stored at 4°C and desorbed for GC-MS analysis within 1 week of sampling, using the purge and trap method. A primary thermal desorption (Turbomatrix 400, PerkinElmer, Waltham, USA) of the carbotraps was conducted at 200°C and under an N_2 carrier flow of 50 ml/min. The desorbed gases were then analyzed by a chromatographic column (CP-FFAP CB 25m x 0.15mm x 0.25µm) maintained at 220°C, using Helium as carrier gas flowing at 1mL/min under a pressure of 310 kPa. Separated compounds were detected by mass spectrometry with two simultaneous acquiring modes. A full scan (FS) acquisition for masses between 20 and 300 uma allowed identification of compounds by comparison with library references and their retention time. A single ion resolution (SIR) acquisition was used to quantify specific masses of the compounds.

3 RESULTS AND DISCUTION

3.1 Composting indicator and final mixture characteristics

The temperature regime of each pilot reactor is presented in Figure 1. On day 4, the compost mixture aerated at 150 L/H reached a peak temperature of 45 °C while those aerated at 350 and 550 reached a peak temperature of 47 and 57 °C. On day 6, the compost mixture aerated at 850 L/h reached a peak temperature of 57 °C while that aerated at 1150 L/H reached 55 °C. Accordingly, aeration rates of 850 and 1150 L/h seemed to delay peak temperature while aeration rates of 150 and 350 L/h seem to limit peak temperatures. The 550 L/h aeration rate produced the quickest and highest peak temperature.

The chemical characteristics of the final mixtures (Table 4) indicate a drop in TC and COD during composting. Concentration drop for TKN and NH^+_4/NH_3 respected levels in aeration rate.

The mass of biodegraded matter was estimated in terms of O_2 consumed (Table 5), which varied slightly with aeration rate as confirmed by a CV under 10%. Only the final moisture content demonstrated a high CV of 10 %, among pilot reactor compost, because the treatment aerated at 350 L/h composted for the shortest period. The high moisture and the low O_2 total consumption for the 350L/h treatment indicated an uncompleted treatment. The aeration rate influenced the time and the number of turning matter.

3.2 Gaseous emissions results

Total gaseous emissions, namely the sum of VOCs, N-NH_3, S- H_2S and mercaptan in µmol/kg MO_0 (mass of organic matter in the original compost mixture) are illustrated in Figure 3a while individual emissions are illustrated in Figures 3(b), 3(c), 3(d) and 3(e), respectively. Except for mercaptans, the quantity of emissions increased with aeration rate. However, for all aeration rates, the quantity of each detected gas always followed the following order: N-NH_3> S-H_2S> Total VOCs> mercaptan. The reduction factor for compound mass emission with the lowest aeration rate, compared to the highest, was 10 for S-H_2S, 5 for N-NH_3, 3 for VOCs and 5.5 for the total gaseous emissions.

The highest total gas emissions were observed during the early stage of the process (Figure 4a). Total gaseous emission, namely total VOCs emission, N-NH_3, S- H_2S and mercaptan emission, are presented in Figures 4(b), 4(c), 4(d) and 4(e), respectively. After 15days, levels of emissions had dropped 90 %. The aeration rate influenced the profile of gaseous emissions. For the treatments at 1150, 850, 550 and 350 L/h, three peak emissions were observed: the first at 5-9 days corresponding to the flow adjustment, and; the second and third peak emissions followed the first and second turning operations. Increasing the aeration rate increased the peak emission intensity. However, at 150L/h the profile emission remained low. For mercaptan, peak emission occurred during the first ten days and was not influenced by the aeration rate variations.

4 CONCLUSIONS

The present study compared gaseous emission composition as a function of aeration rate during the composting of pig slaughterhouse sludge (PSS) with coarse wood chips (WC). The compost organic matter biodegradation level was measured in parallel along with total O_2 consumption. Oxygen consumption and organic matter biodegradation are indicators which were highly influenced by aeration rate.

Except for mercaptan emission, the mass of gaseous emission in terms µmol/kg MO_0, increased with higher aeration rates. However, compound concentration in the gaseous emission always following the order: N-NH_3> S-H_2S> Total VOCs> mercaptan. These results indicated that the aeration rate had an impact on the mechanism generating the various gaseous compounds and higher rates enhanced stripping along with the amount of gaseous emission.

For the 5 aeration rates, the highest gaseous emissions were detected during the first 15 days of process time, and immediately after turning the compost. The present experiment leads to the conclusion that decreasing the aeration rate can reduce peak and over-all gaseous emissions.

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Figure

FIGURE 1 **Composting material temperature**

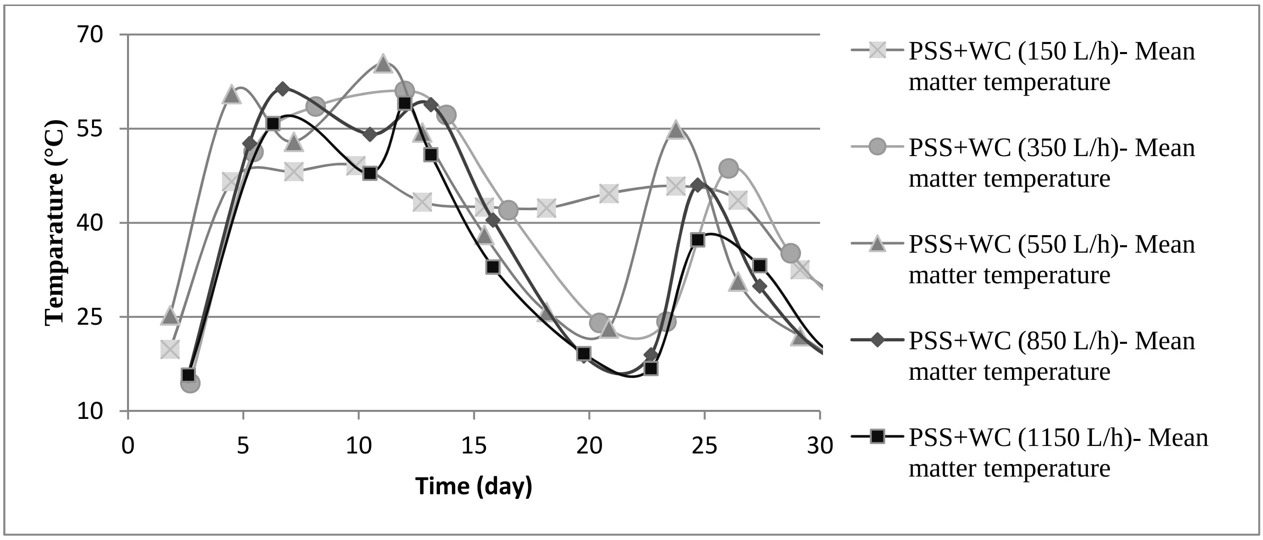
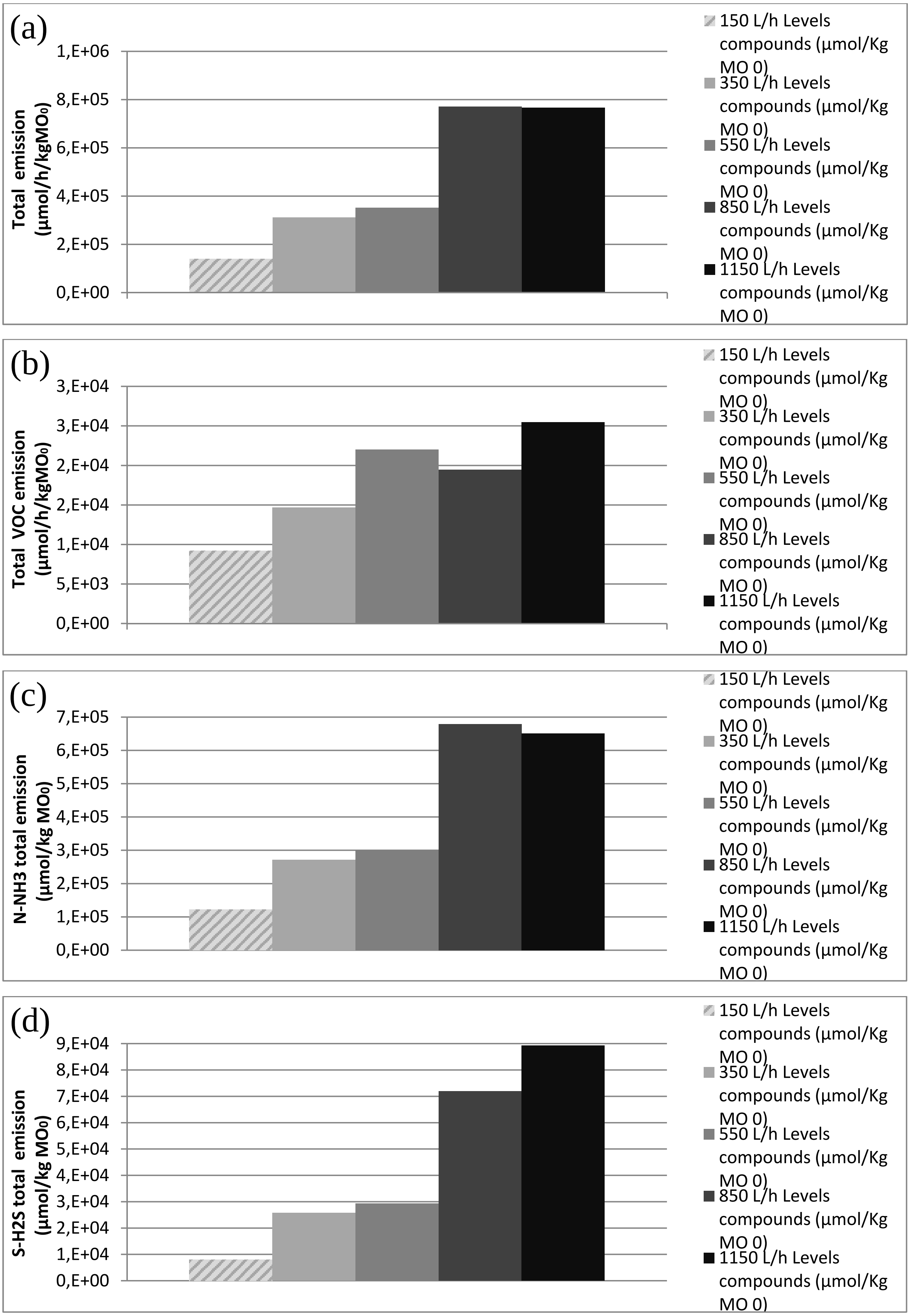


FIGURE 2 **(a) Cumulate amount of total gaseous emissions / (b) Cumulate amount of VOCs emission/ (c) Cumulate amount of N-NH_3 emission/ (d) Cumulate amount of S-H_2S emission/ (e) Cumulate amount of mercaptan emission**


(a)
Total emission (µmol/h/kgMO₀)
1,E+06
8,E+05
6,E+05
4,E+05
2,E+05
0,E+00
150 L/h Levels compounds (µmol/Kg MO 0)
350 L/h Levels compounds (µmol/Kg MO 0)
550 L/h Levels compounds (µmol/Kg MO 0)
850 L/h Levels compounds (µmol/Kg MO 0)
1150 L/h Levels compounds (µmol/Kg MO 0)
(b)
Total VOC emission (µmol/h/kgMO₀)
3,E+04
3,E+04
2,E+04
2,E+04
1,E+04
5,E+03
0,E+00
150 L/h Levels compounds (µmol/Kg MO 0)
350 L/h Levels compounds (µmol/Kg MO 0)
550 L/h Levels compounds (µmol/Kg MO 0)
850 L/h Levels compounds (µmol/Kg MO 0)
1150 L/h Levels compounds (µmol/Kg MO 0)
(c)
N-NH3 total emission (µmol/kg MO₀)
7,E+05
6,E+05
5,E+05
4,E+05
3,E+05
2,E+05
1,E+05
0,E+00
150 L/h Levels compounds (µmol/Kg MO 0)
350 L/h Levels compounds (µmol/Kg MO 0)
550 L/h Levels compounds (µmol/Kg MO 0)
850 L/h Levels compounds (µmol/Kg MO 0)
1150 L/h Levels compounds (µmol/Kg MO 0)
(d)
S-H2S total emission (µmol/kg MO₀)
9,E+04
8,E+04
7,E+04
6,E+04
5,E+04
4,E+04
3,E+04
2,E+04
1,E+04
0,E+00
150 L/h Levels compounds (µmol/Kg MO 0)
350 L/h Levels compounds (µmol/Kg MO 0)
550 L/h Levels compounds (µmol/Kg MO 0)
850 L/h Levels compounds (µmol/Kg MO 0)
1150 L/h Levels compounds (µmol/Kg MO 0)

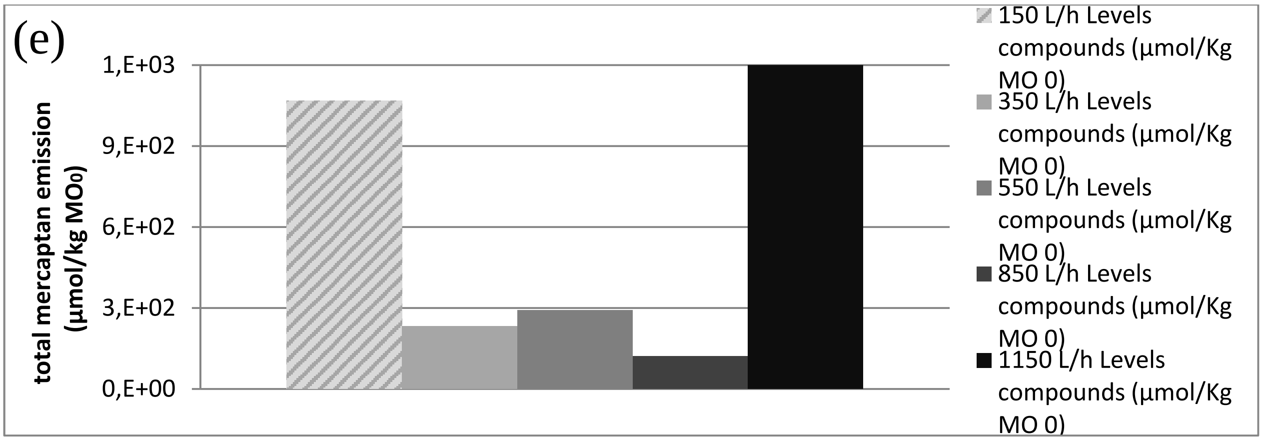
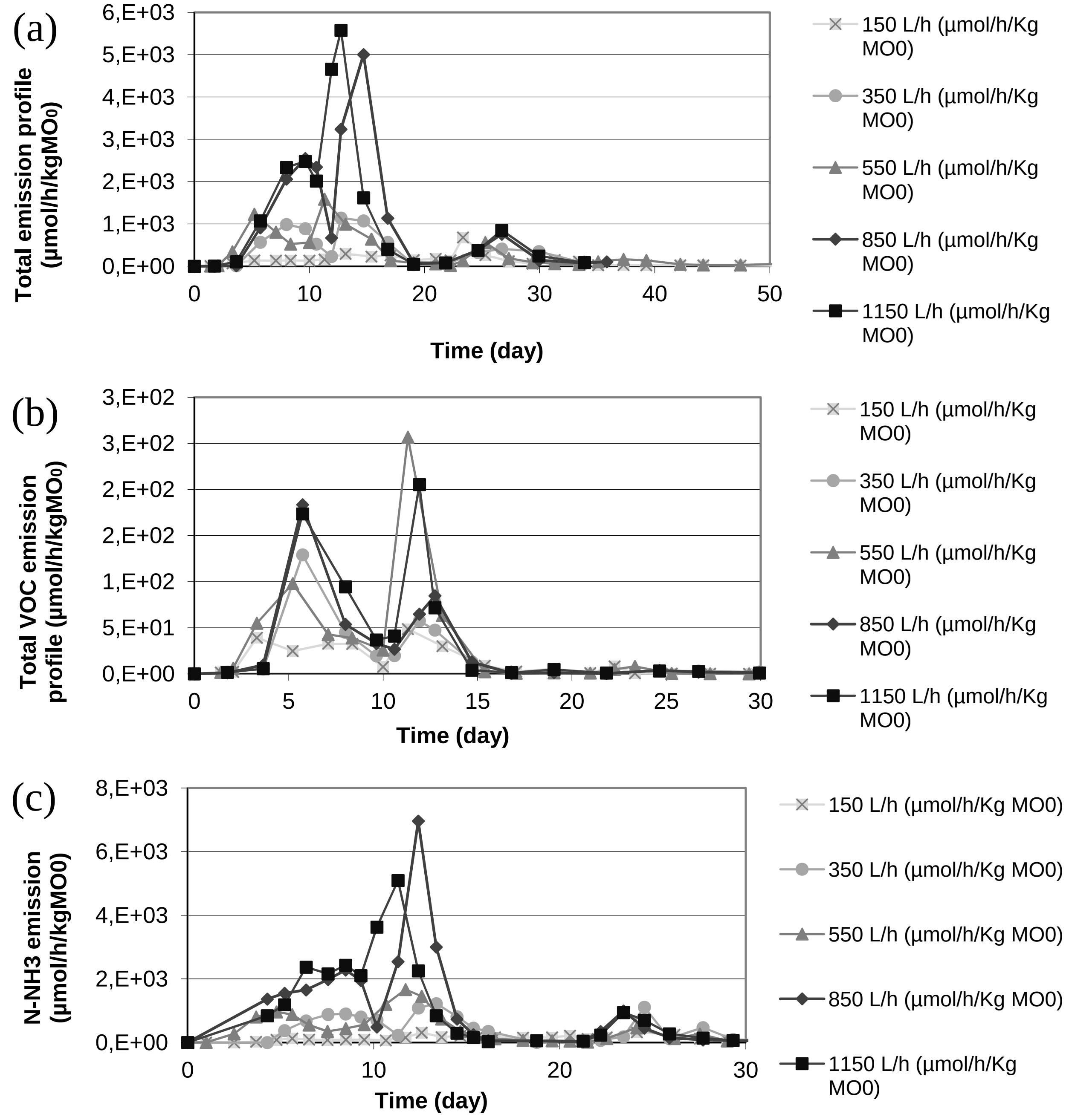
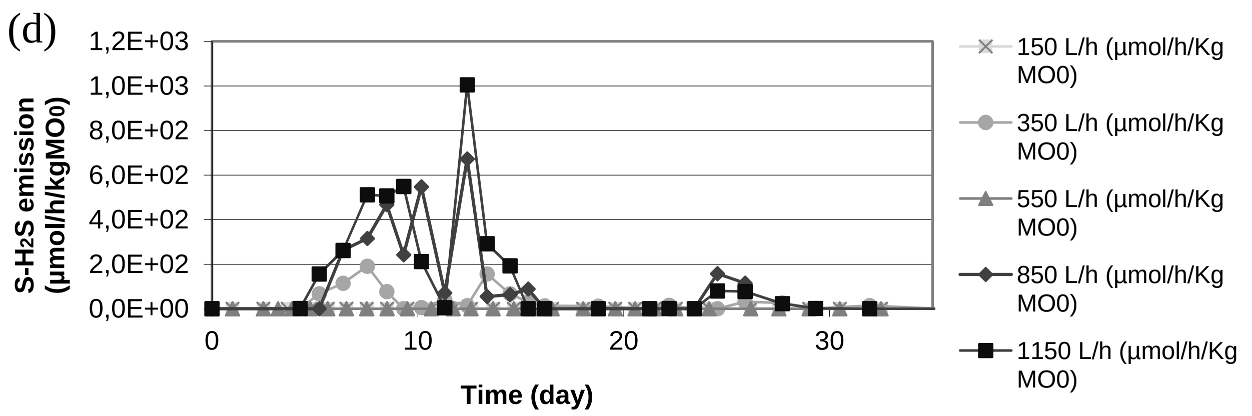


FIGURE 3 **(a) Profile emission of Total emission/ (b) Profile emission of VOCs emission/ (c) Profile emission of N-NH_3 emissions/ (d) Profile emission of S-H_2S emission/ (e) Profile emission of mercaptan emission**





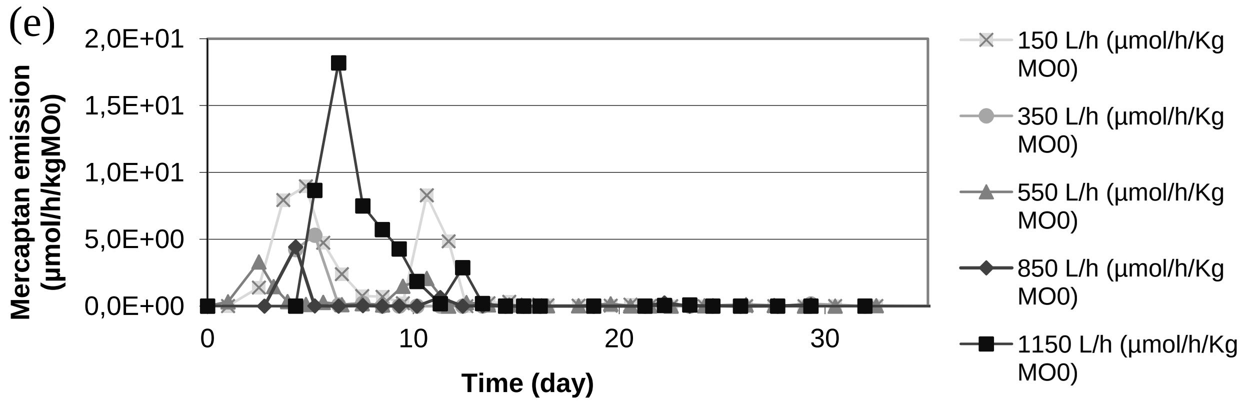


TABLE 1 **Chemical characteristics of pig slaughterhouse sludge and wood chips.**

	PSS (1)	WC 20- 40 mm (1)	PSS (2)	WC 20- 40 mm (2)
Moisture (%)	72,03	27,51	72,59	27,51
OM (% DM)	87,17	99,70	85,48	98,37
COD (mg O_2/ g DM)	1938	1278	1823	1166
TC (mg/ g DM)	596,6	499,7	542,3	500,3
N-NH_4^+ (mg N/ g DM)	6,6	n.m	6,9	n.m
NTK (mg N/g DM)	61,7	1,2	65,6	0,9
TN (mg/g DM)	67,5	1,8	70	2,1

n.m not measured

TABLE 2 **Starting mixture and composting conditions.**

	PSS (1) + WC (1) (150 L/h)	PSS (2) +WC (2) (350 L/h)	PSS (1) + WC (1) (550 L/h)	PSS (2) + WC (2) (850 L/h)	PSS (2) + WC (2) (1150 L/h)	CV (%)
PSS Fresh matter (kg)	90,26	88,34	89,35	87,35	89,31	1,2
WC fresh matter (kg)	75,22	73,61	74,46	72,79	74,43	1,3
WC dry matter (% wet matter)	72,49	72,49	72,49	72,49	72,49	0,0
Dry mass ration	2,16	2,16	2,16	2,16	2,16	0,0
Water added (kg)	20,06	19,63	19,46	17,79	19,85	4,7
Total Mass (kg)	185,54	181,58	183,26	177,94	183,59	1,6
Mixing matter moisture (%)	57,01	57,01	56,91	56,62	57,01	0,3
Mean aeration rate (l/h)	152	356	556	856	1165	
First turning (day)	10	11	10	11	11	
Second turning (day)	21	22	21	22	22	
Third turning (day)	31		31			

Fourth turning (day)	44		44			
End (day)	58	36	58	36	36	
Final mass (kg)	97,4	98,5	87,1	89,8	94,8	5,3
Final moisture (% wet matter)	32,4	41,0	27,6	34,2	35,1	14,2

TABLE 3 **Chemical characterization of the final mixture**

OM_0: Starting organic matter from PSS

	PSS(1)+WC (1) (150 L/h)	**PSS(2)+WC(2) (350 L/h)**	**PSS(1)+ WC(1) (550 L/h)**	**PSS(2)+ WC(2) (850 L/h)**	**PSS(2)+ WC(2) (1150 L/h)**
Moisture (%)	32,4	41,0	27,6	34,2	35,1
OM (% DM*)	95,2	93,7	94,9	94,7	95,0
COD (mg O_2/ g DM*)	1313,0	1394,0	1323,0	1386,0	1334,0
TC (mg/ g DM*)	485,7	504,6	482,7	505,7	497,4
N-NH_4^+ (mg N/ g DM*)	3,9	5,3	3,7	4,7	5,1
NTK (mg N/g DM*)	12,8	18,0	13,6	16,5	18,9
TN (mg/g DM*)	19,9	22,1	19,7	18	19,7

TABLE 4 **Composting process achievement**

	mol O2 / kg OM_0	**Moisture %**	**dT mean**	**dt max**
PSS+WC (1150 L/h)	45.6	35.1	16,7	44,6
PSS+WC (850 L/h)	41.2	34.2	18.3	46,7
PSS+WC (550 L/h)	44.9	27.6	18,6	47,7
PSS+WC (350 L/h)	38.8	41	22,3	48.3
PSS+WC (150 L/h)	46.7	32.4	20,9	33,5
Mean	43.5	34.1	19,4	44,2
CV	7.6	14.2	11,5	13,9

(53) EMISSIONS OF VOCs DURING MUNICIPAL BIOWASTE AND SLUDGE COMPOSTING IN PILOT SCALE REACTORS

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EXECUTIVE SUMMARY

During the composting process, organic matter is mainly transformed by aerobic reactions into a stabilized product. This process involves the production and release of some odorous gases to the atmosphere. There are far fewer published data on VOCs and odours emissions of composting process than emissions of others gases, such as NH_3, N_2O or CH_4 (Mallard (2005)). This article aims to evaluate VOCs and odours emissions during biowaste and sludge composting and to compare these emissions regarding different aeration conditions.

Two series of six composting experiments were carried out in 160L reactors, during 83d.Two different wastes were tested: a mixture of municipal biowaste (BIO) and a mixture of municipal sludge (BOU). Two different aeration strategies were applied to each type of waste: a sufficient aerated modality (BIO+ (Berthe (2012)) and BOU+), and an oxygen limited modality (BIO- and BOU-). The pilot reactors were mechanically turned twice for biowaste and three times for sludge during the first six weeks of composting and their content was screened on the 83th day. Temperatures in the core of the waste and oxygen concentration in emitted gas were continuously monitored. Initial mixtures, mixtures after each mechanical turning and final composts were sampled for physico-chemical analyses and estimation of organic matter stabilization (Van Soest fractionation). Punctual sampling of the gas in headspace was also done in Tedlar® and Nalophane® bags, in order to analyze the specific VOCs concentration in the effluent gas. The sampling was done once a week during the first six weeks and every two weeks afterwards.

Maximal temperature of sufficient aerated experiments was above 70°C and it remained high during several days, whereas the temperature in BIO- and BOU- was never above 55°C. Emitted O_2 concentration in BIO+ and BOU+ was greater than 5%v/v and confirmed that the aeration was not limited.

127 different VOCs were identified for biowaste and 62 for sludge. 30 of them (for each waste) were also quantified. The quantified compounds were classified into six families: terpenes, ketones, sulphur compounds, alcohols, aromatic hydrocarbons and chlorinated compounds. Most of the VOCs appeared during the first six weeks of composting and their variability and concentration decreased during the process.

Results of this study showed that:

- each waste was characterised by different VOCs groups: terpenes and ketones were the major VOCs emissions from biowaste composting, whereas sulphur compounds and monocyclic aromatic hydrocarbons (MAHs) were characteristic from sludge composting,
- the evolution of VOCs concentration during composting were related to the type of waste and the aeration strategy: emissions decreased faster for sludge than for biowaste and for sufficient aerated reactors than for oxygen limited ones,
- odours evolution was similar to total VOCs evolution, but odour concentration was dependant on VOCs nature.

In conclusion, VOCs and odorous emissions were specially detected during the active phase of composting, particularly when the aeration did not fulfil the process oxygen requirements. In order to decrease the olfactory nuisance, it would be necessary to enhance sufficient oxygenation conditions and to control the first part of the composting processes.

1 INTRODUCTION

During the composting process, the organic matter is mainly transformed by aerobic reactions. The process involves the production and the release of odorous and greenhouse gases to the atmosphere. These gases might generate environmental and health impacts for the workers and the neighbourhood of the composting plants. Odorous and gases emissions from plants are a concern for industrials because of the current environmental policies (ICPE-2008 French regulation article 2170, 2008; EPER, 2007; Kyoto protocol, etc.) and the acceptability of the plants by the adjacent residents.

There are far fewer published data on VOCs and odours emissions of composting process than emissions of others gases, such as NH_3, N_2O or CH_4 (Mallard (2005)). Today, the authorities note a lack of data about emissions from composting process and there is a growing demand of knowledge.

The main objectives of this study are to evaluate the emissions of VOCs and odours during municipal biowaste and municipal sludge complete composting process, to compare the effect of the type of wastes on VOCs emissions and to analyse the effect of process management (aeration, mechanical turning and screening) on them.

2 MATERIALS AND METHODS

2.1 Composting experiments

Two composting experiments were performed in six composting reactors during 83 days (12 weeks). The first experiment consisted in composting a mixture of biowaste and screening refusal (BIO), placed in six similar reactors. They were aerated with two different regimes: three reactors were under-aerated (BIO-) and three were optimal aerated (BIO+) as show in Berthe (2012). Total cumulative applied air flow was 2,3 times higher for BIO+ than for BIO-. The second experiment consisted in composting a mixture of sludge, greenwaste and screening refusal (BOU); three reactors were under aerated (BOU-), and three had a not-limited aeration (BOU+), regarding VERI experience. For sludge tests, total cumulative applied air flow was 2 times higher for BOU+ than for BOU-. All the results presented in this paper are the average values of each modality.

In both experiments, all the reactors were mechanically turned several times: biowaste reactors (BIO+ and BIO-) were mechanically turned on the 27th and the 42nd day. Sludge reactors (BOU+ and BOU-) were mechanically turned on the 14th, 27th and 41st day. All the 12 assays were screened on the 83th day

2.2 Pilot devices

For the both experiments, three different pilot scale devices were used during the experiments: six composting reactors, a mechanical turning pilot and a screening pilot.

- Composting reactors: six closed and insulated cylindrical 160L reactors were employed to carry out the experiments. Fresh and dry air was blown from the base of the reactors. Waste core temperature was monitored (platinum-resistance thermometer Pt100, 0-100°C ±1°C) as well as oxygen concentration in emitted gas (electrochemical cell, 0-25%). (Bacheley et al, 2009)
- Mechanical turning pilot: composting reactors contents were individually mixed with this instrument. It has got a 65L cover that enhances the reactors headspace; the cover has a conduct to sample gas in the headspace. The reactors are shaken and turned, as well as aerated in order to mix their content and to monitor gas emissions.
- Screening pilot: this pilot consists of a cylindrical trommel enclosed in a chamber. The confinement permits to sample the gas emissions.

2.3 Solid monitoring

Solid samples were collected before the introduction of the waste into the reactors and after screening by elementary sampling (a variant of NF EN 12579). Additional solid samples were collected by the U44-101 method after each mechanical turning and before screening. Each sample was grounded <1mm and dried at 105°C until constant weight (NF EN 13040). The organic matter (OM) was determined by calcination at 480°C (NF EN 13039). The biochemical composition of composed organic matter was determined using the Van Soest fractionation method (XPU 44-162, AFNOR 2005): separation of soluble organic matters (SOLwater and SOL NDF), hemicellulose (HEM), cellulose (CEL) and lignin fraction (LIGN).All these analyses were realised by the SADEF, COFRAC laboratory specialised in environmental and agronomic analyses.

2.4 VOCs and odours monitoring

Gas, in the headspace of reactors, during mechanical turnings (MT) and screening (SC) was regularly sampled. BIO+/BIO- reactors were sampled on 1, 7, 14, 20, 27 (MT), 35, 42 (MT), 56, 70 83 (SC) days and. BOU+/BOU- reactors were sampled on 1, 7, 14 (MT), 21, 27 (MT), 35, 41 (MT), 56, 70 and 83(SC) days. A lung sampling system was used to collect gas into an inert bag (Tedlar® or Nalophane®). The main advantage of this method is that the sampled gas does not pass through the pump.

VOCs were analysed by TA-TD-GC/MS, this method consists of the concentration of sampled gas on a Tenax® tube, followed by a thermo desorption (320°C) and gas analysis in a gas chromatograph coupled to a mass spectrometer. The column used in GC is specialized on VOCs separation (RTX-VRX; 60m x 1.4Hm x0.25mm).

VOCs analyses were accomplished by internal VERI laboratory. Odours concentration was analysed by a panel of human noses (NF EN 13725) by AROMA CONSULT, COFRAC laboratory. Results, presented in UOE.m^{-3} units, indicate the dilution to apply in order to make the sample not detectable by the half of the panel.

3 RESULTS AND DISCUSSION

3.1 Solid monitoring

3.1.1 Temperature and O_2 monitoring

The FIGURE 1 shows temperature evolution in the core of the waste and residual oxygen concentration in emitted gas.

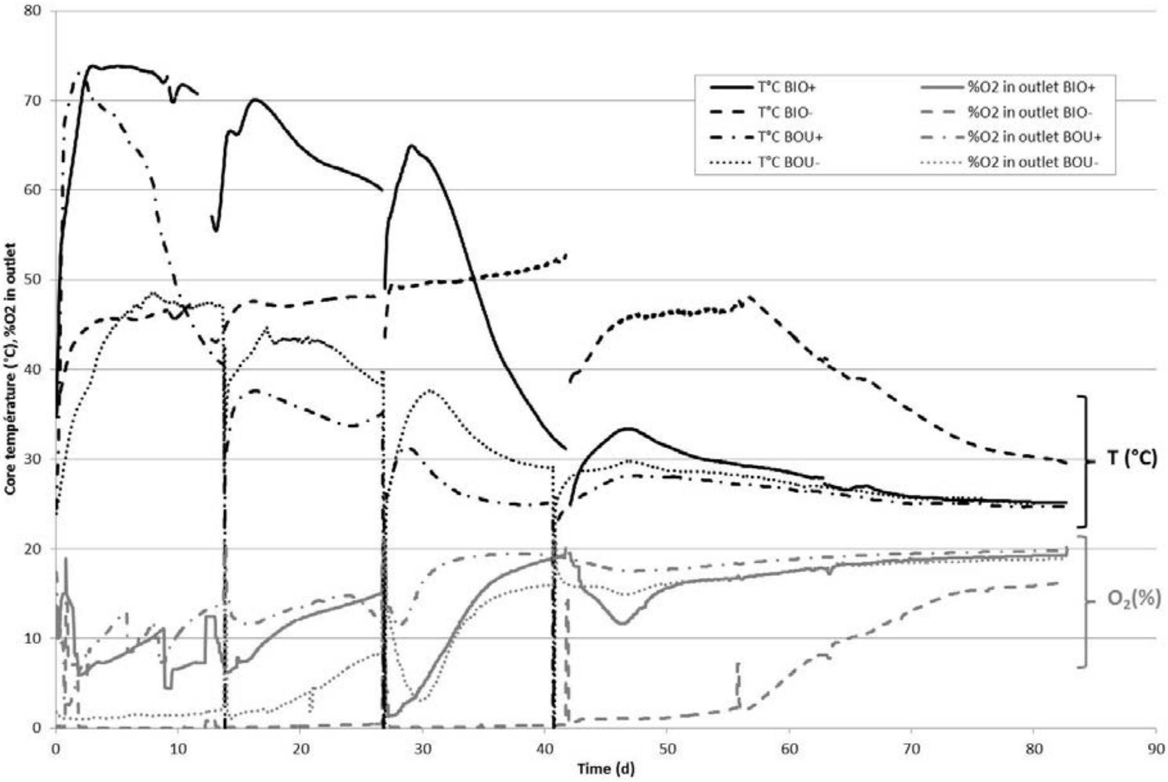

FIGURE 1 Evolution of temperature and oxygen concentration for BIO+/BIO- and BOU+/BOU-

Regarding the effect of aeration on biowaste: first of all, it can be observed that the maximum temperature is 21°C higher for BIO+ than for BIO- (73°C and 52°C respectively). Secondly, the time to reach maximum temperature is different for both modalities: 3 d in average for BIO+ and 41d for BIO-. Thirdly, O_2 concentration in emitted gas is greater than 5% during almost all the period for BIO+(no oxygen limited conditions);on the contrary, O_2 concentration in BIO- emitted gas is lower than 5% during 60 days (oxygen limited conditions).

The effect of aeration on sludge composting was similar: first of all, average maximal temperature for BOU+ is 24°C higher than for BIO- modality (73°C and 49°C respectively). Secondly, BOU+ reached its maximal temperature after 1d of composting, whereas for BOU- the temperature reached its maximum after 9d. Thirdly, average O_2 concentration in BOU+ flow gas is between 5 and 15% (v/v) during 30d, and superior afterwards; in the case of BOU-, average O_2 concentration in emitted gas was inferior to 5%(v/v) during 20d, and superior afterwards. It can be considered that BOU- present oxygen-limited conditions during 20d.

Regarding the waste effect of not-limited oxygen conditions, average maximum temperature was similar for both wastes. However, average temperature rise was slightly faster for the sludge (1d) matrix than for the biowaste one (3d). In addition, high temperature maintain (>60°C) was shorter for sludge matrix (10d) than for the biowaste one (30d). O_2 consumption explains this temperature difference (related to biological activity): a double quantity of oxygen was consumed in the biowaste composting than in the sludge composting (558 vs 337kg O_2 consumed/tDM$_0$).

3.1.2 Evolution of the organic matter biochemical fractionation:

All of the experiments present 3 steps (FIGURE 2): initial consumption of soluble organic fractions (SOL water and SOL NDF), followed by degradation of hemicellulosic and cellulosic fractions (HEM and CEL) and final reorganization of a part of the organic matter into a stable soluble fraction made of humic compounds (Francou (2003)). Final average percentage of organic matter degradation (40% OM loss) and composition are similar for the four modalities at the end of the experiment. Nevertheless, aeration had an important influence in biowaste organic matter evolution (Berthe (2012). This difference was not well-identified on sludge evolution, but soluble phase reorganization seemed to be slower for the limiting oxygen modality.

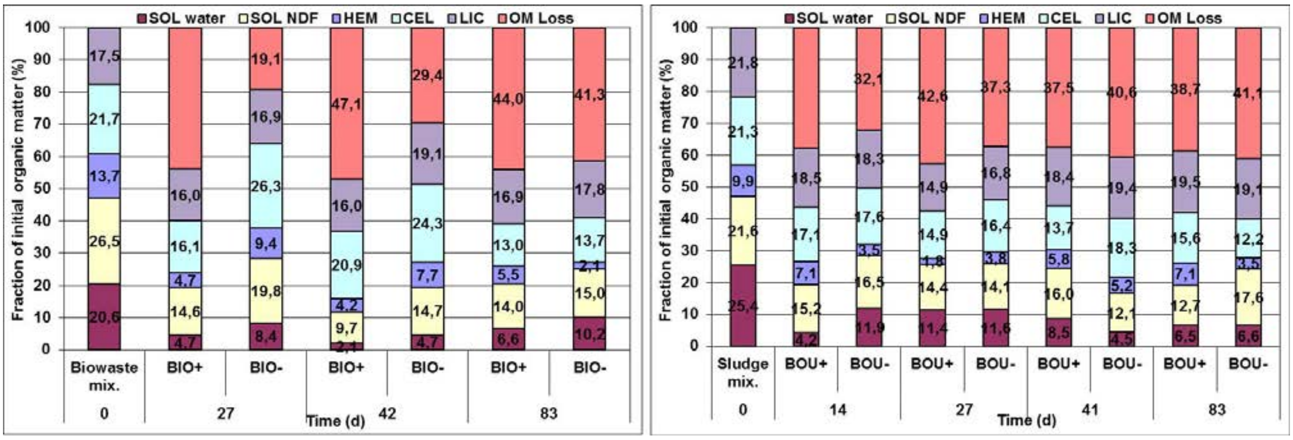

FIGURE 2 Evolution of Organic matter fractionation for BIO+/BIO- (on the left) and BOU+/BOU- (on the right)

3.2 VOCs monitoring

The 0 presents the number of quantified and identified Volatile Organic compounds (VOCs) and the main VOCs families in each experiment. Biowaste presents a larger variability of identified compounds than sludge, 127 and 62 different species respectively.

TABLE 1 Quantified and identified VOCs families

	BIO+/BIO-	**BOU+/BOU-**
Quantified compounds	30 compounds Principal families: terpenes, ketones and sulphur compounds	30 compounds Principal families: sulphur compounds, MAHs, terpenes, and ketones
Semi-quantified compounds	127 compounds 11 families, the principal ones: terpenes, esters, ketones, alcohols	62 compounds 11 families, the principal ones: sulphur compounds, MAHs, terpenes, ketones, alcohols

Terpenes are the most important family during biowaste composting, but they are the third family in concentration for sludge composting, after sulphur compounds and Monocyclic Aromatic Hydrocarbons (MAHs).

Bacheley (2009) and Mallard (2005), have already shown the same distinguishing VOCs families of each waste. The particularity and the novelty of this paper is the temporal evolution of those gases: there is a regular measure that allows comparing the values during a same campaign and between two different wastes.

3.2.1 VOCs monitoring for biowaste (BIO+/BIO-)

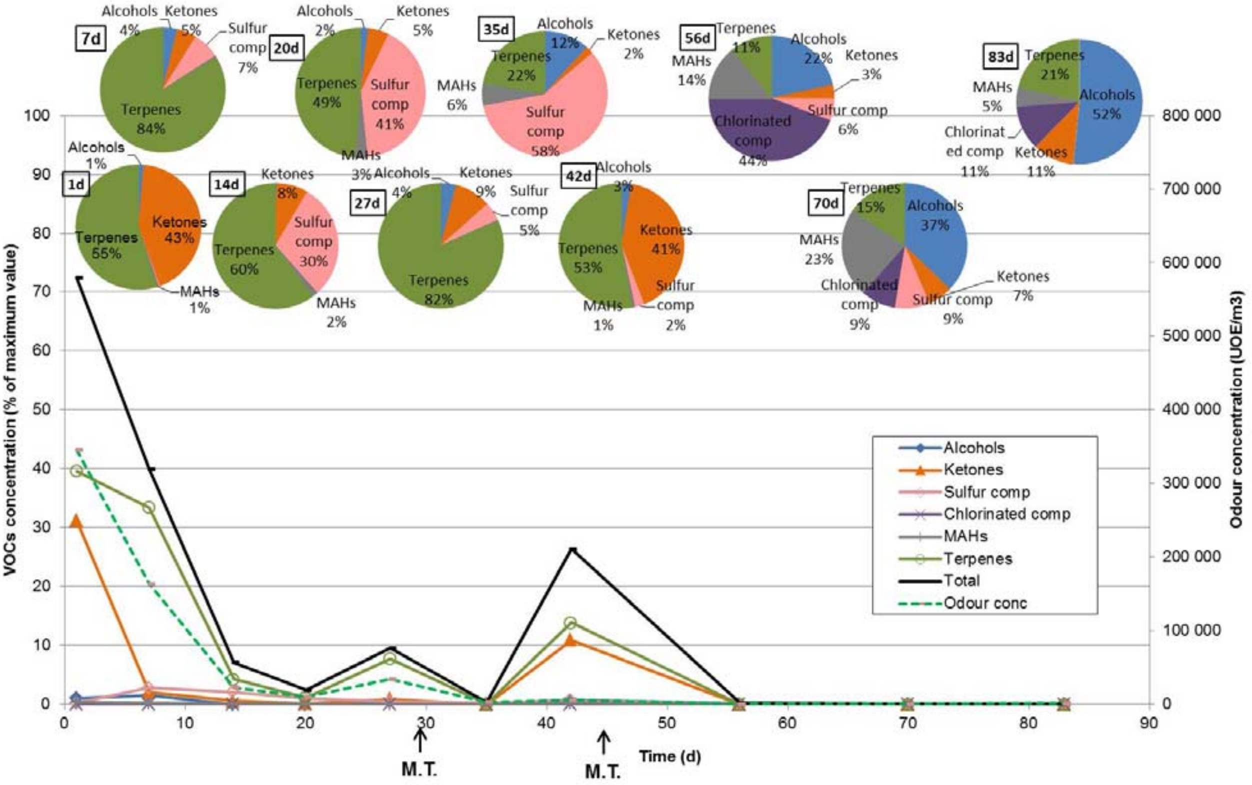

FIGURE 3 Evolution of VOCs families quantitative concentration and odours for BIO+

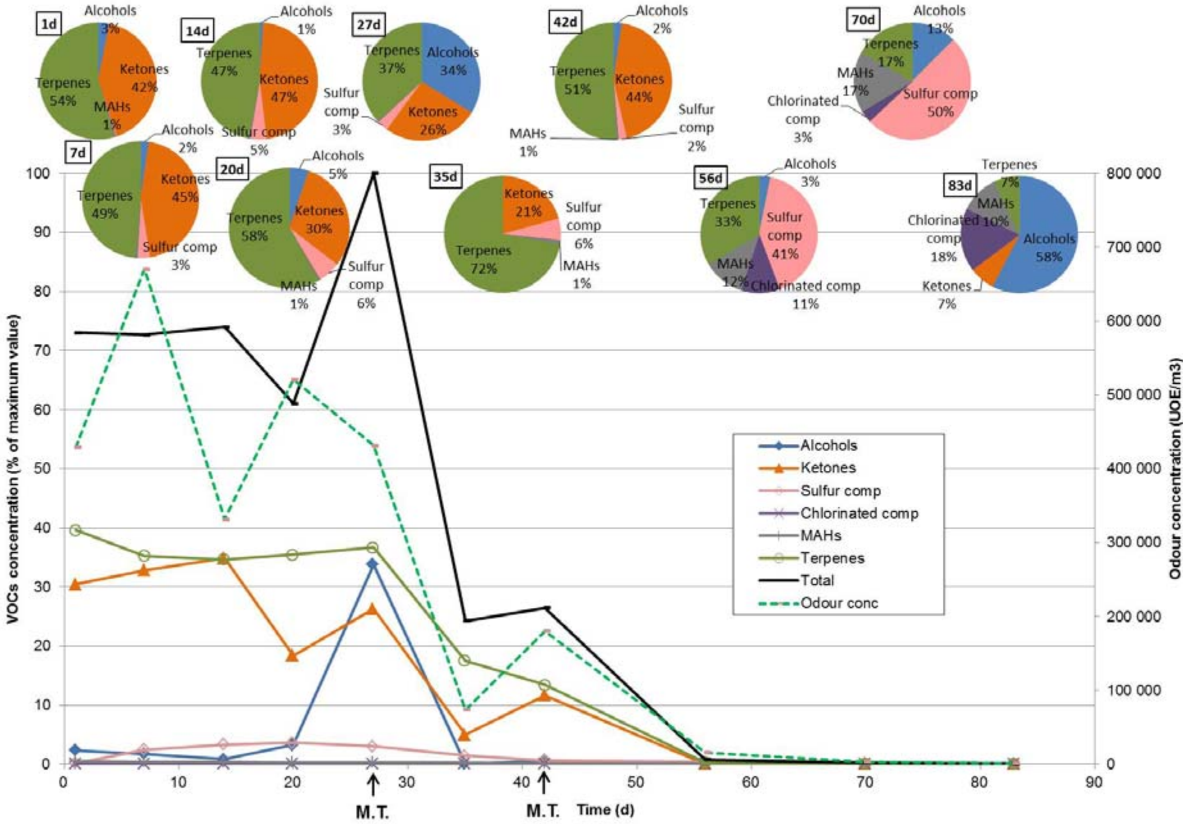

FIGURE 4 Evolution of VOCs families quantitative concentration and odours for BIO-

The figures 3, 4, 5 and 6 represent quantitative VOCs emission and odour concentration, they are the combination of two graphics : the linear one shows the evolution of VOCs in composting (left axis), these results are exposed as percentage of the maximum VOCs quantified emission (BIO-, 27d); it also presents the evolution of odour concentration (right axis). The linear graphic represents discrete values (11 sample times), linking lines are added to make easier their reading. The circular graphics present the proportion of quantified VOCs families in each sample.

Total quantified VOCs during biowaste composting (FIGURE 3 for BIO+ and FIGURE 4 for BIO-), show that the aeration has an important influence on VOCs emission. The two modalities present similar VOCs emission levels the first day of composting; nevertheless, after one week, the BIO+ reactors show a two times lower quantified VOCs concentration than BIO-, this difference increases between the 2nd and 6th weeks of composting (BIO+ concentration was ten times lower than BIO-). In this case, we can observe that, in both modalities, mechanical turnings (M.T.) enhance VOCs emissions, specially the first one for the oxygen-limited modality. Results are surprising for the second mechanical turning for BIO+, odours results (see paragraph 3.3 Relationship between VOCs and odours evolution) do not validate the VOCs evolution in this point, a sampling or analysing problem might have happened; therefore, the biowaste results on this point are not discussed in this paper.

In both modalities, terpenes and ketones are the main quantified VOCs families. The first day, the two modalities present similar concentrations; afterwards, terpenes concentrations are in average ten times higher for the oxygen limited modality than for BIO+ from the 1st week to the 8th and ketones from the 2nd one to the 8th. BIO- reactors also present higher alcohol emissions during the first four weeks, especially during the first mechanical turning when its concentration was one hundred times higher than for BIO+ one. Finally, sulphur compounds emission also appeared to be correlated with anaerobic conditions: an emission peak is observed on the 14th day for both modalities, just after some electrical problems affecting the aeration system; this level of emission remained stable until fifth week in BIO- modality whereas for BIO + this peak is punctual.

3.2.2 VOCs monitoring for sludge (BOU+/BOU-)

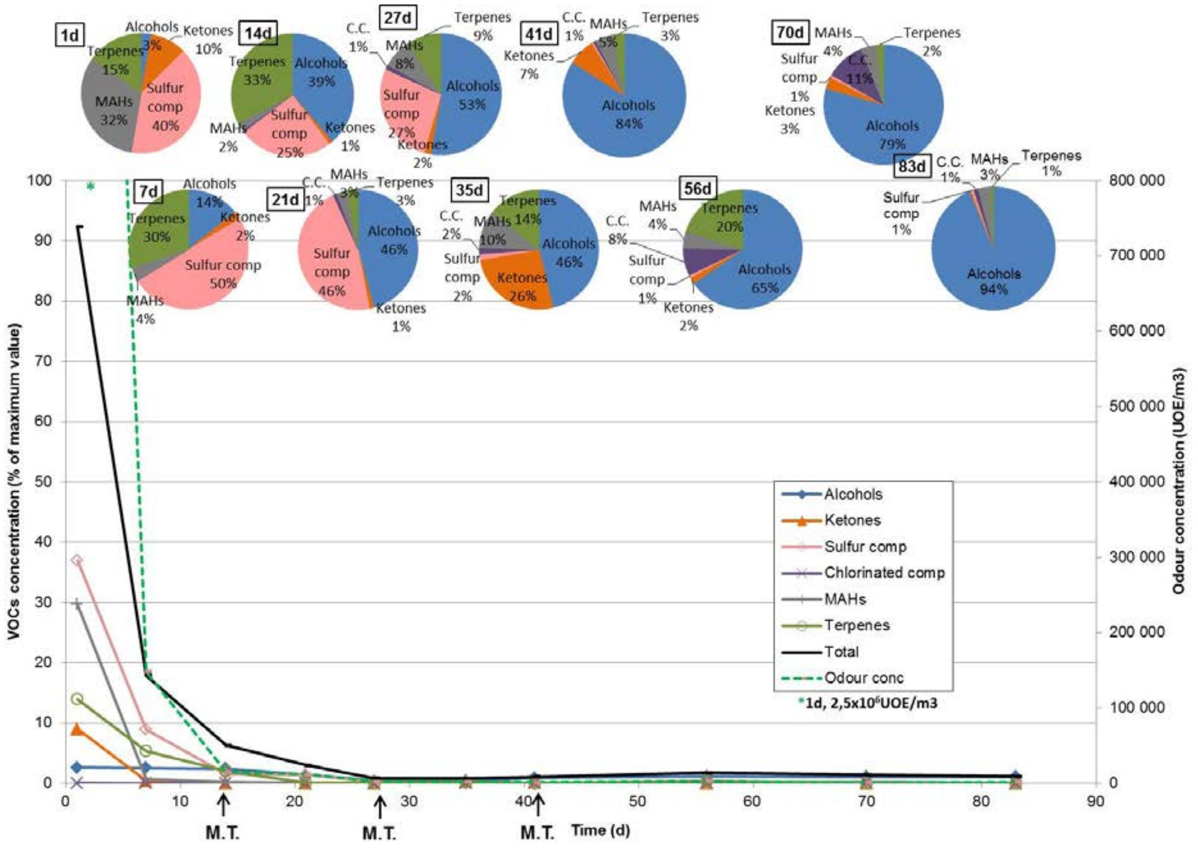

FIGURE 5 Evolution of VOCs families quantitative concentration and odours for BOU+ (N.B. “C. C.” stands for chlorinated compounds)

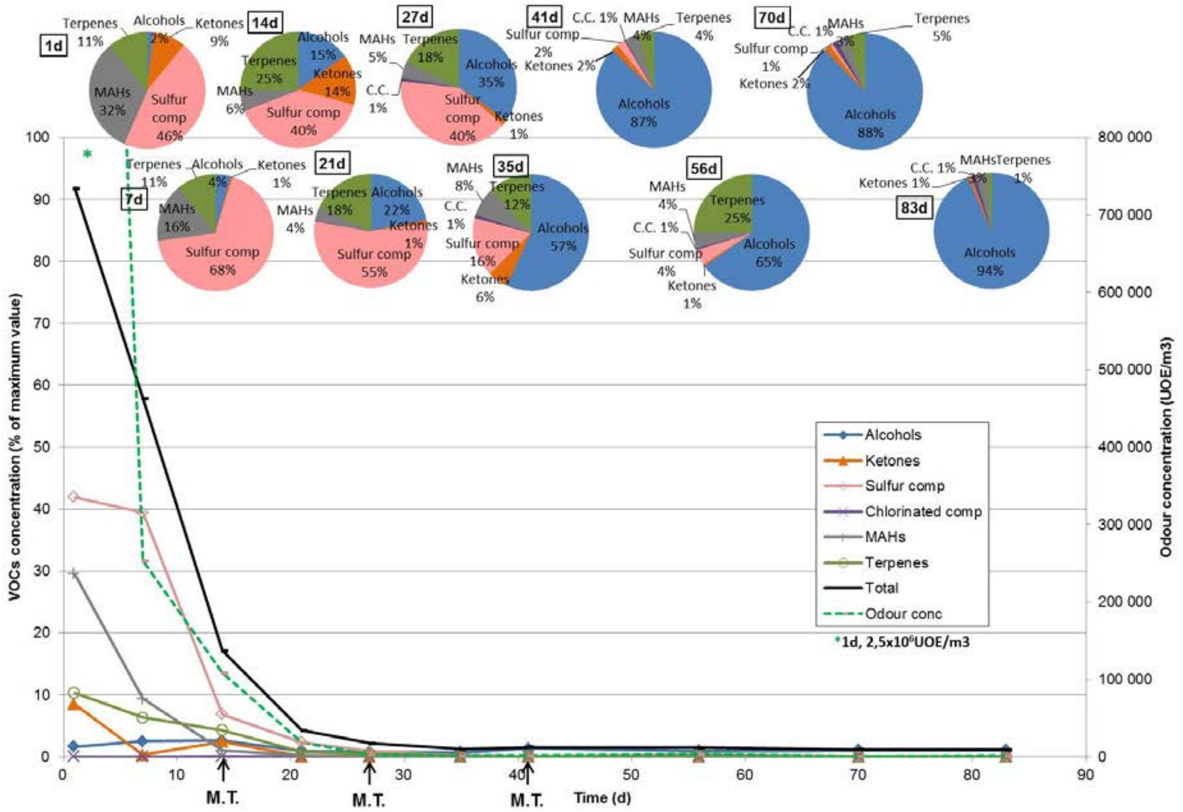

FIGURE 6 Evolution of VOCs families quantitative concentration and odours for BOU-

The evolution of total VOCs concentration for sludge composting (FIGURE 5 and FIGURE 6) is characterized by a deep decrease during the first and second weeks; it remains low and stable during the rest of the composting experiment. The decrease rate is double for the sufficient aerated modality than for the limited one. Mechanical turnings do not seem to affect the total quantified VOCs emission.

Sulphur compounds quantified concentration are majority during the first 21 days in BOU+ modality and the first 27 days in BOU- one. Alcoholic fraction of quantified VOCs becomes the major fraction from the14th day (BOU+) or the 27th (BOU-) days of composting to the end of the experiments. Emitted monocyclic aromatic hydrocarbons (MAHs) concentration is similar for both modalities at the first day, but decreases faster for BOU + than for BOU-. Terpenes and ketones quantified concentrations are similar for the two modalities during all the experiment.

The evolution of VOCs emissions during biowaste and sludge composting in not-limited aeration conditions is shown on FIGURE 3 and FIGURE 5. First of all, the evolution of total VOCs concentration is different for each waste: they are higher and more persistent for biowaste than for sludge. Secondly, mechanical turnings did not have influence on sludge matrix emissions, but their impact was important on biowaste ones.

Terpenes and ketones are the most emitted families in the phase of aerobic fermentation for biowaste (0-6th weeks), whereas sulphur compound where the family that characterised sludge aerobic fermentation. For both wastes, alcohols are the main quantified VOC in curing phase (especially in sludge composting); nevertheless this emission was low.

The results obtained with the semi-quantitative screening (not shown) were similar to the quantitative ones. Same kinetics of total emissions and same VOCs families were found with this method. Because of semi-quantitative units (µg eq toluene/m3 stands for the concentration of toluene that would show the same peak surface in the TD-GC/MS analysis), it is not possible to compare the importance of different VOCs families, but only to hypothesize an approach of their evolution. The main difference between semi-quantitative and quantitative analyses was an important esters emission in biowaste composting tests. Esters of BIO+ presented the same behaviour as terpenes; in the case of BIO-, esters emissions presented high emissions during the first 35 days, and two peaks on the 7th and 27th day.

3.3 Relationship between VOCs and odours evolution

In this section, some aspects for the relationship between VOCs and the odours evolution for biowaste and sludge composting are discussed. Previously, it is necessary to remind that these values have been obtained in pilot reactors, their concentrations are not comparable to other composting systems with different headspace volume or conditions; nevertheless, their evolution might be transposable to other systems. (Bacheley (2009))

The evolution of quantified VOCs concentration and measured odour concentration are presented previously on the FIGURES 3 to 6. Odour concentration presents a similar evolution to total VOCs one. For BIO+, BOU+ and BOU-, maximum odour concentration is measured during the first day of composting and it decreases rapidly; however, BIO- odour concentration is significantly higher than BIO+ one during 42 days; this evolution is not as similar as the other modalities to total VOCs concentration evolution, probably due to the cross effects of VOCs on odours (odours are not additive and their evolution is not always linear with concentration). Individual analyse of the three BIO- modalities showed that their odorous evolution was the least repeatable one of all the experiments.

Maximum odour concentration is reached during first day of sludge composting. These high values seem to be related to sulphur compounds concentration: with a similar total VOCs concentration, sludge reactors presented an odour concentration six times higher than biowaste ones.

4 CONCLUSIONS

In this paper, the behaviour of specific VOCs and odour concentration are compared regarding the type of waste and the aeration management. Temperature and oxygen evolution show two different processes: on the one hand, the not limited oxygen composting (BIO+ and BOU+), where high temperatures (above 70°C) are rapidly achieved; on the other hand, a partially oxygen limited composting, where mesophyll conditions (35 to 55°C) are kept during all the organic matter consumption.

VOCs and odours concentration evolution show that each waste presents its own chemical and odorous signature: the biowaste composting is characterised by terpenes and ketones emissions; whereas the most important VOCs during sludge composting are sulphur compounds and MAHs. These different VOCs mixtures present different odour concentration. For a similar VOCs concentration, BOU+ and BOU- present more intense odour concentration than for biowaste modalities. Mechanical turning effect on VOCs and odours emission is identified during biowaste composting, whereas screening do not affect any of the experiments.

On the one hand, oxygen limitation enhances permanent nuisances throughout the composting process. On the other hand, in all the sufficient aerated experiments, odorant nuisances are specially focused on the active phase of the process, being largely less important in the curing phase. The management of the aeration during the composting process is thus essential and demonstrated in this case, in order to reduce odorous nuisances in the environment of composting plants.

5 ACKNOWLEDGEMENTS

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(245) REVISION OF WASTE TREATMENT INDUSTRIES IED/BREF DOCUMENT - WHAT ARE IMPLICATIONS FOR MBT-OPERATORS?

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EXECUTIVE SUMMARY

Indications on the approach for composting and digestion plants in Germany will be given.
The European BREF documents (**B**est Available Technique **Ref**erence Documents) describe the **B**est **A**vailable **T**echniques (BAT) for low-emission operation of industrial plants which also include waste treatment plants. The definition of the best available techniques in terms of the BREFs is similar to that of the German term state of the art.
The legal basis is the **I**ndustrial **E**missions **D**irective 2010/75/EU (IED Directive) which replaced the **I**ntegrated **P**ollution **P**revention and **C**ontrol **D**irective of 15 January 2008 (IPPC Directive) with effect from 2010.

With the adoption of the IED Directive, the BREF Documents have been gaining increased legal weight. They must be observed in setting permit conditions and constitute a major element of the permitting process. Due to the ongoing progress in technology development, the BREF Documents must be reviewed for currency on a regular basis and revised, if necessary. The review process for the BREF Document starts in 2012.

In preparation of the revision process, documents describing the technical state of the art of German waste treatment plants are currently being developed on behalf of the German Federal Environmental Agency. The project includes data collection from various types of organic waste treatment plants including measurement of emissions in practice.

The Waste Treatment Industries BREF comprises more than 600 pages. In order to have a manageable document stakeholder proposed a sub-division into the Mini-BREFs with one for biological treatment. The German approach proposes a clear differentiation between residual waste treatment on the one hand and composting and anaerobic digestion of source-separated waste streams on the other and also to account for the different requirements for the respective plants, two separate Technical Documents have been prepared:

As results the following statements were made with regard to BAT:

- Engineering measures for emission reduction
- Definition of MBT technologies that are considered to be BAT
- General minimization requirement for consumption figures
- Suitable techniques for waste water treatment
- Suitable techniques for exhaust gas treatment

Moreover, if was possible to indicate at least examples of potential emission ranges of the MBT technologies.
The work on the Draft Technical Document prepared by Wasteconsult international in cooperation with the working group is completed. Comprising 55 pages plus an annex,

This paper describes the updating process of the BREF document focusing on the German contribution to the mechanical-biological treatment technology area. The work for composting and anaerobic digestion plants for biowaste is under progress so only an outlook can be given.

1 FUNCTION AND LEGAL BASIS

The European BREF documents (**B**est Available Technique **Ref**erence Documents) describe the **B**est **A**vailable **T**echniques (BAT) for low-emission operation of industrial plants which also include waste treatment plants. The definition of the best available techniques in terms of the BREFs is similar to that of the German term state of the art.
The legal basis is the **I**ndustrial **E**missions **D**irective 2010/75/EU (IED Directive) which replaced the **I**ntegrated **P**ollution **P**revention and **C**ontrol **D**irective of 15 January 2008 (IPPC Directive) with effect from 2010.
With the adoption of the IED Directive, the BREF Documents have been gaining increased legal weight. They must be observed in setting permit conditions and constitute a major element of the permitting process. Due to the ongoing progress in technology development, the BREF Documents must be reviewed for currency on a regular basis and revised, if necessary. The current (original) Waste Treatment Industries BREF was published in 2006 so that the underlying data date even further back.

The review process for the BREF Document is scheduled to start in 2012. In preparation of the revision process, documents describing the technical state of the art of German waste treatment plants are currently being developed on behalf of the German Federal Environmental Agency.

2 STRUCTURE

The BREF documents are structured into the following main chapters:

1) General Information
2) Applied processes and techniques
3) Current consumption and emission
4) Techniques to consider in the determination of BAT
5) Best available techniques
6) Emerging techniques
7) Concluding remarks

The Waste Treatment Industries BREF comprises more than 600 pages. The chapters listed above contain the information on all the process groups (e.g. biological treatment, chemical-physical treatment, etc.) considered. Due to the many cross-references and the spreading of the information over different chapters, the Document has proved to be difficult to manage. For this reason, both the German expert working groups on the Waste Treatment Industries BREF and the FEAD (European Federation of Waste Management and Environmental Services) have proposed that the Waste Treatment Industries BREF should be split up into self-contained Mini-BREFs on key technologies in future. Currently, a sub-division into the following Mini-BREFs is under discussion:

- Common techniques
- Biological treatment
- Physico-chemical treatment
- Material recovery
- Preparation of waste to be used as fuel

3 THE BREF PROCESS

3.1 In Europe

The BREF Documents are drawn up and revised by the IPPC BUREAU in Seville (Sevilla process) on the basis of the inputs submitted by the EU member countries. For the BREF review process, the member countries submit so-called Wish Lists describing the requested changes with a clear reference to the Main BREF Document including a substantiation of the requested change. The Wish Lists may be complemented by Technical Documents that describe the technical background in greater detail. These Technical Documents are of particular importance if the existing BREF does not yet contain any information on the technology in question or the BREF content needs major revision. Plans are for the IPPC Bureau to take up work on the new BREF by the end of 2012.

3.2 In Germany

3.2.1 Phase 1 (UFOPLAN project 2010-2011)

- Structuring and setting up technology-specific working groups
- Assessment of revision needs
- Collection of up-to-date data from the literature and, if necessary, by questionnaires to be specifically developed for this purpose
- Preparation of Technical Documents (reports)
- Translation of Technical Documents

The preparatory process is coordinated by the German Federal Environmental Agency. The work for the German input is being undertaken under an UFOPLAN project (FKZ 3709 44 305/2) awarded to gewitra GmbH and Wasteconsult international as joint contractors in December 2009. Phase 1 will be finalized shortly.

The contractor-side work on the biological processes presented in this paper was undertaken by Wasteconsult international.

3.2.2 Phase 2 (from 2012)

- Preparation of the Wish Lists
- Document submission to Seville:
  - Wish Lists complemented by
  - Technical Documents from Phase 1
- Support for the further Seville process

4 DRAFT GERMAN TECHNICAL DOCUMENT ON BIOLOGICAL WASTE TREATMENT

4.1 Work process

4.1.1 Expert working group

For the biological processes technology area, a working group was set up in which the following organizations were represented.

- ASA e.V.
- BDE
- BSR
- Fachverband Biogas
- gewitra (Contractor)
- Lobbe (on behalf of BDE)
- MEAB (on behalf of ASA)
- Federal Environmental Agency
- VCI
- VDMA
- VKS im VKU
- Wasteconsult (Contractor)
- Witzenhausen-Institut
- Nehlsen (temporarily)

4.1.2 Procedure

In order to ensure a clear differentiation between residual waste treatment on the one hand and composting and anaerobic digestion of source-separated waste streams on the other and also to account for the different requirements for the respective plants, two separate Technical Documents have been prepared:

- Composting and anaerobic digestion of source-separated waste
- Mechanical-biological treatment of residual waste

The following presentation is limited to the Technical Document on mechanical-biological treatment of residual waste.

- Corresponding section of the BREF 2006 needs major revision
- Preparation of a completely new document

Procedure for document preparation and result:

- Development of a questionnaire for emission and consumption data as well as other information. The questionnaires were created in the form of Excel files comprising the following spreadsheets:
  - General (basic data such as location, throughput etc.)
  - Treatment stages (including systems used)
  - Efficiency (consumption figures)
  - Waste input
  - Output and emissions (see Tables 1 and 2)
- Definition of technology groups (aerobic MBT, MBT with dry anaerobic digestion MBT with wet anaerobic digestion (MBS) and selection of example plants
- Dispatch of questionnaires to one treatment facility per technology via ASA e.V.
- Handover of the completed and anonymized questionnaires to the contractor by ASA e.V.

The analysis of the questionnaires showed the following picture:

- 4 out of 4 of the addressed facilities completed the questionnaire
- Implausibilities, delimitation problems and data gaps in the consumption figures
- Comprehensive data on exhaust gas emissions thanks to the requirements of 30th BImSchV

The data submitted were complemented by literature data. Due to the high quality of the exhaust gas emission data, it was considered worthwhile to conduct a subsequent survey of all German MBT facilities so as to obtain a representative database. In this way, it was possible to obtain emission data from 16 facilities.
This data basis was then used to complete and coordinate the Technical Document.

4.2 Results and work status

The following statements were made with regard to BAT:

- Engineering measures for emission reduction
- Definition of MBT technologies that are considered to be BAT
- General minimization requirement for consumption figures
- Suitable techniques for waste water treatment
- Suitable techniques for exhaust gas treatment

Moreover, if was possible to indicate at least examples of potential emission ranges of the MBT technologies.
The work on the Draft Technical Document prepared by Wasteconsult international in cooperation with the working group is completed. Comprising 55 pages plus an annex, it is even shorter than the corresponding section in the BREF 2006. The draft has been submitted to the representatives of the German Provinces for commenting.

5 BEST AVAILABLE TECHNIQUES FOR MBT TECHNOLOGIES

The final coordination process for the Technical Document is still under way. Essential core statements from the author’s point of view are presented in the following subsections.

5.1 What characterizes the best available techniques?

BAT in terms of the BREF documents is characterized by low emissions and low resource and energy use associated with the operation of the plant (always to be judged against the background of the treatment objective). As well as the type of construction and the processes employed, the plant operating conditions have an influence on emissions and consumption figures.

The resource/energy use and emissions associated with a BAT plant are acceptable if this plant meets the envisaged treatment objectives. For mechanical-biological treatment of residual waste, these are defined as follows:

- Maximum possible degradation of the biodegradable fraction in the output streams going to landfill in order to minimize landfill gas and leachate emissions
- Maximum possible recovery of waste components amenable to material/energy recovery
  - extraction of waste components lending themselves to material recovery / valuable materials
  - production of fractions for energy recovery from high-calorific waste components unsuitable for material recovery

5.2 General engineering measures for emission control

- Unloading stations, feed systems, mechanical and biological treatment stages (up to the end of intensive composting as a minimum) must be fully enclosed. All exhaust gas streams must be captured and treated.
- Delivery vehicle access to the buildings via airlocks or high-speed gates or alternatively, airwall systems or air curtains at the gates (receiving area)
- Maintaining a negative pressure in the buildings
- Effective building ventilation and source capture systems
- Discharge of treated exhaust gas (exhaust air) via a stack
- Largely enclosed design of the mechanical treatment systems
- Liquid-impervious bunker
- Use of wear and corrosion-resistant materials to preclude leakage
- Closed process water and condensate storage tanks
-

5.3 MBT processes constituting BAT

When using fully enclosed intensive composting and anaerobic digestion stages, the following biological process plants are considered to be BAT:

- Aerobic MBT
- MBS
- MBT with part-stream dry digestion
- MBT with full-stream dry digestion
- MBT with part-stream wet digestion (not implemented in Germany)
- MBT with full-stream wet digestion
- MBT / MBS with percolation stage

5.4 Consumption figures

Consumption figures must be minimized. As they depend to a large extent on the selected process and the treatment objective, it is not possible to make any generally valid statements on this issue.

5.5 Waste water

Aerobic maturation systems can be operated with zero waste water discharge, except for minimal quantities. Aerobic MBT plants, by contrast, generate significant waste water volumes due to condensation. Thermophilic part-stream anaerobic digestion processes likewise allow zero-waste water operation (except for a few weeks per year as the case may be).

Full-stream anaerobic digestion systems, in particular wet digestion systems generate 0.3-0.7 m³ waste water per Mg of waste input. With few exceptions, the waste water generated by mechanical-biological treatment plants is sent to external plants for treatment. These are outside the scope of this BREF.
Where the waste water is treated on site, the following processes and process combinations should be considered:

- ultrafiltration / reverse osmosis
- activated carbon adsorption
- biological treatment (normally in combination with another process).

Waste water volumes must be minimized through recirculation and multiple use as far as technically viable.

5.6 Exhaust gas emissions

5.6.1 Exhaust gas management

For the exhaust gas to be treated under controlled conditions, it must be captured. This is also the prerequisite for consistent mass balancing of the emissions. Moreover, fugitive emissions from the system must be minimized. As, in a properly operated plant, the greater part of the emission is generated during the intensive composting phase, a distinction must be made between the delivery building, mechanical treatment, intensive composting and maturation stages. State-of-the-art intensive composting systems are characterized by the following features as a minimum:

- They must be fully enclosed or housed in closed buildings that are provided with airlocks and a forced ventilation system to adjust a slight negative pressure inside the building. Where full enclosure at the feed, discharge or transfer points and during windrow turning is not or only partly feasible, the exhaust air streams must be captured and routed to an exhaust air cleaning system.
- Forced aeration and exhaust air (exhaust gas) capture

Forced aeration or adequate windrow turning intervals are needed to ensure a sufficiently fast biodegradation process and to minimize the formation of anaerobic pockets and resulting methane formation unless minor methane generation is desirable to ensure largely autothermic operation of the RTO.
An enclosure of the maturation stage will not be required if aerobic operating conditions are ensured by forced aeration or sufficiently short turning intervals. As a matter of principle, the maturation stage must be roofed-over to prevent water logging and as a result, insufficient aeration of the composting material.

The anaerobic stages and the downstream aerobic post-treatment stage must likewise be fully enclosed.
Emission reduction potential through the use of low-emission processes and technologies, e.g. exhaust gas minimization through reuse of building exhaust air as process air for the composting process, must be fully exploited to the extent this is technically feasible and economically viable and does not contravene occupational health and safety aspects.
An additional point to be taken into account for intermittently operating anaerobic digestion systems is that the tunnels are purged prior to emptying, e.g. with waste gas from the CHP plant, and that the purge gas is routed to oxidation (gas engine or RTO).

Off-transport of dusty materials must be in closed containers.

5.7 Exhaust gas cleaning processes

The following exhaust gas cleaning processes are considered to be BAT:

- Electrostatic precipitator (only if required as an additional particulate control device)
- Fabric / baghouse filter (only if required as an additional particulate control device)
- Acid scrubber in the case of high exhaust gas ammonia loads
- Biofilter (only for low-strength exhaust gas streams from the mechanical treatment stage and the feed systems)
- Regenerative thermal oxidation
- Co-incineration of exhaust gas in a MSWI, an RDF-fired power plant or in the case of high-strength exhaust gas streams, in a CHP plant

In order to avoid operating upsets and a resulting decline of the removal efficiency, a biofilter must be properly operated. The following operating upsets must be prevented through regular inspections and the adjustment of proper operating conditions:

- Temperature extremes (both low and high temperatures)
- Alkaline (due to $NH_{3)}$ and acidic (due to nitrification) pH values
- Too high or too low a moisture content in the biofilter
- Suitable filter area /space loadings matched to the exhaust air composition

In Germany, the use of a thermal oxidation unit (RTO) is required for exhaust gas cleanup - at least of the high-strength MBT exhaust gas streams - to ensure compliance with the emission limits mandated by 30th BImSchV, and is also considered to be the state of the art / BAT.

6 CONCLUSION: IMPLICATIONS FOR MBT PLANT OPERATORS

In future, the BREF content will be a major element of the permitting process. As the review process based on the national inputs has not yet started at the European level, it is not possible to make any statements on the exact content of the revised BREF at this stage.

The content presumably contributed by Germany to the Seville process should not result in tightened requirements for German plants as these already reflect the state the art and thus are BAT. More stringent requirements may, however, result for plants in some other EU member countries.

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(38) SUGAR CANE TRASK AS AN ALTERNATIVE SUBSTRATE OF BAGASSE FOR PRODUCTION OF THE *PLEUROTUS SAJOR CAJU* MUSHROOM

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EXECUTIVE SUMMARY

The *Pleurotus sajor-caju* mushroom (oyster mushroom) grows naturally on logs, tree stumps and dead wood (Bonatti *et al.*, 2003). Apart from being a high protein food source, this white basidiomycete is increasingly triggering interest in the pharmaceutical field owing to its immeasurable health benefits. Subsequently, the *Pleurotus sajor caju* has several properties acting as immunomodulators, anticancer and antimicrobial agents, having antihypertensive effects and lowering blood lipid concentrations (Daba *et al.*, 2008). Moreover, the mushroom's extensive range of temperature adaptation and its ability to produce a broad spectrum of lignocellulolytic enzymes, thus favouring ease of substrate availability, makes it the easiest, fastest, cheapest to grow, requiring less preparation time and production technology. Likewise, the biotransformation of wastes, high in lignocellulose, by the *Pleurotus* fungi, is of economic relevance to the agricultural sector while dealing with environmental issues concerning waste disposal. In Mauritius, bagasse, a by-product of the sugar cane (*Saccharum officinarum*) is used for the commercial cultivation of the *Pleurotus sajor caju* mushroom. Sugar cane trash is being considered as an alternative substrate for oyster mushroom production. The physicochemical characteristics of bagasse (B) and sugar cane trash (mainly dry leaves) (ST) have been studied and compared in terms of moisture content (MC), total dry solids (TDS), wet bulk density (WBD), particle size (PS), water holding capacity (WHC), pH, electrical conductivity (EC), total volatile solids content (TVS), ash content, carbon content, nitrogen-phosphorus-potassium contents (N-P-K), crude fibre content (CF) and crude protein content. B had a higher MC (62.1%) than ST (40.5%). Conversely, the TDS content in ST was superior (59.5%) as compared to B (37.9%). Particle size in B and ST were similar, ranging from 3.327mm to 6.680mm. WBD for TS was lower (70.2 kg/m^3) relative to B (130.3 kg/m^3). The WHC in B exceeded (684.6%) that of ST (515.6%). pH in B and ST were nearly equivalent, 6.20 and 6.26 correspondingly. However, ST had a higher EC (2.90 mS/cm) parallel to B (0.64 mS/cm). TVS in B was above (94.599%) that of ST (93.378%). In contrast, ash content in ST levelled above (6.622%) that of B (5.401%). The carbon content of B and ST were nearly analogous, 52.56% and 51.88% respectively. The N-P-K content for B was 0.66%, 0.14% and 0.28% correspondingly in comparison to the N-P-K of ST, 0.99%, 0.07% and 1.2%, in that order. CF in B was higher (38.78%) than in ST (35.69%). ST had a superior crude protein content (6.16%) compared to B (4.11%). Substrates B and ST were each weighed to 750g and mixed with 10% calcium carbonate and 10% crushed maize seeds, by mass. The MC for both substrates was adjusted to 70%. Each mix was then packed in polypropylene bags, plugged with PVC tubing (diameter and length = 50mm) and a 50mm-thick sponge. Each substrate had a total of 9 replicates. The 18 bags were then pasteurized at 70°C for six hours. On subsequent cooling, each bag was inoculated with 5% *CC 114 Pleurotus sajor-caju* spawn and then incubated at temperatures ranging from 23°C to 27°C with relative humidity maintained at 80%. The onset of mycelia colonisation could be observed on the fifth day of incubation on both media. The spawn run for B and ST took 26 days and 30 days respectively. The first flush of mushroom was harvested after a 35-day incubation on B and 39-day incubation on ST. A total of three flushes were harvested from both B and ST. The average biological efficiency on bagasse (ABE) was superior (83.82 ± 4.9%) to that of sugarcane trash (74.49 ± 5.2%). This study suggests the possibility of replacing bagasse with sugar cane trash for the cultivation of oyster mushrooms. Still, supplementation of sugar cane trash with other lignocellulosic wastes, like banana leaves, becomes essential to raise the biological efficiency of substratum utilisation.

1 INTRODUCTION

The oyster mushroom is the second most cultivated edible mushroom worldwide after the button mushroom (*Agaricus bisporus*) (Sánchez, 2010). It is consumed worldwide for both gastronomic and medicinal purposes. This gourmet mushroom is rich in proteins, vitamins, fibres, carbohydrates, several minerals and has a low fat content (Bonatti *et al.*, 2003). It is common in Chinese cuisine and progressively being popularised in other countries. The mushroom is typically served raw, in soups or stir-fried with soy sauce. Additionally, both the fruiting body and mycelium contain compounds with a wide ranging antimicrobial activity and have been extensively used in traditional medicine for curing various types of diseases since it also has antiviral, anticancer, antitumor, anti-inflammatory, immunomodulating activities and inhibits cardiovascular diseases (Akyüz *et al.*, 2010). This white-rot basidiomycete grows primarily on hardwoods and numerous pasteurised non-composted and composted lignocellulosic wastes. In Mauritius, the oyster mushroom (*Pleurotus sajor-caju*) is cultivated on crushed bagasse and maize seeds, supplemented with calcium carbonate. Conversely, in a couple of years, a shortage of the bagasse substrate will be expected due to the following reasons:

- The converging of bagasse towards thermal power stations for producing electricity under the Master Plan for Renewable Energy as part of the Long-Term Energy Strategy 2009-2025 report.
- The present closing down of sugar factories with the diversification of agricultural lands.
- The tendency of the bagasse substrate accommodating green moulds and other undesirable fungal growths impairing the colonisation and hence fruiting of *Pleurotus sajor caju*. This has an economic significance because considerable wastage is incurred.

Growers are progressively having problems in obtaining the bagasse substrate. Therefore, a rise in commodity price and a drop in mushroom production is predictable. The challenge right now is to find an alternative substrate using the local feedstocks available. Based on the estimations of Hassuani *et al.* (2005), it is inferred out of the 4 million tonnes of cane produced, waste cane trash is about 611,000 tonnes. This value presents a substantial emerging lignocellulosic source for growing oyster mushrooms. The trash recovery option not only adds an extra revenue to the sugar industry but also provides an environmentally sustainable solution towards alleviating the load of refuse left to compost in the fields and encourages the country towards self-sufficiency in food production. Furthermore, this strategy can be integrated in the present Mauritian agricultural system to enhance farmer income on a sustainable basis.

1.1 Background

Mushrooms are the reproductive organs of the largest and most complex fungi. Neither belonging to the animal nor to the plant monarchy, mushrooms are more closely related to Kingdom Animalia than to Kingdom Plantae (Nikoh *et al.*, 1994). Shalchian-Tabrizi *et al.* (2008), have placed fungi with the animals in the monophyletic group of opisthokonts. Hence, the 2007 classification of Kingdom Fungi recognises seven phyla on the basis of characteristics of their sexual reproductive structures, namely, Microsporidia, Chytridiomycota, Blastocladiomycota, Neocallimastigomycota, Glomeromycota, Ascomycota and Basidiomycota. The last two groups have the greatest economic importance. Ascomycota includes delicacies like truffles and morels while Basidiomycota encloses the majority of mushrooms which are successfully cultivated commercially. Since all times, human beings have been fascinated with mushrooms. Mushrooms were praised by the Pharaos, Greek and Romans as a noble food, providing strength for battle and a gift of God, respectively (Winston, 2012; Daba *et al.*, 2008). Wasser (2005) mentions the first written record of mushroom cultivation dating back to the Sung Dynasty (A.D. 960–1127). Because of their proved nutritional and health-added benefits, the world market for fresh, edible speciality mushrooms including *Agaricus*, *Pleurotus* and *Lentinula* has experienced considerable growth over the past two decades with an annual production estimated from 3,6 MT in 2009 to nearly 6 MT in 2010 (FAOSTAT, 2010). Potential applications of the mushroom's lignocellulolytic enzymes are being considered in recycling lignocellulosic wastes into highly alimentative protein-rich biomass as well as their integration in chemical, fuel, food, textile, laundry, pulp and paper industries along with animal feed production (Diaz *et al.*, 2011; Yadav *et al.*, 2010). Most edible basidiomycetes are saprophytic in nature, degrading lignocellulosic substrate materials and gaining nourishment from the cell wall structural polymer of lignocelluloses producing fruiting bodies (Cheung, 1998). The suitability of the substrate in question and proper environmental conditions defines the two distinct phases in the lifecycle of the basiodiomycete, namely, the vegetative stage, mitotic and meiotic reproduction thus break down of complex nutrients for absorption, and the fruiting stage. Compared to Agaricus and Lentinula, Pleurotus mushrooms are the easiest, fastest and cheapest to grow, require less preparation time and production technology (Mandeel *et al.*, 2005). Hence, they are increasingly being popularised because they have a wide range of temperature adaptability and most of their substrates are readily available (Sales-Campos *et al.*, 2009). Furthermore, Juárez *et al.* (2010) and Sagar *et al.* (2009) state after mushroom harvest, the 'spent compost' can be considered as follows:

- Animal feed as the mushroom mycelium boosts its protein content.
- Substrate for cultivation of other mushrooms.
- Soil conditioner, since it is still rich in nutrients and polymeric components that enhance soil structure.
- Permaculture component.
- Paper manufacture and cardboard production.
- Agents that digest pollutants, like polychlorinated phenols on landfill waste sites as it contains populations of microorganisms able to digest the natural phenolic components of lignin.
- Enzyme production.
- Biogas production.

Generally, almost all the available, lignocellulosic substances are likely to be used as substrate for the *Pleurotus spp*. (Ahmed *et al.*, 2009). Substrates include banana leaves, grass (*Saccharum munja*), coconut coir, coffee pulp, corn cobs, corn leaves, crushed bagasse, molasses waste, mango fruits and seeds, peanut hull, sugarcane leaves, water hyacinth, wheat straw and brassica strawwood chips have also been used (Buah *et al.*, 2010; Dunkwal and Jood, 2009; Cangy and Peerally, 1995). Alkaline additives are added during substrate preparation to maintain a pH favourable to fungus growth while increasing substrate degradation and mushroom yield (Adebayo *et al.*, 2010). The *Pleurotus sajor caju* mushroom was introduced in 1982 in Mauritius by the CATT (Chinese Agricultural Technical Team). This mushroom variety was found to be most suitable to the local agro-climate, furthermore it was a vigorously growing high yielding strain and hence economically viable. The present local substrate formulation for *Pleurotus sajor-caju* mushroom is fundamentally, bagasse as substrate, maize seeds as nutrient source and calcium carbonate as supplement. Bagasse is a by-product of the cane industry. It is the fibrous stalk remaining after the sugarcane (*Saccharum officinarum*) has been crushed for juice extraction. This agro-waste is still abundant through the island as the sugar sector remains among the six economic pillars of the country. The bagasse is allowed to ferment for two to three days and is moistened to 70% water content. The maize seeds and calcium carbonate are blended with the substrate into a homogeneous mixture. Polypropylene bags are filled with the resultant blend. The opening of each bag is fitted with a 50 mm diameter-PVC ring and plugged with a 50 mm length-cubed sponge. The bags are pasteurised, cooled and inoculated with the mycelium spawn in a sterile area. They are incubated in total darkness at 90% to 100% relative humidity. Spawn run takes two to three weeks at temperatures varying from 20°C to 29°C. After complete colonisation, the sponge is removed and a 10 mm-diameter PVC ring is used to replace the previous one. The bags are left open for regular misting, normally twice or thrice a day, depending on ambient temperature. The relative humidity is lowered to 80% to 90%. Primordia formation will occur in three to five days after complete colonisation. Misting is continued until the fruit body is ready for harvest. Slight variations occur in the lifecycle phase time. Also, growth parameters depend on the type of substrate and local agro-climatic conditions. Sugarcane trash is dried sugarcane leaves and constitutes 10–20% of the weight of cane harvested, (Singh *et al.*, 2008; Yadav *et al.*, 1994). The amount of trash produced depends on the plant variety, age of the crop at harvest and soil as well as weather conditions. During the harvesting operation around 70-80% of the trash is left in the field with 20-30% taken to the mill together with the cane stalks as extraneous matter (Pankhurst, 2005).

Research objectives

- Evaluating mycelium growth and mushroom yield on bagasse and sugarcane trash
- Identifying possible substrate characteristics that can favour mushroom yield

2 METHODOLOGY

2.1 Determination of moisture content

70g of the fresh unscreened sample was oven-dried in a pre-weighed aluminium plate at 70 ± 5°C for 24 hours, or until constant weight. The moisture content was determined by the difference in net dry weight of the equilibrated sample and its initial as-received weight divided by its wet mass.

2.2 Determination of total dry solids

The total dry solids was calculated by subtracting the percentage moisture content from 100%.

2.3 Determination of particle size distribution

25 g of fresh substrate sample was placed on a pre-weighed sieve having the largest mesh size, 26.67 mm. The sieve was then fitted on top of the rest 5 pre-weighed sieves (mesh sizes: 26.67 mm, 18.85 mm, 13.33 mm, 6.680 mm, 4.699 mm, 3.327 mm, in that order) together with a base collecting pan, on the Ro-Tap testing sieve shaker. The apparatus was set to sieve for 10 minutes. After this lapse, the mass of the sieves and pan were taken to record the change in weight.

2.4 Determination of wet bulk density

A pre-weighed 1000 mL beaker was gradually filled with fresh sample material to the rim. The bulk density was calculated by the disparity in vessel weight after and before filling, divided by its volume.

2.5 Determination of water holding capacity

A fresh substrate sample was filled into a modified glass cylinder (M_o) having its base replaced with a plastic sieve mesh and secured with water-saturated filter paper. The whole piece was weighed (M_c) and disposed into a 2000-mL plastic beaker. Water was slowly added until the surface of the assembly was just covered by approximately 1cm. After a 24 hour stand, the cylinder-substrate set was removed out of the water and dried from the outside. It was then disposed on a saturated cellulose base covered with a watch glass. After 2 hours, the glass cylinder was re-weighed (M_{moist}) until a constant mass was recorded. The maximum water holding capacity (WK_{max}) was determined using the equation:

$$WK_{max} = \frac{E_{moist} - E_{dry}}{E_{dry}}$$

E_{moist} = mass of wet sample (g) = $M_{moist} – M_o$
E_{dry} = mass of dry sample (g) = $(M_c – M_o) (1 – 0.01W_c)$
M_c = mass of cylinder (g) + mass of wet filter paper (g) + mass of weighed-in sample (g)
M_{moist} = mass of cylinder (g) + mass of wet filter paper (g) + mass of wet sample (g),
M_o = mass of cylinder (g) + mass of wet filter paper (g),
W_c = water content of fresh sample (%)

2.6 Determination of the pH value

To 20 g of the consistently ground oven-dried sample, 200 mL of 0.01 mol/L of calcium chloride solution was added. The mixture was gently stirred for one hour. The pH was determined with a calibrated pH meter.

2.7 Determination of the electrical conductivity

To 20 g of the significantly ground oven-dried sample, 200 mL of deionized water was added. The mixture was continuously stirred on a magnetic stirrer for 2 hours. The electrical conductivity was determined over the collected filtrate using a calibrated electrical conductivity meter.

2.8 Determination of total volatile solids content

5 g of the oven-dried ground sample material was placed in a pre-weighed dry porcelain crucible and burnt up at 550°C in a muffle furnace for 2 hours. The volatile solids content was expressed as a percentage by mass of the dried sample.

2.9 Determination of ash content

The percentage ash was calculated by subtracting the percentage volatile solids from 100%.

2.10 Determination of total carbon content

Total Organic Carbon (TOC) was calculated from the following formula according to Jiménez and García (1992):

$$\text{TOC} = (\%\ \text{Ash})/1.8$$

2.11 Determination of total nitrogen by the Kjeldahl method

15 ml concentrated sulphuric acid was added to 0.5 g of the properly ground oven-dried sample into a boiling tube followed by a Kjeldahl tablet. The mixture was then heat treated at 360°C for 1½ hr in a fume chamber. The consequent bright clear sample was allowed to cool at room temperature, then transferred into a 100-mL volumetric flask and made up to the mark with distilled water. 2 ml of the mixture was pipetted and transferred into a distillation vessel followed by 5.0 ml soda lye. The volatile ammonia was collected in an acid receiver containing 10.0 ml boric acid. The acid was titrated against 0.01M hydrochloric acid using an indicator.

2.12 Determination of phosphorus content

50.0 ml of 0.1 M sulphuric acid was added to 5 g of consistently ground oven-dried sample, agitated in a closed container on a mechanical shaker for one hour and then filtered. To a 100-mL volumetric flask, 80.0 ml of distilled water was added followed by 8.0 ml chromogenic reagent, 8.0 ml ascorbic acid solution , 5.0 ml of the sample filtrate and made up to the mark. Likewise, standard phosphorus solutions (0 to 20 ppm) were prepared in 100-mL volumetric flasks. Full colour development was allowed for 30 minutes. The absorbance of each solution was then measured in a 10 mm optical cell using a calibrated spectrophotometer at 660 nm.

2.13 Determination of potassium content

50.0 ml of ammonium acetate was added to 5 g of homogeneously ground oven-dried sample, agitated in a closed container on a mechanical shaker for one hour. The resultant mixture was then filtered. To a 500-mL volumetric flask, 0.954g of dry potassium chloride was added followed by 1 ml concentrated hydrochloric acid and made up to the mark with distilled water. A series of five potassium standards were prepared into 100-mL volumetric flasks. The absorbance of the sample filtrate was then measured in a 10 mm optical cell using a calibrated flame photometer.

2.14 Determination of crude fibre content

2.0 g of consistently ground oven-dried sample was heated under reflux with 150.0 ml of 0.128M sulphuric acid for 30 minutes. The sample was rinsed three times with 50.0 ml aliquots of hot water and then filtered. The residue was then heated under reflux with 150.0 ml of 0.128M potassium hydroxide for 30 minutes. The sample was then washed with 50.0 ml aliquots hot water and then filtered once again. The consequent residue was washed with 10 ml acetone and dried at 70°C overnight in a forced-convection oven. The dried sample was allowed to equilibrate in a dessicator and afterwards ashed in a muffle furnace at 550°C for 2 hours. Crude fibre content was expressed as percentage loss in weight on ignition.

2.15 Determination of crude protein content

The percentage crude protein was calculated as percentage nitrogen multiplied by 6.25 (Diez and Alvarez, 2001).

2.16 Inoculum preparation

Pure mycelial culture of *Pleurotus sajor caju* was maintained on Potato Dextrose Agar (PDA). For stock culture production, millet grains were used (Futtee, pers.comm, AREU).

2.17 Spawn preparation

Spawn was prepared by inoculating polypropylene bags containing boiled maize seeds mixed with 4% (w/w) calcium carbonate with the stock culture.

2.18 Cultivation method

The bagasse and sugarcane trash were each weighed to 750g and blended with 10% calcium carbonate and 10% crushed maize on a dry weight basis with the moisture content adjusted to 70%. The substrates were then filled in heat resistant bags. The opening of each bag was fitted with a polyvinyl (PVC) ring (diameter and length = 50mm) and plugged with a 50 mm piece of cubed sponge. These were pasteurised at 70°C for four hours in a designed steel drum with a gas burner as heating device and were afterwards allowed to cool gradually to ambient temperature. Each bag was inoculated with the *CC 114* strain spawn at the rate of 5% dry weight of the substrate (Shah *et al.*, 2004). Immediately after, the sacks were transported to the growing room and randomly deposited horizontally, 20 cm apart, on galvanised metal shelves (disinfected by 5% domestic bleach). Incubation was carried out in complete darkness where bags were covered with black plastic sheets. The incubation temperature ranged between 23°C to 27°C. The growing room was equipped with a table fan, which was switched on when the temperature of the room exceeded 30°C, a fluorescent ceiling light (40 W, cool white) and a hygrometer. On complete colonisation of the bagged substrate, the black plastic sheet, PVC rings and sponge plugs were removed. Subsequently, only a PVC ring (20 mm length and 53 mm diameter) was replaced at the opening of each bag. The exposed surface was hand sprayed with tap water once or twice a day to maintain moisture in the bags at 75-80%.

The biological efficiency (B.E.) (yield of mushroom per kg substrate on dry weight basis) was determined by the following formula (Chang *et al.,* 1981):

$$B.E.\ (\%) = \frac{\text{Fresh weight of mushroom}}{\text{Dry weight of substrate}} \times 100$$

3 RESULTS & DISCUSSION

The main physicochemical characteristics of bagasse and sugarcane trash are presented in **Table 1**.

Table 1 Physicochemical characteristics of bagasse and sugarcane trash

Parameter	Bagasse	Sugarcane trash
Moisture Content (%)	62.1 ± 3.0	40.5 ± 0.3
Total Dry Solids (%)	37.9 ± 3.0	59.5 ± 0.3
Particle Size (mm)	3.327 - 6.680	3.327 - 6.680
Wet Bulk density (Kg/m^3)	130.3 ± 2.5	70.2 ± 1.1
Water Holding Capacity (%)	684.6 ± 14.9	515.6 ± 15.1
pH	6.20 ± 0.02	6.26 ± 0.04
EC (mS/cm)	0.64 ± 0.49	2.90 ± 0.08
Total volatile solids content (%)	94.599 ± 5.4	93.378 ± 6.6
Ash content (%)	5.401 ± 4.2	6.622 ± 0.09
Carbon content (%)	52.6 ± 2.4	51.9 ± 0.05
Nitrogen content (%)	0.66 ± 0.06	0.99 ± 0.06
Phosphorus content (%)	0.14 ± 0.01	0.07 ± 0.00
Potassium content (%)	0.28 ± 0.01	1.20 ± 0.02
Crude fibre content (%)	38.78 ± 0.9	35.69 ± 0.49
Protein content (%)	4.11 ± 0.35	6.16 ± 0.40

3.1 Moisture content

The moisture content for bagasse was 62.1% compared to sugarcane trash, 40.5%, which was composed of green and dry leaves. According to Stamets (2000), the moisture content of the substrate to be colonised by the mushroom mycelium should be between 60% and 75%. Therefore, the water level of the two substrates had to be adjusted to 70% before inoculation.

3.2 Total dry solids

The percentage dry solids give an estimation of the sample's total fibre, soluble solids and mineral content on a dry basis (Anwar, 2010). Sugarcane trash had a higher dry solids content, 59.5%, compared to bagasse, 37.9%. Yet, both values fit into the dry matter contents of substrates required for the cultivation of oyster mushrooms. For instance, the total dry solids for wheat straw and rice straw are 65% and 85% respectively (Mangaraj and Kulkarni, 2011; Maas *et al.*, 2008).

3.3 Particle size

Bagasse and sugarcane trash had similar particle sizes, 3.3-6.7%. Iwase *et al.* (2000) and Kok-Kheng (1989), recommend substrate particle size within a minimum range of 1 mm to 3 mm for mycelium development and subsequent colonisation of the *Pleurotus sajor caju* mushroom.

3.4 Wet bulk density

Bagasse had a higher wet bulk density, 130.3 kg/m^3 (Deepchand, 2001) in contrast to sugarcane trash, 70.2 kg/m^3. Lam *et al.* (2007) found that the wet bulk density of wheat straw was 290.7% at 60% moisture content.

3.5 Water holding capacity

Water holding capacity (WHC) provides information on the amount of water that can be absorbed by the bedding material (Changirath *et al.*, 2011). The water holding capacity of bagasse was 684.6% compared to sugarcane trash, 515.6%. According to Ficior *et al.* (2010) substrate mixtures used for the cultivation of *Pleurotus* had water retention capacity ranging from 200-580%.

3.6 pH

Bagasse and sugarcane trash had near to similar pH, 6.2 and 6.3. Sherif and Sherif (2007) found the pH value of bagasse to be 6.6. *P.sajor caju* normally thrives on substrates with pH ranges of 6.0 to 8.0 (Fanadzo *et al.*, 2010).

3.7 Electrical conductivity

Sugarcane trash had an electrical conductivity well above that of bagasse, 2.90 mS/cm and 0.64 mS/cm. Sherif and Sherif (2007) found the EC value of bagasse to be 1.40 mS/cm. According to Choudhary *et al.* (2009) increase in electrical conductivity (normally greater than 6 mS/cm) is almost proportional to the decrease in the number of mushroom pinheads.

3.8 Total volatile solids content

The volatile solids fraction of a substrate comprises the percentage organic matter that can be used directly by the mycelia (Magingo *et al.*, 2004). It refers to the biodegradable content of the substrate consisting mainly of cellulose with the organic matter content lying within a range of 68% to 95%, (Rahman and Imtiaj, 2008). Bagasse and sugarcane trash were within the same value limits of volatile solids content, 94.6% and 93.4%, respectively.

3.9 Ash content

The ash content in bagasse was lower, 5.4% than in the sugarcane trash, 6.6%. Pant *et al.* (2006) recommends substrates having low ash contents for growing oyster mushrooms. Likewise, Khan *et al.* (2008), found the protein content of banana leaves significantly higher compared to six other agricultural wastes. Norouzi *et al.* (2008) also recorded best fruit body production with rice straw + oilseed rape straw (25:75) and oilseed rape straw which had lower ash contents.

3.10 Carbon content

The carbon content in bagasse was slightly above that of sugarcane trash, 52.5% and 51.8%, correspondingly. Donini *et al.* (2009) state the highest yield of *Pleurotus* species in substrates with carbon contents ranging from 55 to 60%.

3.11 N:P:K content

The N:P:K contents for bagasse was 0.66%, 0.14% and 0.28% respectively, compared to sugarcane trash which was 0.99%, 0.07% and 1.2% correspondingly. Torkashvand *et al.* (2008) found the N:P:K content of bagasse to be 0.48%, 0.05% and 0.16% respectively. Mahimaraja *et al.* (2008) found similar values for sugarcane trash nitrogen, phosphorus and potassium contents, 0.53%, 0.1% and 1.10% in that order. Patil *et al.* (2010) found that soybean straw with N:P:K contents, 3.94%, 1% and 2.3% had the highest biological efficiency with the *Pleurotus ostreatus* species. Moreover, Kinfemi *et al.*, (2009) have found similar N:P:K contents for cowpea shells, 1.95%, 0.91% and 0.5% respectively, used for growing *Pleurotus sajor caju* mushroom.

3.12 Crude fibre content

Bagasse had a superior crude fibre content 38.8% compared to sugarcane trash, 35.7%. Most substrates used for oyster mushroom cultivation have a crude fibre content ranging from 28% to 65% as demonstrated by Mshandete and Cuff (2007) and Silanikove *et al.* (1988).

3.13 Crude protein content

Crude fibre measures the cellulose, hemicellulose and lignin content of food (Zakpaa *et al.*, 2010). Sugarcane trash had a higher crude protein content, 6.2%, compared to bagasse, 4.1%. Obodai *et al.* (2003) and Fanadzo *et al.* (2010) recorded the highest biological efficiency in composted sawdust which had an approximate crude protein content of 1%.

3.14 Biological efficiency

Bagasse had a higher biological efficiency, 83.8%, compared to sugarcane trash, 74.5%.

4 CONCLUSION

The difference in the physicochemical values of bagasse and sugarcane trash lied principally in the wet bulk density, water holding capacity and electrical conductivity. The present study has shown that there is a possibility of using sugarcane trash as a promising substrate for the cultivation of the *Pleurotus sajor caju* mushroom. Yet, to increase the biological efficiency of the new substrate, the mushroom strain has to be acclimatised with the sugarcane trash by varying its ratio to bagasse. Furthermore, the new substrate could be supplemented with additives to increase mushroom yield.

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(73) EFFECT OF CO-COMPOSTING OF STRAW AND BIOCHAR ON NUTRIENT AVAILABILITY FROM ANIMAL SLURRY SOLIDS

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EXECUTIVE SUMMARY

Development of intensive live stock farming in Europe has been restricted to a small number of regions. Thus, large amounts of agricultural wastes accrue in these areas making them “hot spots” of nutrient surplus, scattered around Europe. Due to the risk of severe environmental consequences, governmental regulations increasingly limit the allowed application rates of manure on agricultural land.

To sustain animal production in these areas under these regulations, innovative technologies have been developed to facilitate the separation of manures into a liquid and solid fraction. The liquid fraction with its easily available nitrogen is a valuable fertilizer, which can be used locally; whereas the considerable volume reduction of the fibrous fraction enables long-distance transportation. This means that the vast majority of the essential, but increasingly scarce nutrient phosphorous (P), as well as organic nitrogen (N_{org}) and carbon (C) in the slurry can be transported to places where there is a deficiency of these elements.

Composting is one of the means to increase the stability and reduce the mass and volume of the soil amendment products, improving handling and storage characteristics. However, greenhouse gas emissions occur during the composting process and microbiological immobilization might limit the availability of the nutrients to the plants after addition to agricultural soil. Both, the amounts of greenhouse gasses produced and immobilization of nutrients are dependent on and can thus be controlled by the type of bulking agent used to secure sufficient aeration of the material during the composting. The objective of the current study was to investigate the effect of composting bulking agents on the plant nutrient availability, with special interest in P, after soil application of manure solid fraction from biogas digestate.

In this study, an anaerobically digested, dewatered (decanter centrifuge) solid fraction of animal slurry was composted for 28 days in lab scale composting reactors. Both cereal straw and commercially available wood chip biochar were utilized as bulking agents. A combination of liquid extraction methods, application of the “diffusive gradient in thin films” (DGT) technique, a pot experiment in the greenhouse and an incubation study was used to indicate the plant nutritional availability of P in the animal slurry solids and the derived composts, as affected by bulking agent. In addition to the compost products, anaerobically digested animal slurry solids were also added as soil amendments, with or without mixing with biochar or straw.

Phosphorous availability is first assessed through different extractions and DGT-application. Nutrient net release in a depleted loamy soil is then compared with the actual uptake of the nutrients by a barley plant (*Hordeum vulgare*), with explicit focus on phosphorous availability and uptake.

The results will give a first indication of the effect of bulking agent on the quality of compost from animal slurry solids as an organic fertilizer for plant nutrition. Moreover it can be estimated, how the composting process influences the nutrient availability of the solid fractions after soil addition.

Experimentation is on-going, and results will be reported in the presentation at the conference.

1 INTRODUCTION

Development of intensive live stock farming in Europe has been restricted to a small number of regions. Thus, large amounts of agricultural wastes accrue in these areas making them “hot spots” of nutrient surplus, scattered around Europe. Due to the risk of severe environmental consequences, governmental regulations increasingly limit the allowed application rates of manure on agricultural land.

To sustain animal production in these areas under these regulations, innovative technologies have been developed to facilitate the separation of manures into a liquid and solid fraction. The liquid fraction with its easily available nitrogen is a valuable fertilizer, which can be used locally; whereas the considerable volume reduction of the fibrous fraction enables long-distance transportation. This means that the vast majority of the essential, but increasingly scarce nutrient phosphorous (P), as well as organic nitrogen (N_{org}) and carbon (C) in the slurry can be transported to places where there is a deficiency of these elements.

1.1 Background

Composting is one of the means to increase the stability and reduce the mass and volume of the soil amendment products, improving handling and storage characteristics. However, greenhouse gas emissions occur during the composting process and microbiological immobilization might limit the availability of the nutrients to the plants after addition to agricultural soil. Both, the amounts of greenhouse gasses produced and immobilization of nutrients are dependent on and can thus be controlled by the type of bulking agent used to secure sufficient aeration of the material during the composting.

However, processing, i.e. composting of the manure under different conditions might also alter P speciation or binding and hence plant availability. To assess it, conducting growth experiments is time-consuming, while chemical extraction methods are less laborious, but correlate only poorly with actual plant availability.

A faster alternative method to predict the nutrient release capability of a soil amendment is the application of the “diffusive gradient in thin films” (DGT) technique. The DGT mimics the processes in the root zone, where it acts as an ‘infinite’ sink for the nutrient and transport to the sink is limited by diffusion through a well-defined gel. This method has recently been successfully used to assess the P status of different agricultural soils.

1.2 Research objectives

The objective of the current study was

- to investigate the effect of composting bulking agents on the plant nutrient availability, with special interest in P, after soil application of manure solid fraction from biogas digestate and
- to investigate the applicability of a range of chemical extractions in comparison to the DGT technique to predict the plant availability of P from different amendments, derived from manure solids.

2 METHODOLOGY

A combination of liquid extraction methods, application of the “diffusive gradient in thin films” (DGT) technique, a pot experiment in the greenhouse and an incubation study was used to indicate the plant nutritional availability of P in the animal slurry solids and the derived composts, as affected by bulking agent.

An overview of the range of the five investigated products, derived from the digested solid fraction of pig slurry can be seen in Table 1. Details on the applied methods are given in the following sections.

TABLE 1 **Overview on investigated manure derived products**

Solid pig manure fraction	**Type of bulking/mixing agent**	**Abbreviation**
Digestate	none	SOL
Digestate	Straw	SOL+STRAW
Digestate	Biochar	SOL+CHAR
Composted Digestate	Straw	COM+STRAW
Composted Digestate	Biochar	COM+CHAR

2.1 Composting

In this study, an anaerobically digested, dewatered (decanter centrifuge) solid fraction of animal slurry was composted for 28 days in lab scale composting reactors (10 L volume). Both cereal straw and commercially available wood chip biochar was utilized as bulking agents. Gaseous emissions were continuously measured using a photo-acoustic field gas-monitor (INNOVA 1412) in conjunction with a multipoint sampler (INNOVA 1309). Further details can be found in the study of Chowdhury et al. in the ORBIT 2012 proceedings. In addition to the immature compost products, anaerobically digested animal slurry solids were also investigated, with or without mixing with biochar or straw, as a reference material. All materials were stored frozen before analysis.

2.2 Chemical extractions

The five different digestate products were extracted with three extraction liquids. Deionised water, 2% citric acid and 1 molar hydrochloric acid were added to the products at a solid-liquid-ratio of 1:100 and shaken overhead for 16 hours at room temperature. Subsequently the liquid was filtered at 0.45µm and stored at 4 degrees Celsius before phosphate analysis.

2.3 DGT-Application

The products were amended to an inert sand matrix at a product-sand-ratio of 1:100. Deionised water was added to the mixture up to saturation. After 24 hours of equilibration, a DGT-device (DGT Research Ltd, Lancaster, UK) was applied to the surface for 24 hours at room temperature. The resin-gel was extracted with nitric acid and stored at 4 degrees Celsius before phosphate analysis.

2.4 Incubation and growth experiment

All products were applied to a sandy loam from a nutrient depleted field from the experimental farm of the University of Copenhagen in Taastrup, Denmark. The product-soil-ratio was 1:100 with a soil mass of 100 g DM per incubation container. The incubation was conducted for 45 days at 15 degrees Celsius. At day 0, 15, 30 & 45 an aliquot of the soil was extracted on a shaker with deionised water for 1 hour, filtered (0.45µm) and stored chilled before phosphate analysis.

In a similar way 2 kg DM of nutrient depleted soil were mixed with 20 g DM of digestate-product for the growth trial. Barley (*Hordeum vulgare*) was grown in the pots for 45 days, harvested, dried, digested and analyzed for P content in the above soil part of the plant biomass.

3 RESULTS AND DISCUSSION

Since experimentation is still on-going, results will be reported and discussed in the presentation at the conference.

4 CONCLUSIONS

The results will give a first indication of the effect of bulking agent on the quality of compost from animal slurry solids as an organic fertilizer for plant nutrition. Moreover it can be estimated, how the composting process influences the nutrient availability of the solid fractions after soil addition.

Depending on the results, the DGT method could provide a faster way to estimate the nutrient release capability of agricultural waste as an organic fertilizer.

5 ACKNOWLEDGEMENTS

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(105) SUITABILITY OF COMPOSTS FOR AN ACID-LOVING PLANT: HIGHBUSH BLUEBERRY

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EXECUTIVE SUMMARY

Highbush blueberry (*Vaccinium corymbosum* L.) is adapted to soils with high organic matter and pH of 4.5-5.5. Coniferous sawdust is the blueberry industry standard soil amendment and mulch in the Pacific Northwest, USA. However, sawdust prices are increasing, and its availability is becoming limited. Therefore, many blueberry producers are interested in compost as an alternative to sawdust, which might also reduce the need for nitrogen (N) fertilizers. Our objectives were to: (i) estimate elemental S (S^o) rate needed to acidify compost to target pH for blueberry, and (ii) evaluate diverse composts vs. sawdust as soil amendments in plant growth trials under low and adequate N fertilization regimes.

The compost feedstocks were animal manures (horse or dairy manures; solids + bedding) or plant materials [yard debris (grass + woody prunings from urban landscape maintenance), deciduous tree leaves (from street sweeping), mint (leaves and stems recovered after steam distillation of peppermint oil) or bark (aged conifer bark composted with fine sawdust and municipal wastewater treatment biosolids)]. Finished compost pH was usually 7.5-8.5, except for the bark compost, which had a pH of 5.2.

Composts were titrated with dilute sulphuric acid to determine their capacity to buffer pH. Compost acidification to pH 5 required an average of 10 g S^o/kg across all feedstock categories. Dairy and mint composts had the highest S requirement, and the most variable compost acidification requirements (within a feedstock) were found among dairy manure (8-28 g S/kg; n = 5) and horse manure composts (4-12 g S/kg; n = 4). Yard debris compost had moderate S requirement (9 g S/kg). Bark compost did not require acidification.

To evaluate compost suitability, two plant growth trials were conducted in pots in acid mineral silt loam soil (pH 5) amended with a high rate of compost or sawdust (30% by volume). Trials were performed in winter in a greenhouse (4-L pots; Jan-May) and outdoors in summer (14-L pots; May-Sept). Soil pH (after compost amendment) was predicted by the pH buffering capacities of composts. Plant growth was strongly affected by soil pH. Shoot and root growth decreased as soil pH increased from 5 (acidic) to 7 (neutral). Plants usually grew better in plant-derived composts (bark, yard debris, or deciduous tree leaf, but not mint) than in manure-derived composts (dairy or horse). Acidification of composts with finely ground S^o increased plant growth. At low levels of N fertilizer addition (Greenhouse Trial), plants grew better with compost than with sawdust. At a higher N fertilizer rate (Outdoor Trial), plant growth with the best performing composts was equivalent to sawdust. Composts with C:N near 20 produced good growth in both trials. Plant growth response to compost was not related to compost NO_3-N analyses and was good in compost treatments with relatively low NO_3-N. Most composts contained soluble salt (EC) levels considered high for blueberry. Yard, leaf, and bark composts had the lowest EC (< 1), while EC in horse, dairy, and mint composts averaged 1.8. 2.5, and 6.1, respectively (1:10 compost: water method). However, compost EC did not appear to be of primary importance in determining plant response to compost. In fact, plants grew better in composts that had been acidified with S, even though compost EC was elevated by S^o oxidation to sulphate. Salts were rapidly leached from pots in our trials, limiting the duration of plant exposure to high EC.

We conclude that composts with pH < 6 and EC < 2 are ideal soil amendments for blueberry. Composts with higher EC may be acceptable when leaching of salts is assured, or when compost is applied as mulch. Composted woody plant materials (C:N near 20), acidified with finely ground S, are the most promising composts for blueberry. Finely ground S dust reacted quickly in compost, achieving full reaction in 2-4 weeks. The S^o application rate can be customized to match compost buffering capacity using our quick test method (3-d incubation with dilute sulphuric acid).

1. INTRODUCTION

Highbush blueberry (*Vaccinium corymbosum* L.) is a crop of increasing importance in the Pacific Northwest, USA. Between 2008 and 2010, blueberry plantings in the state of Oregon increased from 5700 to 7500 ha, and market value increased from $37 to $63 million USD. Organic and conventional blueberry growers in Oregon are interested in composts as mulches or soil amendments for highbush blueberry production. Blueberry may benefit from nutrients, organic matter, and microbial activity supplied by compost. However, the high pH and high soluble salts present in some composts may adversely affect plant growth.

Blueberry is categorized as a calcifuge plant, well-adapted to acidic soil, with tolerance to high concentrations of plant-available metals (e.g. Mn and Al) in soil (Korcak, 1988). Blueberry responds positively to organic matter addition when grown in mineral soil, but the mechanism responsible for organic matter benefit to blueberry is often unclear. Blueberry growth has been shown to improve when soil is amended with sawdust, pine bark or peat moss. These grower-standard amendments are characterized by acidic pH and low nutrient supply to the plant.

One major benefit of replacing sawdust with compost could be a reduction in N fertilizer requirement. In the Pacific Northwest, blueberry is often mulched with acidic Douglas-fir (*Pseudotsuga menziesii*) sawdust. Some growers also incorporate sawdust into raised beds at planting. Because the biological decomposition of sawdust immobilizes inorganic N, growers must apply additional N in order to meet plant needs (Hart et al., 2006). For growers that produce blueberry under organic certification, fertilizer is expensive, ranging from $7 to $22 USD per kg N.

1.1 Research objectives

- Estimate elemental S (S^o) rate needed to acidify compost to target pH for blueberry (5.0)
- Evaluate diverse composts for their suitability as soil amendments for blueberry

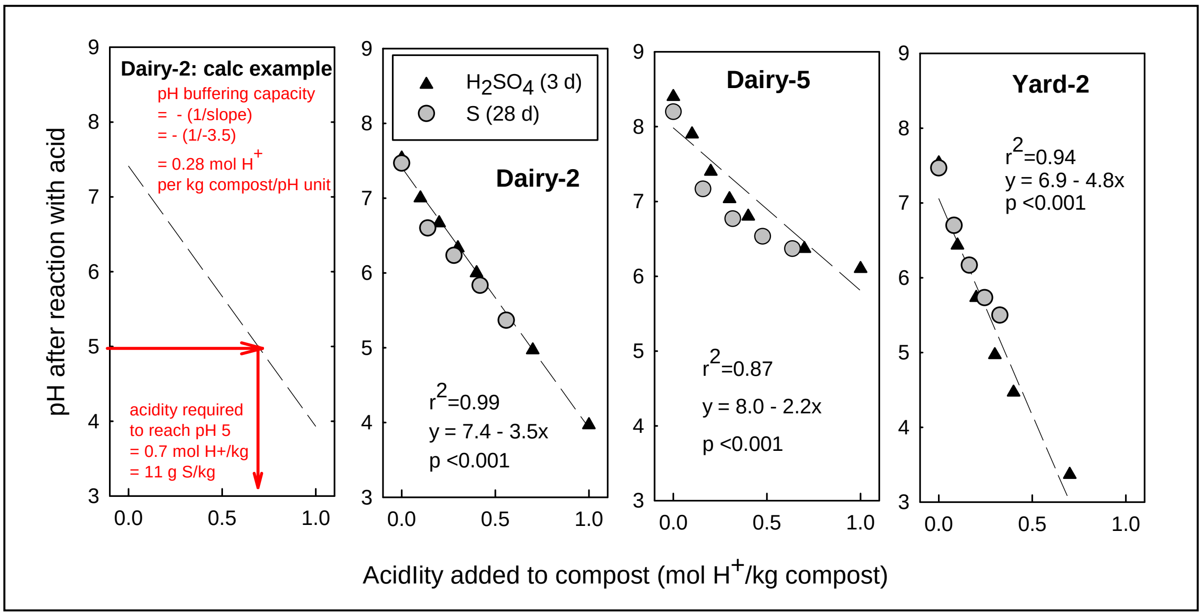

FIGURE 1. **Compost pH buffering capacity was measured by titrating compost with dilute sulfuric acid.** Compost pH was measured 3-d after acid addition. **Left:** Illustration of calculation for Compost pH Buffering Capacity (CBC) and Compost Acidification Requirement to pH 5 (CAR). Linear regression was used to determine the slope of pH change per unit of acid addition, and the result was expressed in units of mol H+/kg compost dry matter per pH unit. **Middle and right:** Titration data used to estimate compost pH buffering capacities for Dairy-2, Dairy-5 and Yard-2 composts. Values obtained in the short term titration (3-d) were also confirmed by adding fine S^o dust to compost. The compost pH resulting from S^o addition was measured after 28 d at 22 ^oC.

2. METHODOLOGY

Compost feedstocks were animal manures (horse or dairy; solids + bedding) or plant materials [yard debris (grass + woody prunings from urban landscape maintenance), deciduous tree leaves (from street sweeping), mint (leaves and stems recovered after steam distillation of peppermint oil) or bark (aged conifer bark composted with fine sawdust and municipal wastewater treatment biosolids)].

Compost EC and pH were determined in a 1:10 ratio of compost (dry wt) to water (Table 1). Total C and N were determined by combustion analysis with direct measurement of C and N in exhaust gas. Nitrate-N was extracted with 2M KCl, filtered, and determined via an automated colorimetric method. Costello (2011) provides a full description of composts and analytical methods.

Laboratory titration to determine the pH buffering capacity of composts. Composts were titrated with dilute sulphuric acid to determine their capacity to buffer pH, and to estimate H^+ addition required to reduce compost pH (Figure 1). This procedure was developed in our laboratory: Step 1: Add moist compost (equivalent of 5 g of dry compost) to 100-mL cups. Step 2: Add 0.25M H_2SO_4 at rates of 0, 1, 2, 3, 4, 6, 8 or 10 mL (1 mL = 0.1 mol H^+ per kg dry compost;). Use a separate cup for each acid addition rate. Add deionized water to make volume to 50 mL (e. g., if adding 2 mL of 0.25M H_2SO_4, then add 48 mL of water). Step 3: Cover cups with lids or plastic wrap to prevent moisture loss. Step 4: Measure compost pH after equilibration for 3 d at room temperature (approx. 22^{o}C). Step 5: Plot 3-d compost pH (y-axis) vs. acid addition rate (Figure 1). The negative reciprocal of the slope of the line is equal to pH buffering capacity of compost (CBC). For example, if compost was titrated with 0 and 0.4 mol H^+ per kg dry compost and pH decreased by one unit, then the pH buffering capacity would be 0.4 mol H^+ per kg dry compost per pH unit. Step 6: Estimate S^o addition required to reach pH 5 using this equation: (compost pH – 5) x (CBC; mol H+/kg per pH unit) x (16 g S/mol H^+) x (1/0.75; assumed efficiency S conversion to H^+). For example, for Dairy 1 compost, we calculated that addition of 8 g S/kg compost dry matter was required to reduce compost pH to 5.0, calculated as: (7.8 initial pH-5.0 final pH; 2.8 unit pH change) x (CBC; 0.13 mol H^+/kg) x (16g S/mol H^+) x (1/0.75).

TABLE 1 **Compost carbon, nitrogen, pH, electrical conductivity (EC) and pH buffering characteristics**. Averaged across feedstock type for composts used in Greenhouse and Outdoor Trials. Compost buffering capacity determined by dilute sulfuric acid titration (Figure 1). S^o acidification requirement (to pH 5) assumes 75% conversion of S to acidity (H^+).

Primary Compost Feedstock	**n**	**C:N**	**Total N**	**NO3-N**	**EC**	**pH**	**Compost pH buffering capacity (CBC)**	**Compost Acid Requirement to pH 5 (CAR)**	**S^o to acidify to pH 5**	
							Average	**Average**	**Average**	**Maximum**
			g/kg	g/kg	mS/cm	1:10	mol H+/kg per pH unit	mol H+/kg	g S/kg	g S/kg
Dairy	5	15	17	1.4	2.5	8.3	0.23	0.8	16	28
Horse	4	20	11	0.6	1.8	7.6	0.15	0.4	8	12
Yard	3	19	13	0.3	0.9	7.6	0.17	0.4	9	10
Leaf	2	19	10	0.6	0.3	8.0	0.17	0.6	12	19
Bark	2	24	12	1.0	0.9	5.0	0.22	0.1	1	2
Mint	2	8	45	2.3	6.1	8.4	0.22	0.8	16	19
				Grower Standard Amendment (Control)						
Sawdust	2	620	0.7	0.001	0.03	4.0				

Plant growth trials to evaluate compost suitability for blueberry.

Two plant growth trials were conducted in pots to evaluate compost suitability. Organic amendment (compost or sawdust) was added to pots at a high rate (30 compost:70 soil; v/v). Trials were performed in winter in a greenhouse (4-L pots; Jan-May) and outdoors in summer (14-L pots; May-Sept). Soil was used in our growth trials (instead of nursery media) because our goal was to evaluate compost suitability for field application. Willamette silt loam soil was collected in bulk and homogenized in a cement mixer. Soil pH was suitable for blueberry (pH = 5.3: 1:2 soil:water method) and it had typical organic matter content (19 g/kg C; approx. 40 g organic matter/kg).

Acidified and non-acidified composts were evaluated in the Greenhouse Trial. Prior to potting, each compost was acidified by reaction with elemental S dust (32 g S/kg compost-C) for 70 d at room temperature (22^{o}C). Because compost-C varied, the S rate varied among composts from 5 to 8 g S/kg on a compost dry matter basis. One compost (mint) received a much higher S rate (11 g S/kg) because its C analysis was high (370 g C/kg).

During each plant growth trial, N was supplied by fertigation every 14 d (Greenhouse Trial) or every 7 d (Outdoor Trial). Fertilizer N was supplied via mineral NH_4-N salts in the Greenhouse Trial, and by fish emulsion in the Outdoor Trial. In both trials, dilute fertilizer solution containing approximately 100 mg NH_4-N/L was applied every 7 to 14 d in sufficient volume to fill pore space. More N was supplied to plants via fertigation during the Outdoor Trial (5 g/plant) than in the Greenhouse Trial (<0.1 g N/plant), due to more frequent fertigation, greater pot volume (larger plants), and longer trial duration (78 d in the Greenhouse Trial, 119 d in the Outdoor Trial).

Whole plants were harvested at the end of each plant growth trial. Shoots were separated into old stem (present in transplant) and new growth (leaves + stem produced after transplanting). In the Greenhouse Trial, root growth was measured from the original root ball using a cm scale (1 = no root growth; 7 = maximum root growth extended across entire pot diameter). In the Outdoor Trial, soil was removed with a high pressure water spray, and then root dry weight was determined.

Greenhouse Trial.

FIGURE 2. **Plant growth trials with blueberry. Compost incorporated at 30% by volume into a mix with mineral soil.**

3. RESULTS AND DISCUSSION

3.1 Estimating elemental S rate needed to acidify compost

Compost buffering capacity, as determined by titration of individual composts (data not shown) ranged from 0.08 to 0.36 mol H+/kg compost/pH unit. The change in pH with acid addition (pH buffering capacity; Figure 1) was considered a linear function for the purposes of determining S^o acidification requirement. However, a few of dairy composts were very strongly buffered between pH 6 and 7 (e.g. Dairy-5 in Figure 1). Compost acidification to pH 5 required an average of 10 g S^o/kg across all feedstock categories. Dairy and mint composts had the highest S requirement. Yard debris compost had moderate S requirement (9 g S/kg). Bark compost did not require acidification. The most variable compost acidification requirements (within a feedstock) were found among dairy manure composts (8 to 28 g S/kg; n = 5) and horse manure composts (4 to 12 g S/kg; n = 4).

The predictive value of measuring Compost Acidification Requirement (CAR) was illustrated in the Outdoor Trial (Figure 3). Soil pH of the compost + soil mixture (Day 76) was not correlated with compost pH, but it was strongly correlated with CAR.

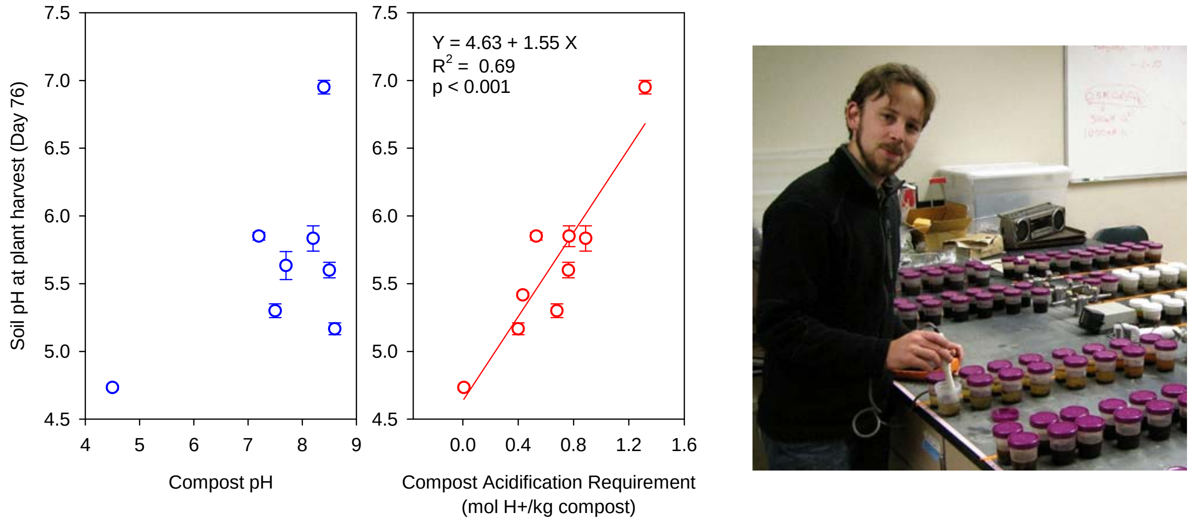

FIGURE 3. Relationships between preplant compost analyses and soil pH measured on Day 76 of the Outdoor Trial. Error bar = SE of mean. **Left:** Soil pH (Day 76) was not correlated with preplant compost pH. **Middle:** Soil pH (Day 76) was strongly correlated with preplant compost acidification requirement [CAR; see Figure 1 for derivation]. **Right:** Author (Costello) measuring compost pH at three days after dilute sulfuric acid addition. Five grams compost was incubated in 50 mL dilute acid in each cup. Each cup (100 mL; covered with lid to prevent moisture loss) represents a single acid rate (mol H+/kg compost) on a compost pH buffer line (Figure 1).

3.2 Compost effects on plant growth

- **pH.** Plant growth was strongly affected by soil pH. Shoot and root growth decreased as soil pH increased from 5 to 7 (Figure 5).
- **Compost acidification (Greenhouse Trial).** Acidification of composts with finely ground S^o increased plant growth (Figure 4). Plants grew better in compost that had been acidified with S, even though compost EC was elevated by S^o oxidation to sulphate. EC (averaged across 10 composts) increased from 1.9 to 3.5 dS/m when compost pH decreased from 7.2 to 5.3.
- **Plant growth response vs. feedstock category (Figures 4 and 7)**. Plants grew better in plant-derived composts (bark, yard debris or leaf) than in manure-derived composts (dairy or horse). Mint compost analysis (Table 1) was similar to that of manure composts (high N, pH, EC, and S^o required for acidification). Plant growth with mint compost was inferior to that of other composted plant materials.
- **Comparison to sawdust.** At low levels of nitrogen (N) fertilizer addition (Greenhouse Trial), plants grew better with compost than with sawdust (Figure 4). At higher N fertilizer rate (Outdoor Trial), plant growth with the best performing composts (derived from plant materials) was equivalent to sawdust (Figure 7).
- **Compost analyses vs. plant growth**
  - Most composts contained salt levels considered high for blueberry (Table 1). The yard, leaf, and bark composts had the lowest EC values (<1). Compost ECs in horse, dairy, and mint composts averaged 1.8. 2.5, and 6.1, respectively, using the 1:10 compost:water method. However, compost EC did not appear to be of primary importance in determining plant response to compost. In fact, plants grew better in composts that had been acidified with S, even though compost EC was elevated by So oxidation to sulphate. Salts were rapidly leached from pots in our trials, limiting the duration of plant exposure to high EC.
  - Composts with C:N near 20 produced good plant growth in both trials.
  - Plant growth response to compost was not related to compost NO3-N analyses. Compost treatments with relatively low NO3-N produced good plant growth. Composts with high NO3-N also had high EC and high K (negative characteristics for use with blueberry).
  - Inferior plant growth with dairy and mint composts (Figure 4) in the Greenhouse Trial was associated with high K in compost (data not shown). Total K (20 to 60 g/kg) and saturation extract K (20 to 100 mg/L) in these composts were very high compared with composts that gave good plant growth. Bark and leaf composts

produced good growth and had much lower K analyses (total K< 5 g/kg; saturation extract K < 10 mg/L). Plants growing in dairy and mint composts in the Greenhouse Trial had extremely high leaf K (10 to 20 g/kg; normal is 4 to 7 g/kg), and low leaf Mg (< 1 g/kg; normal is 1.3 to 2.5 g/kg).

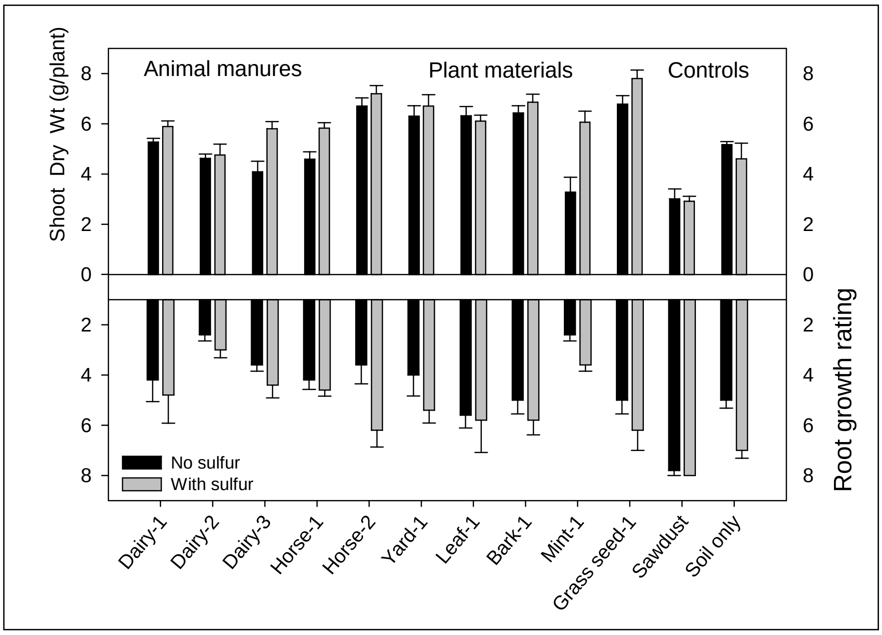

FIGURE 4. Compost effects on blueberry shoot and root growth for acidified compost (with S) and non-acidified compost (no S). Greenhouse Trial. Root growth rating (1 to 7 scale). 1 = no root growth; 7 = roots extended across pot diameter. Error bar = SE of mean. Plants grown in sawdust-amended soil suffered from N deficiency. Fertilizer N rate used in this trial was low (<0.1 g/plant). "With S" = elemental S (32 g S/kg compost-C) was allowed to react with compost for 70 d prior to potting. Average compost pH reduction with acidification was 1.9 units. Contrasts between feedstock categories (P= 0.05): Shoot growth was greater with compost (n = 10) than with sawdust. Plants produced greater shoot and root growth with composts derived from plant materials (n = 5) than with composts derived from animal manures (n = 5).

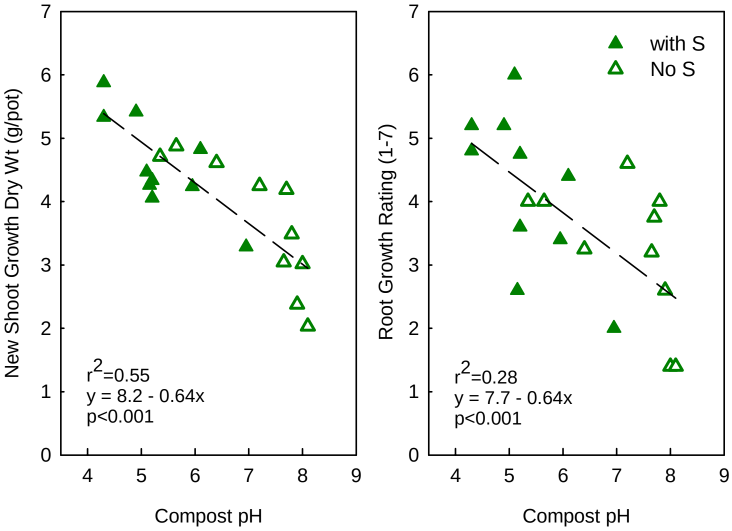

FIGURE 5. **Plant growth declined as compost pH increased. Each data point represents one compost.** "With S" = elemental S (32 g S/kg compost-C) allowed to react with compost for 70 d prior to potting. Average compost pH reduction with acidification was 1.9 units. Greenhouse Trial. **Left:** shoot growth. **Right**: Root growth rating (1 to 7 scale). 1 = no root growth; 7 = roots extended across pot diameter.

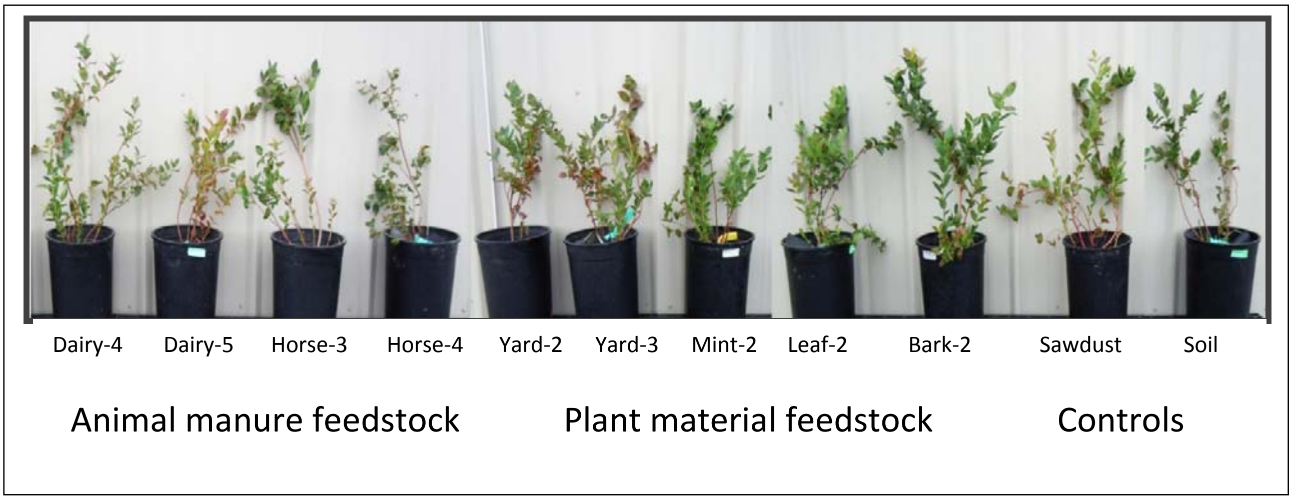

FIGURE 6. **Plants at Harvest (Day 119 after transplanting). Outdoor Trial.** Leaf and bark composts had best growth (equivalent to sawdust). Plants grown with Horse-4 and Yard-2 composts showed water stress in mid-summer, associated with low water holding capacity of soil + compost mixture.

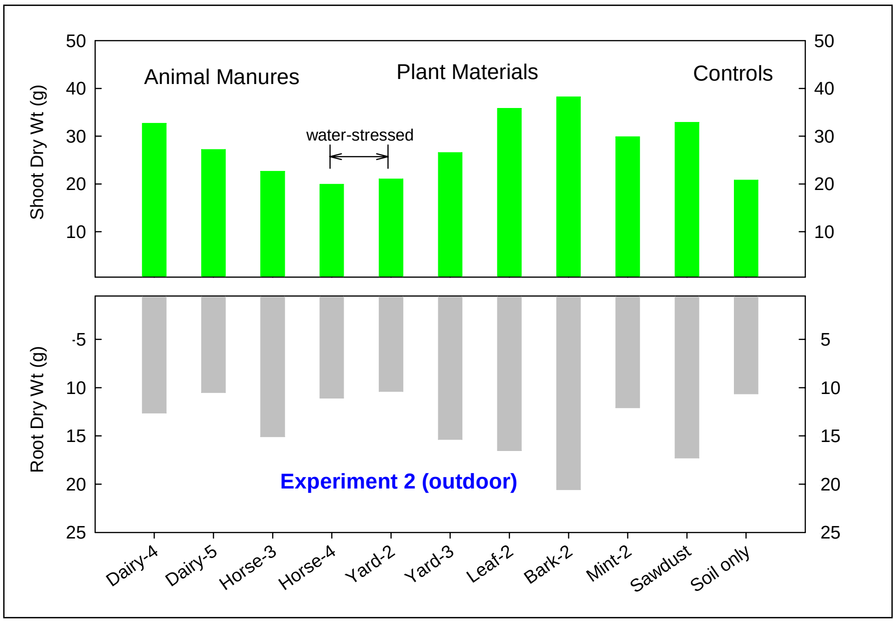

FIGURE 7. **Compost effects on blueberry shoot and root growth. Outdoor Trial.** Pooled SE of mean = 3 g for shoot dry wt, and 4 g for root dry wt. Plants were fertigated weekly with fish emulsion. Plants grown in sawdust had adequate N in this trial. Plants grown with Horse-4 and Yard-2 composts showed water stress in mid-summer, associated with low water holding capacity of soil + compost mixture. Contrasts between feedstock categories (P= 0.05): Plants grew better in composts derived from plant materials than in composts from animal manures. Plant growth was equivalent for composts derived from plant materials vs. sawdust.

4. CONCLUSIONS

We conclude that composts with pH < 6 and EC < 2 are ideal soil amendments for blueberry. Composts with higher EC may be acceptable when leaching of salts is assured, or when compost is applied as mulch. Composted woody plant materials (C:N near 20), acidified with finely ground S, are the most promising composts for blueberry. Finely ground S dust reacted quickly in compost, achieving full reaction in 2-4 weeks. The S^o application rate can be customized to match compost buffering capacity using our quick test method (3-d incubation with dilute sulphuric acid).

We recommend that growers choose composts based on a complete analysis, and apply compost at modest rates on a trial basis. Composts with high K analysis should be avoided, because K does not leach readily from soil, and excess K uptake may injure blueberry plants.

5. ACKNOWLEDGEMENTS

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(237) EFFECT OF COMPOSTED SEWAGE SLUDGE ON HEAVY METAL CONTENT OF THE SOIL AND PLANTS

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EXECUTIVE SUMMARY

A good way for utilizing municipal sewage sludge is its use on agriculture lands after composting. The fate of sewage sludge is unsolved, its large quantity is cumulated on the territories of sewage cleaning plants. We can solve serious environmental problems with sewage sludge treatment, however there are some risks, since sewage sludge may contain heavy metals in high rate.

In the Nyírség region of Hungary there are mostly acidic sandy soils with low humus content. Because of the decreasing quantity of livestock manure, the subsequent delivery of organic matter into the soils could be problematic. Application of sewage sludge (compost) in agriculture is very difficult, because the possible great toxic elements content of sludges and the farmers have to proceed according to stringent lows.

Sandy soils are poor in organic and mineral colloids. Long-term use of organic fertilizers could improve this problem, but in a short-term period only the macro- and micro-element content could be supplied. Due to these properties sandy soils have low fertility. To improve all these characteristics we can use composted sewage sludge.

Experiment was established in spring, 2003 at the Research Institute of University of Debrecen, in Nyíregyháza city, located at NE part of Hungary and was re-treated in 2006 and 2009. The compost was applied at 9, 18, 27 t ha^{-1} doses, ploughed into the soil before sowing. Beside the toxic element content of soil, we studied the effect of the applied compost on triticale, maize and pea in a small-plot experiment in five replicates. The pH of this light textured sandy soil is 4.58, its humus content is 0.31%. In the composting process some additives such as bentonite, rhyolite and wheat straw were used, because these materials have good effects on light textured sandy soils, especially with organic material. In this way, the compost contains 40% sewage sludge, 25% straw, 30% rhyolite and 5% bentonite. Our compost fills the requirements of the Agricultural Ministry Order of 36/2006 (V.18.).

The observed parameters were the followings:

- toxic element content (As, Cr, Cu),
- the quantity of the yield,
- the development of plants.

In the 2008 and 2009 years of the experiment, soil samples were collected twice a year, in April and after harvesting. Samples were sieved to <2 mm, dried at 105 °C and the Cr and Cu concentrations analyses were done after a HNO_3-H_2O_2 digestion by ICP-OES (VARIAN VISTA PRO) and the As concentrations analyses was done by GF-AAS equipments. Plant samples were dried at 105°C and were ground before digestion. The measured elements were arsenic (As), chromium (Cr), and copper (Cu).

One-way analysis of variance and Tukey's test were used to determine the effects of treatments on crop yield and on metal content of plants and soil.

Pea accumulated heavy metals in its root but the heavy metal content of seeds was not increased in the treatments and the values were in the concentration range of crops growing on unpolluted soils. In the case of the other crops treated, there was no or very little increase of the heavy metal content. The results of the metal content stayed under the levels set in the 50/2001 (IV.3.) Goverment Statute. Any significant effect of sewage sludge compost treatments on Cu, As and Cd content of the soil was not found.

1. INTRODUCTION

The use of composted sewage sludge may have negative effects on the increase of toxic elements in the environment, especially its long-term use could enrich these elements in the soils and plants (Vermes, 1998).

Sewage sludge is an organic waste which usually contains high levels of nitrogen and phosphorous as well as significant concentrations of micronutrients (Epstein et al., 1976; Sopper, 1993). The use of sewage sludge in soil has very beneficial effects on the quantity and availability of nutrients, on the restoration of structural stability of the soil and on resistance against soil erosion (Sort and Alcaniz, 1996, Debosz et al., 2002).

Composting offers an effective method for treating sewage sludge by stabilizing organic matter, reducing odour, and killing human pathogens and weed seeds by the heat generated (Riffaldi et al., 1986; Bevacqua and Mellano, 1993). The products can be used as soil conditioner to improve soil physical properties by increasing soil organic matter content. In this way the water holding capacity and aggregate stability are increased, but soil bulk density is reduced (Smith, 1992). Sewage sludge compost also represents a source of nitrogen (N), phosphorus (P), potassium (K) and other trace elements such as zinc Zn, copper (Cu) and molybdenum (Mn) for plant growth (Tester, 1990).

Compost is applied to cropland to maintain and improve soil structure and plant nutrition (Gigliotti et al., 1996). However, the presence of toxic elements in composts is the main cause of adverse effects on animal and human health, transmitted through the food chain from soil, groundwater and plants (Senesi et al., 1999). When a soil is amended with products such as sewage sludge, the toxic elements which these products may hold will be contained within an organic matrix. This may lead to the increased solubility of some of the metals (due to the formation of soluble organic complexes) or to their immobilization and subsequent reduced possibility of being assimilated by the plant (Garcia *et al.,* 1991). Consequently, exactly analyzing the contents of toxic elements in composts is very important for the routine monitoring and risk assessment and regulation of environment.

The aim of the experiment was to demonstrate that application of sewage sludge compost did not cause hazardous accumulation of toxic elements in the soil and plants. During the experiment we would like to follow the movement of toxic elements in the food chain.

2. METHODOLOGY

The experiment was established in spring, 2003 at the Research Institute of CAAES of University of Debrecen, at the town of Nyíregyháza, located in the NE part of Hungary. It was re-treated in 2006, when the field experiment was re-arranged and the size of plots was enlarged. The small-plot experiments where the plots are 19 x 36 m in size, were done in five replications. There are different types of sandy soil in this region, therefore we used bentonite, rhyolite and wheat straw as additives, because these materials have good effects on light textured sandy soils if they are mixed with organic material (e.g. sewage sludge). Our compost fills the requirements of the Agricultural Ministry Order of 36/2006 (V.18.). It contains 40% sewage sludge, 25% straw, 30% rhyolite and 5% bentonite. The compost was applied at 9, 18, 27 t ha^{-1} doses, ploughed into the 0-30 cm soil layer before sowing. We studied the effect of the applied compost on triticale (*x Triticosecale Wittmack*), maize (*Zea mays*) and pea. (*Pisum sativum*). Toxic elements of plant samples were measured after harvesting. In the case of triticale and maize the toxic elements content of grain was measured while the pea plants were divided into three parts (root, straw+pods and grain) and the toxic element content of these parts were measured. Composite soil samples were collected of 5 subsamples in each plot from 0-30 and 30-60 cm soil layers after harvesting of all test plants.

The observed parameters were the followings:

- ***Toxic elements content***, which is important for the nutrition chain. The problem with the toxic elements is that they can accumulate and may cause serious illnesses if they get into an organism. This is also true for the soil as well because it can accumulate toxic elements, too or with the help of soil water these elements may get into the deeper layers which cleaning would be very complicate.
- ***The quantity of the yield,*** which is important for the profit. First of all, the good quality plants can be sold for better profit. It could be harvested much more crop when we used compost, comparing to the control plots.
- ***The development of plants,*** because the primary target is the rapid and equal growth during the growing period. When the growth of a plant population stops and weather is changeable, the deficiency of the crops and the economical damage may be considerable. The strong and healthy developing plants can resist the possible attacks of causative agents (fungi, bacteria and viruses).

2.1 Chemical and statistical analysis

Toxic elements content (Cr and Cu) of the soil and plant samples were measured by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) and the As concentration analyses was done by GF-AAS instruments after digestion with cc. HNO_3 – cc. H_2O_2, according to the Hungarian Standard. The statistical analyses (one-way ANOVA followed by Tukey's test) were performed with SPSS 13.0.

2.2 Toxic elements in the test plants

In the case of pea test plant chromium (Cr) was detected only in the root in the rate of 0.3-6 mg kg^{-1} concentration in 2008-2009 years. These results are similar to the results of Chandraa et al. (2009) who also found the highest Cr concentration in the root of the test plants. The quantity of Cu was the highest in the root and grain of pea in 2008. In 2009 Cu concentration increasing was observed in the root. We measured higher Cu concentration in straw+pods and grain compared to 2008 (Fig.1). The Cu content of test plant was relative low comparing to the plants growing in unpolluted sites.

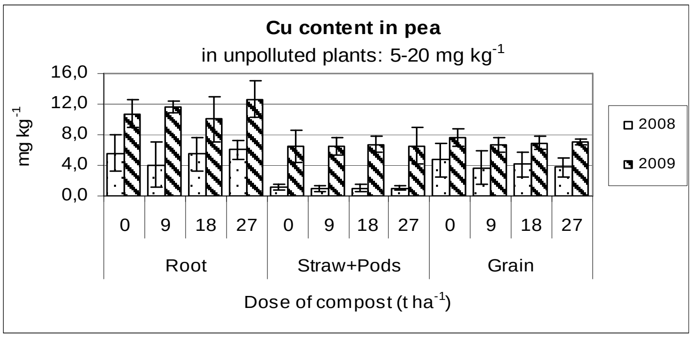

FIGURE 1 **Copper (Cu) content in different parts of pea in the sewage sludge compost treatment.**

The arsenic (As) content was very low in the grain of pea in 2008 and 2009 (Fig.2). In the other parts of the plant was also lower then in the plant growing on unpolluted soil. The measured value was 0.3-0.9 mg kg^{-1}. It can be seen that the As translocates only in small quantity into the grains. Kádár et al. (2000) also measured that the main storage place of the toxic elements is the root. The average arsenic level of the plants living on unpolluted site is marked on the figure and comparing our results to this data it can be seen that the applied compost doses did not cause high As level in test plants.

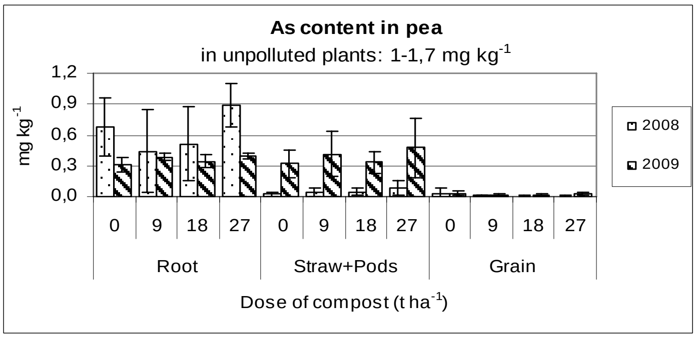

FIGURE 2 **Arsenic (As) content in different parts of pea in the sewage sludge compost treatment.**

Table 1 shows toxic elements content in test plants after the sewage sludge compost treatment. As and Cr were not measureable in triticale and maize grains in 2008. Copper (Cu) concentration was under the 5 mg kg^{-1} in the triticale, but effect of treatments increased the level of Cu in 2008 and 2009. According to Casado-Vela et al. (2007) effect of 6 kg m^{-2} compost application increased Cu content in fruit.

The quantity of Cu measured in maize was higher in the 9 t ha^{-1} and a 27 t ha^{-1} doses compost treated plots, compared to the untreated ones in 2008.

Quantity of Cr was not measurable again in the triticale crop in 2009. The concentration of As showed very low value in the triticale crop.

Extreme weather damaged the maize crop before harvesting in 2009, so we could not measure toxic elements in it.

TABLE 1 **Toxic elements content of test plants after the sewage sludge compost treatment in 2008 and 2009.**

Test plants	**Dose of compost (t ha^{-1})**	**Cr (mg kg^{-1})**				**As (mg kg^{-1})**				**Cu (mg kg^{-1})**			
		2008		**2009**		**2008**		**2009**		**2008**		**2009**	
		Mean	**S.D**	**Mean**	**S.D**	**Mean**	**S.D**	**Mean**	**S.D**	**Mean**	**S.D**	**Mean**	**S.D**
Triticale	**0**	NM	NM	NM	NM	NM	NM	0.058	0.024	3.32	1.88	4.37	0.79
	9	NM	NM	NM	NM	NM	NM	0.044	0.031	4.35	0.28	4.91	0.42
	18	NM	NM	NM	NM	NM	NM	0.051	0.065	3.78	0.38	3.81	0.91
	27	NM	NM	NM	NM	NM	NM	0.066	0.036	4.17	0.49	3.51	0.45
Maize	**0**	NM	NM	ND	ND	NM	NM	ND	ND	2.35	0.16	ND	ND
	9	NM	NM	ND	ND	NM	NM	ND	ND	3.65	2.73	ND	ND
	18	NM	NM	ND	ND	NM	NM	ND	ND	2.46	0.25	ND	ND
	27	NM	NM	ND	ND	NM	NM	ND	ND	2.92	0.54	ND	ND
In unpolluted plants (mg kg^{-1})		**0.1-0.5**				**1-1.7**				**5-20**			

ND, no data, because extreme weather damaged the maize crop before harvesting in 2009.
NM, not measurable, the concentration is under the measuring limit of the instrument.
There is not significant difference between treatments, according to the Tukey's test ($p<0.05$). Therefore we not signed the a, b,… indexes in the table.

Although the soil contained toxic elements in a small quantity, the concentrations of these elements were not measurable in every case in the plant samples. These results proved that quantification of the available part of toxic elements is important for characterization the toxicity of a soil (Rauret et al., 2000).

2.3 Toxic elements in the soil

The soil fertility is important for preservation the environment for the future generation (Várallyay, 1998). Prevention of toxic elements movements into the soil is necessary because removing them is very difficult. If there are toxic elements in the soil, you should examine their movement in the soil-plant system. Our experiments were investigated these relationships. We examined toxic element content of soil of test plants after the sewage sludge compost treatment. Table 2 summarizes the measured values in 2008.

TABLE 2 **Toxic elements content in two layers of soil of test plants after the sewage sludge compost treatment in 2008.**

Test plants	Dose of compost (t ha^{-1})	Cr (mg kg^{-1})				As (mg kg^{-1})				Cu (mg kg^{-1})			
		0-30 cm		30-60 cm		0-30 cm		30-60 cm		0-30 cm		30-60 cm	
		Mean	S.D	Mean	S.D	Mean	S.D	Mean	S.D	Mean	S.D	Mean	S.D
Triticale	**0**	3.76	5.32	1.07	1.01	1.066	0.593	0.432	0.622	3.78	2.83	3.16	0.11
	9	8.96	2.83	1.18	0.98	1.275	1.032	0.561	0.772	6.20	1.18	3.00	0.08
	18	5.69	3.04	0.90	0.73	0.976	0.412	0.718	0.566	6.27	0.27	2.80	0.38
	27	1.52	1.11	1.54	1.36	0.721	0.255	0.489	0.537	3.32	4.77	3.62	1.47
Maize	**0**	0.00	0.00	1.41	0.61	1.028	0.200	0.673	0.640	0.00	0.00	2.86	0.54
	9	1.87	2.85	2.28	1.73	1.316	0.170	0.000	0.000	1.63	2.34	3.36	0.99
	18	0.40	0.80	3.48	3.91	1.089	0.217	0.377	0.524	1.67	2.51	3.98	2.05
	27	0.55	0.85	1.18	0.78	1.162	0.445	0.801	0.191	1.28	1.71	2.94	0.53
Green pea	**0**	0.00	0.00	1.31	0.42	1.133	0.252	0.590	0.799	0.00	0.00	2.77	0.38
	9	0.00	0.00	3.12	2.98	1.276	1.113	0.000	0.000	0.26	0.50	2.74	0.27
	18	0.04	0.07	1.35	1.10	1.309	0.891	0.452	0.629	0.54	1.08	2.38	0.54
	27	0.12	0.23	1.34	0.42	1.001	0.677	0.371	0.521	0.57	1.12	2.73	0.62
approved limit[1] (mg kg^{-1})		**75**				**15**				**75**			

[1] (50/2001. Government regulation)

There is not significant difference between treatments, according to the Tukey's test ($p<0.05$). Therefore we not signed the a, b,… indexes in the table.

Cr content of the soil was below the 75 mg kg^{-1} limit in all treatments in 2008. Concentrations of toxic elements were increased in the treatments in the upper 0-30cm soil layer, but this was not significant. This change was not observed in the deeper soil layer which was indicated that the Cr was not leached from the upper soil layer. On the contrary, Weber et al. (2007) observed an increase of Cr content of soil, but they used higher (72 t ha^{-1}) compost dose in their experiment.

Measured As concentration showed an increase due to the effect of the sewage sludge compost in the soil of the test plants similarly to the Cr, but its concentration is very low. This low As concentration of soil caused the very low As concentration of test plants as it was indicated in the results of plant samples. Hartley et al. (2009) measured also an increase of As content in sandy soil amending with green waste compost.

It can be seen that the amount of Cu increased in the top soil of triticale, but it increased less in the soil of green pea and maize.

The Cu concentrations were increased by the 18 t ha^{-1} compost application. Weber et al. (2007) achieved similar results, where the highest doses of compost resulted a duplication of the Cu concentration. Mylavarapu and Zinati (2009) observed significantly higher levels of Zn, Mn and Cu in soil amended with compost at a rate of 19 t ha^{-1}. Toxic elements were detected in 2008, but it was below the official limit value.

It can be seen toxic elements content of soil of test plants after the sewage sludge compost treatment in Table 3. Cr concentration of soil showed similarly values in both soil layer (5.8-7.7 mg kg^{-1}) in 2009, which neither were above 75 mg kg^{-1} limit. The compost treatment did not increase the content of Cr in the treatment plots.

TABLE 3 **Toxic elements content in two layers of soil of test plants after the sewage sludge compost treatment in 2009.**

Test plants	Dose of compost (t ha^{-1})	Cr (mg kg^{-1})				As (mg kg^{-1})				Cu (mg kg^{-1})			
		0-30 cm		30-60 cm		0-30 cm		30-60 cm		0-30 cm		30-60 cm	
		Mean	S.D	Mean	S.D	Mean	S.D	Mean	S.D	Mean	S.D	Mean	S.D
Triticale	**0**	7.68	1.69	7.26	0.70	2.477	0.327	1.946	0.110	6.16	0.91	5.11	1.17
	9	6.90	0.61	7.38	0.29	2.528	0.133	1.918	0.155	5.98	0.43	5.84	0.42
	18	6.92	0.18	6.37	1.60	2454	0.202	1.835	0.288	5.93	0.33	4.89	0.44
	27	6.95	0.36	5.99	2.36	2.724	0.258	1.893	0.333	6.29	1.03	5.39	1.52
Maize	**0**	6.13	0.39	7.53	1.82	2.509	0.231	1.852	0.326	5.99	0.66	5.20	1.29
	9	6.72	0.94	6.75	2.17	2.383	0.183	1.797	0.189	6.03	1.10	5.41	1.90
	18	6.36	0.51	6.51	2.01	2.361	0.067	1.764	0.182	5.85	0.68	4.04	0.30
	27	5.98	0.13	5.82	1.35	2.353	0.188	1.692	0.126	5.86	0.82	4.23	0.16
Green Pea	**0**	6.78	0.92	6.48	2.50	2.597	0.105	1.726	0.206	6.12	0.11	5.01	0.23
	9	6.72	0.46	7.16	0.37	2.491	0.018	1.882	0.109	6.04	0.53	5.58	0.79
	18	7.16	1.22	7.16	2.77	2.553	0.161	1.781	0.166	6.43	0.15	5.55	1.32
	27	6.84	1.31	6.68	2.81	2.550	0.041	2.040	0.245	7.13	0.65	4.77	0.44
approved limit[1] (mg kg^{-1})		**75**				**15**				**75**			

[1] (50/2001. Government regulation)

There is not significant difference between treatments, according to the Tukey's test ($p<0.05$). Therefore we not signed the a, b,… indexes in the table.

Similar amount of As were measured in both soil depths. It was not observed significant toxic elements raises after compost application in 2009.

The Cu content increased in the 0-30 cm soil layer, but it decreased in the 30-60 cm layer after the sewage sludge compost application. Similarly, a significant increase of total Cu was found after compost application (Mantovi et al. 2005). Any significant effect of sewage sludge compost treatments on Cu, As and Cd content of the soil were not found in our experiment. The applied low compost doses did not cause any extreme toxic element accumulation in it.

2.4 Effect of sewage sludge on the crops

Vigorous development, stronger green colour and healthier crops were observed in treated fields. The yields of treated plants were higher than that of the control ones in 2008 (Fig. 3). These data show that the favourable beginning plant conditions can cause an increase in the crop yield. This quantity of maize yield on this soil type is in the range which can be achieved by application of artificial fertilizers.

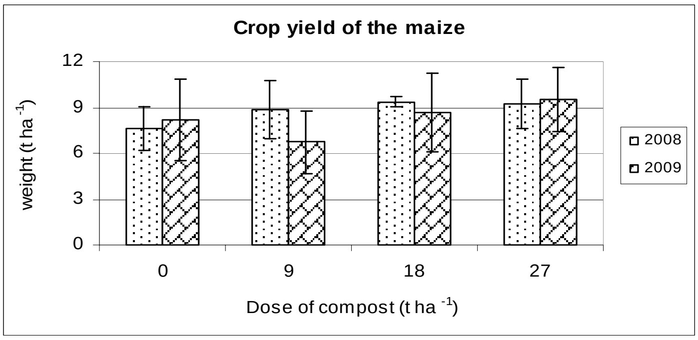

FIGURE 3 **Crop yield of the maize in the sewage sludge compost experiment in 2008-2009.**

Crops of triticale (Fig.4) were higher in the treated plots than in control plots in 2008. It was a higher harvested crop production in all treatments. Cascado-Vela et al. (2007) recorded the sweet pepper crops during crop growth in order to measure the evolution of these properties as a consequence of increasing compost applications. Similar results were obtained by Moreno et al. (1996) who have not found any treatment effect in grain yield after contaminated sewage-sludge compost application.

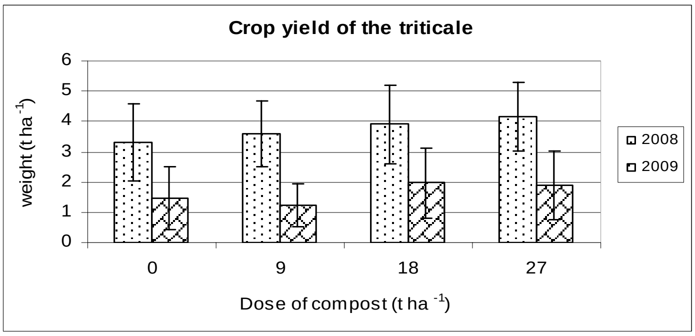

FIGURE 4 **Crop yield of the triticale in the sewage sludge compost experiment in 2008-2009.**

The measured data did not show typical crop production because the weather conditions were unfavourable in 2009. Therefore the effect of treatment was not observed in the data of triticale yield.

3. CONCLUSIONS

There is a regional sewage works in Nyíregyháza, where 150.000 m^{-3} sewage sludges originate yearly. The acidic sandy soils with low humus content are common in the Nyírség region of Hungary. The quantity of livestock manure and other organic matter have decreased last decades. The sewage sludge can be suitable for substituting these matters. Application of sewage sludge (compost) in agriculture is very difficult, because the addition of compost can cause an increase of heavy metal concentrations in soil.

These are the reason, why we decided to produce sewage sludge compost suitable for all plants. The sewage sludge of the town of Nyíregyháza contains relatively low quantity of toxic elements. Our results proved that the repeated use of composted sewage sludge for crop production did not cause any danger for agriculture and food safety. The good quality and the circumspect application of the sewage sludge compost could guarantee its safe utilization.

Amounts of toxic elements in the soils and plants were in the range of toxic elements content of unpolluted soils and plants.

The quantities of toxic elements in food and feed crops are relevant from the point of view of the food safety. Any toxic element accumulation was not found in the grain yield of test plants.

4. ACKNOWLEDGEMENTS

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(240) ORGANIC WASTE COMPOSTS AS POTENTIAL FERTILISERS FOR CEREAL PRODUCTION IN DEVELOPING MARKET CONDITIONS

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EXECUTIVE SUMMARY

Production of waste compost is well established in many European countries. In developing markets, however, the waste composts are not fully accepted as conventional organic fertilisers. The nutrient value of composts must be studied locally. The aim of the study was to investigate the potential fertilisation value of two composts for cereal production.

Two composts were compared. Compost I (C_1) was produced from source separated food and green waste, and category III animal by-products; and composted in aerated covered static piles for 6 weeks. Compost II (C_2) wasproduced from source separated food and green waste, and composted in static enclosed reactor during 2 to 3 weeks. Both composts were matured in open windows for minimum six months. The composts were mixed with mineral soil in volume 0%, 25%, 50%, and 100% of compost, and N-rates of 0 to 200 kg ha^{-1}. The substrate chemical parameters and their effect to spring wheat germination and yield structure were studied in pot trials. Germination and aboveground biomass and number and mass of grainswere measured. Three-month experiment was carried out in controlled conditions in long day conditions (16 h light) and with mean air temperature 22°C in day time.

Despite of relatively similar input materials, and similarities in chemical composition of compost, the plant response was significantly different. Compost C_2 did show good effect on seed germination irrespective of the compost rate in substrate but compost C_1 had higher fertilisation value. Compost C_1 showed a delay in seed germination. Different nitrogen norms didn´t show significant increase in aboveground biomass for compost C_2 but N rates 160 and 200 kg ha^{-1} gave positive effect when using compost C_1. Current study referred to the relevance of plant response experiments with local substrates and plant varieties, particularly in conditions of developing compost markets.

1. INTRODUCTION

1.1 Background

The European Community waste strategy set out a hierarchy for preferred waste-management options already in nineties. The targets for the diversion of biodegradable waste from landfill were published in 1999 (1999/31/EU), and they became a part of national waste legislation in Eastern-European Countries even before EU Membership. Production of waste compost is well established in many Western-European countries. In developing markets, however, the waste composts are not fully accepted as conventional organic fertilisers. More than a decade has passed from setting the targets for the diversion of biodegradable waste from landfill, but recycling of organic wastes have not yet fully recognised as a reasonable waste management method. One of the reasons is poor market situation – potential end-users of the product lack knowledge about solid waste composts, and hesitates using it.

In Estonia, 410000 t of municipal waste was collected in 2010. Waste management has followed the principles of Waste Hierarchy (2008/98/EC) at all times, and there has been a clear shift to recognise disposal as the least wanted method. Composting was foreseen as a preferred method for organic waste recycling in both National Waste Management Plans 2002-2007, and 2008-2013 (The National…, 2002; The National…, 2008). Since 2007, source-separated organic waste is collected in few municipalities, although not all. The dominating composting method is open-windrow, but AgBag System, Comp-Any Agile-Fix technology, tunnel composting, and in-vessel static pile systems along with drum-composting have been recently introduced (Kriipsalu, 2011 a). The largest waste composting facility is Tallinn Waste

Recycling Centre (max 15 000 t y^{-1}). Although compost is produced, the utilisation of it remains unsolved. The reputation of waste compost is low, as there is no historic background in using waste composts. This is a common situation in most Eastern-European countries.

Implementing of composting strategies can be successful only with good marketing of a product, but marketing requires systematic approach to compost research. The producer must know the properties of its compost, especially its nutrient value and plant response. Since the plant varieties, soil, climate and light conditions vary by countries, plant response must be studied locally.

The Estonian Waste Management Association (EWMA) has taken an initiative in promoting recovering of waste materials and products in Estonia. The mission of this non-profit association is to stand for the common interests of the members and to develop waste management in Estonia according to the principles of sustainable development. Since 2009, EWMA has been a leader for cluster programme among waste management companies. Cluster is a co-operation between the companies of similar sectors and research & educational institutions. The main objectives of a cluster are to increase the added value of the companies; and to increase the sales. The EWMA has initiated the Waste Recycling Cluster 2011-2014, which is co-financed by the European Regional Development Fund through Enterprise Estonia. There are 21 partners in the Cluster: waste companies, and three research and educational institutions, which is unique in Baltic States. The main objectives of Estonian Waste Recycling Cluster are to increase waste recycling in Estonia and to process waste into products which are compliant to quality standards, and are certified. The Cluster is focused on a) production of quality compost, b) production of recovered fuels, and c) production of recycled aggregates.

1.2 Research objectives

The aim of the study was to investigate the germination features and potential fertilisation value of compost for cereal production. Compost from two largest producers in Estonia was studied.

2. METHODOLOGY

2.1 Composts, soil, and analytical procedures

Two composts were used in this study. Compost I (C_1) was produced from source separated food and green waste, and category III animal by-products; and composted in aerated covered static piles for 6 weeks. Compost II (C_2) wasproduced from source separated food and green waste, and composted in static enclosed reactor during 2 to 3 weeks. Both composts were matured in open windows for minimum six months. Due to Animal By-Products Regulation (EC 1069/2009), the compost C_1 had to be treated 70 ºC, and 1 h. As actual measurements revealed, the temperature exceeding 70 ºC were maintained for weeks, rather than hours (Kriipsalu, 2011 b), which also resulted in smaller water content, and some loss of volatile compounds. Both composts were sieved 10 mm. The properties of soil, composts, and soil-compost substrates are presented in Table 1.

TABLE 1 **Chemical characteristics of the substrates: bare soil (S) and compost (C_1 and C_2)**

	N kg ha^{-1}	pH_{KCl}	C (%)	N (%)	NH_4-N (mg kg^{-1})	NO_3-N (mg kg^{-1})	P (mg kg^{-1})	K (mg kg^{-1})	Ca (mg kg^{-1})	Mg (mg kg^{-1})
Soil (S)		6,4	3,6	0,3	2,0	18,1	202,9	144,1	1769,7	156,59
C_1		6,6	26,3	3,9	25,4	3765,1	2177,9	7748,4	6274,4	974,5
C_2		6,8	24,6	2,3	17,6	380,0	3192,6	7158,2	3860,7	1275,1
S+C_1	40	6,3	3,3	0,3	1,0	30.2	228,2	182,7	1970,9	174,0
S+C_1	80	6,3	3,6	0,3	0,6	39,2	230,8	196,3	1886,1	172,7
S+C_1	120	6,3	3,6	0,3	0,7	51,5	236,8	211,1	1868,3	171,4
S+C_1	160	6,4	3,7	0,3	0,6	56,4	234,9	211,2	1834,8	165,8
S+C_1	200	6,4	3,7	0,3	1,2	64,5	231,3	214,3	1933,6	172,9
S+C_2	40	6,3	3,4	0,3	0,8	11,9	226,3	181,4	1892,3	174,3
S+C_2	80	6,3	3,5	0,3	1,5	16,4	224,2	194,2	1851,0	176,7
S+C_2	120	6,4	3,5	0,3	1,8	19,9	220,0	195,4	1848,4	170,2
S+C_2	160	6,3	3,2	0,3	1,1	27,2	222,3	214,2	1817,4	169,2
S+C_2	200	6,4	3,6	0,3	1,9	29,4	208,5	223,6	1779,6	172,9

The soil of the experiment originated from humus horizon of *StagnicLuvisol* by WRB (2006) classification, is with sandy loam texture. The soil analyses were carried out at the laboratories of the Department of Soil Science and Agrochemistry, Estonian University of Life Sciences. In 2011 the trial soil was slightly acidic – pH_{KCl} 6.3; C 3.6%; plant available phosphorus 202.9 mg kg^{-1}; potassium 144.1 mg kg^{-1}; calcium 1769.7 mg kg^{-1}; magnesium 156.59 mg kg^{-1}, total nitrogen 0.3% of soil. Air-dried soil samples were sieved through a 2 mm sieve and used to determine soil reaction (pH) in 1MKCl 1:2.5, organic carbon was determined according Tjurin and in order to get the humus content the number from Tjurin was multiplied with the coefficient 1.724 (Vorobeva 1998), the content of total N was measured after Kjeldahl (Procedures for Soil Analysis, 2005). The content of plant available elements (P, K, Ca and Mg) in the soil was determined by the ammonium lactate method.$NH_4^+$and NO_3^-were determined with ionchromatographDionexIC5000. Statistical analyses were performed by Statistica10.0 (Statsoft, 2005).

2.2 Plant species and testing facilities

In the experiment, wheat cv. Manu was used. The germination potential was examined at 20 ºC in darkness, and germination 95% guaranteed the viability of the seed. All experiments were conducted in controlled condition in phytotron, which was maintained at 22/18±2 ºC (day/night). The relative humidity was between 40 % and 60 %. Lights maintained on a 16/8 (day/night) cycle provided a light intensity of about 15 000 lux on the soil surface.

2.3 Germination test

Each container was of length 45 cm, width 28 cm and height 8 cm with some drain holes on the bottom. Mineral soil and compost compound was used in the experiment, where compost content in mixtures was in proportion 0 %, 25 %, 50 % and 100 %. Traditional hand-watering method was used. Soil moisture was adjusted daily by adding distillate water onto the soil surface.

One hundred seeds were sown uniformly in each container to depth of about 1.5 cm. Seed germination was determined by visual seeding emergence and was recorded daily. The test was terminated 7 weeks later. Electrical conductivity (EC) of substrates was measured by the WET Sensor WET-2 (AT Delta-T Devices) five times during the experiment at 10 day intervals.

2.4 Fertilizertest

Each pot was of 11 cm (top) or 8 cm (bottom) diameter and 10 cm height with some drain holes on the bottom, and had a petri dish (12 cm diameter) underneath. Mineral soil and compost compound were used in the experiment, where compost was added what responsive in the field condition on N-rates of 0, 40, 80, 120, 180, and 200 kg ha^{-1}. Five seeds were sown uniformly in each pot to depth of about 1.5 cm. After the seed germination, the seedlings were thinned to leave only three of the most uniform plants per pot and the test was terminated 12 weeks later. The plants were then cut above the soil surface and the fresh biomass was immediately weighed. The dry biomass was measured after oven drying at 80 ºC for 72 h. Experimentwas carried out in four replicates. At the end of the experiment grain mass and number were measured.

3. RESULTS AND DISCUSSION

In the compost $C_2$and soil mixtures the emergence of spring wheat reached the maximum germination point (Table 2) after 7 days. The emerged plants in variant C_2 were 88 to 92%, where the emergence was almost simultaneous in different mixtures, and 14 days later no further germinated plants emerged. The emergence was the same in mineral soil. In the other compost C_1 and its mixtures with soil, a relevant delay of emergence was observed. The emerged plants in C_1 and its mixtures with soil were 8 to 68%. The emergence delay was stronger where the compost concentration was higher. Second emergence was determined 14 days later and by that time the percentage of emerged plants was 36 to 68 (Table 2). The germination of wheat seeds decreased with the increase of compost percentage in the mixture, indicating phytotoxicity by nitrate (NO_3^-) concentration.

TABLE 2 **Effect of the different substrates on the seeds germination**

Substrate	Germination (%)		Substrate	Germination (%)	
	Days of sowing			Days of sowing	
	7	14		7	14
Soil	92	92	**Soil**	92	92
S + C_1 (25%)	68	68	**S + C_2 (25%)**	88	88
S + C_1 (50%)	21	55	**S + C_2 (50%)**	85	85
S + C_1 (75%)	15	43	**S + C_2 (75%)**	92	92
S + C_1 (100%)	8	36	**S + C_2 (100%)**	89	89

The electrical conductivity (EC, mSm^{-1}) expresses availability of nutrients for the plants, both in the substrate and soil. EC in soluble nutritional salt concentration in the substrates withC_1 was statistically significantly higher compared with mineral soil and C_2 mixtures (Figure 1). The substrates where C_1 concentration was 25%, 50% and 75% the plants were growing very rapidly and the leaves were of very intensive green colour which indicates that the nitrate concentration was very high. Wheat plant growth in variant S + C_1 (100%) were slowdown and and no ears were formed. Plants reached growth stages with delay compared to mineral soil and C_2 variant.

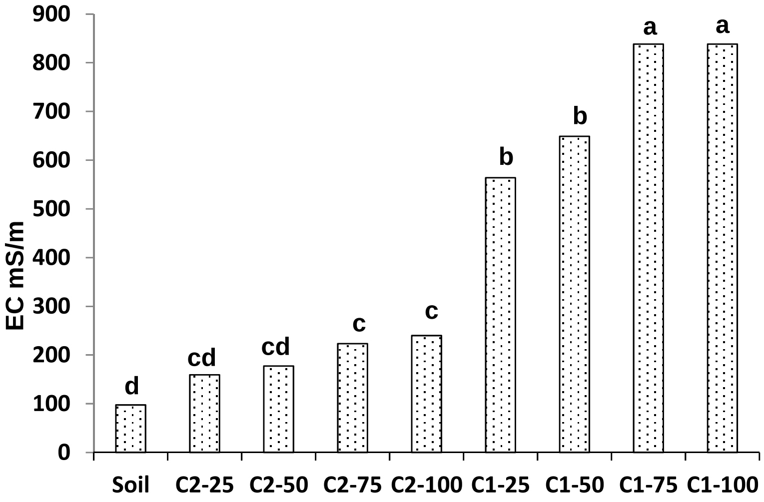

FIGURE 1 **Electrical conductivity (mSm^{-1}) in the soil and mixtures before planting**

In fertilizer test the EC decreased in the soil and mixtures since 15 July because the plants had intensively depleted nutrients during the rapid growth period. EC in C_1variants were higher compared with C_2 variants (Figure 2).

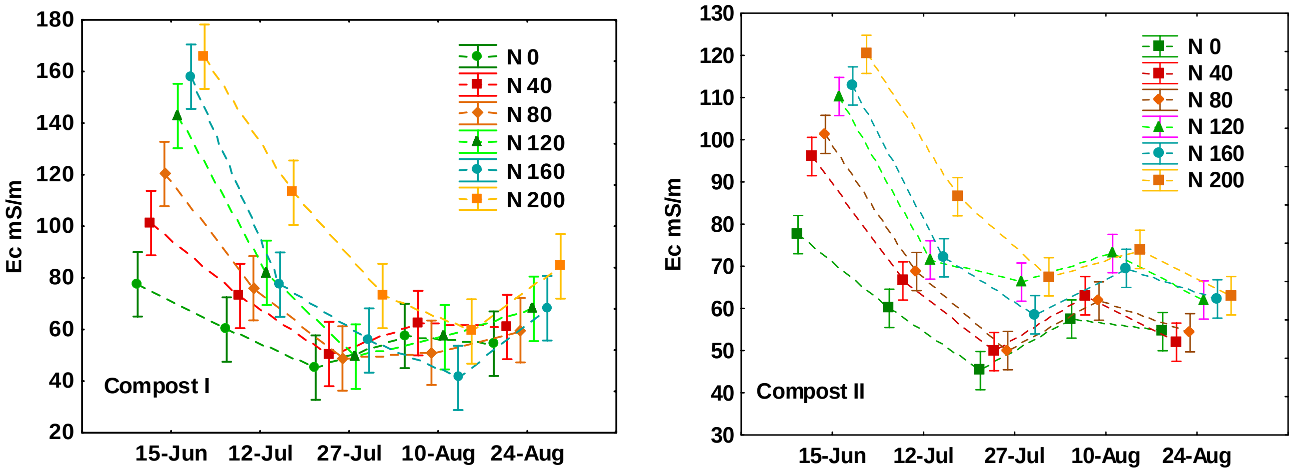

FIGURE 2 **Dynamics of electrical conductivity (mS m^{-1}) in the soil and mixtures: compost I (C_1) and compost II (C_2)**

The value of compost as a fertiliser is demonstrated by the growth of a biomass, and the grain yield of the plant. The dry weight of plants shoot, kernel weight, and number of seeds were higher in variants N160 and N200 with C_1 (Figure 3). Shoot dry weight (g) per plant was higher approximately by 0.9 g (Figure 3), and kernel weight per plant approximately 0.9 g compared with mineral soil (0.4 g).The number of kernel was approximately 28 seeds higher per plant (Figure 4). The effect of compost CII on the shoot dry weight, kernel weight and number of seeds were lower, about at the same order as in the mineral soil.

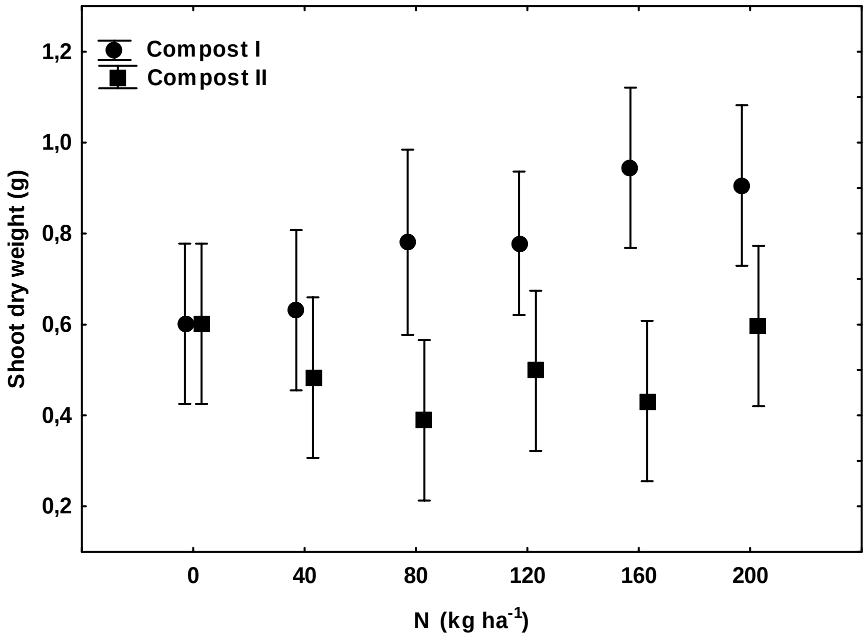

FIGURE 3 **Shoot dry weight (g) per plant**

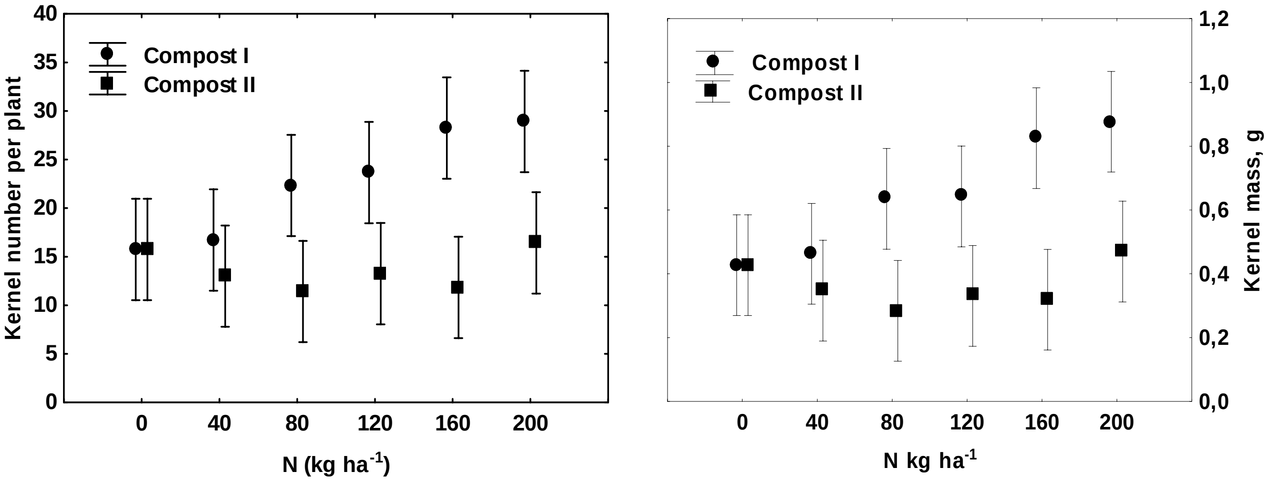

FIGURE 4 **The number of kernel and mass (g) per wheat plant**

4. CONCLUSIONS

Despite of relatively similar input materials, and similarities in chemical composition of compost, the plant response of composts was significantly different. Compost C_2 did show good effect on seed germination irrespective of the compost rate, and the germination was considered excellent. Compost C_1 showed a delay in seed germination, and germination was not complete.

Compost C_1 had higher fertilisation value than C_2. Different nitrogen norms didn´t show significant increase in neither aboveground biomass nor seeds for compost C_2. N rates 160 and 200 kg ha^{-1} gave positive effect when using compost C_1.

Current study referred to the relevance of plant response experiments with local substrates and plant varieties. These findings are particularly importance in defining marketing arguments in conditions of developing compost markets.

5. ACKNOWLEDGEMENTS

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Session 24

(31) COMPARISON OF SAPROMAT AND OXITOP-METHOD FOR ANALYSING RESPIRATION ACTIVITY (AT_4)

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EXECUTIVE SUMMARY

In the run-up for amending the Austrian landfill ordinance, parameters were developed to assess the stability/reactivity of mechanical-biologically pre-treated residual waste. The Landfill Ordinance 2008 (= Deponieverordnung 2008) regulates limit values for Respiration Activity (= “Atmungsaktivität”) $AT_4 < 7$ mgO_2/g DM, Gas Generation Sum $GS_{21} < 20$ Nl/kg DM and alternatively Gas Evolution (= “Gasbildung”) $GB_{21} < 20$ Nl/kg DM). Methods for analysing these parameters were established by the Austrian Standards Institute (OE-NORM S2027 - 1 to 3, 2004a, b and c). As laboratory practice shows, these methods also are used for the assessment of other wastes (e.g. sewage sludge, commercial waste, material from abandoned sites, bio-waste compost). For measurement of respiration activity in Austria mainly two equipments are used - Sapromat® and OxiTop®. Whether respectively to what extent these two methods give same results, is discussed in this paper.

Since 2009 at ABF-BOKU 275 respiration activity tests of samples from different stages of MBT- resp. biowaste composting processes, samples from recultivation layers, materials from landfills resp. abandoned sites and residues from anaerobic treatment were analysed parallel by Sapromat® and OxiTop®. The range of AT_4 for these 275 samples was between 0.1 and 109 mg O_2/g DM (69 of them > 20 mg O_2/g DM). Samples were ground wet to particle size < 20 mm. Water content was optimised by fist test. For Sapromat® test 30-50 g WM were put into 500 ml glass vessels; for OxiTop® test 40-65 g WM were put into 2,500 ml glass vessels. Both tests were started at same day with same well prepared sample in the same laboratory in duplicates each.

As already known there is a really good correlation between Sapromat® and OxiTop® method. First results (169 samples, $R^2 = 0.973$) already were shown at the Sardinia symposium 2011 (Binner and Lechner, 2011). In this former investigation high reactive samples were underrepresented (just 6 samples in the range $AT_4 > 50$ mg O_2/g DM). Thus additional “high reactive” samples were analysed. Correlations were evaluated for each type of material separately as well as for all materials together. OxiTop® shows lower findings than Sapromat®. For low reactive range ($AT_4 < 20$ mg O_2/g DM) readings for all the different materials show very high correlations (R^2 between 0.983 and 0.995) and similar lower findings (mean 89 %, range from 88 to 90 %, just recultivation layers show 84 %) in OxiTop® compared to Sapromat®. The mean errors of prediction are 0.4 to 1.1 mg O_2/g DM. For full range of reactivity coefficients of determination (R^2) are 0.983 to 0.996. Lower readings vary between 90 % and 97 % (mean 95 %, again low reactive recultivation layers show lowest readings 84 %). The mean errors of prediction are 0.4 to 3.1 mg O_2/g DM. The reason for increase of percentage found by OxiTop® seem to be inhibiting metabolic products of degradation, which are discharged more frequently by OxiTop® because necessity of daily opening of system for oxygen supply in case of high reactive material.

Thus we conclude that limit values of Austrian landfill ordinance 2008, which were set by investigations by Sapromat® can be analysed also by OxiTop® equipment. The limit for landfilling ($AT_4 < 7$ mg O_2/g DM) as well as the limit for leaving closed system ($AT_4 < 20$ mg O_2/g DM) can be predicted by a factor 1/0.89 (mean error of prediction of 0.9). For higher reactivity ($AT_4 > 50$ mg O_2/g DM) a factor of 1/0.95 (mean error 2.2) is to be used. Nevertheless investigations for adaptations of Sapromat® method are necessary. Possibly also during Sapromat® test for reactive samples daily opening for “aeration in between” to allow inhibiting compounds to leave the system is to be required.

1 INTRODUCTION

1.1 Background

In the run-up for amending the Austrian landfill ordinance, parameters were developed to assess the stability/reactivity of mechanical-biologically pre-treated residual waste. The Landfill Ordinance 2008 regulates limit values for Respiration Activity (= “Atmungsaktivität”) $AT_4 < 7$ mg O_2/g DM (dry matter), Gas Generation Sum $GS_{21} < 20$ Nl/kg DM and alternatively Gas Evolution (= “Gasbildung”) $GB_{21} < 20$ Nl/kg DM). Methods for analyzing these parameters were established by the Austrian Standards Institute (OE-NORM S2027 - 1 to 3, 2004a, b and c). As laboratory practice shows, these methods also are used for the assessment of other wastes (e.g. sewage sludge, commercial waste, material from abandoned sites, bio-waste compost). For measurement of respiration activity in Austria mainly two equipments are used: Sapromat® and OxiTop®.

1.2 Research objectives

For legislation in Austria and Germany the equivalency of these two equipments is an important question. For control of limit values Sapromat® as well as OxiTop® is accepted although investigations by Sapromat® were the basis for limits of Austrian landfill ordinance (Binner et al., 1999). The objective of this research was the determination of the correlation between the Sapromat® and OxiTop® on a large number of samples (275) with different types of wastes and different reactivity ranges.

2 METHODOLOGY

2.1 Sampling and sample pre-treatment

Samples of various origin (MBT-plants, composting plants, landfills resp. abandoned sites, recultivation layers (from landfill sites) and digesters) with different reactivity status (0.1 to 109 mg O_2/g DM) were taken according to Austrian Standard OE-NORM S2123-1 (2003). Fresh (wet) samples were shredded to particle size < 20 mm. For all samples stabilisation was done by freezing at -22 °C. According to draft of Austrian Standards OE-NORM S2027-4 (2011) defrosting was done gentle by putting samples in a refrigerator (+4 °C) over night.

Next morning approx. 500 g sample by fist test were wetted to a water content which is favourable for aerobic biological degradation. Pre-aeration of samples was done by spreading them in an aluminium tray (layer approx. 1-2 cm) for 5-7 hours at laboratory atmosphere. To avoid drying out samples were controlled periodically. After pre-aeration samples were homogenised again (if necessary some additional water was added).

2.2 Methods for determination

For Sapromat® test (Sapromat E® by Voith Sulzer) 30 – 50 g WM (wet matter) sample (accuracy 0.1 g) were transferred into a glass reaction bottle (500 ml) which is placed in a water bath (20 °C + 1 °C). During aerobic degradation microbes consume oxygen and produce an equivalent volume of carbon dioxide. CO_2 is adsorbed by NaOH grains. By this a negative pressure occurs within the system. By electric energy and $CuSO_4$ solution oxygen is produced until the negative pressure is compensated. The amount of produced oxygen is calculated by the needed electric energy (Binner et al., 2011), which is recorded in periods of 36 seconds.

For OxiTop®-test (WTW) 40 – 65 g WM sample (accuracy 0.1 g) after measurement of volume were transferred into a 2,500 ml glass reaction bottle. Bottles were placed in a climatic exposure test cabinet (20 °C + 1 °C). The produced CO_2 is adsorbed by soda-lime. By this a negative pressure occurs within the system. From negative pressure recorded by an OxiTop-C® measuring head oxygen consumption can be calculated (Binner et al., 2011). The period between two measuring points depends on test-duration (during the whole test-period 360 data points are recorded). Unfortunately the test period is to be chosen when starting the test. If test period is chosen to short (e.g. longer lag-phases occur) test has to be started again and readings have to be accumulated during EXCEL calculations. That is why at ABF-BOKU always a test period of 7 days is used. This leads to a period of 28 minutes between two readings.

For both types of tests 3-hours averages are calculated for estimation of the length of lag-phases (end of lag-phase is reached, when actual three hours average is higher than 25 % of the maximum three hours average). The oxygen consumptions per hour are accumulated over the whole test period (= lag-phase + 4 days). The AT_4 is calculated by oxygen sum after 4 days + lag-phase minus oxygen sum during lag-phase. The results are referred to dry matter basis calculated in mg O_2 /g DM as mean of two repetitions.

Statistic data analysis was done with SPSS 15.0 for windows. A linear regression (y = kx + d) without including constant d in equation (d=0) was used to develop a model for prediction of AT_4 -Sapromat® (y-variable) using values from OxiTop® (x-variable). Correlations are described by coefficient of determination (R^2) and mean error of prediction. Test of significance was set to 0.05, which means that all p-values less 0.05 can be trusted.

3 RESULTS AND DISCUSSION

Already data presented in Sardinia 2011 (Binner et al., 2011) demonstrated lower readings by OxiTop® compared to Sapromat® results. The mean of lower readings compared to Sapromat® was around 88 %. Due to the fact that samples in the high reactivity range (> 50 mg O_2/g DM) were underrepresented instability of the model for prediction was suspected although the huge number of samples (169). Thus further more 106 samples were analysed.

Figure 1 shows correlation between Sapromat® (x-axis) and OxiTop® (y-axis) for 206 samples of different wastes in the low reactivity range (AT_4 < 20 mg O_2/g DM). Compared to the 143 samples presented in Sardinia 2011 (Binner et al., 2011), there are just small changes in R^2 (0.983 to 0.985), k (0.885 to 0.889) and mean error of prediction (1.0 to 0.9) which demonstrates an already very stable model for 143 samples. There are no significant differences between the waste types MBT, abandoned sites and composts (readings of OxiTop® were around 88-90 % compared to Sapromat®); just recultivation material showed little bit more lower OxiTop® results (84 %) than the other materials (details see table 1).

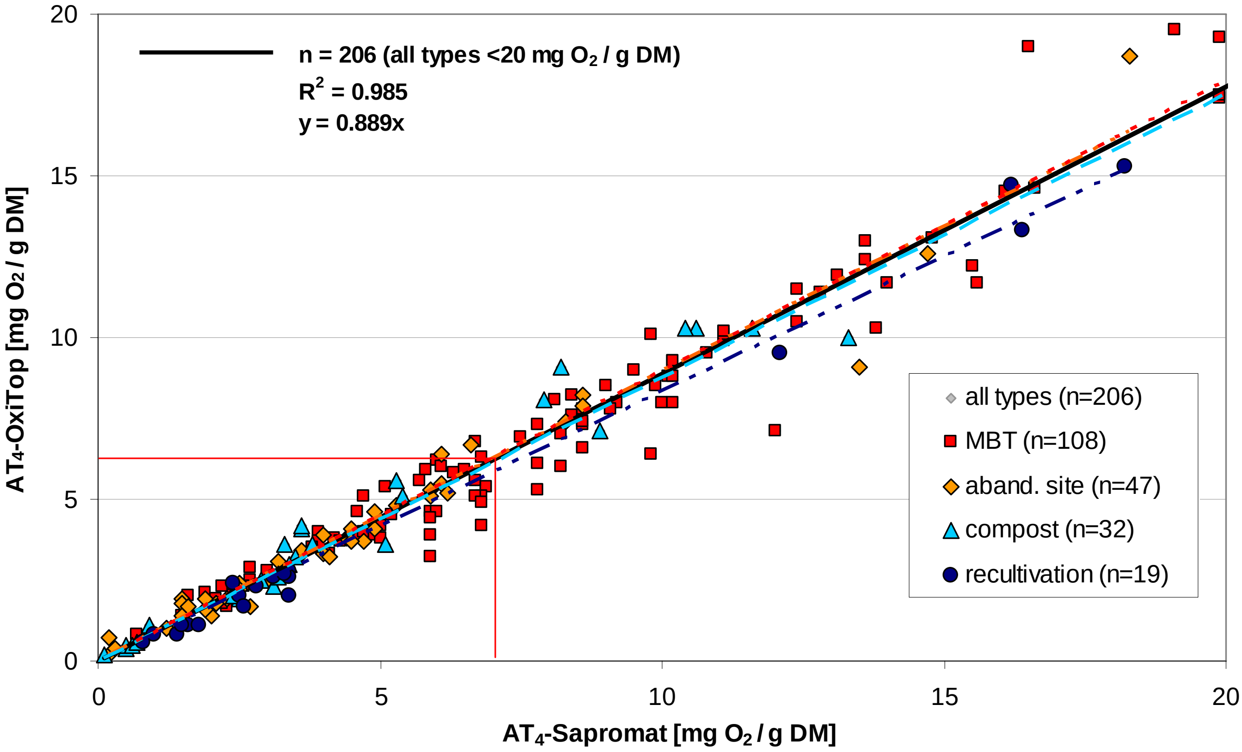

FIGURE 1 **Respiration activity (AT_4) - Correlation between measurement by Sapromat® and OxiTop® for low reactivity range (AT_4 < 20 mg O_2/g DM). R^2, n and y are shown for all types of waste (evaluation for different types of waste see table 1)**

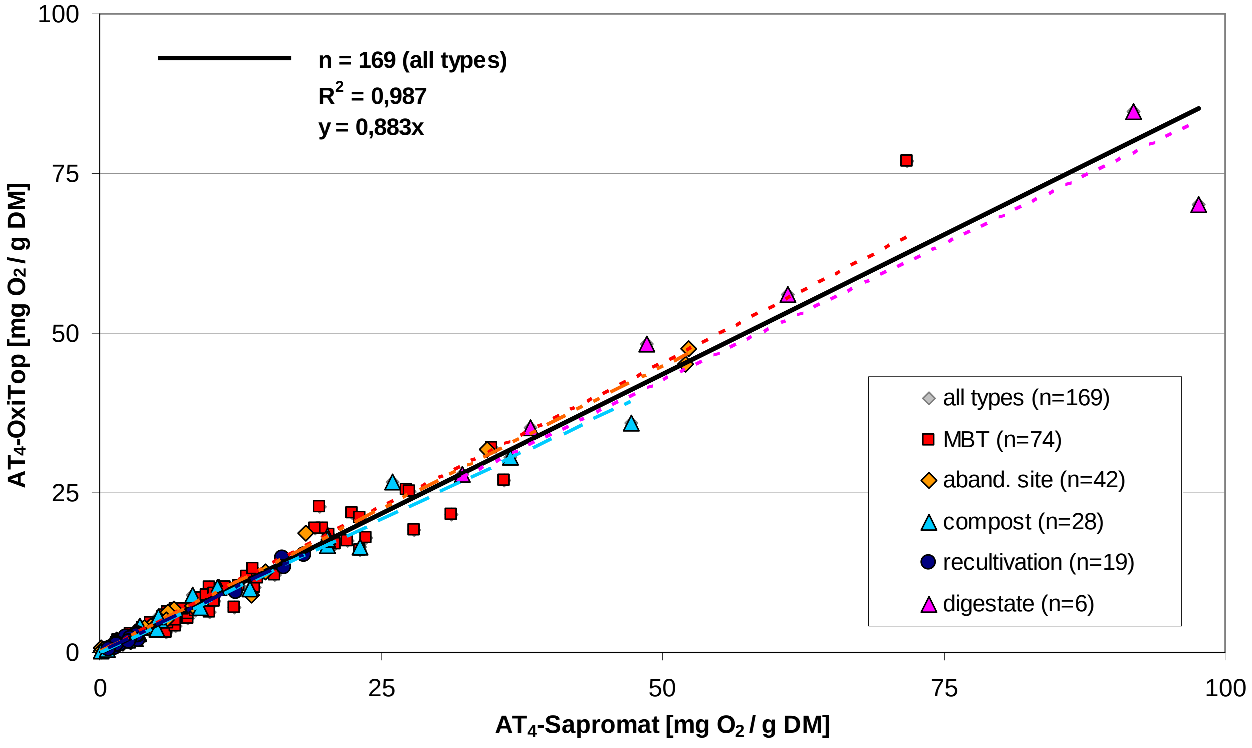

FIGURE 2 **Respiration activity (AT_4) – Correlation for 169 samples between measurements by Sapromat® and OxiTop® for whole range of reactivity (data 2011). R^2, n and y are shown for all types of waste (evaluation for different types of waste see table 1)**

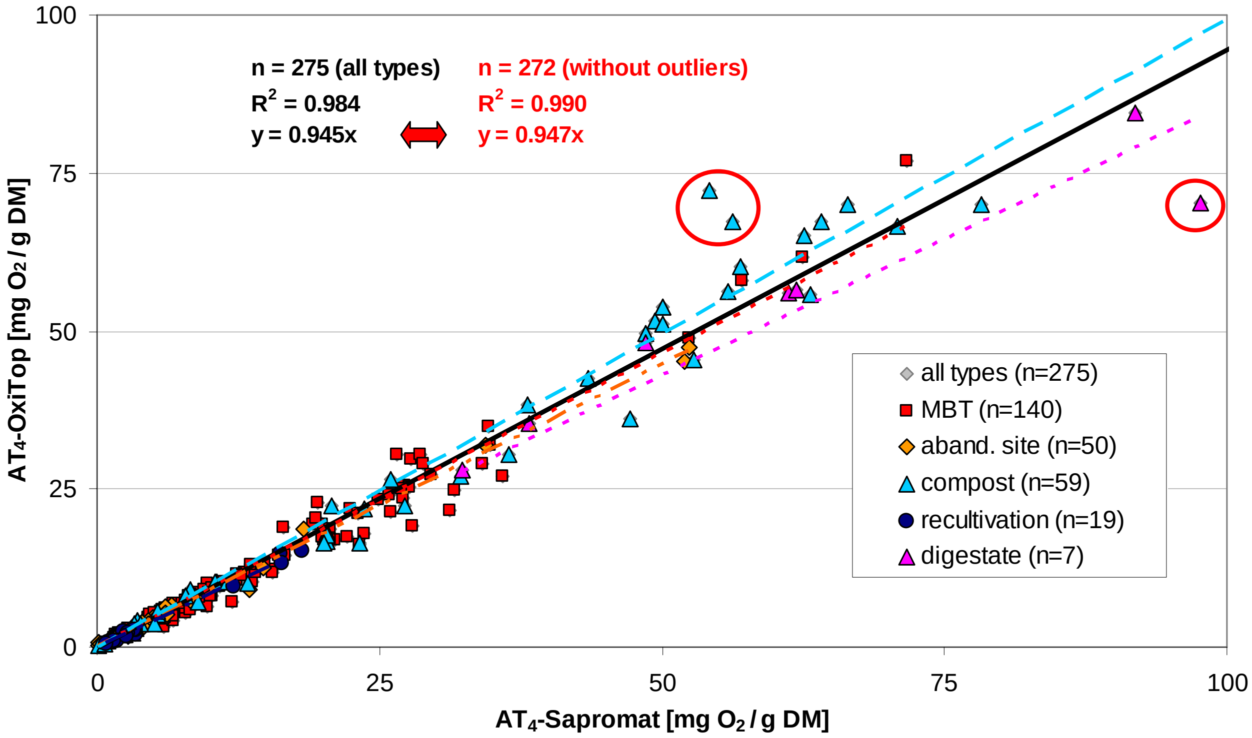

FIGURE 3 **Respiration activity (AT_4) – Correlation for 275 samples between measurements by Sapromat® and OxiTop® for whole range of reactivity (data 2012). R^2, n and y are shown for all types of waste (evaluation for different types of waste see table 1)**

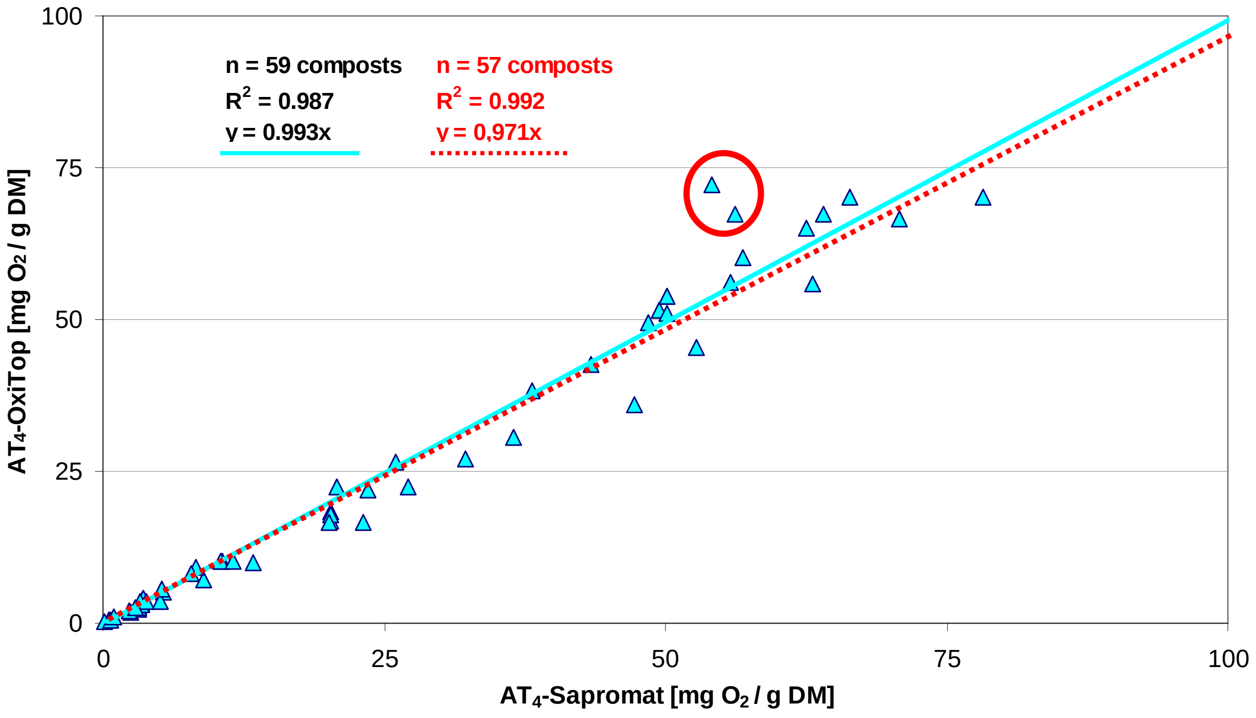

FIGURE 4 **Respiration activity (AT_4) of samples taken during composting of source separate collected biowaste (biobin) from households - Correlation between measurement by Sapromat® and OxiTop® for whole range of reactivity before and after elimination of outliers**

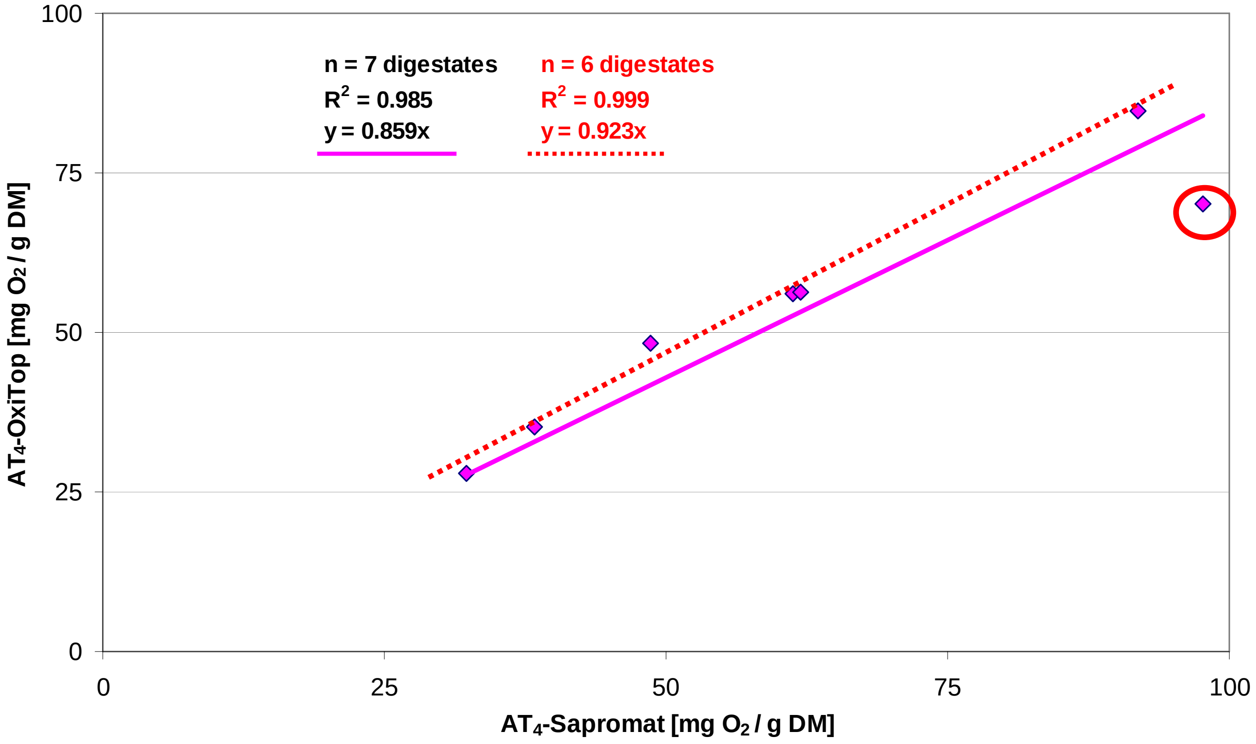

FIGURE 5 **Respiration activity (AT_4) of samples taken from digestates of source separate collected biowaste - Correlation between measurement by Sapromat® and OxiTop® for whole range of reactivity before and after elimination of outliers**

TABLE 1 **Correlation between Sapromat® and OxiTop® measurements for full reactivity range and low reactivity range (all samples and for different types of samples) - coefficients of determination, gradients of linear equation and mean error of prediction (by SPSS 15.0, linear regression, d=0).** ***Results after elimination of outliers are shown in italic stile.***

reactivity range	type of sample	n	coefficient of determination (**R^2**)	gradient of linear equ. (y = **k**x)	mean error of prediction
full reactivity range: $RA_4 = AT_4 =$ 0-109 $mgO_2 * g\ DM^{-1}$	all types	**275**	**0.984**	**0.945**	**2.9**
	all without 3 samples	***272***	***0.990***	***0.947***	***2.2***
	MBT	**140**	**0.985**	**0.930**	**2.1**
	MBT	***139***	***0.985***	***0.912***	***1.9***
	abandoned sites	**50**	**0.996**	**0.895**	**0.7**
	composting	**59**	**0.987**	**0.993**	**4.2**
	composting	***57***	***0.992***	***0.971***	***3.1***
	composting + digestates	**66**	**0.983**	**0.956**	**5.2**
	composting + digestates	***63***	***0.993***	***0.960***	***3.1***
	recultivation	**19**	**0.995**	**0.836**	**0.4**
	digestates	**7**	**0.985**	**0.859**	**7.0**
	digestates	***6***	***0.999***	***0.923***	***1.8***
low reactivity range: $RA_4 = AT_4 <$ 20 $mgO_2 * g\ DM^{-1}$	all types	**206**	**0.985**	**0.889**	**0.9**
	MBT	**108**	**0.983**	**0.897**	**1.1**
	abandoned sites	**47**	**0.984**	**0.896**	**0.7**
	composting	**32**	**0.991**	**0.876**	**0.8**
	recultivation	**19**	**0.995**	**0.836**	**0.4**

Full reactivity range shows a different picture. 169 samples analysed until 2011 (figure 2) also showed just marginal differences between the various materials. OxiTop® readings for MBT, abandoned sites and digestates were around 87 to 91 % compared to OxiTop®; compost and recultivation material showed 83-84 %. The mean for all materials was around 88 %.

The additional samples (figure 3) in the high reactive range mainly were composts and few materials from MBT resp. abandoned sites. Figure 4 shows correlation for 57 resp. 59 samples from composting plants. The readings of OxiTop® increase to 97 % of Sapromat® (two samples marked by ring were skipped as outliers). This is much higher than 83 % calculated by data 2011. MBT increases from 91 % to 93 %, abandoned sites remained at 90 %. The mean over all materials (275 samples minus 3 outliers) increases to 95 % (from 88 % in 2011). The eliminated outliers were 2 composts (figure 4) and one digestate (figure 5). After elimination of outliers the gradient of linear equation for digestates increases from 0.859 to 0.923. For composts respectively for correlation of all samples the gradient decreases from 0.993 to 0.971 resp. remains constant (0.945 to 0.947).

Due to the fact that in Sapromat® there is an even oxygen replacement, whereas OxiTop® has to be opened periodically for oxygen replacement higher readings during OxiTop®-tests for high reactive materials were unexpected. As reason we postulate metabolic products of degradation, inhibiting aerobic microbes during Sapromat® test. Inhibiting effects already were shown by Binner et al. (2007). Thus for respiration activity measurement of reactive samples they proposed additional “aeration in between” during the test (the new Austrian Standard OE-NORM S2027-4 (2011) advises “aeration in between” too).

By opening reaction bottle for oxygen supply inhibiting compounds leave the system. Because of very intensive oxygen consumption for reactive materials OxiTop® has to be opened each day, whereas in Sapromat® “aeration in between” is done just once or twice during the whole test. Thus during standard test in Sapromat® lower readings by inhibition can not be excluded except “aeration in between” also happens on daily basis. For verification of this hypothesis some more tests will follow; results will be presented at ORBIT conference.

4 CONCLUSIONS

275 samples of various wastes (from MBT- and composting plants, abandoned sites resp. landfills, digestates and recultivation layers) - 69 of them in the high reactivity range AT_4 20-109 mg O_2/g DM - were analysed parallel by Sapromat® and OxiTop®. Therefore tests were started at same day with same well prepared sample in the same laboratory.

OxiTop® shows lower findings than Sapromat®. For low reactive range $AT_4 < 20$ mg O_2/g DM readings for all the different materials show very high correlations (R^2 between 0.983 and 0.995) and similar lower findings (mean 89 %, range from 88 to 90 %, just recultivation layers show 84 %) in OxiTop® compared to Sapromat®. The mean errors of prediction are 0.4 to 1.1 mg O_2/g DM.

For full range of reactivity coefficients of determination (R^2) are 0.985 to 0.995. Lower readings vary between 90 % and 96 % (mean 95 %, again low reactive recultivation layers show lowest readings 84 %). The mean errors of prediction are 0.4 to 3.1 mg O_2/g DM. The reason for increase of percentage found by OxiTop® seem to be inhibiting metabolic products of degradation, which are discharged more frequently by OxiTop® because necessity of daily opening of system for oxygen supply in case of high reactive material.

Thus we conclude that limit values of Austrian landfill ordinance 2008, which were set by investigations by Sapromat® can be analysed also by OxiTop® equipment. The limit for landfilling ($AT_4 < 7$ mg O_2/g DM) as well as the limit for leaving closed system ($AT_4 < 20$ mg O_2/g DM) can be predicted by a correction factor 1/0.89 (mean error of prediction of 0.9). For higher reactivity ($AT_4 > 50$ mg O_2/g DM) a factor of 1/0.95 (mean error 2.2) is to be used. Nevertheless investigations for adaptations of Sapromat® method are necessary. Possibly also during Sapromat® test for reactive samples daily opening for “aeration in between” to allow inhibiting compounds to leave the system is to be required.

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(122) EVALUATION OF COMPOST MATURITY BY HOT WATER EXTRACTED (HWP) CARBON

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EXECUTIVE SUMMARY

The water soluble carbon has been proposed by several researchers as a parameter to evaluate compost evolution because its concentration rapidly decreased with the process. The authors established the 0.5 % value as a maximum content of water soluble carbon above which compost could be considered mature.

The aim of this work was evaluate the hot water soluble carbon extracted with Hot Water Percolation (HWP) as an indicator of compost maturity stages.

The composting windrow was built from green wastes on 10. October and destroyed on 21 November. The outside temperature was above 10^{o}C, and from 15 November about 5^{o}C.

The sampling times were 0, 2, 8, 16, 21, 30, 37 and 42 days after establishing the compost wíndrow. The depth of sampling was 40 cm.

Dry matter content, ignition loss (organic-C) and carbon content and spectral properties of HWP extracts were determined. The hot water percolation (HWP) is a new easily applicable soil extraction method (Füleky and Czinkota, 1993) which was adapted to compost analysis. During hot water percolation the desorbable, hydrolizable and easily soluble elements and compounds are extracted by hot water (102-105^{o}C) at 120-150 KPa (Takács-Füleky, 2003). *5* times 100 cm^3 extracts were collected and TOC was determined in the solution. The optical density of these solutions was measured at 254, 465 and 665 nm.

During composting process the amount of total organic carbon significantly decreased. At the same time the hot water soluble HWP-C content decreased from 605 mgL^1 measured on the 0 day of composting to 27.9 mg L^{-1} on the 42 day. It is a strong positive correlation between the HWP-C content of the first 100 cm^3 fraction and the sum of *5* times/100 cm^3 fractions (R^2 =0.9830). Consequently is enough to measure the carbon content and spectral properties of the first HWP fraction. The measured HWP-C content is naturally less than the measured value at other authors (0.5-1.7 *%*) because the hot water percolation (HWP) is a fast, one-two minutes long process. It is a very strong linear correlation between the absorbance at 254 nm and the HWP-C content of extracts (R^2=0.9545).

The tendency of SUVA values is very similar as the results of Said Pollicino et al., 2007. Their results show a sudden increase of SUVA after the 28 day of composting which means the maturity of compost. The E4/E6 values show the same tendency which was demonstrated by Sellami et al., 2008, when the E4/E6 significantly decreases at the maturity stage. Both the HWP-C content measured in 100 cm^3 and 5 times 100 cm^3 extracts at 254 nm and also at 465 and 665 nm show a decreasing tendency during maturation process.

As a conclusion the HWP-C content, the absorbance at 254 nm and the E4/E6 values of hot water percolation (HWP) extracts very well show and demonstrate the maturity stages of composting process, in spite of the fast extraction.

1 INTRODUCTION

Compost maturity has been determined by different workers using different indices based on physical, chemical, biological, biochemical and humification aspects of organic matter. From all of these indices, those related to the water-soluble fraction of the organic matter during composting have a special interest because it is in this fraction that most of the biochemical transformations of the organic matter take place. The intensity of organic matter degradation and the evolution of the water-soluble compounds depend on the kind of material used in starting mixtures. This fraction includes compounds such as sugars, small carbohydrates, aminoacids, small peptides, phenols, carboxillic acids, etc.

The quantification of these parameters during the composting process has led to the conclusion that simultaneously there is a degradation of the organic matter by the microorganisms and also a synthesis as a consequence of the transformation of the simple organic molecules.

Such parameters are: the water-soluble organic carbon (C_{ow}), which concentration rapidly, decreases with the process, the ratios of water-soluble carbon to water-soluble organic nitrogen (C_{ow}/N_{ow}), and water-soluble organic carbon to total organic nitrogen (C_{OW}/N_{OT}), the germination index with *Lepidium Sativum L.*, the quantification of phytotoxic substances such as ammonia, the low molecular weight organic acids, phenols, etc. The proposed values for the maturity: C_{OW}<0.5-1,7 %, C_{OW}/N_{OW} 5-6, C_{OW}/N_{OT} <0.40 (0.55), GI>60. (Sanchez-Mundero et al. 2001 Bernal et al. (1998)

The wide variation in recommandation for this parameter suggests that WSC value should be established depending on compost feedstock (Benito M. et al., 2009).

The concentration of water extractable carbon which included a high proportion of easily bio-degradable organic compounds such as sugars, amino-acids and peptides, decreased during the composting process to reach values ranging from 0.73. Water extractable carbon exhibited a slight increase during thermophylic phase, related to bio-oxidation occurring at relatively high temperature and solublizing the released carbon; it then reached 0.88. Nevertheless the previous water extractable carbon values below 1.7 % suggest a high degree of humification in the composted materials (Sellami F. et al., 2008).

The ratio between the inorganic forms of nitrogen has been used as criterion for assessing the maturity of compost. The nitrate increase, linked to nitrification during composting, resulted in NH_4/NO_3 ratio<1 at the end of the process, suggesting that the final compost had reached maturity.

The composition of cold and hot water-extractable OM was investigated. The C and N concentrations were always lower in the cold (C: 2.69 to 3.95 gkg^{-1}, N: 0.14 to 0.29 gkg^{-1}), than in the hot-water extracts (C: 13.77 to 15.51 gkg^{-1}, N: 0.34 to 0.83 gkg^{-1}). The mass spectra of the hot water-extracted organic matter revealed more intensive signals of carbohydrates, phenols, and lignin monomers (Landgraf et al. 2006).

The C_w/N_{org} ratio decreased during composting in all cases, falling from a wide range in the initial samples (0.89-3.52), to a very narrow range of 0.32-0.66 in the mature samples since the maturity index C_w/N_{org}<0.70. The chemical characteristics of the different composts studied here allowed us to establish the following maturity indices: C/N<12; C_w<1.7 %; C_w/N_{org}<0.55; NH_4^+/NO_3^- < 0.16; NH_4-N < 0.04 % (Bernal, 1998).

Bernal et al. (1998) suggested that a C/N ratio of 20 would reflect a satisfactory degree of compost maturity. They consider that a value below 12 indicates a high degree of compost maturity. Therefore the C/N ratio in the solid phase cannot be used as an absolute indicator of compost maturation due to the large variation that is dependent on the starting materials.

In both composts, the C/N ratio showed a significant decrease throughout the composting process, measuring 67.8 % and 60.11 %, respectively.

Organic carbon in the water extract of immature compost consists of phenolic compounds, sugars, hemicellulose, organic and amino acids, peptides and degradable substances. In mature compost soluble organic carbon is present as humic substances, which are resistant to further degradation thus explaining the increased stability in the compost. (Bernal, 1998)

Fulvic acids decreased gradually during the composting process. Generally fresh and raw composts contain low levels of HA and higher levels of FA compared to mature compost. The C_{HA}/C_{FA} ratio is one of the best parameters related to the degree of compost maturity. The C_{HA}/C_{FA} ratio higher than 1.6 indicates the maturity of the compost (Ko, 2008).

E_6, E_4 and E_2 were calculated, where E is the optical density of the solution and the affixed indexes 6, 4 and 2 correspond to 664 nm 472 nm and 280 nm. The ratios E_2/E_4, E_2/E_6 were determined to describe the level of the humification process intensity. The ratio E_2/E_4 is used as an indicator of the relative amounts of lignin at the beginning of humification. The ratio E_2/E_6 reflects the relation between non-humified and highly humified material. Finally, the E_4/E_6 ratio is the most indicative related to the humification degree. (Sellami, 2008)

The ratio of optical densities of humic acids and fulvic acids at 465 and 665 nm, respectively (E_4/E_6) has long been considered to reflect the degree of condensation of aromatic nucleus of humus indicating its maturity. E_4/E_6 ratio inversely related to the degree of condensation of aromatic network in HA, i.e. a low E_4/E_6 ratio would be indicative of a high degree of aromatic constituents condensation, whereas a high ratio reflects a low degree of aromatic condensation and high proportion of aliphatic structures (Aparna, 2008).

Observations were made on changes in chemical characteristics of water-extractable organic matter during composting. UV absorption at 254 nm of bull water extractable organic matter samples was measured. The measured absorbance was normalized to the concentration of dissolved organic C giving the specific UV absorption ($SUVA_{254}$), which serves as an indicator of the aromatic character of organic matter. During the 250 day composting period the organic C content of water extractable organic matter decreased significantly from 6.0 mgL^{-1} of the initial material to 1.5 mg L^{-1} at the end of the process.

The $SUVA_{254}$ values obtained for bulk water extractable organic matter were constant during the first 28d of composting with an average value of 0.97 L $mg^{-1}m^{-1,}$ but subsequently increased steadily to 1.77 and 3.02 L $mg^{-1}m^{-1}$ by day 90 and 250, respectively. Since the UV absorption of organic solutes is directly proportional to their content of aromatic compounds, these results clearly show a relative increase in the content of aromatic moieties with composting time (Said-Pollicino D. et al., 2007).

The UV absorption of the humic substances was used to evaluate the condensation degree of the humic aromatic nuclei, indicating its maturity. The absorbance at 280 nm corresponds to lignin and aliphatic structure in humic substances at the beginning of the process. The absorbance at 472 nm is the result of the organic macromolecules depolymerisation through microbial decomposition at the humification beginning. The absorbance at 664 nm is characteristic of high oxygen content and aromatic compounds produced in the stabilization phase. The progress of the three ratios (E_2/E_4, E_2/E_6 and E_4/E_6) characterizing the degree of OM maturity (Sellami F. et al. 2008).

The absorbance ratio E_4/E_6 of the humic substances is a traditional parameter to estimate the degree of humification and/or the molecular size (Senesi 1998). At the initial stage of composting, large values of E_4/E_6 ratio have been associated with the presence of smaller size organic molecules or more aliphatic structures and usually with a higher content of functional groups (Chen et al. 1997). With an increase in the composting time, the E_4/E_6ratio decreased significantly, suggested that the carbohydrates and the quinones were oxidized and bound to methoxyl groups and/or alliphantic side chains in humic substances. At the end of composting, an adequate degree of maturity and stability was achieved by a lower E_4/E_6 ratio (Sellami F. et al., 2008).

Specific absorption at 254 nm (ie. measured absorbance divided by the DOC concentration) referred to as specific ultraviolet absorbance (SUVA), has been used for many years to assess the aromaticity of DOC. Specific UV absorbance is used routinely as a criterion for water quality by the drinking water industry. Specific absorbance value, or absorptivity, measured at different key wavelength has also been used to assess a variety of properties, such as aromaticity, hydrophobic content, apparent molecular weight and size, and biodegradability. Key wavelengths selected in this study and corresponding reference works (Jaffrain, 2007).

The aim of this work was to evaluate the hot water soluble carbon extracted with Hot Water Percolation (HWP) as the indicator of compost maturity stages.

2 METHODOLOGY

Composting was made on the composting site in Ökörtelek-völgy with the GORE™ COVER technology. It is a closed system with forced aeration. The cover is a semipermeable membran which helps to ensure diffusion of gases but retains the heat and water vapor. By the helps of this technology the process is 4 weeks.

The composting windrow was built from green wastes on 10. October and destroyed on 21 November. The outside temperature was above 10°C, and from 15 November about 5°C.

The sampling times were 0, 2, 8, 16, 21, 30, 37 and 42 days after establishing the compost windrow. The depth of sampling was 40 cm.

Dry matter content, ignition loss (organic-C) and carbon content and spectral properties of HWP extracts were determined. The hot water percolation (HWP) is a new easily applicable soil extraction method (Füleky and Czinkota, 1993) which was adapted to compost analysis (Füleky et al., 2003)). During hot water percolation the desorbable, hydrolizable and easily soluble elements and compounds are extracted by hot water (102-105°C) at 120-150 KPa (Takács-Füleky, 2003). 5 times 100 cm^3 extracts were collected and TOC was determined in the solution. The optical density of these solutions was measured at 254, 465 and 665 nm.

3 RESULTS AND DISCUSSION

During composting process the amount of total organic carbon significantly decreased (Table 1). At the same time the hot water soluble HWP-C content decreased from 605 mg L^{-1} measured on the 0 day of composting to 27.9 mg L^{-1} on the 42 day. The hot water soluble carbon content of the 10 fractions can be seen on Figure 1. First order kinetic function was fitted to the measured data. It is a strong positive correlation between the HWP-C content of the first 100 cm^3 fraction and the sum of 5 times/100 cm^3 fractions (R^2=0.9830). Consequently is enough to measure the carbon content and spectral properties of the first HWP fraction. The measured HWP-C

TABLE 1 **Hot water extracted (HWP)-c and spectral properties of compost materials**

Days	Org.-C	HWP-C	Σ HWP-C	254 nm	465 nm	665 nm	SUVA	E4/E6
	%	mg L^{-1}	mg L^{-1}	cm^{-1}	cm^{-1}	cm^{-1}	L mg^{-1} m^{-1}	
0	45.4	605.0	1910	6.71	0.310	0.062	1.11	5.11
2	41.02	289.0	807	3.05	0.210	0.056	1.06	3.79
8	20.89	97.6	240	1.52	0.096	0.026	1.56	3.62
16	21.51	133.0	285	1.43	0.144	0.035	1.34	3.21
21	18.78	108.0	233	1.39	0.120	0.044	1.28	2.70
30	20.47	75.7	145	1.15	0.083	0.030	1.53	2.73
37	11.26	49.3	121	1.59	0.075	0.026	3.24	2.80
42	11.83	27.9	86	0.92	0.044	0.025	3.46	1.74

Content naturally less than the measured value at other authors (0.5-1.7 %) (Benito et al., 2009, Sellami et al., 2008), because the hot water percolation (HWP) is a fast, one-two minutes long process. Table 1 contains the measured optical density of HWP extracts at 254, 465 and 665 nm. The changes of HWP-C content during the maturation process can be seen on Figure 2 and the measured absorbance at 254 nm of these extracts on Figure 3. It is a very strong linear correlation between the absorbance at 254 nm and the HWP-C content of extracts (R^2=0.9545).

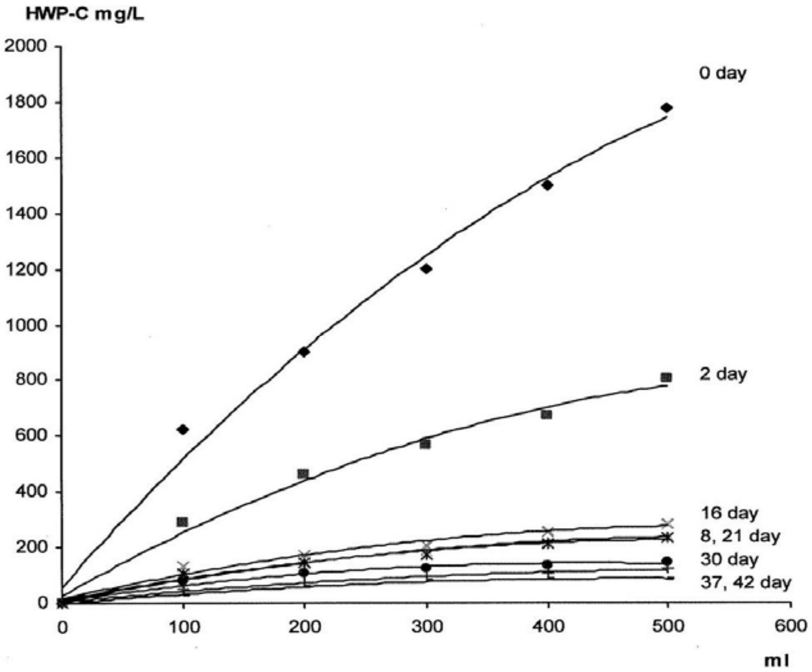
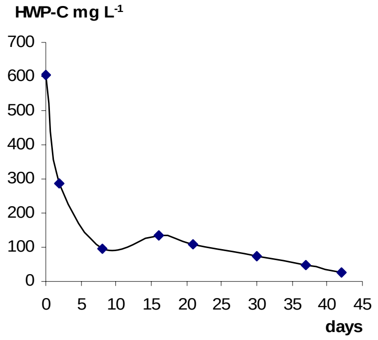


FIGURE 1 **Kinetics of HWP-C desorption**

The specific UV absorbance of HWP extracts at different maturity stages can be seen on Figure 4. The tendency of SUVA values is very similar as the results of Said Pollicino et al., 2007. Their results show a sudden increase of SUVA after the 28 day of composting which means the maturity of compost. The E4/E6 values on Figure 5 show the same tendency which was demonstrated by Sellami et al. 2008, when the E4/E6 significantly decreases at the maturity stage. Both the HWP-C content measured in 100 cm^3 and 5 times 100 cm^3 extracts at 254 nm and also at 465 and 665 nm show a decreasing tendency during maturation process.



FIIGURE 2 **Hot-water carbon (HWP-C) at different maturity stages**

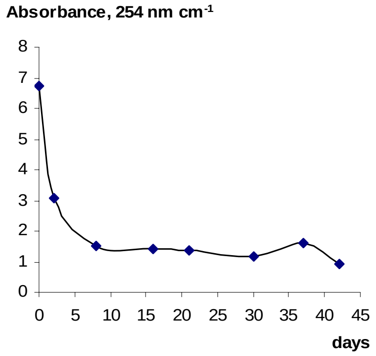


FIGURE 3 **Optical density (254 nm) of the HWP extracts at different maturity stages**

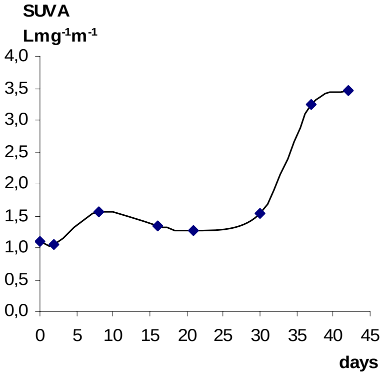


FIGURE 4 **Specific UV absorption (SUVA$_{254}$) of the HWP extracts at different maturity stages**

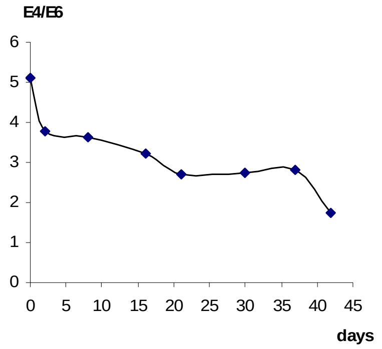


FiGURE **5** **E4/E6 values of HWP extracts at stages different maturity stage**

4 CONCLUSION

As a conclusion the HWP-C content, the absorbance at 254 nm and the E4/E6 values of hot water percolation (HWP) extracts very well show and demonstrate the maturity stages of composting process, in spite of the fast extraction.

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(201) EFFECT OF OPERATING PARAMETERS DURING COMPOST STABILITY RESPIRATION ACTIVITY TESTS

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EXECUTIVE SUMMARY

The goal of this work was to assess the effect of various operating parameters (sample size, temperature, air flowrate) during static and dynamic respiration activity tests that are used to assess compost stability. The material used in all experiments was a MSW compost derived from a commingled MSW composting facility in Athens, Greece. The facility receives raw commingled MSW that go through several pre-processing steps, an active in-vessel composting step of 18 hr and 6 weeks and a final curing period of 5-6 weeks. The material used in this research had a moisture content of less than 30% ww and had been stored in plastics bags for more than 3 years. The static respiration test was performed with 1 L air-tight vessels equipped with Oxi-Top® manometric heads. Five different sample weights were used during the static respiration assay, namely 25, 50, 75, 100 and 125 g (wet weight). Cumulative oxygen consumptions were calculated every 28 min via the pressure drops recorded by the manometric heads over a 7 day period. Carbon dioxide generation was calculated on day 1, 3, 5, and 7, according to Komilis et al. (2011), to allow the assessment of the profiles of respiratory quotients versus time. All runs were performed in triplicates at 20°C and 35°C. The dynamic respiration tests were performed in custom made 5.5 L air-tight reactors made of plexi-glass. A gravel layer placed at the bottom of the respirometers facilitated the uniform distribution of air that entered from the bottom of the reactors. The dynamic experiments were performed in a 36°C±2°C incubation room. Different dynamic experiments were performed with variable wet weights and air-flow rates. The (wet) weights of the compost placed in the dynamic reactors during all runs was 300, 600 and 900 g, whilst air flow rates were kept between 29 ml/min and 78 ml/min; the resulting unit air flowrates ranged from 6 to 30 L air $kg^{-1}VS\ h^{-1}$. Measurements of momentary O_2 consumption and CO_2 generation rates were performed 3 to 4 times daily during a 7 day period. All runs were performed in triplicates. Several dynamic respiration indexes were calculated. Respiratory quotients, cumulative O_2 consumptions ($DCRI_7$) and cumulative CO_2 productions (Total CO_2) were calculated over the 7 day period that experiments lasted. According to the results of this work, the 25 and 50 g (wet weight) sample sizes resulted in statistically similar unit O_2 consumptions and CO_2 productions during the static respiration assay, whilst the 125 g sample size resulted in significantly lower indexes compared to those obtained with the other samples sizes. The static respiratory indexes calculated at 20°C were more than 20% lower than the ones calculated at 35°C. The results from the dynamic respiration tests showed that the highest DRI_{24} (230 mg O_2 kg^{-1} DM h^{-1}) and the lowest respiratory quotient (less than 0.1), was obtained at the 30 L air/kg VS/h unit air flowrate. The highest respiratory quotient was 0.50 and was recorded at the 9 L $kg^{-1}VS\ h^{-1}$ unit air flowrate. The static CRI_7 ($SCRI_7$) at 35°C was 12 g O_2 $kg^{-1}DM$. The corresponding dynamic cumulative respiration index ($DCRI_7$) was 27 g O_2 kg^{-1} DM, and was obtained at a unit air flowrate of 24 L $kg^{-1}VS\ h^{-1}$. Conclusively, dynamic respiration tests led to higher respiration indexes compared to the static tests; in addition, sample size, air flowrate and temperature were crucial during the determination of these stability indexes.

1 INTRODUCTION

1.1 Background

Composting of organic substrates has the primary goal to produce a stable end-product. The term “stability” is related to the microbial decomposition or the microbial respiration activity of the composted matter (Iannoti et al., 1993). There has been quite a significant effort among researchers to characterize stable composts using indexes that are mainly based on the quantification of microbial respiration activity. The most common microbial respiration activity indexes are the ones based on oxygen consumption (or uptake) and carbon dioxide generation (or evolution). Respiration activity tests are classified in two major groups, namely the static and dynamic ones.

The static respiration tests (SRT) are performed at the absence of a continuous air flow; O_2 consumption is often measured via pressure differences or via direct measurements of the O_2 content usually in the gaseous phase (Binner & Zach, 1999; Gea et al., 2004; Iannoti et al., 1993; Komilis and Tziouvaras, 2009; Komilis et al., 2011; Ponsá et al., 2008). On the other hand, dynamic respiration tests (DRT) are using a continuous air flow regime with the goal to achieve an adequate oxygen supply to the microorganisms. These dynamic tests require the precise measurement of air flowrate throughout the process and the measurement of the O_2 and CO_2 contents at the inlet and outlet of the test devices (Adani et al., 2004; Scaglia et al., 2000; Ponsá et al., 2010; Tremier et al., 2005). Sample size, air flowrate and temperature seem to vary among the published respiration tests. The research team of Adani has been using samples sizes of around 10 to 13 kg, during dynamic tests, and suggests that the air flowrate should be continually adjusted during the process to constantly maintain an O_2 content equal to 140 ml L^{-1} air (14% v/v) at the reactor's exhaust gas (Adani et al., 2004; Scaglia et al., 2000). Other researchers have been using sample sizes as low as 40 g and a temperature of 20°C (Binner & Zach, 1999) and 50 dry g at a 35°C temperature (Komilis & Tziouvaras, 2009). Sample sizes of around 100 wet g have been used by the Spanish compost research team of A. Sánchez (Ponsá et al., 2010) during their dynamic respiration tests. The specific team has been using an initial airflow rate of 30 and 20 ml min^{-1} for active and more stable samples, respectively, whilst adjustment of the flow rate was made to maintain the O_2 content in the exhaust gases above 10% (v/v) (Ponsá et al., 2010). The ASTM dynamic respiration method (ASTM, 1996) is performed at 58°C, uses a 500 g wet compost weight per reactor, suggests that the unit air flowrate should not exceed 200 L kg^{-1} waste d^{-1} and maintains exhaust gas O_2 contents always above 6% (v/v) to ensure aerobic conditions. From the above, it is apparent that there is variability in the operating parameters of the respiration tests, namely air flowrate, sample size and temperature.

1.2 Research objectives

Goal of this work was to study the effect of certain operating parameters (sample size, aeration flowrate, temperature) on the respiration activity indexes produced during static and dynamic respiration tests. In particular, the effect of sample size and temperature were investigated during the static respiration assays, and the effect of unit air flowrate and sample size were investigated during the dynamic respiration tests. The same organic substrate (a MSW derived stable compost) was used in both assays.

2 MATERIALS AND METHODS

A municipal solid waste (MSW) compost was obtained from a large commingled MSW composting facility located in Athens, Greece. The compost had gone through an active 18 hr pre-composting step of intensive aeration, a 6 week in-vessel negative aeration composting phase and a curing period of 5-6 weeks prior to its storage in plastics bags. The specific samples that were used in this experiment had been stored in plastic bags for more than 3 years. Approximately 40 kg of the compost was transferred to the university laboratory for use in the experiments. Organic matter (OM) or volatile solids (VS), moisture and dry mass (DM) were determined according to standard methods.

2.1 Static respiration test (SRT)

The O_2 consumption and CO_2 production were measured using 1 L air-tight manometric respirometers acquired by WTW®. All respirometric measurements were performed in triplicates at 35°C in an incubator at the absence of light. Water was added to all composts to achieve a moisture content equal to approximately 50-55% (wet weight basis). O_2 consumption (expressed in g O_2 kg^{-1} DM) was calculated via the pressure drops recorded in the respirometers every 28 min intervals using the principles of the ideal gas law (Komilis et al., 2011). The respirometers were frequently opened, when the pressure decrease inside the respirometer exceeded approximately 100 mbar to aerate its contents. Experiments lasted 7 days. The respiration indexes that were calculated during the SRT was a cumulative O_2 consumption at 7 days ($SCRI_7$) expressed in g O_2 dry kg^{-1} of initial material. The total C-CO_2 produced by each compost was quantified on days 1, 3, 5 and 7 using a titration of an aliquot received from the alkaline trap placed in the respirometer (Komilis et al., 2011). A new 50 mL alkaline trap was placed after each measurement. A static respiration index (SRI_{24}) was defined as the peak average rate of O_2 consumption recorded over a continuous 24 hr period and was also calculated according to Komilis et al. (2011). It was expressed in units of mg O_2 kg^{-1} DM h^{-1}.

2.2 Dynamic respiration test (DRT)

The DRT was performed using a 5.5 L air tight custom made reactor from plexiglass (see Figure 1). The MSW compost sample sizes that were used in the dynamic experiments ranged from 300 g to 900 g wet weight (230 to 690 g dry matter). Deionized water was added to the compost prior to the initiation of a run to achieve an initial moisture content of 55% on a wet weight basis (ww). Branches, cut to approximately 7 cm length, were mixed with the compost at a 4:1 wet weight ratio to provide bulking. The total free air space of the reactor was 5.5 L. Gravel was placed at the bottom 7 cm to provide an air plenum. The substrate (compost + branches) had a variable height, which ranged from 7 to 23 cm during the runs. Air was distributed to the 3 reactors by a small air compressor and via a plain manifold with plastic tubing. The flow to each reactor was adjusted with a glass float type flowmeter provided by Aalborg® (PMR1-016587) with a precision needle valve attached to the inlet; the flow range was 0 to 100 ml min^{-1} and the resolution was 2.5 ml min^{-1}. Only the one top reactor outlet was kept constantly open during the experiments and was periodically connected to a portable gas analyzer. The flow was adjusted to the initial desired level for each experiment and was kept constant throughout the 7-day experimental period. No fluctuation of the flowmeter glass float was observed during the experiments. Blank runs were performed in triplicates using (i) empty reactors and (ii) reactors that contained solely the (wetted) branches that were used during the main experiments to provide bulking. Gas sampling was performed with a portable gas analyzer (GA 94, Geotechnical Instruments Ltd., U.K) equipped with infrared absorption sensors to measure CH_4 and CO_2 and with a galvanic cell type sensor to measure O_2. The gas analyzer was calibrated, and periodically checked, using standard gases provided by AEROSCOPIO Hellas S.A. (Athens, Greece). The standard gases comprised a mixture of CH_4:CO_2 at 49.9%:50.1% (v/v), respectively, and a mixture of O_2:N_2 containing O_2 at 8.06% (v/v). Atmospheric air was also used for calibration purposes assuming a 20.9% (v/v) O_2 concentration and a 0.0% concentration for CO_2. The analyzer's resolution was 0.1% (v/v). The experiments took place in a room that maintained a temperature of 35 ± 2 °C. Runs lasted 7 days. The instantaneous O_2 and CO_2 contents at the reactors' exhaust gases were measured 3 to 4 times daily at approximately equal intervals. Acquisition time per measurement was approximately 5 to 7 min so that to achieve a constant reading by the gas analyzer. The O_2 and CO_2 contents of the ambient air within the incubation room were always measured prior to the initiation and after the termination of a series of measurements. The instantaneous O_2 consumption rate per sampling event was calculated by the formula mentioned in Scaglia et al. (2000). Similarly, the instantaneous CO_2 generation rate was calculated according to formula (1). Both rates were calculated at the incubator temperature.

$$\mathrm{CO_2\ gener} = \frac{(\mathrm{CO_2out} - \mathrm{CO_2in}) * Q * 60 * 12 * 1000^{a}}{22.4 * \dfrac{273 + \mathrm{TempRoom}}{273} * \mathrm{DM} * 1000^{b}} \qquad (1)$$

Q: air flow into the reactor (ml min^{-1}) adjusted at the incubator's temperature; 60: conversion factor (min h^{-1}); 22.4: L of gas per g-mole at standard conditions (0°C, 1 atm); 12: g of C per CO_2 mole; DM: dry matter of material placed in the reactor at time 0 (kg); 1000^a: conversion factor (mg g^{-1}); 1000^b: conversion factor (mL L^{-1}); CO_2 gener: instantaneous carbon dioxide generation rate in mg C-CO_2 kg^{-1} DM h^{-1}; CO_2in: Percentage of CO_2 at the inlet of the reactor, % v/v; CO_2out: Percentage of CO_2 at the outlet of the reactor, % v/v; TempRoom: Incubation room temperature (°C). The respiration indexes that were calculated were: a 24h dynamic respiration index (DRI_{24}), a C-CO_2 index, a cumulative O_2 consumption after 7 days (herein referred to as $DCRI_7$), a cumulative CO_2 generation (Total CO_2) after 7 days, an average respiratory quotient over the 7-day period (RQ aver) and a peak respiratory quotient (RQ peak). The DRI_{24} was taken as the maximum of the averages of the instantaneous O_2 consumption rates that were recorded during a continuous 24-hour period (Scaglia et al., 2000). The C-CO_2 index was calculated based on the same concept, and was taken as the maximum of the averages of the instantaneous CO_2 generation rates recorded during a continuous 24-hour period. The cumulative O_2 consumptions after 7 days ($DCRI_7$) were calculated by numerically integrating the area under the O_2 consumption rate vs time curve from day 0 to 7. DCRIs were expressed in mg O_2 kg^{-1} DM. Similarly, the cumulative CO_2 generation (Total CO_2) after 7 days (expressed in mg C kg^{-1} DM) was also calculated by integrating the area under the C-CO_2 generation rate curve from day 0 to 7. The respiratory quotient (RQ) was calculated by the number of moles of CO_2 generated over the number of moles of O_2 consumed during the same time period.

2.3 Experimental design

The experimental design of the static respiration test was based on a two-factor ANOVA. One factor was the sample size and the other factor was the temperature. The samples size levels were: 25, 50, 75, 100 and 125 g of wet (as-collected wet compost) and the levels of the temperature factor were 20°C and 35°C.

In the case of the dynamic tests, 6 unit air flowrates (UAF) were achieved. The initial experimental conditions are included in Table 1. When UAF above 30 L kg^{-1} VS h^{-1} were attempted in this work, the resulting respiration rates could not be reliably computed. This was due to the fact that practically excess air was entering the reactor in those high UAFs, so that the O_2 content in the exhaust air was always close to the ambient air content (20.9%). In addition, significant CO_2 dilution was apparently occurring so that CO_2 was practically undetectable by the gas analyzer at the exhaust gases. All experimental runs were performed in triplicates under both static and dynamic conditions. Statistics were performed with MINITAB® v.16.

3 RESULTS AND DISCUSSION

3.1 Static respiration test results

Results of the SRT are included in Table 2. The two way ANOVA that was employed during the SRT revealed that both temperature and sample size significantly affected the CO_2 index (at $p < 0.05$). That is, the 35°C led to statistically greater amount of carbon dioxide generated compared to that at the 20°C. In addition, different sample sizes led to different CO_2 generations. The p values of the temperature factor and the sample size factor were 0.001 and 0.005, respectively. The two way ANOVA also revealed that the SRI_{24} was affected to a much greater extent by the temperature factor ($p < 0.001$) than by the sample size factor ($p = 0.044$). On the other hand, the CRI_7 was affected more by the sample size factor (p=0.012) than by the temperature factor (p=0.031). In all cases, however, both factors (sample size and temperature) significantly affected all 3 respiration indexes at $p < 0.05$.

The effect of sample size on the respiration indexes was, then, separately studied per temperature level. According to Table 2, there were statistical differences among the respiration indexes of different sample sizes at the 35°C. Table 2 shows that the $SCRI_7$ was statistically lower at the 25 g and 50 g samples sizes compared to that of the 125 g sample size. Similar results were obtained with the SRI_{24} index. Slightly different results were obtained with the CO_2 index, since both the 100 g and 125 g had statistically lower means compared to the 25 g and 50 g samples sizes. Based on the above, it appears that the 25 g and 50 g sample sizes lead to the highest respiration activities, and as the sample size increases, respiration activity diminishes. At the 20°C temperature, all sample sizes provided statistically similar results for all 3 respiration activity indexes ($SCRI_7$, SRI_{24}, 7 d CO_2). This was partly attributed to the large variability recorded for the 50 g sample size experiment. From the above, it can be deduced that the 35°C temperature leads to a significantly higher respiration activity compared to that at the 20°C. Sample size is particularly important during the SRTs at the 35°C temperature. Apparently, as the sample size within the respirometer increases, oxygen diffusion to the interior of the material is limited leading to a reduced respiration activity of the compost (at 35°C). The 25 g and 50 g samples sizes seem to produce statistically similar results for all 3 respiration indexes at both temperatures. The smaller sample sizes lead to a higher respiration activity, but should be used with caution with heterogeneous materials. Figure 2 shows two static respiration indexes and the respiratory quotient versus time. The oxygen consumption had a steady rate throughout the 7 day period, at both temperatures. This steady consumption rate profile is characteristic of stable composts (Komilis et al., 2011). The increased respiration activity at 35°C compared to 20°C is evident in both Figures 2a and 2b. $SCRI_7$ ranged from around 7 to 12.5 g O_2 kg^{-1} DM (at 35°C) and from 3 to around 10 g O_2 kg^{-1} DM at 20°C. The CO_2 generations varied from around 3 to 5 and from 1 to around 4 g C / kg^{-1} DM at the 35°C and 20°C temperatures, respectively. The lowest and highest SRI_{24} were 28 and 125 mg O_2 kg^{-1} DM h^{-1}, respectively (see Table 2). The RQ ranged from 1 to 1.2 for most sample sizes (Figure 2c) at both temperatures.

3.2 Dynamic respiration test results

The results of the dynamic respiration test and the influence of the respiration indexes by the UAF are depicted in Figure 3. According to the Figure, DRI_{24} ranged from around 120 to 230 mg O_2 kg^{-1} DM h^{-1}, whilst the C-CO_2 indexes ranged from around 2 to 34 mg C kg^{-1} DM h^{-1}. All DRI_{24} values were less than 500 mg kg^{-1} DM h^{-1}, which, according to CEN (2007), is a stability limit for very stable composts. Figure 3a reveals that there was a slight positive correlation between the UAF and the DRI_{24}. A stronger negative correlation, on the other hand, existed between the C-CO_2 index and the UAF. That is, as the UAF increased, the C-CO_2 index decreased. The $DCRI_7$ had a similar profile to DRI_{24} when plotted versus the UAF (see Figure 3b). Similarly to the C-CO_2 index, the cumulative CO_2 generation (Total CO_2) also decreased with increasing UAF (Figure 3b). As a result of the above, the respiratory quotients observed a clear negative linear correlation with the UAF (Figure 3c).

This finding is important, since it indicates that the UAF highly influences the stoichiometry of the oxidation of an organic substrate during composting. In this work, the RQs ranged from around 0.5 (at the lowest UAF) to less than 0.1 (at the high UAFs). Therefore, since RQ were found to be significantly affected by the UAF, a unique stoichiometric equation to represent the aerobic degradation of an organic substrate cannot be developed. The high variability of RQ indicates that this index cannot be used as a reliable stability index. This agrees with the findings of Gea et al. (2004).

3.3 Static and dynamic test results – A comparison

The DRI_{24} calculated during the DRT ranged from around 100 to 250 mg O_2 kg^{-1} DM h^{-1} depending on the flowrate. The corresponding SRI_{24} at 35°C varied from 77 to 126 mg O_2 kg^{-1} DM h^{-1}, i.e. up to 2 times less than the dynamic indexes. Similarly, the $DCRI_7$ ranged from around 15 to less than 30 g O_2 kg^{-1} DM, depending on the unit air flowrate. The corresponding $SCRI_7$ (see Table 2) ranged from around 7 to 12 g O_2 kg^{-1} DM (at 35°C). In this case, it is clear that the static tests produce indexes that are around 2 to 2.5 times less than the corresponding values of the dynamic tests. This agrees with the findings of other researchers (Gea et al., 2004; Scaglia et al., 2000), who reported that dynamic oxygen respiration rates can be up to 5 times higher that the static ones for the same substrate. On the other hand, the dynamic cumulative CO_2 generation after 7 days ranged from less than 0.2 to around 4 g C kg^{-1} DM, whilst the static 7-d cumulative CO_2 amounts ranged from 3.2 to 4.7 g C kg^{-1} DM. Excluding the low value of 0.2 g C kg^{-1} DM, the range of CO_2 values of the dynamic tests is around 1.5 to 4 g C kg^{-1} DM, which is a range comparable to the range obtained by the static tests. Therefore, in the case of CO_2, static and dynamic tests appear to produce comparable indexes.

4 CONCLUSIONS

The conclusions that can be drawn from this work are:

- The oxygen related respiration indexes calculated during dynamic respiration tests are 2 to 2.5 times higher than the corresponding static indices.
- The cumulative carbon dioxide generation indexes are similar during both types of tests (static and dynamic).
- The $DCRI_7$ and the DRI_{24} increase and the total CO_2 index decreases with increasing unit air flowrates.
- RQs are between 1 and 1.2 during static respiration tests. On the other hand, RQ is highly influenced by the air flowrate during the dynamic respiration tests.
- Both sample size and temperature affect the static respiration indexes. A significantly higher respiration activity was recorded at 35°C compared to the 20°C during the SRT. In addition, at the 35°C, as the sample size increased, the microbial respiration indexes reduced.

5 ACKNOWLEDGEMENTS

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Figure

FIGURE 1 **Schematic of air-tight reactor used in the dynamic respiration test**

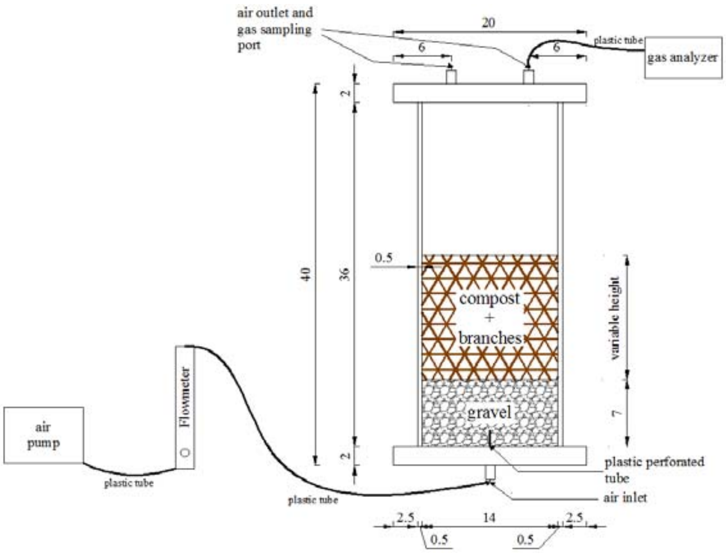

FIGURE 2 **Static respiration test respiration indexes versus time (a: oxygen consumption, b: carbon dioxide generation, c: respiratory quotient)**

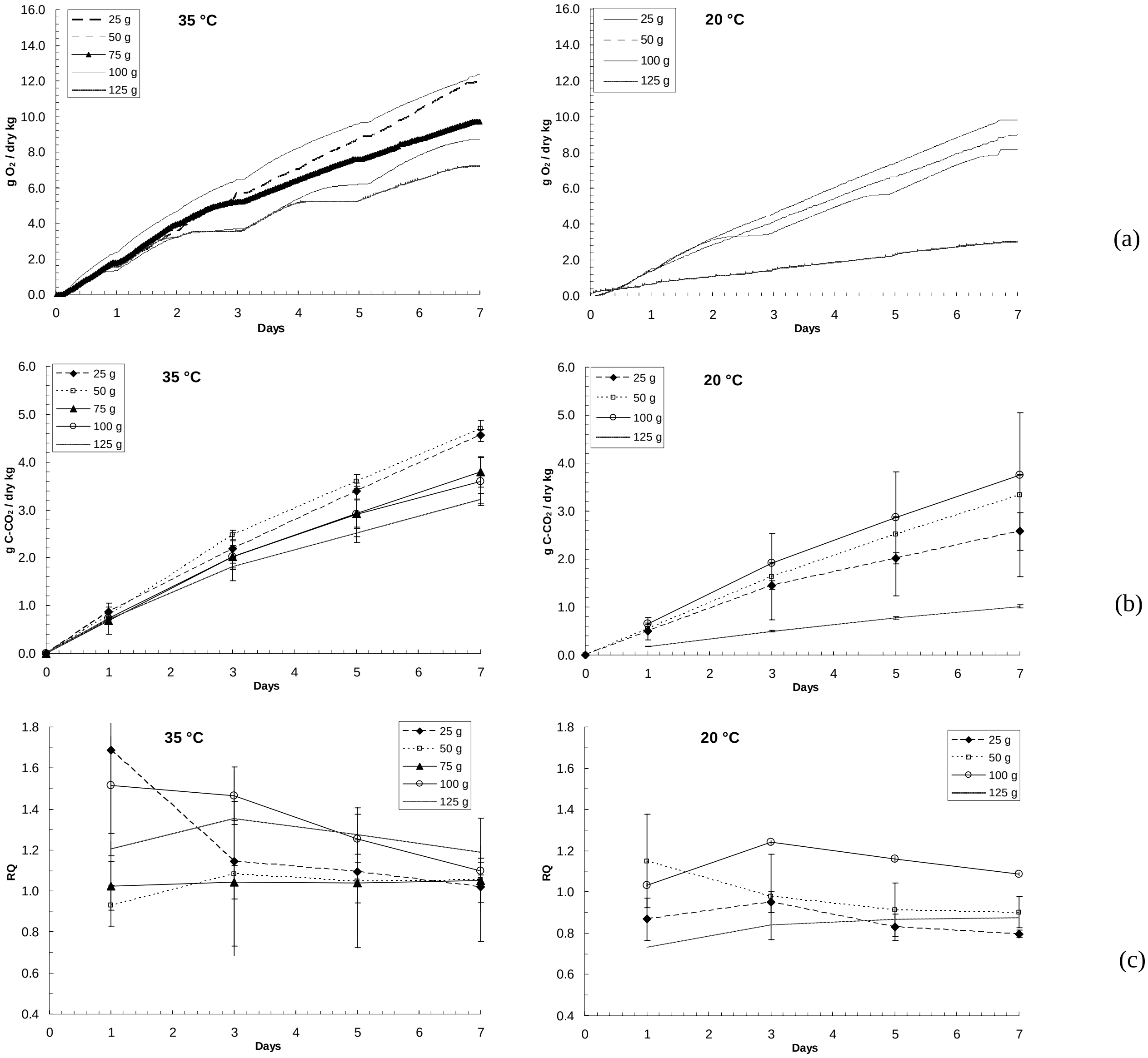

FIGURE 3 **Dynamic respiration indexes versus unit air flowrate**

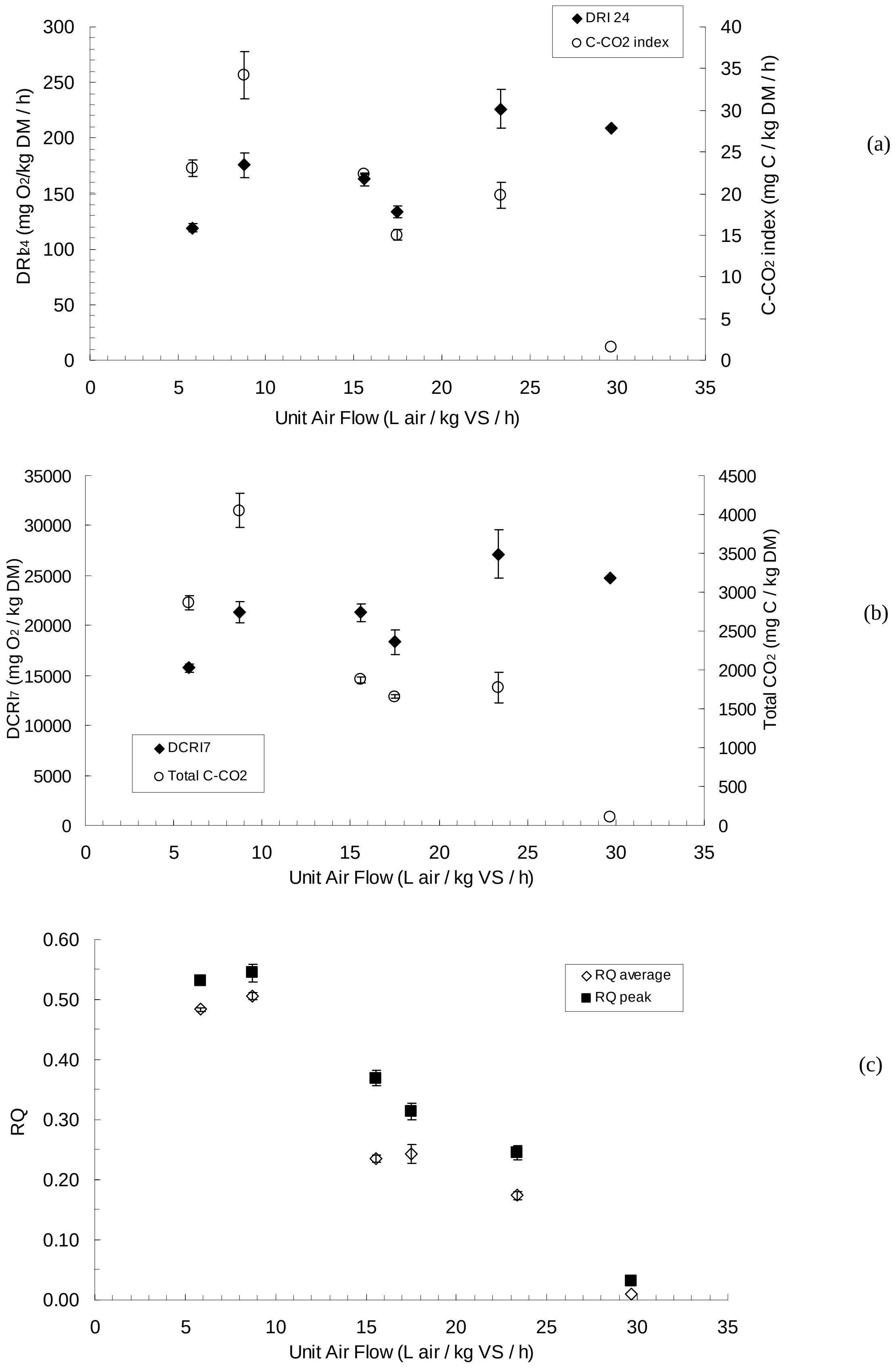

TABLE 1 **Initial experimental conditions of the dynamic respiration test**

Experiment ID	Wet mass in reactor (g)	Dry mass in reactor (g)	Volatile solids placed in reactor (g)	Constant air flowrate (ml min^{-1}) **	Unit air flowrate (L air kg^{-1} VS h^{-1}) **
1	300	231	100	29	17
2	325	250	109	54	30
3	600	462	201	29	9
4	600	462	201	78	23
5	900	693	301	29	6
6	900	693	301	78	16

*: All experiments were run in triplicate, except for experiment 2; **: Calculated at 35±2°C.

TABLE 2 **Results of the static respiration test**

	20°C			**35°C**		
Sample size	**SCRI$_7$**	**SRI$_{24}$**	**7d CO$_2$**	**SCRI$_7$**	**SRI$_{24}$**	**7d CO$_2$**
25 g	9.0 ± 1.4 ^{A}	66 ± 18 ^{A}	2.7 ± 0.47 ^{A}	12.1 ± 1.7 ^{A}	109 ± 3.2 ^{AB}	4.6 ± 0.12 ^{AB}
50 g	9.8 ± 4.6 ^{A}	81 ± 48 ^{A}	3.3 ± 1.7 ^{A}	12.4 ± 2.7 ^{A}	126 ± 2.5 ^{A}	4.7 ± 0.16 ^{A}
75 g	N/M	N/M	N/M	9.7 ± 1.6 ^{AB}	91 ± 20 ^{AB}	3.8 ± 0.32 ^{BC}
100 g	8.2 ± 1.8 ^{A}	85 ± 33 ^{A}	3.8 ± 0.007 ^{A}	8.7 ± 1.4 ^{AB}	82 ± 22 ^{B}	3.6 ± 0.51 ^{C}
125 g	3.0 * ^{A}	28 * ^{A}	1.0 ± 0.06 ^{A}	7.2 ± 0.06 ^{B}	77 ± 1.2 ^{B}	3.2 ± 0.10 ^{C}

SCRI$_7$: Static cumulative respiration index at 7 days (g O$_2$ kg^{-1} DM); SRI$_{24}$: Static respiration index (mg O$_2$ kg^{-1} DM h^{-1}); 7d CO$_2$: Cumulative carbon dioxide generation after 7 days (g C kg^{-1} DM); values are means ± one standard deviation; means, on the same column, that do not share a letter are significantly different; N/M: Not measured; *: one measurement performed during oxygen analysis.

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(231) ABIOTIC PARAMETERS AND MICROBIAL COUNTS AS STABILITY INDICATORS DURING COMPOSTING OF ORGANIC WASTE

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EXECUTIVE SUMMARY

The implementation of the EU Directives concerning the Integrated Pollution Prevention and Control (2008/1/EC) and the water policy (2000/80/EC) have oriented the waste management operations at minimization via recycling and recovery strategies. In the case of the industrial and agricultural waste, composting is often placed within the most feasible options. The performance of the composting process and the some important quality aspects of the end-product (the compost), mainly assessed by stability, are governed by both the composition - biological and elemental- of the wastes and the management strategies. In order to postulate the optimization of the quality of the compost, the combined application of independent methodologies for the determination of microbial activity is needed: the application of well-integrated perspectives and methodologies might hold a key to identify stability indices for composting.

This study reports on assessing compost stability during composting of a variety of organic waste: poultry manure; source separated biowaste; mixtures of sludge and garden clippings sprayed with certain pesticides; mixture with sludge derived from beverage production, paper pulp, mushroom substrate, horse manure; mixture with beverage sludge, mushroom substrate, horse and chicken manure. The different substrates were composted using windrow pilot or full scale systems and monitored, from raw material to mature compost (for approximately 3-5 months). The composting process was monitored through the microflora population evolution and the microbial activity, assessed through the determination of the respiration activity (using the Specific Oxygen Uptake Rate - SOUR test - Lasaridi & Stentiford, 1998a,b) and the stable isotope fractionation. To better interpret the results, a number of abiotic parameters (temperature, pH, electrical conductivity, moisture and volatile solids content) were also recorded throughout each composting process. The succession of the microbial community was studied as assessed by microbial counts. Standard procedures for compost sampling and selective substrates were used for the cultivation and enumeration of the different groups of microorganisms (total bacteria, amylolytic, cellulolytic and proteolytic bacteria, enterobacteria, fungi and yeasts, nitrifying bacteria, bacteria with resistance to antibiotics, total coliforms and *E. coli*).

In all runs, the compost temperature reached thermophilic levels (above 55 ^oC) within the first few days of processing regardless of the inherent differences of the raw materials. The physical and chemical parameters examined varied depending on the composting material. The respiration rate, as determined via the SOUR test, for all substrates increased at the beginning of the active composting phase, as complex compounds were broken down to simpler, more easily degradable ones. Thereafter, the respiration rate gradually declined as to reach values below 2.0 $mgO_2 \cdot g^{-1} VS \cdot hr^{-1}$. Moreover, results indicated that there was a correlation between composting time and some physical and chemical parameters, such as intensity of water evaporation and volatile solids reduction. The temporal evolution of the microbial community structure seems to depict well, albeit in a qualitative manner, the changes in the composting matrix during the process. Results of the microbial community dynamics analysis suggested that for all materials examined the microflora characterising the process was spontaneously developed, with no signs of inhibition, as their metabolic activity drives the process and overrides substrate differences. The observed pattern for $\delta^{13}C$ did not show any statistically significant variation during the composting process, as opposed to findings reported elsewhere. Overall, the SOUR test and the $\delta^{15}N$ signature seemed to offer a good indication of the progress of the organic matter stabilisation process during composting.

1. INTRODUCTION

The implementation of the EU Directives concerning the Integrated Pollution Prevention and Control (2008/1/EC) and the water policy (2000/80/EC) have oriented the waste management operations at minimization via recycling and recovery strategies. In the case of the industrial and agricultural waste, composting is often considered to be among the most feasible options. The performance of the composting process and the certain aspects of the quality of the end-product (the compost), mainly assessed by stability (i.e. the degree to which readily biodegradable organic matter has decomposed- Lasaridi and Stentiford, 1998a), are governed by both the composition - biological and elemental- of the waste and the process management strategies. None of the numerous tests that have been employed for stability specification has proven trouble-free or gained full acceptance (Raj & Antil, 2011; Chang & Chen, 2010; de Guardia et al., 2010; Chroni et al., 2009; Benito et al., 2003). In order to postulate the optimization of the quality of the compost, the combined application of independent methodologies for the determination of microbial activity is needed: a holistic approach to the composting process, which encompasses a full integration of microbiological, physical and chemical methods.

The nitrogen isotopic fractionation ($\delta^{15}N$) signature of the composting matrix appears to be a quite promising, yet still unexplored tool for the estimation of compost stability. Up to date, several studies have used them to examine the fate of organic matter input in soils (Yoneyama, 1996; Balesdent et al., 1988; Cerri et al., 1985), but just two have employed $\delta^{15}N$ in order to monitor composting process (Lynch et al., 2006; Kim et al., 2008). None has linked it with compost stability.

1.1 Research objectives

This study reports on the compost stabilization as assessed by the monitoring of respiratory activity using the Specific Oxygen Uptake Rate (SOUR) test, physical and chemical parameters, microbial counts and a fairly unexplored tool, the nitrogen isotopic fractionation ($\delta^{15}N$).

2. METHODOLOGY

2.1 Composting set-up and sampling

A total of eight different composting processes – the co-composting processes of two different mixtures of non alcoholic beverage sludge BIO.A and BIO.B, the composting processes of poultry manure PM1 and PM2, and the co-composting of anaerobic digested sludge and green waste GWS, GWS.M, GWS.L and GWS.ML – were monitored from raw mixture to stabilised compost. The composition of the mixtures, the system used and the monitoring time of the process are presented in Table 1. The BIO.A and BIO.B trials were set up, respectively, in two replicated windrows in a composting facility, near Akraifnio, Greece. The poultry manure (average density of 375 kg/m^3) that was used in both trials, was obtained from a typical industrial type, layer poultry farm near Oinoe, Greece. The first run, PM.A, was set in a wooden composting bioreactor, in the garden of Harokopeio University, which was designed in a way that facilitates passive convective aeration. The second run, PM.B, was set on-site in the production farm, in a Hosoya composting system which consists of two open, oval shaped channels and one linear channel, and daily receives approximately 30 to 35m^3 of poultry manure. Residence time in the specific trial was set at 19 days.

In all cases sampling intervals were determined by the temperature and the moisture profile of each substrate, given the control of other limiting factors, such as moisture content, aeration etc. Specifically, in the case of PM.B, sampling was determined by the distance that the substrate had travelled from the entrance of the system, as that distance reflects the duration of treatment and compost age.

Composite samples were collected for analyses, from three randomly selected locations from the core of the windrow or bioreactor. Following that, samples were finely ground and mixed. Sub-samples were obtained for analyses using the cone and quartering method. Sampling was scheduled on the installation day (day 0) and at short intervals throughout the thermophilic and the cooling phases of the composting processes. More specifically, sampling took place on days: 0, 3, 7, 10, 18, 25, 40, 53, and 90 for Bio.A; 0, 4, 7, 14, 36 and 56 for Bio.B; 0, 6, 8, 11, 15, 18, 24, 41, 43, and 45 for PM.A; 0, 4, 6, 16, 18, 19, and 30 for PM.B; 0, 7, 15, 21, 30, 36, 43, 51 and 64 for GWS, GWS.M, GWS.L and GWS.ML.

TABLE 1 **Composting trials**

Trial	Mixture	System	Time (days)
PM.A	Poultry manure (fresh & partly composted 1:1 v/v)	Wooden composter ($1.0\ m^3$)	55
PM.B	Poultry manure (fresh & partly composted ~2:1 v/v)	Hosoya system (gate 30 to 35 m^3 daily)	19
BIO.A	Beverage sludge ($11m^3$), mushroom bedding waste ($7.5m^3$), poultry manure ($16m^3$), hippodrome bedding waste ($7.5\ m^3$), and cocoa shells ($3m^3$)	Windrow ($45\ m^3$)	90
BIO.B	Beverage sludge ($7.5m^3$), mushroom bedding waste ($3m^3$), hippodrome bedding waste ($12\ m^3$), and paper pulp ($4.5m^3$)	Windrow ($27\ m^3$)	56
GWS	Anaerobic digested sludge & shredded green wastes (1:5 v/v)	Composter ($1.0\ m^3$)	64
GWS.M	Anaerobic digested sludge & shredded green wastes (1:5 v/v), sprayed with metribuzin	Composter ($1.0\ m^3$)	64
GWS.L	Anaerobic digested sludge & shredded, green wastes (1:5 v/v), sprayed with linuron	Composter ($1.0\ m^3$)	64
GWS .ML	Anaerobic digested sludge & shredded green wastes (1:5 v/v), sprayed with metribuzin and linuron	Composter ($1.0\ m^3$)	64

2.2 Physicochemical analyses

Compost stability was determined in duplicates in trials BIOA., BIO.B, PM.A and PM.B, using a modification of the SOUR test (Specific Oxygen Uptake Rate), run at 30 ˚C (Lasaridi & Stentiford, 1998a, b). SOUR tests were not conducted for GWS. GWS.M, GWS.L and GWS.ML due to equipment failure. Moisture content (MC – % ww), volatile solids (VS – % dw), electrical conductivity (EC) and the pH values were measured in triplicates according to FCQAO, 1994. The reduction of volatile solids content (VSred – %) was calculated on a constant ash content basis (Haug, 1993). Total carbon and nitrogen content and isotopic composition ($\delta^{13}C$, $\delta^{15}N$) of the samples (1.0 ± 0.1 mg, n=4) were determined using an automated combustion elemental analyser interfaced with a continuous- flow isotope ratio mass spectrometer (PDZ Europa, Cheshire, UK); inorganic carbon was assumed to be negligible. Natural abundance of carbon and nitrogen is expressed relative to Pee Dee Belemnite (PDB) and atmospheric N_2, respectively.

2.3 Microbial analyses

Culturable microflora was monitored through the enumeration of total coliforms, total aerobic bacteria, actinobacteria, and antibiotic resistant bacteria. Microbial groups were determined by the dilution plate count technique and the colony forming units (CFU/g DW). All microbiological analyses were performed in triplicates. Total coliforms and *Escherichia coli* were isolated on Chromocult agar (Merck) and incubated for 24h at 37˚C. Total aerobic bacteria were enumerated on Nutrient Agar (LabM) after 2 days incubation at 30˚C, while actinobacteria were estimated according to Clark (1994). Cultivable Erythromycine and Tetracycline resistant bacteria were plated on tryptic soy agar supplemented with 16 μg/ml erythromycin and 30 μg/ml tetracycline, respectively, and incubated at 28˚C for 40 hours (Mitchel Jr et al., 2008).

3. RESULTS AND DISCUSSION

3.1 Composting process

In Table 2, the initial and the last values of pH, VS and EC, as well as the max temperature values and the VS reduction (%) throughout all composting runs, are presented. The temperature variation (Figure 1) went through typical changes for each type of substrate and process used. For BIO.A, temperatures above 50˚C were recorded for 50 days. The highest value (75.6˚C) was reached by day 5. The mesophilic stage of this process lasted approximately 24 hours.

For BIO.B, the highest temperature (66.5˚C) was noted on day 16. In PM.A the highest temperature peak (72.7˚C) was recorded on day 8; the mesophilic phase lasted less than 24 hours, the thermophilic up to 50 days and the cooling phase for 14 days. Ambient temperature levels were reached by day 64. In PM.B the highest temperature reached 65.5˚C on day 4. The thermophilic phase was maintained for all the remaining period of 18 days that the substrate was treated in the system channels. For GWS, GWS.M, GWS.L and GWS.ML the highest temperatures reached were, respectively, 52.0˚C (Day 4) , 51.1 ˚C (Day5 €), 52.1 ˚C (Day 4), 55.2 ˚C (Day 5).

TABLE 2 **Specific abiotic parameters during the composting process. BIO: mixtures of beverage sludge; PM: poultry manure; GWS: mixtures of anaerobic digested sludge and greenwaste. A and B suffixes of the BIO mark the different mixtures used. A and B suffixes of the PM mark the different composting processes used. M and L suffixes of the GWS mark the different pesticide used.**

	T_{max} (°C)	MC (%) day 0/ last sample	pH day 0/ last sample	E.C. (mS/cm) day 0/ last sample	V.S. red (%)
BIO.A	75.6	62.5 / 27.3	7.8 / 9.1	3.01 / 3.55	40.2
BIO.B	66.5	51.2 / 23.6	7.9 / 9.0	1.80 / 2.70	-
PM.A	72.7	47.5 / 48.3	8.8 / 9.7	8.35 / 5.76	41.2
PM.B	65.5	65.8 / 18.6	7.3 / 9.9	3.70 / 3.92	58.8
GWS	52.0	66.6 / 63.4	8.3 / 8.4	2.52 / 1.46	63.5
GWS.M	51.1	63.8 / 59.9	8.3 / 8.3	2.21 / 1.69	65.1
GWS.L	52.3	66.6 / 59.4	8.0 / 8.6	2.13 / 1.94	63.2
GWS.ML	55.2	67.3 / 66.9	8.0 / 8.5	2.39 / 2.12	67.6

The abiotic factors (Table 2) evolved in a manner typical for composting of this type of substrates. The pH values in mixture BIO.A ranged from 7.8 (± 0.16) to 9.1 (±0.01), at day 0 and 53 respectively, while in mixture BIO.B from 7.9 (±0.27) to 9.0 (± 0.38) at day 0 and 56, respectively. In PM.A pH ranged from 8.8 to 9.7, while in PM.B from 7.3 to 9.9. The difference among the initial pH values of PM.A and PM.B, could be justified by the time (about 5 hours) that took the transportation of the PM.A from the farm to the bioreactor, as well as a potent difference in the stabilization rate of the partly composted poultry manure. For the same reason initial EC for PM.A differs significantly from EC for PM.B. EC values and trends differed between the mixtures throughout the composting processes, supporting the hypothesis of Lasaridi & Stantiford (1996) that the EC does not evolve in similar patterns. For mixture BIO.A, EC fluctuated between 3.1 (±0.7) at day 0, and 3.6 (±0.07) at day 53, whereas for BIO2 from 1.8 (±0.07) at day 0 to 2.7 (±0.25) at day 56. For mixtures GWS, GWS.M, GWS.L and GWS.ML the EC values slightly decreased.

3.2 Respiration activity

The respiration rate (SOUR test) for BIO.A, BIO.B and PM.A, PM.B substrates (not shown) was elevated at the beginning of the active composting phase, as complex compounds were broken down to simpler, more easily degradable ones, reaching a value of 8.4 mg O_2/g VS/hr (±0.92) at day 7 for mixture BIO.A, 3.9 mg O_2/g VS/hr (±0.13) at day 10 for mixture BIO.B, 1.13 mg O_2/g VS/hr at day 8 for PM.A and 1.47 mg O_2/g VS/hr at day 4 for PM.B. Thereafter, the respiration rate gradually declined as to reach values below 2.0 mg O_2/g VS/hr by the day 25 for BIO.A and by the day 14 for BIO.B. According to the respiration rate, the stabilisation process lasted longer for mixture BIO.A. Concerning the PM.A, an intense decrease of SOUR values occurred within Day 0 and Day 6, which could be attributed to the presence of ammonium nitrogen in the initial mixture. Leth et al. (2001) reported that according to Reitzer (1996) most of the microorganisms prefer ammonium as nitrogen source, while the uptake of ammonium results in repression of enzymes that catabolise other nitrogen sources (i.e. urea). This repression could be reflected with the reported decrease of respiration activity. Overall, the results of this study confirm the pattern of SOUR variation during composting, and especially the gradual decrease, consistently recorded after the end of the thermophilic phase (Chroni et al., 2009), allowing the parameter to serve as a reliable compost stability indicator (Lasaridi and Stentiford, 1998a).

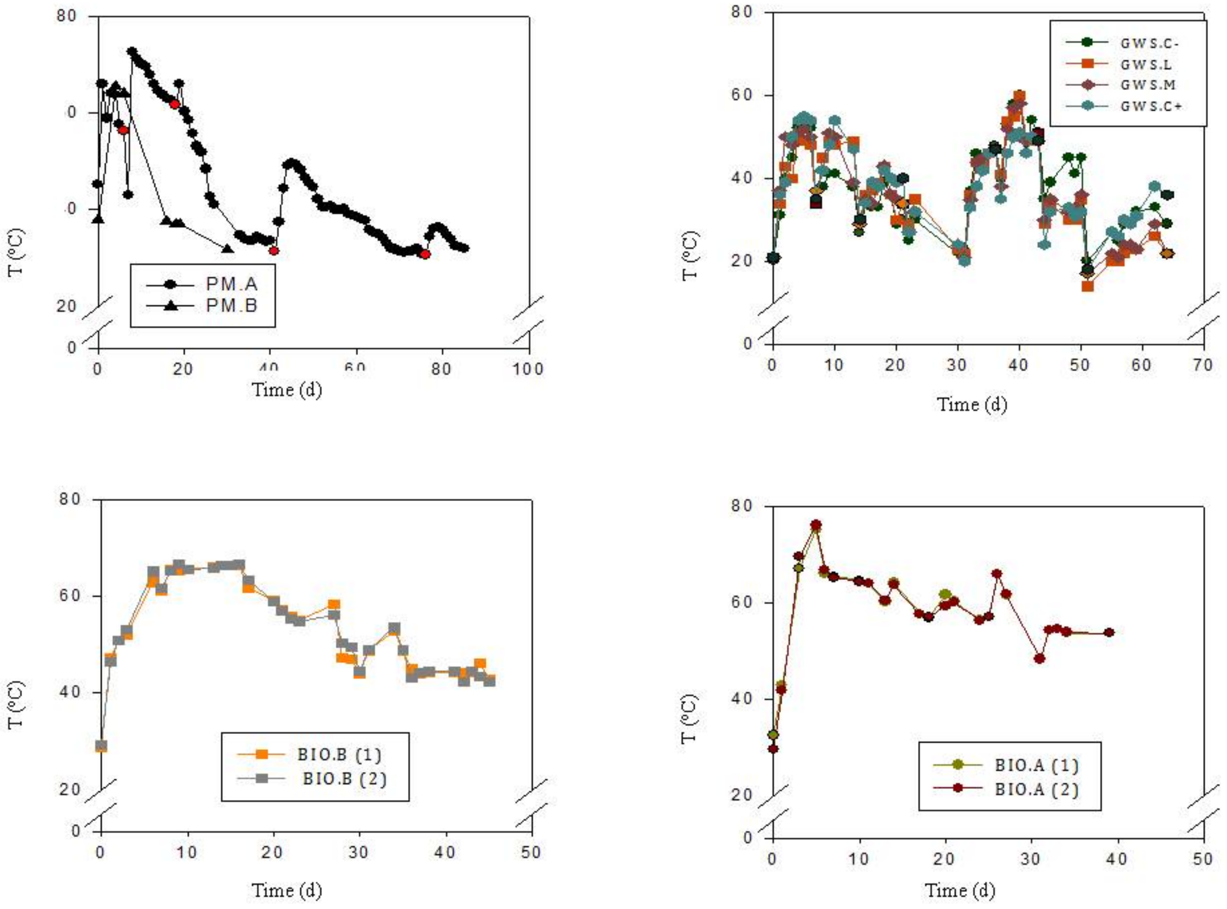

FIGURE 1 **The temperature profiles of the composting processes**

3.3 Changes in δ^{15}N during composting

As opposed to the findings of Lynch et al. (2006) who reported a slight depletion of δ^{13}C , the observed pattern for δ^{13}C did not indicated any pattern. On the contrary, the δ^{15}N changes revealed potentially useful information about the organic matter transformations. During the first days of composting, an increase in δ^{15}N was noted, indicating an intense ammonia volatilization in the lower bulking agent mixture. This is in accordance with the findings of Kim et al. (2008) and is supported by the SOUR-test results reported in section 3.2. As Yoneyama (1996) indicated, mineralization of relatively ^{15}N-depleted nitrogen and re-assimilation of relatively ^{15}N-enriched nitrogen during microbial N turnover may result in ^{15}N enrichment in acid hydrolysable fractions, as compared to non-hydrolysable fractions resistant to microbial cycles. Loss of ammonia from animal manure results in δ^{15}N values above 10‰ or even above 20‰. Hence, it has been suggested that δ^{15}N can be used to trace the fate of manure N in the soil-plant system (Hogberg, 1997) or investigate the bacterial activity in composting systems. Comparing the composting trials, it appears that the type of the composting mixture affects the nitrogen isotopic fractionation, though not the pattern of the increase.

3.4 Microbial monitoring

Referring to population of total coliforms in BIO.A and BIO.B, the population of *E.coli* declined below the detection limit by Day 7 and 14 respectively, while the total coliforms population decreased during thermophilic phase, but re-emerged in the cooling phase. Although the hypothesis of a contamination from the turning machine can not be rejected on the basis of the existing results, it is believed that the recorded emergence of the aforementioned bacterial populations is likely to be caused by the characteristics of a typical windrow management scheme. In PM.A and PM.B population of *E.coli* declined below the detection limit by Day 2 and 4 respectively. Microbial counts of bacteria resistant to Erythromycine and Tetracycline were run on BIO.A, BIO.B. and PM.B (not on the rest of the trials.

The populations of bacteria resistant to Erythromycine and Tetracycline declined during the thermophilic phase of the composting process, but were not decimated in every case.

4. CONCLUSIONS

The results of the microbial population dynamics monitoring suggested that for all materials the microflora characterising the process was developed without signs of inhibition. The microbial metabolic activity overrided substrates differences. The high temperatures are necessary, but not sufficient for the decimation of pathogens. The key players to the latter are the exposure time to high temperatures and the composting management system. Overall, the SOUR-test proved to be useful as an indicator to stabilisation of compost. It also seems that δ^{15}N signature might offer a good indication of the progress of the stabilization process during composting.

5. ACKNOWLEDGEMENTS

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Session 25

(21) AEROBIC PRE-TREATMENT OF MECHANICAL SCREENED MSW : EFFECTS ON LANDFILL ANAEROBIC BIODEGRADATION

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EXECUTIVE SUMMARY

The Municipal Solid Waste (MSW) resulting from Source Segregated (SS) collection follows different fate depending on the waste management system implemented in the several European States. The EU27 mean data shows that about 38% w/w of the 255.000.000 tonnes per year of waste produced is directly disposed in landfill without any pretreatment. This makes landfill one of the most important sources of anthropogenic GHG emission, contributing for many EU states from 3 to 13%. Further, landfill produces also other pollutant substances, as leachate, that has to be correctly managed for avoiding environmental threats.

In that areas that lacks of incineration facilities, the Mechanical Biological Treatment (MBT) of the MSW represents a possible solution for trying to increase the amount of recovered materials and reducing both the mass and the reactivity of the waste disposed in landfill. Combining the MBT with the management of the landfill in bioreactor mode, instead of traditional one, can represent a sustainable solution for waste management in many European areas. Inside bioreactor landfill, biological aerobic and/or anaerobic processes are enhanced for achieving waste stabilization and environmental recover of the site, in a narrowed period (i.e. 10-15 years instead of 40-50 years). The attention of this work has been focused mainly on the MBT treatment before landfilling.

Starting from the Waste Organic Fraction (WOF) arising from the mechanical sorting section of an existing MBT facility, different analysis have been performed on the effect of aerobic pretreatment length both on WOF stability and on its behaviour during anaerobic degradation process. Aerobic treatment has been performed on 500 kg of WOF by two aerobic bins of about 800 litres of volume each one. The Landfill Gas production (LFG) has been evaluated by a modified incubation test, treating about 5 kg of WOF sample with no inoculums. The stability achieved by the WOF after different days of aerobic pretreatment has been evaluated both by a self developed Dynamic Respiration Index (DRI) apparatus and by a fermentation test for the evaluation of the GB parameter.

Both the WOF as produced by the MBT and the WOF samples with different days of aerobic pretreatment have been characterized by Moisture Content (MC) and Volatile Solids (VS) determination.

As expected, longer is the aerobic pretreatment length lower is the humidity of the sample. After the 30 days both the MC and the VS concentration remains quite constant.

The most suitable GB parameter for comparing the different WOF samples suitable is the BG at 42 days, as a consequence of the long lag phase occurring for the WOF with 0 days of aerobic pretreatment. GB at 42 days decreases as the days of pretreatment rises. The DRI confirms substantially these results eve if the trend is less regular. The maximum DRI potential is achieved for the sample with 0 days of pretreatment, about 5,000 $mgO_2/kgVSh$, and the minimum is achieved for the sample with 120 days, about 330 $mgO_2/kgVSh$.

The aerobic pretreatment increases the rapidity of the methanogenic phase, related to one of the WOF with 0 days, achieving its maximum value for the sample with 30 days.

In this case the LFG production related to the VS content of the WOF as withdrawn from the MBT is of about 85 $Nl/kgVS_0$ with a mean methane concentration of about 45% up to 55% v/v.

These results shows the positive effect that aerobic pretreatment can have on the anaerobic biodegradation process of the biodegradable fraction of MSW, aspect that can lead to further benefits if combined with the management of the landfill in sustainable mode.

1 INTRODUCTION

The Municipal Solid Waste (MSW) resulting from Source Segregated (SS) collection follows different fate depending on the waste management system implemented in the several European States. The EU27 mean data shows that about 38% w/w of the 255.000.000 tonnes per year of waste produced (ISPRA, 2011) is directly disposed in landfill without any pretreatment. This makes landfill one of the most important sources of anthropogenic GHG emission, contributing for many EU states from 3 to 13% (EEA, 2011). Further, landfill produces also other pollutant substances, as leachate, that has to be correctly managed for avoiding environmental threats.

In that areas that lacks of incineration facilities, the Mechanical Biological Treatment (MBT) of the MSW represents a possible solution for trying to increase the amount of recovered materials and reducing both the mass and the reactivity of the waste disposed in landfill. Combining the MBT with the management of the landfill in bioreactor mode (Benson et al., 2007; Cossu, 2010; Di Maria and Valentini., 2011), instead of traditional one, can represent a sustainable solution for waste management in many European areas. Inside bioreactor landfill, biological aerobic and/or anaerobic processes are enhanced for achieving waste stabilization and environmental recover of the site, in a narrowed period (i.e. 10-15 years instead of 40-50 years). In this work the effect of aerobic pretreatment of the Waste Organic Fraction arising from an existing MBT facility, on its behaviour during degraded under anaerobic conditions, has been experimentally investigated.

1.1 Background

MBT facilities operating for waste recovery together with mass reduction and stabilization before landfill, consist generally of three main sections. The first section is represented by the in inlet section, in which the MSW resulting from SS collection are directly discharged by the collection lorry. From this first section the MSW, after light shredding, are generally moved to the Mechanical Sorting section were the Dry Fraction (DF), rich of light materials with high Calorific Value (i.e. paper, plastic, cardboard, textile, wood,...), is separated from the Waste Organic Fraction (WOF) that is generally rich of Organic Fraction (OF) and fines (20S) (particles <20mm). The most diffused screening technology are trommels with holes ranging from 30 to 180 mm of diameter, depending on the MSW features and on the scope of the treatment. DF can be further refined by removing the metals content (*e.g.* magnets and eddy currents), by segregating the inert components with aeraulic and ballistic separation, for increasing material recovery including the production of Solid Recovery Fuel for co-combustion purposes. The WOF, after ferrous and non ferrous metals separation, is moved to the MBT biological treatment section. Here WOF is usually aerobically treated for reducing both its biodegradable content and its biological activity, before being disposed off in landfill. The aerobic biological treatment leads also to the reduction of the weight as a consequence of the process loss in Moisture Content (MC) and Volatile Solids (VS) concentration.

The effect of the MBT of MSW on landfilling behaviour have been largely investigated (De Gioannis et al., 2011; van Praagh et al., 2009; Bazema et al., 2007; Komilis et al. 1999; Binner and Zach, 1999) and shows the positive effect that this can have on the environment by reducing gaseous and liquid emissions. Another positive effect of the aerobic pre treatment concerns the possibility of enhancing the anaerobic process occurring in the landfill. Infact, if opportunely performed, aerobic pretreatment can lead to a reduction of the WOF rapid biodegradable material concentration, reducing the inhibition phenomenon occurring when anaerobic degradation occurs in presence of high VS concentration (Schievano et al., 2010).

If the landfill is managed in bioreactor mode instead of the traditional one, exploiting leachate recirculation and other management techniques, the WOF aerobic pretreatment can bust the rapidity of biomethane production leading to further advantages as increase in energy recovery, reduction of the length of the period of Landfill Gas (LFG) production and reduction of the leachate pollutant concentration. In other words, the landfilled waste, along with the landfill site, can be stabilized and environmentally recovered in a shorter period (Fig. 1).

1.2 Research objectives

Aerobic pretreatment of WOF arising from mechanical screened MSW before landfilling, can have relevant influences both on gaseous and liquid pollutant emissions. The aim of this work is focused mainly on the gas emissions of WOF once anaerobic conditions are established in bioreactor landfill.

Depending on aerobic pretreatment length and process conditions, it is possible to achieve different results ranging from a simple reduction of the rapid biodegradable materials content to a more or less high stabilization level of the waste.

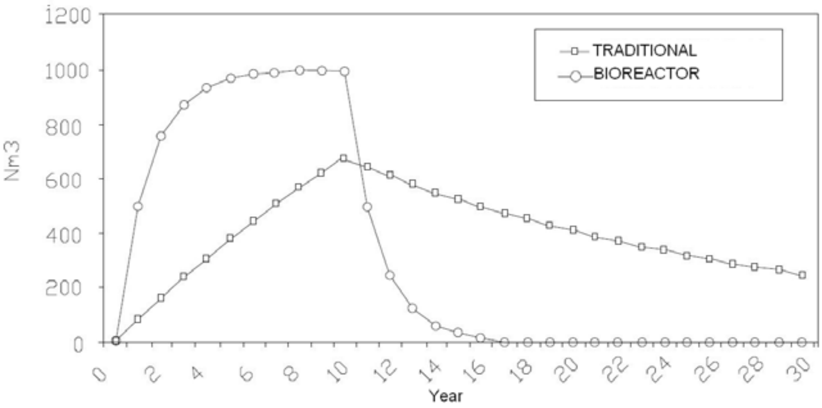

FIGURE 1 **LFG production comparison for a landfill managed in traditional and bioreactor mode.**

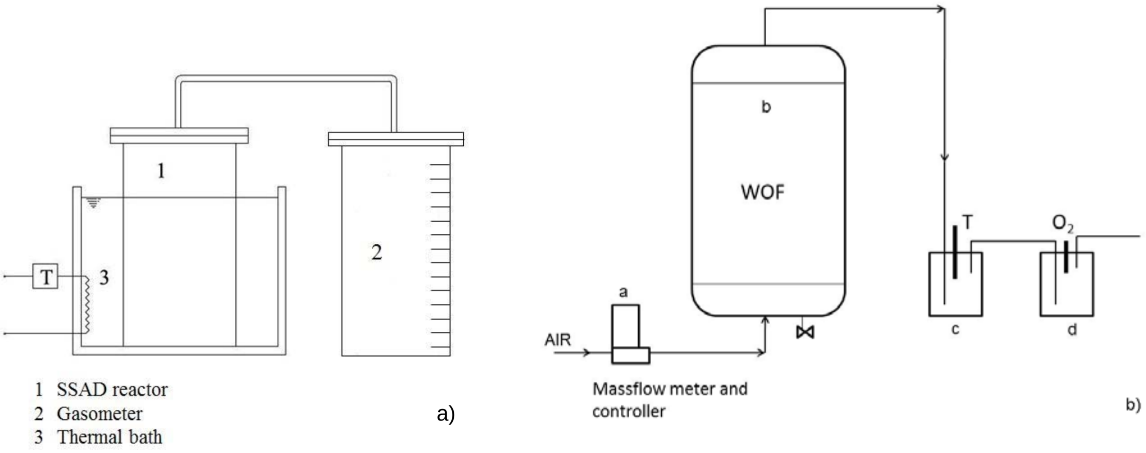

FIGURE 2 **Modified Binner and Zach test a) and PDRI apparatus apparatus b).**

TABLE 1 **WOF component concentration as withdrawn from the MBT.**

Component	**Value (% w/w)**
Plastics	3.90
Paper and Cardboard	1.30
OF	29.4
WEEE	0.10
Metals	0.50
Glass	4.20
20S	58.7
Others	1.90
Total	100

Further, stabilization can represent a reversible condition, by an increase of the H, or a quality of the material that is maintained and eventually increased during the years even in different operating conditions.
The level of the stabilization achieved highly influences the LFG production (Nm^3/tonne VS), the quality (i.e. CH_4 % v/v) and the rapidity of methanogenic phase start once anaerobic conditions are established starts in the landfill body.

All these aspect have to be investigate carefully for forecast in the most reliable way the expected emission from landfill and how these can be managed in the most suitable and sustainable way.

TABLE 2 **Physical characteristics of WOF with different days of pretreatment.**

Days of pretreatment	MC (%)	VS (% db)	pH
0	43.0	50.7	7.51
15	37.6	32.7	7.73
30	20.6	33.7	7.77
60	19.7	28.7	7.55
90	19.8	29	7.16
120(1)	33.9	29.2	7.63

(1)After 95 days added 18%w/w of water.
db = dry basis.

TABLE 3 **Characteristics of the Bio with different days of pretreatment.**

Days of pretreatment	MC (%)	VS (%db)	Corg (%db)
0	67.6	74.0	25.0
15	46.3	49.7	16.8
30	33.5	72.5	34.2
60	20.8	80.0	43.9
90	19.9	76.7	38.1
120(1)	42.7	70.5	28.7

(1)After 95 days added 18%w/w of water.
db = dry basis

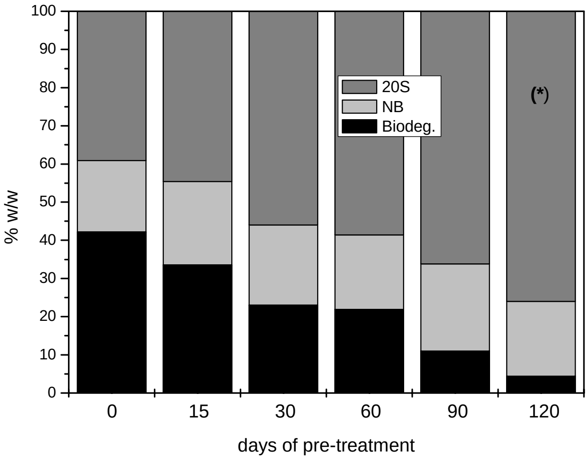

FIGURE 3 **NB, Bio and 20S fractions for the WOF samples with different days of aerobic treatment.**
(*) After 95 days added 18% w/w of water.

2 METHODOLOGY

2.1 Experimental apparatus

Different experimental apparatus have been exploited for simulating the aerobic and anaerobic processes of the WOF. The aerobic pretreatment process has been conduct by two bins, with a volume of 800 litres each one, modified by the creation of an aerated floor through which an electrical fan supplies about $1Nm^3/h$ of air per each bin. The temperature of the WOF under treatment has been monitored by two thermocouple inserted in the waste mass. At different days of aerobic pretreatment, 0, 15, 30, 60, 90 and 120 a given amount of WOF has been sampled from the bins, characterized and then processed in the other apparatus.

The anaerobic process occurring in bioreactor landfill have been simulated by a modified Binner and Zach (1999) test (Fig. 2a), exploiting a 12 litres volume reactor instead of the 1litres proposed. It has also been exploited for evaluating the LFG production of the different WOF samples. Before being inserted in the reactor the sample have been wetted for 24 h and then drained for 6 h for achieving the water-hold capacity. The reactor temperature has been kept at mesophilic conditions (35°C) by the aid of a thermal bath. The mean sample weight results of about 5 kg. No inoculums has been exploited for starting the process.

The WOF samples Biogasification Potential (BP) has been evaluated by an experimental apparatus similar to the one proposed by Trine et al. (2004), exploiting as inoculums the digestate arsing from laboratory run of anaerobic digestion of cow manure. BP test have been performed at mesophilic conditions. The Biogas and LFG methane concentration (%v/v) has been analysed by an infrared sensor (±2%). All the other gas components have been grouped in a balance term.

The stabilization level achieved by the WOF at different days of aerobic treatment has been evaluated both by the Potential Dynamic Respiration Index (PDRI) (Fig. 2b) (Scaglia et al., 2000) and by the GB test (Binner et al., 1999).

2.2 Sampling and characterization

About 500 kg of WOF have been withdrawn from the MBT facility before biological section inlet and manually analyzed for evaluating the weight of the different components (Table 1). The manually separated components have been mixed again before being inserted in the aerobic apparatus, about 250 kg each bin.

At 0, 15, 30, 60, 90 and 120 days of aerobic treatment, about 20 kg of WOF per each bin have been withdrawn for being tested and analyzed. The first analysis concerns the evaluation of three main components fractions as the Non Biodegradable (NB), the Biodegradable (Bio) and the fines (20S) (Fig. 3). Then MC of the sample withdrawn from the bins has been evaluated by mass loss after heating at 105°C for 24h. The solid residues arising from MC evaluation has been heated at 550°C for 24h. By evaluating the further mass loss the VS concentration has been determined. The pH has been evaluated on a sample diluted in demineralised water 1:10 and mixed for 30’ (Table 2).

The Bio fraction has been analyzed separately, after manual sorting for determining the MC, VS and organic Carbon (Corg) concentrations (Table 3). MC and VS have been determined by the same methodology described above whereas the Corg has been evaluated by the Springer and Klee wet dichromate oxidation method (Nelson and Sommers, 1996).

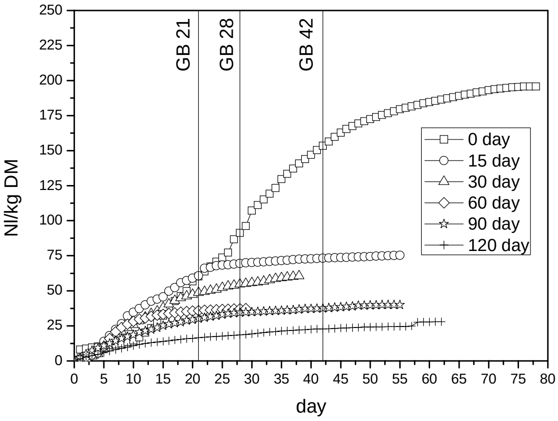

FIGURE 4 **Average GB for the sample with different days of pretreatment. (DM= Dry Matter)**

3 MAIN RESULTS

The WOF composition, as withdrawn from the MBT facility (Table 1), shows that OF and 20S represents together more than 85% w/w. Other Bio components are represented by paper and cardboard with about 1.3 % w/w. The remaining fraction is represented by biologically inert material among which the glass represents the larger fraction with about 4% w/w. The MSW sieving section shows a quite high efficiency in separating the DF from the WOF one.

The aerobic treatment has several effects on WOF feature. A longer period of aerobic process (Fig. 3) causes a reduction in the WOF sample Bio concentration, whereas the 20S concentration results increased. The bacterial activity transforms a fraction of the biodegradable components in to CO_2 and H_2O, reducing also the biodegradable material size and MC.

The NB fraction %w/w remains quite constant in coherence with the process. Similarly (Table 2) longer is the treatment periods lower is the VS concentration. The pH remains quite constant close to neutral value. The 120 days sample shows an increase in MC as a consequence of water addition (*i.e.* 18% w/w) for reducing inhibition effects caused by the low humidity content. Also the Bio component of the WOF sample shows a continuous reduction in MC even if the VS and Corg concentration have a fluctuant trend.

The stability evaluated by the fermentation test (Fig. 4) shows that the most suitable parameter for it representation is the GB42 (*i.e.* after 42 days of fermentation). This is a quite natural consequence of the inhibition phenomenon occurring during the first period of fermentation test for the sample with 0 days. Infact, the GB after 21 and 28 days for this sample results lower than the one achieved by samples with a longer aerobic pretreatment. The GB test results are susbstantially confirmed by the PDRI one (Fig. 5), even if the PDRI shows a less regular trend as the WOF aerobic pretreatment period rises (*i.e.* 30 and 60 days).

The first 15 days of aerobic pretreatment are the one causing the higher drop both in GB42 and in PDRI. Then the WOF stabilization proceeds more slowly achieving the stabilization level required for disposal in landfill (i.e. GB21≤20 Nl/kgDM and PDRI ≤ 1,000 mgO_2/kgVs h) after more than 90 days, and if some corrections are made to main process parameters as water addition for controlling inhibition phenomenon caused by a too low MC level.

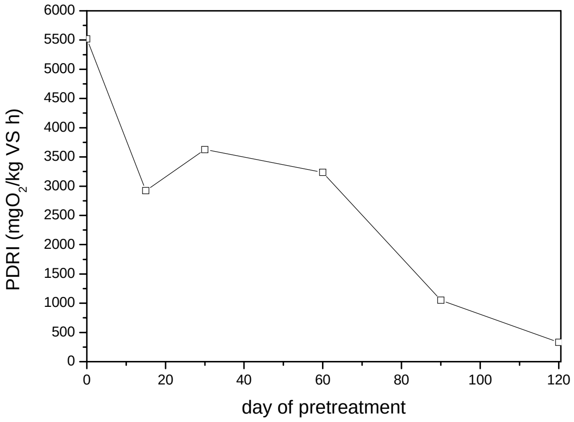

FIGURE 5 **PDRI value for the WOF samples with different days of pretreatment.**

The LFG production ranges from about 10 up to 60 Nl/kg (Fig. 6) being very influenced by the pretreatment period. As expected inhibition phenomenon limits the LFG production for the sample with 0 day whereas the maximum production is achieved by the sample with 90 days. This is also confirmed by the Biogasification Efficiency (BE), defined as the ratio between the LFG yield, expressed in this case as Nl/kgVS, and the BP of the same sample (Fig. 7). BE results quite null for the 0 day sample, ranging from 40 up to 65% for the other samples. For a better evaluation of the aerobic pretreatment effect on gas production this results have been normalized to the unit of VS of the sample with 0 days (VS_0) (Fig. 8). It is possible to see that the maximum LFG yield as Nl/kgVS_0 is achieved for the sample with a pretreatment period from 15 to 30 days.

The results show that the aerobic pretreatmen plays a relevant role in the anaerobic biodegradation process of the landfilled WOF. MC loss during aerobic process, along with VS, reduces the mass and the volume of the waste leading to an increase in landfill life. The sample with 90 days of aerobic pretreatment shows the higher LFG production (Nl/kgVS_0) even if the sample with 15 days shows the higher biomethane production (Nl/kgVS_0) (Fig. 8). On the basis of these results, if the main issue is to achieve high environmental protection, the longer is the stabilization process lower is the GHG emission potential. For renewable energy recovery issue, the optimum length of the aerobic pretreatment period is of about 15 days, able to reduce inhibition phenomena along with the preservation of a large concentration of non stabilized biodegradable substances. In this condition the mean CH_4 concentration in the LFG is higher than 45 % v/v achieving a peak higher than 55% v/v.

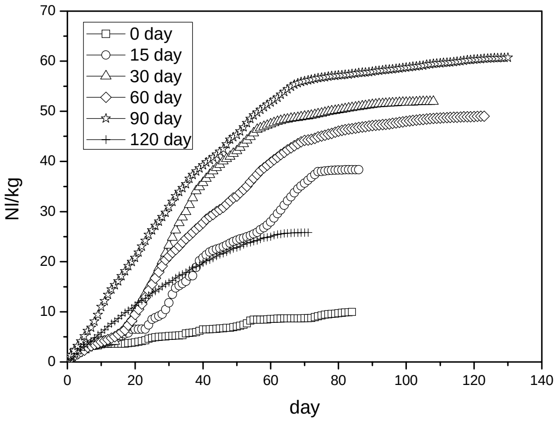

FIGURE 6 **Average LFG production for the WOF samples with different days of aerobic pretreatment.**

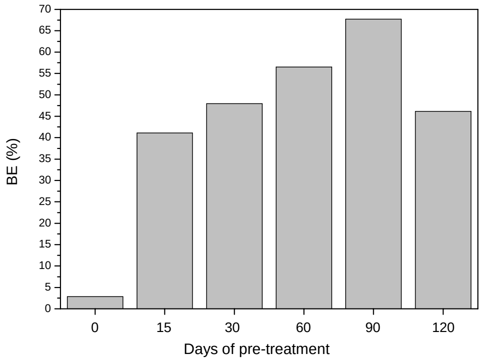

FIGURE 7 **BE for the WOF samples with different days of aerobic pretreatment.**

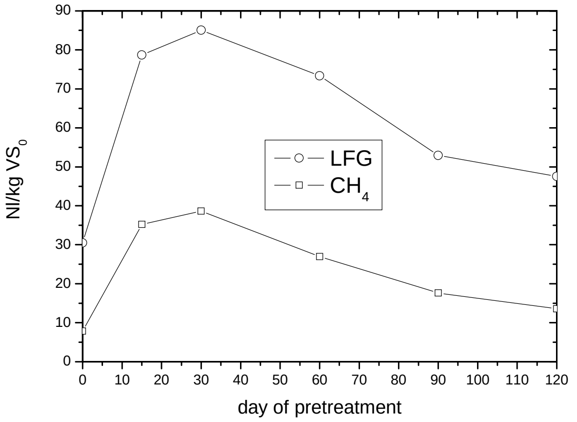

FIGURE 8 **LFG and biomethane yield related to the kgVS$_0$ for WOF with different days of aerobic pretreatment.**

4 CONCLUSIONS

Aerobic pretreatment of the Waste Organic Fraction produced by an existing MBT facility influences largely the anaerobic biodegradation once landfilled. The PDRI ranges from about 5,000 to 330 mgO$_2$/kgVSh respectively for the WOF sample with 0 and 120 days of aerobic pretreatment. Similarly the GB42 ranges from about 200 to 20 Nl/kgDM. The stabilization limits in terms of PDRI and GB are achieved for aerobic pretreatment higher than 90 days.

The landfill gas production ranges from 10 to 60 Nm^3/tonne, achieving its maximum value for the sample with 90 day of anaerobic pretreatment. If this result is normalized to the kgVS of the WOF without aerobic pretreatment, the maximum LFG production is of about 85 Nl/$kgVS_0$ for the sample with 30 days of aerobic pretreatment. Also the maximum CH_4 production, about 40 Nl/$kgVS_0$, is achieved for the same sample.

Maximum environemtnal protection can be achieved by minimizing landfill gas production after WOF aerobic pretreatmetn higher than 90 days whereas maximum energy recovery can be achieved with WOF with about 15 days of aerobic pretreatment.

5 ACKNOWLEDGEMENTS

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(23) EXPERIMENTAL ANALYSIS OF THE AEROBIC STABILIZATION EFFICIENCY OF AN EXISTING MBT PLANT

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EXECUTIVE SUMMARY

The management of the Municipal Solid Waste (MSW) has been strongly improved by the last Waste Framework Directive 2008/98/CE, by increasing the amount of waste materials that have to be reutilized, prepared for reutilization and/or recycled. In any case, even if these goals will be achieved, a large amount of residual MSW from the Source Segregated collection (SS) will need to be properly managed.

Incineration is a quite suitable way for managing these materials even if it is costly and not homogenously spread in the different European Areas. Another diffused solution is represented by Mechanical Biological Treatment (MBT) facilities that can lead to some advantages as producing and high quality Solid Recovered Fuels (SRF) or reducing and stabilizing the mass of the waste to be disposed off. In particular, this last solution was the first exploited in MSW management since many years ago. Currently many of these plants operate in quite different conditions related to the design one. The difference arises from the variation both in waste management strategies and in the waste rates treated. These differences can influence both the mechanical and the Biological Treatment (BT) sections of the facility leading tom some modifications in plant efficiency. The present study analyses the BT section of an existing MBT facility operating the aerobic stabilization of the Waste Organic Fraction (WOF) produced by the mechanical screening of the MSW resulting from SS collection. This BT section is a continuous flow composting plant with an aerated flow through which electrical fans provides the process air. The continuous flow is performed by a crane bridge with screw that moves on the basin area. The screws provide to stir and move ahead the WOF from the inlet to the outlet section.

The analysis has been focused on the evaluation of the main WOF physical and biological activity features during different days of the treatment period. Different sampling points have been fixed on the basin area corresponding to specific mean days of treatment. The stability achieved by the WOF in the different sampling points has been evaluated by a Dynamic Respirometer Index (DRI) apparatus developed by the LAR^n of the University of Perugia, able to evaluate the oxygen uptake rate (mgO_2/kgVSh) of the material.

Main results show that the screws has a relevant influence on some process parameters as temperature, Volatile Solids and Dynamic Respirometer Index potential. The continuous mixing effect combined with the continuous WOF introduction in the basin, produces a temperature profile that in some cases is quite constant during the whole basin length. Similarly for the VS concentration that seems not to vary significantly from the inlet to the outlet section.

The high temperature level profiles combined with the amount of air injected beneath the WOF bed, causes a rapid humidity reduction in the first days of the process that can lead to an inhibition of the aerobic bacteria activity. This is confirmed by the VS concentration but also by the DRI values.

The DRI potential of the WOF at the basin inlet is practically reduced in the first 25-50% meters of the basin length, remaining quite constant in the remaining part.

Some improvement concerning process air regulation along with the possibility of increasing the WOF humidity if becomes to low, can lead to improve the stabilization process efficiency together with a reduction of the global impact of the MBT process.

1 INTRODUCTION

The management of the Municipal Solid Waste (MSW) has been strongly improved by the last Waste Framework Directive 2008/98/CE, by increasing the amount of waste materials that have to be reutilized, prepared for reutilization and/or recycled. In any case, even if these goals will be achieved, a large amount of residual MSW from the Source Segregated collection (SS) will need to be properly managed.

Incineration is a quite suitable way for managing these materials even if it is costly and not homogenously spread in the different European Areas. Another diffused solution is represented by Mechanical Biological Treatment (MBT) facilities that can lead to some advantages as producing and high quality Solid Recovered Fuels (SRF) or reducing and stabilizing the mass of the waste to be disposed off. In particular, this last solution was the first exploited in MSW management since many years ago. Currently many of these plants operate in quite different conditions related to the design one. The difference arises from the variation both in waste management strategies and in the waste rates treated. These differences can influence both the mechanical and the Biological Treatment (BT) sections of the facility leading tom some modifications in plant efficiency. In the following the BT operating conditions of an existing MBT plant working since 94ies has been experimentally evaluated. Waste physical characteristics and stabilization during aerobic BT process has been also analyzed.

1.1 Background

Mechanical Biological Treatment plants are quite diffused in the treatment of the Municipal Solid Waste (MSW) resulting from Source Segregated collection and can operate in two main ways:

1) Maximizing Solid Recovered Fuel from MSW;
2) Maximizing MSW mass reduction and stability before landfilling.

The first approach is pursued by the Mechanical Stabilate Method (MBS) (Wiemer and Kern, 1995) and is mainly base on a biodrying process exploiting the heat realised by the organic fraction during aerobic treatment (Tambone et al., 2011). As a consequence of temperature increase, the process air that flows through the MSW extract efficiently the humidity from the waste leading to an increase of the Net Calorific Value (NCV) (kj/kg) of the material. After this treatment, the biodryied waste can be further refined for reducing impurities and inert content for producing a high quality SRF that can be successfully exploited in co-combustion plants (Friege and Fendel, 2011).

The second approach aims to reduce and stabilize the mass of MSW to be landfilled. This goal is pursued by processing the MSW in two main plant sections. A first section consisting in mechanical and physical screening devices that allows to recover the metals and to separate the inlet waste stream in two main outlet streams. A Dry Fraction (DF) mainly rich of plastics, paper and cardboard, textile, wood, and a Waste Organic Fraction (WOF), mainly rich of Organic Fraction (OF) and fines (20S) (*i.e.* particle <20mm). The DF can be further refined for other recovery aims, included energy recovery, whereas WOF is transported to the Biological Treatment section. In the BT it is usually aerobically treated for reducing the mass and the reactivity of this waste before being disposed off (Bezema et al. 2007; Binner and Zach, 1999; De Gioannis et al, 2009; Di Maria and Valentini, 2011; Komils et al., 1999). Stabilization represents a quality of the material that is kept during time even if the physical and environmental conditions changes. In particular stabilized waste reduces significantly gaseous and liquid pollutant emission once landfilled reducing significantly the impact of this plant on the environment. Further, also landfill management costs can be significantly reduced, in particular during after closure management, if the waste achieves a high level of stabilization.

The MBT facility analiyzed in this work is of the second types and processes about 70,000 tonnes per year of MSW. The waste is firstly discharged by the collection lorries in the inlet section from which are moved into a bag opener. From the bag opener the MSW stream enters the mechanical sorting section, consisting mainly of trommels and metals separation devices, that splits the inlet stream in two main outlet streams, DF and WOF. The DF can be further refined for material recovery aims, included SRF production, whereas the WOF stream is moved to the BT section. This section consist of a concrete basin, with an aerated floor through which electrical fans delivers the air rate required by the aerobic biological process (Fig. 1). On the basin works a crane bridge with screws providing to stir and move the WOF from the inlet section to the outlet fulfilling a continuous stream of waste (Fig. 1b). During the last 15 years many changes have been occurred in the waste management system in which the MBT operates. These change concerns both the amount and the quality of MSW collected, affecting the mechanical and the BT process efficiency.

1.2 Research objectives

Since the 94, when the considered MBT starts operating, many changes have been occurred in the amount and in the composition of the MSW and consequently in the WOF processed by the plant. This can have different effect both on the mechanical and on the biological treatment efficiency and effectiveness.

For trying to improve plant management and to plan possible upgrading options, it is necessary to achieve a deep knowledge of the current performances of the MBT.

In this work the attention has been focused mainly on the BT section even if some consideration on the mechanical section have to be made for evaluating the amount and the composition of the WOF.

Due to the continuous stream flowing through the aerobic basin, the analysis of the only inlet and outlet WOF features is not sufficient for understanding correctly the whole biological process. At a give position inside the BT basin corresponds a given number of days of treatment at which corresponds specific characteristics of the waste. For this reason it is necessary to follow the WOF evolution by withdrawing WOF samples at given position in the basin. In this way it is possible to achieve information about the profiles of the main process parameter along the basin length (*i.e.* days of treatment) that can help in taking the decisions on how improve the current situation.

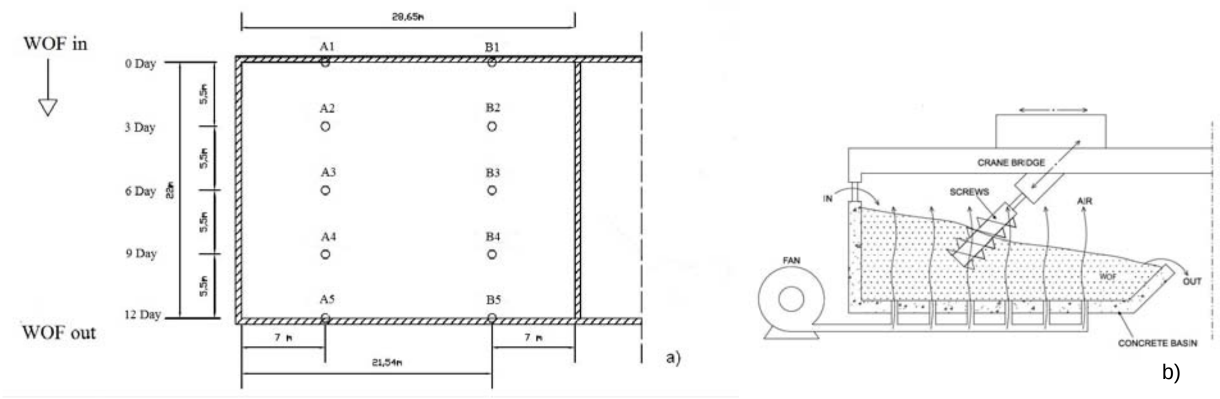

FIGURE 1 **MBT Biological Treatment section basin schematic, plant with sampling points a), section b).**

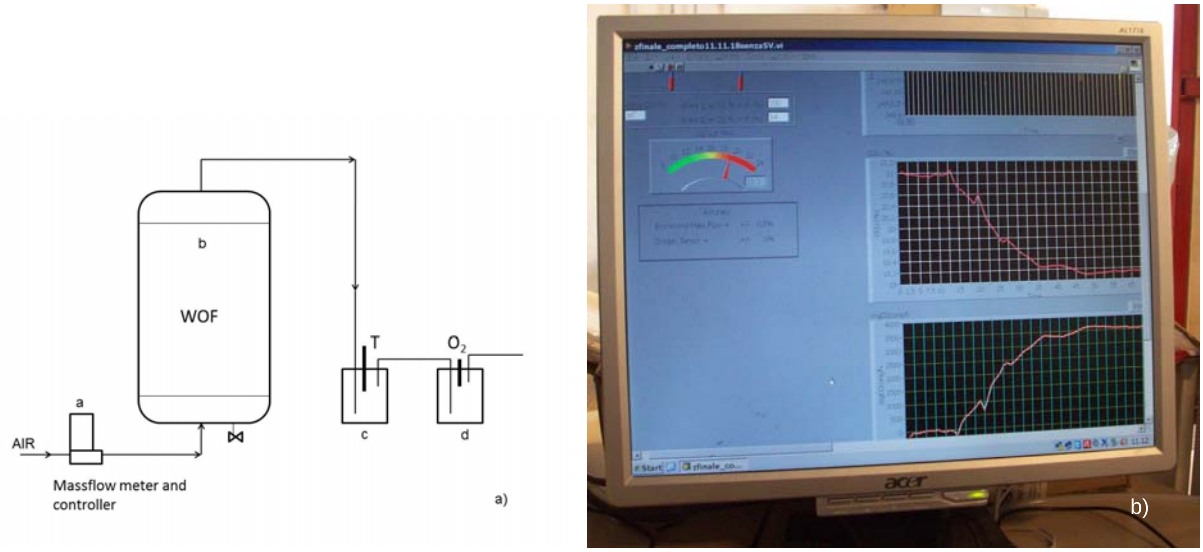

FIGURE 2 **PDRI experimental apparatus schematic a) and control panel b) developed by LAR^n.**

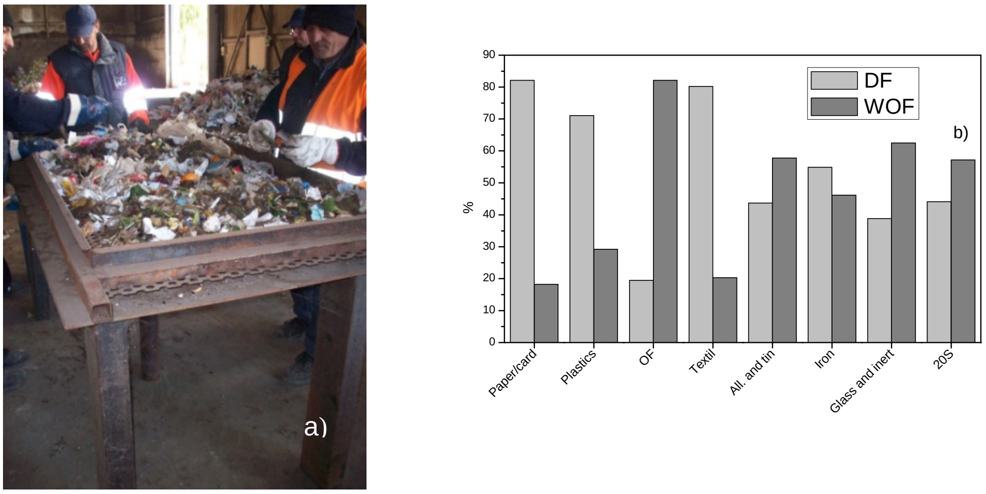

FIGURE 3 **DF and WOF compostion at the MBT mechanical section outlet, manual segregation a), components concentration b).**

2 METHODOLOGY

2.1 Experimental apparatus

The evaluation of the stability achieved by the WOF at different positions along the basin length has been determined by the Dynamic Respirometer Index (Scaglia et al., 2000) performed by an experimental apparatus developed by the LAR^n laboratory of the University of Perugia (Fig. 2a).

The apparatus has been developed according to the UNI/TS 11184-2006 along with a control system implemented by LabView NI (Fig. 2b). Oxygen uptake, internal and external temperatures are continuously monitored along with the air mass rate entering the respirometer. Minimum oxygen concentration in the exhaust air has been imposed at 16% v/v during all the runs. The inlet air rate regulation, if require, is performed by a similar PID logic based on the prevision of the expected oxygen consumption rate. In this way the mass rate regulation is changed dynamically instead of the fixed steps imposed by other commercial apparatus. The respirometer body has been realized in AISI 304 steel for the higher mechanical restistance and for being able to achieve a higher gastight level among the different device components. Infact, for a more correct measure, it is important an absence of external air leakage but also constrain the inlet air flow to pass entirely through the WOF sample avoiding internal leakage. This aim is more difficult to be efficiently and effectively achieved by plastic structures instead of steel ones.

2.2 Sampling and characterization

The sampling points on the whole basin surface have been determinate by the creation of a virtual grid. The grid is composed of two vertical and five horizontal lines (Fig. 1a). The intersection of these lines determines two series of five sampling points A_i and B_i, with i ranging from 1 to 5. At each point two samples of about 15 kg each one have been withdrawn, $A_{i,j}$ and $B_{i,j}$, with j ranging from 1 to 2, at a deep of about 0.5m starting from the surfaces of the WOF bed. For a better comprehension of the process evolution, sampling operation has been synchronized by the mean waste retention time inside the basin (*i.e.* about 14 days).

The sample humidity (H) has been determinated by weight loss after heating at 105°C for 24h. Volatile Solids (VS) concentration has been evaluated by heating at 550°C for 24 h the dry residues (TS) of the H test. The temperature in the sampling points has been manually measured by a thermocouple about 1 m deep related to the WOF surface. In the same points the exhaust air quality has been measured by a portable gas analyzer measuring the gas composition %v/v. CH_4 and CO_2 concentrations are measured by infrared sensors whereas the O_2 and H_2S concentrations are measure by electrochemical cells.

A given amount of the withdrawn samples, about 10kg, has been wetted until achieving the 75% w/w of water hold capacity, according to UNI/TS 11184-2006 procedure, and then inserted in the DRI apparatus for the evaluation of the oxygen uptake ($mgO_2/kgVSh$).

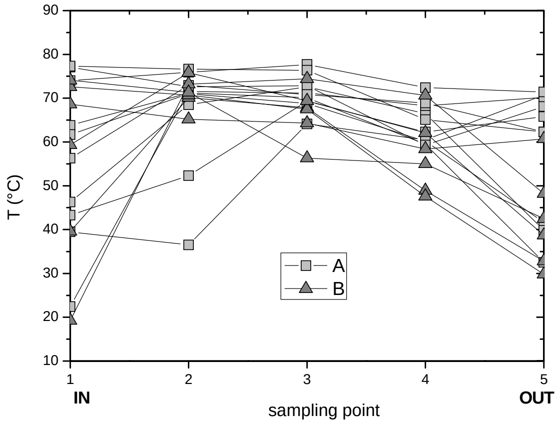

FIGURE 4 **Temperature profiles for the different basin sampling points.**

3 MAIN RESULTS

The evaluation of the screening efficiency of the mechanical section of the MBT has been performed by several mass balances and composition analysis, by manual segregation (Fig. 3a) of the DF and WOF at this section outlet.

The WOF represents meanly about the 49% w/w of the whole MSW treated, and it is mainly composed of OF and 20S (i.e. particles with <20mm size) (Fig. 3b).

The mean amount of air supplied by the electrical fans at the beneath of the basin is of about 4Nm^3 per each kg of VS at basin inlet. Globally this corresponds to about 1,100 Nm^3 per each tonne of WOF treated. The temperature profiles measured during the sampling operations at different sampling points shows (Fig. 4) that the maximum values is achieved at the middle of the basin length, corresponding to about half of the retention period (*i.e.* 6 days).

In some cases temperature is quite constant with a slight reduction from the inlet to the outlet section whereas in other cases there is a rapid grow in the first 5.5 meter followed by a decrease in the last 11 meters for achieving values similar to the inlet ones. This particular phenomenon is a consequence of the stirring and moving effect produced by the screws that moves forward the WOF causing a mixing effect among WOF with different days of treatment.

The expected temperature profile, with a minimum values for the sampling points $A_{1,j}$ and $B_{1,j}$, a maximum around the $A_{2,j}$ and $B_{2,j}$ position followed by a constant decrease in the points $A_{3,j}$-$A_{4,j}$ and $B_{3,j}$-$B_{4,j}$, can be subverted. Depending on the instant at which the new WOF has been charged and at which the screws transits on the analyzed basin stripe, along with other operating conditions, temperature profiles can be significantly influenced.

A similar effect can be found in the profile of the VS (Fig. 5). The stirring and mixing effect of the screws leads to a quite constant value of the VS concentration from the inlet and outlet section in spite of the expected reduction.

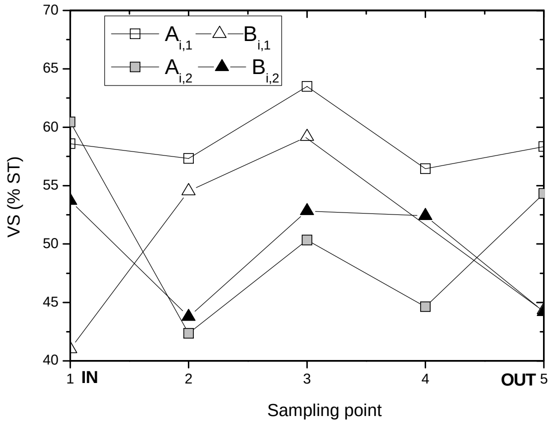

FIGURE 5 **VS profiles for the different sampling points.**

Another relevant phenomenon that can contribute to inhibiting the VS reduction along the basin length is represent by the strong H reduction (Fig. 6). Mean H value at basin inlet ranges from about 40 up to 55% w/w and is rapidly decreased in the first 11 meters (*i.e.* sampling point 3). The relevant H reduction is a consequence of the combined effect of temperature levels and of the air rate flowing through the WOF under treatment. When H becomes lower than 40% w/w, biological activity results inhibited causing a slight further reduction of VS content.

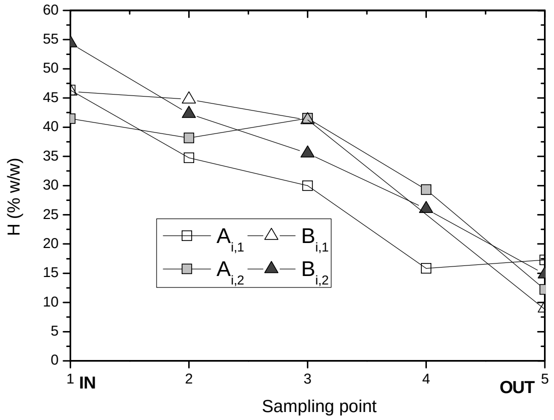

FIGURE 6 **H profile for the different sampling points.**

The amount of air supplied by the fans results able to provide an oxygen concentration in the exhaust gas higher than 15 % w/w. This avoids the risk of anaerobic conditions formation as confirmed by the CO_2 concentration that corresponds to the reduced O_2 fraction %v/v (Fig. 7).

The stabilization achieved by the WOF during the aerobic process (Fig. 8), shows that the DIR potential ranges from about 3,800 $mgO_2/kgVSh$, at the inlet section, to about 2,100 $mgO_2/kgVSh$ at the outlet section. The experimental results shows that the inlet WOF has a very variable DRI and the minimum value is generally achieved in the first half of the basin length, instead of the basin outlet. This is a consequence of the afore described phenomena including screws, delay between crane bridge transit and fresh WOF introduction and H loss. Meanly the BT section is able to reduce the bacterial activity expressed as oxygen uptake per kg VS per h, of about 45% compared to the one of the inlet material. The minimum DRI potential of about 1,500 $mgO_2/kgVS$ h at basin outlet has been achieved for the sample $A_{5,1}$.

On the basis of these results the BT section shows to have the potential for treat successfully the yearly WOF rate (tonne/year) even if some improvement seem necessary for increase the process efficiency and effectiveness. The air rate supplied by the fans results adequate in the first part of the basin and almost high in the second one. Anyway this aspect is difficult to improve considering the structural features of the basin. This can be a consequence of the inhibition phenomenon caused by the strong H reduction that leads to a significant decline of the oxygen uptake. As consequence, this limits also the stabilization process rapidity as results from the DRI analysis. A possible solution for trying to increase the stabilization efficiency, is represented by the addition of water for bring the H concentration back to acceptable values to comply with a quite efficient biological activity. The water addition has to be made around the half of the basin length in a quantity of about 8% w/w. The effect of this modification is currently under investigation.

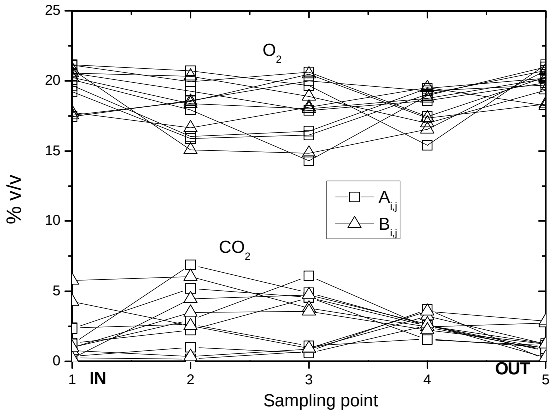

FIGURE 7 **Oxygen and Carbon dioxide concentration of the exhaust air in the different sampling points.**

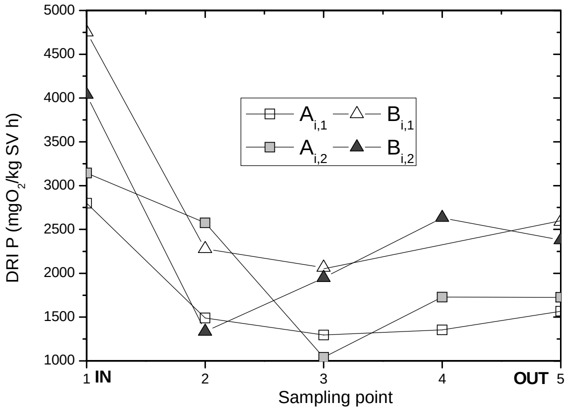

FIGURE 8 **DRI potential for the different sampling point.**

4 CONCLUSIONS

The results of the experimental analysis performed on the continuous flow aerobic Biological Treatment section of an existing Mechanical Biological Treatment plant show many particular aspects affecting both the section efficiency and the biological process evolution. The stirring and moving effects caused by the screws of the crane bridge on the processed Waste Organic Fraction plays a relevant role in the whole process development. A first consequence can be found on the mean temperature profiles. The maximum temperature values are generally achieved at the half of the basin length, i.e. after about 6 days of treatment. Further, generally high temperature values are maintained until the basin outlet. Similarly the VS content remains quite constant during the process. The rapid humidity reduction during the first half length of the basin, more than 50% of the inlet one, causes inhibition of the bacterial activity, leading to a substantial interruption of the stabilization process as measured by the oxygen uptake. Infact, the Dynamic Respirometer Index drops significantly in the first 5-10 meters of the basin length, maintaining a quite constant value in the remaining length. The basin seems to be able to process and stabilize the whole amount of WOF yearly treated even if some improvement for biological process control have to be realized. Increasing waste humidity content, when necessary, along with an optimization of air delivery, can have positive effects both on stabilization and energy efficiency of the MBT plant.

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(74) MECHANICAL BIOLOGICAL TREATMENT OF MUNICIPAL WASTE: IMPACT OF RETENTION TIME OF THE ROTARY DRUM REACTOR

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EXECUTIVE SUMMARY

Mechanical Biological Treatment (MBT) is a proven technology for the treatment of municipal solid waste (MSW). The number of MBT plants is increasing significantly in recent years. In a significant number of these plants, the three major elements are a Rotary Drum Reactor (RDR) for preparation of the waste, an anaerobic digestion system for organic matter degradation and energy recovery followed by a composting step for organic matter stabilisation. Although industrial feedback exists, there is a lack of scientific results at industrial scale. For that reason, Veolia Environnement Recherche et Innovation (VERI), with the support of the Agence de l'Environnement et de la Maîtrise de l'Energie (ADEME), carried out a research project to evaluate the performance of an MBT of residual municipal solid waste (rMSW) including a 24-m RDR from the VEOLIA-operated industrial site of Champagne sur Oise, the VERI semi-industrial (21m^3) dry anaerobic digester at Graincourt les Havrincourt and the VERI pilot-scale (160L) composting reactors at Limay. The objective of the project was to evaluate the impact of the retention time of the RDR, which varied between 1.5 and 4 days, on the performance of the biogas production in the mesophilic anaerobic digestion, the compostability of the digestate and the agronomic value of the final product.

Two three-month experiments were conducted considering the two retention times (1.5 and 4 days) of the RDR. The incoming rMSW and both flows of the screen at 40 mm following the RDR were characterized using the MODECOM^{TM} (MéthODE de Caractérisation des Ordures Ménagères, 1993) methodology and physical and biochemical parameters in order to determine the performance of the equipment. The comparison between the composition of the rMSW used during each test and the average composition of the French rMSW shows that the material used during both tests could be considered as classical rMSW. No real impact on the quantity of Volatile Solids (VS) was observed whereas a modification of the particle size was pointed up.

Weekly three tons of the below 40 mm fraction were fed into the anaerobic digester. It was operated at 35°C and 30 days of retention time. The anaerobic digestion performance was evaluated in terms of biogas production and quality and also in terms of process stability (NH_4^+, VFA, TAC, and pH). In terms of biogas production, the results obtained show that, during both experiments, around 65 % of the Biochemical Methane Potential (BMP) of the feed was expressed in the reactor. There was no significant difference between both experiments indicating that there was no significant impact of the retention time in the RDR on the amount of organic matter made available for anaerobic digestion.

In both experiments, the digestate was analyzed in order to determine its ability to be composted in terms of organic matter availability and simultaneously composting test were conducted in order to evaluate the agronomic value of the stabilised final product. The dehydrated digestate was composted alone. The results showed low VS degradation due to the degradation of most of the VS during anaerobic digestion. Nevertheless a VS stability difference seems to exist between the two experiments maybe due to a longer retention time in RDR for the second experiment.

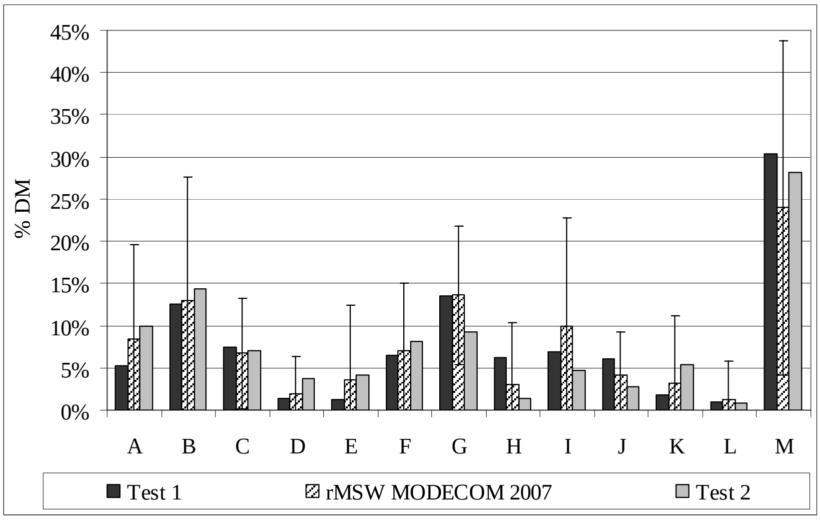
1. INTRODUCTION

Waste management is a priority in Europe. According to the EU Directive 1999/31/EC, the biodegradable Municipal Solid Waste (MSW) fraction allocated in landfills has to be reduced to 35 % (in weight) of the quantity produced in 1995 at the latest in 2017. As a response to this directive the number of Mechanical Biological Treatment (MBT) plants is increasing significantly in recent years in France. The three major elements on these plants are a Rotary Drum Reactor (RDR) for preparation of the waste, an anaerobic digestion system for Volatile Solids (VS) degradation and energy recovery followed by a composting step for VS stabilisation. Although industrial feedback exists, there is a lack of scientific results at industrial scale. For that reason, Veolia Environnement Recherche et Innovation (VERI) carried out, with the support of the Agence de l'Environnement et de la Maîtrise de l'Energie (ADEME), a research project to evaluate the performance of a MBT process on residual Municipal Solid Waste (rMSW) including a RDR, a semi-industrial dry anaerobic digester and pilot-scale composting reactors. The main objective of the project was to evaluate the impact of the RDR retention time (1.5 day and 4 days) on the performance of the biogas production in the mesophilic anaerobic digestion, the digestate compostability and the agronomic value of the final product.

2. METHODOLOGY

2.1 Residual Municipal Solid Waste

The residual Municipal Solid Waste (rMSW) used during both tests was obtained after separating glass, plastic, cardboard, cans and paper by selective collection. A MODECOM^{TM} characterisation (see page 4: MODECOM^{TM}) on dry basis was conducted for each test. The obtained results and their comparison to the French rMSW (MODECOM^{TM} 2007) are presented in Figure 1.



A	Green and food waste
B	Paper
C	Cardboard
D	Composite material
E	Textile
F	Sanitary textile
G	Plastic
H	Other combustible
I	Glass
J	Metals
K	Other noncombustible
L	Dangerous waste
M	Very small < 20 mm

Figure 1 : Comparison of the composition between used-rMSW and the French classical rMSW (MODECOMTM 2007) in DM (Test 1: rMSW composition before 1.5 day pre-treatment in the RDR; Test 2: rMSW composition before 4 days pre-treatment in the RDR)

The rMSW used in both tests is comparable to the “average” French rMSW (MODECOM^{TM} 2007). It is important to notice the important range observed for each category due to the heterogeneity of the rMSW. For example, the composition of green and food waste is double from one test to another and still included in the variation of the MODECOM.

In addition, the used rMSW had more than 50 % of fermentable material (52 % and 55 % for test 1 and 2 respectively) which is green and food waste, paper, cardboard, composite, sanitary textile, and the organic fraction of the very small (< 20 mm). Therefore, the used waste seems to be interesting for biological processes.

2.2 Rotary Drum Reactor and Screening

A Rotary Drum Reactor (RDR) is a rotating cylinder with variable length and diameter (length > 20 m, diameter > 3) with an internal surface protected against corrosion. Knifes are disposed on the internal surface in order to open bags which contain MSW. This equipment is usually used as a pre-treatment in MSW biological treatment processes.

The RDR used, in the current study, was 24 m long and of 3.5 m diameter. The rotation speed was set at 1 rpm during day-time and 0.5 rpm at night-time. RDR are mostly operated with addition of both air and water.

Nevertheless, during this study, the RDR was operated without addition of either air or water due to a faulty material for aeration and enough-wet rMSW.

The RDR outflow was directly screened at 40-mm: the <40 mm fraction was kept and used in the anaerobic digestion process and the >40 mm fraction was eliminated.

2.3 Anaerobic digestion

The anaerobic digestion of the <40 mm fraction from both tests was performed on a 21 m^3 semi-industrial pilot plant located in the North of France. A detailed description of the pilot plant can be found in Lemaire *et al.* (2005).

Operational parameters

The dry anaerobic digestion was operated under the following main operational conditions:

- The operational volume in the digester was maintained around 14 m^3.
- The DM content of the feed was adjusted to 40 % with water before feeding the reactor.
- The system was operated at an average Hydraulic Retention Time (HRT) of 30 days.
- Each experiment was conducted during three months that represent three HRT: the first one in order to achieve stability of new operational conditions, the last two in order to obtain representative results.
- Three tons of <40 mm RDR output rMSW were weekly “semi-continuously” fed (Monday, Wednesday and Friday).
- The average Organic Loading Rate (OLR) was about 10.2 kg VS/m3/day.
- The temperature was kept constant under mesophilic conditions, 35°C, for both tests.

Several parameters (pH, VFA/TAC ratio and ammonia content), see Figure 2, were followed during both experiments in order to evaluate process stability. The daily follow-up of those parameters, presented in Figure 2, proves the biologically stability of the system for both tests. In fact, the pH of the digestate remained between 7.0 and 8.0 (with an average of 7.5 ± 0.2 for test 1 and 7.2 ± 0.1 for test 2), which are the favourable conditions for micro-organisms development (Ananthanarayan and Jayaram Paniker, 1978). The VFA/TAC ratio (VFA in gCH_3COOH/kg of sample and TAC in $gCaCO_3$/kg of sample) was stable and constantly below 0.8 (average of 0.3 ± 0.1 for both tests) indicating a great stability of the system (Ehrig, 1983). In addition, with an average ammonia concentration of 0.9 ± 0.3 $gN\text{-}NH_4^+$/L for test 1 and 0.5 ± 0.1 $gN\text{-}NH_4^+$/L for test 2, the system was clearly below the ammonia inhibition concentration (Grady *et al.*, 1999).

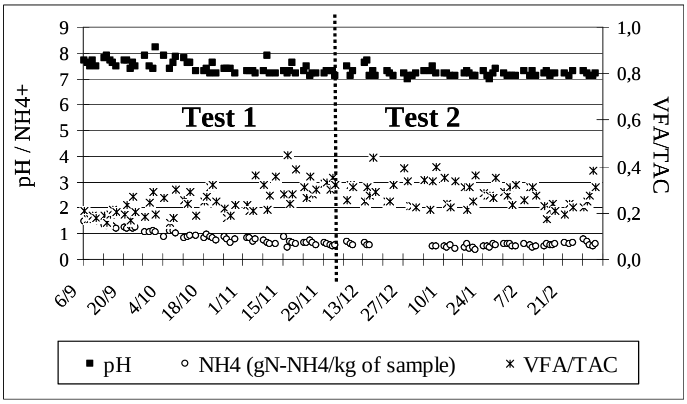


Figure 2: Daily follow-up of pH, ammonia and VFA/TAC ratio during both tests

2.4 Composting

This step consisted in composting the dehydrated digestates from anaerobic digestion. Dehydrated digestates were obtained by pressing digestates. Tests were performed in 160 L, closed and insulated reactors (Bacheley *et al.*, 2008). The first experiment was done with dehydrated digestate obtained from rMSW that spent 1.5 day in the RDR (test 1) and the second one with rMSW that spent 4 days in the RDR (test 2). Each experiment lasted 4 weeks. The temperature and O_2 concentration were continuously monitored. Several analyses were made at the beginning of composting (t_0) and after the 4 weeks of composting (t_4) (see 2.6).

2.5 Technical aspect to get samples - Quartering method

To divide a big sample in a smaller one (easier to study, to move…) and still representative: the whole sample was first mixed and divided in four equal parts, then two of them were kept (the two other eliminated) and mixed again. These operations were repeated until the wanted final size of the sample is obtained.

2.6 Analysis

- **The Dry Matter (DM) content** was analysed according to the French Standard NF U 44-171 by drying at 105°C. The sample DM content is expressed in percentage of Fresh Matter (FM).
- **The Volatile Solids (VS) or Organic Matter (OM) content** was measured according to the French Standard NF EN 13-039 by drying at 550°C during 4 hours at least. The sample VS content is expressed in percentage of FM.
- **The MODECOM characterisation** was conducted according to the French Standard AFNOR XP X 30-466 on the dried sample. The sample was divided in three particles sizes: > 100 mm, 20-100 mm and < 20 mm. The > 100 mm and 20-100 mm fractions were divided in 12 categories: green and food waste, paper, cardboard, composite material, textile, sanitary textile, plastic, other combustible, glass, metals, other non-combustible and dangerous waste. The sample characterisation is expressed in percentage of each category on DM.
- **The pH** was measured according to the French Standard NF EN 13-037 after calibration with buffer solutions and sample dilution with demineralised water.
- **Total Alkalinity (TAC) and total Volatile Fatty Acids (VFA) contents** were measured by titration following a VERI internal method (inspired by Anderson and Lang, 1992). The result is a concentration of basic species (TAC) and a concentration of total VFA.
- **Ammonium content (N-NH_4^+)** was measured using a two-steps method that includes a KCl extraction and the Berthelot colorimetric titration.
- **The Biochemical Methane Potential (BMP)** was measured in batch anaerobic digestion tests with inoculum, nutrients and buffer solutions. The objective was to get the highest biogas production from the sample (detailed of the INRA laboratory internal method was not given).
- **Van Sœst** analysis was conducted according to the French Standard XP U 44-162 consisting in successive fractions extraction using increasingly intensity extractants. The objective of this analysis is to characterize the organic matter in four biochemical fractions: soluble, hemicellulose, cellulose and lignin (considered from the most biodegradable to the least). Each fraction is expressed in percent of VS.
- **Carbon mineralization in 3 days (Cm3)** was conducted according to the French Standard XP U 44-163. The sample is mixed with soil and CO_2 emissions are recovered in a basic solution and then determined by titration.
- **The Indicator of Residual Organic Carbon (IROC)** was measured according to the French Standard XP U 44-162. The IROC traduces the stability of the material, ranging from 0 (less stable) to 1 (more stable). It was calculated using the following expression:

IROC = 445 + 0.5 x %Soluble - 0.2 x %Cellulose + 0.7 x %Lignin - 2.3 x Cm3.

3. RESULTS AND DISCUSSION

3.1 Rotary Drum Reactor

The main impact of the pre-treatment in the RDR was on the particles size. Figure 3 presents the evolution of the particle size in % of input DM.

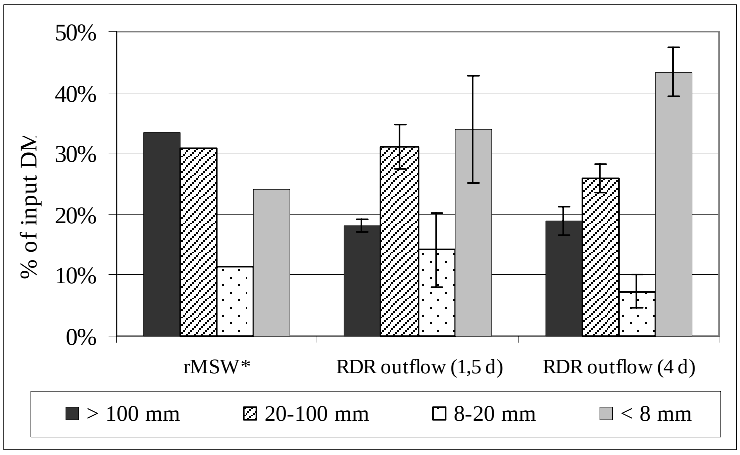


Figure 3: Evolution of the particles size of rMSW in % of input DM
****Only one characterisation had been done for rMSW***

The particles size reduction occurred in two steps:

- 1st step, 0 - 1.5 days (test 1): the >100 mm fraction decreases by half (from 34% to 18 %) which corresponds to an increase of other smaller fractions (especially those <20 mm: from 11 % to 14 % for the 8-20 mm fraction and from 24 % to 34 % for the <8 mm fraction)
- 2nd step, 1.5 - 4 days (test 2): the 8-100 mm fractions decrease (from 31 % to 26 % for the 20-100 mm fraction and from 14 % to 7 % for the 8-20 mm fraction) which corresponds to another increase of < 8 mm fraction (from 34 % to 43 %).

This RDR particle size evolution trend has already been observed by Crepel *et al.* (2009). However, the observed particle size reduction in the current study was less important for a rMSW treated in a RDR for 4 days (the particle size distribution after 4 days in the RDR was the same in both studies, although the >100 mm fraction represented only 34 % in the rMSW used in the current study and 50 % in the study of Crepel *et al*., 2009).

Another important aspect was to evaluate the impact of the RDR on the organic matter. No significant degradation of organic matter was observed at any retention time on the RDR. On the contrary, Crepel *et al.* (2009) observed 2% organic matter loss after 1 day in the RDR and 6% after 4 days. These different observations could be explained by the operational conditions of the RDR during the current study (no air and water addition in the tube).

3.2 Anaerobic Digestion

Once the biological stability has been checked for both experiments (see section 2.3), it is possible to evaluate and compare the anaerobic digestion process performance for each test. The observed average weekly methane production (in Nm^3/t VS fed) for both tests is presented on Figure 4. It is compared to the methane production obtained during the mesophilic anaerobic digestion of non RDR pre-treated rMSW (crushed and screened at 40 mm) (Bellegarde *et al.*, 2011). The average measured BMP values for the input material used in each test were 278 ± 85 Nm^3/t VS, 305 ± 56 Nm^3/t VS and 237 ± 32 Nm^3/t VS for test 1 and 2 of the current study and for the study conducted without RDR respectively. The difference between the BMP values obtained for a crushed rMSW and the rMSW pre-treated in the RDR could be explain by the different plastic content of the <40mm fraction in each case. In the case of the crushed rMSW, a more important fraction of plastics goes to the <40mm fraction due to the crushing. While in the case of the rMSW pre-treated by RDR, the fraction of plastics introduce in the <40mm fraction by the effect of the RDR is smaller. Therefore the organic matter could be the same but the anaerobic biodegradability is lower in the case of the crushed rMSW.

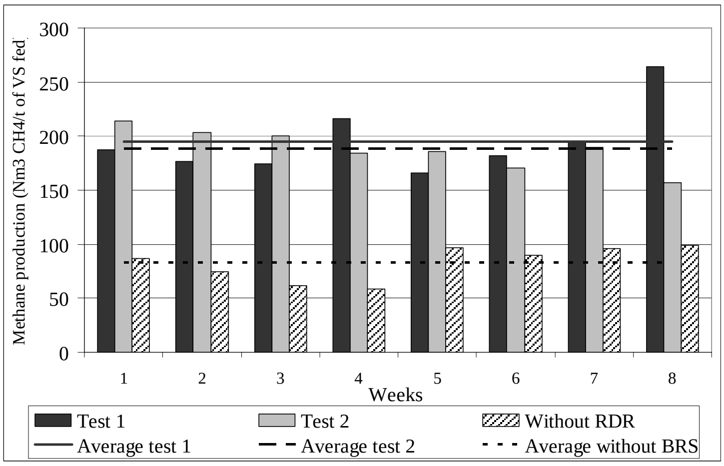


Figure 4: Methane production per organic matter fed and per week

A statistical analysis (student test with a risk of 5%) of the average methane production for both tests (195 ± 32 Nm^3 CH_4/t of VS fed for test 1 and 188 ± 18 Nm^3 CH_4/t of VS fed for test 2) showed no significant difference. In addition, the analysis of the expressed BMP during each experiment indicated that 69 % and 63 % of the BMP was expressed for test 1 and test 2 respectively. Therefore, there is no impact of the RDR retention time in anaerobic digestion performance.

The difference compared to the performance observed of the test conducted without RDR (average methane production = 83 ± 16 Nm^3 CH_4/t of VS fed; 35 % of the BMP expressed) is quite important. The pre-treatment in RDR optimizes the anaerobic digestion performance by almost doubling the expression of the BMP.

In order to study the evolution of the organic matter through the anaerobic digestion process, a Van Sœst profile of the input and output was conducted. Figure 5 shows the results obtained for both tests.

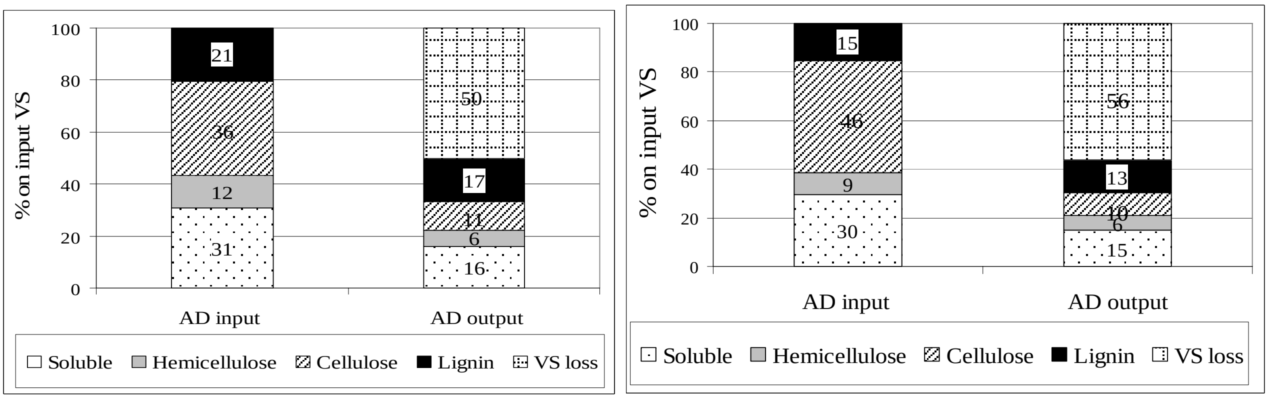


Figure 5: Evolution of the organic fractions (Van Soest) during anaerobic digestion for test 1 (on the left) and test 2 (on the right)

Similar profiles and evolutions were observed for both tests. In both cases a degradation of 50-55% of organic matter was observed. Two main compartments are degraded: the soluble fraction which decreased from 31 % to 16 % in test 1 and from 30 % to 15 % in test 2 and mostly the cellulose and hemicellulose fraction (from 48 % to 17 % for test 1 and from 55 % to 16 % for test 2).

3.3 Composting

The indicator of residual organic carbon in soils (IROC) was calculated in order to characterize the stability of the organic matter in dehydrated digestate at t_0 and t_4. IROC increased during composting (2.8 and 27.6 % increase for test 1 and 2, respectively), indicating organic matter stabilization throughout the composting process.

Initially the organic matter of the dehydrated digestate was more stable in test 1 than in test 2. Indeed, the IROC measured for test 1 was higher than for test 2 (70 and 55 % VS, respectively). This difference of organic matter stability at the beginning of the composting process is also evident with the results of the organic carbon mineralization during 3-day incubation in soil (Cm3). In fact, results of both tests showed a great difference. At the beginning of composting

(t_0), organic matter was more available for microorganisms in the second experiment. Indeed, 9.4 % of VS have been biodegraded after 3 days of incubation for the dehydrated digestate of test 2 while only 3.9 % for the dehydrated digestate of test 1. Cm3 obtained for test 2 after 4 weeks of composting (3.4 %) was higher than Cm3 for test 1 (3.2 %) indicating that the organic matter of the test 2 at t_4 can still be further stabilized through composting.

A characterisation of the organic matter was done based on the Van Soest profiles (Figure **6**). The objectives were to characterize the organic matter before and after composting and to evaluate its evolution during composting. The results observed for test 1 showed a transfer from the cellulose fraction to the hemicellulose fraction indicating a reorganization of the organic matter. On the contrary, the results obtained for test 2 showed a transfer from the cellulose fraction to the soluble fraction indicating active organic matter degradation. These results are consistent with the difference of stability outlined before.

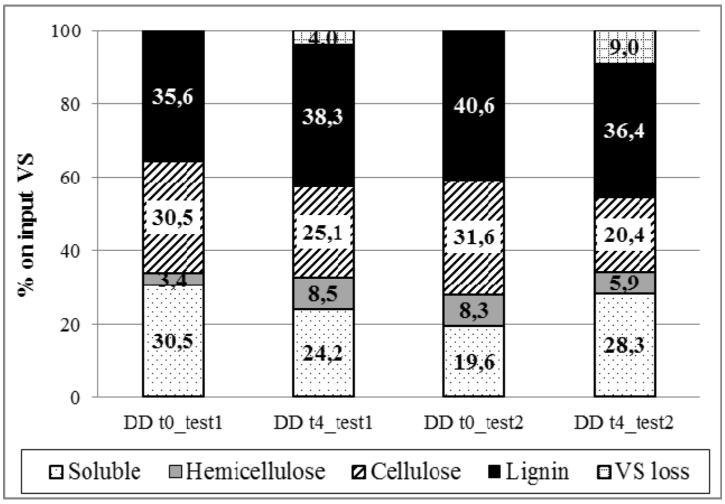


Figure 6 : Van Soest profiles for dehydrated digestate at the beginning of composting and after 4 weeks of composting

The difference of stability was also observed on the evolution of the temperature during composting. The temperature reached during the composting of the digestate in test 2 was higher than in test 1 (Figure 7). However in both cases the maximum temperature reached was 50°C. It is probably insufficient from hygienisation point of view. An additional composting test was conducted in order to improve temperature rise. The digestate from the anaerobic digestion was blended with a fresh bulking agent (green waste (GW)). The ratio used for the new mixture (DGW) was one volume of digestate for one volume of green waste. Both a greater temperature rise (T_{max} ≈ 60°C) and organic matter degradation (20.5 % of initial VS content) were observed during the composting of the new mixture (Figure 7).

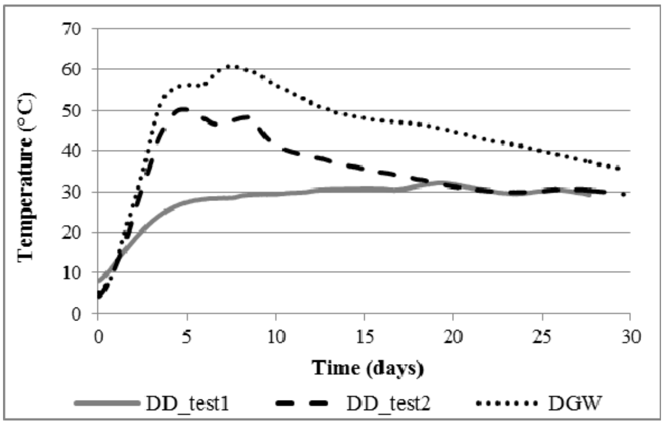


Figure 7 : Temperature evolution during composting for three different mixtures

The difference of stability observed between both tests could be due to the different retention times in the RDR. 4 days in RDR seems to facilitate the access of microorganisms to organic matter improving its biodegradation and consequently the temperature rise during composting.

The composting of dehydrated digestate, although feasible, gives low organic matter degradation due to the important VS loss during anaerobic digestion. The rate of organic matter degradation and the temperature rise during the composting process can be improved by addition of a fresh bulking agent like green waste.

4. CONCLUSION

During the current study, an increase in the retention time (1.5 and 4 days) of the RDR leads to an evolution in the particle size from the bigger fractions to the smaller ones. On the contrary, no degradation of organic matter was observed at any retention time. Other studies observed a greater shift from the bigger to the smaller fractions and degradation of organic matter at similar retention times.

The different retention times in the RDR showed no impact on the biogas production of the anaerobic digestion of the <40mm fraction of the RDR outflow. However, the presence of the RDR increased significantly the expression of the biogas production potential (as the rate of BMP expression) compared to the anaerobic digestion of a comparable rMSW non-pre-treated in the RDR, as shown in previous studies.

Additionally, the retention time in the RDR had a slight impact on the organic matter stability before composting and the evolution during the composting process. In both cases, the digestate presented low organic matter content, degraded during anaerobic digestion, and for that reason a low rise of the temperature was observed during composting. An enhancement of the process could be achieved by mixing the digestate with green waste (1/1, V:V). Although the composting products respect some of the parameters of the French standard NF U 44-051, further research is required to optimize the composting process and to evaluate the possible agronomic valorisation of the final product.

It is important to notice that, although this study shows no major impact of the retention time of the RDR on the performance of the treatment, short retention times can induce very important operational problems such as clogging and are not recommended by constructors. The experiments, although realized in an industrial plant, were conducted during short periods of time and not under continuous operation.

5. ACKNOWLEDGEMENTS

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(100) INNOVATIVE REVAMPING MBT PLANTS – A PORTUGUESE CASE STUDY

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EXECUTIVE SUMMARY

Biodegradable municipal waste technologies in Mechanical and Biological Treatment Plants (MBT plants) are gaining momentum throughout Europe and the world. These waste management technologies consist of several mechanical and biological stages that progressively recover and stabilise the biodegradable matter under controlled anaerobic and/or aerobic conditions.

At the same time, Materials Recovery Facilities (MRF plants) are being built in order to sort by different material the packaging waste at-source collected.

Typically, both types of facilities are being independently built and managed with innovative technologies such as sorting of organic material through UV scanning at MBT plants or optical recognition & scanning sorting at MRF plants. Despite its success and knowing both facilities do not process the same type of waste, one believes significant synergies can still be gained from integrating both technologies.

The role of MBT in waste management is predicted to grow for the foreseeable future, with the primary aim of MBT plants being the recovery of a large percentage of recyclables from mixed waste streams. Once this has been achieved, the main objective of the subsequent biological section of the treatment is to produce a material with low environmental impact fit for land application.

These new generation and more sophisticated solid waste treatment facilities are quite costly and technically difficult to operate. Hence, the costs associated to these technologies must be taken into account.

This paper presents a case study of an MBT plant operating in Portugal (Resiestrela`s company) that has been upgraded in 2011. Resiestrela's Composting Plant was built in 2001 and was dimensioned to treat 35.000 Mg of MSW and composting 20.000 Mg of biodegradable waste per year. An extra investment of € 2,5M was needed to revamp the plant that is nowadays processing 70.000 Mg of MSW and 30.000 Mg of biodegradable waste per year. This plant is now able to combine MRF high technology (optical separation equipment) with organic recovery processes, making it possible to treat both waste chains in a profitable manner.

Cost estimation is a basic requirement for municipal solid waste policy planning and on the face of scarce availability of data, this paper intends to present and describe the investment costs as well as new operational revenues and operational costs regarding new data in order to do a cost-benefit analysis.

After the upgrade, the plant not only increased the compost production, but mostly increased the amount of recyclables materials recovered from roughly 1% to more than 6,5% of the input in MSW. This enables the plant to increase significantly the revenues regarding recyclables materials sells and also diminish the amount of waste disposed into landfill.

The preliminary results indicate automation of MBT plants as an adequate and cost effective solution to achieve recycling goals in the 2011-2016 time-frames.

1 INTRODUCTION

The production of municipal solid waste (MSW) is an inevitable consequence of today's consumer society. This stream of solid waste generated by households and by commercial establishments and institutions (considered equal to households) includes food scraps, product packaging, grass clipping, small furniture, clothing, bottles, newspapers and batteries. It does not include medical, commercial and industrial hazardous or radioactive wastes, which must be treated separately. Portugal produces approximately 6 million tons of MSW each year and finding sustainable and cost-effective alternatives to the disposal of MSW in landfills represents a major challenge to the waste management industry.

The introduction of the EU Council Directives 99/31/EC and 2004/12/CE resulted in the development of new waste management strategies in Europe. The first Directive primary goal is to prevent or reduce as far as possible the negative environmental effects from landfill biodegradable waste disposal. The second Directive promotes separation at source and packaging waste recovery. Only waste that is not suitable for re-use, recycle or recovery should be disposed off in sanitary landfills.

In this context, alternative biodegradable municipal waste processing technologies are gaining importance throughout Europe and the world. These waste processing management technologies consist of several biological stages that progressively reduce and stabilise the biodegradable matter under controlled anaerobic and/or aerobic conditions.

In many countries previously heavily dependent on landfill, and embarking on organic waste recovery for the first time, it makes good environmental and economic sense to start by building composting facilities. Composting can be by simple windrowing, and the first waste streams treated are usually just those derived from curbside collection of garden waste, which is readily mixed and composted in most climates. However, when the propose is to reduce significantly biodegradable waste going to landfill and achieve the UE targets, a improve scheme of source separated organic waste collection must be implemented or a sophisticate mechanical treatment upstream the composting stage might be design in order to produce a compost which fulfils the legal requirements. Nevertheless, these solid waste treatment facilities are quite costly, and it must be stressed that not only are the markets limited for the compost products produced, but also the value of this material is very difficult to realize as a financial return.

So far limited information is available for getting reliable cost estimates, but processing unsorted waste using innovative technology in order to maximize recyclable materials in MSW recovery can be economically advantageous. And under the correct conditions, MSW in MBT plants have been shown to compost effectively, behaving in a similar to green waste-derived composts. Hence, recycling and composting integrated in a unique plant are seen as attractive waste management options.

At the same time and as a result of the implementation of the second directive mentioned above regarding the packaging waste recovery, Materials Recovery Facilities (MRF plants) are being upgraded in order to maximize the sorting of selectively collected recyclable materials by different types of material: glass, paper/cardboard, plastics (distinguishing PE, HDPE, Brick, Film and mix plastic) and metals (ferrous and non-ferrous).

1.1 Portuguese background

On the second half of the nineties, Portugal started a revolution in municipal solid waste management. At the time, the municipalities began to associate with each other to spin off management companies with the objective of managing – collect, transport, treat and recover – the municipal solid waste produced in those municipalities and, at the same time close the existing dumps. Today, 26 municipal solid waste companies subsist and the access to some process of municipal solid waste treatment or recovery is now available to all the population.

EGF - Empresa Geral do Fomento, S.A. is Águas de Portugal Group's sub-holding company responsible for guaranteeing the treatment and recovery of waste within a framework of environmental and economic sustainability. The management of waste treatment and recovery systems is carried out by 11 concessionary companies created in partnership with the Municipalities. These companies process more than 3,5 million tons (Mg) of MSW per year, serving about 60% of the Portuguese population living in 174 Municipalities. Its universe is constituted by several infrastructures that can be seen in Table 1.

The definition of the national sector strategic plan for the period 2007-2016 (PERSU II) limit the quantity of biodegradable waste going into landfills (35% of the quantity existing in 1995 for 2020).

In addition, like other countries in EU, Portugal has the objective of recycling 60% of the total package units placed on the market, considering a minimum of 55% for each material. The goals for each EGF companies were defined together with SPV – Portuguese green dot society, and EGF companies share the objective of sending to the recycling industry more than 240 thousand Mg of recyclable materials in 2011, representing a contribution greater than 50% of the country goals to that year. National legislation intends to increase those quantities until 2016 in 25%, which is an ambitious objective.

TABLE 1 **MSW Treatment Facilities of EGF universe.**

Existing MSW Treatment Facilities (2012)		**Global capacity (kMg/year)**
Biodegradable Waste Valorisation		**~1085**
Composting of MSW	3	~300
Anaerobic Digestion SSOW	1	~35 (+30)
Anaerobic Digestion MSW	7	~700
Composting of green waste	3	~20
Energy Valorisation		
Incineration plant	1	~600
Sanitary Landfills	22	
Selective Collection and Sorting	18	
Transfer Stations	48	

1.2 Mechanical and Biological Treatment plants (MBT plants)

The role of MBT in waste management is predicted to grow for the foreseeable future, with the primary aim of MBT plants is to recover a large percentage of recyclables from mixed waste streams. Once this has been achieved, the main objective of the subsequent biological section of the treatment is to produce a material with low environmental impact fit for land application.

Using a range of technologies, MBT plants are nowadays designed to separate the fraction smaller than 40-80mm (mechanically sorted organic residuals - MSOR) of the fraction greater than 80mm which is rich in recoverable recyclables materials (glass, paper/cardboard, plastics, metals).

In Portugal, there are 11 organic waste recovery plants in operation, belonging to different companies, namely Lipor, Braval, Amagra, Resiestrela, Resinorte, Tratolixo (Amtres), Amarsul, Valnor, Suldouro, Valorlis and Valorsul. 7 of these 11 plants belong to EGF group. Throughout the next year EGF Group expects to start operating 4 more Mechanical Biological Plants for MSW Treatment (ERSUC, Amarsul and Algar). In this context, by the end of 2012 there will be a total of 11 organic waste valorisation plants operating in Portugal belonging to EGF group.

Although separation at source collection programs for recyclable materials are well installed in major European countries, there is still a significant amount of recyclable material not properly separated at-source which end up at the unsorted Municipal Solid Waste fraction (MSW) and directed to MBT plants. In addition, the new and more ambitious quantity goals that should be accomplished in the period 2011-2016 called for an update of the facilities in order to increase sorting capacity and recyclable recovery wherever it is (unsorted or source separated streams).

Several companies manage and build their MBT plants so they can also rely on the mechanical pre-treatment to accomplish their individual goals, through the recovery of recyclable materials remaining in the household waste.

The increase of sorting capacity of the plants was, traditionally, attained by employing additional workers to sort, in the hand sorting cabin, the fraction greater than 80mm rich in recoverable recyclables materials. Though, the increase of employees means a directly increase on operational costs. On the other hand, despite its value as recyclable materials, most of this waste cannot be, in an efficient and intensified way, recovered by these plants and end up being dispatched to sanitary landfill.

An alternative to the traditional and conservative solution might be the use of high technology including optical recognition & scanning sorting equipment at the sorting line of equipment that classifies automatically the materials by type (plastic/non-plastic; PE/HDPE/film/brick…).

Processing unsorted waste using innovative technology in order to maximize recyclable materials in MSW recovery can be economically advantageous. Therefore, an evaluation of the investment and maintenance costs, along with efficiency improvements is needed.

The applied innovative technology usually minimizes the need of human work. Though, workers are still important since the greater efficiency of the equipment represents according to the experience of several facilities, frequently a lower quality of the stream recovered. Then, workers are typically associated with quality control of the sorted materials (negative sorting) and management of bale material.

2 RESIESTRELA’S COMPOSTING PLANT

Resiestrela, S.A. is the company responsible for the treatment of the Municipal Solid Waste (MSW) produced in the Centre/Northeast region of Portugal, approximately 70 thousand tons per year. It covers a population of nearly 65 thousand inhabitants.

This system consists of several operational facilities: 14 collection centres; 1 landfill (including biogas recovery and CPH unit); 1 Materials Recovery Facility; 1 composting plant; 8 transfer station.

Resiestrela’s Composting Plant was built in 2001 by the consortium HLC/CGEA/Hagen/TVD/Conegil and it cost roughly13M€.

FIGURE 1 **Resiestrela’s Composting Plant, sanitary landfill and WWTP.**

2.1 Process description before upgrading

The Composting Plant receives MSW from 14 Municipalities and the mechanical treatment includes operations of screening, and magnetic separation as well as manual sorting, aiming to remove the greater flow of recyclable materials (plastics, glass, paper/cardboard and metals) and to separate organic materials from the rest. Organic material is dispatched to biological treatment and recyclable materials are sent to the respective recycling units, receiving a motivation fee from the entity that manages the integrated systems of packaging waste management. Materials rejected by the process are dispatched to landfill.

The biological treatment process consists of a composting building with automatic feeding and turning machine where the material spends nearly 1 month. After pre-composting, it is maturated in a post composting hall during roughly 2 months. After that the compost is refined by passing it through a vibrant sieve and a densimetric table with a cyclone system to remove light and heavy contaminants.

The exhausted air extracted from the intensive composting building (30.000m3/h) is treated in a biofilter.

The plant is dimensioned to treat mechanically 30.000 Mg of MSW and biologically (aerobic process) 20.000 Mg of biodegradable waste per year.

The plant receives waste 7 days per week, processing in 5 days, 8 hours per day (only one shift), 250 days per year.

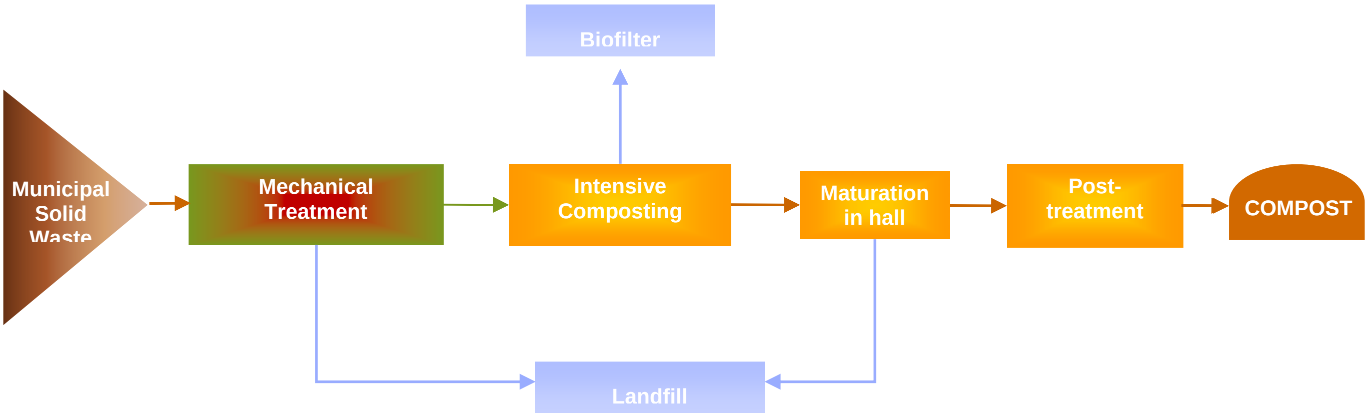

FIGURE 2 **Process flow diagram of Resiestrela's Composting Plant.**

2.2 Process description after upgrading

Due to several constraints in the last years, the availability of the composting plant has been significantly lower than expected. The main problems the plant was facing were:

- Closed and full plastic bags in the end of the mechanical line (refuges stream), due to the lack of a bag-opener;
- Difficulty in recovering recyclable materials due to the exceeding load of waste on the hand sorting conveyors
- Intensive maintenance works demanded of the turning machine (and the original supplier ceasing)

Therefore, in 2010, it was decided to revamp the plant in order to increase biological treatment capacity, maximize recyclable material recovery and solve several operational problems. Hence, a public tender process was promoted to convert the existing plant and an investment of 2,5M€ was done, which included:

- bag opener at the head of the pre-treatment line;
- hand sorting cabin (2 positions) at the end of the mechanical treatment line;
- magnetic separator to increase the recovery of ferrous materials capacity;
- ballistic equipment to separate rolling and heavy materials from flat and light materials (to promote the sorting capacity and quality, downstream);
- automatic screening and sorting equipment to maximize the recyclable materials recover (3 optical sorting equipment to separate i) plastic film; ii) plastic/non plastic materials/brick packages; iii) PE, HDPE, mix plastics);
- bale press and Teletruck for sorted materials and bale management;
- wheel loader to feed MSOR into composting building and form the windrows;
- 10 blowers for forced aeration of compost windrows;
- turning machine for the compost (side-turn machine).

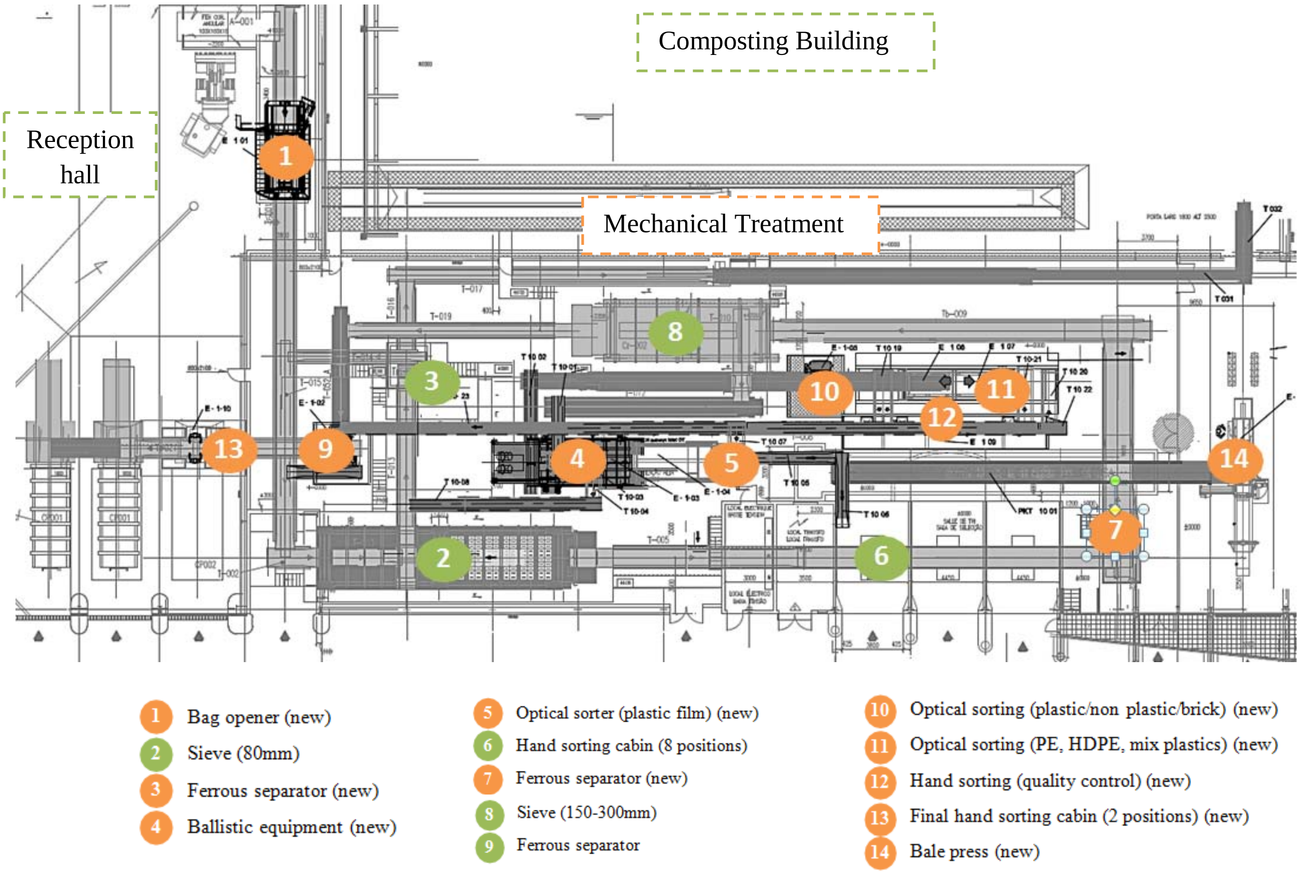

FIGURE 3 **Mechanical Treatment (revamped) of Resiestrela's Composting Plant.**

Hence, nowadays, the mechanical treatment includes operations of bag-opening, screening, and magnetic separation as well as manual sorting, aiming to remove the greater flow of plastic film (greater dimensions), glass and paper/cardboard, a ballistic equipment and, complementary, automatic sorting to separate the maximum of PE, HDPH, Brick, mix plastic (see Figure 3). The fraction lower than 80 mm is dispatched to biological treatment that occurs in the original composting building but without the automatic feeding and turning machine used before. The feeding is done by the wheel loader and the windrows are mainly static and forced aerated (negative mode). In the post composting hall, half of the piles are also forced aerated (positive mode) and frequently turned by the new turning machine.
Currently, the plant is processing 6 days/week, 8 hours per day (one shift only), and will be able to treat mechanically more than 60.000 Mg of MSW and biologically (aerobic process) roughly 30.000 Mg of biodegradable waste per year.

2.3 Operational costs & revenues

Between 2001 and 2010, before revamping the plant, an average of 25.000 Mg of MSW were processed (mechanical treatment) and 15.000 Mg of organic waste was composted (biological treatment), per year. In those conditions, the plant was able to recover annually roughly 250 Mg of recyclable materials (only ~1% of total MSW treated) by hand sorting.

After the revamping work, a great increase of capacity is foreseen. The plant is able to treat all the waste produced and collected in the region (~60.000-70.000 Mg of MSW), and according to the figures of the last 6 month after the start-up, an amount of recyclable material greater than 6,5% of the input of MSW can be recovered. That means a significant increase comparing to the original mechanical treatment scheme. The biological treatment has also been upgraded and amplified (intensive aeration), and roughly 30.000 Mg of MSOR obtained in mechanical treatment can be recovered in the composting building.

TABLE 2 **Capacity and main products of the plant.**

Capacity	Before up-grading Mg/year	After up-grading (estimative 2012) Mg/year
Mechanical treatment (MSW treated)	**25.000**	**60.000**
Biological treatment (MSOR treated (organic waste))	**12.000**	**30.000**
Recyclables recovered	**500**	**3.900**
Ferrous and non-ferrous metal	250	900
Plastics (PE, HDPE, Brick, Film)	250	3000
Compost selling	**2.000**	**3.000**

Considering those average conditions, operational revenues and operational costs for both periods were estimated and are summed up in Table 3.

TABLE 3 **Major operational revenues and operational costs, before and after upgrade of the plant.**

Revenues	Before upgrade €/year	After upgrade €/year
Sales recyclables	**75 k€**	**795 k€**
Ferrous and non-ferrous metal	12.5 k€	45 k€
Plastics (PE, HDPE, Brick, Film)	62.5 k€	750 k€
Sales compost	**20 k€**	**30 k€**
Gate fee	**1.2 M€**	**2.9 M€**
Costs		
Supplies and External Services	**239.2 k€**	**334.9 k€**
General consumables	1.8 k€	35.2 k€
Process consumables	60.5 k€	232.0 k€
Maintenance	33.5 k€	60.5 k€
Special Services	143.4 k€	62.9 k€
Staff costs	**133.8 k€**	**263.5 k€**

Regarding recyclable materials revenues, it is important to clarify that SPV (national Green Dot Society) provides operators, such as Resiestrela, with incentives fees differentiated by the type of material, and for the amount of recyclable materials sold in the recycling market (to promote this extra taskforce and contribution to EU targets achievement). Since the actual market value in this sector is irregular, it was considered an average value of 50€/Mg for metallic materials and 250€/Mg for all plastics (PE, HDPE, Brick, Film).

In Table 4 it is shown the results of 6 month monitoring of recyclables materials recovery by type of material, immediately after the start-up of the revamped plant. As presented below, the type of material most sorted is plastic film followed by ferrous material and paper/cardboard. In overall, an amount of recyclable material greater than 6,5% of the input of MSW can be recovered.

TABLE 4 **6 month monitoring of recyclables materials recovery by type of material.**

Recyclable material	min. (%/MSW)	max (%/MSW)	average (%/MSW)
PE	**0.38%**	**0.70%**	**0.52%**
HDPE	**0.24%**	**0.31%**	**0.27%**
Brick	**0.42%**	**0.56%**	**0.49%**
Film	**2.26%**	**3.31%**	**2.74%**
Mix plastic	**0.62%**	**0.75%**	**0.66%**
Glass	**0.02%**	**0.10%**	**0.06%**
Ferrous	**0.82%**	**1.08%**	**0.97%**
P/C	**0.55%**	**1.07%**	**0.70%**

Concerning gate fee revenues, it is important to stress that the figures shown in Table 3 regard only to the gate fee charged to the MSW delivered in Composting Plant. Resiestrela charges a gate fee for the MSW delivered in the system which is the same fee independently of waste treatment destiny (composting plant, material recovery facility plant, landfill…). The gate-fee is annually adjusted to the expected profits of the whole system for that year. The gate fee has decreased in 2012 mainly due to the greater profits expected after the composting plant upgrade. For the compost, it was considered an average price of 10€/Mg.

In regard to the major costs of the plant, general consumables (Table 3), include expenses for office consumables, communication, insurances, literature, advertising, training programmes, external services, etc. Process consumables include expenses for utilities such as water, electricity and gas, fuel, other liquids and lubricants. Maintenance includes tools, equipment, vehicles and building maintenance. Special Services includes cleaning/housekeeping and other specialized services.

3 RESULTS AND DISCUSSION

When planning an upgrade of an existing MBT plant in order to achieve better recyclable materials recovery targets, there are both technical and economic aspects to be considered.

On one hand, when deciding which equipment should be included in the pre-treatment line and how it should be dimensioned, one has to stress that the knowledge of the market and the eventual willingness of governments is essential to plan which kind of materials should be recovered, so that the investment may be worthwhile. Otherwise, after the investment done, the company can face the difficulty of selling the materials recovered in the plant.

On the other hand, the correct design of the equipment is essential to achieve greater capacity, efficiency and efficacy. Since raw material is municipal solid waste, that includes food scraps, product packaging, grass clipping, small furniture, clothing, bottles, newspapers, paint, batteries, etc., the stream to be treated is significantly contaminated, in opposite to the experience faced in Materials Recovery Facilities, treating at-source packaging waste. Frequently the loading flux is also significantly higher to those in MRF plants so that the capacity has to be much greater. Thus, when designing and predicting the line concept it must be stressed that the line must accommodate a greater flow and lead with a greater level of contaminants (that difficult the scanning tasks). According to the experience obtained in EGF group facilities, ballistic equipment where rolling and heavy materials are separated from flat and light materials, promotes downstream capacity and efficacy on scanning and sorting the different kind of materials. Though, it represents a significant increment on the investment costs (200 - 400k€). When upgrading an existing plant it must be factored the size of this kind of equipment. Note that the greater the superficial area of the equipment, the higher residence time and consequently efficiency on the separation capacity. Additionally, the automatic scanning and sorting equipment should be dimensioned above the nominal capacity or, at least, never under dimensioned, since the quality of the sorting capacity can be extremely affected by the loading conditions.
An economic analysis of this upgrading project in Resiestrela's composting plant was done to evaluate the advantages of including automatic sorting systems in an existing MBT plant.

The figures are presented in terms of cost per ton of MSW that is treated per year in the plant (60.000), believing that it is the most meaningful way of representing the total operating costs and allowing comparison and extrapolation to other plants (Table 5). Note that only the difference promoted by the upgrade (descripted in section 3) is considered and includes the intervention on composting capacity. Besides the great increase of total capacity of the plant, the difference in gate fee revenues are not considered since it is independent of material treatment inside the system for the overall of the company.

There are some aspects to be factored into the economic analysis:

- The investment cost (10 years of depreciation period was considered) was included in the annual net costs. However, it usually differs significantly from case to case due to the EU financial programs commonly associated to this kind of facilities. In this case, the project was, indeed, subsidized by EU financial programs in nearly 70%. Besides that, the real investment costs of the project was considered so that it can be easily analysed;

- Recyclable materials revenues also differ significantly from case to case in EU. There are different financial programs and government initiatives to be factored. In Portugal, SPV (national Green Dot Society) provide operators with incentives fees differentiated by the type of material (independently of the surplus actual market value) to promote this extra contribution to EU targets achievement;
- When more recyclable material is recovered in pre-treatment line, it will have great impact not only in the increase on the revenue, but also in reducing of landfill waste disposal. The so called opportunity cost of this project is the cost of the amount of recyclable material recovered increase if it would be landfilled. The landfill disposal treatment costs of Resiestrela's company is estimated at 12€/Mg of MSW.

TABLE 5 **Operational revenues and operational costs due to the upgrade.**

Increased Revenues	€/MgMSW	
Sales recyclables	**12.07 €**	
Ferrous and non-ferrous metal	0,39 €	
Plastics (PE, HDPE, Brick, Film)	11,68 €	
Sales compost	**0,15 €**	
Operational Revenues (A)		**12,22 €**
Increased Costs		
Supplies and External Services	**3,86 €**	
General consumables	0,56 €	
Process consumables	2,86 €	
Maintenance	0,45 €	
Staff costs	**2,16 €**	
Operational Costs (B)		**6,03 €**
Upgrading Investment	**4,17 € ⬄**	
⬄Costs Depreciation - 10 years (C)		**0,42 €**
Opportunity costs (landfilling) (D)		**0,68 €**
Upgrade net costs (A-B-C+D)	**6,45 €**	

As a result of the upgrade, there is a significant increase of the running costs of the company, due to mainly process consumables and staff costs. Indeed, the number of employees was increased (typically associated with quality control of the sorted materials, management of bale material and turning compost new machine) and the plant has increased the labour time in 1day/week. The increase in process consumables is mainly due to the higher expenses in electricity (more ~100k€/year) and fuel (more ~50k€/year). Despite the expected increase in maintenance needs, usually connected to more equipment involved, Resiestrela's composting plant was already facing several problems in the past concerning the automatic turning machine and other equipment installed in the mechanical treatment so that the impact of the new and higher technologic equipment installed is not significant. When estimation is need, 1 to 3% of the investment cost is commonly used.

Additionally, this upgrade of the plant enabled the company to treat the "at source" separated recyclable material also in this facility. It is a great improve in efficiency for Resiestrela's company since the existing MRF plant does not have automatic sorting equipment installed (only hand sorting is done), and the total amount processed per week in this facility is nowadays processed in one single day in the mechanical treatment of the composting plant. Nonetheless, those figures were treated independently and not considered in this study.

Recyclable materials recovery efficiency can be easily increased by processing the refuges flow. Some tests during the start-up were done (at the end of the dayshift) in order to treat the amount of refuges stored during the day without hand sorting work (only automatic sorting) and it was observed a significant increase mainly in plastic film recovery. Unfortunately, as the composting plant is already at its full capacity, it is not possible to over treat this flow without an increase in the labour time (implying an increase on staff costs) so the experimental tests were abandoned. Nevertheless when extra capacity takes place it is recommended to analyse and quantify the benefits of this extra recovery ability.

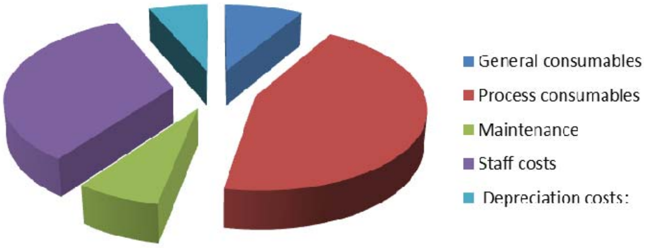

FIGURE 4 **Operational and investment costs differences breakdown observed after upgrade.**

4 CONCLUSIONS

Municipal solid waste from undifferentiated collection may be subject to different processes of treatment and recovery, whereby waste that is confined in sanitary landfills can be gradually broken down by material fraction not suitable for recuperation, recycling or recovery in its different forms.

Combined plants, where recyclable recovery is directly integrated in composting plants, show many advantages and all those involved in the planning and design of waste treatment facilities should consider these benefits. Processing MSW using innovative technology in mechanical pre-treatment, maximizes the recyclable materials recovery.

The plant of present case study is now able to combine MRF high technology (optical recognition & scanning sorting) with organic valorisation processes, making it possible to treat both MSW and packaging waste chains in a profitable manner. Increases on recyclable materials recovery from roughly 1% to more than 6,5% of the input were obtained. Besides resulting in the significant increase of the revenues regarding recyclable materials sells, it also contributes to lengthening the useful life of the landfills and in the promotion of the use of endogenous energy resources.

5 FURTHER CONSIDERATIONS

10 of the 11organic Waste Treatment Plants of EGF group mentioned above, process mechanically unsorted municipal solid waste being able to recover a percentage of the recyclable material still present in domestic waste. In 2012, it is expected to recover about 50.000 Mg/year of recyclable material, complementary to the amount source-collected and sorted in MRF plants. Some of these MBT plants had already foreseen similar schemes in order to maximize recyclable materials recovery. Assuming the same increase of efficiency obtained at Resiestrela’s composting plant to all those existing plants of EGF group, figures could reach up to 65.000 Mg/year, contributing significantly to the packages recovery UE and national targets. (~240.000 Mg/year).
Nevertheless, it has to be stressed that when investment and operational costs are analysed, EU funds and willingness of governments providing operators with financial and tax reduction incentives toward carbon emissions reduction and national targets achievement contribution must be factored. Hence, one can conclude that the results indicate automation of MBT plants as an adequate and cost effective solution to achieve recycling goals in the 2011-2016 time-frames if incentives to fulfil EU targets take place.

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(236) MECHANICAL-BIOLOGICAL WASTE PROCESS OF MUNICIPAL SOLID WASTE: PILOT LABORATORY PROJECT

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EXECUTIVE SUMMARY

From October 2008 to May 2009, the Centre de recherche industrielle du Québec (CRIQ) performed a pilot laboratory project on behalf of the City of Montreal, pertaining to energy recovered from municipal solid waste (MSW) through mechanical-biological treatment (MBT), a process which combines mechanical components (grinding and sifting) with a biological component (biological drying). This project was intended to develop the optimal production conditions required for producing a viable, energy-recovered by-product with desirable end-user characteristics (cement plants, cogeneration plants, boiler houses, etc.).

Work performed included:

- Sampling and characterization of various types of MSW available throughout the City of Montreal territory;
- Assessment of the need to create mixtures from various types of available MSW;
- Seeking information as to the desired characteristics required for energy recovery of fuels produced through MBT processes;
- Performance of laboratory and large-scale MBT tests related to MSW in order to assess the need of using biological drying procedures on MSW or on individual fractions after sieving. These tests were intended to determine the proportions and characteristics of the various individual fractions obtained during the sieving processes of MSW;
- Research and selection of equipment required for MBT processes and research of Quebec equipment manufacturers available in this area;
- Interpretation and mainstreaming of results leading to the development of a MBT supply chain process specifically designed for City of Montreal MSW.

Results from the work performed have shown that:

- The quantity of waste generated per household is approximately 11.3 kg/week/household and this waste has a moisture content of approximately 40%;
- The ‘compostable material’ category constitutes more than 50% of MSW and has a promising calorific value of 20 megajoules per kg on a dry basis;
- The coarser fraction (above 100 mm) is the most interesting fraction with respect to energy recovery. It represents 50% or more of all domestic garbage, has a moisture content of 15%, is not strong-smelling and does not require stabilization. The chlorine content of this fraction presently represents one of the main constraints in that based on criteria of 2 Quebec cement plants, the requirements were not met.
- The intermediate fraction (between 50 and 100 mm) must be dried and stabilized in order to consider its use as a fuel;
- The fine fraction (less than 50 mm) which represents approximately 10% of all MSW and contains a significant proportion of organic matter also has the highest moisture content and the lowest calorific value. Biological drying will lower the moisture content to approximately 20% and, at the same time, stabilize it biologically. The fine fraction which is biologically dried could be used as backfill in engineered landfill sites.

1 INTRODUCTION

In their overall development plan, the City of Montreal, in 2009 (City of Montreal, 2009) stated it was proposing to implement a mechanical biological treatment (MBT) plant for MSW derived from a three-way collection system.

MSW consists of residential materials collected through a three-way collection system: 1) MSW, 2) recyclable material and 3) compostable material. The MBT system is comprised of the sifting and grinding of MSW followed by the biological drying of these elements. The treatment enables the drying of MSW, thereby facilitating the use of recovered energy by third parties who have the required equipment (cement plants, cogeneration plants, etc.). MBT allows for the preparation and fractioning of MSW making possible the recovery of each individual fraction at the most appropriate location in accordance with end-users' specific needs. Biological drying processes are based on the same principles as composting processes but with respect to drying, the thermophilic phase (45 to 70°C) is amplified to dry the material as quickly as possible. Biological drying processes are not intended to produce compost. This project was one of the preliminary phases required for the development of a pilot plant (25,000 tons) as well as a full sized plant by 2014 (estimated capacity of 500,000 tons).

1.1 Research objectives

The project's aim was to determine, through various tests and trials performed on MSW, optimal conditions required for producing a viable energy-recovered by-product with desirable end-user characteristics (cement plants, cogeneration plants, boiler houses, etc.) through a MBT (mechanical-biological treatment) process.

2 METHODOLOGY

The preliminary phase of this project entailed the sampling and characterization of various types of MSW produced throughout the City of Montreal territory. Three municipalities from the Island of Montreal that had implemented a three-way collection system on their territory, Pointe Claire, Westmount and Côte Saint-Luc, were targeted as sampling areas. For each of these areas, a sampling of MSW taken from approximately 30 households over three streets was collected for each sampling campaign. Three different sampling periods were arranged over three months, from October to December 2008. MSW collected was weighed separately on a calibrated Toledo weighing platform with a 100 kg capacity and an accuracy factor of 0.05 kg. Afterwards, a sub-sample of 400 litres, on average, was taken from matters collected, using the quadrant method, and was sorted in order to determine its composition in accordance with 12 specific categories: 1) paper and cardboard, 2) glass containers, 3) other glass products, 4) metal and aluminum containers, 5) other metals, 6) plastics, n° 1 to 5 and n° & 7, 7) plastic n° 6, films and other plastics, 8) compostable material, 9) bulk waste and construction, renovation and demolition (CRD) waste, 10) hazardous MSW, 11) textiles, 12) others. For this sampling, the identification of categories used was adapted from other characterization projects for MSW performed in Quebec (Recyc-Québec and Éco-Entreprises, 2007). Analytical methods used for the project are described in table 1.

A search for information pertaining to characteristics required for energy recovery of fuel materials produced through the MBT process was performed by contacting businesses that need this type of fuel. This phase entailed the identification of criteria prescribed by potential users (cement plants, cogeneration plants, etc.) of MSW used as an alternative source of fuel. In order to achieve this, users of this type of fuel were identified and contacted: Boralex, Cascades and Kruger (cogeneration plants) and Lafarge Canada and Ciments St-Laurent (Holcim) (cement plants). Market requirements relevant to residual- type fuels were defined and characteristics relating to this type of product were identified. Lastly, air emission standards that had to be adhered to for cogeneration end cement plants were identified (MDDEP 2009). This information helped establish criteria which could restrict the use of MSW as recovered energy sources of fuel.

Pilot laboratory and large-scale tests regarding grinding and sifting procedures were performed on MSW in order to assess whether or not to use biological drying procedures on MSW or individual fractions. These tests also helped determine the proportions and characteristics of the various fractions obtained during the sifting process. Most pilot laboratory tests were performed on MSW which had not previously gone through a sifting process. This approach was designed to understand the nature of MSW and to determine if certain-sized fractions showed recovery possibilities without using the sifting phase. To achieve this goal, MSW was emptied manually by opening the bags of garbage with a knife. Analysis of this waste compared to critical composting parameters (moisture content, total organic matter content, total nitrogen content (TKN), carbon to nitrogen ratio, porosity) (Mustin 1987) was performed in order to determine the necessity of performing optimized composting formulations.

TABLE 1 Analysis method

Analysis	Method or reference
Total organic matter (TOM)	ASTM D2974
Moisture content	ASTM D2974
Ash content	ASTM D2974
Metals: Al,Sb,Ag,As,Ba,Be,Bi,Cd,Ca,Cr,Co,Cu,Sn, Fe,Li,Mg,Mn,Hg, Mo,Ni,Pb,K,Se,Na,Sr,Ti Tl,V,Zn	MA200-Mét.1,1 R3: ICP
Total halides (in Cl)	Combustion method and titration of chloride
Total fluorides	Alkali fusion (internal method), EPA13A
Total sulphur	Combustion method: LECO S-144 DR
Gross calorific value	ASTM E 711, Calorimetric bomb
C,H,N	LECO CHN-2000
Total organic halogens (in Cl)	18-11-02, V1,R1: Calorimetry
Self-heating	Performed in 20 litre insulated pail
Bulk density	Apparent density

Figure 1 introduces the sequence of tests conducted in a laboratory as well as large-scale tests. Sifting laboratory tests were performed using a mix of MSW previously collected and characterized. A mix of 3 m^3 of MSW (household waste) derived from the second characterization campaign from Pointe-Claire, Westmount and Côte Saint-Luc was undertaken. Equipment used for the size classification of MSW mix was a double-pole vibrating sifter (915 x 1,220 mm) with a 20 degree angle. Openings in strainers used were 102, 76, 50 and 25 mm respectively.

Large-scale grinding tests were performed using a hammermill for timber grinding. Sifting was performed with a trommel screen with a mesh size of 50 mm as this type of equipment is readily available. Subsequently, some research work on equipment used in MBT processes (grinding and sifting) was performed using our own personal contacts. Specialized Quebec equipment suppliers were contacted, allowing us to select more precise grinding and sifting equipment.

Biological drying tests (composting) were performed inside insulated 120 litre reactors which have been used by CRIQ for many years. Air is injected inside the reactors under a distribution grate and allows for a uniform aeration of material. Control of temperature evolution and of the desired drying level is performed by adjusting the ventilation rate. This type of reactor can simulate the center of a larger pile of material since temperatures can be maintained within the range of optimal composting temperature, between 50 and 60 °C (Haug 1993). These tests were normally performed for a period of 2 to 4 weeks. The progressive ventilation method meant maintaining the temperature between 50 and 60 °C for as long as possible, with a suitable ventilation rate. The variable ventilation method meant a variation of ventilation flow alternating between a temperature of 50 to 60 °C followed by a reduced temperature (below 30 °C). Three to four sequences of low and high levels of ventilation were performed to verify the material's self-drying capacity, meaning until the temperature ceased increasing

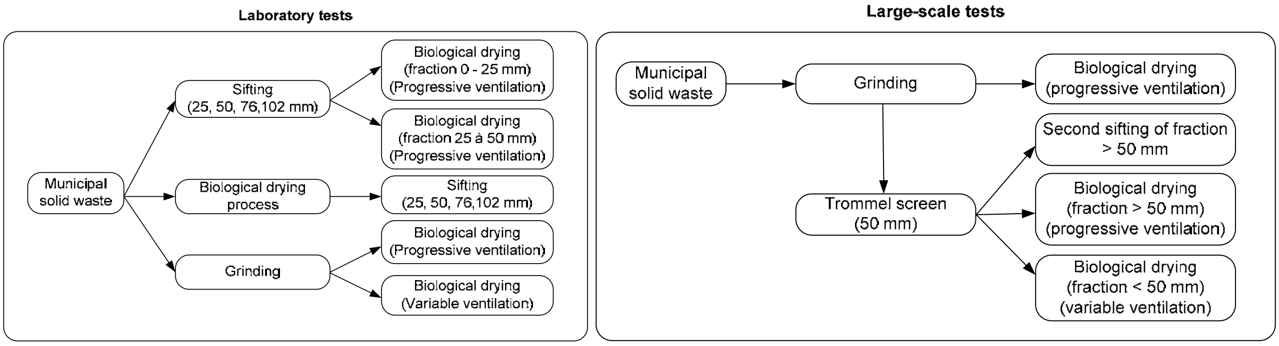

FIGURE 1 Sequence of tests performed (laboratory and large-scale tests)

Lastly, with the help of the effects on the individual materials used in each trial, we were able to integrate results for each test performed and thereby design a processing chain system allowing a large portion of MSW derived from a three-way collection system to be put into place.

3 RESULTS AND DISCUSSION

The presentation of results is divided into six sub-divisions: 1) characteristics of untreated MSW; 2) fractionation tests (grinding and sifting); 3) biological drying tests; 4) summary of characteristics for various fractions; 5) energy recovery from various fractions; 6) proposed MBT supply chain process.

3.1 Untreated MSW

- The quantity of waste generated per household was estimated at approximately 11.3 kg/week/household;
- Compostable material represented more than 50% of MSW;
- Categories with a high energy content, such as paper, for instance and various types of plastic constitute nearly 25% of all domestic garbage;
- The difference in the composition of MSW material collected in the three sample areas (Pointe-Claire, Côte Saint-Luc and Westmount) was insignificant;
- Depending on the seasons, analysis results may have been impacted by the composition of MSW. However, this aspect was not taken into account within the present project framework;
- Untreated MSW exhibits a moisture content of approximately 40%;
- Untreated MSW displays a meaningful calorific value (20 MJ/kg on a dry basis), but a moisture content which is too high (40%) for energy recovery as such, without any treatment;
- Chlorine content in untreated MSW would represent one of the major constraints in the development process of waste as an alternative fuel.

3.2 Fractionation tests: grinding and sifting

- The coarser fraction (above 100 mm) which represents more than 50% of all MSW and which exhibits a moisture content of 15% is the most interesting fraction with respect to energy recovery (calorific value above 20 MJ/kg on a dry basis);
- The coarser fraction (above 100 mm) is not strong-smelling;
- The fine fraction (less than 50 mm) which represents approximately 10% of all domestic garbage and which contains a major proportion of organic matter is the fraction with the highest moisture content and displays the lowest calorific value (< à 10 MJ/kg w.b.). This fraction must be stabilized.
- The fraction comprised between 50 to 100 mm exhibits a high moisture content (above 30%) and must be dried biologically for utilization as heating purposes;
- During the grinding process, all garbage bags must first be opened to avoid strong odour emissions during subsequent phases (biological drying, storage, handling);
- It is highly recommended to remove ferrous metals in the various stages within the treatment process.

3.3 Biological drying

- Untreated MSW and fine fractions (less than 50 mm) did not require the addition of any inputs for the biological drying process to begin and be efficiently achieved;
- Fine fractions have a higher moisture content than untreated MSW and biological drying processes allow the fractions to efficiently be brought back to a minimal moisture content of approximately 20%;
- In order to meet cement plant criteria standards, the moisture content of MSW would have to be reduced by a minimum of 15%;
- For biological drying to be effective, 24/24 forced ventilation must be implemented. As well, MSW must be stirred to create some consistency within the drying process;
- Using a variable ventilation method (alternating from high to low levels) allows for a faster and more effective drying process than the progressive ventilation method which, however produces a more stabilized, compostable result;
- Biological drying of the coarser fraction (above 100 mm) would not be required as the moisture content is almost identical to that required by cement plants (15%);
- Biological drying processes do not alter the calorific value (on a dry basis) of MSW;

- Biological drying processes produce a dry residue which is relatively deodorized over a period of approximately 20 to 30 days;
- Biological drying processes allow for a reduction of volume of approximately 15 to 20% and a reduction of mass by approximately 25 to 35% and, as a whole, dried MSW does not exhibit any odour but rather smells like compost;
- Fine fractions (less than 50 mm) which are dried biologically may be used as backfill in engineered landfill sites. These fractions represent approximately 10 to 15% of the volume and mass of untreated MSW.

3.4 Summary of characteristics for various fractions

Table 2 introduces a summary of characteristics for individual MSW fractions.

TABLE 2 **Summary of observations relating to various fractions of MSW derived through MBT**

Type of fraction	Mass proportion (%)	Drying requirement	Calorific value	Odour	Stabilizing requirement
Fraction > 100 mm	50	No: M.C. 15 %	High > 20 MJ/kg d.b.	Slight	No
Fraction from 50 to 100 mm	20	Yes: M.C. > 30 %	Medium to high	Strong-smelling	Yes
Fraction < 50 mm	10	Yes: M.C. > 40 %	Low < 10 MJ/kg d.b.	Strong-smelling	Yes

- d.b.: dry basis; w. b.: wet basis; moisture content: M.C.
-

3.5 Energy recovery from various fractions

For energy to be recovered from one or several MSW fractions, the chlorine content would appear to be one of the major constraints. Based on criteria obtained from 2 Quebec cement plants, this parameter was not satisfied. We think the presence of chlorine in MSW is caused by the presence of PVC (polyvinyl chloride) which is widely used in packaging material.

The 2 cement plants (Ciment Lafarge and Holcim) have expressed interest in this type of MSW and have found that the fraction of milled MSW (> 100 mm) sent to them by CRIQ met their requirements in terms of moisture content, calorific value and size grading. However, the chlorine content was too high with respect to their acceptance criteria. However, it is important to mention that the representative from Holcim specified that over a short-term period, the total limit value for halogens expressed in chlorine of 500 ppm would be increased to 1,500 ppm, so over a longer term there would be no limit other than stack emissions.

Consequently, it would appear as though chlorine content (presence of PVC material) is a hindrance to energy recovery from MSW and additional work must be done. Elsewhere around the world, energy recovery from solid recovered fuel (SRF) is practised. Despite less stringent regulations in certain countries and the use of facilities which are better equipped for handling this type of fuel, managers of these types of residue often have difficulty finding buyers for their products. In Quebec, the use of energy recovered from SRF would only be adaptable to cement plants, incinerators or gasification plants at this time. Cogeneration plants in Quebec are designed to use biomass (wood, bark, etc.) and are not suitable for burning residues such as SRF.

3.6 Proposed MBT supply chain process

The interpretation and mainstreaming of results led to the development of an MBT supply chain process for MSW for the City of Montreal (see figure 2).

Notwithstanding the chlorine concentration, the process would produce 2 fractions which could potentially be recovered as energy fuel (> 100 mm and 50 to 100 mm). These fractions constitute nearly 70% of all domestic waste. It would also be possible to recover nearly 3% of ferrous metals from the combustible fraction. In addition, non-recoverable MSW constitutes approximately 17% and 14 % of mass loss have been observed through evaporation during biological drying.

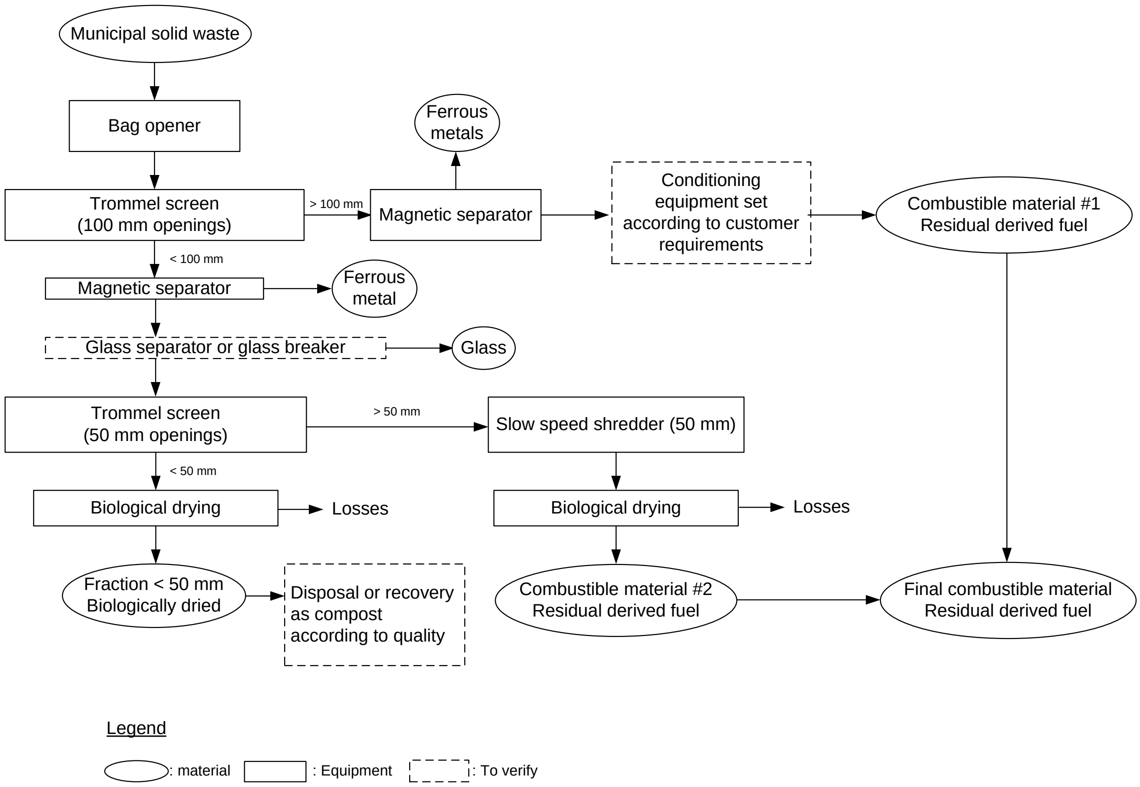

FIGURE 2 **Diagram of proposed MBT for MSW collected throughout the City of Montreal territory**

The proposed MBT chain is made up of equipment mostly used on an industrial scale. However, according to the information that was available during the project, there was only one MBT plant in Canada, the Dongara plant located near Toronto. Main equipment includes a bag opener, two trommel screens, a slow speed shreeder, a minimum of two magnetic separators, and infrastructure that enables biological drying. The proposed chain does not include conditioning of recoverable products (grinding, briquetting, etc.) as potential end-users have different acceptance criteria.

5 CONCLUSIONS

In conclusion, major elements forming an MBT supply chain process were identified as well as limits and constraints inherent in MSW treatment. The proposed chain would allow the production of 2 fractions which could potentially be recovered as energy fuel (> 100 mm and 50 to 100 mm). These fractions constitute nearly 70% of all domestic waste. It would also be feasible to recover nearly 3% of ferrous metals. In addition, non-recoverable MSW constitutes approximately 17% of the total mass. It would also be possible to recover 3% of ferrous metals. Furthermore, the performance of the project has raised several questions:

- Which materials contain chlorine and how do we remove them?
- What exactly is the chlorine concentration and variation contained in the recoverable fractions? Given the nature and heterogeneity of MSW, it would be important to develop a sampling method which would allow a more reliable identification of the composition of MSW.
- What impact would the recovery of MSW have on the quality of fractions produced?
- Would it be possible to extract plastic material from biologically dried fractions smaller than 50 mm and recover energy while adhering to cement plant criteria standards?

5.1 Recommendations

The next phase of the present project should be dedicated to the identification of chlorine sources, evaluation of the variation of MSW characteristics using a more elaborate sampling procedure (10-15 samples at a time), as well as the identification of adjustments that need to be made to the MSW treatment process. These tests could be performed with specialized industrial equipment and solid recovered fuel should meet criteria governing cement plants and other potential end-users of energy sources derived from MSW.

6 ACKNOWLEDGEMENTS

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Session 26

(157) DETERMINATION OF DISCARDED FOOD AND PROPOSALS FOR A MINIMIZATION OF FOOD WASTAGE IN GERMANY

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EXECUTIVE SUMMARY

The research project focussed on two topics. First, food waste along the value-added chain for food has been accounted and second, recommendations to contribute to a reduction in food waste were worked out.

The determination of the amount of discarded food in Germany revealed a total amount of 10.970.000 tons food per year.

By projecting and transferring the result from previous research the following fluctuation margin of the amounts of food waste from the food industry emerges: 210,000 – 4,580,000 tons per year. The median value of this projection is around 1,9 million tons per year. Thus 17 percent of the determined food waste is in industry.

Projecting and transferring the results from international studies also yields major fluctuation margins in the trade sector: 460,000 – 4,79 million tons per year. The median value of this projection is 550,000 tons per year. This includes also the wholesale sector, for which wholesale markets were analysed by means of an example. For this sector a total amount of food waste of 43,500 – 87,000 tons per year was determined. This corresponds to approximately a percentage of the amount handled each year. This fluctuation margins covers only organic waste and in some cases also includes plant and flower waste disposed together with food. The actual amount of food disposed of at German wholesale markets is likely to be smaller.

The total amount of food waste from the large-scale consumers – for example restaurants, hotels, hospitals, schools – is between 1,5 and 2,3 million tons per year. The median value of this projection is approximately 1,9 million tons per year. The catering industry accounts for the largest share of food waste, where a fluctuation margin of 837,000 – 1,015,000 tons per year was calculated. The second largest amount of food waste comes from in-company catering, followed by the accommodation sector. The total amount of food waste from the large-scale consumer corresponds to a share of 17 percent. About half of this amount could be avoided.

The private households create the largest quantities of food waste. The total amount of food waste from households in Germany per year is between 5,8 to 7,5 million tons. These are discarded through the residual waste or organic waste containers or through other routes such as self-composting, feeding to pets or in the sewer system. So households in Germany accounts for about 61 percent of the total food waste. On average, every citizen throws away about 82 kilos of food every year. The amount of avoidable and partially avoidable food waste from households in Germany corresponds to a monetary value of 16,6 to 21,6 billion EUR per year or approximately 200 to 260 EUR per capita and year. For an average household of four, this means that each year avoidable and partially avoidable food waste worth approximately 935 EUR is disposed of as residual waste, in organic waste containers and the sewer system, through self-composting or fed to pets.

In the project a variety of preventive measures that contribute significantly to the avoidance of food waste has been identified, categorized, described and evaluated. The results show 34 promising approaches from different countries, which are explicitly discussed regarding the transferability to Germany. Primarily, these best practice examples relating to consumers, large-scale consumers, trade and the food industry. Political action and scientific research projects were also discussed.

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1. INTRODUCTION

1.1. Background

The Food and Agriculture Organization of the United Nations (FAO) assumes that roughly one-third of the food produced globally for human consumption, or about 1.3 billion tonnes per year, is lost or wasted. At the same time, around 925 million people are suffering from hunger and malnutrition according to the FAO.

Throughout the value-added chain of foods (production, processing, transport, trade, and disposal), we draw upon natural resources and consume considerable amounts of them. These natural resources are wasted if food that has already been produced ends up not being consumed. This results in negative impacts on the environment (e.g. land use, eutrophication, adverse effects on biodiversity, CO_2 emissions, etc.).

In addition, it is both ethically and socially unacceptable not to make use of food. This widens the gap even further between prosperity and poverty, affluence and malnutrition and industrialised and developing countries. Besides these social and ethical aspects, the costs to society and to the individual stakeholders should also be pointed out.

In Germany, too, the issue of food waste received a great deal of attention last year. Studies, press reports, radio and TV broadcasts on this subject were released on a regular basis and engaged socio-political attention. A wide audience gained an insight into the subject of food waste thanks to public events (e.g. podium debates, fairs) and the coverage surrounding the movie “Taste the waste.”

The Federal Consumer Protection Ministry also raised the issue of reducing food waste by launching the campaign "Each meal (time) is precious" with a view to enhancing the appreciation of food. Early 2012, the European Commission also put the subject on the policy agenda and set the goal of curbing the amount of needless food waste by 50 percent EU-wide.

This goal is an ambitious one and can only be achieved if reliable data on the quantities of food waste generated are collected beforehand throughout Europe. Up until now, these data have not yet been available for Germany. Neither has there been any systematic overview of measures, initiatives and expert discussions to date that might point out ways of reducing food waste from farm to fork. Decision-makers therefore lacked a scientifically substantiated basis on effective action to minimise food waste.

1.2. Goals and terms of reference

The project was aimed at estimating the quantities of food waste generated in Germany on the basis of statistics, research, literature, surveys, expert consultations and specific studies on a random basis. Beyond that, proposals on how to reduce the amount of discarded food were developed. In doing so, the research team differentiated by food industry, wholesale and retail trade and consumers (large-scale consumers and households). An estimation of food waste in the farming sector involves a great deal of effort. It is investigated in separated studies and was therefore not covered in this research project.

The project comprises research into the data currently available and expert discussions, with an extensive analysis of data and literature on quantities, measures and initiatives and expert discussions in other EU countries and industrialised nations (e.g. USA, Australia). On the basis of the researched database, the research team estimated the amount of food waste in Germany, formulated recommendations for preventive measures, whilst at the same time identifying existing data gaps and outlining proposals for their removal.

The researched measures and initiatives with regard to optimised food management were assessed, bearing utility aspects in mind. Aspects of efficiency and transferability to the environment prevailing in Germany played an important role in the assessment.

As a result, feasible best practice examples are identified which, if transferred to Germany, may be expected to exert positive effects on food management and, in particular, on the avoidance of food waste.

1.3. Definitions and delineation of the field of investigation

The stakeholders in the food value-added chain surveyed by the project are shown in the following Figure.

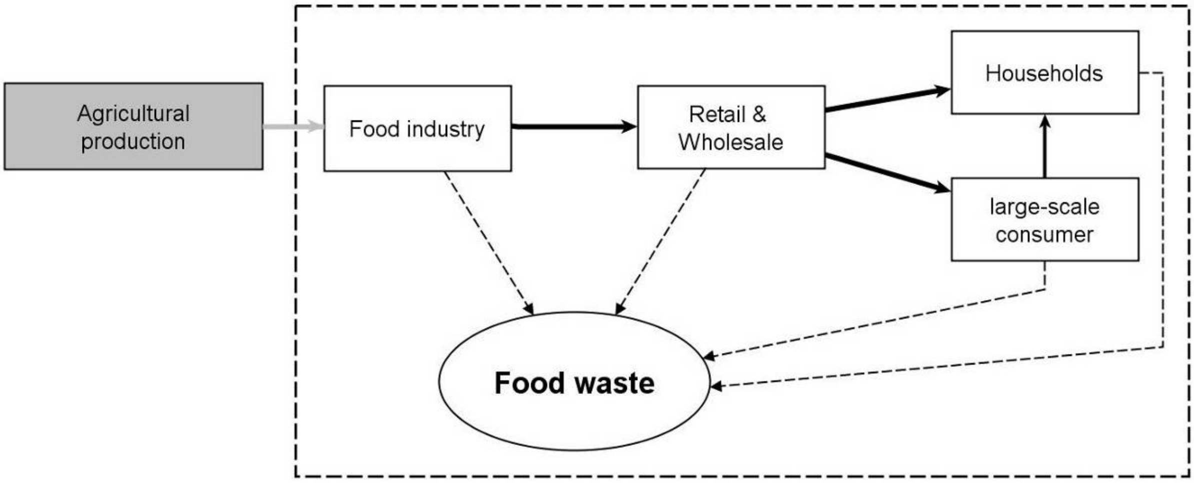

Figure 1: **Areas of the value-added chain for food under examination**

Definition: food waste

The term food waste comprises

- leftover food from
  - agricultural production,
  - (further) processing of food,
  - wholesale and retail trade,
  - kitchens of large-scale consumers,
  - private households and
- raw and processed foods that are fit for consumption.

Food waste is further subdivided into

- Avoidable food waste: still fully fit for human consumption at the time of discarding or would have been edible if they had been eaten in time
- Partly avoidable food waste: generated because of different consumer habits (e.g. bread crusts, apple skins). This category also covers mixtures of avoidable and unavoidable waste (e.g. leftover food, canteen waste, etc.)
- Unavoidable food waste: usually arises when food is being prepared and is discarded. This mainly encompasses both non-edible constituents (e.g. bones, banana peels) and edible ones (e.g. potato peels)

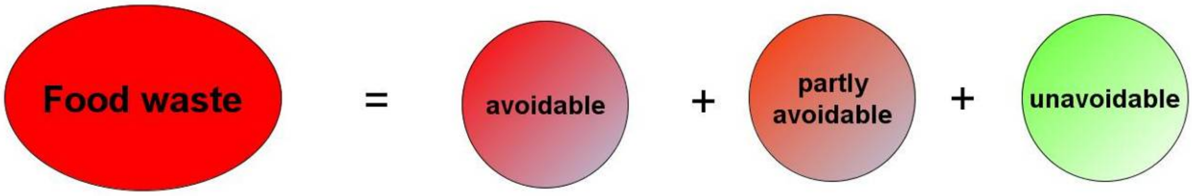

Figure 2: **Breakdown of food waste by avoidability**

2. DETERMINING THE QUANTITIES OF DISCARDED FOOD IN GERMANY

2.1. Methods to estimate the amount of discarded food in Germany

2.1.1. Food industry

To start with, existing studies and statistics were analysed for the investigation into the food industry.

In the absence of sufficient usable data on food waste produced by the industry, a questionnaire was drawn up to determine the quantity of waste in consultation with the Federation of German Food and Drink Industries

(*Bundesvereinigung der Deutschen Ernährungsindustrie (BVE*)). The questionnaire was distributed by the BVE and affiliated trade associations.

In addition, a revised questionnaire was distributed directly to most food business operators. Feedback as regards content and additions made by individual enterprises were incorporated in the revision.

2.1.2. Retail & Wholesale

At first, existing literature on trade (national and international studies, statistics, etc.) was analysed. These results were transferred to Germany with due regard to the methods used in each case.

In order to verify the database generated, trade associations for retail trade and for wholesale markets and other relevant stakeholders (e.g. food waste disposal firms) were contacted. Knowledge gained from these expert consultations and complementary on-the-spot checks were taken into account.

2.1.3. Large-scale consumers

The triangulation method was used for the analysis of large-scale consumers. Triangulation refers to a combination of more than one approach to produce results (cf. Blaikie, 1991). Thus, several calculation approaches were developed for the individual types of management of large-scale consumers as part of this research project to ensure best possible estimations of the amounts of food waste. In addition to the calculation regarding individual types of management, the total amount for all large-scale consumers was also estimated. The results were then compared.

The calculation routes and data sources that were used for the individual types of management can be found in the long version of the research project. The results for the individual types of management were compared with the total estimation for large-scale consumers.

2.1.4. Private households

The following framework conditions must be taken into consideration when estimating/projecting the amount of food waste from private households: There are different ways to dispose of food waste (cf. Figure 3) of which the municipal waste collection system covers only a part. Like the amount of waste in general, the amount of food waste is subject to several influential factors (time, area studied, household type and size, time budget, income situation, personal lifestyle). This means that in reality there is no such thing as a typical average household that can serve as a statistical reference.

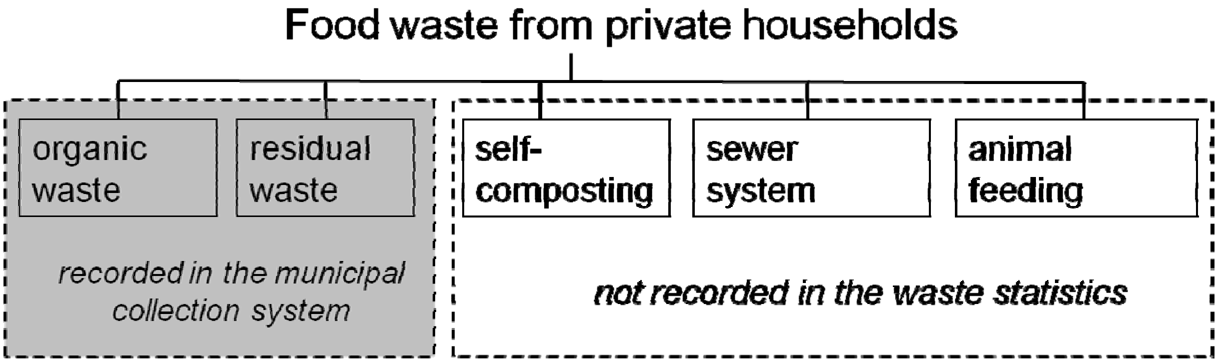

Figure 3: Disposal routes of household food waste

Projection approach:

- Critical evaluation of existing estimations for Germany. It has been found that the available data cover only parts of total food waste (e.g. no beverages, only food waste in the municipal collection system, only avoidable food, etc.).
- The amount of food waste was therefore extrapolated on the basis of data from waste collection analyses in Germany and from comparable national and international studies. The data were prepared and compiled (in comparable form wherever possible). The extrapolation was based on waste data (amounts and composition of waste) and was complemented by transferring data from supply side studies or from other regions wherever this was necessary due to a lack of data availability:
  - Estimation of the amount of food waste
  - Estimation of the composition of food waste

2.2. Results of the projections

2.2.1. Overview

Figure 4 shows the fluctuation margins of estimated food waste in Germany broken down by the areas of the food value-added chain that were analysed.
If one simplistically adds the mean values of these fluctuation margins, the total amount is 10,970,000 tonnes of food per year (cf. Figure 5).

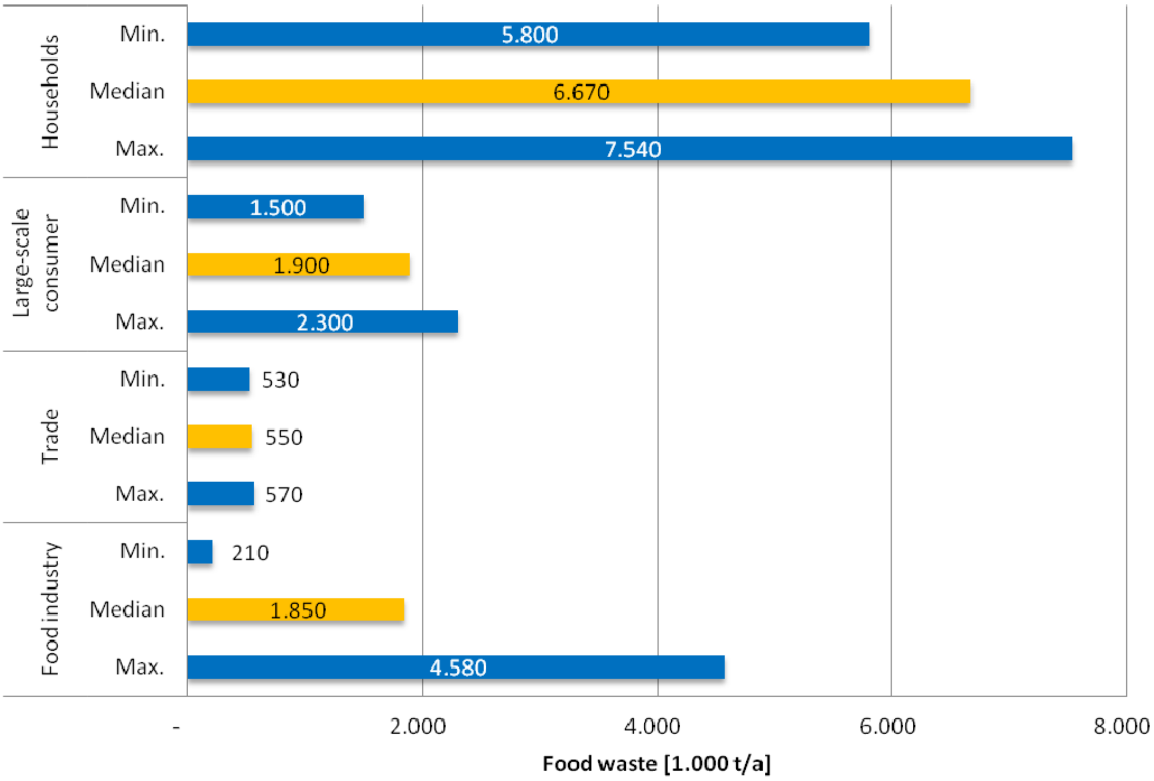

Figure 4: **Range of food waste in Germany by areas of the food value-added chain**

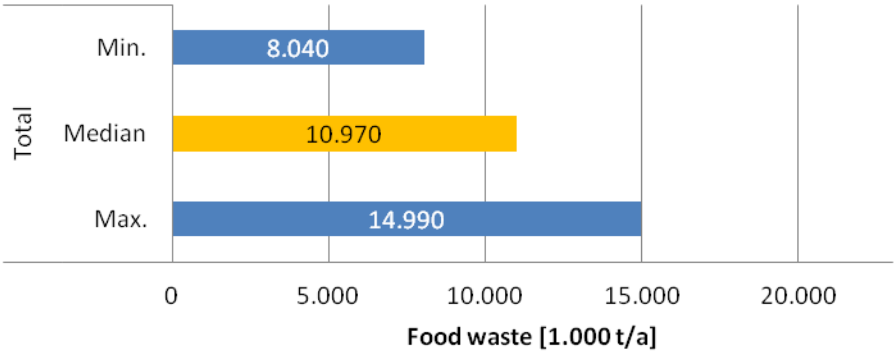

Figure 5: **Range of total food waste in Germany**

The percentage distribution of the calculated total amount is shown in Figure 6. One can see that of the analysed groups, households account for approximately two thirds of the amount of waste. The food industry and large-scale consumers each have a share of 17 percent. Compared with the other analysed groups, food waste from the trade sector accounts for only a small share of the total amount of waste. One must keep in mind that large parts of this amount are given to charities (cf. 2.2.2).

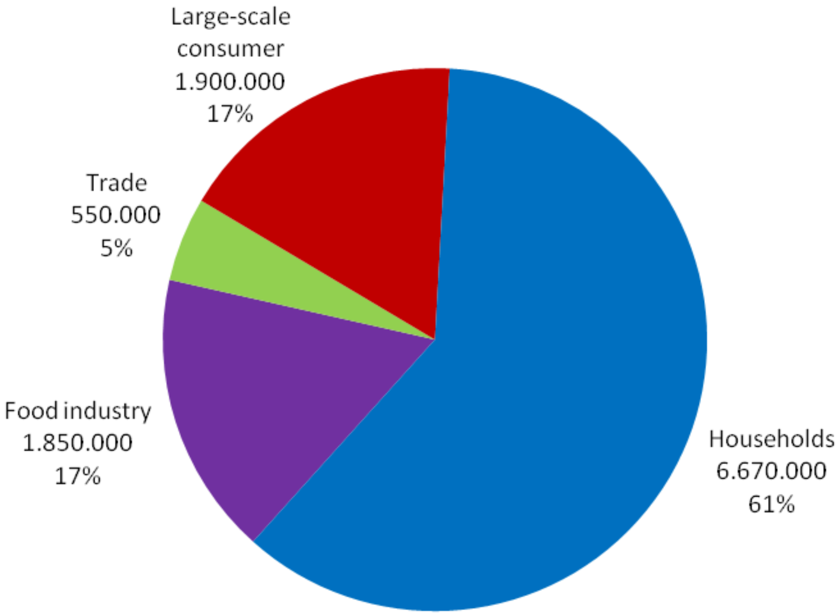

Figure 6: **Distribution of food waste by value chain sectors for food (median) [t/a]**

2.2.2. Food industry

By projecting and transferring the results from international studies the following fluctuation margin of the amounts of food waste from the industry emerges: 210,000 – 4,580,000 tonnes per year. The median value of this projection is 1,850,000 tonnes per year.

The fluctuation margin for industrial food waste is very large. This is due to the fact that the data basis is very unclear and that the industry has no clear definition of food waste. For instance, food waste given to third parties for utilization or fed to animals is not considered waste in some studies while it is included as such in others.

Some questionnaires were sent to trade associations and to food industry enterprises to be able to quantify the amounts mentioned above. Due to the low response rate it is impossible to calculate quantitative results.

From the available data material, however, qualitative findings can be deduced. Most enterprises surveyed stated that good and far-sighted operational planning should counter avoidable food waste caused by overproduction and bad planning. Yet, it is nearly impossible to avoid food waste at this stage of the value-added chain. The demand for food is not always constant. Also, retained samples are stored for quality assurance. Their purpose is primarily to guarantee flawless and safe condition of products at least until their best-before date. These foods are then disposed of. Production losses and faulty batches caused by technical problems constitute an additional source of waste.

2.2.3. Retail & Wholesale

Projecting and transferring the results from international studies also yields major fluctuation margins in the trade sector: 460,000 – 4,790,000 tonnes per year. The median value of this projection is 750,000 tonnes per year and exhibits the largest deviation to the fluctuation margin compared with other sectors. Thus the maximum value is more than five times higher than the median value, which suggests that these are outliers. The reason is a total loss of ten percent in American trade, which serves as the basis of this value. Other European studies and conversations with relevant stakeholders do not confirm such high loss rates for the German trade sector.

In addition to the calculations, it was intended to conduct a direct survey to estimate the amount of food waste in the trade sector. However, the answers to nearly all enquiries – of both retailers and the federal association – contained a reference to a previously published study by the EHI Retail Institute GmbH. It was therefore impossible to obtain additional data.

The study conducted by the EHI Retail Institute GmbH shows that the German food retail trade is affected by a turnover loss of 1.1 percent per year in lost food, which corresponds to approx. 310,000 tonnes per year (EHI Retail Institute GmbH, 2011). This amount does not contain food given to charities. The true food losses in the retail sector are thus higher and amount to approximately 500.000 tonnes per year. It is undisputed that giving food to charities and food banks is a meaningful way to avoid waste that can possibly be intensified.

The amount of waste established for the wholesale sector, for which wholesale markets were analysed by means of an example, was approximately 43,500 – 87,000 tonnes per year. This corresponds to approximately 0.5 percent of the amount handled each year. However, this fluctuation margins covers only organic waste and in some cases also includes plant and flower waste disposed of together with food. Due to this inhomogeneous composition of the amount of organic waste, the actual amount of food disposed of at German wholesale markets is likely to be smaller.

2.2.4. Large-scale consumers

The amount of waste from large-scale consumers has been calculated both in general and for the individual types of management among large-scale consumers. Wherever possible, several calculation approaches were stated within the individual types of management to be better able to assess the scale. A summary of the results for the individual management types and for large-scale consumers in general with the respective reference year can be found in the long version of the study.

The catering industry accounts for the largest share of food waste from large-scale consumers with its range having been calculated to be between 837,000 and 1,015,000 tonnes per year. However, it was only possible to apply one approach for the calculation of this range as further information on food waste per capita from literature varies greatly. The second largest amount of food waste among large-scale consumers comes from in-company catering with between 147,000 and 402,000 tonnes per year, followed by the accommodation sector with 186,000 tonnes per year. Retirement and nursing homes account for 93,000 to 145,000 tonnes of food waste per year, followed by schools with 75,000 to 87,000 tonnes per year and hospitals with 65,000 tonnes per year. Food waste from universities, day-care centres for children, prisons and the German armed forces (*Bundeswehr*) are estimated at less than 41,000 tonnes per year each.

The comparison of the results from the different calculation routes shows that the level of food waste from large-scale consumers gathered by adding the individual sectors and by the general projection across the entire sector is comparable. Total food waste is between 1,500,000 and 2,298,000 tonnes. The fluctuation margin results from the above-mentioned uncertainty related to the data and could be only be reduced by more in-depth studies.

It was not possible to estimate to which extent the food waste was avoidable or unavoidable on the basis of the literature for individual management types at hand. According to Müller (1998), approximately 56 percent of food waste from large-scale consumers is avoidable and approximately 48.5 percent according to information provided by Baier and Reinhard (2007). When applying the more current value of 48.5 percent, 756,600 to 1,114,530 tonnes of food waste from large-scale consumers would be avoidable.

2.2.5. Private households

In Germany an estimated 5.05 million tonnes of food waste is discarded via the municipal waste collection system each year, i.e. along with residual waste or in organic waste containers. This corresponds to 62 kg/(E*a) per capita. Approximately 70 percent of this amount is found in residual waste or in organic waste containers. However, this amount corresponds only to a small part of the total food waste as it is also disposed of through other routes such as self-composting, feeding to pets or in the sewer system. However, due to the uncertain data we can only roughly estimate the additional amounts being disposed of through these routes. It ranges between 0.75 and 2.49 million tonnes per year or between 9 and 30 kg/(E*a). It is not possible to make a reliable estimation as to how much of this amount is attributed to self-composting, sewer system and feeding.

The total amount of food waste from households in Germany per year is between 5.8 to 7.5 million tonnes. Taking the median value of this range in order to simplify, the annual amount of food waste is 6.7 tonnes or 81.6 kg/(E*a) of which about three quarters (76 percent) enter the municipal waste collection system.

Table 1: Avoidability of household food waste in Germany

	Percentage by weight	Per capita kg/(E*a)		Total Germany in million tonnes		Family of four kg/a	
		from	to	from	to	from	to
In the municipal collection system	100 %	61.8		5.05		247.2	
unavoidable	35 %	21.6		1.77		86.5	
partially avoidable	18 %	11.1		0.91		44.5	
avoidable	47 %	29.0		2.37		116.2	
Total (all disposal routes)	100 %	71.0	92.2	5.80	7.54	284.0	368.8
unavoidable	35 %	24.9	32.3	2.03	2.64	99.4	129.1
partially avoidable	18 %	12.8	16.6	1.04	1.36	51.1	66.4
avoidable	47 %	33.4	43.3	2.73	3.54	133.5	173.3
Σ avoidable and partially avoidable	65 %	46.2	59.9	3.8	4.9	184.6	239.7

Table 3 shows that 47 percent of food waste is avoidable and that 18 percent is partially avoidable. This corresponds to 2.37 million tons or 29 kg/(E*a) of avoidable food waste in the municipal collection system and 0.91 million tonnes or 11.1 kg/(E*a) of partially avoidable food waste. In addition there is waste being disposed of through other routes. Taking again the median value for the sake of simplicity, the total amount is 3.14 million tonnes or 38.4 kg/(E*a) of avoidable food waste and 1.2 million tonnes or 14.7 kg/(E*a) of partially avoidable food waste.
Figure 7 displays household food waste broken down by its avoidability according to Figure 2.

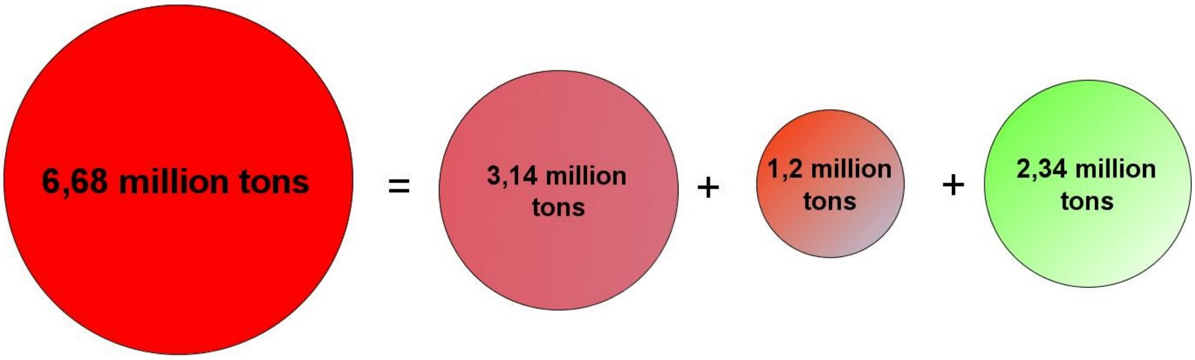

Figure 7: Amount of food waste from households in Germany

Due to a lack of sufficiently detailed and representative data for Germany, only an approximate estimation on the composition of food waste for each product group can be carried out (on the basis of data by Cofresco [2011] and Barabosz [2011]). Figure 8 shows the composition of avoidable and partially avoidable food waste. Fruits and vegetables make up the largest share of avoidable and partially avoidable food waste.

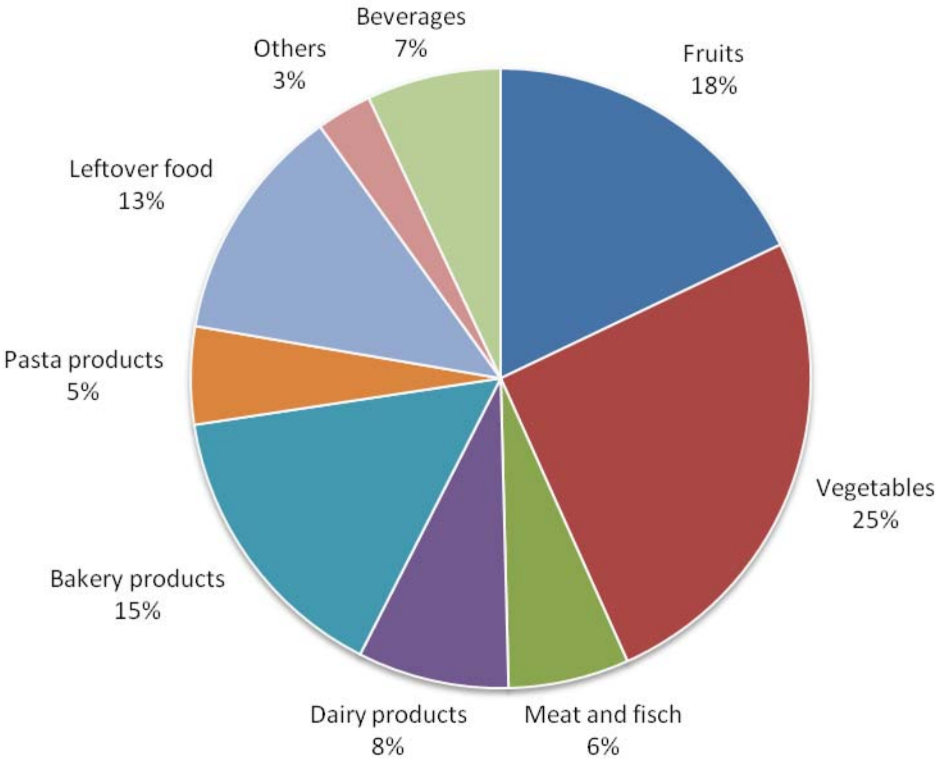

Figure 8: **Composition of avoidable and partially avoidable food waste from households in Germany by product group (percentage by weight)**

The amount of avoidable and partially avoidable food waste from households in Germany corresponds to a monetary value of 16.6 to 21.6 billion EUR per year or approx. 200 to 260 EUR per capita and year (cf. Table 4). For an average household of four, this means that each year avoidable and partially avoidable food waste worth approximately 935 EUR is disposed of as residual waste, in organic waste containers and the sewer system, through self-composting or fed to pets. Private consumer spending for food and non-alcoholic beverages by German households in 2010 totalled 151.14 billion EUR (Statistisches Bundesamt Deutschland, 2011). This means that avoidable and partially avoidable food waste accounts for between 10 and 14 percent of expenditures for food and non-alcoholic beverages.

Table 2: **Monetary value of avoidable and partially avoidable food waste from households in Germany**

Monetary value of avoidable and partially avoidable food waste	Per capita EUR/(E*a)		Total Germany in billion EUR		Family of four EUR/a	
Range	203.5	263.8	16.6	21.6	814	1,055
Mean value	233.7		19.1		934.6	

3.1 PROPOSALS FOR A MINIMIZATION OF FOOD WASTAGE

3.1. Strategies and concepts to reduce the amount of food discarded in other countries

With a view to working out proposals to reduce the amount of food thrown away in Germany and to point out options for action in order to implement various measures in the individual food chain sectors, the research team, first of all, researched existing measures at home and abroad. Existing measures were identified with the help of the literature and Internet research, through scientific exchange and expert consultations, and by interviewing individual initiators of preventive measures.

These include concrete preventive measures, scientific papers, political acts, technical products, and social initiatives (food distribution programmes).

Not all initiatives covered are aimed at reducing food waste. Individual projects pursue other objectives, as a matter of priority, such as a reduction of packaging waste in retail trade or sustainability campaigns in enterprises. Yet, they can result in a decrease in food waste by creating synergy effects. In this regard, pilot projects to optimise processes in the food industry or initiatives for sustainable consumption are worth mentioning, for example.

A total of 360 preventive measures were identified. These were classified, described and their results, if any, compiled. An assessment system (utility analysis) was developed in order to make preventive measures of the same category comparable. Particular regard was given to the transferability of the measures to Germany.

The results show 34 promising approaches, which are described in detail in the long version of this project. Compiled as a best practice catalogue, it identifies specific options for action and recommendations to prevent food waste.

3.2. Key Recommendations for action

Food waste should be permanently reduced at all stages of the food value-added chain. In the interest of sustainable consumption, the appreciation for food needs to be enhanced in large sections of the population. The recommendations for action provide food chain policy-makers, consumers, and stakeholders with reference points for decision-making when conceiving successful strategies for reducing food waste.

Drawing up a national roadmap represents a major step. This roadmap would foster the creation of the necessary framework conditions for preventing food waste in Germany.

First of all, objectives were articulated (along the same lines as the *Roadmap to a resource-efficient Europe: halving food waste by 2020* (European Commission, 2011). An agenda for action was devised as the next step. This agenda sets out measures that will help meet the objectives. Afterwards, a research agenda will be devised in order to close existing gaps in data and knowledge.

The active involvement of all relevant stakeholders in the food chain (agriculture, industry, trade, households, restaurant and catering sector, policy-makers, educational establishments, social institutions, etc.) is needed in order to halve avoidable food waste by 2020. A high level of acceptance and the involvement of all stakeholders can only be secured by objectifying the debate. This can be done, for one, by determining reliable figures through the research agenda and, secondly, by avoiding apportionment of blame.

Joint and expedient implementation of the preventive measures primarily requires a great willingness of all stakeholders to communicate and an open and trusting approach in the provision of experience, information, and knowhow. To this end, all stakeholders must engage in intensive networking throughout the food chain and transcend the limits of individual stages of the value-added chain. It is strongly recommended that round table talks will be held involving all relevant stakeholders in the value-added chain, e.g. farmers, producers, distributors, large-scale consumers (mass and institutional caterers, catering firms, hotel and restaurant industry), final customers, churches and environmental, educational and social welfare organisations (FOOD, SCP, 2009).

The farming sector, food industry, retail trade, hotel and restaurant industry, scientific community, and policy-makers can be brought together by initiating an Internet-based network for reducing food waste. Alongside an exchange on innovative technologies and solutions, the most innovative collaborative activities of participating businesses will be conferred with an award. Furthermore, the participants will benefit from the reciprocal exchange of experience and from neutral advice (No Waste Network, 2011).

The subject of food waste represents a major element towards increasing the regard in which foodstuffs are held. Platforms on more sustainability in the food chain are the key to coping with future challenges arising in the provision of food supplies (Foresight, 2011). We should strive for a more environment-friendly design of foods and greater transparency in the value-added chain (Verduurzaming Voedsel, 2009).

A database of best practice measures from the food chain, in the form of an open source, will enable the stakeholders to benefit from the experience gained from successfully implemented measures. In addition, such a portal can be used as an advertising medium for the enterprise's own projects and thus boost the image of the enterprise itself (Experiencebox, 2007).

Enterprises are called upon to take food waste into account in their environmental management systems and to identify it in their environmental statements. It should be examined whether food waste ought to be considered, in future, as a sub-criterion in various environmental certification schemes or when awarding environmental standards.

The subject matter should be integrated in training schemes in the fields of production, processing, trade, and catering trade. Incentives should be provided for holding of and staff participation in training schemes, advanced vocational training, and workshops. Staff members should be encouraged to act as disseminators and role models in the responsible handling of food.

In order to estimate and assess developments in the prevention of food waste and relevant action, constant collection of data or monitoring are essential. Based on scientific findings, these should be rapidly developed and introduced.

In spite of all the measures presented here, it will not be possible to completely prevent food waste. Even if a substantial reduction proves possible, foods will still be disposed of as waste. It is therefore absolutely necessary to leave the quantities generated in the substance cycle and to use their energy content, as appropriate. Depending on the suitability, food waste should be composted or used to produce renewable energy (biogas production). We should aim at expanding separate biowaste collection with a view to the recycling of food waste. This is also enshrined in the new Closed Substance Cycle and Waste Management Act (KrWG) (Deutscher Bundestag, 2012), which will make the separate collection of biowaste mandatory as of 1 January 2015.

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(175) FOOD WASTE FROM CANTEENS

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ABSTRACT

Since some years, one important topic of scientific work at the University of Stuttgart is the monitoring, evaluation and minimization of food waste. In this context ISWA-Institute at University of Stuttgart is cooperating in an international investigation project:

“GreenCook - transnational strategy for global sustainable food management”

GreenCook is elaborated in the frame of Interreg IVB NWE (North West Europe). The project-period is lasting from 2010 – 2013. Project-partners come from Belgium, France, Netherlands, UK and Germany.

GreenCook comprises –amongst others – pilot actions (living labs) with the intention to reduce food wastage. One of the ISWA-activities within GreenCook is the analysis of mass flows of food and the generation of food waste in canteens. Therefore as a first pilot study the university canteen at the largest university campus in Stuttgart (approximately 5000 meals per day) has been investigated. All relevant food and waste streams have been determined and balanced, e.g. input of food, storage, preparation, presentation / buffets, meal rests etc..

The meal rests on the plates – as well as rests from buffets etc. have been weighted and its composition was captured. The weighing of food waste has been supported by questionnaires, in order to verify reasons for wasting food.

As one important result, a input-output-balance of the investigated university-canteen could be elaborated, showing the relevant mass flows of food and food waste.

Avoidable waste streams could be identified and possible measures for a minimization of food waste where identified. Another very important outcome of this pilot action was the establishment of a monitoring system in the canteen, helping to verify the efficiency of implemented measures.

In addition to that, a standardized methodology for the investigation and monitoring of any individual canteen is currently elaborated and will be tested and calibrated within additional analysis in other university canteens in the metropolitan area of Stuttgart.

Within the oral presentation the adopted methodology for investigation, measuring, evaluation and monitoring of food waste in canteens will be explained. Results from implemented measures will be illuminated. Transferability to other canteens in the international context – as well as comparisons from similar analysis within GreenCook will be shown.

Finally the standardized methodology for the investigation and monitoring of any canteen will be demonstrated as well as first results from its application.

(209) PRELIMINARY ASSESSMENT OF FOOD WASTE GENERATION IN HOUSEHOLDS IN GREECE

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EXECUTIVE SUMMARY

According to Waste Framework Directive (2008/98/EC), waste prevention are the measures taken before a substance, material or product has become waste, that reduce the quantity of waste, the adverse impacts of the generated waste on environmental and human health or the content of harmful substances. Food waste is among the priority topics for waste prevention initiatives. Food waste occurs in nearly every household throughout the year. The amount of edibles discarded depends on several factors with varying significance for each household. For instance, socio-demographic backgrounds as well as consumption patterns or cooking habits are likely to influence the magnitude of food discarded. Food waste reduction is known to have a very large tonnage diversion potential, as it is the largest single waste stream in the municipal solid waste, while wasted food accounts for significant greenhouse gas emissions and consumer expenditure.

In order to account for the attitudes and behaviours of households in Greece towards food waste generation and prevention, a preliminary research was conducted. The research took place via the use of a structured questionnaire in the greater Athens area during February and March of 2012. The participants were selected randomly. The aim of our research was:

- To identify the attitudes of the respondents regarding food waste and food waste reduction.
- To identify certain consumer behaviour practices that can yield to the reduction of food that is thrown away
- To explore the effect that the sociodemographic variables have on the attitudes and the behaviour of the households on the generation as well as the prevention of food waste

The main results of our study indicate that 45% of the respondents replied that the amount of food that they throw away is small. More than half of those who said that they throw food away replied that they are very much disturbed by this fact and they try hard to avoid it. In terms of the amount of food waste, six out of ten of the respondents replied that it is less than the respective amount 12 months ago. Based on the responses, the main reason for that is the financial recession that Greece is facing.

Regarding their shopping habits aiming at reducing the amount of food waste generated, six out of ten replied that they are compiling a list before going for food shopping while one in two replied that they buy only what is necessary. This conservative behaviour is altered during the holiday periods: seven out of ten respondents replied that they cook a larger amount of food. However, more than 80% of them replied that they keep the extra amount of food and consume it in the days that follow. A major source of food waste is resulting from the food products that they are past their expiration dates. Our results indicate that milk, eggs and cheese are the items that consumers are very conscious about their expiration date since our results indicate that more than 80% of the respondents always check their expiration date. On the other hand, fruits are the food items that consumers are careless regarding their expiration date and thus it is more probable to be thrown away.

In conclusion, even though the research is still at its early stages, the preliminary results indicate that people in Greece have positive attitudes towards food waste prevention and that their habits, driven to a large extent by the financial recession, are close to the literature suggestions for reducing food waste generation. The results generated by this research will be of paramount importance since this research is the first one conducted in Greece in the field of food waste.

1 INTRODUCTION

According to Waste Framework Directive (2008/98/EC), waste prevention are the measures taken before a substance, material or product has become waste, that reduce the quantity of waste, the adverse impacts of the generated waste on environmental and human health or the content of harmful substances. The waste hierarchy has been applied as a guideline in waste prevention and management legislation and policy and prevention is addressed as its first priority.

Food waste is among the priority topics for waste prevention initiatives. Food waste occurs in nearly every household throughout the year. The amount of edibles discarded depends on several factors with varying significance for each household. For instance, socio-demographic backgrounds as well as consumption patterns or cooking habits are likely to influence the magnitude of food discarded. Overall, the international evidence suggests that waste prevention benefits will be derived from a ‘package’ of measures (Cox et al., 2010). Among the waste prevention measures modeled by Gentil et al. (2011), the prevention of food waste (in particular meat waste) has the highest benefits. Moreover, UK evidence suggests that the greatest tonnage diversions towards waste prevention can be achieved on food waste, home composting and bulky waste (Cox et al., 2010).

Food waste reduction is known to have a very large tonnage diversion potential, as it is the largest single waste stream in the municipal solid waste (MSW), while wasted food accounts for significant greenhouse gas (GHG) emissions and consumer expenditure. In the UK, where extensive research on the subject has been carried, it was found that one third of the food bought by households, of approximate value of €19 billions, becomes waste. Estimations indicate that up to 60% of this waste could theoretically be avoided (WRAP, 2009). It is very important to know if these characteristics are specific to the UK or extent to other countries.

Food waste is directly related to the organic fraction of the MSW. Food waste is biodegradable, being the biggest source of methane generation. An even bigger environmental problem is represented by the emissions of greenhouse gases and consumption of natural resources during the life cycle of food, i.e., its production, processing and transportation. Food waste reduction, as a waste prevention measure, will aid the compliance with the EU Landfill Directive (1999/31/EC), i.e., the progressive reduction of landfilling of biodegradable municipal waste to 35% of the 1995 level by 2020.

1.1 Research objectives

Based on the pivotal work by WRAP (2009) there is a large number of behaviours that can have a positive impact on food waste generation and, consequently, to the efforts towards food waste prevention. Such behaviours include meal planning, cupboard checking and list making before going for food shopping, proper storage of food items, use of food leftovers, cooking the right amount of food, and careful use of expiration date labels. Thus, the aim of our research was:

- To identify the attitudes of the respondents regarding food waste and food waste reduction.
- To identify certain consumer behaviour practices that can yield to the reduction of food that is thrown away
- To explore the effect that the sociodemographic variables have on the attitudes and the behaviour of the households on the generation as well as the prevention of food waste

2 METHODOLOGY

The research took place via the use of a structured questionnaire in the greater Athens area during February and March of 2012. Eighty six (86) participants were randomly selected and completed the questionnaire. The results of questionnaires give quantitative information like kind and frequency of behaviours and habits that result in wasted food and reasons for those habits on the basis of self reported behaviour of the respondents. Furthermore, information about interrelated conditions (e.g. level of employment, age of household members) and behaviour (e.g. buying, cooking and diet habits) can be gathered. Attitude and behaviour surveys are used in waste prevention efforts (Kurisu and Bartoleto, 2011; Sharp et al., 2010); they are used to set a baseline at first, and later to evaluate changes in attitude or behaviour (Sharp et al., 2010).

3 RESULTS

The demographic information of the sample is given in Table 1. The two columns on the left present the characteristics of the household; columns on the right present the characteristics of the respondents, respectively. The majority of the respondents have either a full time job or they are retired; they are mostly between 35-54 years of age; they hold a university or higher education degree; they live with their families in apartment buildings in homes that they own. 48% of the respondents are the ones mainly responsible for the food supplies of their households. Moreover, 63% of them are the ones mainly responsible for the preparation of food in their households.

TABLE 1 **Demographic characteristics of the sample**

Household characteristics	%	*Employment status*	%
Single person	17.4	Full time job	65.1
Roommates	5.8	Part – time job	3.5
Couple	15.1	Unemployed/Student	3.5
Family (w/ young children)	31.4	Freelancer	9.3
Family (all adults)	30.2	Retired	18.6
Home characteristics	%	*Educational background*	%
Single family home	25.6	Compulsory education	9.3
Apartment building	74.4	Secondary education	18.6
Own home	75.6	University	43.0
Rent home	24.4	Postgraduate studies	29.1
		Age	%
		18-34	20.9
		35-44	34.9
		45-54	27.9
		>55	15.3

3.1 Attitudes regarding food waste

The first section of the questionnaire was dealing with the attitudes of the respondents regarding the generation of food waste and the efforts or actions taken, if any, that aim at reducing food waste. The first question was “How much of the total food items – whether prepared/cooked or not – do you throw away into the bin?” 5.8% replied “a reasonable amount”, 12.8% replied “some”, 45.3% “a small amount”, 31.4 % “hardly any”, while 4.7% replied “none”.

The next question was “To which extent are you concerned or bothered about the amount of food waste that your household throws away?” 55.8% replied “a great deal”, 34.9% replied “a fair amount”, 7% “a little”, and finally 2.3% replied “not very much”.

The following question was “How much effort do you and your household go to in order to minimise the amount of uneaten food you throw away?” 44.7% of the respondents replied “a great deal”, 45.9% responded “a fair amount” while 8.2% replied “a little”.

TABLE 2 **“Which of following actions yielded the reduction of food waste that is thrown away during the past 12 months?”**

	%
I only buy food that is needed	54.4
I prepare carefully a weekly meal schedule	40.4
I try to buy food stuff, only after I consume whatever I have at home	40.4
I started cooking the leftovers from previous days	10.5
I estimate better the food portions that we consume	26.3
I buy more local/Greek food products	17.5
I judge better how long it takes for a food item to rotten	10.5
I use better the food labels “best before” and “expiration date”	10.5
I make better use of the freezer	19.3
I make a list before going for food shopping	63.2
I check the cupboards at home before going for food shopping	52.6

The next question asked the respondents to compare the current amount of uneaten food that is thrown away with the respective amount 12 months ago. 32.6% replied that the amount remained the same, 20.9% replied that the food thrown way decreased a little, while 45.3% replied that the amount thrown away decreased a lot.

The next question is aiming at identifying the actions that yielded the difference in the amount of food that is thrown away reported in the previous question (see Table 2). Making a list before going for food shopping is the most popular action (63.2), followed by buying only the necessary food (54.4%) and checking the cupboards before going for food shopping (52.6%).

The next question aimed at identifying which was the driving force behind the actions described in the previous question. Interestingly enough, more than 60% of the respondents mentioned that the financial recession that Greece is facing is the main reason that forced them to take the action mentioned previously. The reason that ranked second was the change in the consumer patterns, while the third reason reported was a change towards a more environmentally friendly lifestyle.

3.2 Consumer behaviour towards fresh food products

The next set of questions was dealing with the consumer behaviour regarding fresh products. The first question was “From which source are you buying the food supplies for the everyday needs of your household?” As shown in Figure 1, supermarkets are the main source for buying food followed by the open markets, which are very popular in Greece. Interestingly enough, internet buys of food stuff is not an option for the participants. The next question was along the same lines “Which source of food supply do you trust the most?” Supermarkets followed by the open markets are the most trusted options (see Figure 2).

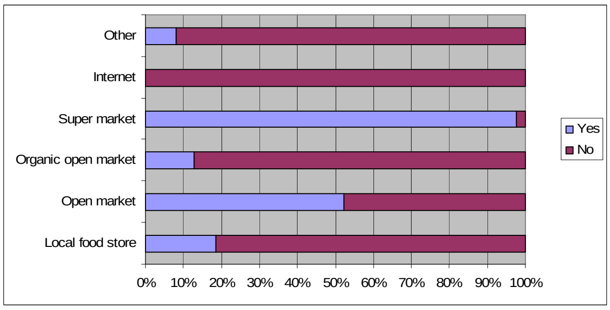

FIGURE 1 **“Where from are you buying food needed for everyday cooking”**

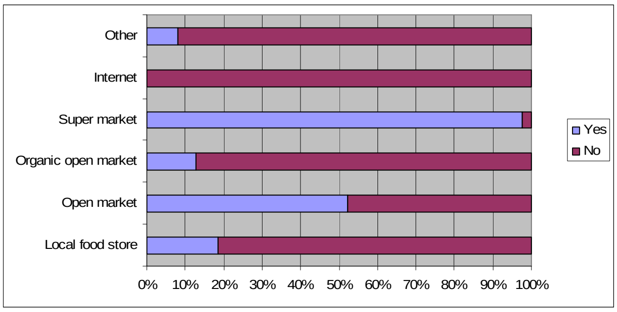

FIGURE 2 **“Which source of food supply do you trust the most?”**

The next question was dealing with certain behaviours that yield to lower food waste generation. Checking the cupboards in order to know exactly what kind of food supplies are at home is the most popular option, as 90% of the respondents do it always or very often (Figure 3). Making a list of the necessary supplies is the next best option while the exact meal programming for the next week is the least preferred option among the three.

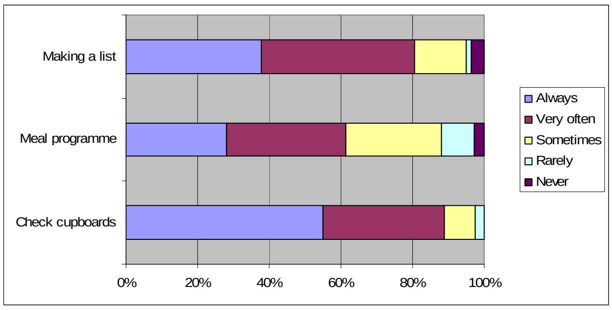

FIGURE 3 **“Before buying food, which of the following are you doing?”**

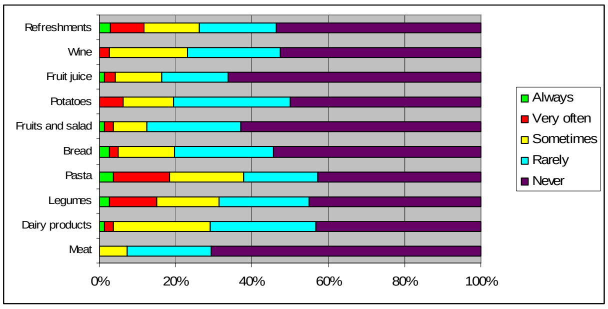

FIGURE 4 **“To which extent are you buying the following items, offered in better prices because they are close to their expiration date?”**

The next question aimed to identify which is the impact of lower prices on the supply of food. The results presented in Figure 4 show that the food items which are more likely to be bought close to their expiration date, because they are offered at better prices, are pasta, legumes and dairy products. On the other hand, meat, fruits, vegetables, bread and potatoes are not bought by the consumers close to their expiration date, even thought they are offered in better prices.

On a relevant question, more than 90% of the respondents never or rarely throw away fresh food supplies because they don’t like it; In addition, more that 75% of the respondents never or rarely throw away fresh food items because of their expiration date. Both of these latter numbers are indicative of the careful consideration for fresh food supplies, both in terms of quantity as well as quality, by the households.

A common practice in Greece is the cultivation of fruits, vegetables or both by consumers. Approximately 25% of the respondents mentioned that they have access to cultivated fresh fruits and/or vegetables. Of those, 29% have access to both fruits and vegetables, 17% to just vegetables while 4% have access to just fruits. These fruits and vegetables are coming mainly (56%) from cultivated land or small gardens (27%).

3.3 Consumer habits during food storage

The next set of questions was dealing with consumer behaviour during food storage. The first question was “How do you preserve the following food items after opening?” The results are presented in Figure 5. Fresh meat and fish, sausages, cheese, juices, wine and cooking oil are preserved mostly air tight closed, while fruits, potatoes and vegetables are kept in their opened package. The next question was “How do you store the following food items?” The results are presented in Figure 6 respectively. Bread, potatoes and fruits are mostly stored in open spaces while meat, fish, sausages, cheese, juices and food leftovers are refrigerated.

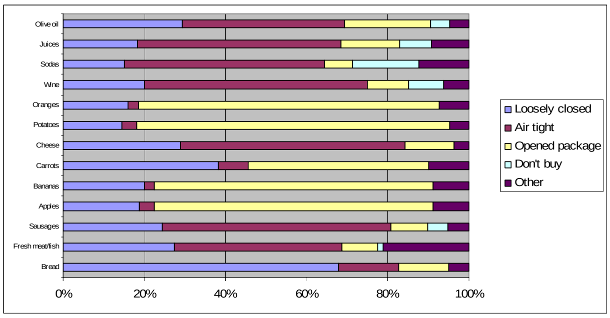

FIGURE 5 **“How do you preserve the following food items after opening?”**

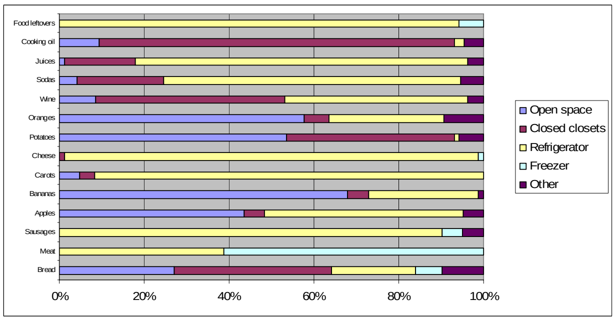

FIGURE 6 **“How do you store the following food items?”**

The next question was dealing with the expiration date stamp found on the food items. The results for various food items are presented in Figure 7. Milk, eggs and cheese are the items that consumers are very conscious about their expiration date since our results indicate that more than 80% of the respondents always check their expiration date. On the other hand, fruits are the food items that consumers are careless regarding their expiration date.

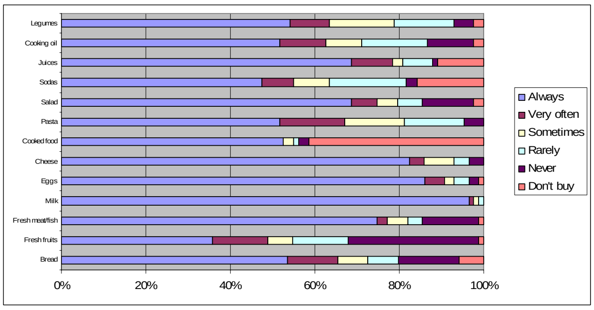

FIGURE 7 **“How often do you check the expiration date stamp?”**

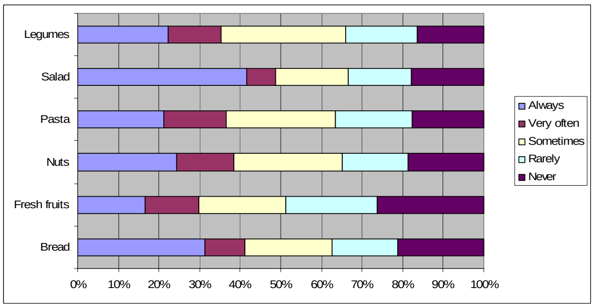

FIGURE 8 **“How often do you throw away food items which are past their “best before” date but don’t have any signs of deterioration?”**

Figure 8 presents the responses to the next question. Salad, bread and nuts are the food items more frequently thrown away if they are past their “best before” date, even though they don’t present any obvious sign of deterioration. On the other hand, fresh fruits, legumes and pasta are those items kept past their “best before” date. Approximately 20% of the respondents always throw away the food items if they are past their “best before” date. The respective percentage for salad is doubled, probably because salad is a food item that is preferred to be eaten fresh

3.4 Food preparation at home

The next couple of questions was dealing with certain options regarding the preparation of food at home. Table 3 presents the frequency of those options. Based on our results, eating home with friends seems to be a very frequent habit. Approximately four out of ten are eating out or ordering food at least 1-2 times per week. Also, one in three respondents with a frequency of 1-2 times per week is either receiving cooked food from their parents or are cooking for others, including their parents. Figure 9 presents the comparison in the frequency of the given options compared to those 12 months ago. It is evident that the frequency of eating out, ordering food and buying pre-cooked food has been reduced, probably due to the financial recession. On the positive side, the frequency of family members sharing the same meal, cooking for friends and relatives, and getting cooked food from parents has been increased.

TABLE 3 **How often are you…**

	5-7 times/week	3-4 times/week	1-2 times/week	Less than once a week	Never
Eating out or ordering delivery (%)	14.3	17.9	38.1	25.0	4.8
Eating pre-cooked food (%)	0.0	6.1	23.2	58.5	12.2
Eating the same food with all family members (%)	0.0	0.0	11.0	25.6	63.4
Eating at home with friends (%)	67.5	26.3	3.8	2.5	0.0
Taking food from parents (%)	0.0	2.4	29.4	58.8	9.4
Cooking for others (%)	2.5	7.5	26.3	25.0	38.8

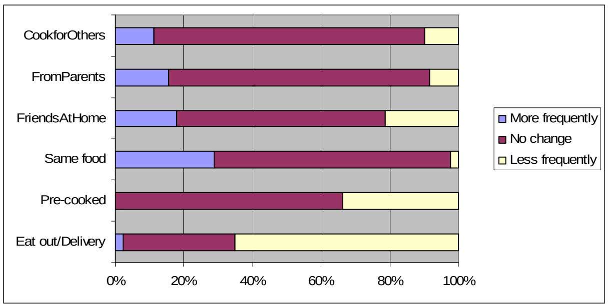

FIGURE 9 **"Compare the frequency of the given options with those 12 months ago"**

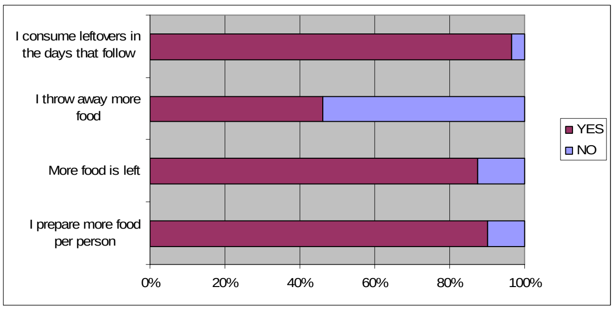

FIGURE 10 **"Which of the following sentences are representative of your behaviour during the holiday season?"**

Because holiday periods are notorious for over-consumption of resources of all kinds, there is a question regarding the behaviour of the household during the holidays (e.g. birthday parties, Christmas, etc.). From the results presented in Figure 10, it is evident that more food is prepared, left and discarded during the festive periods. The following question was "How often do you discard cooked food from the following items?" The results are presented in Figure 11. Salad, bread and rice are the three food items that are discarded the most. In the final question of this section, approximately 60% of the respondents replied that they are always or most of the times thinking that the size of the portion is too large, when cooking or serving a meal (see Figure 12).

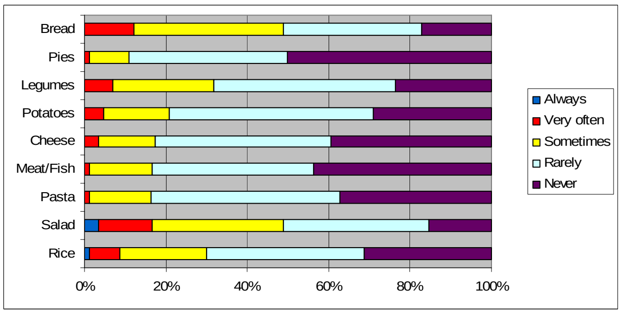

FIGURE 11 **"How often do you discard cooked food from the following items?"**

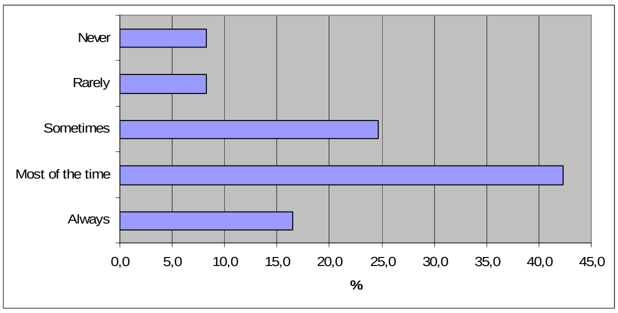

FIGURE 12 **"When cooking or serving a meal, how often are you thinking that the portion size is too large?"**

3.5 Food waste management

Regarding the management of food waste, the leftovers from the plates are discarded in the waste bin while the food left in the kitchenware (e.g. saucepans) is kept in the refrigerator. Of those that keep the food left, 80% consume it later. The next set of questions was dealing with the identification of the disposal routes of the food waste. Food items were classified in three major categories: (i) cooked food, (ii) fruits and vegetables, and (iii) liquid food waste (e.g. milk, coffee, tea, juices). Our results indicate that the major disposal route for cooked food is the waste bin followed by its use as animal feed. For fruits and vegetables the major disposal route is again the waste bin. Regarding the food waste in liquid form, it ends up in the drain as expected. 90% of the respondents had never tried to compost any food waste. Of those who tried composting, the waste stream in focus was the leftovers from fruits and vegetables.

4 DISCUSSION

The results presented in this paper are the preliminary output of an ongoing research effort that aims to identify the attitudes and behaviour of consumers in Greece regarding food waste generation and prevention. Even though the sample presented here is quite small, the sociodemographic profile of the participants is representative of the contemporary societal picture in the country: they are people who either have a full-time job or they are retired; they are highly educated and live with their families in apartments that they own. Half of the respondents have the sole responsibility of buying the food products for their households while 63% have the responsibility of preparing the everyday food. Thus, their expressed attitudes and behaviour via the questionnaires are valid towards fulfilling the aims of our research.

Regarding their attitudes, 45% of them replied that the amount of food that they throw away is small. More than half of those who said that they throw food away replied that they are very much disturbed by this fact and they try hard to avoid it. In terms of the amount of food waste, six out of ten of the respondents replied that it is less than the respective amount 12 months ago. Based on the responses, the main reason for that is the financial recession that Greece is facing. Overall, the expressed attitudes can be characterised as very positive since the households try to minimise the amount of food that is thrown away.

Regarding their shopping habits aiming at reducing the amount of food waste generated, six out of ten replied that they are compiling a list before going for food shopping while one in two replied that they buy only what is necessary. Both of these statements are positive towards preventing the generation of food waste. Moving on to the fresh food products, which are very fragile and thus very prone to be wasted, most of the respondents buy them from the super markets or the open-air markets due to the fact that those are the two most trusted sources of fresh food supply.

Proper food storage is a key issue in avoiding food waste. In terms of consumer habits for food storage, bread, potatoes and fruits are mostly stored in open spaces while meat, fish, sausages, cheese, juices and food leftovers are refrigerated. Fresh meat and fish, sausages, cheese, juices, wine and cooking oil are preserved mostly air tight closed, while fruits, potatoes and vegetables are kept in their opened package.

A major source of food waste is resulting from the food products that they are past their expiration dates. Our results indicate that milk, eggs and cheese are the items that consumers are very conscious about their expiration date since our results indicate that more than 80% of the respondents always check their expiration date. On the other hand, fruits are the food items that consumers are careless regarding their expiration date and thus it is more probable to be thrown away.

Regarding food preparation habits, our research reveals that the frequency of eating out, ordering food and buying pre-cooked food has been reduced compared to 12 months ago, probably due to the financial recession. On the other hand, the frequency of family members and friends having meals at home has been increased. Both of these results indicate that people prefer to prepare the meals for their family at home. One more aspect that we looked into was the consumer behaviour during the holiday periods. Seven out of ten respondents replied that they cook a larger amount of food. However, more than 80% of them replied that they keep the extra amount of food and consume it in the days that follow.

Finally, our results indicate that the major disposal route for cooked food is the waste bin followed by its use as animal feed. For fruits and vegetables the major disposal route is again the waste bin. Regarding the food waste in liquid form, it ends up in the drain as expected. 90% of the respondents had never tried to compost any food waste. Thus, there is a great opportunity for the diversification of the food waste stream towards composting. However, major efforts are required for the development of the necessary infrastructure and the training of the consumers.

5 CONCLUSIONS

The results of a quantitative research, conducted via the use of a structured questionnaire in order to identify the attitudes and behaviours of households in Greece regarding the food waste generation and reduction, have been presented. Even though the research is still at its early stages, the preliminary results indicate that people in Greece have positive attitudes towards food waste prevention and that their habits, driven to a large extent by the financial recession, are close to the literature suggestions for reducing food waste generation. Once the research is completed, further exploration on the effect of the sociodemographic characteristics of the respondents on their attitudes and habits will be conducted in order to identify possible policy suggestions aiming at preventing food waste generation by the households in Greece.

The results generated by this research will be of paramount importance since this research is the first one conducted in Greece in the field of food waste. Moreover, the results will serve as a yardstick in order to establish and promote waste prevention activities for households, an action that is expected to produce positive environmental and financial results in both the household level in addition to the overall waste management system in Greece.

6 ACKNOWLEDGEMENTS

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(229) STATISTICAL ANALYSIS OF RESIDENTIAL FOOD WASTE CAPTURE RATES IN ITALY AT MUNICIPAL AND REGIONAL LEVEL

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EXECUTIVE SUMMARY

This paper analyses capture rates of source separated food waste, focusing on data investigated from official public waste reports of Italian Local Authorities and Waste Monitoring Agencies, which show details at the level of the individual municipalities.

Source separation of residential food waste, performed with the typical Italian scheme (small kitchen buckets with compostable bags) has spread throughout the country at a steady pace since 1998; currently, almost half of the municipalities in Italy have implemented this kind of collection of residential food waste.

Today, it’s possible to outline a detailed overview of the performances of this system in different areas of the country.

As regional averages include municipalities not collecting residential food waste, they are not representative of the many local best practices; thus, a statistical analysis has been performed on single municipal data considering just those collecting residential food waste.

The statistical evaluation has been performed on a data set including 2.335 municipalities, equalling about 20 million inhabitants and 54% of total food waste collected in Italy, using two different indicators (kg/inhabitant and percentage on total waste collected).

The results show that in some regions and provinces residential food waste collection has already reached a defined target, while in other there are still huge possibilities of optimization.

In this work, the average potential and target for northern Italy has been defined as 15% of total municipal waste collected, with many best practices going well beyond that. For the southern regions this potential increases to 25%, with many municipalities easily achieving 30% of overall MSW collected. In particular, the Sardinia Region could possibly become a leading case study for many Mediterranean regions, with more than 30% food waste captured out of the total MSW and the introduction of specific “stick and carrot” regulations.

These average target levels, statistically derived, give an overview of future possibilities of improvement, both in areas historically involved in this kind of collection and in those which have recently implemented it. In Italy, 2.200.000 t of residential food waste have been collected in 2009; according to the trends and targets outlined, source separation of residential food waste could get to more than 6 million tonnes, paving the way for 100 new composting or anaerobic digestion facilities.

1 INTRODUCTION

1.1 Background

Source separation of organics plays a pivotal role in an integrated waste management system.

In Italy, this kind of collection was developed as an intensive scheme aimed at keeping separate food waste and yard waste out of landfills. First implemented in the middle of the 90s, the most effective scheme is based on the use of 8 to 10 liter buckets with certified compostable bags to be kept in the kitchen, and curbside collection based on small vehicles with a typical frequency of two times a week.

FIGURE 1 **A key feature of the Italian food waste collection scheme: collection without yard waste, with small compostable bags certified according to the European standard EN 13432**

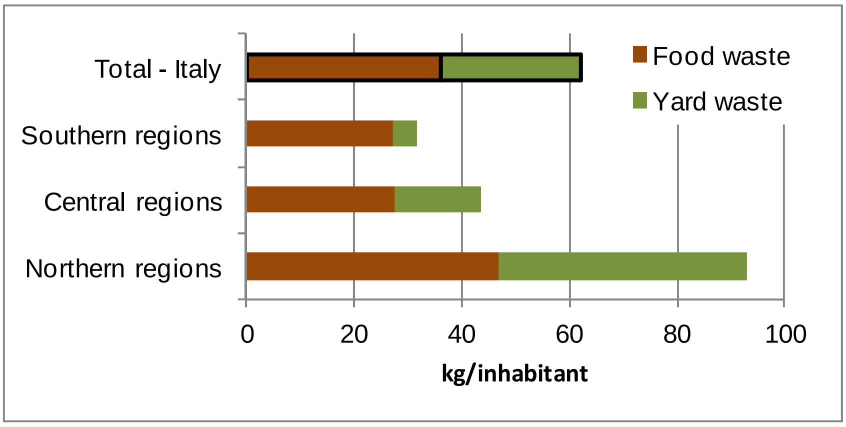
At first it was well implemented in the northern regions, where lack of landfills and increasing disposal prices quickly acted as strong drivers to start this new kind of collection. Now, the Italian model of intensive residential food waste collection achieves impressive diversion rates without higher costs [i], and is catching on in Spain and the UK [ii].

In 2009, in Italy 3.743.719 tonnes of residential organics (food waste plus yard waste) were collected, averaging 62,0 kg/inhabitant, with strong differences in the three macro areas of the country, as shown in tab. 1. and in fig. 2.

TABLE 1 **Source separation of residential organics in Italy, 2009.**

		Food waste		**Yard waste**		**Total organics**	
	Inhabitants	**Total (t)**	**kg/ inhabitant**	**Total (t)**	**kg/ inhabitant**	**Total (t)**	**kg/ inhabitant**
Northern regions	27.568.435	1.290.122	46,8	1.276.528	46,3	2.566.651	93,1
Central regions	11.890.464	327.654	27,6	189.579	15,9	517.234	43,5
Southern regions	20.881.429	565.771	27,1	94.064	4,5	659.835	31,6
Total - Italy	*60.340.328*	*2.183.547*	*36,2*	*1.560.171*	*25,9*	*3.743.719*	*62,0*

FIGURE 2 **Average captures of residential food waste and yard waste in the three macro areas in Italy**



These two fractions are now gained the largest portion of recyclables collected, and the quantity of compost produced (1.400.000 tons in 281 composting plants [iii]) has pushed the development of markets, legislation, quality and assurance schemes, with an important role played by the national Composting Association (CIC, Consorzio Italiano Compostatori).

1.2 Data source

This study targets food waste collection specifically. As stated above, this is widespread in Italy, particularly in northern regions, but there's still a lack of official national statistics that provide detailed quantities of food waste, which are needed in order to make comparisons at a small scale between municipalities in different parts of the country.

The main source of data for municipal waste in Italy is the annual report published by the National Environmental Agency (ISPRA, Rapporto Rifiuti). In this report data is aggregated at a provincial and regional level, which is indeed a good amount of detail; in Italy there are 110 Provinces, with an average of 551.000 inhabitants/province, and 20 Regions, averaging 3.030.000 inhabitants/region.

The only drawback is that this report usually publishes aggregated data on "organics", combining the amount of food waste and garden waste collected. This approach might be appropriate in central European countries like Germany, where the collection of food waste is co-mingled with yard waste, but the evaluation of the typical Italian ISSO (Intensive Source Separation of Organics) scheme requires these quantities to be acknowledged separately.

Fortunately, on the last available report, which addresses data from 2009, there is an annex with more detailed information and separate columns for food waste and yard waste. So it was possible to perform some comparison and evaluation at a provincial and regional level.

The "real numbers" about capture rates come only when going deeper, looking at data related to single municipalities. In Italy we have 8.092 municipalities, and typically the implementation of food waste collection doesn't happen at the same time inside a Region or a Province, because even if there are regional and national recycling targets to achieve, often each local authority has its own contract with a waste collection company and decides individually on the collection scheme to be adopted in its territory.

Because of this, averaging the collection rates at a provincial and regional level often "flattens" the number by including municipalities which don't have ISSO schemes implemented.

With this work, extensive data mining has been performed on various reports published by local authorities at a provincial or regional scale, and available online or officially in other ways; it was then possible to build solid statistics based on a large number of local authorities actually collecting food waste separately.

From this analysis some important indicators about capture rates and targets for food waste were extracted.

The local data sets were analyzed, extracting numbers and indicators from the tables shown and gathering them in a single large database. They were selected by looking primarily at the following features:

- Detailed data at municipal level;
- Evidence of specific food waste collection, overall municipal waste, total separate collection, inhabitants;
- Presence of enough municipalities collecting food waste in the area covered by the single data set;
- Availability on the internet with tables in pdf or xls format.

The main source of this detailed data has been the reports published yearly by many Provincial Waste Monitoring Offices (OPR, Osservatori Provinciali Rifiuti) or by Regional Waste Authorities [iv],[v],[vi],[vii], partly by asking for data from some public bodies [viii]. Table 2 lists the area covered by the database that was generated.

The purpose of this data processing is specifically to evaluate food waste captures in municipalities that already have reached a “steady state” having well implemented source separation (see filtering criteria in chapter 2.2), so it is correct to merge databases that are not strictly related to the same reference year. Anyway, if multiple time series were available, the latest year was used, as displayed in table 2.

TABLE 2 **Areas covered by this study**

Area	Years monitored	Number of municipalities with food waste collection (most recent data)	Number of inhabitants collecting food waste	Total captures per year (t)
Provincia di Salerno	2006, 2007	86	884.136	57.467
Regione Marche	2010	99	991.106	35.554
Provincia di Torino	2001-2009	265	2.262.570	140.586
Regione Sardegna	2005-2009	370	1.642.358	151.012
Regione Veneto	2010	555	4.896.576	347.008
Regione Lombardia	1998-2009	896	8.265.861	417.497
Provincia di Rimini	2005-2009	17	347.183	18.908
Provincia di Benevento	2009	47	147.064	8.299
TOTAL		**2.335**	**19.436.854**	**1.176.332**
% on overall Italy		**28,8%**	**33,0%**	**53,9%**

Food waste collection has a long history in the northern regions compared to some southern situations, like Sardinia Region, but the results are very promising, so they were included in the analysis.

It is noteworthy how the selected sample is representative of the whole country, as it involves almost 20.000.000 inhabitants; moreover, as per total quantities of food waste collected, the database covers 54% of total national food waste.

2 METHODOLOGY

2.1 Choice of the correct indicator

In order to compare municipalities with different population size, the specific per capita indicator is the common choice. It must be pointed out that it is biased to some extent, because it’s calculated on the number of residents; thus comparing municipalities with different non-resident populations (tourists, commuters) would supply unreal results.

Another indicator was chosen to overcome this problem: namely, the percentage of food waste out of total municipal solid waste collected. This way, it doesn’t matter if food waste is collected from residents or non-resident citizens. Specific captures in kg/inhabitants were still calculated in order to make comparisons with international reports.

2.2 Removal of biased values

Evaluating the sample, a possible biasing factor could occur including municipalities with partial participation of food waste collection, in terms of:

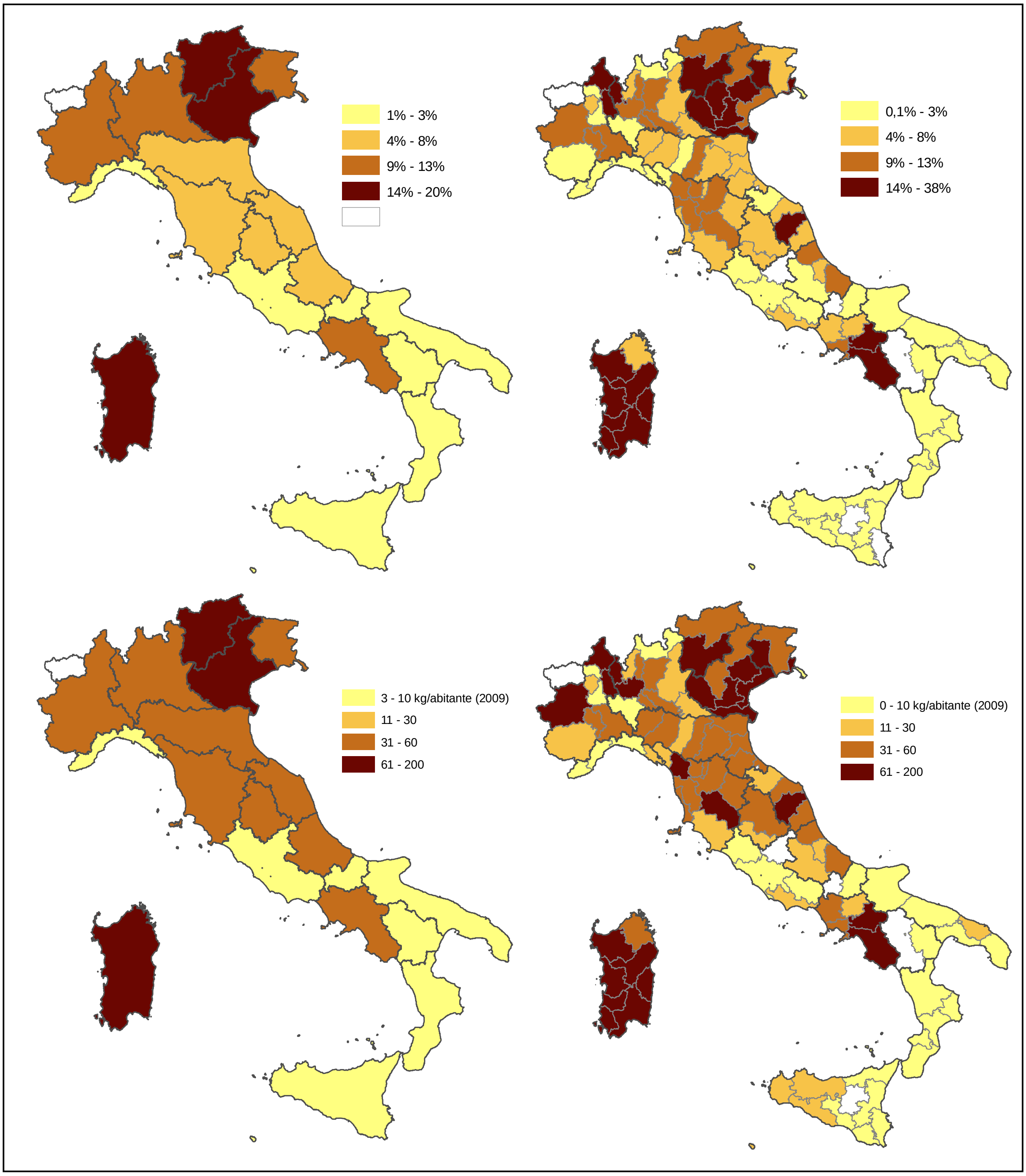
- Time, i.e. those which started initial food waste collection during the year considered in the database;
- Population, i.e. with only a part of citizens involved in this kind of collection.

Including the above mentioned cases, the average capture rate calculated based on the whole year could be biased; to avoid this problem, a minimum threshold of 3% food waste on total MSW was required in order to include them in the database as municipalities performing food waste collection.

3 RESULTS

3.1 National survey: provinces and regions

FIGURE 3 **Specific captures of food waste in Italy, year 2009. The two top maps are plotted with food waste as percentage of total MSW; at the bottom, maps show kg/inhabitant.year values.**



Maps shown in figure 3 give a snapshot of the current situation in terms of specific food waste captures in Italy. It's clear that when averaged based on Regions, captures aren't representative of the municipalities that are actually collecting food waste efficiently. Looking at averages on Provinces, the deeper detail gives more information, especially in Regions like Lombardy where there are significant differences in terms of implementation of food waste collection.

It can be pointed out that:

- **Regional averages** give a true overview only in Regions where food waste collection is widespread and has been established for many years (e.g. Veneto)
- **Provincial averages** highlight territories performing as best practices in Regions where food waste is still in an early implementation phase (e.g. Province of Macerata – 16,9%, in Region Marche – 7,0%)
- In order to define feasible targets to be achieved by implementing food waste collection, we must look at statistic on **single municipalities**, calculating averages **only** on those who collect food waste.

3.2 Detailed survey on municipalities

Normal (Gaussian) distribution has been calculated on every set of municipal data in order to evaluate the average capture rate and the dispersion of single values around it.

A higher and sharper peak in the plot of a normal distribution represents a province or region where every single municipality has almost the same capture rate of food waste, indicating a widespread and well established collection scheme, with few modifications with respect to a certain standard. For instance, this is the situation in Lombardy and Veneto.

A flatter and larger Gaussian plot indicates a system still in an evolution phase, with important feasible improvements that can be done, like in Salerno province or in Sardinia region.

In figure 6, values are represented with Box-Whisker plots showing the minimum, maximum, 1st quartile, average, 3rd quartile. The outcome of this chart is that central and especially southern regions have higher captures of food waste comparing with the northern ones.

This was already stated in literature [ix], and it mainly relates to regional habits and traditions such as more home cooking than the northern regions, with meals richer in vegetables and fruit, but is highlighted with a statistical evidence in this study. In Sardinia region, and in provinces such as Salerno and Benevento, values of 20-30% of food waste in total MSW are quite common, while in northern regions they are reached only by the best performing municipalities.

FIGURE 4 **Normal distribution of food waste captures in the local datasets considered (% of total MSW)**

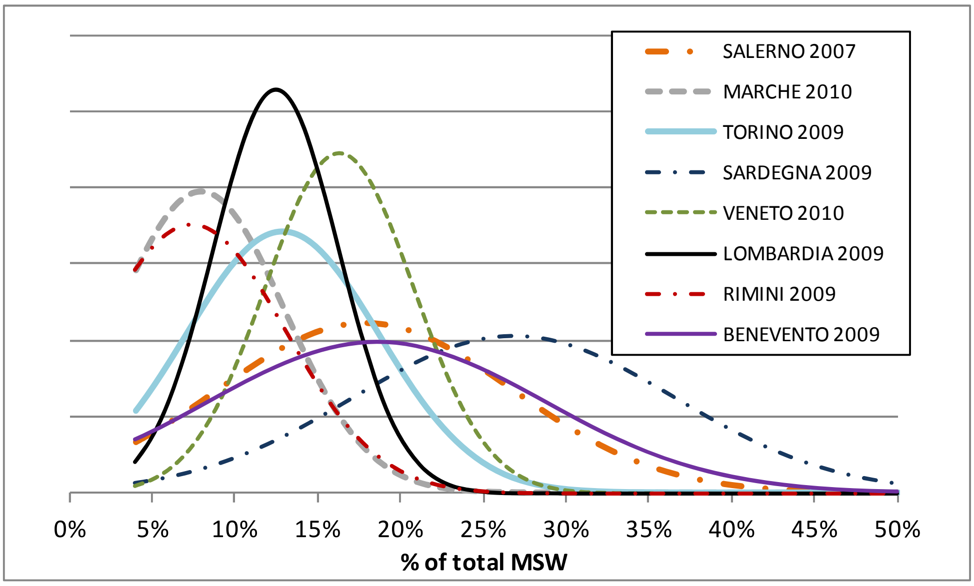


FIGURE 5 **Normal distribution of food waste captures in the local datasets considered (kg/inhabitants.year)**

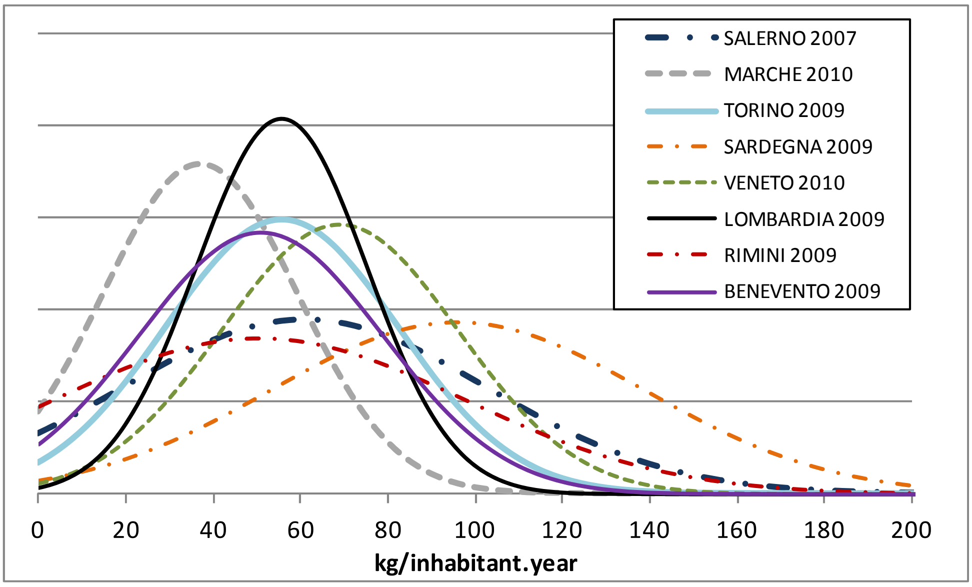
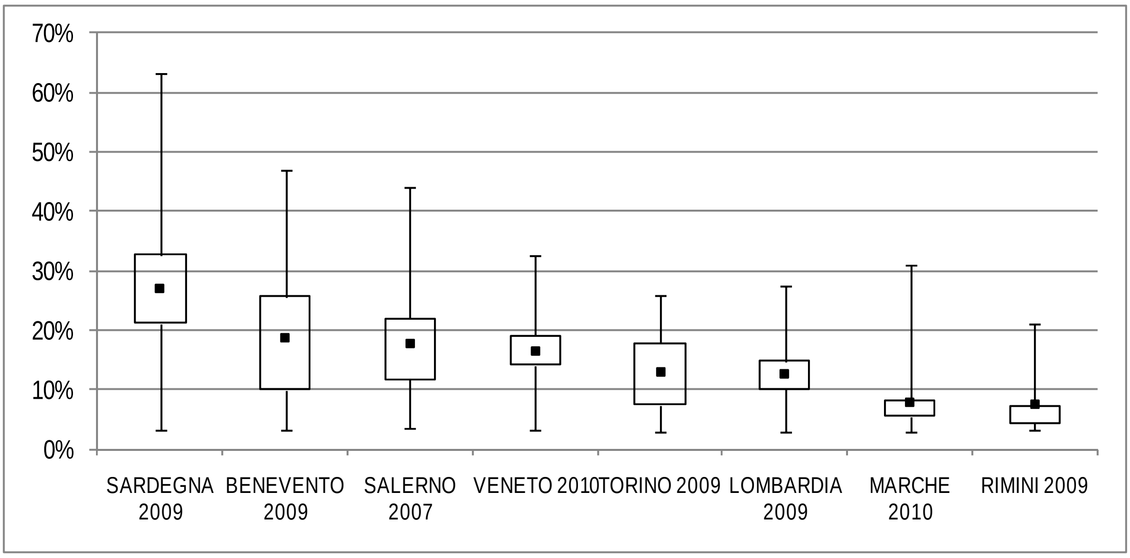


FIGURE 6 **Food waste captures in the local datasets considered (% of total MSW). Box-Whisker plots showing the minimum, maximum, 1st quartile, average, 3rd quartile.**



3.3 Extrapolation of targets for food waste captures

It’s clear that average targets to be set as a reference must be different in northern and southern regions. A general target could be 15% of total MSW in the north, 25% in the south. Expressed in kg/inhabitant, this is about 75 in the north and 125 in the south.

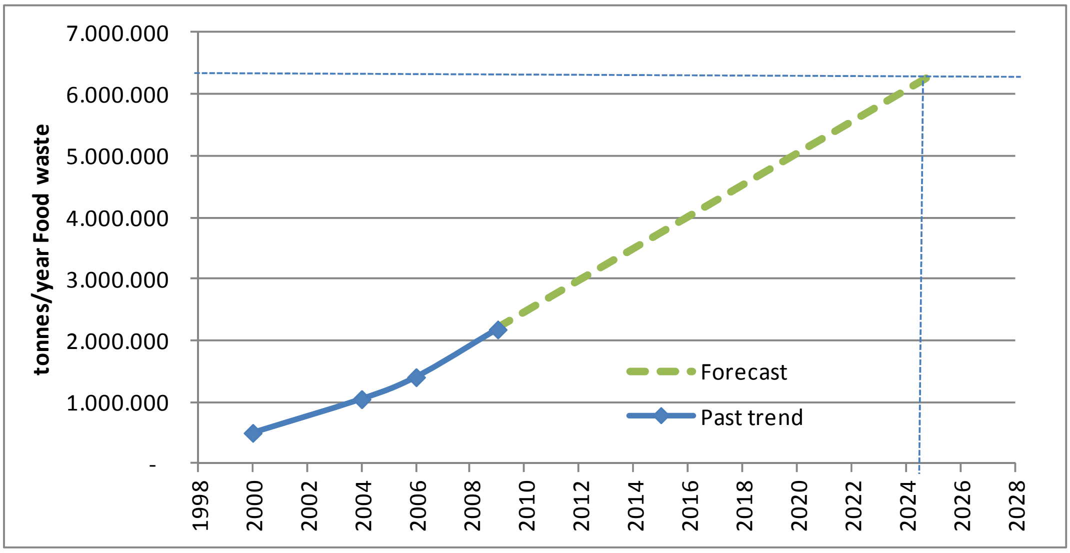
It must be pointed out that these target are to be considered as realistic and feasible, as they are based on averages; municipalities performing as best practices could easily go up to 20% in the north and 35% in the south.

Applying these targets at a national scale, it appears that food waste collection could go up to more than 6.000.000 tonnes, almost three times the current capture. Looking at the past trend in food waste collection, using a simple linear extrapolation it seems this target could be reached in 12 years from now, by 2024, but recent drivers could make us reach the goal sooner.

TABLE 3 **Targets for food waste capture at a national scale**

	Total MSW (tonnes 2009)	Total Food waste captured (tonnes 2009)	Target food waste capture (% on MSW)	Target food waste capture (tonnes)	% of target food waste currently captured
Northern regions	18.474.196	1.569.842	15%	2.771.129	56,6%
Southern regions	13.635.713	613.705	25%	3.408.928	18,0%
Italy	***32.109.909***	***2.183.547***		***6.180.058***	***35,3%***

FIGURE 7 **Linear extrapolation as a forecast for reaching the average targets in food waste captures.**



4 CONCLUSIONS

This study allowed for the identification of some aggregated targets in terms of food waste capture, based on a huge dataset at a municipal level.

First of all, central and southern Italian regions showed a strong potential in reaching very high targets (average 25% on total MSW), since the early implementation phase of the collection. Northern regions, where there's a more established practice of collecting food waste separately with the intensive curbside scheme, have reached a lower average capture (target 15% on total MSW), perhaps due to less home cooking and more packaged food usage.

These targets, recalculated at a national level, show that currently in Italy just 35% of the average food waste potential target is collected, providing plenty of possibility to further implement this collection scheme, especially in southern regions where diversion from landfill is most important, because they mainly rely on them as a disposal option and this approach must be changed according to EU and national regulation. In terms of quantity, almost 4.000.000 more tonnes of food waste could be collected, which from an industrial point of view translates into 100 new composting / AD facilities to be built.

Recent drivers (legislative obligation for collecting residential food scraps and national targets for 65% overall source separation rate at community level; increasing economic and environmental benefits from composting and anaerobic digestion of food waste, with respect to landfill and incineration) could speed up the decision by municipalities to start intensive source separated organics (ISSO) collection, to achieve these targets. Moreover, on January 1st 2011, Italy introduced a nation wide ban on non compostable plastic single use shopping bags. The exemption of certified compostable carrier bags is giving a substantial contribution in terms of outreach and communication to the citizens, minimisation of contamination by non compostable plastics, reduction of residues generated during pre- and post-treatment stages at composting facilities. An important remark is about how quickly these high captures could be reached, in as short as one month after implementing a good food waste collection scheme from scratch, if the right instruments are given to citizens (buckets, compostable bags, outreach and education campaigns).

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Session 27

(62) STUDY OF THERMAL CONDUCTIVITY IN ORGANIC SOLID WASTES BEFORE COMPOSTING

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EXECUTIVE SUMMARY

In France, like in all developed countries, the amount of solid wastes generated per year has increased continuously since the 1960’s. To hold back this trend, waste policies have been set up, as illustrated by current EU waste policy and its five main priorities: prevention, reuse, recycling, recovery and disposal. Composting can be defined as the process whereby aerobic micro-organisms convert organic substrates into compost: a hygienic, biostable product that can be beneficially applied to land (Haug, 1993; Mohee & Mudhoo, 2005). Therefore, it fits perfectly with the fourth priority, recovery, which fosters extraction of useful material or energy from wastes. In this context, interest towards composting has increased continuously during the last few years. During treatment, micro-organisms breakdown organic matter and produce carbon dioxide, water and heat. Heat generated by biological activity modifies moisture content and temperature conditions. These changes result in the appearance of a temperature peak in first days of treatment and most pathogens are killed by the high temperatures reached (around 70-80°C), turning waste into a hygienic product.

Among the various physical parameters taking part in the composting process, thermal conductivity seems to be of major importance, and could be used as an indicator to follow heat transfers within the organic matrix. However, as all physical parameters involved in the process, the initial preparation of the substrate (adjustment of moisture content or C/N ratio, addition and mixing with bulking agent …) has an influence on the physical parameters involved in the process (such as bulk density, Free Air Space or air permeability) and thus, on thermal conductivity. Moreover, difficulties often occur in composting experiments because the effects of compaction on physical properties are ignored, or information about these effects is lacking. As soon as the pile of waste is built, the settlement of the composting matrix begins. This settlement, called primary settlement or physical compressive settlement (Gourc et al., 2010; Yue et al., 2008) is related to the vertical load and leads to compaction. Despite its major importance, until now little has been written on thermal conductivity in composting, in particular about its link with compaction. That’s why this study focus on it and aims to investigate how it evolves with (i) compaction or depth within the pile of waste, (ii) preparation parameters of the substrate and (iii) temperature. This investigation was carried out on mixtures of urban sludge and wooden palettes used as bulking agents.

To understand how preparation parameters of the mixtures would affect thermal conductivity, two moisture content (50 and 65%), two types (fresh and recycled) and two meshes of bulking agent (< and > 20 mm) were tested. The influence of compaction (or depth) was evaluated in two steps. First, a “Schaub-Szabo” device (strongly inspired by the apparatus described in *Schaub-Szabo and Leonard* (Schaub-Szabo & Leonard, 1999)) was used to get depth-bulk density profiles in the different substrates. Then, these bulk densities were re-created in a modified air pycnometer where thermal conductivity was measured with a thermal probe directly embedded in the composting sample. Therefore, a link between thermal conductivity and compaction (or depth) could be established. On the other hand, the study of the impact of temperature on thermal conductivity was carried out in 10 liter cells where biological activity was prevented by a nitrogen atmosphere. The cells were filled with the same eight sludge-wooden palettes mixtures as before, and a thermal probe was put directly inside each sample. Then, they were put in different constant-temperature baths with target temperature from 5 to 75°C.

In this study, thermal conductivity increased with depth and a statistical analysis highlighted the fact that it was only significantly impacted by moisture content (among the three preparation parameters cited above). Moreover, the impact of temperature on thermal conductivity was clear and a linear relationship between these two parameters could be established. Each correlation was specific to the substrate but with a similar slope. These results are interesting in two ways: first, until now little has been written on thermal conductivity in organic solid wastes, and in particular about its link with compaction.

Besides this originality, the data obtained can now be used in numerical modeling to get a more thorough and accurate way to model heat transfers, an essential part of modeling composting systems.

1. INTRODUCTION

Nowadays, over 800 million tons of wastes are generated each year in France. To control the rise in the amount of waste produced since several decades now, waste policies have been set up in France as in most developed countries to fight this phenomenon. Current waste policy in the EU follows five main priorities, which in a hierarchical order are: prevention, reuse, recycling, recovery and disposal. Prevention encourages the community to reduce the amount of waste generated and to be more efficient in the use of resource. If waste cannot be prevented, as many of the materials as possible should be recovered, preferably by reuse and recycling. Energy and material recovery aims to foster extraction of useful material or energy from wastes. And disposal, as the least desirable option, must be handled with maximum precautions to minimize negative environmental outcomes. As a consequence, composting fits perfectly in the frame of material recovery and that is why during the last few years, interest has grown towards it as a way to treat organic wastes.

Composting can be defined as the biological organic matter decomposition process which occurs in aerobic conditions. At first sight, it could be seen as a quite simple process: with the proper mixture of water, oxygen and nutrients, micro-organisms breakdown organic matter of waste to produce compost. Yet, at micro scales it involves complex processes. These processes are of three types: biological processes and heat and mass transfers. Biological processes are linked to the activity of the decomposing micro-organisms which, provided with oxygen, turn organic matter into compost and produce heat, water and other gaseous compounds as well (mainly carbon dioxide). Their biological activity (consumption of oxygen and production of water and carbon dioxide) leads to mass and volume reduction of the substrate and organic matter stabilization. Furthermore, most pathogens are killed when the peak of temperature (70-80°C) is reached in the first days of treatment.

In return biological activity generates heat, leading to changes in moisture content and temperature conditions. Regarding heat transfers, thermal conductivity is a key role parameter. It is impacted by numerous parameters and among them moisture content, contact between particles and temperature seems to be of great importance. Contact between particles is link to particle size: the thinner the material, the more contacts there is. As moisture content, particle size is a preparation parameter which is set when the substrate is prepared.

As soon as built, the pile of waste is subject to compaction because its physical structure is unable to bear its own weight. One solution to that issue, widely used, is to add some bulking agents to solidify the pile. Nevertheless, in addition to the fact that these bulking agents added should not exceed a certain amount (to keep an acceptable C/N ratio for instance), this primary settlement or “physical compressive settlement”, as some authors called it (Gourc et al., 2010; Yue et al., 2008), cannot be avoided and its impact on initial physical characteristics should not be ignored, as it is usually the case because of the lack of information about it.

The original idea of this paper is to focus on thermal conductivity as it seems to be a physical parameter of major importance in regards to heat transfers in composting and yet, not actually well-considered in the existing literature. Therefore, this study aimed to investigate the links between thermal conductivity and (i) compaction or depth within the pile of waste, (ii) preparation parameters of the substrate and (iii) temperature. All experiments were carried out on urban sludge-bulking agent mixtures, with two different moisture contents, two particle sizes of bulking agent and two types of bulking agent (but quite similar anyway).

In order to perform thermal conductivity measurements at different heights (or under different degrees of compaction) we proceed in two steps, using two sets of apparatus. First a “Schaub-Szabo” device (named after its designer) was used to simulate compaction occurring in the pile and link compaction, depth and bulk density. Then, the sample was placed in another device, called “CPP” device, where the previous measured bulk densities were recreated. Here, thermal conductivity was measured using a thermal probe directly embedded in the sample. On the other hand, to evaluate the impact of temperature on thermal conductivity, measurements were made with thermal probes placed inside 10 liter cells, filled with substrate and where biological activity was inhibited with a nitrogen atmosphere.

2. MATERIALS AND METHODS

2.1 Sample preparation and characteristics of materials

The substrate tested in these trials was an urban sludge mixed with bulking agents. Two types of bulking agents were used: recycled and fresh wooden palettes. The urban sludge came from a wastewater treatment plant, the recycled wooden palettes used as bulking agents were collected from a composting platform and had already undergone a dozen composting cycles while the fresh ones were directly bought from a retailer.

For each urban sludge – bulking agent mixtures, two moisture contents (50% and 65%) and two meshes of bulking agents (< 20 mm and > 20 mm) were tested, leading to a total of eight different mixtures. As on the industrial site, the volumetric ratio of the sludge/bulking agent mixtures was fixed at 1/3 (which corresponded to a dry mass ratio of 0.147).

To minimize changes in the fresh materials, the sludge was sealed in plastic bags, stored frozen at -20°C and thawed as needed in a refrigerated room at 4°C. Likewise, the prepared mixtures were kept at 4°C between two sets of measurements in the “Schaub-Szabo” and the “CPP” devices.

2.2 Experimental setup

2.2.1 “Schaub-Szabo” device

The experimental device used to simulate compaction and to determine the variation of bulk density with depth was inspired by the device designed by Schaub-Szabo & Leonard (Schaub-Szabo & Leonard, 1999). It consisted of a cylindrical Plexiglas container for the material and a set of weights with a platform scale to apply vertical loads. The container was approximately 700 mm high with an inside diameter of 388 mm; and a fill line was marked 400 mm from the bottom. Inside the container, a bottom grid allowed potential water loss (leachates) from the sample, and the container was perforated at the bottom so that the water can be collected in a recipient placed below.

The recipient containing the sample was weighed; the sample was placed in the container until it reached the fill line, and the recipient was weighed again. The mass of the sample (M_1) was determined by difference and the bulk density of the first layer (BD_1) calculated according to:

$$BD_1 = \frac{M_1}{V_{fl}}$$

where V_{fl} is the volume to fill line (0.04729 m^3).

To determine the bulk density in the second layer (BD_2), known masses were placed on top of the sample to apply a load equivalent to the mass M_1 to the sample in the container. The masses used were steel discs with a diameter of 380 mm (slightly less than the Plexiglas container) and adjusted by adding a known volume of water on top of them. The loading was applied for 24 hours, until the material stabilized to a constant volume.

At the end of the compression, the settlement of the matrix (h_2) was recorded. Then, the loading system was removed and fresh material was added to get a 400 mm (h_0) height. The mass added (m_2) was calculated as follows:

$$m_2 = BD_2 \cdot V_{fl} \cdot \left(1 - \frac{h_2}{h_0}\right)$$

The above procedure was repeated, layer by layer, to simulate six layers of material, which corresponded to a pile of waste of 2.4 m high. A relationship between bulk density and depth for each mixture was finally obtained.

2.2.2 “CPP” device and thermal conductivity measurement

The bulk densities previously measured in the “Schaub-Szabo” device were recreated in a “CPP” device, which was originally used to measure porosity and permeability of compacted materials (“C” stands for compaction, “P” for porosity and “P” for permeability in the acronym “CPP”). It was the same apparatus as described in Druilhe, Benoist et al. (Druilhe et al., 2008) with a thermal probe directly embedded in the composting material. Thermal conductivity measurements were conducted at each bulk density calculated before in the “Schaub-Szabo” device. Thus, the relationship between thermal conductivity and depth was brought to light.

To recreate the various degrees of compaction, the sample was placed in a removable basket designed to hold about 40-60 L of mixture. Once the basket loaded and the volume known, the bulk density of the sample was modified by adjusting the depth of a perforated compression plate, manually controlled by an airtight screw. Thermal conductivity was measured with an unsteady state probe method which is a commonly used technique on porous materials (Chandrakanthi et al., 2005; Iwabuchi et al., 1999; Van Ginkel et al., 2002). The thermal probe consists of a heating wire and a thermocouple which measures the temperature at this source. When supplied with a constant electric power Q, the temperature of the probe increases as a function of time. After a certain period of time, the elevation of temperature reaches an asymptotic regime. Therefore, the graph of the rise in probe temperature versus the logarithm of the time gives a straight line, and the thermal conductivity λ ($W.m^{-1}.K^{-1}$) can be calculated from the slope A:

$$\lambda = \frac{Q}{4\pi \cdot A}$$

Practically, the probe was embedded directly at middle height into the composting material. The electric power was supplied by a constant power generator and the thermocouple of the probe was connected to a data acquisition terminal. Thermal conductivity measurements were directly made with the dedicated software.

2.2.3 Thermal conductivity cells

In parallel, the impact of temperature on thermal conductivity was also investigated. The experiment was carried out in 10 liter cells filled with substrate. Thermal conductivity was measured with a thermal probe placed inside the sample; and the same protocol described in 2.2.2 was applied to the eight sludge-bulking agent mixtures studied. To ensure that temperature was the only changing parameter during the experiment, biological activity had to be inhibited. Indeed, the physical structure of the matrix could be modified under the effect of micro-organisms breaking down organic matter. Therefore, the cells were supplied with nitrogen to inhibit biological activity during the measurements.

The cells were put in different constant-temperature bath with target temperatures ranging from 20 to 75°C, by steps of 5°C. In addition, thermal conductivity measurements were performed right after the samples were taken out of the refrigerated room (and before they reached the ambient temperature) at 5, 10 and 15°C.

As said earlier, the peak of temperature in composting usually reaches temperature values around 70-75°C, that is why the maximum target value was set at 75°C.

2.3 Statistical analysis

A statistical analysis was carried out to identify which factors and their interactions had a significant impact on thermal conductivity. The four factors were considered were depth, moisture content and particle size and type of bulking agent (temperature, although studied in this paper, was not used as a factor in the statistical analysis). Four independent variables were used (namely depth, moisture content, particle size and type of bulking agent) and two normalized values, -1 and +1, were considered for each of them. For depth, these two values corresponded to the first (-1) and sixth layers (+1) – from -0.4 to 0 m and from -2.4 to -2 m in depth respectively - simulated with the “Schaub-Szabo” device, for moisture content to 50 (-1) and 65% (+1), for particle size to < 20 mm (-1) and > 20 mm (+1), and for the type of bulking agent to recycled (-1) and fresh (+1) wooden palettes. Therefore, a negative impact of the variable “type of bulking agent” on one of the physical responses would mean that this response decreased when switching from recycled palettes to fresh ones.

The significance of variable effects and interactions was determined using a Student test at a confidence level of 95% ($P < 0.05$). All statistical analyses were performed with the software Statgraphics (Centurion XV, Warrentown, Virginia, USA).

3. RESULTS AND DISCUSSION

3.1 Impact of depth, moisture content, particle size and type of bulking agent on thermal conductivity

The results of the statistical analysis are presented in Table 1 below. It displays the impact of a given parameter or interaction on thermal conductivity, if it is whether positive or negative and its significance.

Table 1: Statistical analysis [+: positive impact, -: negative impact, +++/---: $P \leq 0.0001$, ++/--: $0.0001 \leq P \leq 0.01$, +/-: $0.01 \leq P \leq 0.05$, (+)/(-): $P \geq 0.05$ (non significant)]

	Thermal conductivity
Depth (D)	(+)
Moisture content (MC)	++
Particle size (PS)	(-)
Bulking agent (BA)	(-)
D/MC interaction	(+)
D/PS interaction	
D/BA interaction	
MC/PS interaction	--
MC/BA interaction	(-)
PS/BA interaction	++

As previously stated by some researchers before (Ahn et al., 2009; Chandrakanthi et al., 2005), thermal conductivity increased with compaction (or increasing bulk density) due to a reduction in the void space. On Figures 1 and 2 below, thermal conductivity seemed to increase with depth as well but the statistical analysis revealed that depth alone didn't have a significant impact and neither in interaction with another factor (see Table 1).

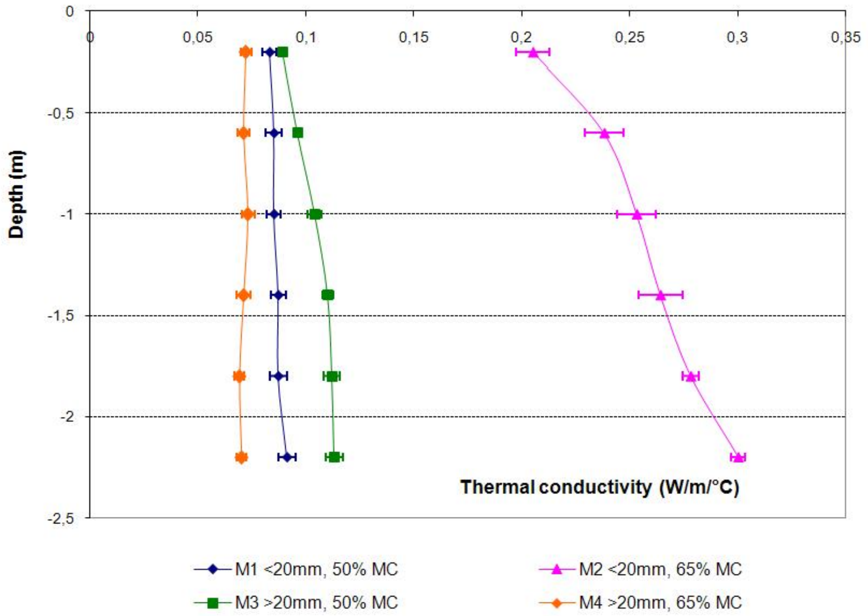

Figure 1: Thermal conductivity as a function of depth at different moisture contents and particle sizes (sludge – recycled palettes mixtures)

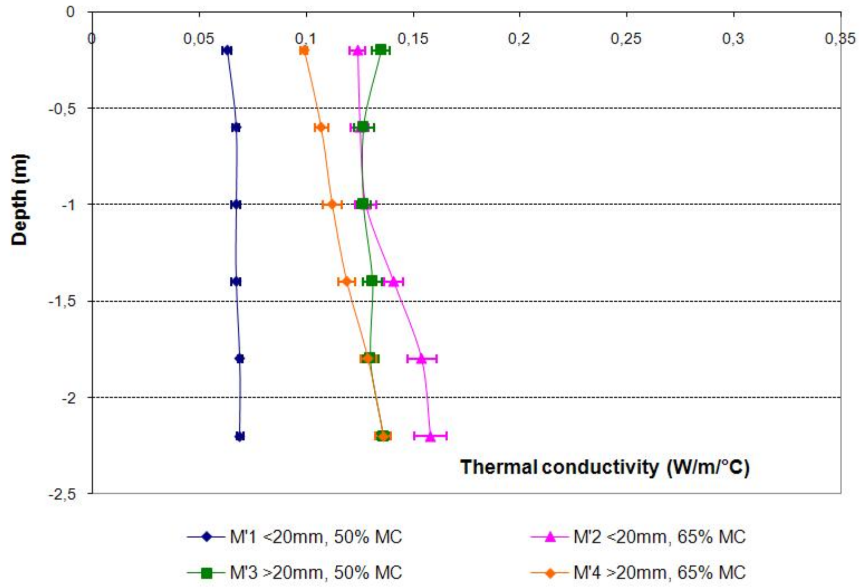

Figure 2: Thermal conductivity as a function of depth at different moisture contents and particle sizes (sludge – fresh palettes mixtures)

Among the three preparation parameters tested (moisture content, particle size and type of bulking agent), the statistical analysis shows that thermal conductivity was significantly influenced by only one factor, moisture content, and increased with it. This result was expected since in theory, thermal conductivity of water is higher than of air (0.06 $W.m^{-1}.K^{-1} > 0.026 W.m^{-1}.K^{-1}$). Therefore, by increasing moisture content, the pores of the organic matrix were filled with water and then, thermal conductivity increased. These results were consistent with the existing literature, even if the experiments were performed on different substrates such as borage seeds (Yang et al., 2002), grain dusts (Chang et al., 1980), beef manure (Houkom et al., 1974), dairy cattle feces mixed with sawdust (Iwabuchi et al., 1999), leaf composts (Chandrakanthi et al., 2005) or compost-bulking agent materials (Ahn et al., 2009).

The type and particle size of bulking agent, although they did not have significant impacts alone, intervened with significant interactions (as shown in Table 1). The first interaction was negative and between moisture content and particle size. It meant that the increase in thermal conductivity with an increase in moisture content was maximal at low particle size. It also meant that at high particle size (> 20 mm), the impact of moisture content on thermal conductivity became negative. Another interpretation of this interaction is that, at high moisture content (65%), thermal conductivity tended to increase when particle size decreased. These results were in agreement with the study of Ahn, Sauer et al. (Ahn et al., 2009) who observed an increase in thermal conductivity for eleven different composting materials when grinding them from 10 cm to 0.5 mm. An explanation to this phenomenon was that decreasing particle size created more thermal contacts between particles which, combined with high moisture content, led to an increase in thermal conductivity.

The second significant interaction brought to light was positive and between particle size and the type of bulking agent (see Table 1). The tendency of thermal conductivity to decrease when switching from recycled palettes to fresh ones was significant with particles < 20 mm: 0.083-0.3 W/m/°C with recycled palettes and 0.063-0.158 W/m/°C with fresh ones.

3.2 Evolution of thermal conductivity with temperature

As shown on Figure 3, thermal conductivity (λ) clearly increased with temperature (T). Between 5 and 75°C, thermal conductivity increased between two and five times depending on the mixture. Moreover, a linear relationship of the form $\lambda = a \cdot T + b$ was obtained, with correlation coefficients ranging from 0.946 to 0.989. *b* coefficients were all different, meaning that each correlation was specific to the mixture studied. However, they all had a similar slope *a* (0.003-0.004 $W/m/°C^2$).

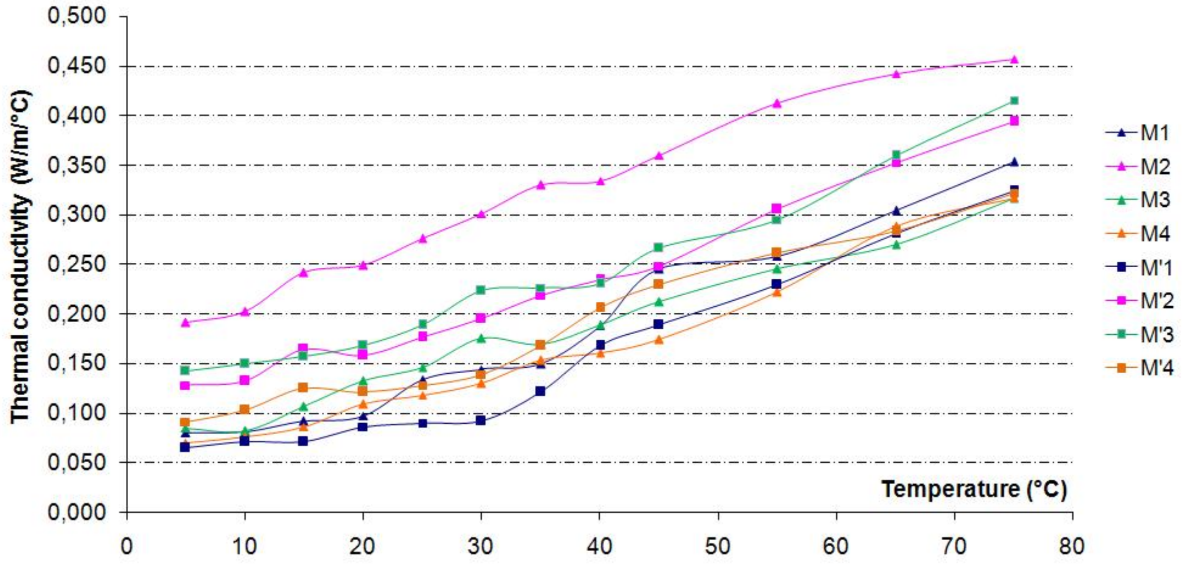

Figure 3: Thermal conductivity as a function of temperature at different moisture contents and particle sizes

These are interesting results because, although thermal properties are very important in composting, information on their values for various composting materials is lacking. In this way, the data provided by this study could be very useful, in particularly regarding numerical modeling.

Furthermore, these results were consistent with the little literature currently available. Van Ginkel (Van Ginkel et al., 2002) also showed that thermal conductivity of a mixture of chicken manure and wheat straw increased with temperature; and their relationship showed an almost perfect linearity according to the authors. Unfortunately, they did not give the value of the slopes they obtained so we could not compare. Similarly, Yang et al. (Yang et al., 2002) obtained a mathematical equation linking thermal conductivity of borage seeds to temperature and moisture content.

4. CONCLUSION

In this paper, the influence of three preparation parameters of the substrate (moisture content, particle size and type of bulking agent), depth (or compaction) and temperature on thermal conductivity in organic solid wastes was investigated. Knowledge about thermal conductivity in such substrates and its link with depth (or compaction) is quite poor at present. That is why this paper aimed to give some answers about what parameters influence thermal conductivity in organic solid wastes before composting.

Among the three preparation parameters tested and depth, thermal conductivity was only significantly influenced by moisture content. Contrary to what could have been expected from theory, depth (or compaction) didn’t significantly impact thermal conductivity. On the other hand, temperature had a clear impact and a linear relationship between these two parameters was brought to light. Each correlation was specific to the substrate but with a similar slope. However, it is important to note that such results are only valid in the sludge-wooden palettes mixtures tested, and similar studies should be carried out on other substrates.

The data experimentally obtained can now be used in numerical modeling to improve existing heat transfers models, an essential step to get a more thorough and accurate modeling of whole composting systems.

5. NOMENCLATURE

λ	Thermal conductivity ($W.m^{-1}.K^{-1}$)
A	Numerical coefficient (-)
BD_i	Bulk density of the layer i ($kg.m^{-3}$)
FAS	Free Air Space (-)
h_i	Settlement of the layer i (m)
h_0	Initial level before settlement (m)
$M1$	Sludge-recycled wooden palettes mixture (particle size < 20mm, 50% moisture content)

M2	Sludge-recycled wooden palettes mixture (particle size < 20mm, 65% moisture content)
M3	Sludge-recycled wooden palettes mixture (particle size > 20mm, 50% moisture content)
M4	Sludge-recycled wooden palettes mixture (particle size > 20mm, 65% moisture content)
M'1	Sludge-fresh wooden palettes mixture (particle size < 20mm, 50% moisture content)
M'2	Sludge-fresh wooden palettes mixture (particle size < 20mm, 65% moisture content)
M'3	Sludge-fresh wooden palettes mixture (particle size > 20mm, 50% moisture content)
M'4	Sludge-fresh wooden palettes mixture (particle size > 20mm, 65% moisture content)
Q	Dissipated electrical power per unit length ($W.m^{-1}$)
T	Temperature (°C)
V_{fl}	Volume to fill line (m^3)

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(72) LARGE SCALE COMPOSTING MODEL

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EXECUTIVE SUMMARY

One way to treat the organic wastes accordingly to the environmental policies is to develop biological treatment like composting. Nevertheless, this development largely relies on the quality of the final product and as a consequence on the quality of the biological activity during the treatment. Favourable conditions (oxygen concentration, temperature and moisture content) in the waste bed largely contribute to the establishment of a good aerobic biological activity and guarantee the organic matter stabilisation with limitation and control of odorous and greenhouse effect gaseous emissions. Several approaches (0D biochemical reducing, see Pommier et al.2007, effective 1D modelling coupling transport and biochemical) have been made to understand the behaviour of such systems. In this paper we will present a 2D numerical model using Darcy scale equations for heat and mass transport coupled with a biochemical reactive scheme. Then, we will solve that system (using experimental measurements on reactivity and transport coefficients) with a commercial code (COMSOL TM). The model described here is based on the biological model presented in Trémier et al 2005 coupled with an upscale transport model detailed in Hénon 2008 which takes into account the major components of the gas phase: N_2, O_2, CO_2 and also H_2O. This is a crucial point because of:

- The reaction rate, depending on the moisture content (humidity comes from the initial condition of the sludge but also from the reactive scheme because reactions produce water),
- heat content, very sensitive to the evaporation rate in the sludge.

It has been shown in Pujol et al 2011 that the impact of drying could be important on the reactivity but also that the pseudo component air could not be sufficient to represent the drying in the sludge.

The process studied was a closed reactor composting process (180 m^3 rectangular box) with positive forced aeration. The air was blown from the bottom of the reactor, via two ventilation pipes. In the upper part of the reactor, air was sucked and led to a biofilter treatment system. The treated waste was a mixture of sewage sludge and bulking agent that was composted during four weeks without turning. Several informations were recorded during the treatment like temperature evolutions at different locations (see Henon et al. 2009 for more details about the temperature recording). We have validated this code by comparing the temperatures obtained through the simulations with those recorded during the experiments.

After this step of validation and a discussion on final composition of the organic matter in the experiments compared to the ones estimated by simulations, we have used this numerical model as an optimization tool. Modifying the initial, boundary and operating conditions we have been able to determine the best conditions to this particular composting process. A whole set of conditions is discussed in the paper.

1. INTRODUCTION

Composting is a very complex phenomenon where organic wastes are converted into a stable material by microorganisms. They generally consume O_2 injected from air, and the organic matter inside the sludge. Some exhaust gases will be generated mainly H_2O, CO_2 but also heat which can allow the matter to be sanitized (porous medium must be maintained at more than 55°C for 3 days according to Golueke 1983). Then, this compost can be used soil improver by enriching the soil nutrient and organic content.

The process studied in our case consists in a closed reactor with positive aeration. In figure 1 on the upper left part, the geometry is briefly reminded. Air is distributed using several holes made in two veins. The homogeneity of the distribution is one of the key points, because if we need to provide microorganisms enough oxygen, we also have to avoid important drying which may block the reactive scheme. Under certain conditions (temperature and humidity), microorganisms cannot play their role and all the problem is to keep an equilibrium between temperature/water content/air distribution. The effects of humidity have particularly discussed in Pommier et al 2007.

In figure 1, the different scales of the problem appear. If we have a look at the industrial scale, we can define a REV scale where an effective transport model can be derived from the transport processes in a detailed image of the porous medium so called the pore scale. The biofilm phase can also be homogenised because it appears as a porous medium and seeing it like an effective medium requires an averaging process made recently in Aspa et al. 2011.

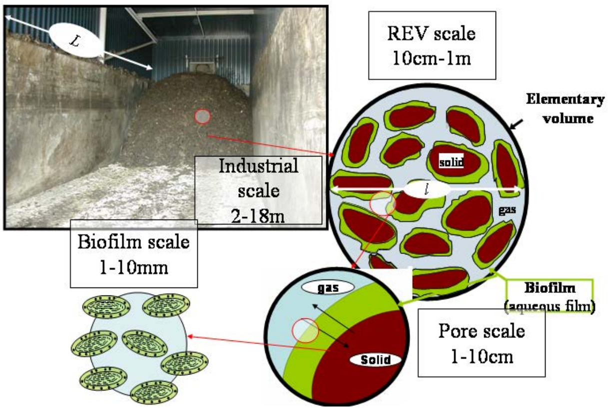


Figure 1: Industrial composting process and the different scales associated, from the biofilm scale (1mm) to the industrial one (10m)

The other important point is the water content in the sludge and its prediction during composting. According to Richard et al. 2002, the moisture content must be controlled in order to maintain an efficient process. We will see this more in details further but in the reactive scheme, there is a high dependence upon the water content on the global reactivity. This could be interesting if we could follow humidity inside the wastes but it's challenging because of the assumptions underlying the results of the measures we will have (how a large scale measurement could be a good indicator of the local water content?).

Thus, a macroscale model could give some indications and hopefully indicates how to optimize the process in term of aeration.

2. METHODOLOGY

We will recall the local scale equations, leading to the macroscale system fully coupled with biodegradation terms determined thanks to respirometric studies. In a last subsection, the parameter values missing in order to close the system will be explained.

2.1 Local scale model

At the pore scale, the set of governing equations are based on the basic fluid mechanics concepts. The medium is seen like a continuum; so, we can write the Navier Stokes equations in the gas phase (we supposed the fluid phase to be immobile) with a no slip condition upon the solid phase and the fluid phase.

For the mass transport equations, in the gas phase, classical convection and diffusion equations are used, with flux continuities on the interfaces between gas and liquid phase. For solid component, we only use classical ODE forms, with an accumulation part and a reactive term. The heat transport is a convective conductive equation in the gas phase, and a conductive equation in the solid phase. Then, we average the whole set of equations to obtain the macroscale model. To do that, we use the classical theory and theorems of the volume averaging process and the reader could see some details in Puiggali & Quintard.1992. In our case, we will only present the final system.

2.2 Darcy's scale model

Averaging all the equations over a REV, we can express a set of equations at the Darcy's scale. The mass conservation for the gas phase is given by the continuity equation expressed like $\varepsilon \frac{\partial \rho_g}{\partial t} + \bar{\nabla} \cdot \left(\rho_g \bar{u}_g \right) = \dot{m}_{gsO_2} + \dot{m}_{gsCO_2} + \dot{m}_{gsH_2O}$, where ε is the porosity, ρ_g the gas density and $\bar{u}_g = -\frac{\bar{\bar{K}}}{\mu_g} \cdot \left(\bar{\nabla} p_g - \rho_g \bar{g} \right)$ is the intrinsic average velocity of the gas phase, given by the Darcy's law. In equation 1, $\dot{m}_{gsH_2O}$ is the exchange term between the le gas and the immobile water phase. $\dot{m}_{gsO_2}$ and $\dot{m}_{gsCO_2}$ are the reactive terms corresponding respectively to O_2 consumption and CO_2 production. They are given by the following relations..

$$R_{O_2} = \left[\frac{1-Y}{Y} \cdot \mu_{opt}\left(T, C_{sH_2O} \right) \cdot \frac{MB(t)}{K_b + MB(t)} \mathrm{X}(t) + b(T) \cdot (1-f) \cdot \mathrm{X}(t) \right] \times M_{O_2}$$

$$R_{CO_2} = P_{CO_2} \cdot \frac{R_{O_2}}{M_{O_2}} \cdot M_{CO_2}$$

In our case, we use the classical assumption that gas phase is ideal, so: $\rho_g = P_g / RT \sum_{i \in g} \frac{\Omega_{g,i}}{M_i}$, where P_g is the gas pressure, $\Omega_{g,i}$ the mass fraction of component I in the gas phase and M_i its molecular weight.

Gas species are transported in the gas phase and a general advection dispersion reaction equation is used.

$$\varepsilon \rho_g \frac{\partial \Omega_{gi}}{\partial t} + \varepsilon \Omega_{gi} \frac{\partial \rho_g}{\partial t} + \Omega_{gi} \bar{\nabla} \cdot \left(\rho_g \bar{u}_g \right) + \rho_g \bar{u}_g . \bar{\nabla} \Omega_{gi} + \bar{\nabla} \cdot \left(\varepsilon \rho_g \bar{\bar{D}}^*_{gi} \cdot \bar{\nabla} \Omega_{gi} \right) = \pm R_i,$$ where $i = CO_2, O_2, N_2$ ($R_{N2}=0$).

We see that the dispersion transport appears in this equation. We have chosen to use a classical model defined like:

$$\bar{\bar{D}}^*_{gi} = \underbrace{\frac{D_{gi} \bar{\bar{I}}}{\tau}}_{\text{diffusive term}} + \underbrace{\left[\alpha_T \left\| \bar{u}_g \right\| \bar{\bar{I}} + \left(\alpha_L - \alpha_T \right) \frac{\bar{u}_g \bar{u}_g}{\left\| \bar{u}_g \right\|} \right]}_{\text{dispersive term}},$$ where $\bar{\bar{I}} / \tau$ is the tortuosity tensor for an isotropic medium.

α_L represents the longitudinal dispersivity coefficient and α_T the transversal one.

Water vapour transport is governed by one equation in the gas phase.

$$\varepsilon \frac{\partial \rho_g \Omega_{g,H_2O}}{\partial t} + \bar{\nabla} \cdot \left(\rho_g \Omega_{g,H_2O} \bar{u}_g \right) = \bar{\nabla} \cdot \left(\varepsilon \rho_g \bar{\bar{D}}^*_{gH_2O} \cdot \bar{\nabla} \Omega_{g,H_2O} \right) + \dot{m}_{gsH_2O}$$

But also, one in the solid phase:

$$\left(1 - \varepsilon \right) \frac{\partial \rho_s \Omega_{sH_2O}}{\partial t} = R_{H_2O} + \dot{m}_{sgH_2O}$$

Following Puiggali and Quintard 1992, we assume that the local equilibrium assumption is valid in our composting problem, so, no differences between an average value of solid humidity and the one in the gas exist. This imposes that the gas phase is at the equilibrium so that $\Omega_{gH_2O,eq} = \Omega_{gH_2O}$. This allows us add the two preceding equations and to obtain the one we will now use.

$$\left(1 - \varepsilon \right) \frac{\partial \rho_s \Omega_{sH_2O}}{\partial t} = R_{H_2O} - \varepsilon \frac{\partial \rho_g \Omega_{gH_2O}}{\partial t} - \bar{\nabla} \cdot \left(\rho_g \Omega_{gH_2O} \bar{u}_g \right) + \bar{\nabla} \cdot \left(\varepsilon \rho_g \bar{\bar{D}}^*_{gH_2O} \cdot \bar{\nabla} \Omega_{gH_2O} \right)$$

We will calculate $\Omega_{gH_2O,eq} = X_{sat} M_{H_2O} \sum_{i \in g} \frac{\Omega_{i,g}}{M_i}$ assuming that at the equilibrium, we have $T = T_{sat}$, and $X_{H_2O,eq} = X_{sat}$.

Local equilibrium for temperature is also assumed, and the equation takes the following form:

$$\varepsilon\rho_g\sum_{i=1}^{N_i}Cp_{gi}\Omega_{gi}\frac{\partial T}{\partial t}+(1-\varepsilon)\sum_{k=1}^{N_k}Cp_{sk}\rho_{sk}\frac{\partial T}{\partial t}+\left(\sum_{i=1}^{N_i}Cp_{gi}\Omega_{gi}\right)\rho_g\overline{u}_g\cdot\vec{\nabla}T=-T\left\{\varepsilon\rho_g\sum_{i=1}^{N_i}Cp_{gi}\frac{\partial\Omega_{gi}}{\partial t}+\rho_g\sum_{i=1}^{N_i}Cp_{gi}\left(\overline{u}_g\cdot\overline{\nabla}\Omega_{gi}\right)\right\}$$

$$-T\left(\sum_{i=1}^{N_i}Cp_{gi}\Omega_{gi}\right)\left\{\frac{\partial\left(\varepsilon\rho_g\right)}{\partial t}+\vec{\nabla}\cdot\left(\rho_g\overline{u}_g\right)\right\}-T\left\{(1-\varepsilon)\sum_{k=1}^{N_k}Cp_{sk}\frac{\partial\rho_{sk}}{\partial t}\right\}+\overline{\nabla}\cdot\left(\overline{\overline{\Lambda}}_{eff}\cdot\overline{\nabla}T\right)+\Delta H_{O_2}R_{O_2}-\Delta H_{H_2O}$$

$$\left[\varepsilon\rho_g\frac{\partial\Omega_{gH_2O}}{\partial t}+\rho_g\overline{u}_g\cdot\overline{\nabla}\Omega_{gH_2O}-\overline{\nabla}\cdot\left(\varepsilon\rho_g\overline{\overline{D}}^{*}_{gH_2O}\cdot\overline{\nabla}\Omega_{gH_2O}\right)+\Omega_{gH_2O}\left(-R_{O_2}+R_{CO_2}+\dot{m}_{gsH_2O}\right)\right]$$

Where Cp_{sk} and Cp_{gi} are respectively the heat capacity for the k component in the solid phase and the heat capacity for the i component in the gas phase. Finally, other reactive terms necessary in the biodegradation process are summarized in table 1 and the definitions and values of this biodegradation process are given in Table 3.

Table 1: biodegradation scheme

Micro-organisms growth kinetics	$\frac{d\mathrm{X}(t)}{dt}=\mu\left(T,C_{sH_2O}\right)\cdot\frac{MB(t)}{K_B+MB(t)}\cdot\mathrm{X}(t)-b(T)\cdot\mathrm{X}(t)$
Rapidly biodegradable consumption kinetics	$\frac{dMB(t)}{dt}=-\frac{1}{Y}\cdot\mu(T,C_{sH_2O})\cdot\frac{MB(t)}{K_b+MB(t)}\mathrm{X}(t)+K_h(T)\cdot\frac{MH(t)/\mathrm{X}(t)}{K_{MH}+MH(t)/\mathrm{X}(t)}\mathrm{X}(t)$
Slowly biodegradable consumption kinetics	$\frac{dMH(t)}{dt}=-K_h(T)\cdot\frac{MH(t)/\mathrm{X}(t)}{K_{MH}+MH(t)/\mathrm{X}(t)}\mathrm{X}(t)$
dry matter consumption	$R_{MS}=-C_{MS}\cdot R_{O_2}$

2.3 Parameters

Our model needs two types of parameters. Firstly, transport parameters must be specified and measured. Then, kinetic parameters must be estimated in order to be coupled with the transport problem. All of them are given in the next tables and the value used in our study case is given.

Table 2: Parameters appearing in the macroscale equations

Parameters	notation	Source	Value	Units
ε	Porosity	Experiments (Druilhe et al.2008)	0.3	-
μ_g	dynamic viscosity in the gas phase	Handbook of Chemistry and Physics	1.78×10^{-5}	*kg/m/s*
K	Permeability	Druilhe et al. (2008)	3×10^{-7}	m^2
τ	tortuosity	Kallel et al. (2004)	2	-
α_L	Longitudinal dispersion coefficient	Experiment	0.4	*m*
α_T	Transversal dispersion coefficient	-	0.2	*m*
ΔH_{molO_2}	Enthalpy of reaction O2 consumption	Bailey and Ollis (1986)	3×10^5	$J/mol\,O_2$
Λ_s	Solid thermal conductivity	Trémier (2004)	0.5	*W/m/K*

Table 3: Values, sources and determination modes of biodegradation parameters.

Constant	notation	Source	values	Units
f	Inter matter fraction		0.2	-
Y	Rendement de production de biomasse		0.69	-
K_b	Half saturation constant for hydrolysable matter		0.8	mol O_2/m^3
K_{MH}	Half saturation constant *X/MH*		6.8	-
$a_{\mu opt}$	kinetic coefficient micro organism growth		1.27×10^{-5}	s^{-1}

$b_{\mu opt}$	Optimum kinetic coefficient micro organism growth	Respirometric tests	9.05×10^{-5}	s^{-1}
b_{opt}	Optimal value for death micro-organisms		1.53×10^{-5}	s^{-1}
k_{hopt}	Optimal kinetic for hydrolysis		2.56×10^{-5}	s^{-1}
$T_{\mu\max}=T_{K\max}=T_{b\max}$	Maximum temperature for growth kinetics, hydrolysis and death		80	$°C$
$T_{\mu\min}=T_{K\min}=T_{b\min}$	Minimum temperature for growth kinetics, hydrolysis, and death		0	$°C$
$T_{\mu opt}=T_{Kopt}=T_{bopt}$	Optimal temperature for growth kinetics, hydrolysis, and death		40	$°C$
P_{CO_2}	Stoichiometric coefficient CO_2 consumption		0.8	mol_{CO_2}/mol_{O_2}
P_{H_2O}	Stoichiometric coefficient H_2O production		1.5	mol_{H_2O}/mol_{O_2}
C_{MS}	Stoichiometric coefficient MS consumption		0.021	mol_{MS}/mol_{O_2}
G(T)	Temperature function		See after	-

Using these coefficients, we can define non linear functions for the biodegradation scheme introduced in Trémier et al 2005. Let's define G(T), it is a function depending from several parameters and especially from $T_{\mu\max}=T_{K\max}=T_{b\max}$, $T_{\mu\min}=T_{K\min}=T_{b\min}$, $T_{\mu opt}=T_{Kopt}=T_{bopt}$ and can be written using a cardinal function (see Rosso et al 1995).

$$G(T)=\frac{(T-T_{b\max})(T-T_{b\min})^2}{(T_{bopt}-T_{b\min})\left[(T_{bopt}-T_{b\min})(T-T_{bopt})-(T_{bopt}-T_{b\max})(T_{bopt}+T_{b\min}-2T)\right]}$$

Firstly, $b(T)$, $K_h(T)$ and $\mu(T,C_{sH_2O})$ that will be used in order to define respectively the dependence of micro organisms growth with temperature, the hydrolysis rate and biodegradable matter consumption can be written as:

$$b(T)=b_{opt}\times G(T),K_h(T)=k_{hopt}\times G(T),\mu(T,C_{sH_2O})=\left[a\mu_{opt}\ln(C_{sH_2O})-b\mu_{opt}\right]\cdot\frac{C_{gO_2}}{K_{gO_2}+C_{gO_2}}G(T)$$

At this point, we clearly exhibit the high dependence with temperature for the biodegradation (from $G(T)$) but also with water content with the $\left[a\mu_{opt}\ln(C_{sH_2O})-b\mu_{opt}\right]$ term. Stoechiometric coefficients P_{CO_2}, P_{H_2O}, and C_{MS} are determined empirically using the balance equation introduced in Trémier, 2004 $C_xH_yO_z+\left(x+\frac{y}{4}-\frac{z}{2}\right)O_2\rightarrow xCO_2+\frac{y}{2}H_2O$.

In this section, biodegradation kinetics and transport parameters have been set. Reactive terms come also from an averaging process, because we measure the reactivity in a porous medium composed of several REV. Those represent a macroscale behaviour which have been coupled with the macro scale model presented. Determining the parameters missing by measurements, we are now able to use this numerical tool and try to check if it matches with experimental data.

3. COMPARAISON BETWEEN INDUSTRIAL SCALE EXPERIMENTS AND SIMULATIONS

In order to validate our model and to use it as an optimization tool for the industrial process, an experimental campaign was made during one month and some temperature recording and balances have been made for H_2O and dry matter. The temperature recording was presented in Henon et al. 2009, but the following figure 2 will summarize the locations of the recorders and dimensions. The first results obtained are given in figure 3.

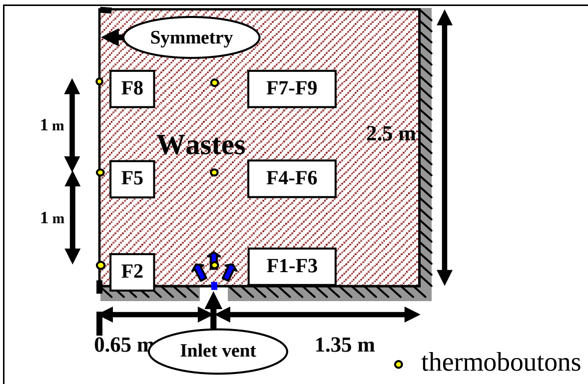


Figure 2: sensors of temperature disposed in the waste during the composting process. Only one half is represented as the global process is symmetric.

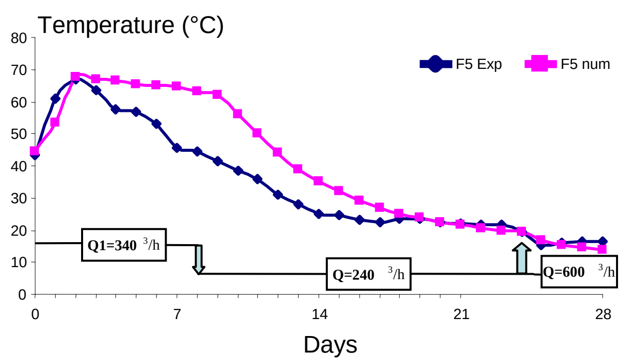


Figure 3: comparison between experimental data and numerical prediction for F5 thermocouple.

During the experiments, the aeration was supplied as follow:

- Flow rate equal to Q1= 340m^3/h during 9 days.
- Flow rate equal to Q2= 240m^3/h during 16 days.
- Flow rate equal to Q3= 600m^3/h during 3 days.

No turning of the composting mixture has been made during this experiment in order to keep the sensors at their initial locations but also to be able to analyse the distribution of water without any perturbation due to mixing.

On Figure 3, we can see a good agreement, between predicted and measured values. In the middle part, we observe that the numerical model overestimates the temperature from the 3rd to the 18th day. Several over locations have been checked and the results look the same. At this step, we can explain these differences mainly by some heterogeneity in biomass concentration in the waste but also from the drying process which is not limited in our model. No sorption isotherm was added in order to limit the evaporation process. This is confirmed in table 4, H_2O losses are more important in our numerical case, but we represent fairly well the dry matter losses, and that confirms the good accuracy of our model compared to experimental data.

Table 4: Final balance for H2O and dry matter from the experiment compared to numerical simulation.

	Experimental balance	Numerical balance
H_2O losses (% of the initial mass)	72.1	93
Dry matter losses (% of the initial mass)	10.58	12.9

4. OPTIMIZATION

The above model was used to study several aeration protocols in order to find the best way to achieve sanitation process, i.e a temperature of 55°C maintained during 3 days according to Golueke 1983. Other couples temperature/time have been proposed by Dumontet et al. 1997 (50° during 9 days) or in De Bertoldi et al (55 to 65°C for 1 to 3 days). All the tests made are summarized in the table 5. We have simulated changes of the flow rate (240m3/h except in M2 which is the base case), the aeration (2 inlets in M5 & M6), heated the ground and air (M4 & M5) or simulated mixing (in M4 & M6). These are the model specifications:

- M1: Base case : flow rate constant equal to $240\ m^3/h$ during all the process (28 days)
- M2: Flow rate varying (like in the results presented before).
- M3: Identical to M1, with a mixing (15th day)
- M4: Ground and air heated at 30°C + mixing 15th day
- M5: Two inlets with constant flow rate ($240\ m^3/h$) + ground and air heated at 30°C
- M6: Identical to M5, mixing 15th day.

Table 5: Results of the optimization tests with highlights on sanitation.

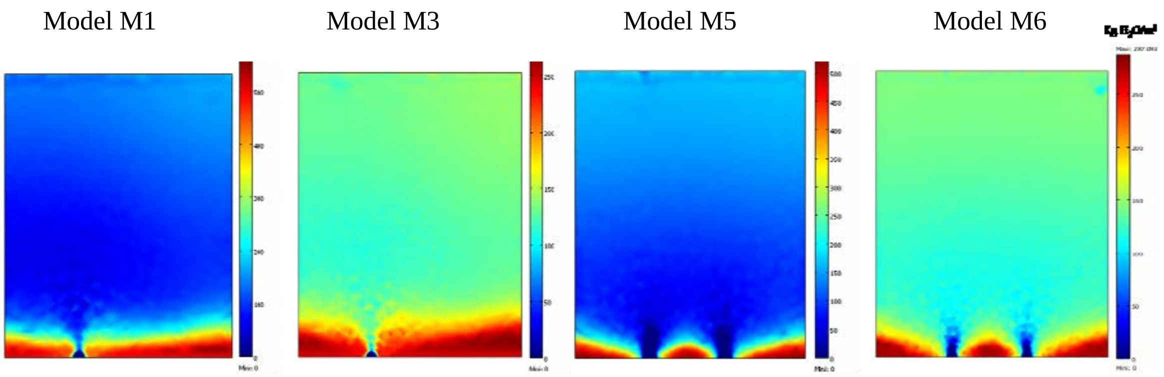
X = reached hygienisation 0 = no sanitation		Hygienisation		
		55 ° C, 3 days (Golueke, 1983)	50 ° C, 9 days (Dumontet et al., 1997)	55 to 65 ° C, 1 to 3 days (From De Bertoldi et al., 1988)
M1	Top	X	X	X
	Central part	X	X	X
	Bottom	0	0	0
M2	Top	X	X	X
	Central part	X	X	X
	Bottom	0	0	0
M3	Top	X	X	X
	Central part	X	X	X
	Bottom	0	0	0
M4	Top	X	X	X
	Central part	X	X	X
	Bottom	0	0	0
M5	Top	X	X	X
	Central part	X	X	X
	Bottom	0	0	X
M6	Top	X	X	X
	Central part	X	X	X
	Bottom	0	0	X

All the tests without any mixing (M1, M2, and M5) give the same results from the sanitation point of view. Only the upper and central part of the whole domain can reach the temperature of 55°C during more than 3 days. In the M5 and M6 cases, another part of the domain can be sanitized but according to De Bertoldi et al., 1988. One way to achieve this goal is to mix the wastes and to put the upper part in the bottom for the last period of the composting process. The final balances give the following results. The mixing effects are mainly a less important drying, but it is not sensible as we estimate a global balance. For dry matter, no important differences can be seen.

Table 6 : H2O and dry matter balances over the different optimization tests

	M1	M2	M3	M4	M5	M6
H_2O losses(% initial mass)	64.5	69.5	63	64	65.5	64
Dry matter losses (% initial mass)	20.5	21	21	20	20	20

But, if we make a close-up on water content, at the end, after 28 days composting, the differences appear clearly. Due to mixing, H_2O is more homogeneous in the waste and it seems that no limitation due to insufficient water content exists. Two inlets homogenise air flux and consequently the water content inside the waste.


Model M1

Model M3

Model M5

Model M6

Figure 4: Comparison of apparent volumetric mass H_2O fields for from letf to right M1 and M3 (M1 +mixing) and M5 and M6 (M5 + mixing). Water content is maximum in red and equal to zero in dark blue.

5. CONCLUSIONS

We have presented a new model of reactive transport in porous media where biological reactions take place. This is developed at the so called Darcy's scale, *i.e*, a scale where the influence of the porous medium appears in effective coefficients. This model takes into account several couplings, drying and also a biological model.

After a validation test by comparing the results given by the model to the one obtained during an experimental campaign, we have used this to test several situations in order to optimize the sanitation process but also the dry matter consumption and H_20 evaporation. It appears that it seems necessary to mix the waste once during the process in order to ensure that all the porous medium reach the sanitation criteria, except in one case, injecting the same flow rate using two inlets and maintaining the ground and incoming air inside the waste at 30°C. So getting back the heat from the outlet to preheat the incoming air could allow, in some cases, to reach the sanitation criterion.

The next step in our case will be to take into account the evolutions of transport parameters and to validate our model on several wastes using pilot experiments. This has begun and several input data are now available from Huet et al. 2012. He has shown some important variations on porosity and permeability inside the waste during the composting process. Taking that into account could allow us to have a more accurate model.

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(85) EXTERNAL INFLUENCES ON THE ENERGY EFFICIENCY OF COMPOSTING PLANTS

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EXECUTIVE SUMMARY

With regard to energy efficiency as a major tool to reduce the use of fossil energies and to enable the conversion to a sustainable energy system, also biological waste treatment technologies have to improve their specific energy efficiency i.e. the energy used for a specific amount of biological waste utilised. To increase the energy efficiency of composting plants two general approaches seem to be viable.

The first approach is to take a look at the composting plants itself. What technology is used, which kind and which quantities of biological waste is utilised, and which amount of energy is used (see Figure 1 left side). Technologically similar plants show considerable differences in their specific energy efficiency. Based on that fact it should be possible to identify the factors relevant for the energy efficiency.

The second approach investigates the composting plant not as an isolated phenomenon, but looks at it as part of the whole waste utilisation chain, from the arising of the waste till the application of the waste product. This chain strongly influences the size and technology of a plant and therefore its energy efficiency. Although this has little meaning for existing plants, it can explain some aspects of their current energy efficiency and might avoid unjust comparisons.

Both approaches were followed in a still on-going research project with the goal to increase the energy efficiency of composting plants in Germany. To provide data for the internal efficiency factors a survey was undertaken. This survey covered all 440 composting plants subjected to the Federal German Compost Quality Assurance Organisation (BGK). These plants, which exclusively utilise source separated organic waste, represent 75% of the total composting capacity in Germany, which is at around 10 Tg – ten million tons – annually (BGK, 2011).

For the second approach a mathematical model of the utilisation chain was developed and implemented with GoldSim® into a computer model. Data from the survey and from literature can be used to run specific scenarios, which compare the influences on the energy efficiency from internal and external factors. Figure 1 (right side) shows the general model structure, explained in detail on the following pages, positioning the composting plant within the utilisation chain.

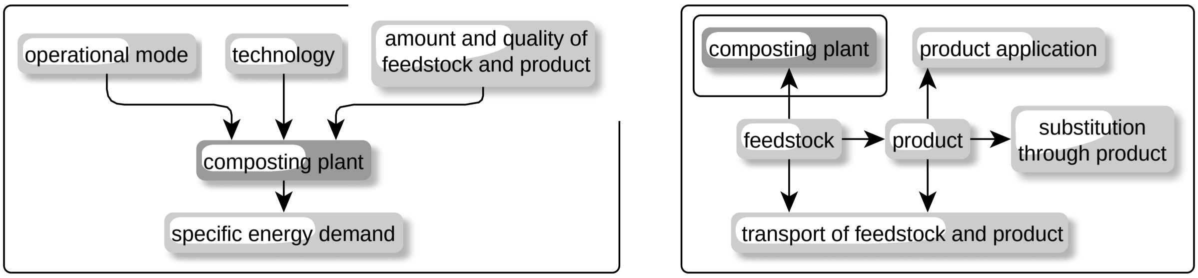


FIGURE 1 **Approach 1 (left) and Approach 2 [Model structure] (right)**

Expected prior to the survey, most plants could not provide detailed energy consumption of single parts or processes, since they have only one electricity meter and one bill for diesel. Notwithstanding, enough data about the overall energy efficiency of different plants was collected to draw the conclusion that aeration and its operational mode is one key internal factor. And since the decision about aeration or no aeration depends strongly on the amount and quality of the feedstock, this internal factor is also influenced by an external factor, whereas most external factors are also influenced by other external factors.

Although, based on literature, the energy demand of aeration can be decreased by simple changes of the operation mode; a composting plant without aeration would in most cases still be more energy efficient. But, as shown on the following pages, energy efficiency depends also on hardly influenceable factors. In addition, some plants might be less energy efficient than others, but in turn they might be more efficient in land use, feedstock utilisation or emissions.

1 INTRODUCTION

In contrast to the utilisation of organic waste through anaerobic digestion or combustion, composting yields no usable energy, apart from organic heat which provides an intensive germ reduction and evaporates a lot of the substrates water content. The aim of composting is primarily to produce an organic fertiliser respectively a valuable soil amendment and not to convert organic carbon into energy. Notwithstanding, like all other waste treatment technologies also composting should be as energy efficient as possible.

1.1 Background

Many studies address energy efficiency as part of evaluating technologies for treating organic waste. A recent example is a study about the costs and benefits of an optimised bio-waste utilisation (UBA 2010), published by the Federal Environmental Agency. While this is a good approach to evaluate among others the energy efficiency of one or more technologies, it helps little to improve the existing treatment plants.

In Germany exist around 950 composting plants with a combined treatment capacity of approximately 10 Tg (BGK, 2011). The combined treatment capacity for all organic waste – composting plants included – is at around 13 Tg (DESTATIS, 2010). It can be said that composting plays a key role in the treatment and utilisation of organic wastes in Germany. Therefore, finding ways to improve its energy efficiency is a worthwhile goal.

1.2 Research objectives

The first of two main objectives was to find out where in detail composting plants where not as energy efficient as possible. This should provide knowledge for plant operators to improve the energy efficiency in the short-term, through changes of the plant operations, e.g. different operational modes for the aerators. In the midterm, improved plant designs and technological updates, e.g. ventilation shaft systems with little friction loss should improve the energy efficiency further.

The second main objective should provide decision makers in administration and policy with a sound knowledge base to improve the plants energy efficiency in the long-term, through the identification of external influence factors. These factors include e.g. necessities for energy intensive aeration and exhaust air treatment.

2 METHODOLOGY

To achieve the first objective, data about the energy use of composting plants had to be collected and analysed. The results were partly integrated into a computer model which was developed to achieve the second objective. Since the results of this research are heavily influenced by the application of the model, the second subchapter will detail its development and function.

2.1 Data collection

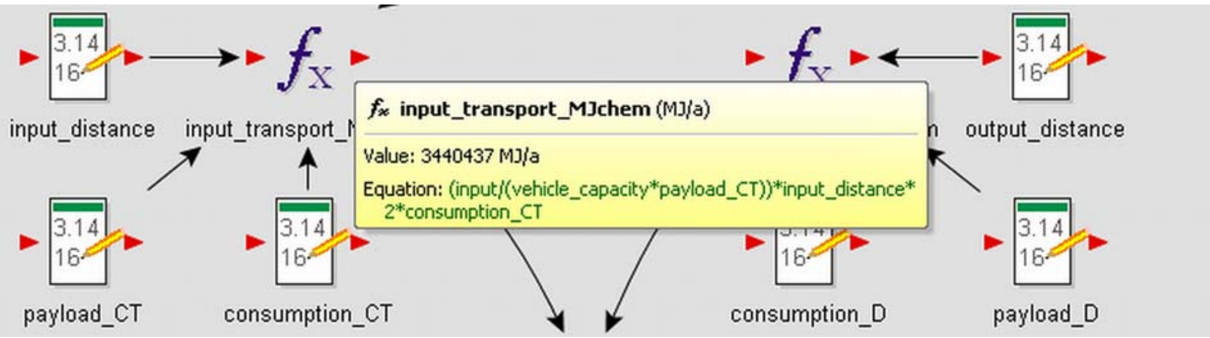
To provide appropriate measures and strategies for composting plants to improve their current energy efficiency a survey was undertaken. The survey was done with an electronic form in Portable Document Format, which could after completion either be printed or directly sent via e-mail. Contacts which did not reply in time were requested to send back the form a second and if necessary a third time, which increased the response rate considerable. The survey covered all 440 composting plants subjected to the Federal German Compost Quality Assurance Organisation (BGK). These plants represent 75% of the total composting capacity in Germany, which is at around 10 Tg annually (BGK, 2011). In total 59 plants with a combined treatment capacity of 1.2 Tg took part in the survey, which means a response rate of 16%, calculated in treatment capacity. Therefore, a good database was provided to investigate several aspects of the plants operations. However, as expected prior to the survey, most plants could not provide detailed energy consumption of single parts or processes. The main reason for this is that at most plants there is only one electricity meter and one bill for diesel. To obtain detailed data, several meters would have to be installed and the fuel consumption of single machines would have to be accurately recorded. Nonetheless, the total energy efficiency of several composting plants, together with data relating to external influence factors could be obtained.

2.2 Model building

Based on a combination of data from the survey and data from literature a theoretical model of the organic waste utilisation chain was created. The theoretical model was then refined with respective equations into a mathematical model, which was implemented as a computer model, using the simulation software GoldSim®. With different scenarios it is possible to compare the impact of external and plant internal energy consumptions on the whole utilisation chain.

A model should always be as transparent as possible in order to be evaluated and possibly improved by a large number of people. This goal is pursued in the on-going development of the current model and implemented through some principles. The first principle is the visualisation of system borders through the graphical arrangement of calculation elements. Figure 4 on the following page shows the main visualisation of the model how any user of the model would see it. The different coloured areas represent all parts of the system and the system boundaries at once. In case of a further development with addition of more complexity, the many more calculation elements would be combined to related groups, which would be illustrated as group elements in the main visualisation again. In effect this would create a model hierarchy.

This principle also allows avoiding oversized and hardly comprehensible equations. Figure 2 shows on the left side three data elements which provide input to calculate the annual energy demand for the collection and transport (CT) of the organic waste. The automatically drawn arrows represent always a flow of data, which allows a fast understanding of the calculation methods and prevents wrong links between elements during the building of the model. For a deeper understanding of the calculation methods pop-up windows allow a quick look at the condition and function of any element.



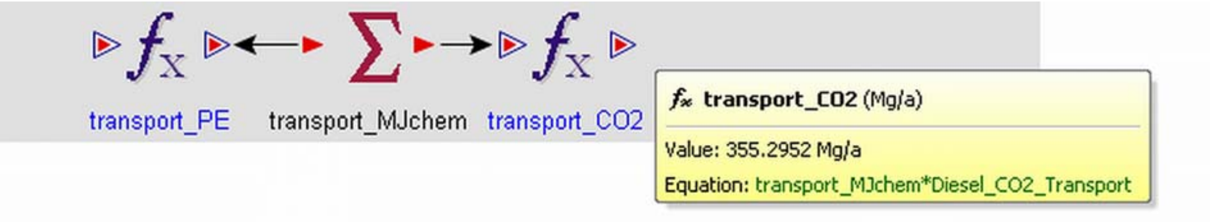
input_transport_MJchem (MJ/a)

Value: 3440437 MJ/a

Equation: (input/(vehicle_capacity*payload_CT))*input_distance*2*consumption_CT

FIGURE 2 **Model detail**

In order to preserve a comprehensive view of the model it is necessary to limit the display of elements respectively their relation to other elements. The pop-up window in Figure 3 shows the equation to calculate the CO_2-emissions caused by transport. The conversion factor necessary for this calculation (Diesel_CO2_Transport), a data element is not visible in the overview because many other calculation elements use it too and the many relation arrows would obstruct the view.



transport_CO2 (Mg/a)

Value: 355.2952 Mg/a

Equation: transport_MJchem*Diesel_CO2_Transport

FIGURE 3 **Model detail**

Another principle which shall provide a fast comprehension of the model is the use of easy recognisable icons for every element. This principle in not adopted yet, but it is quite easy to implement and should yield a considerable benefit. For example, the developer and user would not have to read the name of the element but could directly navigate within a coloured area to a CO_2-Icon to look at its value or equation.

Since the model can be evaluated by all partners of the on-going project, it will undergo several changes until the end of the project and hopefully also beyond that point. Although the development is done with proprietary software, the transparency of the model should allow an easy adaption to other modelling software, if necessary.

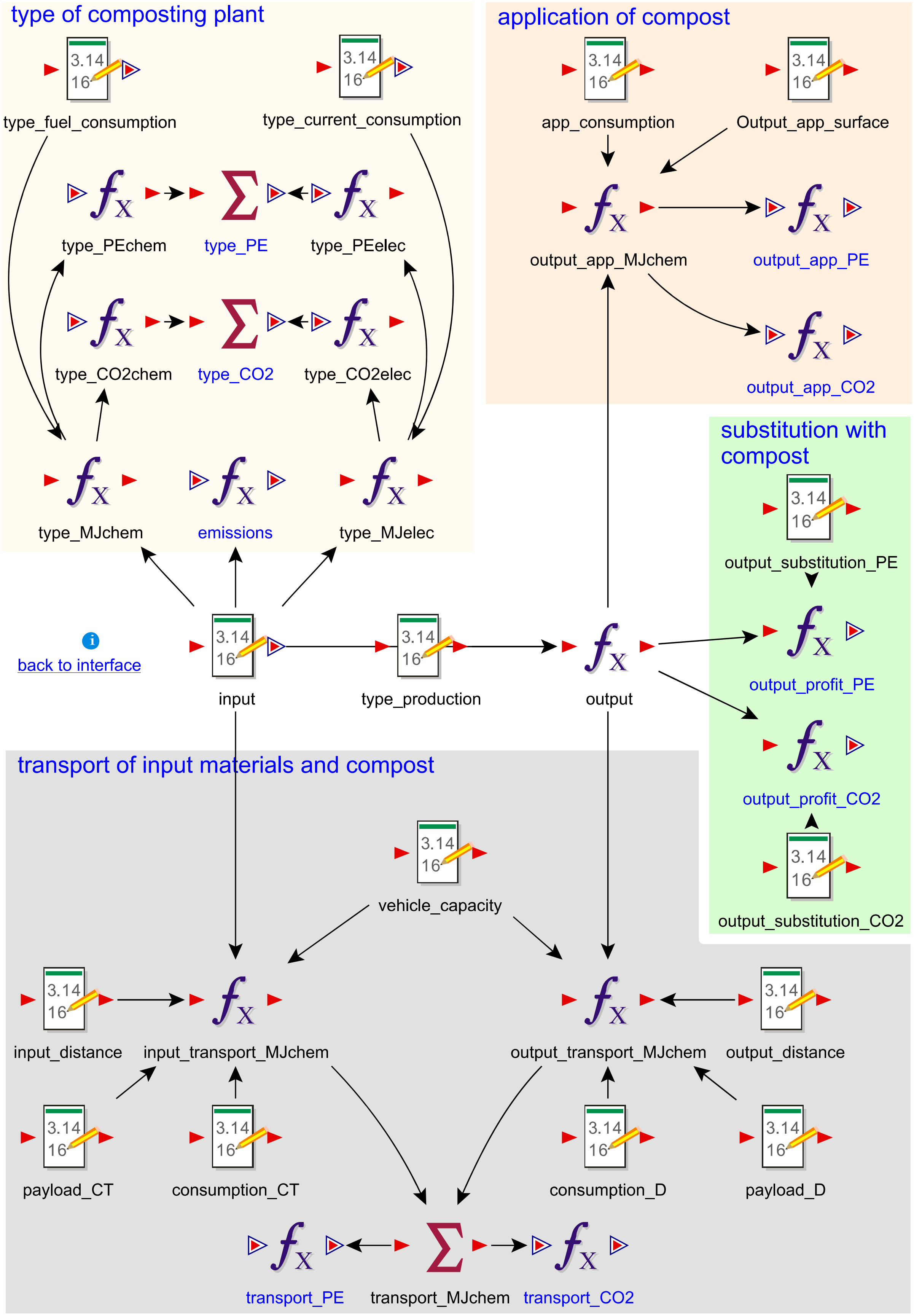


FIGURE 4 **Model overview**

3 RESULTS

Based on the survey, the average energy demand in form of electricity and diesel for treating one Mg organic waste in an enclosed and aerated composting plant is around 95 $\text{MJ}_{el}/\text{Mg}_{Input}$ and 65 $\text{MJ}_{di}/\text{Mg}_{Input}$. An open and not aerated plant has an average demand of 4 $\text{MJ}_{el}/\text{Mg}_{Input}$ and 92 $\text{MJ}_{di}/\text{Mg}_{Input}$. Although there is hardly any data on single machines, it is possible to conclude from these overall energy demands that the aeration is a key energy consumer within a composting plant. Also Springer (2011) could substantiate the assumption that aeration is the key factor for the specific energy demand of aerated composting plants. Nolden (2000) investigated the influence of different aeration modes on the energy demand and on the decomposition process. He found out that with a relatively easy change of the aeration mode the energy demand of the aeration could be reduced by 36 %.

The current version of the model is not yet usable to evaluate the most important external influences on the energy efficiency of composting plants. These are the amount and type of organic waste to be utilised, the demand for products based on organic waste, the existing development around a composting plant, and all laws and regulations which affect them in turn. This last factor is probably the most difficult to integrate into the model since it consists of a number of complex regulations which partly vary regionally and which require many data to provide a usable result.

One factor which is already included in the model is the electricity mix, which comes in form of the primary energy factor. This factor determines on a large scale how much primary energy is used by composting plants and hence, how good their primary energy efficiency is. Especially plants with forced aeration are currently adverse affected, since the ventilation demands usually a lot of electricity, which has a high primary energy factor. Therefore, with the progressing change of the electricity mix towards more renewable energies, the primary energy balances improve automatically over time. In practice it would already be possible for electricity intensive plants to improve their balance today. They just would need to purchase solely electricity from renewable resources.

4 DISCUSSION

Although energy efficiency is also important for environmental considerations, in the end only economic parameters – mostly calculated short-term – decide which kind of composting plant is built and how it is operated. The energy consumption has an economic impact especially on the operation costs of composting plants. Assuming the current prices for electricity and diesel being at 0.05 €/MJ_{el} (0.18 €/kWh) and 0.04 €/MJ_{di} (1.40 €/l), the average energy costs for treating organic waste would be at around 7.50 €/Mg_{Input} for an enclosed and aerated plant. For an open and not aerated plant it would be at around 4.00 €/Mg_{Input}. Compared to the income per treated Mg organic waste, which is depicted in Figure 3 for several composting plants, the influence of energy efficiency could be characterised as quite low. Therefore, as long as energy prices stay at that level, there is probably not much incentive provided by financial considerations. As a consequence, subsidies or a change of regulations would be necessary to stimulate the improvement of energy efficiency.

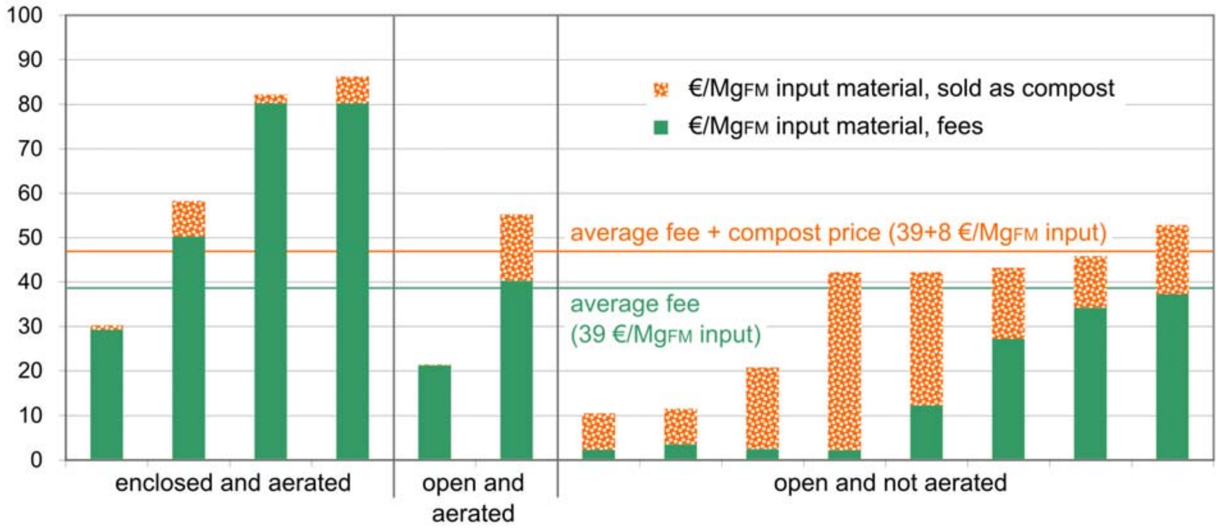


FIGURE 5 **Fees and compost prices of 14 composting plants (Meyer et al. 2011)**

A viable option to achieve the required stimulation might be through an energy performance certificate in the style of similar certificates for buildings. Springer (2011) developed in his dissertation an online database which can compare the overall energy efficiencies with data provided by the plant operators. Beside the direct benefit for the operator – knowing the rank of the own plant compared with similar other plants – this online service could be the first step to generate energy performance certificates for composting plants. These would bring transparency to the energy requirements of the compost sector and could improve the public perception in regard to costs and benefits of compost. It would probably be an ideal addition to the compost quality assurance provided by the BGK (2011).

5 CONCLUSIONS

To improve the current energy efficiency of single composting plants it is helpful to compare them with similar plants in order to recognise possible weaknesses in their operation. The comparison of the overall specific energy demand – energy used to treat and utilise a specific amount of organic waste – is a good starting point. To determine and improve the specific issues responsible for inefficiencies, it is necessary to investigate further in each case, since existing data covers mostly only the overall consumption of electricity and diesel.

The improvement of the energy efficiency of composting plants in the long run – with technological updates and new constructions - should also be based on external influence factors. These are, among others the amount and type of organic waste to be utilised, the demand for products based on organic waste, the existing development around a composting plant, and not to forget all laws and regulations which affect them in turn. They all influence to a large extend the decisions about size and technology of a composting plant and therefore also its energy efficiency. This realisation should also prevent any unjust comparisons between technologically different plants.

The current version of the computer model described in this paper is not yet sufficient to provide a thorough evaluation of all external influence factors in regard to the energy efficiency of composting plants. However, it is already capable to compare the internal energy demand with external energy demands from the waste utilisation chain before and after the composting plant. A further development of the model, with the maintained developmental principles in mind, could provide a better understanding of external influences and with that enable better policies to increase the energy efficiency of the whole compost sector.

6 ACKNOWLEDGEMENTS

The Authors wish to thank the German Federal Ministry for the Environment, Nature Conservation and Nuclear Safety (BMU) for financial support of the project “Increasing the energy efficiency in biological waste treatment” (FKZ-No. 03KB022B), within which the data collection and model building, described in this paper, was undertaken.

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(88) MULTI-COMPONENT HEAT AND MASS TRANSPORT MODEL FOR COMPOSTING PROCESS: EXPERIMENTAL VALIDATION

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EXECUTIVE SUMMARY

Composting consists in an aerobic process where the organic matter from wastes is biodegraded and converted into a stable granular material called compost. Even if composting is considered to be based on natural phenomenon, it is governed by very complex mechanisms, involving many parameters such as quality and accessibility of the nutriments for micro-organisms, local oxygen content, temperature of the medium, pH, moisture content, and so on (Mustin, 1987, Diaz *et al.*, 2007;). These parameters directly affect biodegradation kinetics. Moreover, they are interdependent and impacted by the operating conditions, which make difficult the understanding of the process.

In order to understand these mechanisms, a multi-component transport model including a biological model and taking into account the drying phenomenon has been developed. This is the first time such a model is carried out. And in general, existing models in literature ignored the drying phenomenon. However, it has been demonstrated in Pujol *et al.* (2010) that the effects of drying on the composting process could have a dramatic impact and must be considered in composting models.

The aim of this paper is to confirm these results, and validate the model. In order to do that, experiments at pilot scale have been carried out. Gas concentrations and temperatures were recorded and compared with those obtained by numerical simulations. An analysis of this comparison is presented. It shows that the model reproduces quite well what it can be observed during experiments, especially for temperature and oxygen (but still a coefficient problem for carbon dioxide). Moreover, the organic matter balance is very good since there is less than 3% of error between simulated and experimental results.

1 INTRODUCTION

1.1 Background

Composting is a process whereby biodegradable organic material from waste is converted into a stable granular material called compost. The biodegradation of this organic fraction is operated by aerobic micro-organisms, which consume oxygen from air and organic matter to produce mainly carbon dioxide, water and heat. This leads to a rise in temperature over several consecutive days allowing the sanitation of the product. Applying the obtained compost to land improve soil structure and enrich the soil nutrient content.

From an industrial point of view, composting consists in controlling the organic waste biodegradation in order to reduce stabilization times, improve and control compost quality, and limit environmental and sanitary impacts. Since the efficiency of industrial composting processes is largely controlled by oxygen availability within the waste, the acceleration of the biological activity is generally obtained by improving oxygen input into waste piles: windrow turning and/or forced aeration of the windrow.

Nevertheless, the overall efficiency of the process is also controlled by environmental factors, such as temperature, moisture content, or drying phenomenon coupled with biological activity. These parameters and interactions could partially be determined by experiments at pilot and/or industrial scales. But because of the existing difficulties to control the operating conditions, modeling seems interesting and quite relevant.

Therefore, using a model allows understanding the mechanisms and selecting the most relevant parameters. In addition, the comparison of scenario may be useful to optimize the composting methods and reduce time and costs for operational issues.

1.2 Research objectives

A deep understanding of the phenomena which occur during composting seems to be a necessary step to improve the process' control and to produce good compost by limiting environmental and health impact. In order to do that, modelling is an interesting tool, since simulating the behaviour of the waste during composting would help to better understand the complexity of the phenomena and their interactions. This would also allow identifying the parameters, or couple of parameters, which could be levers to cope with operational issues. Eventually, such a model could be the basis of a steering tool at an industrial scale and could help improve the process much as from the point of view of the product quality as of energy consumption.

Hence, this paper presents the development of a model that takes into account heat and mass transport phenomena coupled with aerobic biodegradation and drying. The model is implemented under COMSOL Multiphysics®. In a previous work (Pujol *et al.*, 2011), different local equilibrium and non-local equilibrium approaches were evaluated to simulate drying phenomena at the Darcy's scale. The model was then used to simulate the impact of drying on the composting process, when no external water is added to the waste, in order to understand the relative importance of the different mechanisms. It has shown that the drying must be taken into account, since its impact on evaporated water, reached temperature and degraded organic matter could be very important. Then, it has been concluded that the model should be compared with experimental results in order to validate the model's results. This is the aim of this paper.

2 COMPOSTING MODEL

We want to use a model which is distributed (2D model), and allow to describe the multi-component reactive transport, including drying phenomena and biological mechanisms. The studied media can be considered as a porous medium. Therefore, the model must take into account the inherent specificity of these media and, hence, comes within the scope of multi-scale problems.

2.1 Upscaling features and hypotheses

When developing transfer models in reactive porous media coupled with drying phenomena, one of the main difficulties lies in the necessity to take into account the mass and heat transfers within the porous medium. As for gas/liquid equilibrium processes, continuity equations governing mass and heat transfers are only valid for continuous media. Consequently, these equations need to be considered at pore scale, because continuous phases can only be distinguished at this scale.

Nevertheless, given the actual (and future) computing power, it is not possible to simulate, at pore scale, the dynamic behaviour of a massif of wastes the size of a composting windrow. Then, the solution is to obtain a transfer model at an intermediary scale, equivalent to several sizes of pores, and called Darcy scale. Several methods have been developed to get this kind of models: volume averaging, homogenization method … This kind of approach allow developing equations system at Darcy scale in the case of drying of a porous medium. Volume averaging applied to drying in porous media has been detailed in numerous works (**Erreur ! Source du renvoi introuvable.** ; **Erreur ! Source du renvoi introuvable.** ; **Erreur ! Source du renvoi introuvable.** ; **Erreur ! Source du renvoi introuvable.** ; **Erreur ! Source du renvoi introuvable.** ; **Erreur ! Source du renvoi introuvable.**) which have been a base for the development of our own model. The upscaling is not presented in this paper, and we refer the reader, for this part, to the literature. Thanks to this method, a multi-component transfer model including a biological model and drying phenomena has been carried out. Nevertheless, several hypotheses are assumed before obtaining the final macroscopic model:

- The developed model takes into account three phases: gas, liquid and solid.
- The matrix of wastes is considered homogeneous.
- Only the gas phase is considered mobile. Here, we made the important restrictive assumption that the liquid water is immobile given the low saturation. This approximation is based on the few amounts of leachate produced in composting plants compared to the water initially present in the wastes.
- Four species are considered in the gas phase: oxygen, carbon dioxide, water vapour and nitrogen.
- Dissolution of gas species in the liquid phase is neglected.
- Effective diffusion of the species in the gas phase is considered isotropic.

- The thermal local equilibrium hypothesis is considered, which means that the temperatures in the three phases are the same.
- The local chemical equilibrium hypothesis is assumed for water. However, for numerical reason, a local non-equilibrium formulation is used to express the exchange term between gas phase and liquid phase which is expressed as equation (1) (Pujol *et al.*, 2011).

  $$\dot{m} = \sigma\varepsilon_g\rho_g\left(\omega_{H_2O,g,eq} - \omega_{H_2O,g}\right) \quad (1)$$

  σ is inversely proportional to the time required to obtain the equilibrium within the pore (s^{-1}). A value of 2.5 s^{-1} is taken (Pujol *et al.*, 2011). ε_g is the porosity, ρ_g is the gas density, $\omega_{H_2O,g}$ is the mass fraction of vapour water, and $\omega_{H_2O,g,eq}$ is the mass fraction of vapour water at equilibrium, given by equation (2).

  $$\omega_{H_2O,g,eq} = \frac{P_{sat}}{P_g} M_{H_2O} \sum_{i=1}^{n} \frac{\omega_{i,g}}{M_i} RH \quad (2)$$

  P_{sat} is the vapour pressure defined by Antoine equation (3):

  $$P_{sat} = \exp\left(\left(C_1 - \frac{C_2}{T + C_3}\right) \times \ln(10)\right) \quad (3)$$

  and RH is the sorption isotherm governing the equilibrium between the liquid phase and the gas phase for water. Its expression is based on Oswin expression (4):

  $$RH = \frac{\left(\frac{MC}{A(1-MC)}\right)^{\frac{1}{B}}}{1 + \left(\frac{MC}{A(1-MC)}\right)^{\frac{1}{B}}} \quad \text{(Oswin, 1946) (4)}$$

Now that the hypotheses have all been set up, the global model can be introduced.

2.2 Model presentation

The equation of mass conservation in the gas phase is described by equation (5).

$$\frac{\partial(\varepsilon_g\rho_g)}{\partial t} + \vec{\nabla}.(\varepsilon_g\rho_g\vec{u}_g) = \dot{m} + R_{O_2,g} + R_{CO_2,g} \quad (5)$$

$\vec{u}_g$ is the intrinsic average velocity given by equation (6).

$$\vec{u}_g = -\frac{\kappa_a}{\varepsilon_g\eta_g}\left(\vec{\nabla}P - \rho_g\vec{g}\right) \quad (6)$$

In Equation (2), $\dot{m}$ is a mass exchange term between the gas phase and the liquid phase for water typically linked to the drying phenomenon (evaporation/condensation). $R_{O_2,g}$ and $R_{CO_2,g}$ are terms for the oxygen consumption and carbon dioxide production due to the biological activity.
The gas phase behaves like an ideal gas, it is then possible to use equation (7).

$$\rho_g = \frac{PM}{RT} \quad (7)$$

The mixture molar mass is given by equation (8).

$$M = \frac{1}{\sum_{i=1}^{n} \frac{\omega_{i,g}}{M_i}} \quad (8)$$

The mass conservation for each species *i* in gas phase is given by equation (9).

$$\frac{\partial(\varepsilon_g\rho_g\omega_{i,g})}{\partial t} + \vec{\nabla}.(\rho_g\vec{u}_g\omega_{i,g}) = \vec{\nabla}.\left(\varepsilon_g\rho_g\overline{\overline{D}}^*_{ig}.\vec{\nabla}\omega_{i,g}\right) + R_{i,g} \quad (9)$$

where $i = O_2, N_2, CO_2 \; (R_{N_2,g} = 0)$.

The equation for water vapor is given by equation (10).

$$\frac{\partial(\varepsilon_g\rho_g\omega_{H_2O,g})}{\partial t} + \vec{\nabla}.(\rho_g\vec{u}_g\omega_{H_2O,g}) = \vec{\nabla}.\left(\varepsilon_g\rho_g\overline{\overline{D}}^*_g.\vec{\nabla}\omega_{H_2O,g}\right) + \dot{m} \quad (10)$$

Water can be found in the gas phase but it is mainly present in the liquid phase where the mass conservation of water is described by equation (11).

$$\frac{\partial\left(\varepsilon_l \rho_l \omega_{H_2O,l}\right)}{\partial t} = -\dot{m} + R_{H_2O,l} \quad (11)$$

$R_{H_2O,l}$ is the term of water creation due to the degradation of organic matter:

Given the assumption of local equilibrium which has been made for the temperature continuity, the heat equation can be written as (12).

$$\left(\varepsilon_g \rho_g C_{pg} + \varepsilon_l \rho_l C_{pl} + \varepsilon_s \rho_s C_{ps}\right)\frac{\partial T}{\partial t} + \rho_g C_{pg} \vec{u}_g . \vec{\nabla} T = \vec{\nabla}.\left(\lambda^* \vec{\nabla} T\right) - \dot{m}\Delta H_{vap} + R_T \quad (12)$$

ΔH_{vap} is the enthalpy of vaporization of water. R_T is the heat production due to the biological activity:

Finally, the model used to describe the organic matter evolution is neither too simple nor too complex. Complexity of biological models is due to the number of bacterial populations and the considered division of the substrate. The model chosen for this work is inspired from the one developed by Pommier *et al.* (2008). The organic matter is divided in three solid fractions: the first one is readily hydrolysable (X_{Rb} (kg.m^{-3})), the second one is slowly hydrolysable (X_{Sb} (kg.m^{-3})), and the third one is inert (X_I (kg.m^{-3})). The two first fractions are progressively hydrolysed in the biofilm, producing a readily assimilated soluble fraction, (S_r (kg.m^{-3})). This is the fraction that is directly consumed by the bacteria. Only one population is considered, X_a (kg.m^{-3}). Bacterial growth is modelled by a Monod equation with a limitation by the quantity of available S_r. These micro-organisms also consume oxygen ($R_{O_2,g}$), and produce carbon dioxide ($R_{CO_2,g}$), water ($R_{H_2O,l}$) and heat (R_T). Mortality of these bacteria is taken into account in the model, creating inert material and readily biodegradable solid fraction. All these phenomena have already been presented in Pujol *et al.* (2011) and are described by Equation (13) to Equation (17).

$$\frac{\partial X_{Rb}}{\partial t} = -\gamma_T \tau k_{Rh} X_{Rb} + \gamma_T \tau \varphi_{O_2}\left(1 - f_I\right) b X_a \frac{0.844}{0.706} \quad (13)$$

$$\frac{\partial X_{Sb}}{\partial t} = -\gamma_T \tau k_{Sh} X_{Sb} \quad (14)$$

$$\frac{\partial X_I}{\partial t} = \gamma_T \tau \varphi_{O_2} f_I b X_a \frac{0.844}{0.706} \quad (15)$$

$$\frac{\partial X_a}{\partial t} = \gamma_T \tau \varphi_{O_2} \mu_{\max} \frac{S_r}{K_{S_r} + S_r} X_a - \gamma_T \tau \varphi_{O_2} b X_a \quad (16)$$

$$\frac{\partial S_r}{\partial t} = \gamma_T \tau k_{Rh} X_{Rb} \frac{0.9375}{0.844} + \gamma_T \tau k_{Sh} X_{Sb} \frac{0.9375}{0.844} - \gamma_T \tau \varphi_{O_2} \frac{\mu_{\max}}{Y_a} \frac{S_r}{K_{S_r} + S_r} X_a \frac{0.9375}{0.844} \quad (17)$$

γ_T, τ and φ_{O_2} are limitation terms of the biodegradation due to thermal effect (Rosso *et al.* (1993)), moisture limitation effect (Pommier *et al.* (2008)) and oxygen availability (Mason (2006)):

$$\gamma_T = \frac{\left(T - T_{\max}\right)\left(T - T_{\min}\right)^2}{\left(T_{opt} - T_{\min}\right)\left[\left(T_{opt} - T_{\min}\right)\left(T - T_{opt}\right) - \left(T_{opt} - T_{\max}\right)\left(T_{opt} + T_{\min} - 2T\right)\right]} \quad \text{for } T_{\min} < T < T_{\max} \quad (18)$$

$$\tau = \begin{cases} 0 & \text{if } \omega_{\mathrm{H_2O,l}} \le \omega_{\min} \\ \dfrac{\omega_{\mathrm{H_2O,l}} - \omega_{\min}}{\omega_{\max} - \omega_{\min}} & \text{if } \omega_{\min} < \omega_{\mathrm{H_2O,l}} < \omega_{\max} \\ 1 & \text{if } \omega_{\mathrm{H_2O,l}} \ge \omega_{\max} \end{cases} \quad (19)$$

$$\varphi_{O_2} = \frac{\omega_{O_2}}{K_{O_2} + \omega_{O_2}} \quad (20)$$

Terms of production and consumption are described by equations (21) to (24).

$$R_{O_2,g} = -\gamma_T \tau \varphi_{O_2}\left(\frac{1 - Y_a}{Y_a}\right)\mu_{\max}\left(\frac{S_r}{K_{S_r} + S_r}\right)\frac{X_a}{0.706} \quad (21)$$

$$R_{CO_2,g} = \gamma_T \tau \varphi_{O_2} \left(\frac{Y_{CO_2}}{Y_a} \right) \mu_{\max} \left(\frac{S_r}{K_{S_r} + S_r} \right) \frac{X_a}{0.706} \quad (22)$$

$$R_{H_2O,g} = \delta \gamma_T \tau \varphi_{O_2} \left(\frac{1 - Y_a}{Y_a} \right) \mu_{\max} \left(\frac{S_r}{K_{S_r} + S_r} \right) \frac{X_a}{0.706} \quad (23)$$

$$R_T = \frac{4.6 \times 10^5}{M_{O_2}} \gamma_T \tau \varphi_{O_2} \left(\frac{1 - Y_a}{Y_a} \right) \mu_{\max} \left(\frac{S_r}{K_{S_r} + S_r} \right) \frac{X_a}{0.706} \quad (24)$$

The commercial software COMSOL Multiphysics® has been chosen to carry out the numerical simulations based on this system of equations. The consistency of the model has already been demonstrated in Pujol *et al.* (2011). Therefore, it is now necessary to compare experimental results with simulation's ones in order to examine the quantitative and qualitative validity of the developed model.

3 PRESENTATION OF THE STUDY

3.1 Experiments presentation

The experiments led in VERI (Limay) have been carried out at pilot scale and consist in the simulation of the composting process of wastes in a controlled environment. To do that, four cylinders of about 156 L have been used (FIGURE 1), in order to test the repeatability of the process. Identical conditions have been imposed for all the pilots.

A grid at the bottom of the pilot allows a homogeneous injection of air. This system allows supplying oxygen for aerobic bacteria within the studied wastes which is here a mix of biowastes and green wastes. Composting process by forced injection can then be simulated. Three different flow rates are set, Q1, Q2 and Q3. Q2 can be set at different time for each silo, because it depends on the moment when a certain temperature (defined by the experimenter) is reached. Q3 is set at the same moment for each pilot. Each modification of aeration results in a decrease (approximately divide by 2) of the previous rate.

The upper part of each pilot is closed by a flat top which can favour the condensation of the water vapour from the outlet gas. Then, the produced condensate can fall down within the wastes and influence the process, at least in the top part. This particularity should be taken into account in the results' discussion.

These experiments have allowed collecting data about the temporal evolution of temperature and gas concentration (oxygen and carbon dioxide) in the massif of wastes. In fact, grids have been directly installed in each cylinder where 14 temperature captors (thermocouples) and 6 gas probes have been fixed (the probes take a sample of the gas which is then analysed by micro-chromatography). FIGURE 1 indicates the location of each captor. Finally, the matter is characterized, providing amount of dry matter, organic matter, inert matter and the moisture content at the beginning and at the end of the experiment. This database can then be used for the validation of the developed model. Therefore, simulations have been carried out, reproducing experimental conditions used for the studied pilots.

(a) (b)

FIGURE 1 **(a)Photography of a cylindrical pilot used for the experiments and (b) location of the captors and the probes in the pilot.**

3.2 Presentation of the simulation case

It is necessary to define correctly the geometry on which the simulations will be carried out. Since, here, the studied domain is a cylinder, and because we assume that the matrix of wastes is homogeneous in the pilot, the best solution seems first to use the half of a 2D-slice and to work in a cylindrical coordinate system. But we want this model to be able to simulate the composting process on windrow geometry where this kind of system is not adapted. This is the reason why the model is directly written in a Cartesian system. Therefore, the geometry used for the simulations is a 2D slice of the silo, illustrated by FIGURE 2.

Only the heat equation is solved in the wall in order to simulate the thermal losses. A thermal exchange with the outside is taken into account. The intrinsic properties of the studied waste (mix of biowastes and green wastes) come from measures carried out at VERI (Limay) and LISBP (INSAT). Biological parameters were measured via respirometry at 20°C in Sapromat.

Simulation results are now compared with experimental results, in order to make sure of the good behaviour of the model.

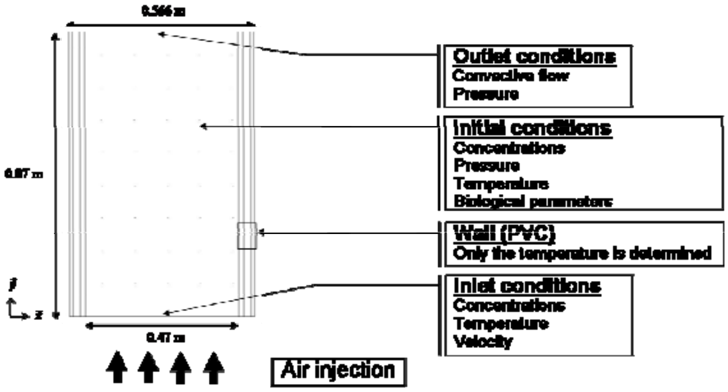


FIGURE 2 **Geometry and (initial and boundary) conditions used.**

4 RESULTS

4.1 Comparison between experimental and simulated results

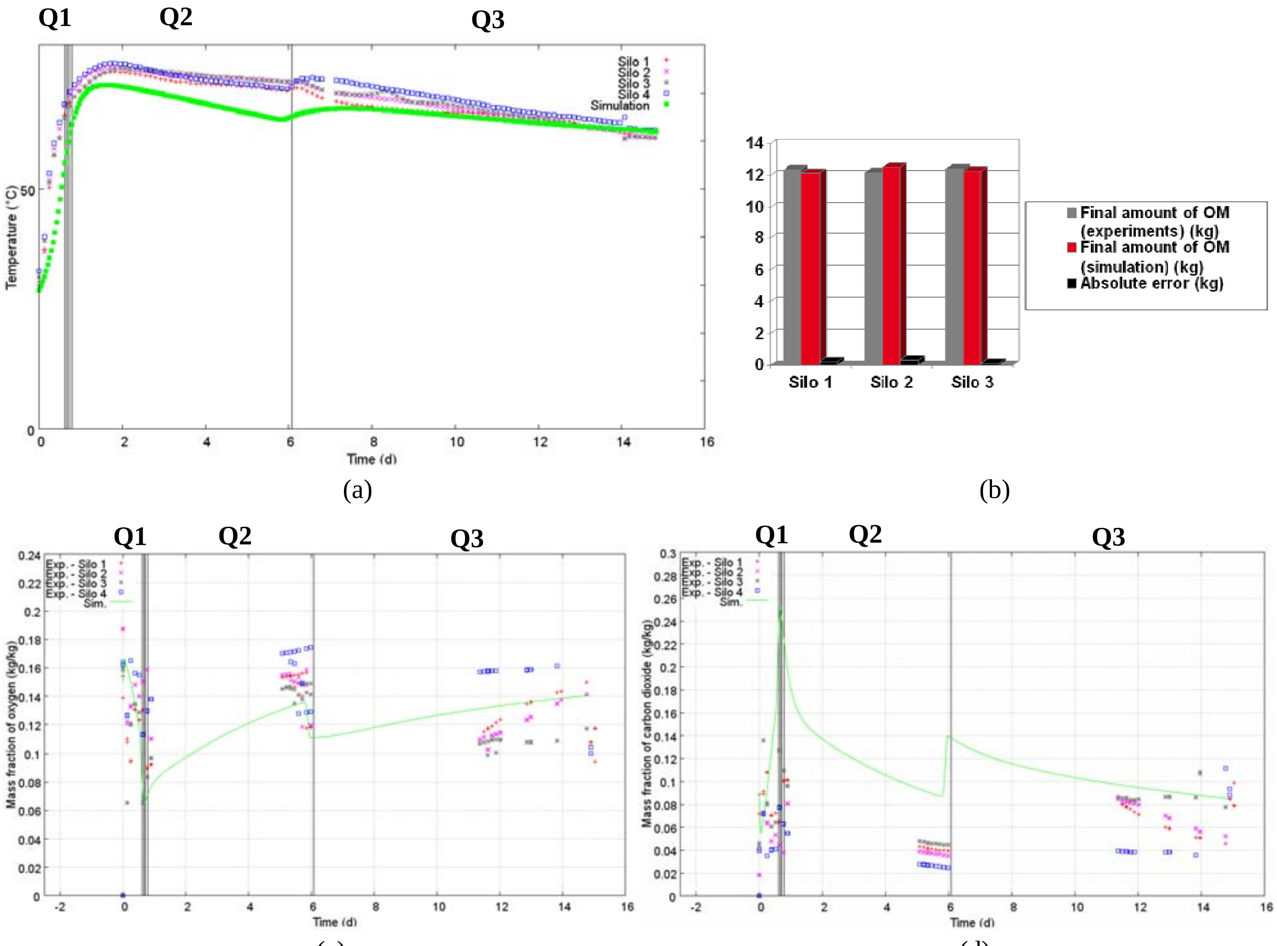
It has been decided to check the evolution of the temperature at T5 and the gas concentrations at O2 (FIGURE 3). These points have been chosen because they are located in the centre of the pilot: therefore, they are more representative of the composting process than the other points because less influenced by the boundary conditions.

Concerning the temperature, a difference of 3°C can be observed between the maximal reached temperatures from experiments and the ones from simulation. Moreover, experimental results and simulated results are quite close during all the process. But it can be noticed that the simulated temperature is “late” compared with experimental results. This can be explained by the fact that biological reaction has already begun when the experiment starts. Therefore, until the second aeration modification, simulated temperature is underestimated, with a maximal difference of 8°C just before Q2.

The oxygen concentration is well described by the model, but it’s not the case for the carbon dioxide. At the beginning of the process, concentration’s evolution is a bit erratic. Nevertheless, the model seems to catch well the tendency of this evolution, with an error that can reach 0.08 kg/kg. Between day 5 and 6, oxygen concentration is first underestimated, and then follow the experimental data. On the other hand, an important overestimation of carbon dioxide production is observed, even if the evolution profiles seem identical. At the end of the process, oxygen concentrations perfectly fit with those measured. Carbone dioxide concentrations are still overestimated, but are closer of the experimental data than between day 5 and 6.

It is not possible to have the amount of organic matter in the pilots at each moment. So, it is only measured at the beginning and at the end of the process. The quantities measured at the beginning are the values used as initial conditions for the biological variables in the model. Only final amount of organic matter are compared. The maximal error reached in this test between the final values of organic matter’s amount is equal to 2.71%.

These results are very satisfying and are good indicators of the good behaviour of the global model when limitations by water, oxygen and temperature are not too sensible. These results are very encouraging, even if other configurations must be tested to validate definitely the way the degradation of the organic matter is taken into account.



(a) (b)

(c) (d)

FIGURE 3 **Comparison between evolution of temperature (a), organic matter balance (b), and evolution of oxygen (c) and carbon dioxide (d) concentrations of experimental and simulations results.**

5 DISCUSSION AND CONCLUSIONS

Comparison between experimental and simulation results have pointed out that, globally, the model is able to follow the evolution of temperature and gases concentration during composting process at pilot scale. The results are quite promising, since the observed variables are the one in the centre of the geometry which are the most representative of the process. For the temperature, the maximal difference between simulation and experiments is lower than 8°C. This difference is certainly due to the fact that there is a delay in the simulated reaction compared to the experimental one. This delay has directly an impact on the maximal reached temperature and, therefore, the way the temperature decrease during the process. But because the intensity of the biological reaction must be equivalent in both cases, the impact on the oxygen production is not very important and we still get pretty good results. However, it has been noticed that the amount of produced carbon dioxide is overestimated. The biological activity is not the cause of these results: the parameter used for the carbon dioxide production applies to the total carbon dioxide produced: gas and dissolved, and not the gaseous part only. So this parameter should be corrected in order to describe the good evolution of carbon dioxide in the gas phase. Finally, the organic matter balance is very positive, since the error between simulation and experiments is lower than 3%.

Even if the model can give quite good results, improvements must be brought to the model, especially concerning the rise of the temperature at the beginning of the process.

Several explanations are being considered, but the most plausible seems to be that the reaction has already begun in the pilot when the first measures are done, which is not taken into account in the model. If this part is taken into consideration, it will surely enhance the results obtained, at least for temperature evolution. Concerning carbon dioxide evolution, as it has been said, the coefficient of carbon dioxide production must be reconsidered in order to fit with what we want to express: the production of carbon dioxide in the gas phase.

It would be interesting now to test other kind of wastes to make sure the model well describes the composting process, no matter what matrix is used. Finally the next step will be to test the model at an industrial scale and validate it the same way we did at pilot scale. If the model well reproduces what it can be observed on windrow, it could then be used to optimize the process (aeration conditions, windrow shape, compost quality, …) or to compare different scenarios (different pore sizes, different mix ratio, differences between indoors processes and outdoors ones…).

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Session 28

(51) COMPOST PELLETIZATION INCREASES THE PHYTOAVAILABILITY OF PHOSPHORUS IN COMPOST

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EXECUTIVE SUMMARY

Compost pellets, known as convenient soil amendments, have recently been attracting attention as the most readily abundant alternative source of soil nutrients, with the rising prices of chemical fertilizer as a backdrop. The objective of this study was to investigate the effect of pelletization on phosphorus (P) phytoavailability in compost. Greenhouse experiments were conducted to compare P availability between two forms of compost, pelletized compost (PE) and the corresponding raw material (RM) combined with a single superphosphate (SSP), using komatsuna (*Brassica Campestris* L.) and buckwheat (*Fagopyrum esculentum* Moench) as indicator crops. The compost used in this study was a mixture of cattle manure compost and poultry litter (1:1; v/v). The results indicate that PE fertilized plants yielded more komatsuna shoots than RM fertilized plants. Total P uptake increased curvilinearly with application rates for all three fertilizer materials. The relationship between P uptake and rate of P application fit well to a square root model. The average P recovery rate of PE was 12.5% and 24.5% for komatsuna and buckwheat, respectively, while that of RM was 8.6% and 16.3%, respectively. The P recovery rate of PE was comparable to that of SSP. The improvement in phytoavailability of compost P by pelletization can be explained by considering that pelletization could limit the contact of soil with compost P, possibly decreasing P precipitation and adsorption on soil colloids. Although further research is needed under field conditions and on more soil types for fertilizer application recommendation, the molding of compost into pellets appears to be desirable for not only improving crop yields and economic returns but also minimizing soil eutrophication and environmental problems.

1 INTRODUCTION

The annual amount of livestock excrement in Japan was estimated to be approximately 87 Mt, which is equivalent to 21% of the 419 Mt industrial waste discharged in 2007. (Ministry of the Environment Government of Japan, 2010) Approximately 90% of livestock excrement is now available as fertilizer and/or soil amendment for farmlands. In Japan, livestock excrement is mainly processed into compost; however, its application as organic fertilizer for farmlands is problematic. Even after processing livestock excrement into compost, widespread distribution is difficult because of low nutrient density and its bulkiness. On a local scale, even if suitable processed compost is available, crop farmers lack the necessary labour and machinery for appropriate application. This challenge leads to the common practice of excessive application, which causes soil eutrophication and environmental problems.

In recent years, the rising prices of chemical fertilizers, P in particular, have led farmers to consider alternatives. Animal manure compost contains a certain amount of P as well as nitrogen (N) and potassium (K). Unlike that in fertilizer, total P in compost is mostly citrate soluble and water insoluble. Thus, compost pellets have gained popularity through their convenience in transportation, handling, and field application (Yakushido, 2000). By molding compost into pellet form, weight and volume is reduced by approximately half. In addition, manures from different sources can be mixed to balance NPK nutrients.

In its dry form, the chemical composition of pelletized compost (PE) is approximately equal to that of the corresponding raw material (RM) (Kano et al., 1997; Hara, 2005). However, the effect of pellets on plants differs from that of ordinary compost because the pellet shape persists in upland soils for comparatively long periods (Hara, 2001; Kubotera et al., 2009). Using swine manure compost, Hara (2005) reported that N immobilization and nitrification were restricted, while N mineralization was invariant in pellet form. A sand column experiment revealed a delayed release of nutrients and an increase in released K in pelletized composts compared with that in traditional ones (Haraguchi et al., 2008). However, limited documentation is available on the effects of pelletization on P phytoavailability in compost. Our objective was to evaluate the effect of pelletization on phosphorus (P) phytoavailability of compost that composed of beef cattle manure compost and broiler litter. The study was conducted under greenhouse conditions with two non-mycorrhizal plants to compare P availability between ordinary compost, pelletized compost, and commercial inorganic P fertilizer.

2 METHODOLOGY

2.1 Compost and soil

In this study, PE and the corresponding RM obtained from a compost manufacturer operated by a local agricultural cooperative (JA-Kikuchi Yuki Shien Center Koshi) were examined to determine P phytoavailability. RM was composed of beef cattle manure compost and broiler litter (1:1; v/v). Because composts can contain hard materials such as stones, the mixture was broken into fragments smaller than 2 mm in diameter to prevent damage to the pelleting machine. RM was formed into cylindrical pellets 8 mm in length and 5 mm in diameter using a disk-die type pelleting machine and was then dried in a greenhouse to reduce the water content to less than 15%. In dry form, the chemical compositions of the two composts were minimally affected during pelletization (Table 1). The soil used in this study was an Ap horizon of Hydric Pachic Melanudands collected from the experimental field of National Agriculture and Food Research Organization Kyushu Okinawa Agricultural Research Center (NARO-KARC) (32° 52′ N, 130° 44′ E). The soil showed a high P sorption capacity and low available P, as indicated in Table 2. The soil was ground to pass through a 5-mm sieve for the following experiment.

2.2 Greenhouse experiment

Comparisons of P availability between the two forms of compost and an inorganic P source (single super phosphate; SSP) were conducted under greenhouse conditions using komatsuna (*Brassica Campestris* L. var. Rakuten) and buckwheat (*Fagopyrum esculentum* Moench var Kitawasesoba) as indicator crops. These crops were selected because they are known as non-mycorrhizal species; thus, the consideration of arbuscular mycorrhiza mediated P uptake was unnecessary. Each P source was applied at various rates to a 1/5000 a Wagner pot filled with the aforementioned soil, which adjusted to field moisture capacity, as shown in Table 3. Application not including P (-P control) was also examined. In addition, a sufficient amount of N was applied to all pots at the rate of 450 mg kg^{-1} as ammonium sulphate in PE and RM pots. N was added in addition to that originating from the compost that was determined to have a 40% availability of total N content. A sufficient amount of K was also added to all pots at the rate of 374 mg pot^{-1}. As in PE and RM pots, K was added in addition to that originating from the compost that was determined to have a 100% availability of total K content.

The pots were arranged in a completely randomized design with three replicates. The experiment was conducted in a greenhouse at 24 °C (day)/ 14 °C (night). Komatsuna seed (12 per pot) and Buckwheat seed (20 per pot) were planted. After 14 days, both plants were thinned to three plants per pot. Komatsuna was grown for another 20 days; buckwheat was grown for an additional 56 days. In the flowering period, buckwheat plants were moved outdoors to encourage entomophilic pollination and fruition. Field capacity was maintained by periodic weighing and addition of tap water as required. At 34 days and 70 days after sowing komatsuna and buckwheat seeds, respectively, the above-ground portion of the plant was harvested. Komatsuna was dried in a forced air oven at 70 °C for 48 h, while buckwheat was dried at 35 °C. Dry matter weights and seed yields were examined, and the triplicate samples were pooled for the following chemical analysis.

2.3 Chemical and data analysis

Komatsuna and buckwheat shoots were ground using a Wiley mill to pass through a 1-mm screen. Buckwheat grain was fined down with an IMF-800DG food mill (Iwatani; Tokyo, Japan). Uniform portions of ground samples were digested in H_2SO_4–H_2O_2 (Mizuno & Minami, 1980). P in the digest was determined by a molybdate blue color method (Murphy and Riley, 1962) on an ultraviolet–visible spectrophotometer. P uptake (mg P pot^{-1}) was calculated on a dry weight basis by multiplying P in the respective tissues with its dry matter weight and on a whole-plant basis by adding the P contents of grain and shoots.

Analysis of covariance (ANCOVA) was performed on yield and the P concentration of plant tissues using the Fit Model platform in JMP 8 software (SAS Institute, Cary NC), with material as a categorical independent variable and rate as a continuous independent variable. The relationship between P uptake and rate of P application was fitted to a square root model (Eq. 1) using the nonlinear regression platform in JMP8, where Y is the P uptake; X is the rate of P applied; B and C are the square root and linear coefficient, respectively; and A is the common intercept, substituting the P uptake value of the control treatment. The square root model is practical for modeling fertilizer response studies in many cases (Nelson et al., 1985) and has been used for fertilizer recommendation purposes (Colwel, 1979). For the obtained models, an F test (Motlusky and Ransnas, 1987) was conducted to test the null hypothesis that there is no overall difference between PE and RM curves. The P recovery rate (z%) was calculated by Eq. 2 using the coefficient obtained from Eq.1:

$$Y = A + BX^{1/2} + CX \qquad \text{(Eq. 1)}$$

$$z = 100 \times \frac{Y - A}{X} = 100 \times \left(\frac{B}{X^{1/2}} + C \right). \qquad \text{(Eq. 2)}$$

3 RESULTS

3.1 Buckwheat

Dry weight changed significantly with an increase in P application rate while remaining relatively constant among fertilizer materials. Grain weight increased with an increase in the P application rate. In contrast, shoot weight decreased with an increase in P application rate (Figure 1, Table 4). P concentrations in buckwheat grain changed significantly with an increase in the P application rate with insignificant change among fertilizer materials. In contrast, P concentrations in buckwheat shoots did not change appreciably among treatment combinations in this study. Total P uptake increased curvilinearly with application rate (Figure 2). The relationship between P uptake and rate of P application fit well to the square root model, and the F test revealed a significant difference between PE and RM curves. However, the P uptake rate among fertilizer materials was drawn near in higher P application rate. Average P recovery rate, calculated in the range of X = 40 to 170 was 24.5%, 15.9%, and 24.0% for PE, RM, and SSP plants, respectively.

3.2 Komatsuna

Significant differences in the dry weight of komatsuna were observed among treatment combinations in this study. Shoot weight was generally lower for plants fertilized with RM than for those fertilized with SSP or PE (Figure 1, Table

4). P concentrations in plant tissues changed significantly with an increase in the P application rate with insignificant change among fertilizer materials. Similar to that observed in buckwheat, the total P uptake increased curvilinearly with the application rate (Figure 2). The relationship between P uptake and rate of P application fit well to the square root model, and the *F* test revealed a significant difference between PE and RM curves. The average P recovery rate, calculated in the range of X = 40 to 170, was 12.5%, 8.6%, and 12.6% for PE, RM, and SSP plants, respectively.

4 DISCUSSION

Compost pellets have a smaller surface area for contact with soil and greater concentrations released products at the surface of pellets compared with raw materials. Granule size is an important physical characteristic of phosphate fertilizers which influences plant response (Bouldin & Sample, 1959). Previous research indicates that, in general, plant response increases with a reduction in granule size. Bauer et al. (2007) reported that ryegrass showed no P uptake response to 2.0- to 4.0-mm particles of calcium phosphate recovered from liquid swine manure despite the relatively high availability of 0.5- to 1.0-mm particles. This result can be explained by the following theory. As granule size increases, fertilizer particles are more unlikely to make contact with plant roots, particularly in cases of slightly soluble fertilizer and soil with extremely low phosphate diffusion coefficients.

In contrast, Williams and Lipsett (1969) determined that the coarse particle sizes of superphosphates were most effective for pastures on soils with high sorption and were subject to leaching. As stated in the Introduction section, compost pellets persists in upland soils for long periods; thus, a decrease in P precipitation and adsorption on soil colloids is possible. Haraguchi et al. (2008) measured early nutrient release patterns from composts and detected a delayed release of P from compost pellets. As not mentioned in previous research, this result was due to high local salt concentration, which reduced P release from compost. Urano et al. (2005) reported that applied compost P persisted longer in a soil exhibiting high sorption when swine manure compost was applied in pellet form. In addition, animal manure compost, a common P source, simultaneously affects P sorption and solubility reactions by providing a source of carbon and stimulating biological activity, and by releasing organic ligands or acids from decomposition processes (Iyamuremye & Dick, 1996; Haynes & Mokolobate, 2001). Therefore, the improvement of phytoavailability of compost P by pelletization can be explained by physical and chemical shielding effects from P precipitation and adsorption on soil colloids.

However, it should be noted that the present results were obtained in greenhouse experiments, and further research is needed to evaluate the agronomic effectiveness of pelletization of compost in field trials, particularly for long-term effects and using other test crops and on other soil types.

5 CONCLUSIONS

Compost pellets have potentially higher P phytoavailability compared with corresponding RM. Although further research is needed under field conditions and on additional soil types for accurate fertilizer application, the molding of compost into pellets appears to be desirable for not only improving crop yields and economic returns but also minimizing soil eutrophication and environmental problems. It is notable that compost pellets are superior sources for providing nutrients that are insolubilized in soil, such as P in an acidic soil.

6 ACKNOWLEDGEMENTS

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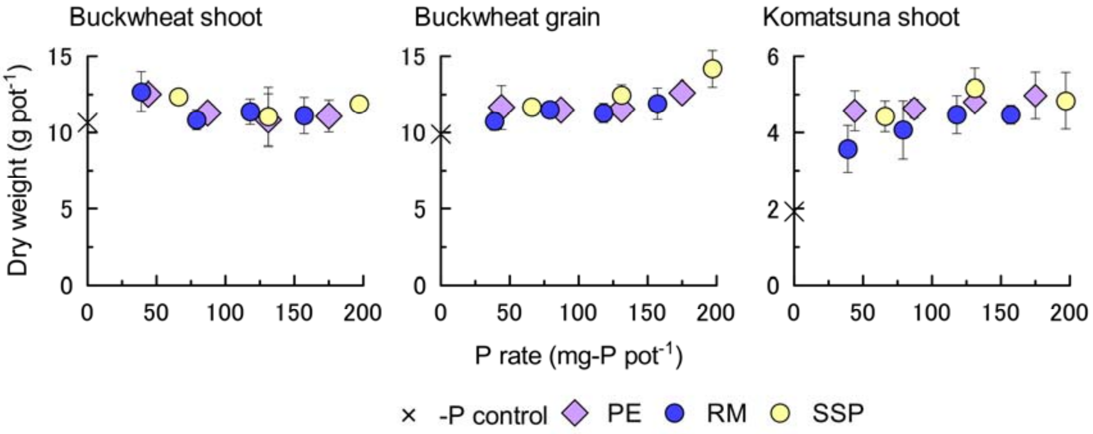
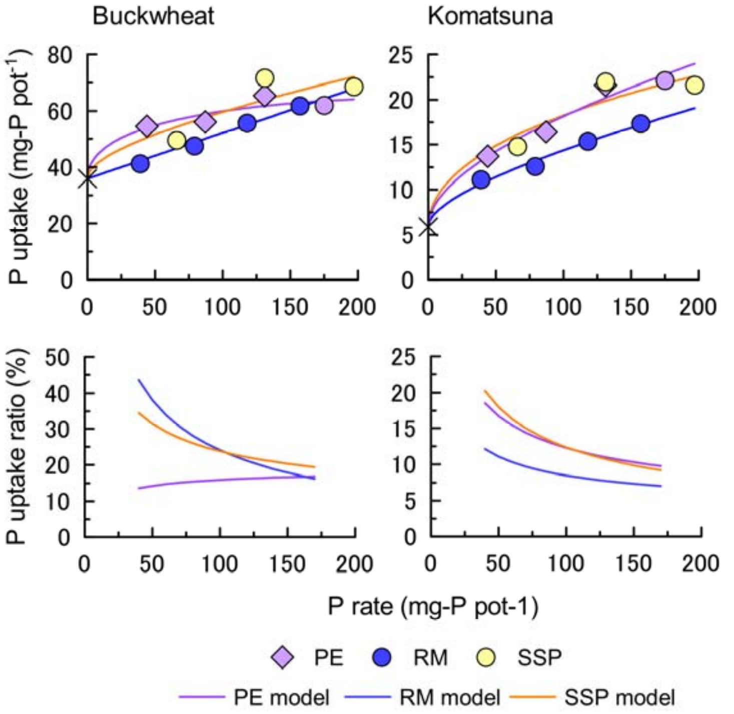


FIGURE 1 Effect of fertilizer materials and P application rates on yield

Data are average of triplicates of the experiment. Standard error bars are shown when the size was larger than that of the symbol.



Attached table. Calculated coefficient from model

	A	*B**	*C**
Buckwheat			
PE	35.9	3.378±0.898	−0.097±0.078
RM	35.9	−0.400±0.178	0.199±0.016
SSP	35.9	1.858±2.830	0.052±0.229
Komatsuna			
PE	5.85	1.079±0.320	0.015±0.028
RM	5.85	0.628±0.156	0.022±0.014
SSP	5.85	1.357±0.773	−0.012±0.063

*Estimate ± S.E.

FIGURE 2 Effects of fertilizer material and rate on P uptake and recovery

Data are fitted to a square root model: $Y = A + BX^{1/2} + CX$

TABLE 1 Chemical characteristics of two compost forms

	C^1	N^1	P^1	K^1	Ca^1	Mg^1	$m.c^{1,2}$	pH^3	EC^4
PE	353	35.3	20.1	27.0	120.4	8.1	63.5	-	-
RM	360	35.6	19.5	26.1	125.3	8.1	210.0	8.38	5.75

^{1}g kg^{-1} dry weight basis ^{2}Moisture content ^{3}1:10 H_2O ^{4}1:10 H_2O, dSm^{-1}

TABLE 2 Selected physicochemical properties of the soil examined in this study

Texture	**pH (H_2O)**	**EC (dSm^{-1})**	**T-C ($g\ kg^{-1}$)**	**T-N ($g\ kg^{-1}$)**	**Av-P ($g\ kg^{-1}$)**	**Ex-K ($g\ kg^{-1}$)**	**PAC[1] ($g\text{-}P_2O_5\ kg^{-1}$)**	**CEC ($cmol_c\ kg^{-1}$)**
HC	353	35.3	20.1	27.0	120.4	8.1	22.9	36.0

[1] Phosphate adsorption coefficient [2] Cation exchange capacity

TABLE 3 Overview of the soil treatment

Material	**applied ($g\ pot^{-1}$)**	**P rate ($g\text{-}P\ pot^{-1}$)**
-P control	0	0
PE	2.33	44
	4.65	87
	6.98	131
	9.30	175
RM	2.54	39
	5.08	79
	7.62	118
	10.17	157
SSP	0.86	66
	1.71	131
	2.57	197

TABLE 4 Effects of fertilizer materials and rate on plant yield and P concentration in tissues

Material	**Buckwheat shoot weight $g\ pot^{-1}$**	**Buckwheat grain weight $g\ pot^{-1}$**	**Komatsuna shoot weight $g\ pot^{-1}$**	**Buckwheat shoot $g\text{-}P\ kg^{-1}$**	**Buckwheat grain $g\text{-}P\ kg^{-1}$**	**Komatsuna shoot $g\text{-}P\ kg^{-1}$**
-P Control	10.7	9.9	1.93	0.60	2.37	3.04
PE group[1]	11.4	11.8	4.75 a[2]	0.92	3.24	3.89
RM group[1]	11.4	11.5	4.24 b	0.79	3.04	3.51
SSP group[1]	11.8	12.4	4.75 ab	0.87	3.12	3.83
ANCOVA						
Material (M)	NS	NS	*(0.0199)	NS	NS	NS
P rate (P)	*(0.0450)	**(0.0016)	**(0.0071)	NS	**	**
M × P	NS	NS	NS	-	-	-

[1] Least squares means, which show the response values for levels of a nominal effect. The response values are adjusted for P rate in the model to examine the effect of each variable.
[2] Means followed by the same letter indicate no significantly difference (Tukey HSD).

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(64) COMPOSTING FOR INCREASING THE FERTILIZER VALUE OF CHICKEN MANURE: EFFECTS OF FEEDSTOCK ON P AVAILABILITY

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EXECUTIVE SUMMARY

Soils in Northern Belgium under agricultural use are low in organic carbon. Application of compost as a source of stabilized organic matter is one strategy for maintaining or increasing the soil organic carbon content. Compost for this purpose can be prepared at the farm level, giving the farmer the opportunity to recycle organic wastes. To meet fertilization standards to reduce nutrient leaching, especially the total P content of the composts limits the application rate. Use of poultry manure as organic fertilizer is restricted due to its low N/P ratio. For some types of manure, adding woody material or straw allows to process the manure to a high quality compost. Including chicken manure in compost may result in a stable product rich in nutrients and organic matter, with a higher C/P value. This research was set up to find out optimal feedstock composition for composting poultry manure at the farm in order to create a soil improver with a higher value as organic fertilizer.

We aimed at determining the effect of feedstock composition on P content and availability in compost when small-scale on-farm composting was applied. The research is based on two compost experiments with 3 treatments each.

In the first compost trial, several feedstock materials were compared for composting with 7.5-10 vol% of fresh chicken manure. The tested feedstock materials were wheat straw, grass clippings, poplar bark, compost and grass hay. Based on chemical characteristics, the compost with 42.5 vol% bark in the feedstock had the highest quality as soil improver.

In the second trial, the effect of the amount of chicken manure in the feedstock mixture was assessed, i.e. 10, 17 or 20 vol%. The tested feedstock materials were wheat straw, grass clippings, poplar bark, willow wood chips, and grass hay. Due to dry weather conditions, intensive follow-up of the process (e.g. regular moistening) was necessary. The second composting experiment was characterized by high temperatures over a long period, indicating that the applied feedstock mixture served as a long-term C source for the process. Results from the second composting experiment indicate that 10 vol% of fresh chicken manure is the upper limit for reducing nutrient losses and achieving a fertilizer with a sufficiently high N/P ratio.

In general, compost characteristics varied due to differences in feedstock characteristics and progress of the composting process. Besides general characteristics, cell wall components and total and plant-available P and N concentrations were measured in the composts. Availability of P was assessed in ammonium acetate extracts at pH 4.65, and in extracts of 0.25 M NaOH and 0.05 M Na_2-EDTA. Based on the analysis of cell wall components of the feedstock mixture and the composts, the biodegradation potential was calculated for each of the composts.

One compost from each compost trial 1 and 2 was selected for application in the field. A field trial with leek was set up to assess the P release from composts based on poultry manure, pure poultry manure and other organic fertilizers, of which some were based on manures as well.

Preliminary conclusions are that poultry manure can be recycled through composting in an organic fertilizer with optimal nutrient ratio and organic matter content. P in the poultry manure can thus be recycled when appropriate feedstock materials are selected and the amount of fresh manure is restricted.

1 INTRODUCTION

Use of poultry manure as organic fertilizer is restricted due to its low N/P ratio. For some types of manure, adding woody material or straw allows to process the manure to a high quality compost. This research was set up to find out optimal feedstock composition for composting poultry manure at the farm in order to create a soil improver with a higher value as organic fertilizer.

2 METHODOLOGY

2.1 Compost trial

The research is based on two compost experiments with 3 treatments each. Two separate compost trials were executed in open air on a site with concrete floor at the experimental farm of ILVO in Merelbeke. The feedstock materials used in the composting process were sampled at the start in four replicates. Temperature and CO_2 levels in the composts were monitored to detect the need for compost turning. The composts were regularly mixed with a Tractor-pulled Sandberger ST300 windrow compost turner and covered or additionally rewetted when necessary. At time of sampling, four samples per row or pile (each based on 10 subsamples) were taken and sieved over a 20 mm mesh and stored in the refrigerator at 4°C. The separate feedstock materials and composts were weighed on a weigh bridge when compost piles were set up and at each sampling time. When necessary, water was added to the compost with a drip irrigation system of the “Uniram” type, at an hourly rate of 8 litre water per meter windrow.

In the first compost trial, several feedstock materials were compared for composting with 7.5-10 vol% of fresh chicken manure. The mixture of each of the three treatments consists of chicken manure, wheat straw and fresh grass clippings. These feedstocks were completed with a mixture of grass hay and compost, tree bark and grass hay, respectively (Table 1, compost A, B and C). Feedstock mixtures had a C/N content of 30/1, which is generally considered an ideal starting ratio for composting. On September 22nd 2010, the three compost piles were set up and composted in a windrow system with a length of 20m per compost type. The windrows were covered with semipermeable fabrics to avoid too high water contents. After 10 weeks of composting, the composted materials for compost A and C were moved and stored in 2 piles. These piles and the windrow for compost B were sampled on January 5th 2011, 15 weeks after the composting process started.

In the second trial, the effect of the amount of chicken manure in a feedstock mixture was assessed, i.e. 10, 17 or 20 vol% of chicken manure. The tested feedstock materials were wheat straw, grass clippings, bark, wood chips, and grass hay (Table 1). The wood chips were harvested in a plot with *Salix viminalis* in the short forestry plantation of ILVO. The composting was started on February 18th 2011. Irrespective of the continuously high temperatures and activity, the three composts were weighed, sampled and stockpiled on 15th of May 2011. Besides the compost trial, a 50:50 dry matter ratio mixture of green waste compost and fresh chicken manure was stockpiled for the same period (February 18th until May 9th) and sampled afterwards.

2.2 Compost analyses

Sample preparation of composts for chemical tests, determination of dry matter content, moisture content and laboratory compacted bulk density was executed according to EN 13040. Electrical conductivity (EC) (EN 13038) and pH-H_2O (EN 13037) in the substrate samples were measured in a 1:5 soil to water suspension. Determination of organic matter (OM) content and ash in the composts or substrates was done according EN 13039. Extraction of water soluble nutrients and elements (NO_3-N, NH_4-N, SO_4, Cl, Na) was executed according to EN 13652, and NO_3-N, SO_4 and Cl were measured with a Dionex DX-600 IC ion chromatography (Dionex, Sunnyvale, CA). Ca, K, Mg, P, Mn, Zn, Cu and Fe were extracted in ammonium acetate and measured by CCD simultaneous ICP-OES (VISTA-PRO, Varian, Palo Alto, CA). Total N concentration in the compost samples was determined with the Dumas Method (elemental analysis) according to EN 13654-2. As an indicator of compost stability, oxygen uptake rate (OUR) is calculated from the oxygen consumption due to microbial activity of 20g compost (<1cm fraction) in 200ml buffered nutrient solution during 5 days of shaking in a closed Oxitop respirometer. OUR is expressed as mmol/kg OM/hour. Total element concentrations for Cd, Zn, Mn, K, Mg, Ca and P were measured by CCD simultaneous ICP-OES (VISTA-PRO, Varian, Palo Alto, CA) after having been ashed and digested with HNO_3 (p.a. 65%). Availability of P was assessed in ammonium acetate extracts at pH 4.65 (as described above), and in extracts of 0.25 M NaOH and 0.05 M Na_2-EDTA. Neutral detergent fibre (NDF), acid detergent fibre (ADF) and acid detergent lignin (ADL) content in the dried compost samples was determined with an Ankom220 Fiber Analyzer extraction unit according to Van Soest et al. (1991). Based on NDF, ADF and ADL content, the biodegradation potential was calculated as (hemicellulose + cellulose)/lignin.

2.3 Field trial

The short term P release of different compost products was tested in a full factorial field trial with 4 replicates per treatment in a horticultural field with leek at Inagro (Rumbeke-Beitem). Different organic amendments including fresh chicken manure, compost B, compost E and the stockpiled mixture of compost and chicken manure were applied on June 8th in the field to reach an equal P dose (110 kg P_2O_5/ha), and were compared with a blank treatment. The upper 23cm of the soil was sampled on July 27th for all replicates separately, and samples were incubated for 3 weeks at 15°C and 70% relative humidity. Readily available P was measured with ICP-OES in the beginning and after 3 weeks of incubation in a 1:5 (w/w) 0.01M $CaCl_2$ extract. The effect of treatment and sampling time was tested with 2-way ANOVA (STATISTICA 10).

3 RESULTS AND DISCUSSION

3.1 Compost trial 1

In the first compost trial, the need for turning the compost was highest shortly after starting the composting, with temperature peaks over 65 °C. Especially mixture B was highly reactive, and needed to be turned 11 times during the trial. Mixture A and C were turned 6 and 10 times, respectively. CO_2 levels were higher for compost A and C than for compost B, especially during the first week. Maximum values were 12 to 14%. Only compost B needed additional water, which was supplied during 5 full days. Compost A and C were supplied with water during 1 and 3 days, respectively.

Total and 0.25M NaOH-extractable P concentrations were higher in the composts than in the initial feedstock mixtures (Fig. 1) due to the mass reduction during composting, but the 0.25M NaOH-extractable P concentrations were relatively lower for the composts than for the feedstock mixtures (Figure 1), pointing at P stabilisation during composting.

Concentrations of Cd, Zn and Mn in the compost are affected both by concentrations in the feedstock mixture and the mass reduction during composting. Cd concentrations were relatively high in the poplar bark, leading to higher concentrations in compost B than for compost A and C. The concentration in the composts was lower than the legal criterion of 2 mg Cd/ kg DM compost and 400 mg Zn/ kg DM compost, respectively.

Feedstock characteristics for compost trial 1 (Table 2) illustrate the differences between grass hay and bark: the tree bark used in compost B had a higher C/N ratio than the grass hay used in compost A and C. The feedstock mixture of compost B had a lower degradation potential, which is reflected in a lower mass reduction than for the other 2 composts (Table 5). After composting, a lower biodegradation potential was measured for compost B than for compost A and C (Table 5).

Based on chemical characteristics of the composts, the compost with 42.5 vol% bark in the feedstock (compost B) had the highest quality as soil improver (Table 3), i.e. the highest OM content (on DM base), lowest EC, lowest OUR and highest C/N and C/P ratio.

3.2 Compost trial 2

Due to dry weather conditions, intensive follow-up of the process (e.g. regular moistening) was necessary in the second compost trial. This experiment was characterized by high temperatures over a long period, indicating that the applied feedstock mixture served as a long-term C source for the process.

A positive correlation was found between the total and NH_4-acetate extractable P concentrations of the 6 composts of trial 1 and 2 (R = 0.88). NH_4-acetate, $CaCl_2$/DTPA and 0.25M NaOH-extractable P concentrations were lower in the composts D, E and F than in the stockpiled mixture of chicken manure and compost (Figure 1), pointing at a stabilisation of P in the composting process. For the stockpiled chicken manure, both the high OUR and the low NH_4-N/NO_3-N ratio point at an incomplete composting and partly anaerobic circumstances (results not shown). For trial 2, all composts had a similar biodegradation potential, but the stockpiled mixture of compost and chicken manure had a higher biodegradation potential than the composts (Table 5). The mass reduction for the three composts of trial 2 was clearly higher than for those of trial 1, while a low mass reduction was observed for the stockpiled manure (Table 5).

Cd concentrations were high in the poplar bark and the wood chips (Table 4), leading to high Cd concentrations in the composts (Table 3). The wood chips were harvested in a plot with *Salix viminalis* in the short forestry plantation of ILVO, and higher Cd concentrations for this species were observed previously in bark and wood (Vandecasteele et al., 2008). Zn and Mn concentrations were high in the chicken manure (Table 4). The Cd concentrations in the composts exceed the legal criterion of 2 mg Cd/ kg DM compost in compost D, E and F, while the legal criterion of 400 mg Zn/ kg DM compost is exceeded for compost E and F (Table 3).

The highest stability is found for compost E: it has the lowest OUR value and the highest NO_3 concentration. Compost D has a better N/P and C/P ratio than compost E and F. These results indicate that 10 vol% of fresh chicken manure is the upper limit for reducing nutrient losses and achieving a fertilizer with a sufficiently high N/P ratio (Table 3).

3.3 Field trial

P release from the applied composts and manures in the field was tested in an incubation trial. No significant effects between treatments ($p > 0.05$) and between sampling time ($p > 0.05$) in readily available P were observed. These results indicate that the short-term P release in the soil was slow, both for the tested products and for the blank treatment.

4 CONCLUSIONS

When comparing P availability in the composts and the feedstock mixtures or stockpiled chicken manure, results point at a stabilisation of P in the composting process. All composts had a similar biodegradation potential, but the stockpiled mixture of compost and chicken manure and the feedstock mixtures had a clearly higher biodegradation potential than the composts. Mass reduction during composting was related to biodegradation potential of the feedstock mixture. Based on chemical characteristics, the compost with 42.5 vol% bark in the feedstock had the highest quality as soil improver. Results indicate that 10 vol% of fresh chicken manure is the upper limit for reducing nutrient losses and achieving a fertilizer with a sufficiently high N/P ratio. The short-term P release in the soil was slow, both for the tested products and for the blank treatment.

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Figure

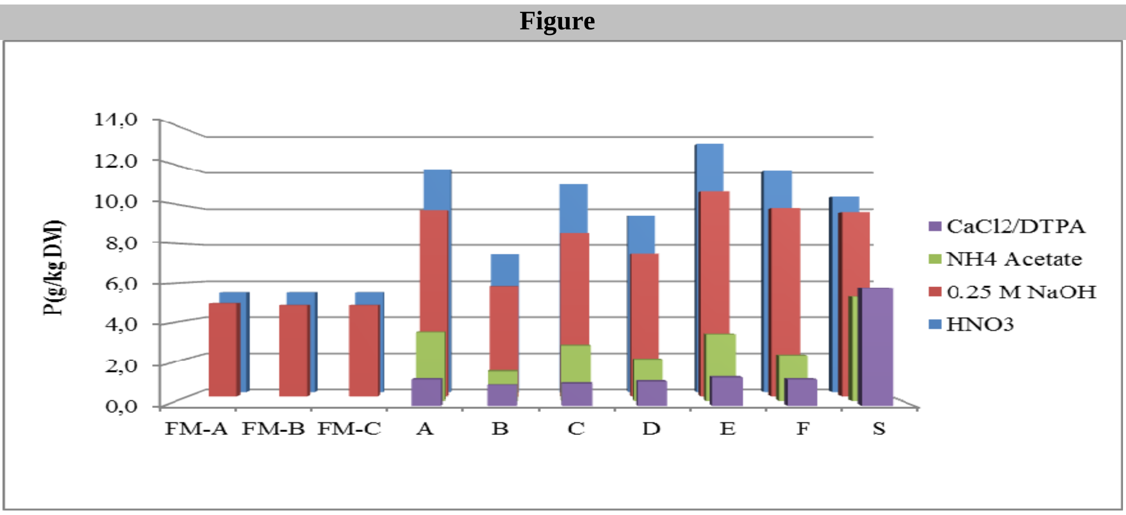

FIGURE 1 **P concentrations in 4 extracts for feedstock mixtures (FM) and composts of the three composts (A-B-C) in trial 1, the three composts (D-E-F) in trial 2, and the stockpiled mixture of compost and chicken manure (S).**

TABLE 1 **Feedstock composition (vol%) of the three composts (A-B-C) in trial 1 and the three composts (D-E-F) in trial 2**

Compost	Chicken manure	wheat straw	grass clippings	poplar bark	wood chips	grass hay	compost
A	10%	30%	15%	-	-	25%	20%
B	7,5%	30%	20%	42,5%	-	-	-
C	7,5%	30%	20%	-	-	42,5%	-
D	10%	15%	21%	27%	18%	9%	-
E	17%	12%	17%	27%	18%	9%	-
F	20%	13%	13%	27%	18%	9%	-

TABLE 2 **Chemical properties of the feedstock used in compost trial 1 (OM: organic matter, DM: dry matter, Ntot: total N). Values are averages ± standard deviation of 4 samples per feedstock**

Variable	**wheat straw**	**grass hay C**	**grass hay A**	**poplar bark**	**Chicken manure**	**grass clippings**
OM (%/DM)	95,1 ± 0,3	92,4 ± 0,5	91,2 ± 2,7	90,3 ± 0,5	42,8 ± 1,9	88,2 ± 0,6
DM (%)	36,8 ± 3,4	63,5 ± 5,8	16,8 ± 3,3	46,5 ± 1,9	62,7 ± 0,6	8,9 ± 0,7
bulk density (g/l)	73 ± 0	27 ± 0	55 ± 0	205 ± 2	533 ± 20	80 ± 0
Cd (mg/kg DM)	0,15 ± 0,02	0,11 ± 0,02	0,17 ± 0,05	1,72 ± 0,18	0,31 ± 0,01	0,12 ± 0,01
Zn (mg/kg DM)	26 ± 9	40 ± 2	64 ± 10	222 ± 8	244 ± 209	70 ± 29
Mn (mg/kg DM)	15 ± 1	117 ± 12	125 ± 54	62 ± 9	225 ± 135	77 ± 2
C/N (-)	117 ± 7	51 ± 3	34 ± 11	58 ± 8	8,25 ± 0,75	24 ± 1
N/P (-)	7,61 ± 0,7	5,13 ± 0,84	5,93 ± 0,4	7,07 ± 0,65	1,94 ± 0,56	4,94 ± 0,08
C/P (-)	901 ± 128	264 ± 31	203 ± 67	410 ± 59	15,87 ± 3,28	120 ± 4
Ntot (kg/tonne DM)	4,52 ± 0,26	10,00 ± 0,60	16,02 ± 4,90	8,80 ± 1,07	29,03 ± 3,52	20,28 ± 0,53
P_2O_5 (kg/tonne DM)	1,38 ± 0,21	4,53 ± 0,51	6,18 ± 1,69	2,87 ± 0,40	35,53 ± 5,94	9,4 ± 0,27
K_2O (kg/tonne DM)	3,16 ± 0,08	21,2 ± 2,01	14,30 ± 4,23	7,94 ± 0,31	19,87 ± 7,96	42,69 ± 0,99
Na_2O (kg/tonne DM)	0,10 ± 0,02	1,16 ± 0,07	0,73 ± 0,19	0,26 ± 0,01	2,76 ± 2,46	1,09 ± 0,31
CaO (kg/tonne DM)	4,04 ± 0,48	5,60 ± 0,35	9,06 ± 1,62	37,17 ± 2,02	56,02 ± 54,81	6,68 ± 0,45
MgO (kg/tonne DM)	1,16 ± 0,02	1,95 ± 0,18	2,49 ± 0,46	2,89 ± 0,38	5,39 ± 3,13	2,84 ± 0,09

TABLE 3 **Chemical properties of the produced composts. Values are averages ± standard deviation of 4 samples per compost. Compost trial 1: compost A, B and C; compost trial 2: compost D, E and F**

Variable	**A**	**B**	**C**	**D**	**E**	**F**
pH-H_2O (-)	8,56 ± 0,14	8,59 ± 0,06	8,31 ± 0,05	8,7 ± 0,1	8,7 ± 0,1	8,6 ± 0,1
EC (µS/cm)	3385 ± 323	945 ± 109	2453 ± 217	801 ± 163	1404 ± 73	804 ± 151
NO_3-N (mg/l)	114 ± 15	40,5 ± 9,01	57,4 ± 24,05	17,8 en < 5	133,78 ± 21,99	43,38 ± 5,25
NH_4-N (mg/l)	45,83 ± 53,64	<5	15,45 ± 22,90	< 5	< 5	< 5
OM (%/DM)	36,8 ± 0,2	51,0 ± 2,7	30,8 ± 1,8	65,1 ± 1,1	58,9 ± 1,9	55,9 ± 2,1
OUR (mmol/kg OM/h)	7,20 ± 1,26	4,05 ± 0,81	11,05 ± 0,64	7,38 ± 0,79	4,88 ± 0,86	5,88 ± 2,05
DM (%)	37,5 ± 1,5	34,5 ± 0,6	36,0 ± 1,9	37,0 ± 0,3	42,1 ± 1,1	34,3 ± 0,5
bulk density (g/l)	544 ± 15	568 ± 4	553 ± 11	502 ± 5	487 ± 7	642 ± 9
Cd (mg/kg DM)	0,72 ± 0,06	1,14 ± 0,07	0,35 ± 0,08	2,92 ± 0,17	2,44 ± 0,06	2,42 ± 0,15
Zn (mg/kg DM)	341 ± 7	280 ± 30	303 ± 54	372 ± 16	513 ± 31	456 ± 23
Mn (mg/kg DM)	489 ± 47	231 ± 10	339 ± 77	340 ± 53	426 ± 32	512 ± 89
C/N (-)	10,40 ± 0,68	17,68 ± 2,94	10,88 ± 1,35	18,45 ± 0,35	14,78 ± 0,89	15,98 ± 1,51
N/P (-)	1,64 ± 0,21	2,23 ± 0,64	1,42 ± 0,24	2,04 ± 0,10	1,67 ± 0,18	1,63 ± 0,09
C/P (-)	17,19 ± 1,81	38,35 ± 4,86	15,38 ± 1,22	37,99 ± 1,69	24,88 ± 3,93	26,12 ± 2,24
Ntot (kg/tonne DM)	19,65 ± 1,23	16,38 ± 2,75	15,90 ± 1,92	19,58 ± 0,34	22,23 ± 0,70	19,53 ± 1,17
Nmin (kg/tonne DM)	0,78 ± 0,25	0,22 ± 0,04	0,36 ± 0,02	0,09 ± 0,05	0,67 ± 0,10	0,21 ± 0,02
P_2O_5 (kg/tonne DM)	27,70 ± 2,88	17,29 ± 2,49	25,79 ± 1,83	22,01 ± 1,27	30,84 ± 4,08	27,57 ± 1,85
K_2O (kg/tonne DM)	34,35 ± 2,75	14,24 ± 0,47	31,29 ± 7,47	20,08 ± 0,63	24,64 ± 0,81	17,09 ± 1,01
CaO (kg/tonne DM)	96,06 ± 1,53	71,34 ± 1,94	84,83 ± 13,29	74,04 ± 3,04	103,43 ± 7,03	103,12 ± 2,60
MgO (kg/tonne DM)	12,12 ± 0,48	7,95 ± 0,23	10,04 ± 0,38	8,18 ± 0,27	10,4 ± 0,59	9,66 ± 0,45

TABLE 4 **Chemical properties of the feedstock used in compost trial 2 (OM: organic matter, DM: dry matter, Ntot: total N). Values are averages ± standard deviation of 4 samples per feedstock**

Variable	**wheat straw**	**grass hay**	**wood chips**	**poplar bark**	**Chicken manure**	**grass clippings**
OM (%/DM)	93,9 ± 0,7	85,9 ± 3,3	87,5 ± 0,7	96,7 ± 1,8	59,1 ± 0,6	84,8 ± 3,4
DM (%)	15,7 ± 1,5	25,4 ± 4,0	32,3 ± 1,5	46,9 ± 5,1	49,9 ± 1,2	15,2 ± 2,3
bulk density (g/l)	99 ± 2	147 ± 14	335 ± 18	291 ± 12	551 ± 6	133 ± 11
Cd (mg/kg DM)	0,13 ± 0,01	0,18 ± 0,04	3,91 ± 0,56	3,01 ± 0,27	0,30 ± 0,01	0,27 ± 0,04
Zn (mg/kg DM)	27 ± 6	116 ± 36	190 ± 8	150 ± 10	589 ± 21	73 ± 15
Mn (mg/kg DM)	24 ± 3	174 ± 18	75 ± 32	59 ± 6	469 ± 9	162 ± 56
C/N (-)	82 ± 5	24 ± 4	64 ± 4	85 ± 7	9,4 ± 0,2	17 ± 1
N/P (-)	9,86 ± 1,00	5,49 ± 0,46	8,94 ± 1,09	6,75 ± 0,70	2,48 ± 0,02	7,64 ± 0,43
C/P (-)	818 ± 120	136 ± 28	575 ± 60	575 ± 19	23,5 ± 0,8	132 ± 4
Ntot (kg/tonne DM)	6,38 ± 0,33	19,80 ± 2,22	7,63 ± 0,53	6,35 ± 0,61	34,88 ± 0,75	25,03 ± 3,42
P_2O_5 (kg/tonne DM)	1,49 ± 0,19	8,35 ± 1,50	1,97 ± 0,19	2,16 ± 0,04	32,24 ± 0,97	7,55 ± 1,30
K_2O (kg/tonne DM)	3,38 ± 0,71	32,27 ± 8,98	8,42 ± 0,93	3,39 ± 0,13	29,67 ± 1,07	22,42 ± 8,99
CaO (kg/tonne DM)	4,39 ± 0,21	14,20 ± 3,13	35,58 ± 2,58	10,46 ± 0,34	121,12 ± 6,69	6,43 ± 0,77
MgO (kg/tonne DM)	1,45 ± 0,14	3,91 ± 0,88	2,43 ± 0,13	0,96 ± 0,01	12,12 ± 0,72	3,07 ± 0,55

TABLE 5 **Biodegradation potential (Values are averages ± standard deviation of 4 samples) and mass reduction extracts for feedstock mixtures and composts of the three composts (A-B-C) in trial 1, the three composts (D-E-F) in trial 2 and the stockpiled mixture of compost and chicken manure (S)**

Sample	**Biodegradation potential**	**% Mass reduction**
Feedstock mixture A	5,7 ± 0,2	
Feedstock mixture B	2,8 ± 0,4	
Feedstock mixture C	7,3 ± 0,3	
Compost A	1,5 ± 0,1	24%
Compost B	1,2 ± 0,1	9%
Compost C	1,8 ± 0,2	21%
Compost D	1,3 ± 0,1	40%
Compost E	1,3 ± 0,1	36%
Compost F	1,3 ± 0,1	47%
S	2,4 ± 0,2	5%

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(193) TESTING N AND P AVAILABILITY FROM ORGANIC FERTILIZER PRODUCTS FOR APPROVAL PURPOSES

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EXECUTIVE SUMMARY

Organic fertilizer products are a potential source of nutrients that can enhance growth of agricultural crops. Interest in these products has increased due to the recent variation in costs of inorganic fertilizers and values of crop products. The Finnish fertilizer product legislation requires that for marketing an organic fertilizer product the applicant must explain *e.g.* the used raw materials and their origin, the chemical and biological composition and physical characteristics. Furthermore, the applicant has to define the recommended application rates and the instructions for safe use of a new organic fertilizer product. Defining the recommended application rates for the new organic fertilizer products requires fast and reliable laboratory methods. The objective of this study was to evaluate two laboratory methods for estimating organic nitrogen (N) and phosphorus (P) availability.

Organic fertilizer products were collected from several on-going projects. N mineralization tests were run according to the protocol used in previous N mineralization tests of plant residues (Jensen et al. 2005). In these incubations 1-3 g of tested fertilizer product was incubated with 50 g of sandy soil on 15°C. Soil samples were taken after 0, 4, 11, 22, 42, 81, 168 and 217 days after the start of incubation. Soil samples were extracted with 2 M KCl and analyzed for ammonium and nitrate N (Lachat auto-analyzer) with three replicates. The soil inorganic N concentrations were compared against incubated soil without any added fertilizer product. The measurement of P availability was based on modified Hedley fractionation (Sharpley and Moyer, 2000), where the fertilizer product was extracted with water, 0.5 M $NaHCO_3$, 0.1 M NaOH and 1 M HCl with a ratio of 1:60.

The N mineralization tests show whether organic fertilizer products cause N mineralization or immobilization. At the beginning, N fertilization effect is determined by the content of inorganic N both in incubation and on field. Digested pig slurry and digested sewage sludge had a high initial amount of inorganic N. During the incubation the decomposition of digested pig slurry led to decreased soil inorganic N concentrations. Dried industrial waste originating from dairy company induced very high N mineralization during the incubation. Water and $NaHCO_3$ soluble fractions dominated in Hedley fractionation of animal manure samples, with median values at 32 and 22 %, respectively. Organic fertilizer products originating from sewage sludge usually had very low P solubility and thus most of P could be found in NaOH or HCl fractions. In the municipal waste samples, the median values of Hedley fractions were generally found at: 2 % water soluble, 5 % $NaHCO_3$ soluble, 67 % NaOH soluble and 22 % HCl soluble.

Long-term soil incubation can be regarded as the standard measure of soil N mineralization potential. However, the incubations are expensive, time-consuming and labor-intensive, while simple chemical indices explain N mineralization rather poorly (Ros et al. 2011). One protocol to be followed could be ISO standard 14328 (2011), where fertilizer product is mixed into the control soil, incubated at 20 °C, with inorganic N concentrations analyzed after at least 2 and 4 weeks. It is economical and relatively easy to analyze a large number of waste samples using the Hedley fractionation method compared with more expensive and time consuming techniques (Toor et al. 2006).

For the approval purposes of product types, N mineralization test and Hedley fractionation appear to be promising estimates of organic N and total P availability. In addition, the users and manufactures of organic fertilizers and soil improvers can benefit from the suggested methods.

1 INTRODUCTION

Organic fertilizer products are a potential source of nutrients that can enhance growth of agricultural crops. Interest in these products has increased due to the recent variation in costs of inorganic fertilizers and values of crop products. The Finnish fertilizer product legislation (Parliament 2006, MMM 2011) requires that for marketing an organic fertilizer product the applicant must explain *e.g*. the used raw materials and their origin, the chemical and biological composition and physical characteristics. Furthermore, the applicant has to define the recommended application rates and the instructions for safe use of a new organic fertilizer product. Defining the recommended application rates for the new organic fertilizer products requires fast and reliable laboratory methods. The objective of this study was to evaluate two laboratory methods for estimating organic nitrogen (N) and phosphorus (P) availability.

2 METHODOLOGY

Organic fertilizer products were collected from several on-going projects. N mineralization tests were run according to the protocol used in previous N mineralization tests of plant residues (Jensen et al. 2005). In these incubations 1-3 g of tested fertilizer product was incubated with 50 g of sandy soil on 15°C. Soil samples were taken after 0, 4, 11, 22, 42, 81, 168 and 217 days after the start of incubation. Soil samples were extracted with 2 M KCl and analyzed for ammonium and nitrate N (Lachat auto-analyzer) with three replicates. The soil inorganic N concentrations were compared against incubated soil without any added fertilizer product. The measurement of P availability was based on modified Hedley fractionation (Sharpley and Moyer, 2000), where the fertilizer product was extracted with water, 0.5 M $NaHCO_3$, 0.1 M NaOH and 1 M HCl with a ratio of 1:60.

3 RESULTS

3.1 Tests of nitrogen mineralization

The N mineralization tests show whether organic fertilizer products cause N mineralization or immobilization (Figure 1). At the beginning, the N fertilization effect is determined by the content of inorganic N contained in the fertilizer products both in incubation and on field. Digested pig slurry and digested sewage sludge had a high initial amount of inorganic N added in the incubation. While the incubation proceeded, the decomposition of digested pig slurry led to decreased soil inorganic N concentrations. The highest N mineralization during the incubation was induced by dried industrial waste. The origin of the dried industrial waste was municipal sewage sludge dominated by dairy company waste water.

3.2 Fractionation of phosphorus

Phosphorus fractions soluble in water and $NaHCO_3$ dominated in Hedley fractionation of animal manure samples, with median values at 32 and 22 %, respectively (Figure 2). Outlier in the water soluble fraction was determined from the separated liquid fraction of digested pig slurry from biogas process, where high solubility of P can be expected.

Organic fertilizer products originating from sewage sludge usually had very low P solubility. There the most of P could be found in NaOH or HCl fractions (Figure 3). In the municipal waste samples, the median values of Hedley fractions were generally found at: 2 % water soluble, 5 % $NaHCO_3$ soluble, 67 % NaOH soluble and 22 % HCl soluble. The outlier in the water soluble fraction was found from composted biowaste sample. The outliers in the NaOH and HCl soluble fractions were found from lime-stabilized sewage sludge samples.

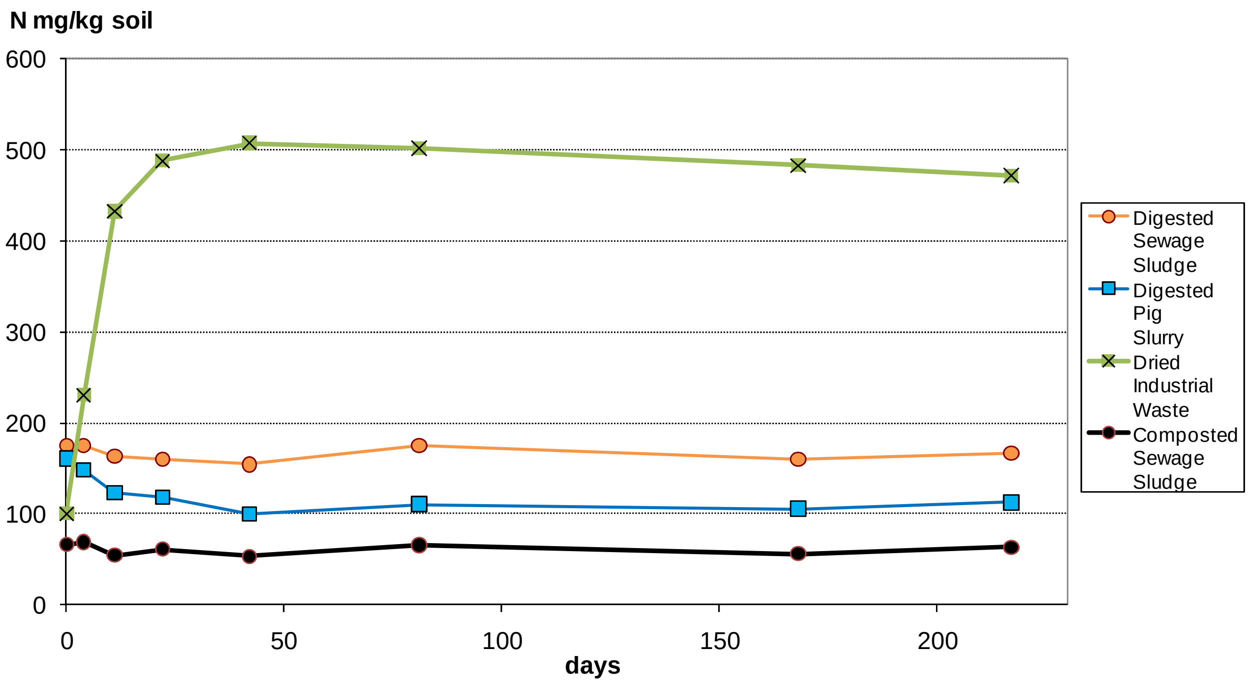

FIGURE 1 **Inorganic N in the incubation experiments of four types of soil amendments**

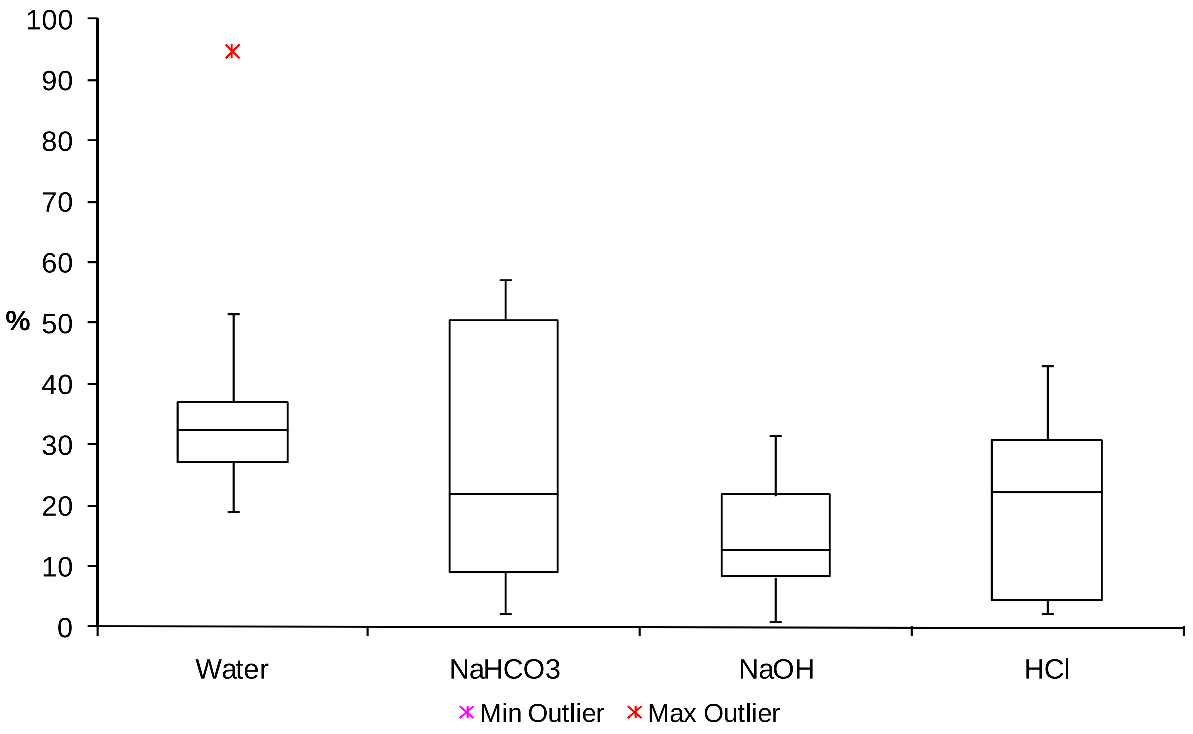

FIGURE 2 **Box-plot distributions of Hedley fractions (% of total P) from biogas treated cattle and pig slurry samples (n=12).**

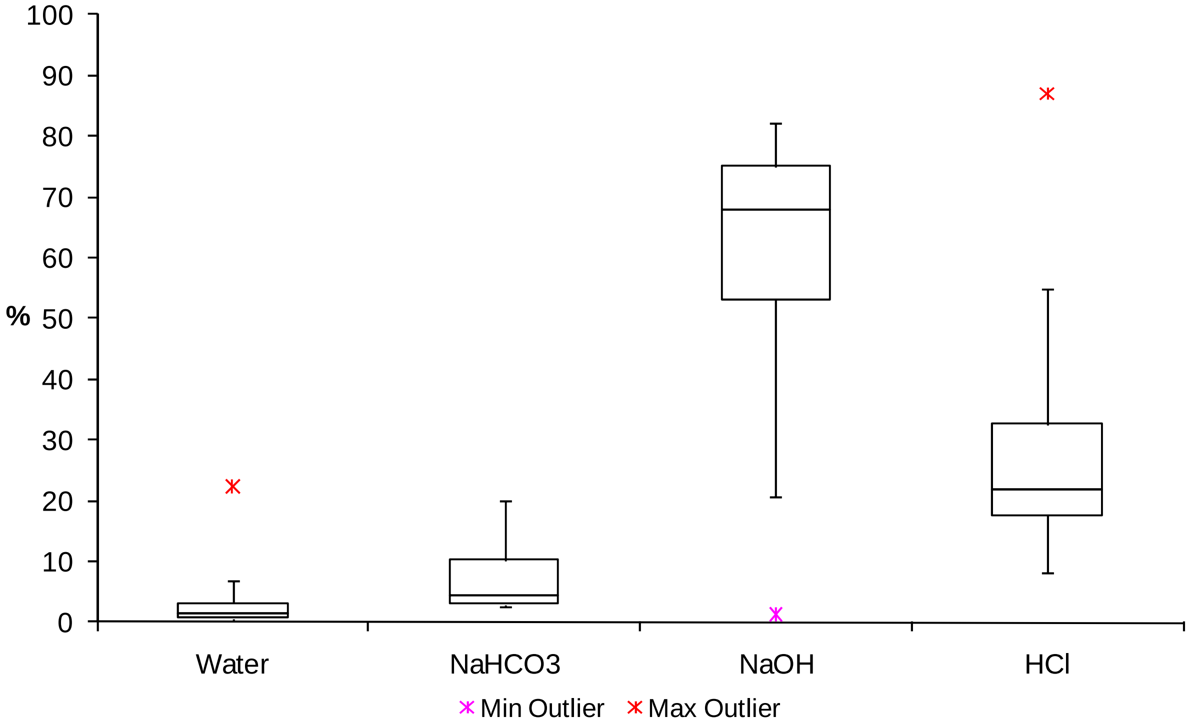

FIGURE 3 **Box-plot distributions of Hedley fractions (% of total P) from municipal waste samples (n=24)**

4 DISCUSSION

Long-term soil incubation can be regarded as the standard measure of soil N mineralization potential (*e.g.* St Luce et al. 2011). Most probably it provides a good approximation of the soil N supply from organic fertilizer products. Soil incubations can be conducted with different procedures varying with temperature, moisture *etc.* One slightly less time-consuming protocol to be followed could be ISO standard 14328 (2011). There the fertilizer product is mixed into the control soil and inorganic N concentrations are analyzed after at least 2 and 4 weeks. The incubation is run at 20° C and the effect of fertilizer products on the soil inorganic N contents is compared against respective concentrations of the control soil.

The main problem with incubations is that they are expensive, time-consuming and labor-intensive. However, simple chemical indices explain N mineralization rather poorly (Ros et al. 2011). Incubations based on ISO standard with duration of 4 weeks may describe the mineralization potential of organic fertilizer products reliably. In these experiments, the levels of inorganic N were stabilized by the sampling of 22nd day. In order to attain practical testing procedures and to define instructions for safe use of organic fertilizer products a fast and reliable incubation method is beneficial.

Several publications have previously produced data from the use of Hedley fractionation with organic fertilizer products (e.g. Huang et al. 2008, He et al. 2010). Toor et al. (2006) stated that it is economical and relatively easy to analyze a large number of waste samples using the Hedley fractionation method compared with more expensive and time consuming techniques such as NMR. The P fractions soluble in water and in $NaHCO_3$ have been described as plant available P. In case the pH of fertilizer product is clearly alkaline, it might be necessary to incubate the fertilizer product with acidic soil and to follow the alterations of P concentrations.

5 CONCLUSIONS

For the approval purposes of product types, N mineralization test and Hedley fractionation seem to give promising estimates of organic N and total P availability. The recommended methods can also be utilized in development of Finnish Agri-Environmental Program. In addition, the users and manufactures of organic fertilizers and soil improvers can benefit from the suggested methods. A fast and reliable testing method will enhance the operations for all actors in the field of organic fertilizers.

6 ACKNOWLEDGEMENTS (IF ANY)

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(213) PLANT AVAILABLE NITROGEN AND PHOSPHORUS FROM COMPOSTED WASTE MATERIALS

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EXECUTIVE SUMMARY

Current EU legislation on waste management (2008/98/EC) and conservation of water systems (91/676/EEC) has seen a growth in the area of compost research. Traditionally the commercial crop growing and agricultural industries have used inorganic fertilisers as the main source to supply nutrients to crops and plants. This study aims to investigate nitrogen (N) and phosphorus (P) release from composted waste materials from numerous different feedstock materials. Much of the literature available on compost research is limited to studies investigating N availability and to a lesser extent P as a nutrient source. This study will also help to gain knowledge of release rates for both N and P. There is a great importance in understanding N and P availability from composted waste. The reasons for this being that it is essential to apply a sufficient amount of compost to ensure the necessary uptake of N and P by the plant whilst minimising the possibility of nutrient run – off which may have adverse effects on the environment.

An incubation study was conducted to investigate available N and P from the various composts over a four week period. The N incubation was conducted at 25°C whilst the P incubation was conducted at 30°C. Rates of application were bases on compost total N and total P as an N and P equivalent to chemical control (calcium ammonium nitrate (CAN) and single superphosphate (SSP)).

Initial results show that available N displayed a more consistent trend than P. The manure wastes were the only category to show an increase in available N over the incubation period. The catering/food waste group was the only other group to exhibit an available N level above 10% during the incubation. The other three groups were all less than 10% initially before dropping to less than 1% by the final extraction. The P results were more varied. Manure wastes had a high initial available P ~40% but had a rapidly decreasing supply, similar to the control. Both biowaste and green waste had an increase in available P at the second extraction before decreasing at the last extraction. The municipal and catering/food wastes had the steadiest available P levels over the four week incubation.

The results have shown to be quite interesting in relation to comparing and contrasting both N and P figures for each composted waste category. Early indications show that N availability is more predictable from each feedstock whereas P availability is quite varied. Current ongoing tests and characterisations of which the results will soon be presented are being used to further quantify N and P availability from different composted wastes.

1. INTRODUCTION

Current EU legislation on waste management (2008/98/EC) and conservation of water systems has seen a growth in the area of compost research. Traditionally the commercial crop growing and agricultural industries have used inorganic fertilisers as the main source to supply nutrients to crops and plants. The aforementioned legislation coupled with the ever increasing cost of N and P based fertilisers has seen the use of compost become more feasible. This study aims to investigate N and P release from composted waste materials from numerous different feedstock materials. Much of the literature available on compost research is limited to studies investigating N availability and to a lesser extent P availability as a nutrient source or on a select sub – category of composted waste. Consequently this study will also help to gain knowledge of release rates for both N and P. These studies of N and P release from compost are investigated with numerous chemical and biological characterisations. Two separate trials will be conducted in which N release rates will be analysed in one and P in the second.

Compost has been found to improve soil structure and organic matter content and also N supply to plants and thus may reduce the input of mineral N fertilizers in conventional agriculture and provide a useful nutrient source in organic farming, respectively (Erhart et al. 2005). Studies have shown that the composting process immobilizes N in litter and produces humus, a source of organic materials and slow release-nutrients (Paul & Clark 1996). Understanding N dynamics is of paramount importance for crop nutrition in terms of application rates as the availability of N to plants is low with the majority of compost N (>90%) bound to the organic N – pool (Amlinger, et al. 2003). Many plants require a high level of N for optimum growth. Understanding N dynamics is very much important from a nutrient management aspect as getting the application rate is of key importance to delivering sufficient N to the plant whilst minimising loss to sensitive parts of the environment.

Modern use of phosphorus for crop production is sourced almost entirely from non –renewable sources i.e. rock phosphate (Van Vuuren et al. 2010). In agricultural systems P is needed for the accumulation and release of energy associated with cellular metabolism, seed and root formation, maturation of crops (especially cereals), crop quality and strength of straw in cereals (Prasad and Foster 2006). Increasing world population growth increases the demand on food production. There is now a greater emphasis on the increasing need for renewable sources of phosphorus. Generally it has been accepted that P release from compost is 100% of the total P, that is it is equivalent to chemical fertilizer e.g. Superphosphate.(BGK 2006) However a literature study carried out recently has indicated that phosphorus availability from composts can vary depending on feedstock (Prasad, 2008). Information on P release is essential if compost is to be applied efficiently, that is, the right amount of P is applied in soil that is adequate for optimising plant growth and P is not lost in ground water or other water bodies and thus has a negative effect on the environment (Prasad 2008). Accurate prediction of P availability and plant P recovery may help tailor compost applications to plant needs and minimise the build – up of bioavailable P which can contribute to eutrophication of sensitive water courses (Magette et al. 2007)

2. MATERIALS AND METHODS

2.1 Composts Investigated

Fourteen composts were sourced from Ireland, Austria, Belgium and Germany. The composts were broadly categorised into five different categories depending on their feedstock material. The categories included; manure waste, bio - waste, green waste, catering/food waste, municipal waste. A set of characterisation studies was conducted on the materials. These characterisations were chemical and biological.

2.2 Incubation Trial

The fourteen composts were used for the incubation trial. The composts were adjusted to approximately 50 - 54% moisture. The composts were sieved to 4mm. The composts were added to 100g of soil at ‘field capacity’. Composts were added at a low and high rate. The rate of compost addition was based on total P and total N results and as equivalent rates to single superphosphate (SSP) and calcium ammonium nitrate (CAN) respectively.

2.2.1 N Incubation

The N incubation was conducted on the fourteen composts with calcium ammonium nitrate (CAN) used as the chemical control and soil as the untreated control. A low index P soil (sieved to 4mm) was used for the trial. The quantities of compost added were based on total N results and as equivalent rates to CAN. For each of the composts, the moisture levels were adjusted to approximately 50%. The composts were added at two rates; 10mg N (low rate) and 20mg N (high rate) per 100g of soil at field capacity. The contents were thoroughly mixed in 200 ml volume plastic containers. Each container was sealed with lids with holes to allow for air intake/release. The containers were incubated for 30 days at 25°C in the dark. Moisture levels were monitored every second day and adjusted as required. Sampling was conducted at 0, 1, 2 and 4 weeks. Sampling was conducted by taking 12.5g from each pot. A stratified sampling method was used to ensure a representative sample was taken for each sample. The samples were extracted in 0.5M KCl. Analysis was conducted using the Aquakem 600 discrete analyser.

2.2.2 P Incubation

The P incubation was conducted on the fourteen composts with single superphosphate (SSP (8%)) used as the chemical control and soil as the untreated control. The quantities of compost added were based on total P results and as equivalent rates to SSP. The composts were added at two rates; 5mg (low rate) and 10mg (high rate). The rest of the preparation was done as per the N incubation. The containers were incubated for 30 days at 30°C in the dark. Sampling was conducted at 0, 2 and 4 weeks. Sampling was conducted by taking 5g from each pot. A stratified sampling method was used to ensure a representative sample was taken for each sample. The samples were extracted using Olsen's Reagent (0.5M $NaHCO_3$). Analysis was then conducted using the Perkin Elmer UV spectrophotometer probe at 880nm using the ammonium molybdate reactive method (Murphy & Riley 1962).

3. RESULTS and DISCUSSION

Table 1 highlights the individual composted wastes used for the trial. Included in the table are the feedstock materials with the composting process type and process duration.

Table 1. Compost samples with feedstock materials and production details.

Sample	Feedstock Material	Composting	Process Length
GW 01	Green Waste	Windrow	13 weeks
GW 02	Green Waste	Windrow	10 weeks
BW 01	Biowaste	In Vessel (15 days) + Windrow	36 weeks
BW 02	Biowaste	Enclosed aerated system	7 weeks
CFW 01	Mixed (Seafood)	Indoor Enclosed Windrow	20 weeks
CRW 02	Food Waste	Aerated Static Pile Composting (Indoors)	6 weeks
CRW 03	Catering Waste	In vessel Tunnels and aerated Floor	10-14 weeks
MuW 01	Food Waste	Aerated Static Pile Composting (Indoors)	8 weeks and then left to mature until needed
MuW 02	Organic Fertiliser (Biowaste + Green waste)	Windrow	16 weeks
MW 01	Horse Manure	Windrow	8 weeks
MW 02	Chicken Manure with Seaweed	Pile composting Indoors	25 weeks
MW 03	Chicken Manure	Pile composting Indoors	18 weeks
MW 04	Broiler Manure (sweepings, feathers)	Pile composting Indoors	21 weeks
MW 05	Manure Compost (mix)	Composted in Tunnel	2 weeks

Table 2 gives details of chemical and biological characterisations completed on each of the composts. Included in this table are the total N, P and K (potassium) values. The final value in the table is the oxygen uptake rate (OUR) (Prasad et al., 2010)

Table 2. **Characteristics of the composts used.**

Sample	pH	EC (mS/m)	Total N (%)	Total P (%)	Total K (%)	C/N Ratio	OUR (mmol O_2/kgOS/h)
GW 01	7.72	1.84	1.99	0.36	1.61	12.63	4.76
GW 02	7.40	4.56	1.34	0.30	0.41	10.16	8.43
BW 01	7.66	2.35	1.45	0.24	1.3	19.27	1.00
BW 02	6.45	0.89	2.04	0.33	0.9	10.10	7.95
CFW 01	7.64	4.96	2.38	1.14	1.1	10.80	n.d
CFW 02	6.98	4.12	1.85	1.71	1.03	12.40	2.66
CFW 03	7.02	4.65	2.50	0.32	1.04	9.98	3.64
MuW 01	8.05	0.33	1.61	0.39	1.26	16.39	5.27
MuW 02	8.28	1.40	2.40	0.63	1.25	14.99	6.33
MW 01	6.25	2.51	3.45	0.43	3.57	10.05	6.84
MW 02	6.31	6.82	3.82	1.38	2.96	11.05	25.97
MW 03	6.25	5.03	3.54	1.98	3.36	12.17	20.47
MW 04	5.30	8.64	4.41	1.34	0.58	9.96	55.72
MW 05	6.02	2.75	1.03	1.81	0.74	10.02	48.12

n.d=not determined

The N incubation was conducted for 4 weeks with extractions analyses results presented as total organic N. Total N results expressed as percentage of N application rate over four week period of incubation (Fig. 1.) Results are expressed on a logarithmic scale to incorporate the large gap present between composted waste results and the chemical control.

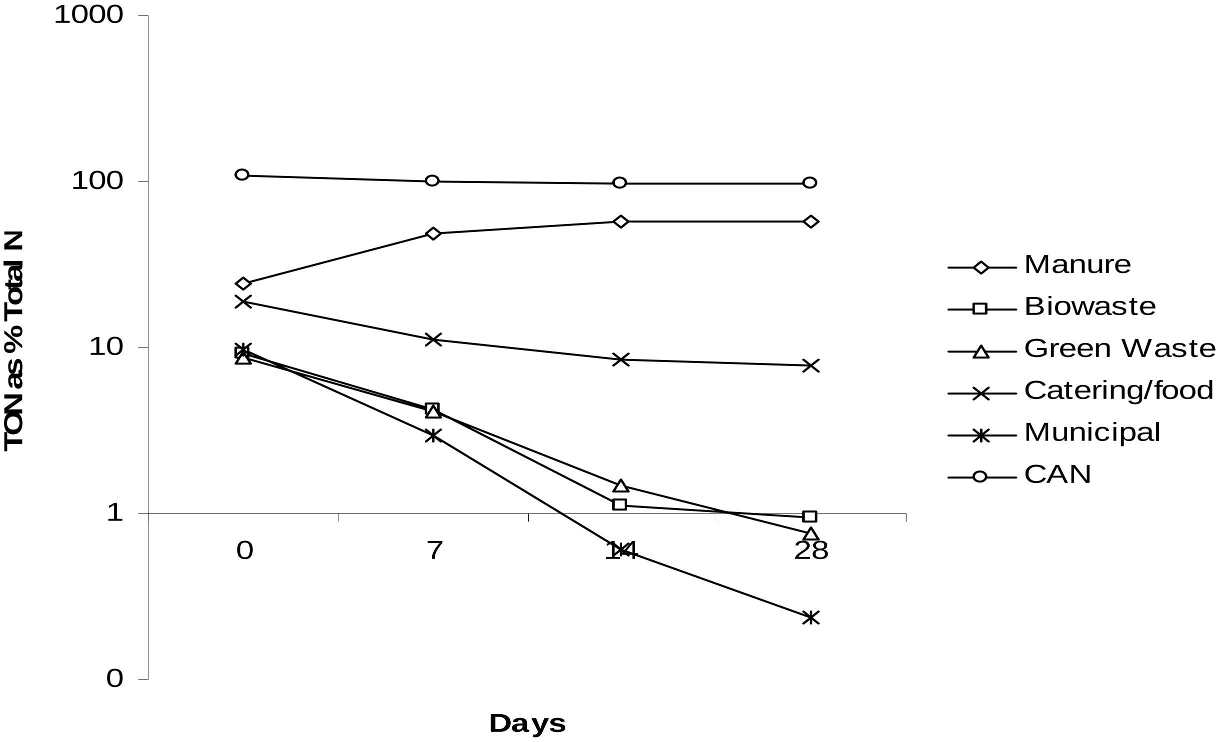

Fig. 1. TON (NO_2 + NO_3-N) expressed as percentage of compost N minus soil value (control) availability incubated for 4 weeks on a logarithmic scale.

The CAN exhibited N availability of 100% approximately. The CAN availability was stable over the four week incubation period. Out of the five composted waste categories manure wastes were the only group to exhibit an increasing supply of N. The initial available N from the manure wastes was 25% approximately. This was value increased to 56% at the final extraction. The catering/food waste group was the next best group in terms of available N. This group had an initial available N of 18%. This decreased to 8% by the final extraction. The final three categories of biowastes, green wastes and municipal waste exhibited an initial available N of less than 10% and this decreased to less than 1% by the final extraction. The 1% figure was an average of the composts. It must be noted that some of the individual results gave a negative value highlighting the immobilisation of N in the compost. These results are generally in agreement with Fuchs et al. 2010 who carried a similar incubation trial for 8 weeks on a large number of different types compost e.g., for agricultural use, for horticultural use etc and found that release of N was <5% and N immobilisation was <5%.

Olsen’s P incubation extraction analyses results were presented as Olsen’s extractable P as a percentage of P application rate minus soil (Fig. 2.) The P results presented more varied trends than the N results.

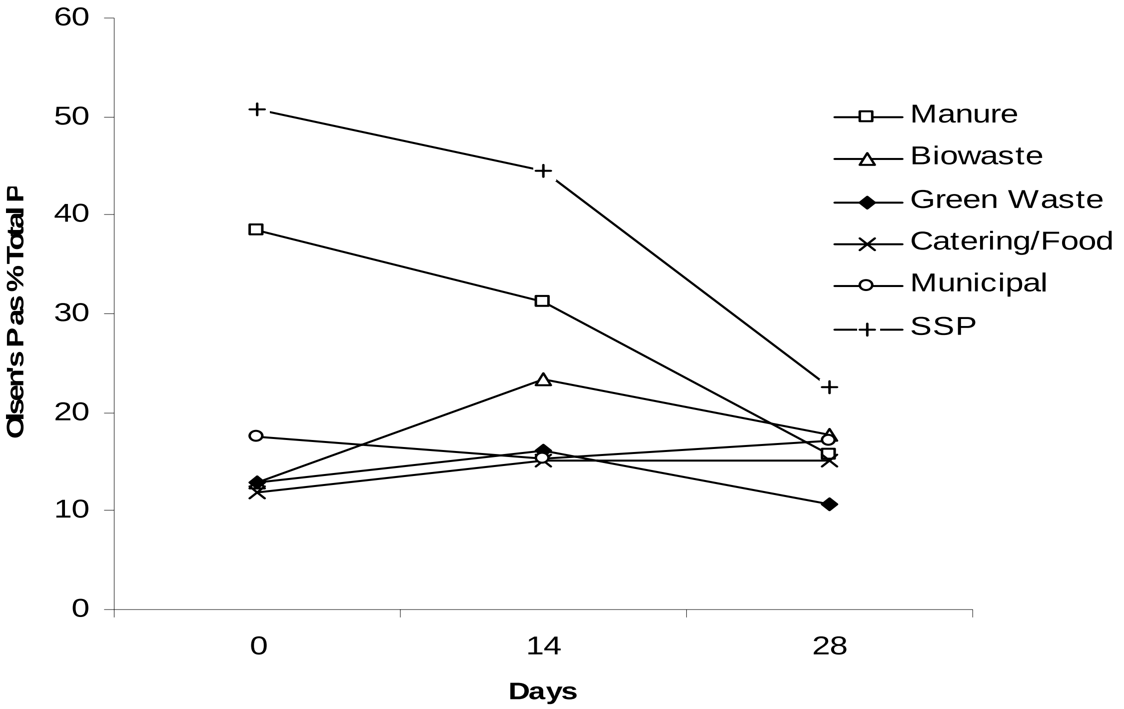

Fig. 2. Olsen's P results expressed as percentage of total compost P availability minus soil (control) incubated for 4 weeks.

The chemical control SSP exhibited the highest availability of P showing an initial available P rate of over 50%. This decreased quite rapidly to 22% at the final extraction. The manure waste group had a significant level of available P at the initial extract with 38% available P. The manure wastes available P decreased quite rapidly similar to the control with approximately 15% available P at the final extraction. The remaining four groups were generally below 20% in available P over the four week incubation. The biowaste showed an increase at the second extraction. The initial value being 13% and this increased to 23% at the second extraction. Green waste displayed a similar trend to biowaste with and increase in available P at the second extraction before decreasing again for the final extraction. There is no obvious explanation for this trend in the results. Both catering/food and municipal wastes exhibited steadier release rates over the incubation period.

From the results presented it is clear that for N availability the feedstock materials have a more distinctive trend and are more predictable. The P results show much more contrasting trends with fluctuations in P availability across different feedstock categories. The category which presented the most diverse set of results was the biowaste category. This is mainly due to the fact that within this category of composted waste materials there are numerous different types of feedstocks. It's these various different types of feedstocks that probably lend to varying trends in N and P availability. The composted manures on the other hand were much more uniform in N and P availability. MW1 gave the lowest available N and P over the incubation period. Green waste, biowaste and municipal waste groups show a low available N initially less than 10%. This indicates the immobilisation of N during the composting process.

It must be stressed that categorisation of composted wastes by feedstock based on N and P availability is generally not possible. This is due to the large variety of compost types and the different composting processes which have been undertaken in the production stage. A greater emphasis must be placed on the understanding N and P availability from composted waste materials. There is a need to develop tests that will be able to predict the uptake of P and N by plants.

There is a potential saving of 100,000 tonnes/ CO_2 per year. This figure is based on the composting of 250 kg of biodegradable material produced by each inhabitant per year. The assumption is that the material is composted and the population of Ireland is 4m (Pers. Comm. F. Amlinger). 400,000 tonnes of compost will replace 60,000 tonnes of N. One kg of mineral N fertiliser uses energy equivalent of 2kg of oil.

The addition of compost to agricultural/horticultural land will improve physical properties of the soil, meaning less energy will be required for soil management. This project has a potential to reduce the amounts of fossil fuels used in the agriculture by a significant amount.

4. CONCLUSION

The results have shown to be quite interesting in relation to comparing and contrasting both N and P figures for each composted waste category. Further testing and characterisation is ongoing which the results of will be presented soon.

5. ACKNOWLEDGEMENTS

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Session 29

(43) AN INNOVATIVE ASSAY TO MEASURE ANAEROBIC BIODEGRADABILITY OF ORGANIC WASTES IN TWO DAYS

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EXECUTIVE SUMMARY

Anaerobic digestion is a biochemical process, where, in the absence of oxygen, bacteria break down organic materials to produce biogas. Many different kinds of biomass can be used as substrates, such as energy crops, putrescible organic wastes, manure, water-treatment sludge, algae or a combination of waste materials. Since the mid 90’s, anaerobic digestion is seen as an alternative to stockpiling of waste and to the world’s finite fossil-fuel resources (Ahring, 2003). Until now anaerobic biodegradability has been conventionally estimated by Biochemical Methane Potential (BMP) or predicted by models with biochemical characterization as data inputs (Buffière et al, 2006). However, these two approaches show important drawbacks. They are time consuming (at least 30 days for measuring BMP), expensive, cumbersome (handling difficulties) and do not meet the requirements for operational waste management. Therefore, fast screening methods on industrial site should be developed to predict the energetic added-value of organic inputs, on their own or under appropriate mixtures. We present an innovative assay to quickly determine the anaerobic biodegradability of organic wastes. Based on this new assay, we have developed the ENVITAL® kit for operators to quickly perform the assay directly on-site for multiple wastes.

Our assay is designed on a 96-well fluorescence-compatible microplate for screening capacity. Each well contains phosphate buffer, a fluorogenic redox indicator, a standard solution or the organic sample and filtered digester sludge as inoculum. Anaerobic conditions are created by sealing the microplate with an appropriate layer of paraffin. The microplate is then incubated and agitated in a microplate reader à 35°C for three days. Automatically each hour, anaerobic biodegradability is monitored by direct measurement of the biosensor fluorescence intensity. Standard solutions are also simultaneously monitored to select the optimal time for activity data treatment. BMP-equivalents are finally derived from correlations previously established with our BMP database developed on a wide panel of wastes.

We have optimized our assay by investigating the impacts of various parameters such as the inoculum and its conservation, the state of the waste, the sample preparation, and the standards. The ENVITAL® kit shows good repeatability of the acetate standard curve with an optimum incubation time of 25H-34H, depending on the inoculum and its conservation before analysis. The linear regression coefficient for the standard curve is 0.98 for all test conditions. We performed our assay on 16 different organic samples, ranging from 0.5 to 98.4 $LCH_4.kg^{-1}$. The standard deviation of the ENVITAL® measurement of the triplicate samples is less than 8%. BMP-equivalent values derived from our assay are in strong agreement with measured BMP using the classic technique, with a linear regression coefficient of 0,88. The prediction error with measured BMP values as reference was from < 5% to 20% (corresponding to BMP <. 10 $LCH_4.kg^{-1}$ Raw Matter).

ENVITAL® enables operators to measure up to 40 BMP-equivalent within less than 3 days for both liquid and solid samples. It can be used to optimize waste collection and management by quickly screening wastes for their energy potential. It can also serve to design digestion operations and to maximize energy performance by improving waste mixtures.

1. INTRODUCTION

Anaerobic digestion is a natural process, by which biomass can be transformed by naturally occurring microorganisms into methane. Bioenergy production has gained a lot of industrial interest in the last decades while energy prices were constantly rising. The number of operating biomethane production plants has been fastly growing in most countries and a lot of new projects are being designed. In the meantime, the technology has been improving with optimized digesters, more efficient biogas cleaning units, and operating costs have also been improved. However, despite the growing interest and industrial relevance of anaerobic digestion, there is still a strong impediment when measuring the biomethane potential (BMP) of a given biomass. The classic method consists in placing a biomass sample in a flask along with an appropriate inoculum and to directly measure the produced biogas over a period of at least 21 days, usually 30 to 40 days (Owen et al, 1979, Hansen et al, 2004). The market price of this analysis lies between 300 and 500 € per samples. It is expensive and the analytical data come at a time, which is no longer relevant to the industrial process. Putting in storage tons of daily arriving biomass for this period of time is almost impossible for a plant operator. Therefore, there is a strong need for analytical innovation on this crucial parameter, used for dimensioning production units and for the daily management of highly diverse biomass.

In a very interesting review, Lesteur et al. (2010) have scanned the various new possibilities to assess BMP. All these methods appear to be indirect assessment of the anaerobic biodegradability. In these methods, measurement of other parameters is used to predict BMP. The first possibility comes from the measurement of aerobic biodegradability during a few days, usually five, to derive the anaerobic activity (Ponsà et al, 2008). This approach is of interest as it uses an inoculum to assess real biodegradability. However, the huge diversity of microbes involved in anaerobic metabolism can not be represented in an aerobic inoculum, leading to large discrepancies between assessed and real BMP values. BMP can also be predicted from the chemical composition analysis of the biomass, either elemental composition (C, H, O, N) or by families of compounds (carbohydrate, protein and fiber content). The composition is measured using classic analytical techniques and results are introduced in a prediction model based on registered correlations (Buswell and Mueller, 1952; Buffière et al 2006). Such indirect measurements require strong pre-established correlations. They are slightly representative of the large chemical variability of biomass but totally independent from the biodiversity of an inoculum. Another approach has gained interest for predicting BMP and is based on spectroscopic techniques, such as UV-Visible spectroscopy or more notably infrared spectroscopy, either mid- or near-infrared (Muret et al, 2010; Lesteur et al, 2011). In this case, the chemical diversity of the sample is described in a much finer fashion as spectroscopic techniques gather a lot of rich information regarding the families of compounds and their interactions. The sample needs to be lyophilized for infrared techniques but the data acquisition is then very fast. Here again, strong correlation models are required to translate complex spectroscopic data in a single BMP value. These models are usually waste specific. More recent techniques have been developed on almost exhaustive characterization of biomass, namely pyrolysis-GCMS and advanced oxidation processes (Jarde et al, 2003; Pognani et al, 2011; Roig et al, 1999). These techniques generate enormous amounts of data representing fragments of the complex biomass chemical structure. These structures are then reconstituted, which enables the correlation with BMP values. All these indirect predictions of BMP are clearly providing operators with potential analytical solutions that drastically reduce the time required to assess BMP. However, they are still expensive as they require elaborate instruments, are not always available on-site and share the characteristics of being chemically-based, not using an inoculum to assess BMP of a given sample.

Our work consisted in developing a much faster direct measurement of BMP enabling the on-site processing of the analysis. This was done using an innovative approach to the direct measurement. Instead of monitoring the biogas produced during the incubation, we used a highly sensitive fluorescent probe to monitor directly *in cellulo* the extent of anaerobic metabolism. This is derived from a system that was developed for aerobic biodegradability (Dudal et al., 2006, Rocher et al., 2011) and is performed on a 96-well microplate format enabling multiple samples analysis at the same time. The microplate is incubated in the reader and the BMP equivalents are simply derived using an acetate calibration.

2. MATERIALS AND METHODS

The different steps required to prepare up to 40 assays on one microplate require less than two hours and are graphically presented in Figure 1. The assay kit is commercially available under the name ENVITAL® (Envolure, Montpellier, France).

2.1. Sample preparation

Most effluents and solid waste samples can be analyzed through this assay, exception made of very dense materials such as bones. Sample preparation consists in an initial grinding of a representative sample using a 600-W food grinder followed by suspending in a specific buffer. The suspension is further grinded and diluted at a given dilution ratio appropriate for the type of sample (provided in the assay protocol).

We have worked with as a series of different samples ranging from various sludges (developed from both agro-industrial and urban wastewaters) to raw biowastes (food and agro-industrial wastes). This study presents a set of 16 samples representing these different families. All samples were sent to an accredited laboratory for classic BMP measurements.

2.2. Inoculum preparation

Various kinds of inoculum can be used in the assay, deriving from a digester, wastewater treatment plant, lab digester, anaerobic microbial cell culture, lyophilized strains or consortium, etc. The microbial concentration should be adjusted with the concentration of the fluorescent probe. This step is performed only the first time an inoculum is being used to quantify BMP on various samples. The inoculum is filtered through a 1.2-µm PES filter before use.

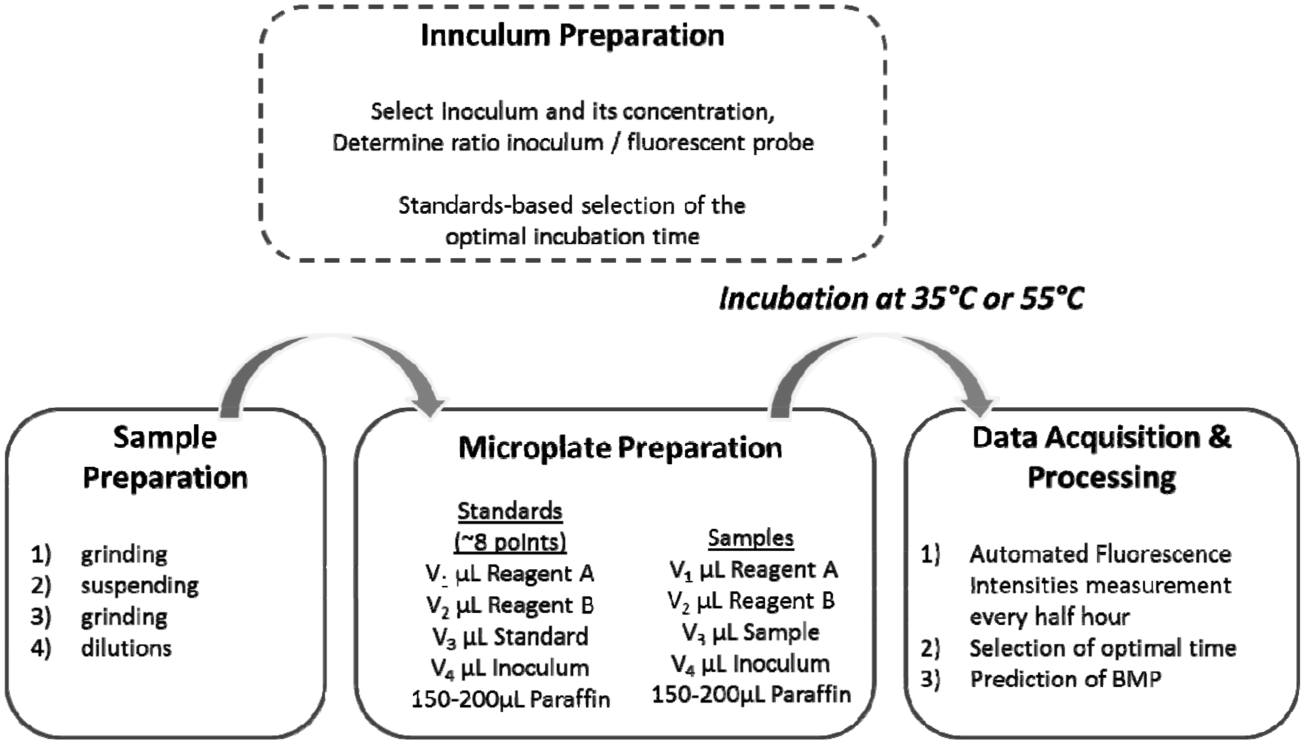

FIGURE 1 **Description of the assay protocol**

2.3. Microplate preparation

The assay consists in performing an acetate calibration curve along with the samples and using this calibration to estimate the adequate incubation time (see next section). In a high quality quantitative assay, two columns of the microplate (a total of 16 wells) are dedicated to the duplicated calibration consisting of a range of acetate concentrations. Two more columns are used for control samples. These are library samples with pre-measured BMP, belonging to the types of samples of interest to the operator. Therefore, up to 40 samples can be fully quantified in a single kit, along with standards and control samples.

In all cases, the fluorescent reagent is mixed with the sample or standard and with the inoculum inside the microplate well. The wells of the microplate are then sealed using pre-warmed paraffin, to avoid the presence of head-space and further oxygen transfer into the well. The microplate used in the assay is a clear bottom microplate, inserted in the reader for a bottom-mode reading.

2.4. Data acquisition and processing

The reader (BIOTEK FLx800 using the Gen5 software) incubates the microplate at 35°C or 55°C, according to the operator's requirements, shakes the microplate regularly and acquires and stores the reading data every half hour automatically during the whole time of the incubation. This time of incubation is set according to the quantification mode used in the assay. The fluorescent indicator is monitored at λex=540 nm to λem=600 nm. Using the calibration mode, the reader software evaluates the quality (R^2) of the calibration curve at every reading (usually every half hour) during 50 hours. It then automatically selects the incubation time for which the calibration has provided the best quality. The sample results corresponding to that selected time are used to quantify BMP based on the control samples fluorescence measured at the selected incubation time.

3. RESULTS

The microplate reader automatically acquires fluorescence readings every 30 minutes throughout the 50 hours of the incubation. At each of these readings, the software generates a calibration curve from the fluorescence intensities emitted from the standards wells. Figure 2 presents the kinetic evolution of the fluorescence intensity for each standard concentration of the calibration curve (from 0 to 2000 mg acetate /L).

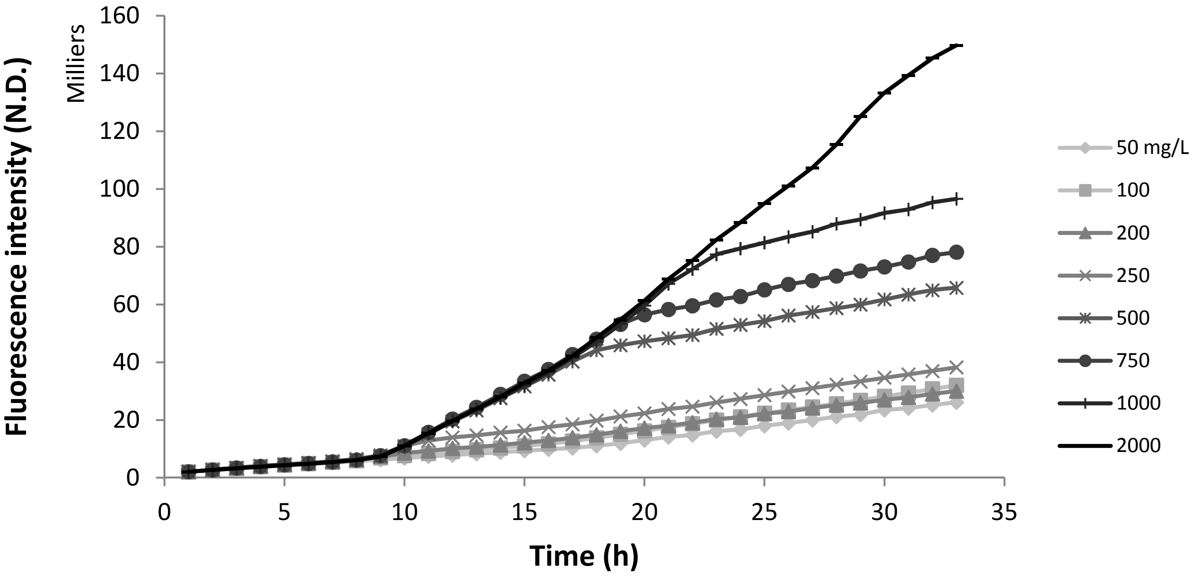

FIGURE 2 **Incubation kinetics for the standards**

The initial ten hours of the incubation do not exhibit much activity. In the paraffin-covered microplate well, anaerobic conditions establish during this period of time. This observed lag phase probably corresponds to the activation of anaerobic microbial communities and their adaptation to the well conditions. After ten hours, the fluorescence intensities generated by the various amounts of substrates start to differentiate. More available substrate generates higher microbial uptake and consequently higher levels of fluorescence. After 25 hours, the calibration curve properly establishes and it reaches an optimum at 33 hours in this case, with a correlation coefficient of 0.99 (Figure 3).

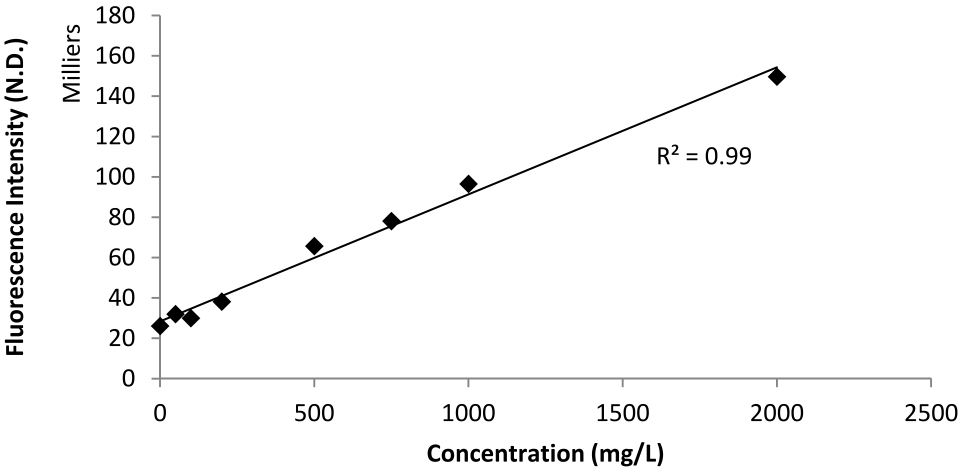

FIGURE 3 **Calibration curve**

Once this adequate incubation time is specified, the fluorescence intensities of the control samples are read and directly related to their pre-registered BMP. The standards give the reading time, at which the controls provide the quantitative link to the BMP. This quantitative link is used to assess the BMP for the samples. The results for the samples described in this paper are presented in Figure 4.

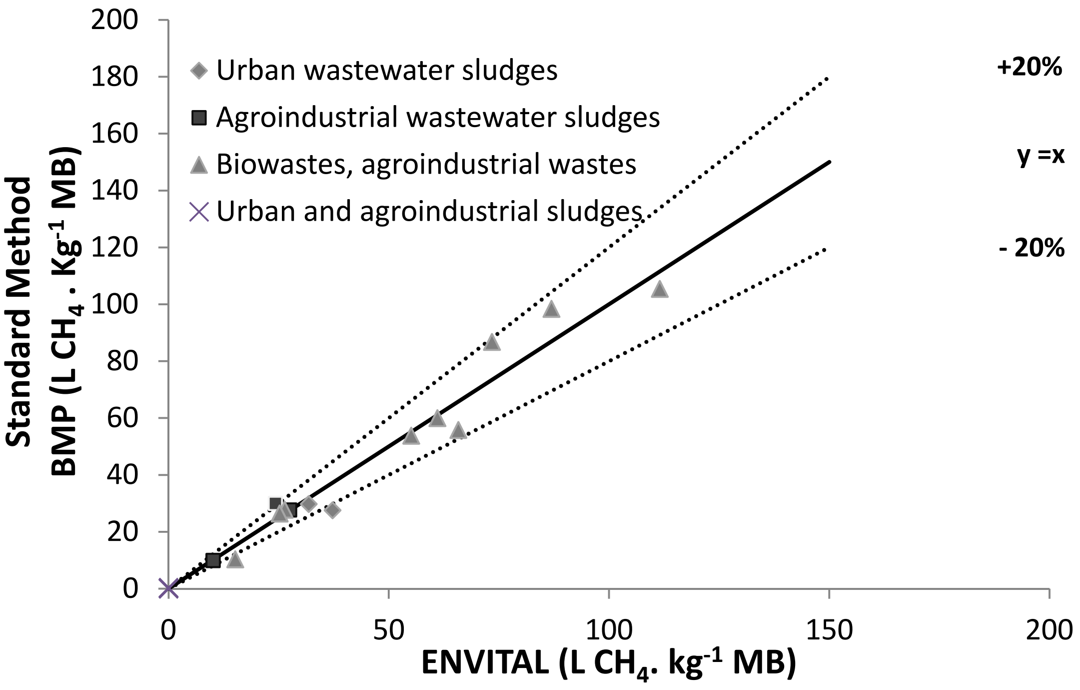

FIGURE 4 **Quantification of BMP from the ENVITAL assay**

Figure 4 shows a strong correlation between the results obtained with the ENVITAL® assay and the results gathered from the accredited laboratory for BMP measurements. Throughout a wide range of bioenergy potential, the differences between both assays do not exceed 20%. Sludges exhibit lower potential than raw biowastes. This can be explained by the fact that a lot of the organic material initially contained in the non-treated wastewaters has already been aerobically degraded during the wastewater treatment process. Therefore, the ability of the remaining substrates to generate microbial metabolism is decreased. In opposition, raw biowastes show promising bioenergy potential. These differences are commonly observed and have been previously reported (Luna del Risco and Dubourguier, 2010).

4. DISCUSSION

The results clearly show that the ENVITAL® assay is successful at predicting BMPs for a wide range of liquid to solid samples. We have built the control samples library with representative samples from the most commonly found families of residual biomass: sludge, agro-industrial effluents, food wastes, etc. This provides the assay with sample-type specific quantification and ensures a much higher quality assessment of BMP.

The new approach described here consisting in a direct *in cellulo* measurement of the metabolism under anaerobic conditions using a specifically designed fluorescent probe provides both high sensitivity and much shorter incubation time. Microbial uptake of the slightest amount of available substrate generates oxidation-reduction processes in the cells, which activate strong fluorescence emission from the probe. The fluorescence intensity is a highly sensitive marker of differences in early substrate utilization, which enables the prediction of later accumulation of end-products such as methane. One important asset of our methodology in comparison to other new methods (Lesteur et al., 2010) lies in the fact that it integrates both chemical and microbial diversities. Anaerobic digestion is a highly complex microbial mechanism as it compiles the sequential activities of different microbial communities using different electron acceptors (Li et al, 2011, Zinder, 1984). Consequently, two very different BMP values can be obtained using two different inoculums. Predicting BMP independently from this biodiversity can lead to major upsets or can only be limited to a specific digester.

One consequence of our direct measurement mode is the possibility to use this assay for evaluating the inoculum quality. This can be done by using the control samples on various inoculums or on the same inoculum sampled at different times. Troubleshooting digesters can be extremely difficult as it is usually operated as a black box. Using the ENVITAL® assay can help to quickly assess the level of activity given by the inoculum. One can imagine a microplate where different wells would be used with control samples amended with various limiting factors such as electron acceptors, inhibitors, other nutrients or micronutrients sources, etc. This would provide the operator with a quick and thorough check-up of the inoculum, along with possible recommendation for improving the digester’s performance.

The microplate format provides the operator with high-throughput analytical potential. As explained above, it can be used to assess the inoculum’s performance, but it can also be used to assess substrates mixture. Some of our work has involved the analysis of very homogeneous wastes, for instance agro-food residues containing mostly sugars. This type of residues is not the most appropriate substrate for methane production. However, when mixed with different and complementing substrates, the methane production can drastically increase. With classic BMP measurements being expensive and time-consuming, operators usually do not try to assess the potential of substrates mixtures. With the microplate format and a response in less than two days, this becomes very easy. Various mixture ratios can be assessed on the same plate, along with addition of micronutrients, modification of pH, or of any possible limiting factor.

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(108) RE-EVALUATION THE PARAMETERS IN COMPOSTING AND ANAEROBIC DIGESTION

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EXECUTIVE SUMMARY

Background of the work: The basic theories of composting and anaerobic digestion (AD) are supposedly well known. However, constant odour problems in the compost after tunnel composting and low energy yields in the dark fermentation process may be due to an inadequate understanding of some of their basic parameters. In biochemistry it is taken as read that an increase in temperature means an increase in biochemical activity as well. Composting, however, is supposed to be an exception: when the process temperature exceeds 60°C, the activity of the micro-organisms is claimed to be inhibited. Estimations of the performance capacity of the microbes carrying out these processes focus on the consumption of the atmospheric oxygen. The role of bound oxygen as oxidizing agent is all but neglected. The microbes' utilisation of the carbon is thought to be selective: lignin is held to be highly resistant while cellulose is held to be highly biodegradable. In acetogenesis of the AD the contribution of volatile fatty acids (VFAs) is supposed to be based on the acetic acid and higher VFA homologues. Formic acid is not believed to play an important role; indeed, reasons for its non-existence are not even addressed.

Objective of the work: addressing and answering he following questions: 1) are microbes able to utilise the chemically bound oxygen in substrates as oxidizing agent, and 2) should lignin be taken as a biodegradable constituent of plant material? Further important questions are 3) does the composting really differ from other biochemical reactions with respect to temperature behaviour, and 4) does the contribution of VFA homologues in AD acetogenesis begin with formic acid?

Main results: According to field experiments, it is evident that the efficiency of the composting process increases when the composting temperature increases from 60°C to 75°C. Composting thus does not necessarily differ from other biochemical reactions with respect to temperature behaviour. The important consequences of the mineralisation are: 1) the absolute ash content of the substrate does not change, and 2) the relative ash content increases due to the decrease of organic matter. When the relative ash content in aerobic or anaerobic mineralisation increases e.g. from 10% to 20%, the organic matter content of the substrate decreases by 55%. The contents of carbon and nitrogen in the residual mass tend to increase slightly while the content of hydrogen tends to decrease slightly. Therefore, microbes need to be able to utilise the chemically bound oxygen in the substrate as well. Subsequently respirometric experiments on the efficiency of composting need to be re-evaluated by taking into consideration the role of chemically bound oxygen in the substrate as a microbial source of oxygen, in addition to atmospheric oxygen. The elemental composition and the amount of humic substances should be expressed in reference to ash-free organic matter in order to avoid the suppression by the increasing ash content.

The zero-level atmospheric oxygen condition in composting should be described by the word 'hypoxic'. It indicates that although there is a shortage of atmospheric oxygen, conditions are not anaerobic, considering the possibility of utilizing the bound oxygen. For practical application, it is important to note that too-high intensity aeration (0.5 – 1.9 m^3/s) dries out the compost mass and may trigger the formation of offensive odours which remain in the compost mass after the reactor phase. According to Crawford and Crawford (1976), 47% of the ^{14}C marked natural lignin is degraded by microbes as $^{14}CO_2$. Lignin should be considered a biodegradable constituent in composting and in AD, like other organic compounds. Its rate of degradation may be slower than that of carbohydrates. Overcoming this slow-down is the challenge to practical processing.

Formic acid cannot be detected by GC-FID in the presence of other VFA homologues. Theories of AD need to be re-evaluated by considering formic acid as one of the VFA components in the acetogenesis. In order to produce CH_4 from CO_2, microbes need excessive hydrogen, even up to three times more than the stoichiometry of the reaction $CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O$ would suggest. This restricts the production of hydrogen in dark fermentation instead of methane.

Conclusion: Re-evaluating these parameters will broaden and clarify the theoretical and practical understanding of composting and AD.

1. INTRODUCTION

The basic theories of composting and anaerobic digestion are supposedly well known. However, constant odour problems in the compost after tunnel composting and low energy yields in the dark fermentation process may be due to an inadequate understanding of some of their basic parameters.

Composting and anaerobic digestion are mineralisation processes in which organic matter is partly mineralised to carbon dioxide and water/methane, and partly transformed to humus. Mineralization has a profound influence on all aspects of composting. Although being an important parameter, the mineralization rate is not too often assessed; however, in the studies where it is done its significance is obvious. Bernal et al. (1998) composted seven mixtures of organic wastes in a pilot plant using Rutgers static pile composting system. After 70 days of incubation they obtained a 25% mineralization of carbon as calculated from TOC. Hartz et al. (2000) determined mineralization rates of 19 manure and compost samples. The mineralization of manure C averaged 35% of the initial C content in 24 weeks, while compost C mineralization averaged 14%. Depending on the conditions of the composting mineralization rates between 15 to 50% can be achieved in a few months.

In the classical microbiological biochemistry of composting it is assumed that the energy of molecular oxygen is used and two molecules of water are formed (e.g. via oxidative phosphorylation), while oxygen of carbon dioxide comes from the substrate not from oxygen of air (e.g. via Krebs cycle). So estimations of the performance capacity of the microbes carrying out these processes focus on the consumption of the atmospheric oxygen. Importance is not given to the role of the chemically bound oxygen as oxidizing agent, even though the substrate contains nearly as much oxygen as carbon.

The microbes' utilisation of the carbon is thought to be selective: lignin (consisting of aromatic groups) is held to be highly resistant to microbial biodegradation, while cellulose (consisting carbohydrate groups) is held to be highly biodegradable (Lynch and Wood, 1985). Lignin degradation in soil is rather difficult to study – it is solubilised in alkaline solvents, and so non-humified lignin degradation products cannot be differentiated from those of humified materials. Some have solved this equation simply by putting an equal sign between humus and lignin, which is not necessarily the correct method. In anaerobic digestion there also is an assumption that lignin generally passes through ruminants unmodified (Hatfield and Fukushima, 2005). According these assumptions lignin should actually accumulate in composting and anaerobic digestion.

Although some researchers tend to assume that aromatic compounds are highly resistant to microbial degradation,it is widely accepted that aromatic compounds are degraded by microbes (Zouari and Ellouz, 1996; Budzinski et al., 1998; Arvin et al., 1989). Crawford and Crawford (1976) marked natural lignin with the ^{14}C from which 47% liberated microbially as $^{14}CO_2$. In environmental biotechnology, the microbiological degradation of aromatic compounds is so prevalent and important an issue that microbial populations specialised for that purpose can be patented (Markl et al., 1998).

In biochemistry it is taken as read that an increase in temperature means an increase in biochemical activity as well. Composting, however, is supposed to be an exception: when the process temperature exceeds 60^oC, the activity of the micro-organisms is claimed to be inhibited (Finstein et al., 1980, Golueke 1991). This assumption is argued by respirometric studies. In a review by Regan and Jerris (according to Golueke 1991), the highest oxygen uptake was at the temperature of 45^oC. However, for hygienic reasons, Golueke set the optimum composting temperature between 55 to 60 ^oC. As a consequence of this assumption, composting temperatures are kept below this limit by increased aeration

A full-scale study was organized to determine the best composting practices for The Helsinki Metropolitan Area Council in 1993 (Tolvanen et al., 1998). In winter 1993-1994 the temperatures in the windrows reached 60^oC in two weeks and in a three-month period the maximum composting temperature was 70^oC. In one windrow the temperature even rose to 84^oC fourth months from the start of the process. The City of Jyväskylä adopted source separation and collection of kitchen biowaste in 1996, and a large-scale windrow composting study was conducted as well (Koivula et al., 2000). In order to better control the odours, windrow height was reduced to 1.5-2 m which was one meter less than in the Helsinki-based study. In low windrows the temperature rose to 85^oC in the winter of 1997. High temperatures had no obvious harm to the process, rather, the odorous emissions were at a relatively low level.

In anaerobic digestion, the contribution of volatile fatty acids (VFAs) is supposed to be based on the acetic acid and higher VFA homologues. Formic acid is not believed to play an important role; indeed, reasons for its non-existence are not even addressed.

1.2 Research objectives

The aim of this research is to study the importance of some parameters in composting and anaerobic digestion. The following questions are addressed: 1) are microbes able to utilise the chemically bound oxygen in compost mass as oxidizing agent, and 2) should lignin be taken as a biodegradable constituent of plant material? Further questions are 3) does the composting process really differ from other biochemical reactions with respect to temperature behaviour, and 4) does the formation of VFA homologues in anaerobic digestion begin with formic acid?

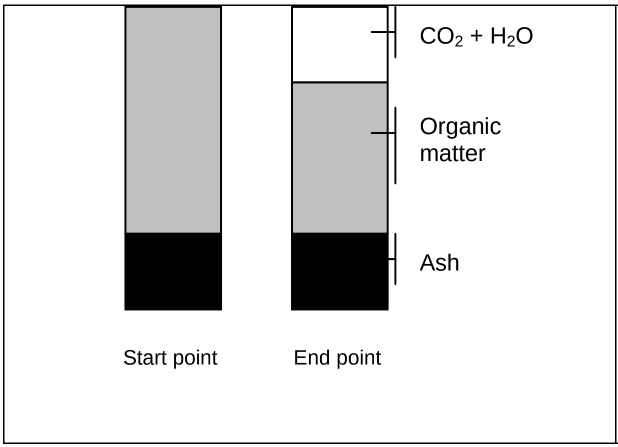
2. METHODOLOGY

Deductions are made by mathematical calculations based on the quantification of mineralisation in composting and AD, by the review of the research work of the author of this article, and by the review of the literature.

3. RESULTS AND DISCUSSION

3.1. Mineralization

The important consequences of the mineralisation are: 1) the absolute ash content of the substrate does not change, 2) the relative ash content increases due to the decrease of organic matter. Tim Haug (1993) presents an elaborated scheme for calculating the mineralisation. Haug’s formula can be visualised as shown in Figure 1.



Composted mass at the start: 1000 t.
Ash content at the start: 10% (= 100 t)
Ash content at the end: 20% (= 100 t)

Remaining composting mass at the end can be calculated as follows:

$$\frac{20\ x}{100} = 100\ \text{t} \quad \Leftrightarrow \quad x = \frac{10000\ \ \text{t}}{20} = 500\ \ \text{t}$$

Remaining organic matter at the end point is 500 t – 100 t = 400 t. Thus, 500 t or 55% of organic matter is mineralised.

FIGURE 1. **Calculation of the loss of organic matter with the aid of absolute and relative ash content. In the case of anaerobic digestion, mineralised products are CO_2 + CH_4; otherwise the reasoning is the same.**

As seen in Figure 1, just by measuring the ash content, one gets a quantitative picture of the consumption of organic matter in the composting/anaerobic digestion process. Due to the decrese of organic matter, the results should be expressed on the basis of ash-free organic matter. Otherwise the time-dependent increase of the relative ash content during the processes will suppress the time-dependent trends of the elemental composition and the humic substances.

In anaerobic digestion an average volume of methane gas can be calculated from the mineralization data as well: Let us suppose that 1) the ash content is 20% at the beginning and 40% at the end So out of 1000 t of starting material, 500 t of organic matter is mineralized as methane and carbon dioxide (biogas).

$m_{CH4} + m_{CO2}$ = 500000 kg, and m = n*M (n = moles and M = molecular weight), so
n_{CH4}* M_{CH4} + n_{CO2}*M_{CO2} = 500000 kg
$m_{CH4} = V_{CH4}$*ρ_{CH4} ja $m_{CO2} = V_{CO2}$*ρ_{CO2}, so
n_{CH4}*V_{CH4}*ρ_{CH4}+n_{CO2}*V_{CO2}*ρ_{CO2} = 500000 kg
In biogas is an average of 66% CH_4 and 34% CO_2 by volume base, so $n_{CH4} = 2n_{CO2}$.
The densities are: $\rho_{CO2} = 1.96$ kg/m^3 and ρ_{CH4},= 0.74 kg/m^3, so we obtain an equation
2 V_{CO2}*ρ_{CH4} + V_{CO2}*ρ_{CO2} = 500000 kg
2*V_{CO2}*0.74 kg/m^3 + V_{CO2}*1.96 kg/m^3 = 500000 kg,
V_{CO2} = 145348 m^3 ~ 145300 m^3 and V_{CH4}*= 290696 m^3 ~ 290700 m^3

3.2 Role of bound oxygen in the microbial metabolism in composting

Composting and anaerobic digestion can be considered to be the early stage of the coalification series: when calculated on the ash-free basis, the amount of carbon tends to increase while that of oxygen tends to decrease in composting (Heimonen and Hänninen, 1997, Koivula et al., 2000). When considering the overall loss of organic matter, it means that the microbes must utilise bound oxygen even at rate a bit higher than they utilise the bound carbon. In the respirometric measurements only the uptake of atmospheric oxygen is measured, and the mineralization due to the consumption of the bound oxygen is not observed. A natural consequence is that dry matter determinations based on the respirometric oxygen consumption do not coincide with the actual dry matter loss calculated on the basis of gravimetric determinations (Iannotti et al., 1993).

Let us suppose that 30% of the carbon in the starting material is mineralized in composting. Carbon dioxide contains two oxygen atoms per one carbon atom, which means that the starting material should contain 60% of oxygen for that purpose alone. Additionally, of course, the starting material should contain that oxygen which remains in the residual composted matter. So the total oxygen content in the starting material should be around 80-90%, which is impossible.

The assumption that chemically bound oxygen would not be an oxidizing agent need to be revised so that microbial metabolism utilizes the bound oxygen in addition to the atmospheric oxygen. When microbes use the bound oxygen, they need less atmospheric oxygen. The validity of taking +55^o C to 60^{o}C as the optimum composting temperature may be called into question.

3.3 Microbial diversity vs. increasing composting temperature

An often presented fear is that temperatures above +60^{o}C would be harmful for the composting microbes. There are, however, field studies in which composting temperatures within the range of 70–75^o C caused no obvious harm to the microbial diversity. Rather, in composting at elevated temperatures of source-separated kitchen biowaste, the hyperthermophilic bacteria at 75^{o}C were present in relatively high numbers in the composts rather soon from the start, and their concentrations were highest during the highest composting temperatures (Table 1).

TABLE 1. **Bacterial count of source separated kitchen biowaste compost conducted at four different incubation temperatures. Amount of bacteria is expressed as x10^6 cfu/g compost (Tolvanen et al., 1998).**

Age of the compost	Psychrophilic bacteria (7 ^{o}C)	Mesophilic bacteria (37 ^{o}C)	Thermophilic bacteria (55 ^{o}C)	Hyperthermophilic bacteria (75 ^{o}C)
2 weeks	3.15	80	50	5.7
1 month	7.85	0.65	12.35	3
6 weeks	0.024	1.25	3.05	0.87
2 months	23.5	40	8.5	3.65
3 months	15	120	15	2
6 months	56.6	14	12.5	2

Miyatake and Iwabuchi (2005) clarified the characteristics of thermophilic bacteria in cattle manure compost. They investigated enzymatic activity and species diversity of cultivated bacteria at 54, 60, 63, 66 and 70^{o}C. The highest level of thermophilic bacterial activity was observed at 54^{o}C. Following an increase in temperature to 63^{o}C, a reduction in bacterial diversity was observed. At 66^{o}C, bacterial diversity increased again, and diverse bacteria including *Thermus* spp. and the thermophilic *Bacillus* spp. appeared to adapt to the higher temperature. At 70^{o}C, bacterial activity measured as superoxide dismutase and catalase activity was significantly higher than at 66^{o}C.

During primordial times temperatures may have been higher than today. Microbes were among the first living organisms on this planet. One would expect that during evolution microbes acquired resistance to high temperatures. In composting, some microbes with lower optimum operative temperature can enter a dormant stage and survive.

When the temperature decreases and reaches the optimum level for those microbes, they can again become operative. Therefore, high temperatures do not necessary endanger the diversity of the microbial population.

3.4 Hypoxic conditions in composting

Atmospheric oxygen is fed into the system to guarantee aerobic conditions. In the active phase of composting it may locally and temporarily be exhausted even though aerobic conditions still overall prevail. In those locations of the heap or windrow the process is in **hypoxic condition**. The anaerobic process is carried out in airtight reactors specifically to prevent the access of atmospheric oxygen, and to achieve overall anaerobic conditions. It is very unlikely that methanogenic bacteria could become operative when the compost is under hypoxic conditions.

Presently the zero-level atmospheric oxygen condition is described as anaerobic. Use of the term 'anaerobic' is further misunderstood as implying anaerobic process conditions suitable even for methanogenic bacteria to operate. Use of the word 'hypoxic' in these occasions clarifies the situation, by indicating that although there is a shortage of atmospheric oxygen, conditions are not anaerobic.

Due to insufficient availability of atmospheric oxygen, facultative composting microbes change their mode of action from aerobic to hypoxic and the process begins producing more odorous compounds. It may not have much influence on the welfare of microbes – perhaps their metabolic activity in terms of temperature is a bit lower. Humans, however, are so sensitive to the foul odours generated that composting as a technological process is endangered when compost smells: nothing else then matters.

Odour generation in hypoxic mode seems to take place particularly between 45^o and 55^o C. At higher temperatures the thermal movements in the chemical bonds of the substrate increase in speed. Bound oxygen then becomes more feasible in terms of energy to be utilised by microbes. They compensate for the shortage of atmospheric oxygen, and when the temperature is high enough, the utilisation of bound oxygen is facilitated, and there is less of a need to change to hypoxic metabolism. Less odorous emission is generated and the overall composting efficiency increases, even though the uptake of atmospheric oxygen decreases.

3.5 Drawbacks due to a too-high atmospheric oxygen flow in composting

The fear of reaching a hypoxic stage if enough atmospheric oxygen is not available is encountered by increasing the aeration rate. Aeration of compost piles is kept at a rather high level, even 0.5 to 1.9 m^3 per second in some tunnel composting applications. However, in high flow conditions the air is guided through a few main channels. High flow in main channels sucks the air from small adjoining channels. Microbes there respond to the lack of oxygen and start to function in hypoxic mode, producing odorous gases, part of which remain in the mass. It may also cool the compost mass so that hyperthermophilic bacteria would not become fully operative. In spite of watering, the composting mass is then also dried. In curing windrows this dry mass may be prone to self-ignition. Paradoxically, by maximising airflow in order to try to improve the composting prerequisites, processing costs are increased, and the overall outcome is actually worsened.

3.6 Degradation of lignocellulose in composting and anaerobic digestion

Carbohydrates have been evolved earlier than lignin, and already then undergone the humification process as well. Sea plants do not contain lignin, and obviously they have been humified in the same manner over the eons. It would be logical to suppose that the later evolution of lignin would not have caused a change in the mode of action of humification, rather lignin has adopted as an additional group of compounds to consume. Carbohydrate structures are not completely mineralised in the humification. Parts of the structures are merely transformed by microbial metabolism first to secondary carbohydrates, and then to humus. This means that carbohydrate structures are recycled in humification. It gives to them a kind of dynamic stability, recalcitrance in the environment. Lignin structures on the other hand are not recycled via microbial metabolism in humification. Lignin can be considered a disposable constituent in humification (Hänninen, 2010).

From an evolutionary perspective, an important advantage for ruminants would be that they could also utilize the energy content of lignin. That is why it is feasible to suppose that microbes in the digestive tract of a cow or a termite could also degrade lignin.

This conclusion can be supported by the fact that a microbial consortium by enrichment culture on (aromatic) syringate can be isolated from the gut of a termite. Such a culture was used to improve the anaerobic degradation of olive-mill waste-water (Hamdi et al., 1992).

Lignin content is most commonly determined by the Klason or 72% (v/v) H_2SO_4 acid procedure. The method was originally developed in the early 1900s by Klason using 64 to 72% H_2SO_4 to dissolve away all of the polysaccharides, leaving lignin as an insoluble residue. The amount of extracted lignin is then determined gravimetrically. Determination is a valid method for wood and grasses, as it is known that they contain lignin.

However, modifications in the extraction procedures may influence the yield of lignin, so that even the same plant may produce different yields of lignin (Hatfield and Fukushima, 2005). So the method is not selective even in this case.

The Klason lignin method, however, is not a specific and selective method to identify lignin in matrices which are not known to contain lignin. Applying the Klason lignin method, for example, to peat produces an acidic extract the weight of which can be determined gravimetrically. According to Czapek (1899) peat does not contain lignin, so peat precipitate produced with Klason lignin method cannot be identified as lignin.

If lignin did not degrade during humification at all, it should accumulate. The relevance of this possibility can be studied mathematically. The normal ash content of legume-grass forages is close to 9.0% by DM basis (Hoffman and Taysom, 2004). The ash content of cow dung is around 27% (Chirag, et al., 2011). The average lignin content of grass is 17%, thus 1000 kg of grass contains 90 kg of ash and 170 kg of lignin. When a cow eats 1000 kg of this grass, it produces 333 kg of dung (Figure 1), of which the lignin content should be 51%. When 1000 t of this dung is further treated anaerobically to the mineralisation rate of 40% of ash, the amount of ash free of hydrolysis residue (digestate) will be 405 t. This amount cannot contain 510 t of lignin, so it must degrade considerably both in the rumen of the cow and in anaerobic digestion reactor.

3.7 Volatile fatty acids in anaerobic digestion

In the acid state of anaerobic digestion, facultative bacteria disintegrate soluble compounds into simpler ones. The intermediate products are volatile fatty acids (VFAs) such as acetic, propionic and butyric acids. However, anaerobic digestion theory does not account for the existence of formic acid – the starting homologue of VFAs. Nor are reasons presented for the non-existence.

VFAs are generally measured by a gas chromatograph (GC) using a flame ionisation detector (FID), a universal detector. The only exception is formic acid, which FID cannot detect in the presence of other VFA homologues – this is one likely reason for the non-existence of formic acid in the theory. But non-existence in GC-FID detection does not necessarily correspond to an actual non-existence in nature. When formic acid was derivatised for detection by GC-FID, its existence was possible to demonstrate among the VFAs formed in composting (Himanen et al., 2006).

The activation energy of formic acid is low, so it disintegrates rather easily into carbon dioxide and hydrogen, according to the following formula:

$$HCOOH \rightarrow CO_2 + H_2 \qquad (1)$$

Microbes can utilise this hydrogen as cheap starting energy in acetogenesis to overcome the more energetic activation barriers of higher VFA homologues.

The theory of anaerobic methanogenesis presupposes that an important step would be the reaction:

$$CO_2 + 4H_2 \rightarrow CH_4 + 2\,H_2O \qquad (2)$$

Hydrogen is in fact a better fuel than methane, and for the retrieval of hydrogen the reaction stoichiometry seems very advantageous if these four moles of hydrogen are saved. This has been attempted in the dark fermentation process by stopping the reaction before it enters the methane stage. The energy yield, however, has been only one-tenth of that amount, which can be obtained if the process is not interfered (Gómez et al. 2009, Kothari et al., 2010). The reason is quite obvious: to be feasible the reaction (2) needs a 2- to 3-time excess of hydrogen (Stephenson and Stickland, 1933).

When microbes are deprived of hydrogen, their most important source of starting energy, their overall functioning will take place at a much lower efficiency.

4. CONCLUSIONS

Composting and anaerobic digestion are considered to be an established, widely used and well-studied processes. However, in this paper some established statements are put into question with the aim of trying to open a new round for discussion on the theory of composting and anaerobic digestion. The mineralization in aerobic and anaerobic processing should be acknowledged, and elemental composition and the amount of humic substances should be expressed in reference to ash free organic matter to avoid the suppression by the relative increase of the ash content.

Atmospheric oxygen is not the sole source of oxygen for microbial oxidation in composting. Consequently in composting process respirometric measurements alone are not a reliable indication of microbial efficiency, especially in temperatures exceeding 55-60°C. Respirometric experiments need to be re-evaluated by taking into consideration the role of chemically bound oxygen in compost mass as a microbial source of oxygen, in addition to atmospheric oxygen. The temperature frame of microbial count measurements needs to be increased up to 75°C for estimating and understanding the changes of microbial diversity at temperatures above 55-60°C.

For practical application, it is important to note that too-high intensity aeration dries the compost mass and may trigger the formation of offensive odours which remain in the compost mass after the reactor phase. It may also cool the compost mass so that hyperthermophilic bacteria would not become operative which actually slows down the overall efficiency of the composting.

Degradation of aromatic compounds and lignin by microbes is well documented in practical applications, even at the level of accepted patents. Therefore lignin should be considered a biodegradable constituent in composting and anaerobic sciences as well in order that real life would have better reflection of the established facts. The rate of lignin degradation may be slower than, for example, that of carbohydrates, and overcoming this slow-down is the challenge to practical processing. It seems that microbes are omnipotent in degrading the organic matter.

Theories of anaerobic digestion need to be re-evaluated by considering the contribution of formic acid as one of the VFA components. In order to produce CH_4 from CO_2, microbes need excessive hydrogen, even up to three times more than the stoichiometry of the reaction $CO_2 + 4H_2 \rightarrow CH_4 + 2\ H_2O$ would suggest. This severely restricts the possibilities of producing hydrogen in dark fermentation instead of methane.

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(123) MONITORING BIODEGRADABILITY OF LIGNOCELLULOSIC BIOMASS DURING ANAEROBIC DIGESTION

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EXECUTIVE SUMMARY

Energy crops and crop residues can be digested anaerobically either alone or as co-substrate to produce biogas, a versatile renewable energy source. In the agricultural sector, this biomass is often co-digested with animal manure and slurry, one of the most significant agricultural waste streams. In spite of the growing number of agricultural biogas plants using agricultural waste and energy crops, the use of process simulation models for predicting and defining plant behaviour is still very limited, mainly due to the complexity of the process and the heterogeneity of the substrates. Also, the lack of standardized substrate characterisation methodology contributes to this issue. The chemical oxygen demand (COD), widely used as a mass balance term in modelling of wastewater treatment plants, could be applied in this field. However, COD is difficult to measure in the case of heterogeneous and solid substrates, including samples with high solids content. This, in turn, has limited its use to date to evaluate substrate biodegradability over time and methane production potential in the field of co-digestion of agricultural by-products. This research paper reports and analyses experimental results on the applicability of COD as a substrate characterisation and process monitoring parameter in the context of lignocellulosic biomass digestion, as collected from the research work currently being undertaken at the University of Luxembourg. The reliability of this parameter was investigated using different silages. Four sets of batch experiments were carried out using maize silage (12.17gVS/l), grass silage (14.58gVS/l), and two co-digestion mixtures of 40% maize and 60% grass (18.31 gVS/l) and 70% maize and 30% grass (12 gVS/l) in 1 litre reactors running under mesophilic conditions. The reactors were inoculated with pre-acclimatized inoculum from Beckerich agricultural biogas plant in Luxembourg. Soluble and particulate COD, volatile solids (VS), total solids (TS), volatile fatty acids (VFAs), and gas production, including composition, were monitored over a period of 24 days. A few adapted analytical procedures for measuring COD in solid and heterogeneous substrates and samples with high suspended solids concentration, in comparison to standard methods widely used for wastewater, have been proposed in recent years but these have rarely been applied and validated in the field of digestion of agricultural by-products. The COD methodology adopted in this study was developed by the process research group at the University of Luxembourg as a modification of the DIN 38414-S9 considering some suggestions from Raposo et al. (2008). The modified COD method was applied to different substrates (i.e. grass, maize silages, and cellulose) and also to monitor particulate COD (pCOD) evolution during anaerobic digestion of the different silages. The soluble COD (sCOD) method as established in the ISO 15705 standard method was modified for these experiments as the digestate has relatively high suspended solids concentration. First, the measured substrate COD concentration was compared with the theoretical oxygen demand for each substrate. COD recovery obtained was 96% for cellulose, 92.5% on average for the different maize silages tested and 91.5% on average for the grass silages tested. For all different batch series, the pCOD decreased overtime as a result of the hydrolysis of the particulate fraction of the substrate. The pCOD evolution along with the methane accumulative yield suggested that the solubilisation of the substrate was slower in the case of the co-digestion experiments. sCOD, on the other hand, initially increased sharply as a result of the solubilisation of the substrate and subsequent acidification and then decreased as digestion progressed. After 9 days of digestion, 77%, 71%, 69% and 48% substrate to methane conversion rates had been achieved for the experiments fed with grass silage, maize silage, the 70% maize/30% grass mixture and the 40% maize/60% grass mixture, respectively. Measuring the pCOD and sCOD together with biogas and VFAs concentration during digestion allows, not only to estimate the degradation advancement through COD mass balance, but also to obtain relevant information about the solubilisation of the particulate fraction (hydrolysis step).

1 INTRODUCTION

1.1 Background

Biogas has a significant role to play in combating climate change. It is a versatile renewable energy source, as it allows for a variety of applications, including electricity and heat production and vehicle fuels. The fermentation process is a multi-step biological process where the organic carbon is converted into CO_2 and CH_4. It can be divided into four phases with different microorganisms participating in each of them, namely hydrolysis, acidogenesis, acetogenesis and methanogenesis. The theoretical biogas yield of any substrate is defined by its content of carbohydrates, proteins, and fats, which have each different gas yields and bio-availabilities.

Biogas can be produced through anaerobic digestion of different organic substrates. In the agricultural sector in particular, the lignocellulosic biomass (e.g. energy crops and crop residues) can be digested anaerobically either alone or, most commonly, as co-substrate to produce biogas. Indeed, slurry and manure, one of the most significant agricultural waste streams, are often co-digested together with energy crops in order to increase the gas yield because of their higher methane potentials. This is particularly the case of countries such as Germany and Austria, where dedicated crops are grown in some areas with the sole purpose of biogas production.

In spite of the growing number of agricultural biogas plants using agricultural waste and energy crops, the use of process simulation models for predicting and defining plant behaviour is still very limited, mainly due to the complexity of the process and the heterogeneity of the substrates. Only recently efforts have been put into the modelling of the anaerobic digestion of energy crops as the main substrate or co-substrate. Therefore, documented studies in this area are still limited. Recent examples include Lübken et al. (2007) (cattle manure and fodder for cows); Thamsiriroj & Murphy (2010) (grass silage); Wichern et al. (2009) (grass silage); or Koch et al. (2010) (grass silage). These studies apply the Anaerobic Digestion Model N°1 (ADM1) (Batstone et al., 2002) to simulate the fermentation of energy crops (mainly grass silage). The ADM1, like other models, uses COD, which is an indirect measure of the organic matter, as the chemical component base unit. Nevertheless, the application of the ADM1 to energy crops has been reported to be problematic. The main challenges are related to the substrate characterisation methods for the incoming digester feed (i.e. lack of a standardized method) and the associated model definition of the enzymatic disintegration and hydrolysis steps (Koch et al., 2010; Lübken et al., 2007), which has been considered as the rate-limiting step in anaerobic digestion by different authors. However, COD is difficult to measure in the case of heterogeneous and solid substrates, or in the case of mediums with high solids content. The loss in accuracy when applying to solid substrates standard methods widely used for water and wastewater, such as the standard methods 5220 B-open reflux and 5220 D-closed reflux colorimetric of the American Public Health Association–American Water Works Association–Water Pollution Control Federation (APHA–AWWA–WPCF), has been previously highlighted (Raposo et al., 2008).

A few adapted analytical procedures for measuring COD for solid and heterogeneous substrates and for samples with high suspended solids concentration have been proposed in recent years (Raposo et al. 2008; Yadav et al. 2006) but these have rarely been applied and validated in the field of digestion of agricultural by-products. During digestion, VFAs are commonly monitored over time in the digestate, most rarely soluble COD (sCOD) (e.g. Raposo et al., 2006; Wichern et al., 2009), while particulate COD (pCOD) or total COD is not usually assessed. In some cases, COD evolution has been estimated from available data for other parameters, i.e. converting volatile solids (VS) data into COD (Lübken et al, 2007; (Thamsiriroj & Murphy, 2010). As for substrate characterisation, while Lübken et al. (2007) applied the German Standard Methods for the examination of water, wastewater and sludge (DIN 38414-S9), Koch et al. (2010) used the ThOD to transfer measurements to COD.

1.2 Research objectives

The aim of this study was to assess the experimental applicability and reliability of COD as a substrate characterisation and process monitoring parameter in the context of lignocellulosic biomass digestion, to be measured through an adapted method for COD determination recently proposed by the process research group at the University of Luxembourg as a modification of the DIN 38414-S9 considering the suggestions by Raposo et al. (2008). A detailed method development description is provided by Sibisi-Beierlein and Greger (2012). Two objectives were established:

- To apply the adapted method to measure the particulate COD in different substrates and to compare the results with the Theoretical Oxygen Demand (ThOD), estimated from van Soest and Weende analysis, in order to determine the reliability of this parameter for solid substrate characterisation.
- To apply the modified method to monitor the evolution of both the particulate and soluble material (pCOD and sCOD) of the substrate during digestion of two different energy crops and to perform a COD balance for the conducted experiments.

2 METHODOLOGY

2.1 Anaerobic digestion experiments

Four sets of batch experiments were carried out using maize silage (12.17gVS/l), grass silage (14.58gVS/l), and two co-digestion mixtures of 40% maize and 60% grass (18.31 gVS/l), and 70% maize and 30% grass (12 gVS/l) in 1 litre reactors running at mesophilic conditions. Maize and grass harvested in May and January 2009 respectively were used in the assays. The reactors were inoculated with pre-acclimatized inoculum from Beckerich agricultural biogas plant in Luxembourg. Each experimental series counted with 11 reactors with 750g of inoculum and one control ("blank") reactor without addition of substrate running in parallel and kept in water baths at 39°C.

2.2 Analytical methods

2.2.1 Determination of particulate and soluble COD for substrate characterisation

Total COD in the substrate was calculated as the sum of the pCOD and sCOD. pCOD was measured according to an adapted method based on the German standard method DIN 38414-S9, originally developed to measure the organic content in sludge and sediments. The suggestions made by Raposo et al. (2008), i.e. to simplify the initial method, were also taken into account. The samples were dried at 105°C and grinded to pass through 1mm sieve before measurement. A detailed description of the modified method can be found in Sibisi-Beierlein and Greger (2012). The sample is heated at 148°C for 2 hours with potassium dichromate as the oxidant and silver sulphate as the catalyser in a strong sulphuric solution. A potentiometric back titration method was used to measure the reaction end-point (i.e. remaining dichromate in the sample). Reagents were prepared as suggested by Raposo et al. (2008) but the content of mercuric sulphate (HgSO4) in the digestion reagent, which is added to avoid the chloride interference, was adapted to 2g according to the measured chloride concentration in the inoculum and digestate. The samples were measured in triplicates and duplicates.

The adapted method for COD determination was applied to 3 different types of substrates: Cellulose, maize silage and grass silage. Cellulose (microcrystal cellulose; Euro OTC) was selected as it is a pure and homogenous substrate present in most agro-wastes, while grass and maize are very popular substrates in Central Europe's agricultural biogas plants (Amon et al., 2007). In the case of maize and grass, silages from different harvesting periods and origins were analysed.

In order to assess the accuracy of the applied adapted method for pCOD measurement in solid substrates, the results thus obtained were compared with the Theoretical Oxygen Demand (ThOD) calculated on the basis of the carbohydrates, proteins, lipids and inerts content in each case, measured through near-infrared spectroscopy (NIRS) by the Luxembourgish Administration for agricultural technical services (ASTA). These fractions correspond to the particulate fraction of the COD. The results from the spectophotometer analysis were calibrated on the basis of a database for crops, soils and organic waste, REQUASUD, from an association of nine Belgian agricultural laboratories. The substrates were thus fractioned into crude protein (RP), crude fat (RL), crude fibre (RF), acid detergent lignin (ADL) and N-free extract (NfE). The sum of RF and NfE represents the total carbohydrate content and ADL represents the lignin (Lig) content, which is assumed not to be degradable and allocated to the inert fraction. Carbohydrates were further divided into hemicellulose (neutral detergent fibre (NDF) minus acid detergent fibre (ADF)) and cellulose (ADF–ADL). NDF and ADF were measured according to van Soest and Wine (1967) method using a Fibretherme FT 12 apparatus.

Four fractions were considered for all substrates for the calculation of the total ThOD for each substrate, as suggested by Koch et al. (2010): the fractions of proteins (RP), lipids (RL), carbohydrates (RF+NfE-ADL) and lignin (ADL). The basic molecule of starch, cellulose and hemicellulose is glucose and so these 3 components were summed up as one fraction (Ch). Once the composition was estimated for each substrate considered through NIRS, the amount of each fraction was calculated (as gTS) for 1 gram of Fresh Matter (FM) and then multiplied by the conversion factor, the ThOD (gO_2/gTS) for each fraction calculated from the known elemental composition as shown in Table 1 using equation 1. For each substrate analysed, the total ThOD (for 1g FM) was estimated as the sum of the ThOD of each fraction according to equation 2.

For a substance with a general chemical formula $C_aH_bO_cN_d$, $ThOD(gO_2/g_{substance}) = \frac{16(2a+0.5(b-3d)-c)}{12a+b+16c+14d}$ Eq.1

$$Total\ ThOD = TS \times \left[(RL \times ThOD_L) + (RP \times ThOD_P) + ((RF + NfE - ADL) \times ThOD_{Ch}) + (ADL \times ThOD_{Lig})\right] \quad [gO_2] \qquad \text{Eq. 2}$$

where TS is the total solids content (%FM) and $ThOD_X$ is the ThOD of each fraction (gO_2/gTS)

TABLE 1 **Theoretical oxygen demand of different fractions**

Fraction	**Formula**	**ThOD (gO_2/gTS)**
Carbohydrates	$C_6H_{10}O_5$	1.19
Proteins	$C_5H_7NO_2$	1.42
Lipids	$C_{57}H_{104}O_6$	2.90
Inerts (Lignin)	$C_{10.92}\ H_{14.24}O_{5.76}$*	1.56

*The elemental composition was taken from Nadji et al. (2009)

Regarding the sCOD present in the incoming substrate, it was considered to correspond to the VFA fraction, which is volatilised during drying. Literature values were used for the estimation of the VFAs concentration, in particular values reported by Weiβbach and Strubelt (2008 a; 2008b), who collected VFAs data for both maize and grass silages. Reported average values were used for both substrates. For maize silage, 9.98 g/kg, 0.28g/kg, 0.01g/kg, 0.09g/kg, 0.06 g/kg, 0.01 g/kg, and 15.20 g/kg concentrations for acetic acid, propionic acid, iso-butyric acid, butyric acid, iso-valerate, valeric acid and lactic acid respectively were used. For grass silages, 8.27g/kg, 0.45g/kg, 0.19g/kg, 2.06g/kg, 0.36 g/kg, 0.1g/kg, and 14.63 g/kg concentrations for acetic acid, propionic acid, iso-butyric acid, butyric acid, iso-valerate, valeric acid and lactic acid respectively were used. The concentrations were then converted to COD using stoichiometric equations and summed up.

2.2.2 Anaerobic digestion monitoring

pCOD, sCOD, VS, TS, VFAs, and gas production, including composition, were monitored over a period of 24 days. pCOD was measured in the reactor with the adapted method (section 2.2.1).. For the measurement of the sCOD, the ISO 15705 standard method was modified, given the relatively high suspended solids concentration in the digestate. Details of the adapted methodology are given in Sibisi-Beierlein and Greger (2012). In brief, the samples were first sieved through 1mm sieve and then centrifuged for 10 minutes at 13000 rpm (Minispin, Eppendorf, Germany). The aliquot was then filtered through a 0.45 μm membrane filter and finally sCOD was determined spectrophotometrically with the Dr Lange® (Hach Lange, Germany) cuvette tests. The sCOD was only measured in the co-digestion batches. The VS and TS were determined according to EN ISO 12879 and 12880. The Samples (60 ml in duplicate) were dried 24h (for TS) at 105°C and incinerated 24h at 550°C for VS determination. The individual VFAs concentrations were quantified by gas chromatograph (GC) using a "FOCUS" GC instrument from Thermo Electron SpA with a Econo-CapTM-1000 (Grace) capillary column using Helium as a carrier gas and flame ionization detector (FID). The gas volume was measured according to the water displacement principle in gas meters filled with a highly concentrated salt solution (30g / 100ml) to prevent the solution of gas in the liquid. The gas volume was measured and expressed at standard temperature and pressure (STP). The composition of the gas, CH_4, H_2S, CO_2 and O_2 were determined with a gas analyser (Biogasmonitor BM2000, Ansyco) equipped with an infrared cell (for CH_4 and CO_2) and internal electrochemical cells (for O_2 and H_2O measurement).

3 RESULTS AND DISCUSSION

3.1 COD of substrates used in lignocellulosic biomass digestion

Table 2 presents the average composition of different maize and grass silages as measured with the NIRS method (Weende fractions) and chemical analysis method (van Soest fractions). The obtained values, which were used for the calculation of the ThOD for each substrate, are in line with those found in literature for equivalent harvesting periods (e.g. Amon et al. (2007) for maize and Wichern et al. (2009) for grass).

TABLE 2 **Main components of different grass and maize silages analysed**

Parameter	Units	Maize 05/2009	Maize 10/2010	Maize 06/2011	Maize 10/2011	Grass 01/2009	Grass 06/2010	Grass 09/2008	Grass 2007
TS	% FM	34.08	31.95	19.04	22.84	30.53	36.93	50.56	19.58
VS	% FM	32.33	30.60	18.47	21.80	27.16	34.01	45.12	16.88
CODp	gO2/kgTS	1193.94	1010.00	1141.12	1148.83	1030.00	853.47	1182.00	1164.00
Raw ash (Ra)	% FM	4.06	4.39	3.36	4.39	9.98	11.15	8.37	17.59
Raw protein (RP)	%TS	6.94	8.06	7.45	9.07	14.69	19.08	16.19	16.87
Raw fibre (RF)	%TS	20.22	21.50	22.66	24.03	29.54	29.85	25.56	29.34
Raw lipid (RL)	%TS	2.86	2.00	3.07	2.54	2.61	3.56	3.28	4.14
NfE*	%TS	65.92	64.05	63.46	59.97	43.18	36.36	46.60	32.06
NDF	%TS	44.70	49.82	54.53	54.53	56.56	50.70	56.56	57.37
ADF	%TS	26.22	26.86	28.47	28.39	29.81	27.77	29.47	41.72
ADL	%TS	3.50	3.51	3.88	4.01	3.86	3.84	4.64	3.98
Hemicellulose	%TS	18.48	22.96	26.06	26.14	27.31	22.92	27.09	15.65
Cellulose	%TS	22.72	23.35	24.59	24.38	25.95	23.93	24.83	37.74

*Calculated value

Table 3 shows the results obtained for the ThOD of the different substrates analysed and also the experimental values of pCOD obtained using the modified method. The date next to each substrate indicates the harvesting season.

TABLE 3 **Comparison between the ThOD and the experimental pCOD values for different substrates tested**

	ThOD (gO2/gTS)	COD experimental			
		Average (gO2/gTS)	SD (gO2/gTS)	R.S.D. (%)	Recovery(%)
Cellulose	1.185	1.14	0.0258	2.27	96.01
Maize 05/2009	1.215	1.19	0.0086	0.72	98.25
Maize 10/2010	1.199	1.01	0.0141	1.40	84.22
Maize 06/2011	1.230	1.14	0.0115	1.01	92.79
Maize 10/2011	1.213	1.15	0.0006	0.05	94.73
Grass (01/2009)	1.160	1.03	0.0141	1.37	88.79
Grass (06/2010)	1.172	0.85	0.0140	1.64	72.79
Grass 09/2008	1.197	1.18	0.0028	0.24	98.75
Grass 2007	1.102	1.16	0.0000	0.00	105.67

The recovery results for the cellulose, which was systematically used as internal standard for control purposes, showed high recovery values, of 96%, similar to those obtained by Raposo et al. (2008) for the same substrate applying their modifications proposed. For the heterogeneous substrates tested, namely the different maize and grass silages, the comparison between the ThOD and the experimental values was also highly satisfactory. Indeed, for the maize silages, the recovery ranged from 84% up to 98% while in the case of grass the recovery varied from 72% up to 105%. It should be noted that the variability observed in the recovery for each substrate (representing the difference between the experimental COD measurements and the estimated ThOD) could be attributed to possible deviations in the Weende and van Soest analysis results. The results also showed that with the adapted method for determining pCOD, the reproducibility of the results was generally good and suggested that incomplete oxidation of the substrate does not occur. As regards the sCOD, the TVFAs content in the feed of the different batches as calculated from literature values and the different loading, were 0.32 gCOD for the maize batch, 0.59 gCOD for the grass batch, 0.37 gCOD for the batch with the 70% maize/30%grass mixture, and 0.4 gCOD for the batch with 40% maize/ 60%grass mixture.

3.2 COD evolution during digestion

The specific methane production (SMP) was calculated for each experiment after 15 days by dividing the methane volume (Nl) by the amount of substrate added, expressed in gVS. The values obtained were 0.36, 0.33, 0.34 and 0.26 Nl CH4/g VS for the assays using grass silage (14.58gVS/l), maize silage (12.17gVS/l), the co-digestion mixture 70% maize/30% grass (12 gVS/l), and the co-digestion mixture 40% maize/ 60% grass (18.31 gVS/l) respectively. Thus the SMP was very similar for the assays fed with maize silage, the grass silage, and the mixture 70% maize/30% grass, and considerably lower (almost 30% lower) for the assay fed with the mixture of 40% maize/ 60% grass, suggesting some sort of inhibition, probably caused by the higher loading. The authors also examined the evolution of the pCOD, measured with the adapted method, the TVFAs (sum of C2 to C5 VFAs), and the cumulative methane production. These are presented in Figure 1 for the assays fed with grass silage and the 40% maize/ 60% grass mixture. For the later, the sCOD evolution is also presented. The COD equivalent of each of the VFAs measured was calculated using the following factors: 1.07 (C_2), 1.51 (C_3), 1.82 (C_4), and 2.04 (C_5). The methane conversion factor used to express cumulative methane production in gCOD is 2.85 gCOD/l_{CH4}.

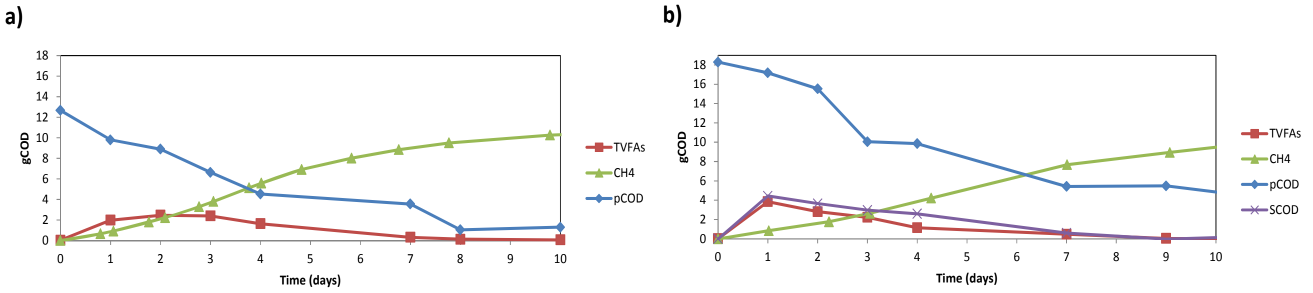

FIGURE 1 **Evolution of TVFAs, cumulative methane production, and pCOD for the grass silage assay (10.93 gVS added) (a) and the 40% maize/ 60% grass mixture assay (13.73 gVS added) (b). sCOD is also presented in figure 1.b).**

For all different experiments, the pCOD displayed a clear decreasing trend as a result of the hydrolysis of the particulate fraction of the substrate, thus providing useful information about the hydrolysis dynamics in each case. sCOD initially increased sharply as a result of the solubilisation of the substrate and subsequent acidification and then decreased as digestion progressed. It was observed in the assays fed with the two mixtures tested that this parameter followed a similar trend to that of the TVFAs during digestion. When considering the cumulative methane evolution along with the substrate solubilisation evolution, it is possible to grasp the dependence of these parameters on the organic loading but also on the substrate composition. The maize silage assay presented the fastest substrate degradation the first 4 days of digestion as could be expected from its measured composition (e.g. the maize silage used had a higher content of easily degradable carbohydrates, i.e. lower NDF). In line with aforementioned results, the substrate to methane conversion rate after 9 days of digestion was higher in the case of the mono-digestion assays (77% and 71% for the grass and maize silages assays, and 69% and 48% for the assays fed with the 70% maize/30% grass mixture and 40% maize/ 60% grass mixture respectively).

3.3 COD balance

As COD cannot be destroyed during anaerobic digestion, only redistributed, mass balances can be done in terms of this parameter. For a COD mass balance at any time in a digester, it is important to take into account that:

- The influent COD and that in the reactor during digestion can be divided into a soluble (sCOD) and particulate (pCOD) fractions, both of them composed of biodegradable and non-biodegradable compounds.
- In the substrate, the sCOD mainly corresponds to the VFAs, while in the digester this fraction includes VFAs but also other soluble components such as monomers (hydrolysis products).
- During digestion,

– The methane production is determined by the amount of substrate that has been solubilised into monomers and then into the different VFAs.

– Bacterial biomass is included in the particulate fraction.

– The COD of CO_2 is zero.

The COD balance in terms of COD for a batch system is illustrated in Figure 2.

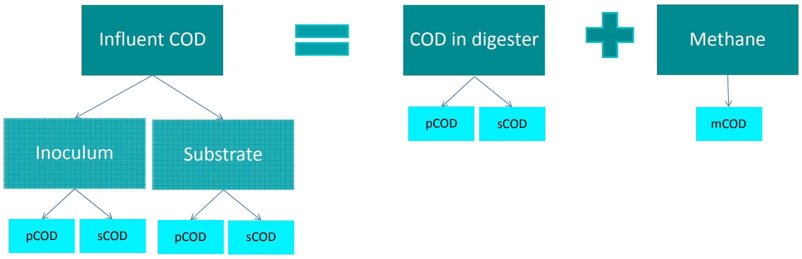

FIGURE 2 **COD balance for a batch reactor**

Table 4 presents the COD balance for both co-digestion assays fed with a mixture of 40% maize and 60% grass (18.31 gVS/l) and a mixture of 70% maize and 30% grass (12 gVS/l). The COD fractions are indicated in each case for both the influent and in the system as measured after 9 days. The COD removal at that time was 71% for the 40% maize/60% grass mixture and 79% for the 70% maize/30% grass mixture. The calculated COD recovery (before and after 9 days of digestion) was of 92% and 98% for the assays fed with mixtures of 40% maize/ 60% grass and 70% maize/30% grass respectively. These recovery results are quite satisfactory and suggest a good accuracy of the proposed adapted methods for estimating the sCOD and pCOD. On the other hand, it is important to highlight that the COD recovery is sensible to changes in the VFAs concentrations in the substrate, estimated from literature values, which presented a wide range for both substrates (for example the measured concentration of acetic acid for grass silage can vary from 2.53 up to 20.67 mg/gFM).

TABLE 4 **COD balance for the 2 co-digestion experiments with maize and grass silage**

		Fraction	**Unit**	**40% Maize/60% Grass (18.31gVS/l)**	**70% Maize+30% Grass (12gVS/l)**
Influent	**Substrate**	pCOD	gCOD	18.29	11.06
		sCOD	gCOD	0.40	0.37
	Inoculum	pCOD	gCOD	30.88	41.25
		sCOD	gCOD	5.35	7.26
After 9 days digestion	**Digestate**	pCOD	gCOD	36.36	43.57
		sCOD	gCOD	5.33	7.32
	Methane	COD in CH_4	gCOD	8.94	7.87
COD recovery			%	92	98

4 CONCLUSIONS

Overall, this study contributes to validate in the field of lignocellulosic biomass and in particular energy crops anaerobic digestion a method previously proposed by Sibisi-Beierlein and Greger (2012) based on the German Standard DIN 38414-S9 considering the suggestion of Raposo et al. (2008) for the determination of COD. The results suggest that the proposed modified method can be appropriate for measuring the organic content of solid substrates used commonly in agricultural biogas plants and that COD could be used with confidence as a substrate characterisation parameter. Moreover, the study suggests that the proposed COD methodology to measure pCOD and sCOD can be applied with confidence during digestion of different silages, either alone or as co-substrate, to monitor the solubilisation of the substrate, thus allowing getting a better insight into the hydrolysis phase. The results presented here can serve as the basis for further work to establish a standardised method for the determination of COD in the field. Nevertheless, further work is till necessary as to test the methodology during digestion of other substrate and mixtures and also to better define the sCOD content in energy crops, which in this study was taken as average VFAs concentration reported in literature.

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(134) PREDICTING THE BIOCHEMICAL METHANE POTENTIAL OF ORGANIC WASTE BY NEAR INFRARED SPECTROSCOPY

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EXECUTIVE SUMMARY

The biochemical methane potential (BMP) evaluates the ultimate amount of methane produced by any given waste or biomass under anaerobic conditions. This value is currently one of the most important parameter for the design and control of anaerobic digestion plants and more specifically in co-digestion plants where a broad rang of substrates can be treated. However, BMP test is time consuming (30-50 days) and expensive, making the current protocol non-adapted for industrial plants management and optimisation. New technologies providing fast determination of BMP become thus necessary. It has been demonstrated that Near Infrared Spectroscopy (NIRS) is a suitable method for fast prediction of a wide range of organic parameters for plant biomass, waste, or soil. It is a spectroscopic method using the infrared region of the electromagnetic spectrum (800-2500 nm). The prediction of the reference value is only based on spectral data and thus do not need any chemical analysis. Its suitability has been demonstrated for the monitoring of anaerobic digestion process, the control of the incoming feedstock and finally for the prediction of BMP in case of municipal solid waste (MSW) and meadow grasses.

The anaerobic co-digestion of organic solid waste is increasingly popular. It indeed offers several advantages in terms of biogas yield as well of as diversity of waste treated. Therefore, fast waste characterisation and more specifically BMP determination becomes crucial for industrial plant management and process monitoring. In that context the use of NIRS to predict BMP potential is a promising alternative. For most of the NIR models used in anaerobic digestion, calibrations were performed for one type of waste or substrate. These models would not therefore be able to take into account the variability of a wide range of waste.

The aim of our study was therefore to develop a NIRS calibration model for the prediction of the BMP value in a co-digestion context, that is, for a broad range of organic waste such as MSW, agro-industrial waste, meat waste, vegetables, fruits, crops…

A total of 296 samples including most of the substrates treated by anaerobic co-digestion were used for NIRS calibration and validation. Spectra collection and BMP tests were conducted in duplicates and for both, on dried and 1 mm grinded samples. The NIRS calibrations were performed by partial least square (PLS) regression between spectra and BMP values, and for different spectra transformations (multiple scatter correction, standard normal variate, de-trend and derivatives).

The NIRS predictions of the BMP values were satisfactory (Root Mean Square error = 40 ml $CH_4.kg^{-1}$ VS_{fed}; Relative Standard Error = 12.7%). The integration of the entire substrate diversity in the model remained nevertheless difficult due to specific organic matter properties and the high level of uncertainty of the BMP values. The addition of more samples in the dataset in order to perform local calibrations would probably improve this point. Calibrated with 224 samples, the determined model allows the prediction of the BMP for a large range of organic substrates.

1 INTRODUCTION

The Biochemical Methane Potential (BMP) evaluates the ultimate amount of methane produced by any given waste or biomass under anaerobic conditions. The information provided by the BMP value is important when evaluating potential substrates and for optimizing the design and functioning of an anaerobic digester (Raposo *et al.*, 2011a).

The anaerobic co-digestion of organic solid substrates, defined as the anaerobic treatment of a mixture of at least two types of substrates, is increasingly popular in Europe (Alvarez *et al.*, 2010). It indeed offers several advantages in terms of biogas yield as well as the diversity of substrates treated. The feasibility of adding new co-substrates in an already established process have been tested for many kind of substrates such as meat waste (Buendia *et al.*, 2009), fat, oil and grease waste (Long *et al.*, 2011), municipal solid waste (Hartman et Ahring, 2005), fruits and vegetables (Callaghan *et al.*, 2002), household waste, sludge and manure (Angelidaki and Ahring, 1997), etc.

Because of the wide range of BMP values of the different substrates considered in anaerobic co-digestion projects, the knowledge of the BMP value of a new substrate is crucial before its addition in an existing process or for the design of the industrial plant for its treatment.

However, the BMP test is time consuming (30-50 days) and expensive, making the current protocol non-adapted for industrial plant management and optimisation. New technologies providing fast determination of BMP become thus necessary. Several biochemical models have already been developed to predict faster the BMP value (reviewed in Lesteur *et al.*, 2010). Although a relatively good estimation of the BMP value is obtained in a shorter time with these models, time consuming laboratory experiments remain necessary.

Alternative methods have been studied. It has been demonstrated that Near Infrared Spectroscopy (NIRS) is a suitable method for fast prediction of a wide range of organic parameters for plant biomass, waste, or soil. It is a spectroscopic method using the infrared region of the electromagnetic spectrum (800-2500 nm). The prediction of the reference value is based only on spectral data without any chemical or biological analysis requirement. Moreover, a huge advantage of the NIRS is that many parameters, either complex or composition dependant, can be successfully predicted by one simple NIR spectra acquisition. Its suitability has been demonstrated for the monitoring of anaerobic digestion process (Jacobi *et al.*, 2009; Lomborg *et al.*, 2009; Reed *et al.*, 2011; Ward *et al.*, 2011), the control of the incoming feedstock (Jacobi *et al.*, 2011) and finally for the prediction of BMP of municipal solid waste (MSW) (Lesteur *et al.*, 2011) and meadow grasses (Raju *et al.*, 2011).

The use of NIRS to predict the BMP value is a promising alternative. However, the current published NIR methods for BMP estimation are limited to one type of substrate. For that reason, they are not suitable for the diversity of substrates than can be treated in a co-digestion context. The build-up of NIRS models taking into account this variability remains thus necessary.

The aim of our study is to develop a NIRS calibration model for the prediction of the BMP values in a co-digestion context, that is, for a broad range of organic substrates (MSW, agro-industrial waste, meat waste, vegetables, fruits, crops…).

2 METHODOLOGY

2.1 Data sample set

The data sample set included 296 organic samples: 57 agro-industrial waste (AGRO), 1 macro algae (ALG), 20 biowaste (BIO), 4 energy crops (ECROP), 11 fatty waste (FAT), 14 meat waste (MEAT) , 2 co-digestion mix (MIX), 66 municipal solid waste (MSW) , 42 plants and vegetables (PLANT), 18 agro-industrial sludge (SL_{agro}), 30 sewage sludge from wastewater treatment plants (SL_{wwtp}) and 31 stabilised municipal solid waste (STAB) .

The agro-industrial waste samples included solid food processing waste and non-conformed end products whereas the agro-industrial sludge samples only included sludge produced during the wastewater treatment on the agro-industrial plants. The municipal solid waste samples included fresh MSW collected on different mechanical-biological treatment plants and at different points of the sorting process. The stabilised MSW samples were collected during landfill drillings. The biowaste category contained household organic waste originated from source separated collection and catering waste. The meat waste category is mainly composed of slaughterhouse waste or stale meat. The fatty waste was collected on industrial sludge digesters that use such kind of substrate to increase methane production yield. The sewage sludge samples were collected on different wastewater treatment plants and at different points of the process.

The plants and vegetables category included agricultural waste (residues from wheat, barley…) and vegetables (potatoes, tomatoes…). Four samples of energy corps (maize, switch grass), 1 sample of macro algae and 2 industrial co-digestion mixes were also included in the data sample set.

2.2 Sample characterisation

In order to conduct the NIRS measurement, all samples were oven-dried at 40°C or freeze-dried and then grinded at 1 mm.

The Total Solid (TS) and Volatile Solid (VS) contents were determined according the French standards NF EN 12880 and NF EN 12879.

For all samples, except the wastewater sludge, the BMP was determined on freeze or oven dried and grinded samples. In the case of municipal sludge, the BMP tests were performed on fresh samples. All tests were performed in 0.5 L serums bottles, under mesophilic conditions and with a substrate/inoculum ratio ranging from 0.2 and 0.5 g of $VS_{substrate}$ per gram of $VS_{inoculum}$. The cumulated biogas production was measured until the end of the production (from 30 to 90 days) and analyzed by micro-gas chromatography. The tests were carried out in duplicate. More details about the method are available in Angelidaki *et al.*, (2009) and Hansen *et al.*, (2004). All BMP values are expressed in ml $CH_4.g^{-1}$ Volatile Solid fed (VS_{fed}). The Standard Deviation of repeatability (SDr) for BMP assays was computed as:

$$SDr = \sqrt{\frac{1}{n}\sum Variance(duplicate\ of\ each\ sample)}$$

With n the number of duplicated samples

2.3 NIR Analysis

The NIR spectra was recorded on a Fourier-transform NIR spectrophotometer (Antaris II, Thermo electron, USA) in the range of wavenumber from 10000 to 4000 cm^{-1} (equivalent to wavelength ranged from 1000 to 2500 nm) with a step of 8 cm^{-1}. For each sample, two different spectra were recorded on a rotary cup spinner (68 scans) and both absorbance spectra were averaged.

2.4. Data processing

In order to reduce the baseline variation and to enhance spectral features, the following pre-treatments have been tested: Standard Normal Variate, SNV (Barnes *et al.*, 1989), Detrend, Dt (Barnes *et al.*, 1989), and first and second derivative using the Savistsky-Golay algorithm (Savistsky and Golay, 1964) with smoothing calculated over 7 data points on both sides.

The spectra information of the entire data sample set was studied using Principal Component Analysis (PCA). Two hundred and forty three samples were selected for calibration and 53 samples were used for validation (Table 1). The validation samples were chosen after ordering the BMP values in the range of 150 to 600 ml CH_4.g-1 VS_{fed} and by selecting 1 over 5 samples. Calibration was performed by Partial Least Square (PLS) regression on transformed absorbance spectra with leave-one-out cross validation procedure. During the calibration procedure, samples were discarded as outliers for two reasons. First, because they strongly influence the model (leverage > 5 times the average level of all the samples). Secondly, if the residuals (difference between the predicted and the measured reference value) were greater than 30, 50 and 80 ml $CH_4.g^{-1}$ VS_{fed} for samples with a BMP value ranging from 0 to 100, 100 to 150 and more than 150 ml $CH_4.g^{-1}$ VS_{fed}, respectively.

The accuracy of prediction and the robustness of models were evaluated by the validation sub-set.

The quality of models was evaluated by the coefficient of determination (R²) and the Root Mean Square Error (RMSE) and the bias for predicted values:

$$R^2 = \frac{\sum(\hat{y}_i - \bar{y})^2}{\sum(y_i - \bar{y})^2} \qquad RMSE = \sqrt{\sum_{i=1}^{n}\frac{(\hat{y}_i - y_i)^2}{n}}$$

With $\hat{y}_i$, the predicted value of the sample i in the data sample set, $\bar{y}$, the average of the measured values of the data sample set, y_i, the measured value of the sample i in the data sample set and n, the number of samples in the data sample set.

The quality of predictions was also evaluated with the Ratio of Performance to Deviation (RPD = Standard deviation/RMSE) and the Relative Standard Error (RSE), calculated as the ratio between the RMSE for predicted values and the average of the validation data sub-set (expressed in %). For RPD values, five levels of prediction accuracy were considered (Saeys *et al.*, 2005). An RPD value below 1.5 indicates that the calibration is not usable. An RDP value between 1.5 and 2.0 reveals a possibility to distinguish high and low value, while an RPD value between 2.0 and 2.5 makes approximate quantitative predictions possible. For an RPD value between 2.5 and 3.0 and above 3.0, the prediction is classified as good and excellent, respectively. All transformations and calibrations were performed with The Unscrambler 9.8 software (CAMO Software AS, Norway).

TABLE 1 **Distribution of samples in the calibration and validation data sets.**

	Calibration data set	Validation data set
All	243	53
AGRO	46	11
ALG	0	1
BIO	18	2
ECROP	3	1
FAT	9	2
MEAT	10	4
MIX	2	0
MSW	51	15
PLANT	33	9
SL_{agro}	16	2
SL_{wwtp}	28	2
STAB	27	4

3 RESULTS AND DISCUSSSION

3.1 Samples characteristics

The VS content of the entire data sample set varied between 0.9 and 97.9% of total solid (TS) (Fig. 1). The BMP values varied between 0 and 1344 ml $CH_4.g^{-1}$ VS_{fed} with 75% of the values included between 196 and 376 ml $CH_4.g^{-1}$ VS_{fed}. The lowest BMP values were observed for stabilised MSW samples and wastewater sludge, due to organic matter stabilisation occurred during landfilling (Berthe *et al.*, 2008) and wastewater treatment (Dignac *et al.*, 2000). The highest BMP values were measured for agro-industrial samples, fatty waste and meat waste. The average of the BMP values for the samples included in the calibration data set and in the validation data set were similar (291 ± 179 and 317 ± 105 ml $CH_4.g^{-1}$ VS_{fed}, respectively).

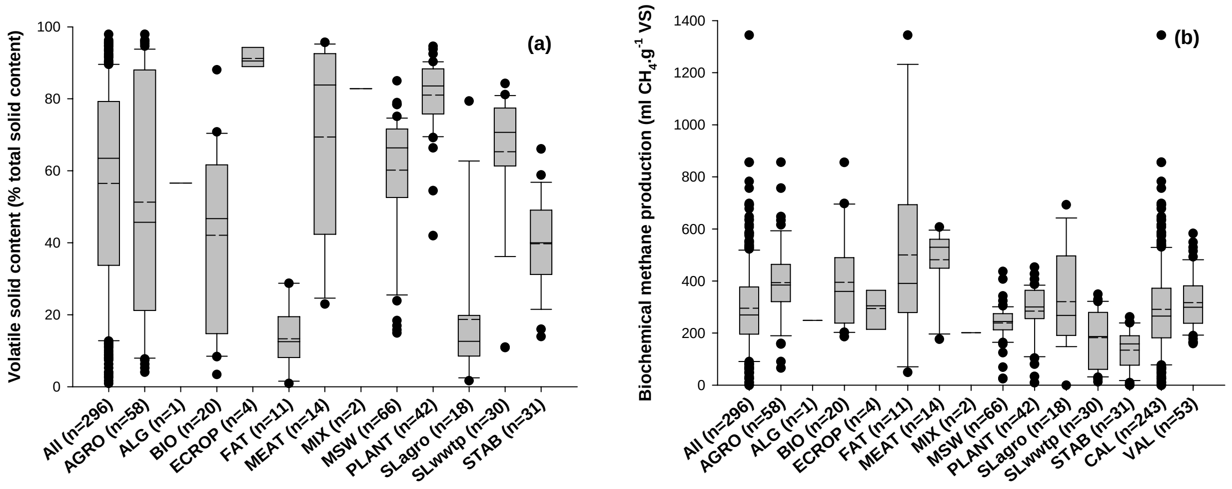

FIGURE 1 **Fig. 1. Volatile solid contents (a) and biochemical methane potential (b) of the entire data sample set separated based on the categories.**The boundary of the box closest to zero indicates the 25th percentile, the black and dashed lines within the box marks the median and the mean respectively, and the boundary of the box farthest from zero indicates the 75th percentile. The error bars above and below the box indicate the 90th and 10th percentiles. The outlying points are indicated in black.

3.2 Quality of BMP predictions by NIRS for a wide range of organic substrates

The best prediction of the BMP values for the entire data sample set was obtained for spectra transformed with SNV, Dt and second derivative procedures ($RMSE_P$ = 40 ml $CH_4.g^{-1}$ VS_{fed}, RPD = 2.61, RSE = 12.7%, Table 2, Fig. 2). Satisfactory predictions were also obtained with SNV, Dt and 1st derivative transformed spectra ($RMSE_P$ = 42 ml

$CH_4.g^{-1}$ VS_{fed}, RPD = 2.53, RSE = 13.2%). In both cases, the RPDs were between 2.5 and 3.0. Therefore, the predictions are classified as good (Sayes *et al.*, 2005).

In order to appreciate the accuracy of the elaborated model, a comparison between the errors of the predictions and the errors inherent to the experimental protocol for BMP measurement can be done. In our study, the SDr was 19 ml $CH_4.g^{-1}$ VS_{fed} for the entire data sample set and reached 34 ml $CH_4.g^{-1}$ VS_{fed} for samples with BMP values above 550 ml $CH_4.g^{-1}$ VS_{fed}. However, the SDr did not include errors due to experimental conditions (as the error of reproducibility did. The error of reproducibility is indeed estimated by performing many repetitions of BMP tests for one sample and modifying chosen parameters of the protocol (e.g. BMP assays performed by different operators and with different inoculums). The error of reproducibility was not measured in our study. It was nevertheless the case in other ones using the same BMP protocol. It was estimated at 32 ml $CH_4.g^{-1}$ VS_{fed} for ethanol (Lesteur *et al.*, 2011) and at 38 ml $CH_4.g^{-1}$ VS_{fed} for cellulose (Hansen *et al.*, 2004). Finally, the $RMSE_P$ of the NIR-model remained lower than the variability evidenced during the “17 laboratories” ring test set up by Raposo *et al.* (2011a), where the variability of the BMP values for mung beans reached 63 ml $CH_4.g^{-1}$ VS_{fed} and 78 ml $CH_4.g^{-1}$ VS_{fed} for 2 different inoculum to substrate ratios. The error of BMP predictions by NIRS was therefore in the range of the experimental error and thus confirmed the suitability of the NIR technique for a fast and accurate BMP estimation.

As already indicated by Lesteur *et al.* (2011) for MSW samples, the prediction of BMP by NIRS for heterogeneous samples appeared more accurate than most of the predictive methods based on biochemical characterization. Even in the case of similar accuracy (Triolo *et al.*, 2011), no chemical analyses are required when using the NIR method.

Increasing the diversity of samples in the dataset generally causes a reduction in the calibration accuracy (Peltre *et al.*, 2011; Stenberg *et al.*, 2004). For that reason, the accuracy of our predictions remained poorer than those obtained for mono-substrate calibrations by Lesteur *et al.* (2011) for MSW and Raju *et al.* (2011) for meadow grasses. On the other hand, due to the same higher diversity of substrates, the elaborated model presented a larger validity range for BMP values compared to mono-substrate models. In the current study, the validation data set ranged from 160 to 582 ml $CH_4.g^{-1}$ VS_{fed} whereas it ranged from 87 to 322 ml $CH_4.g^{-1}$ VS_{fed} and from 51 to 405 ml $CH_4.g^{-1}$ VS_{fed} in the study of Lesteur *et al.* (2011) and Raju *et al.* (2011), respectively. The RPD value (2.61) is moreover higher than in the mentioned studies, confirming the accuracy of the elaborated model for a fast prediction of BMP values for a wide range of organic substrates in a co-digestion context.

Finally and considering that both calibration and validation sets will be joined for using the model in routine (Dardenne, 2010), the final NIR-model will be based on 224 samples (outliers excluded). As far as we know, in the literature, no NIR-model for BMP prediction was calibrated with as many samples (reviewed in Jacobi *et al.*, 2012).

TABLE 2 **Calibration and validation statistics of different PLS prediction models. All transformations and calibrations were performed on the entire range of spectra (999-2500 nm). RMSE and RSE are expressed in ml $CH_4.g^{-1}$ VS_{fed} and %, respectively.**

	Calibration (n= 243)						Validation (n=53)			
	Out	$RMSE_C$	F	R^2_C	$RMSE_{CV}$	R^2_{CV}	$RMSE_P$	R^2_P	RPD_P	RSE
Raw spectra	79	49	5	0.79	53	0.76	62	0.64	1.70	19.7
SNV	77	41	6	0.89	46	0.87	54	0.73	1.94	17.2
SNV-Dt	76	38	6	0.89	43	0.87	57	0.71	1.86	17.9
SNV-Dt(15,2,1)	79	33	7	0.92	41	0.89	42	0.84	2.53	13.2
SNV-Dt(15,2,2)	72	36	7	0.92	42	0.86	40	0.85	2.61	12.7

Out: Outliers, c: calibration; cv: cross-validation; p: prediction; F: number of PLS factors, SNV: standard normal variate; Dt: Detrend; (23,2,1): derivatives parameters: the window length for data smoothing, the polynom degree and the derivative order respectively

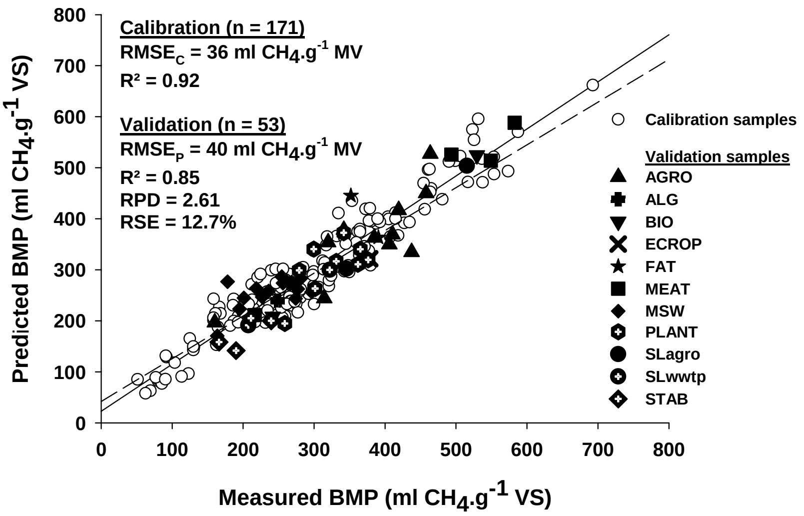

FIGURE 2 **Predicted versus measured BMP values for model calibrated on SNV-Dt(15,2,2) transformed spectra.**

3.3 Integration of substrates in the elaborated model: relation with its robustness

Nevertheless, 72 samples were discarded as outliers during the calibration procedure with the SNV-Dt(15,2,2) transformed spectra, (Table 2). It seemed difficult to integrate all the considered types of substrate in the NIR-model.

Detection of outliers is very important for the robustness and the accuracy of a PLS model. Following the above mentioned criteria for outlier determination, three types of outliers can be identified: outliers with respect to the reference value, outliers with reference to the spectra and outliers in relation to both the reference value and the spectra data (Abookasis and Workman, 2011).

TABLE 3 **Distribution of outlier samples discarded during the calibration procedure with the SNV-Dt(15,2,2) transformed spectra**

	Range of BMP value			
	All	<100	[100-550]	>550
Total	72	24	35	13
AGRO	16	2	8	6
ALG	0	0	0	0
BIO	6	0	3	3
ECROP	0	0	0	0
FAT	6	1	3	2
MEAT	2	0	2	0
MIX	1	0	1	0
MSW	6	2	4	0
PLANT	7	3	4	0
SLagro	5	1	2	2
SLwwt	11	8	3	0
STAB	12	7	5	0

More than half of the samples identified as outliers, 37, were extreme values outside the 10th and 90th percentiles of the entire data sample set (24 and 13 outliers with BMP values below 100 ml CH$_4$.g^{-1} VS$_{fed}$ and above 550 ml CH$_4$.g^{-1} VS$_{fed}$, respectively, Table 3). It represents that more than 50% of the discarded samples were extreme values. It seems difficult to build a model for the extremes of the BMP values range due to the lack of samples with BMP values located in these areas.

Another important group of outliers, 23, were included on either the wastewater sludge or the stabilized MSW category (11 and 12 outliers, respectively). The integration of this kind of samples in the model seems difficult, probably due to the high level of stability of the organic matter of the samples (Berthe et *al.*, 2008; Dignac *et al.*, 2000). Similar conclusions were also evidenced by Peltre *et al.* (2010) for the NIR prediction of an aerobic respirometric index in sludge samples. In the case of wastewater sludge, it can also be explained by the fact that the BMP measurement was not performed on freeze-dried and grinded samples, but on fresh samples. The BMP value for fresh sludge samples can indeed differ due to the removal of volatile compounds during the freeze-drying or to the modification of organic matter accessibility during grinding (Raposo *et al.*, 2011b).

Finally, 27 samples were considered as outliers without belonging to either the wastewater sludge or the stabilised MSW group and without being characterized by an extreme BMP value. It remained more difficult to explain the detection of these group of outliers that can be identified with respect to the reference value, to the spectra or both. As already indicated, the experimental measurement of the BMP can have a high level of error and can greatly differed form the theoretical methane potential calculated from elemental composition (Davidsson *et al.*, 2007; Hansen *et al.*, 2004; Raposo *et al.*, 2011a). The difference can be explained by an inhibition caused by the accumulation of intermediates, as observed by Hansen *et al.* (2004) for fat and proteins. Obtaining the theoretical BMP becomes not possible. But the NIRS is able to analyse the organic matter of the sample without the impact of the inhibitions. Therefore, samples with “suspect” measured BMP value could not be include in the calibration process. More investigations remained nevertheless necessary for the improvement of sample integration in the model and therefore for the increase of its robustness.

3.4 Practical implementation and perspectives for model improvement

The presented NIR prediction of BMP requires the preparation of the samples by freeze-drying and grinding before the acquirement of the spectra (at least 48 hours for both operations). This step is nevertheless necessary in the case of very heterogeneous samples such as MSW. Replacing the experimental BMP assay by the NIR prediction on freeze-dried and grinded samples will make possible to obtain the BMP value with a satisfying accuracy in 2 or 3 days compared to the at least 30 days required for the classical measurement.

As evidenced by the difficulty to integrate all the substrates in our model, the protocol for the use of the NIR technique in routine should include the evaluation that the new spectra is in the range of the spectra variability of the model. Practically, the spectrum of each new sample should be projected on the range of spectra variability defined by the calibration and the validation samples. If the spectrum of new sample is not included in this validity range of spectra, the BMP value of this new sample should not be estimate by the NIR technique but measured by the experimental protocol.

cEven if the elaborated model has a relatively high level of robustness, the ways of improvement are indeed the addition of new samples with extreme BMP values or samples that would increase the spectra validity range.

In order to develop to improve both the accuracy and the robustness of the BMP prediction by NIRS, local calibration procedures are an alternative to be considered (Naes *et al.*, 1990; Shenk *et al.*, 1997). In such procedures, a local algorithm operates by searching for, and selecting, samples in large databases containing spectra similar to the sample being analysed. The selected samples are then used to compute a specific calibration equation, based on PLS regression, for predicting the constituents of an unknown sample. It has been evidenced that local calibration significantly improves the quality of the prediction in the case of data sample set with a high level of variability (Barthès *et al.*, 2010; Zamora-Rojas *et al.*, 2011). The optimisation of the algorithm is performed by varying the number of samples included in the local calibration and depending to the type of substrate used for calibration (from 30 to 200). The addition of new samples in our data sample remains therefore crucial for the development of such NIR calibrations. This kind of approach appears nevertheless as a very promising step toward the development of a global method for a fast determination of the BMP for any kind of organic substrate.

Finally, the prediction of other parameters, in addition to the estimation of the BMP value, also appears as an interesting approach for using the NIR characterization method in order to elaborate well-balanced co-digestion mixture.

4 CONCLUSIONS

With an error of prediction of 40 ml $CH_4.kg^{-1}$ VS_{fed}, the prediction of the biochemical methane potential by NIRS appeared as a suitable and accurate method for a fast characterisation of substrates before their treatment in anaerobic co-digestion units.

The integration of the entire substrate diversity in the final model of the substrate remained nevertheless difficult because of the specific organic matter properties of some kind of substrates and of the high level of uncertainty that could affected the BMP values. The use of local calibration procedures would probably this integration. For that more samples will be necessary.

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Session 30

(86) OPTIMIZATION OF COW MANURE AND SEWAGE SLUDGE CO-DIGESTION USING DISINTEGRATION TECHNOLOGIES

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EXECUTIVE SUMMARY

The environmental problem of manure management and the decreasing availability of land for its disposal are forcing the development of new waste management strategies. Anaerobic digestion is an attractive treatment for cow manure, since it produces biogas, and a stabilized digestate that can be reused as organic fertilizer (Neves et al., 2008). Co-digestion of animal manure with sewage sludge offers economic and environmental benefits due to cost-sharing by processing multiple waste streams with complementary characteristics in order to improve the methane production and prevent inhibition problems due to ammonia toxicity. In this study, several pretreatment technologies such as thermal hydrolysis (Valo et al., 2004), enzymatic hydrolysis and ultrasounds, have been selected to compare them and optimize the anaerobic co-digestion of cow manure from slaughterhouse and sewage sludge by the most effective pretreatment.

Cow manure was treated with different disintegration technologies in order to compare the effects between them. Then, a mixture of thickened primary and biological sludge sampled from a municipal WWTP was mixed with cow manure according to a ratio previously defined (25% COD of the final mixture belongs to cow manure). This ratio was selected as optimum after carrying out different Biological Methane Potential (BMP) tests to three different ratios (15, 25, 50% COD). The three different disintegration technologies were also applied to the selected mixtures in order to optimize the co-digestion process, looking also for the best configuration. For the analysis control S*tandard Methods* were used and BMP tests were carried out following a previously developed protocol (Angelidaki et al., 2009). Furthermore, other physical parameters were characterized in order to assess the implementation of co-digestion mixtures and pretreatments in a continuous operation (filterability, centrifugability and rheology).

In a first phase the influence of the three pretreatments in raw cow manure was compared by BMP tests. Ultrasound was the only one that increased the methane production of raw substrate, improving it by 19%. Moreover, a higher methane production during the first days was observed for ultrasounds and thermal hydrolysis in comparison to raw substrate, what reflects that the hydrolysis rate is improved by these pretreatments. Then, each pretreatment was applied to the mixture of cow manure with primary and secondary sludge. It was observed that the methane productivity increased by all pretreatments, reaching similar values close to 500 $mLCH_4/gVS_{added}$. The effect of pretreatments was also noticed in the first days of productivity, observing a quick start-up of the production during the first five days, reaching the 75% of the final production. Although ultrasounds have the highest value for methane potential, thermal hydrolysis presents some operational and economic advantages (energy integration capacity, continuous implementation) to be selected for a further cow manure co-digestion study. In accordance with previous results, thermal hydrolysis was optimized by the evaluation of three configurations: hydrolyzed cow manure, hydrolyzed secondary sludge or hydrolyzed mixture of cow manure and secondary sludge. Hydrolyzed samples present for all configurations a higher methane potential than the raw mixture (288 $mLCH_4/gVS_{added}$). The highest increase (>30%) is obtained for the configurations on which hydrolysis is applied to secondary sludge alone and to the mixture of secondary sludge with cow manure. Other tests focused in the assessment of physical properties showed that thermal hydrolysis improves considerably the hydrodynamic properties of the samples, especially filtration and viscosity.

While pretreatments to raw cow manure have not shown improvements on biomethane potential (except for ultrasounds), co-digestion increases the production of biogas due to the complementary effect of the substrate and sludge, which is improved by the action of all pretreatments. Thermal hydrolysis advantages convert this pretreatment in the best option for a further optimization in which the next configurations, hydrolyzed mixture and hydrolyzed secondary sludge, have shown the best results for cow manure and mixed sludge co-digestion.

1. INTRODUCTION

Manure production is one of the most problematic environmental concerns of waste management in cattle farming sector. Anaerobic digestion has become a common technology to deal with this waste disposal, converting it in an energy recovery process where biogas is obtained.

1.1. Background

Many tons of animal manure are produced annually worldwide. Nearly three-quarters of the manure is from dairy and cattle, with the remainder coming from swine and poultry operations (Zhiyou et al, 2003). Currently, the disposal of manure is predominately done through land application, which causes greenhouse gas emissions, ecological system eutrophication, and groundwater contamination. (Ying et al, 2009) However, recent environmental and regulatory restrictions in the animal sector such as the decreasing availability of land for manure disposal are forcing the development of new waste management strategies.

An alternative is to change manure from a disposal problem to a bioresource for value-added products, a waste with an energy content of about 13.4 MJ/kg (Klass, 1998). Anaerobic digestion is an attractive treatment of organic wastes such as cow manure, since it produces biogas, a renewable energy source, and a stabilized digestate that can be reused as organic fertilizer (Neves et al, 2008). Moreover, the anaerobic digestion technology can also be used to control malodorous emissions (Comino et al, 2009). The importance of this technology results in considerable environmental benefits (Chynoweth, 2004) and can be an additional income source for farmers.

However, cow manure biodegradation presents some limitations. On the one hand, the high content of fiber contained in the manure limits the overall efficiency of anaerobic digestion because the degradation of recalcitrant fiber is very slow (Ying et al, 2009). In the other hand, ammonia toxicity can cause inhibition at concentrations of 3 g/L. Thus, anaerobic digestion of cow manure requires hydraulic retention times greater than 30 days (Borja et al, 1993).

Co-digestion of animal manure and other types of organic wastes offers economic and environmental benefits due to cost-sharing by processing multiple waste streams with complementary characteristics in a single facility in order to improve the methane production and prevent inhibition problems. There are several advantages to using animal manure for co-digestion: it is a good substrate for dilution of toxic wastes, it is also a source of nutrients, trace metals, vitamins and other compounds necessary for microbial growth and finally it plays a role in neutralizing pH and improving buffering capacity (Angelidaki and Ellegaard, 2005). Several studies have reported the economic and environmental benefits of co-digestion of multiple substrates, such as manure and other waste streams (Holm-Nielsen and Al Seadi, 1998)

Whereas a lot of works have been focused on the enhancement of anaerobic digestion of sewage sludge by thermal treatments (Bougrier et al., 2008; Valo et al., 2004) or ultrasounds cavitation (Donoso-Bravo, Pérez-Elvira et al. 2010), very few studies concern disintegration of manure to increase their conversion into biogas. The best results for thermal hydrolysis were obtained with temperatures in the range of 140-180 ºC where the optimum was 170ºC (Yoneyama et al., 2006). This value is in the range of optimal temperature generally reported for thermal hydrolysis of sewage sludge (Bougrier et al., 2008).

Among other technologies, enzymatic hydrolysis has been tested with several substrates such as corn stover (Kaar and Holtzapple, 1998; Varga et al., 2003), wheat straw (Cacchio et al., 2001), rice hull (Sharma et al., 2001), fruit pomace (Avelino et al., 1997) and sugarcane bagasse (Zheng et al., 2002) but no data has been found on hydrolyzing manure (Zhiyou et al, 2003). Enzymatic hydrolysis presents advantages such as a high yield of glucose production, low environmental impact, and mild reaction conditions.

1.2. Research objectives

In this study, several pretreatment technologies such as thermal hydrolysis, enzymatic hydrolysis and ultrasounds, have been selected to compare them and optimize the anaerobic co-digestion of cow manure from slaughterhouse and sewage sludge.

2. METHODOLOGY

2.1. Materials

Thickened primary (SS1) and biological sludges (SS2) were sampled from a municipal WWTP every week and kept refrigerated at 4ºC. Cow manure (CM) comes from slaughterhouse. The characterization of the substrates included Total Chemical Oxygen Demand (CODt), Soluble Chemical Oxygen Demand (CODs), Total Solids (TS), Volatile Solids (VS), Total Kjeldahl Nitrogen, (TKN), Ammonium, (NH4+) and Total Phosphorus, (P). Values are summarized in *Table 1*:

TABLE 1. Substrates characterization

Parameter	**Units**	**Cow Manure**	**Sludge**	
			SS1	**SS2**
CODt	g/kg	258.8	188.2	83.9
CODs	g/kg	-	10.3	6.3
TS	g/kg	221.6	167.5	71.2
VS	g/kg	208.5	115.6	54.9
TKN	N-g/ kg	0.25	4.69	5.75
NH_4^+	N-g/ kg	-	0.29	0.24
Grease	g/kg	18.3	-	-
P	P-mg/ kg	71.,9	2.5	29.2

Thickened primary (SS1) and biological sludges (SS2) were mixed in a 1:1 weight ratio (according to the common ratio in the WWTP) and then cow manure was added in a proper ratio. These mixtures have been previously studied in a first phase considering different Chemical Oxygen Demand (COD) ratios: 15%, 25% and 50%. The selected optimum ratio, after carrying out different Biological Methane Potential (BMP) tests, was 25%COD (expressed as CODmanure/CODmixture). It was identified a decrease in the methane production for this ratio in comparison with the one obtained for mixtures with lower ratios, possibly due to ammonium inhibition effect. Therefore the assessment of the application of pretreatments to this type of mixture is a challenge in order to know if it can overcome this limitation. Previous studies showed that other pretreatments can avoid this kind of inhibition (Chen et al., 2008). The highest COD percentage (50%) exceeds the cow manure limit weight percentage (15%), so it has not been considered.

2.2. Experimental procedures

Different disintegration technologies (thermal hydrolysis, enzymatic hydrolysis and ultrasounds) and several configurations (pretreatments applied to the cow manure, to the secondary sludge alone or to the cow manure-secondary sludge mixture) were studied. The pretreatments were selected based on COD solubilisation, increase in biogas production, pathogen reduction and dewaterability, as well as the availability to carry out the test at lab-scale. A brief description of the equipments to carry out these pretreatments is enclosed:

- *Thermal Hydrolysis:* The hydrolysis plant was equipped with 2L reactor where the hydrolysis of the substrate/sludge takes place for 30 minutes, which is fed by an 8 bar steam. It is connected to a flash tank where the steam explosion is carried out, and the final product is obtained (*Figure 1*). The use of steam is an advantage for this treatment due to possibilities to be implemented in a full scale plant for energy recovery and a continuous operation; it also provides better hydrodynamic properties to the final mixture.
- *Cavitation by ultrasounds:* The ultrasound set-up is shown in Figure 2 (This equipment converts electrical energy in mechanical vibrations, which are transmitted to the sample by a sonotrode).
- *Enzymatic treatment:* The substrate, the enzymatic solution and the buffer solution (for a pH control) are introduced in 300 mL closed bottles and stirred at a desire temperature (37ºC) and pH (5.3). The process is carried out by the addition of a commercial protease (from *Aspergillus oryzae*) which assures a high activity. The requirements of a continuous control and the high costs are some of the drawbacks to use it.

FIGURE 1. Thermal hydrolysis plant.

FIGURE 2. Ultrasound set-up equipment

In a first phase, each pretreatment was applied to cow manure in order to compare the effects between each treatment and biogas production in BMP tests. In a second phase, the cow manure was pretreated with secondary sludge and then mixed with primary sludge following the ratios previously defined in order to compare the effect of co-digestion and to select the best pretreatment. Primary and biological sludges were used to make the final mixture and reproduce real conditions, but only biological sludge was pretreated in all cases (the SS1 is mixed in before digestion). Biological sludge is composed mainly of biomass and hardly degradable carbohydrates added to the sludge particles. It also contains easily degradable proteins only available with a break of the cellular wall. Pretreatments break these cells and help to the biodegradability of biological sludge. (Pérez-Elvira S.I. et al, 2006). For this reason, pretreatments are only applied to biological sludge.

In a third phase, an optimization of the co-digestion using the selected pretreatments was carried out. In this case thermal hydrolysis was applied to three different configurations of co-digestion with cow manure, primary and biological sludge: hydrolyzed cow manure, hydrolyzed secondary sludge or hydrolyzed mixture of cow manure and secondary sludge. In each case, the pretreated sample was then properly mixed with primary sludge to obtain the same mixing ratio than in previous assays.

2.3. Analytical methods

2.3.1. Characterization of samples

Standard methods (Apha, 2005) for substrates characterization were applied to determine the next parameters: TS/VS total and volatile solids; COD chemical oxygen demand; TKN total Kjendhal nitrogen; NH_4^+ ammonium; VFA volatile fatty acids; grease; phosphorus.

2.3.2. Biochemical methane potential assays

The Biological Methane Potential (BMP) assays were performed following a protocol based on standardized assays for research purposes (Angelidaki and Ellegaard, 2005; Angelidaki et al., 2009). The vials volume was 300 mL and a substrate-inoculum ratio of 1:1 in terms of VS was applied. The incubation temperature was 35ºC. The inoculum was WWTP mesophilic digested sludge (after a pre-incubation period of 2 days at 35ºC). Periodical monitoring analyses of biogas production by pressure meter and biogas composition by gas chromatography were performed during the tests. Methane potential or BMP was expressed as the net volume of methane per g of initial substrate VS content.

2.3.3. Assessment of physical properties

To assess the implementation of co-digestion mixtures and pretreatments in an industrial plant, other tests such as filterability, centrifugability and rheology tests were performed to study the hydrodynamic characteristics of the pretreated mixtures. These tests were very relevant in terms of assessing the impact on mixing requirements, digestate dewaterability and handling properties. The measured parameters were: Filtration Constant (FC), Capillary Suction Time, Centrifugability and Rheology (by assessing viscosity curves) (Apha, 2005).

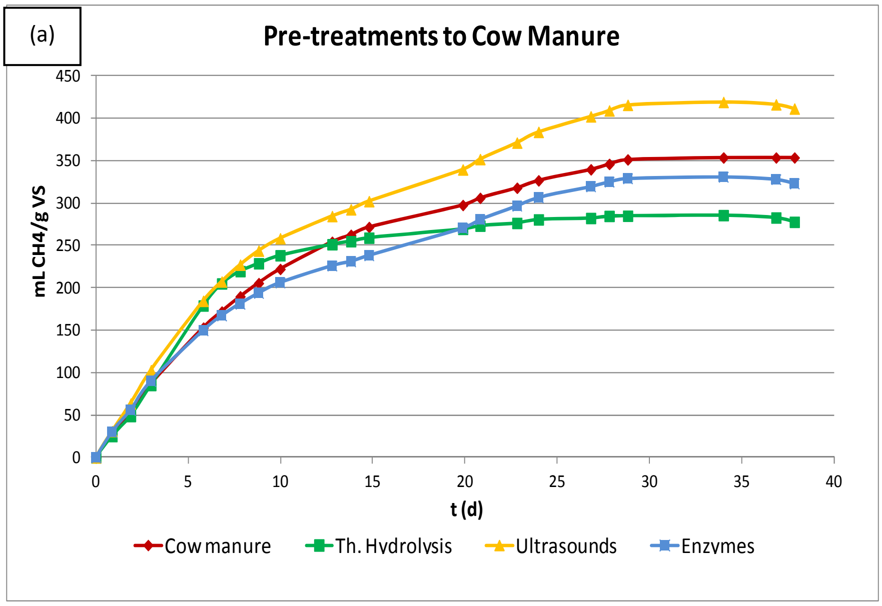
3. RESULTS AND DISCUSSION

3.1. Phase 1: Pretreatments to cow manure

In a first phase the influence of the three pretreatments applied to the raw cow manure was compared. *Figure 3a* shows the methane production evolution for the cow manure alone and after the application of each pretreatment. Results indicate that ultrasound pretreatment is the only one that increases the methane production of raw substrate. Moreover, it can be observed that the rise in the biogas production is higher during the first seven days, where ultrasounds and thermal hydrolysis presented a higher biomethane production increase. This means there is a first stage in cow manure degradation that could be improved by the application of these pretreatments. Additionally, ultrasounds are the only one that provides a higher methane potential respect the non-pretreated cow manure with an overall increase of 19%. Percentage removal showed a wide range of values: while thermal hydrolysis has the highest values for volatile and total solids (50% and 28% respectively), ultrasounds present better results for COD removal (62%), which are acceptable values in both cases.

3.2. Phase 2: Pretreatments and co-digestion of mixtures

Then, each pretreatment is applied to a mixture of cow manure and secondary sludge (then mixed with non-pretreated primary sludge for co-digestion) according to the ratios described in the methodology. The methane potentials are shown in *Figure 3b* for each substrate and for the pretreated mixtures. The biodegradability tests (*Figure 3b*) showed that co-digestion increases the productivity in all pretreatments respect to the non-pretreated one, this can be due to the adjustment of nutrients and the increased content of easily degradable matter (Pérez-Elvira S.I. et al, 2006). Ultrasounds are the best treatment, followed closely by thermal hydrolysis and enzymatic. In these cases, the organic matter removal is lower than before and very similar for all the samples with values between 20-23% for VS and 30-40% for COD. So, it can be said that the small fraction of matter that has been removed presents a good biodegradation yield. Disintegration factors have also been determined as the increase of the ratio *soluble COD/total COD* before and after pretreatments. In general showed high values for all the pretreatments (106% thermal hydrolysis, 188% enzymatic hydrolysis, 78% ultrasounds) which means that a high solubility of the organic matter takes place in all of them, especially in the enzymatic pretreatment. It can explain the high production rate that takes place during the first five days (reaching about 75 % of the total methane production) of the tests in the case of pretreated samples, since the hydrolysis step is not any more a limiting step due to the high COD solubilization.



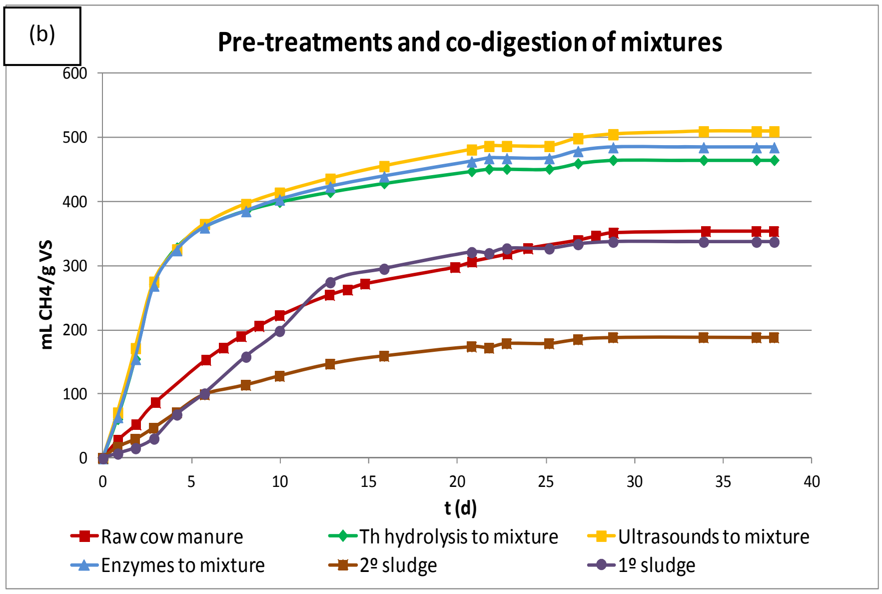


FIGURE 3. Methane production per unit of VS added during batch tests: (a) Phase 1 and (b) Phase 2.

Relating both phases (*Table 2*) it can be observed that co-digestion assays presented a better effect by the action of pretreatments. Although ultrasounds have the highest values for methane potential, thermal hydrolysis presents some operational and economical advantages, such as high energy-efficiency, foaming problems reduction, better sludge dewaterability, guarantee of pathogen removal (Pérez-Elvira S.I. et al, 2006), odours emission control. Therefore, it offers more possibilities to be the best pretreatment option for a further cow manure co-digestion implementation.

TABLE 2. Final methane productivities in batch tests from phases 1 and 2, and % of increase.

	COW MANURE		CO-DIGESTION	
	mL CH_4/g VS	%	mL CH_4/g VS	%
Raw	346	-	304	
Th. Hydrolysis	278	-20	465	53
Ultrasounds	411	19	510	68
Enzimes	323	-7	485	60

3.3. Phase 3: Thermal hydrolysis and co-digestion of mixtures by different configurations

According to previous results it was decided to optimize the thermal hydrolysis pretreatment for a co-digestion of cow manure and mixed sludge. Three configurations have been tested in order to identify which is the best option to optimize the process. Biodegradability results of co-digested mixtures, shown in *Figure 4*, indicate that all the configurations of thermal hydrolysis pretreatment lead to an improvement in terms of methane production regarding the non-pretreated mixture, of at least 16%. The most promising mixtures are the ones applied to the secondary sludge or to the mixture of the secondary sludge with cow manure, with increases of 30% of the final methane production. Even though hydrolyzed cow manure presents a significant increase in the first seven days the final biogas production is lower than the other ones measured. Removal results were similar in all configurations for both VS (40-43%) and COD (~46%).

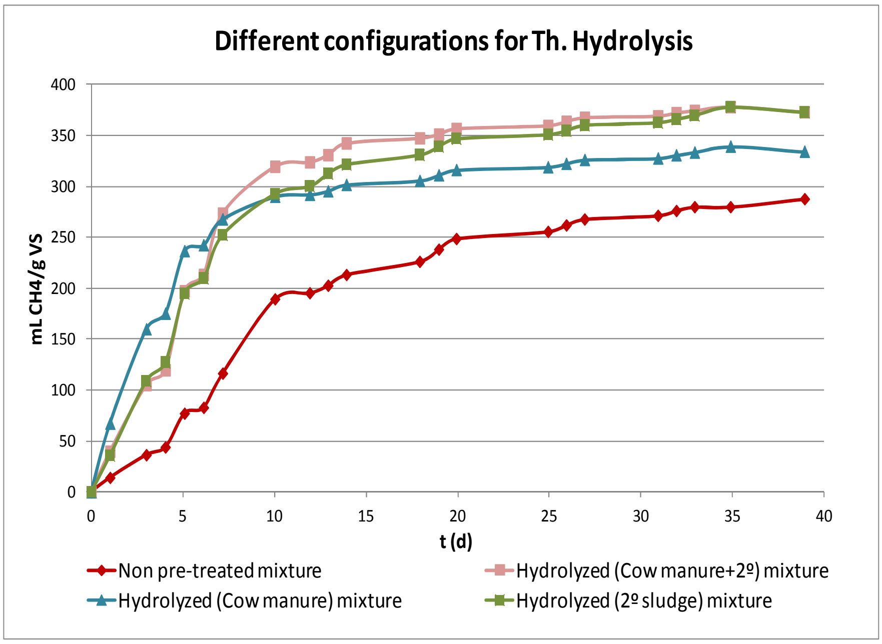


FIGURE 4. Methane production per unit of VS added during batch tests: Phase 3

The hydrodynamic characteristics of the hydrolyzed mixtures indicated that thermal hydrolysis improves the filterability, capillarity (higher FC and lower CST) and viscosity (graphic not shown), specially in the hydrolyzed cow manure mixture (FC=0.028cm^2/s; CST=112s versus FC=0.0021cm^2/s; CST=5222s of non-pretreated sample). Meanwhile, the mixture without pretreatment presents good results for centrifugability: good separation of liquid and solid phases (70% separated liquid and 100% solid recovery in cake) and higher solid concentration in cake, (25%TS). These results indicate that hydrolyzed mixtures have better hydrodynamic properties, since mixing properties, dewaterability, and viscosity of sludge become more favourable for a continuous operation.

4. CONCLUSIONS

Pretreatments applied to raw cow manure have a negative effect for thermal hydrolysis and enzymatic pretreatment concerning its biodegradation; meanwhile ultrasounds have a positive effect with an increase of 19% in the methane production. Co-digestion increases the production of biogas for all pretreatments, thanks to the complementary effect of the substrate and sludge that turn the substrate in a good and easily biodegradable mixture. This increase is improved by the action of the pretreatments, providing a faster hydrolysis process. Thermal hydrolysis presents high-quality results concerning methane potential, and the advantages in terms of operational digestate post-management and digester mixing that this method offers turn it in the best pretreatment for cow manure. Optimization of thermal hydrolysis results in an equal biogas production between hydrolyzed mixture and hydrolyzed secondary sludge (30% higher than non-hydrolyzed one). The assessment of physical parameters indicates that hydrolyzed cow manure presents the best results in terms of hydrodynamic properties, but in all cases those of the hydrolyzed mixtures are improved, regarding the non-pretreated one, in view of a continuous process. Therefore, co-digestion of cow manure and sewage sludge presents a potential application in a real scale, provides an increase of the sludge biogas production and a potential improvement of the process by using thermal hydrolysis pretreatment,

5. ACKNOWLEDGEMENTS

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(112) KINETIC STUDY OF THE MESOPHILIC AND THERMOPHILIC SEWAGE SLUDGE ANAEROBIC DIGESTION

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EXECUTIVE SUMMARY

Wastewater treatment plants generate sewage sludge as a by-product of the physical, chemical and biological processes used during the wastewater treatment. In these plants, anaerobic digestion has been used, for more than a century, to stabilize the sewage sludge produced. At the present time, most anaerobic digesters are operated at mesophilic conditions. However, the need to improve the process feasibility, by means of increasing the biogas yield and reducing the sludge disposal costs, has increased the interest in thermophilic conditions. In this topic, the thermophilic anaerobic digestion offers some potential advantages over conventional mesophilic anaerobic digestion: (i) an increase of the biological and chemical reaction rates, (ii) an increase of the organic matter removal, (iii) a higher solubilization of the particulate organic matter and (iv) an enhancement of the pathogenic organism destruction. However, its industrial implementation have been limited by some drawbacks that are unavoidable: (i) an elevated energy requirement for heating the digester, (ii) a higher risk of process destabilization, (iii) a poorer sludge dewaterability and (iv) a higher odour potential. The aim of this study was to improve the knowledge of the metabolic pathways involved in the mesophilic and thermophilic sewage sludge anaerobic digestion. To achieve this goal, the volatile fatty acids profile in a continuous stirred tank reactor was evaluated during 48 hours after the feeding. , moreover, the performance of mesophilic and thermophilic sludge digestion was compared to give an overview of the process. In the present study, two jacketed semi-continuous stirred tank reactors with a working volume of 2.5-L were used. The operational temperature of the mesophilic digester (37ºC) and the thermophilic digester (55ºC) were ensured by circulating water from a heated water bath, while the digesters medium was continuously mechanically-stirred at 70 r.p.m. The hydraulic retention time of the mesophilic digester was set at 12 and 9 days, whereas the hydraulic retention time of the thermophilic digester was fixed at 9 and 6 days. The digesters were, once a day, manually fed and purged; where the raw sewage sludge, used as food supply, was a mixture of primary sludge (60% in weight basis) and waste activated sludge (40% in weigh basis) adjusted, with deionised water, to obtain a solid concentration of 30 g TS L^{-1}. The results of the study showed the influence of the temperature conditions and hydraulic retention time in process performance. Those results were further understood when the volatile fatty acids profile (analysed at 0, 1, 3, 6, 12, 24, 28, 32 and 48 hours after feeding) was superimposed with the biogas flow rate profile. With regard to the results obtained, the nearly constant specific biogas yields and removal efficiencies, for both temperature conditions, when the hydraulic retention time was decreased; together with the no accumulation of volatile fatty acids, suggested that the anaerobic digestion process was limited by disintegration-hydrolysis step. Furthermore, thermophilic digested sludge exhibited poorer dewaterability properties than mesophilic digested sludge. Finally, the volatile fatty acids behaviour showed clearly differences between mesophilic and thermophilic anaerobic digestion. Mesophilic digestion presented low acid concentrations, with acetate as the predominant acid; in contrast, thermophilic digesters showed higher levels of volatile fatty acids prevailing propionate and i-valerate over the others. Despite these differences, a clear correlation between total volatile fatty acids evolution and biogas flow rate could be observed.

1 INTRODUCTION

Wastewater treatment plants generate sewage sludge (SS) as a by-product of the physical, chemical and biological processes used during the wastewater treatment. In these plants, anaerobic digestion has been used, for more than a century, to stabilize the SS produced. At the present time, most anaerobic digesters are operated at mesophilic conditions. However, the need to improve the process feasibility, by means of increasing the biogas yield and reducing the sludge disposal costs, has increased the interest in thermophilic conditions.

Thermophilic anaerobic digestion (TAD) offers some potential advantages over conventional mesophilic anaerobic digestion (MAD): (i) an increase of the biological and chemical reaction rates, (ii) an increase of the organic matter removal, (iii) a higher solubilisation of the particulate organic matter and (iv) an enhancement of the pathogenic organism destruction. However, its industrial implementation have been limited by some drawbacks that are unavoidable: (i) an elevated energy requirement for heating the digester, (ii) a higher risk of process destabilization, (iii) a poorer sludge dewaterability and (iv) a higher odour potential. Volatile fatty acids, generated during the anaerobic digestion process, are know to be the most important intermediates since they are the principal precursors of the methane production as well as the majors indicators of process imbalance. The identification and the evolution of the VFA in the course of time can improve the knowledge of the metabolic pathways involved in the AD process, specifically between acid consumer microorganisms and acid producer bacteria. In this topic, several authors have studied the evolution and relationship of the VFA in batch and/or continuous digesters either after feeding the digester or after feeding a VFA solution. The aim of the present study was to evaluate the VFA profile in a continuous stirred tank reactor during 48 hours after the feeding as well as the biogas production rate. Organic matter removal efficiency and effluent sludge dewaterability were also analysed and discussed.

2 METHODOLOGY

2.1 Anaerobic digestion set-up

Two identical jacketed semi-continuous stirred tank reactors (S-CSTR) with a working volume of 2.5-L were used in this study. All systems were equipped with an on-line biogas measuring device (Ritter MGC-1). The operational temperature of the MAD (37 ºC) and the TAD (55 ºC) were ensured by circulating water from a heated water bath (Haake DC40). The hydraulic retention time (HRT) of the MAD was set at 12 and 9 days, while the HRT of the TAD was fixed at 9 and 6 days. The digesters were, once a day, manually fed and purged. The raw sewage sludge (SS), used as food supply, was a mixture of primary sludge (PS - 60% in weight basis) and waste activated sludge (WAS- 40% in weigh basis) adjusted to obtain a solid concentration of 30 g TS L^{-1}. Both PS and WAS were obtained from a municipal WWTP of the Barcelona metropolitan area (Spain). In the plant, the PS and the WAS are separately centrifuged (sampling point) and mixed before its mesophilic AD treatment. After collection, the samples were properly mixed and stored at 4 ºC until its utilization. Table 1 summarizes the characteristics of the mixture of PS and WAS used as food supply during the study.

Table 1. Characterisation of both raw sewage sludge

Influent characterization

	Units	
TS	g L^{-1}	31.3
VS	g L^{-1}	22.9
COD_T	g O_2 L^{-1}	41.5
pH	-	6.4
Total Alk.	mg $CaCO_3$ L^{-1}	2,231
VFA	mg L^{-1}	1,814
- Acetic acid	mg L^{-1}	916
- Propionic acid	mg L^{-1}	377
- i-Butyric acid	mg L^{-1}	65
- n-Butyric acid	mg L^{-1}	276
- i-Valeric acid	mg L^{-1}	116
- n-Valeric acid	mg L^{-1}	65

2.2 Analytical procedure and methods

The digesters performance was continuously monitored by pH, biogas production and periodical analysis of total alkalinity. Once constant values of these parameters were reached, steady-state conditions were considered to be achieved. At stationary conditions, analyzes to characterize influent and effluent were carried out for all the HRTs evaluated. Total solids (TS), volatile solids (VS), total chemical oxygen demand (COD_T) were determined following the guidelines given by the standard methods 2540G and 5220D respectively (APHA, 2005); where the losses of VFA compounds during the solids determination were taken into account and then combined to give a final TS and VS value (Kreuger et al., 2011). Alkalinity and volatile fatty acids (VFA) were used as indicators of process stability.

Total (TA) alkalinity was determined by titration method (2320B-APHA, 2005), while individual VFA (acetate, propionate, i-butyrate, n-butyrate, i-valerate and n-valerate) were analyzed by a HP 5890-Serie II chromatograph equipped with a capillary column (NukolTM) and flame ionization detector. The samples used for VFA analysis were previously acidified, centrifuged and filtered through a 0.45 μm nitrocellulose filter. The capillary suction time (CST), used to determine the digestate dewaterability, was determined with a Tritron Electronics Ltd. 304M CST analyser.

3 RESULTS AND DISCUSSION

3.1 Anaerobic digestion performance

The start-up of both digesters (MAD and TAD) was carried out with mesophilic digested sludge from the WWTP. Hence, the MAD obtained directly the anaerobic consortia from the collected inoculum; whereas the thermophilic biomass was adapted through a single step temperature change, from mesophilic to thermophilic conditions (Bouskova et al., 2005). To assure the steady-state conditions for each HRT, i.e. constant pH, VFA concentrations and biogas production rates, a lag time of 30 – 40 days was waited before the digesters evaluation. Digesters evaluation was carried out for about 60 days. Moreover, it could be assured that the reported values were obtained under stable conditions since the volatile fatty acids-to-total alkalinity ratio were, for all the HRTs, lower than 0.01 and 0.25 for MAD and TAD respectively; values below the reported inhibiting conditions (EPA 1979, Balaguer et al., 1992, Callaghan et al., 2002).

Table 2. Characterization of mesophilic and thermophilic effluent

	Units	Mesophilic		Thermophilic	
Operational conditions					
Temperature	ºC	37		55	
HRT	days	12	9	9	6
OLR	g VS L_R^{-1} day^{-1}	2.0	2.6	2.6	3.6
Effluent characterization					
TS	g L^{-1}	23.8	24.2	24.6	22.8
VS	g L^{-1}	15.2	15.5	15.4	14.7
COD_T	g O_2 L^{-1}	26.1	27.4	28.5	29.7
pH	-	7.4	7.4	7.6	7.6
Total Alk.	mg $CaCO_3$ L^{-1}	3944	4177	4315	4205
VFA	mg L^{-1}	22.0	23.1	1057	603
- **Acetic acid**	mg L^{-1}	22.0	23.1	103	84
- **Propionic acid**	mg L^{-1}	n.d	n.d	660	195
- **i-Butyric acid**	mg L^{-1}	n.d	n.d	12	19
- **n-Butyric acid**	mg L^{-1}	n.d	n.d	n.d	n.d
- **i-Valeric acid**	mg L^{-1}	n.d	n.d	282	302
- **n-Valeric acid**	mg L^{-1}	n.d	n.d	n.d	n.d
CST	s	61.0	69.0	127.5	139.2
Removal efficiency					
$VS_{removal}$	%	35.4	34.0	34.3	32.7
$COD_{removal}$	%	35.9	34.4	30.2	29.1
Biogas characteristics					
Biogas production	L_{biogas} day^{-1}	1.55	2.03	1.91	2.61
PEB-V_R	L_{biogas} L_R^{-1} day^{-1}	0.62	0.81	0.76	0.88
PEB-SV_{added}	L_{biogas} g VS_{added}^{-1} day^{-1}	0.32	0.31	0.29	0.29
PEB-COD_{added}	L_{biogas} g COD_{added}^{-1} day^{-1}	0.18	0.18	0.17	0.15

Table 2 shows the effluent characterization, the biogas production and the process efficiency of the MAD and the TAD for each studied HRT. As can be seen, the biogas production of the MAD increased as the HRT was shortened and therefore, the OLR increased. However, the specific biogas yield remained practically constant with an average value of 0.31 L_{biogas} VS_{added}^{-1} day^{-1}. The obtained specific biogas production it is in agreement with the literature, which has reported specific productions from 0.20 to 0.55 L_{biogas} VS_{added}^{-1} day^{-1} (Roberts et al., 1999, Benabdallah et al. 2007, Coelho et al., 2011, Bolzonella et al., 2012, Duan et al., 2012).

The biogas production of TAD, as happened with MAD, increased as the HRT decreased and the OLR augmented. Nevertheless, the specific biogas yield presented an average value of 0.29 L_{biogas} VS_{added}^{-1} day^{-1}. Once again, these specific biogas yields concur with the obtained in previous studies, that have reported productions from 0.15 to 0.50 L_{biogas} VS_{added}^{-1} day^{-1} (Bouskova et al., 2005, Bolzonella et al., 2009, Ferrer et al., 2010, Nges et al., 2010, Coelho et al., 2011). The organic matter removal efficiency, shown by the VS and COD_T degradation (see Table 2), was slightly decreased when the HRT was shortened for both operating temperatures. Concretely, for MAD and TAD, the decrease was only about 1.5 percentage-points. These results and the no accumulation of VFA suggested that the AD process, for both temperature conditions, was limited by disintegration-hydrolysis step. Therefore, the soluble organic compounds end up yielding methane avoiding an organic overload. It should be pointed that, the methane content in the biogas may have fluctuated among the HRT, and therefore the removal efficiencies obtained would match with the specific methane yield more than with the specific biogas yield. Finally, sludge dewaterability seems to be clearly affected by the digestion operating temperature. As expected, thermophilic digested sludge exhibited, indicated by a higher CST value, poorer dewaterability than mesophilic digested sludge, which is in accordance with previous studies (Zhou et al., 2002, Riau et al., 2010, Coelho et al., 2011). Zhou et al. (2002) reported that thermophilic effluents present higher amount of soluble substances, such as extracellular proteins, that difficult sludge dewaterability.

3.2 Behaviour of volatile fatty acids

At stationary conditions, the VFA concentration was below 25 mg L^{-1} for all the MAD HRTs, where acetate represented the main compound. In contrast, TAD presented higher VFA, which ranged from 600 mg L^{-1} to 1100 mg L^{-1}, being the propionate and i-valerate the predominant acids. The higher levels of VFA at TAD is due to low substrate affinity of some thermophilic organisms (Kim et al., 2002, Song et al. 2004) and higher sensitivity of thermophilic biomass to inhibitory compounds (Gavala et al., 2003). It is important to notice that, contrary to expected, the VFA level for TAD was lower for 6-day HRT than for 9-day HRT. Specifically, the propionate decreased a 70% after the 6-day HRT volatile fatty acids evolution (before performance analyzing). Higher levels of propionate have been found under higher hydrogen partial pressures (Ozturk, 1991, Kim et al., 2002, Song et al., 2004); this fact suggests that the hydrogen partial pressure decreased during the experimentation, fostered by methanogenic hydrogen consumption, enhancing the activity of propionate-degraders which led to lower propionate concentrations. Regarding to the evolution of the VFA, figures 1 and 3 show, for all the HRT, the profile of total volatile fatty acids (TVFA- expressed as the sum of acetate, propionate, i-butyrate, n-butyrate, i-valerate and n-valerate) during the mesophilic and thermophilic digestion combined with the biogas flow rate. The evolution of the volatile fatty acids concentration was carried out during 48 hours, digester medium samples were taken 0, 1, 3, 6, 12, 24, 28, 32 and 48 hours after the feeding.

Table 3. Reactions for fatty acid oxidizing organisms (Batstone et al., 2003)

VFA	Reaction	
Acetate	$CH_2COOH \rightarrow CH_4 + CO_2$	Eq.1
Propionate	$CH_3CH_2COOH + 2H_2O \rightarrow CH_3COOH + CO_2 + 3H_2$	Eq.2
i-Butyrate	$CH_3(CHCH_3)COOH + 2H_2O \rightarrow 2CH_3COOH + 2H_2$	Eq.3
n-Butyrate	$CH_3CH_2CH_2COOH + 2H_2O \rightarrow 2CH_3COOH + 2H_2$	Eq.4
i-Valerate	$CH_3(CHCH_3)CH_2COOH + 2H_2O + CO_2 \rightarrow 3CH_3COOH + H_2$	Eq.5
n-Valerate	$CH_3CH_2CH_2CH_2COOH + 2H_2O \rightarrow CH_3COOH + CH_3CH_2COOH + 2H_2$	Eq.6

3.2.1 Mesophilic anaerobic digester

For MAD, the TVFA profile did not differed quite much from the acetate profile, since the acetate was the main acid, as the other acids were not normally found after few hours; only low concentrations of i-butyrate were detected after 48 hours at 9-day HRT (13 mg L^{-1}). So, although the acetate concentrations between HRT were different, the same trend was observed. The main pathway to remove the acetate from the system is via aceticlastic methanogenesis (see equation 1) while it is generated by several routes, mainly: sugars, amino acids, and fatty acids degradation (see equations 2-6). Firstly, the acetate concentration increased during the first hour, when the peak concentration was reached; during this period, the amount of acetate consumed by the aceticlastic methanogens was lower than the amount generated by acetate producer bacteria. Moreover, this increase was more pronounced as the HRT was shortened probably due to the more substrate available.

Secondly, the concentration of acetate decreased until it reached a plateau, at 12 hour for 12-day HRT and about 20 hour for 9-day HRT; which mean that the amount of acetate generated was less than the consumed, as a result of the biodegradable organic matter exhaustion. Finally, the acetate concentration remained constant until the 48 hours, suggesting that equilibrium between the acetate produced and consumed was achieved. Also, it can be observed a correlation between the flow rate and the TVFA profile (see Figure 1), and therefore the acetate profile. As can be observe, the profile of the TVFA concentration matches the evolution of the biogas flow rate for all the HRT evaluated. Generally, during the first hours the reduction of the TVFA concentration led to a lower biogas flow rate; later on, when the TVFA reached the plateau a constant and lower biogas flow rate was observed. This concordance makes sense as acetate, the main VFA, is known to be the principal methane precursor (McCarthy and Smith 1986, Wang et al. 1999).

Regarding the MAD, the TVFA profile did not differed quite much from the acetate profile, since the acetate was the main acid, as the other acids were not normally found after few hours; only low concentrations of i-butyrate were detected after 48 hours at 9-day HRT (13 mg L^{-1}). So, although the acetate concentrations between HRT were different, the same trend was observed.

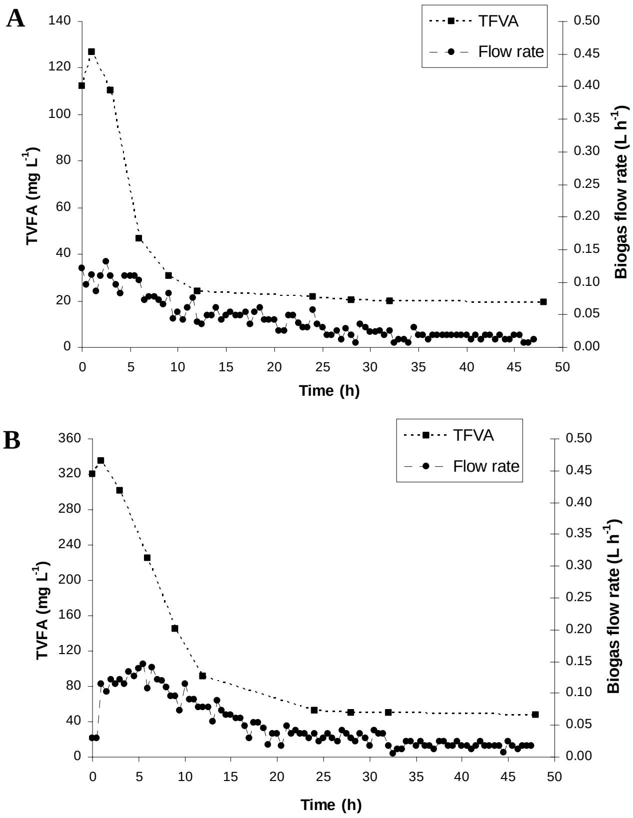

Figure 1. TFVA and biogas flow rate profile for: (A) MAD 12-day HRT, (B) MAD 9-day HRT.

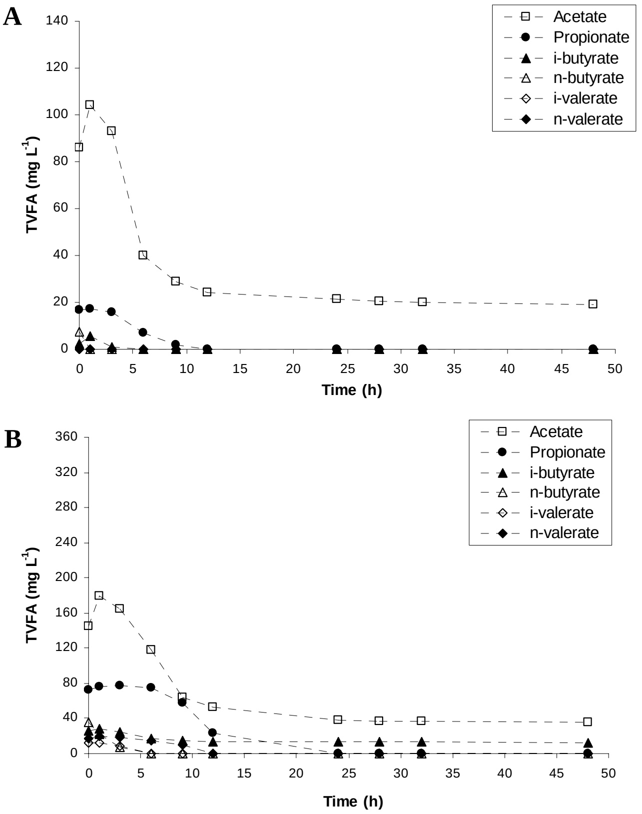

Figure 2. Individual VFA profile for: (A) MAD 12-day HRT, (B) MAD 9-day HRT.

3.2.2 Thermophilic anaerobic digester

As aforementioned, in TAD, predominant acids were propionate and i-valerate. However, the TVFA profile was more similar with the acetate profile than the profile showed by the main acids; even though some differences were found. As happened with the MAD the maximum acetate concentration increased as the HRT was decreased, probably due to the higher organic loading rate (Song et al., 2004). Normally, the maximum acetate concentration was reached three hours after the feeding with acetate concentrations 30% higher than the initial ones (0 hours). Then, the acetate concentration showed a rapid decline until 12 hours. From then on, the acetate concentration decreased slightly to achieve a constant level at approximately 28 hours that remained stable up to the end of the cycle. These steps match as the aforementioned for MAD, since biogas production was observed from the beginning. The propionate concentration increased after the feeding, reaching a maximum concentration at 10 hours for 9-days HRT and 3 hours for 6-days HRT. Afterwards, the concentration started to decrease moderately never reaching a constant concentration during the 48 hours.

The slow dynamics of propionate could be due the low metabolic activity and high sensitivity of propionate-degrader bacteria (Ozturk, 1991, Pind et al. 2002). The literature also reported product inhibition of propionate-degraders by acetate and hydrogen (Wang et al., 1999, Pind et al., 2002), which can affect negatively to the degradation rate. However, in this case the inhibition did not seem a plausible explanation, since the acetate inhibiting concentration were found to be more than 1,400 mg L^{-1} (Wang et al. 1999), much higher than the concentrations of acetate during this study. The degradation of both butyrate isomers led to acetate as product (see eq.3-4) by the same organisms (Batstone et al., 2003), however they showed the different trends. n-butyrate concentration gradually decreased until was not detected at 9 hours after the feeding for 9-day HRT and 24 hours for 6-day HRT. In contrast, i-butyrate concentration increased until 3-6 hours after feeding, decreasing to constant level about 10 mg L^{-1} at 9-day HRT and 20 mg L^{-1} at 6-day HRT. These could be due various reasons: (1) the structural differences between two forms, which could imply different degradation rates, where the degradation rate of n-butyrate was higher than i-butyrate (Wang et al., 1999, Şenturk et al., 2010). (2) The reciprocal isomerisation between the two forms; although Wang et al. (1999) observed that the isomerisation rate of n-butyrate to i-butyrate was greater than vice versa. Valerate isomers are degraded throught different pathways, n-valerate can be degraded via β-oxidation (see eq. 6) while i-valerate cannot (see eq. 5) (Batstone et al., 2003). Both compounds showed the same tendency, even though the concentration of i-valerate was notably higher. To be specific, n-valerate concentration increased during the first hours after the fed. Then, the concentration decreased until it was not detected after 10 hours for 9-day HRT and after 28 hours for 6-day HRT. i-valerate presented a slight increase after the feeding followed by a decrease until a constant level was reached. As abovementioned, the biogas flow rate kept a close correlation with the TVFA behaviour. As expected, the highest flow rate was obtained when the TVFA decreased rapidly, whereas constant flow rate, showed by a null slope, were hardly seen. In fact, null slopes were only obtained for the last 4 hours of the 6-day HRT. These facts could evidence that further the contribution of acetate to methane production, the minor pathways of propionate and butyrate fermentations should be taken into account, as they were found to be a 20% of the total methane production (Mackie et al. 1981, Ahring et al., 1987).

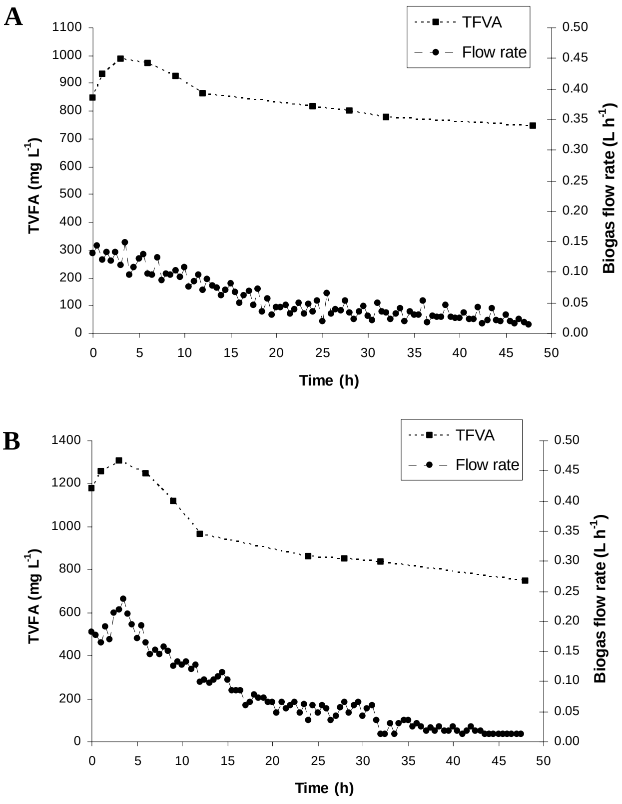

Figure 3. TFVA and biogas flow rate profile for: (A) TAD 9-day HRT, (B) TAD 6-day HRT.

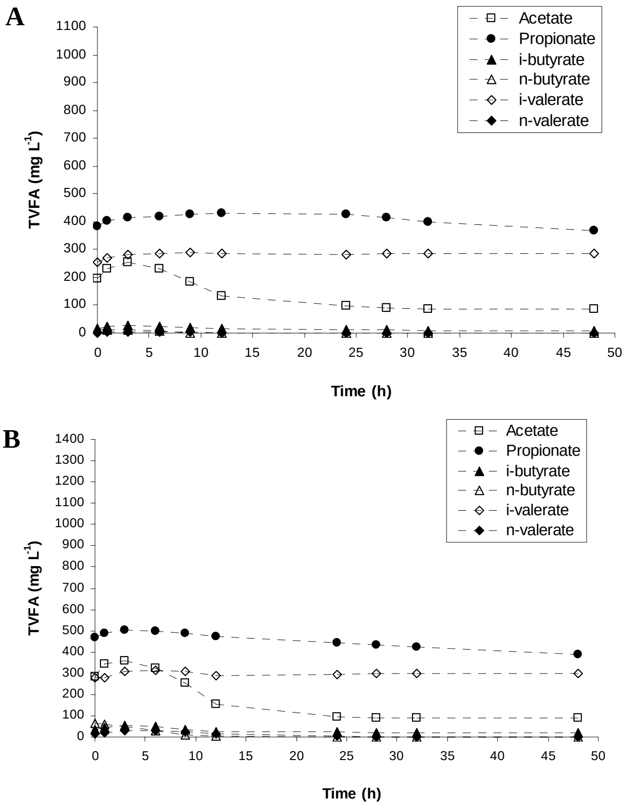

Figure 4. Individual VFA profile for: (A) TAD 9-day HRT, (B) TAD 6-day HRT.

4 CONCLUSIONS

Anaerobic digestion of sewage sludge showed stable performance for mesophilic and thermophilic digestion at the evaluated hydraulics retention time, showing a specific biogas yield that remained practically constant at 0.31 L_{biogas} VS_{added}^{-1} day^{-1} for mesophilic anaerobic digestion and 0.29 L_{biogas} VS_{added}^{-1} day^{-1} for thermophilic anaerobic digestion. The organic matter removal efficiency, showed a slightly decrease when the HRT was shortened for both operating temperatures. These results and the no accumulation of volatile fatty acids suggested that the anaerobic digestion process, for both temperature conditions, was limited by disintegration-hydrolysis step. Furthermore, thermophilic digested sludge exhibited poorer dewaterability properties than mesophilic digested sludge. Finally, the volatile fatty acids behaviour showed clearly differences between mesophilic and thermophilic anaerobic digestion. Mesophilic digestion presented low acid concentrations, with acetate as the predominant acid; in contrast, thermophilic digesters showed higher levels of volatile fatty acids prevailing propionate and i-valerate over the others. Despite these differences, a clear correlation between total volatile fatty acids evolution and biogas flow rate could be observed.

5 ACKNOWLEDGEMENTS

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(148) AGRICULTURAL AND INDUSTRIAL WASTES CO-DIGESTION: FEEDSTOCK STUDY AND MASS BALANCE EVALUATION

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EXECUTIVE SUMMARY

2006 and 2011 French regulations regarding biogas valorisation through feed-in tariffs gave a recent boost to the development of anaerobic digestion activities in France: electricity produced from biogas is currently purchased between 112 and 200€/MWhe (in function of installed electrical power) against slightly more than 50€ before 2006. As a consequence, total installed electrical power from agricultural and industrial wastes co-digestion (<600kWe) had raised from less than 1MWe before 2006 to more than 5MWe in 2011 (Ademe, 2011).

In order to assess such co-digestion market opportunities, CIRSEE team, in partnership with AgroParisTech experimental farm and SITA Ile de France, engaged a preliminary characterisation campaign. Both agricultural and industrial wastes were identified and sampled. Liquid and solid manures, clean and rotten maize silage, food and grease wastes were characterised for pH, dry and volatile matter (DM and VM) and total and ammonium nitrogen contents. To assess anaerobic biodegradability, batch incubation tests were performed in controlled environment during 60 days on each "substrate" and on a specific mix of them, which corresponds to a typical feedstock with half agricultural and half industrial wastes inputs. This first part of the study allowed comparison of substrates characteristics and assessment of co-digestion effects by comparing mixed and individual materials degradation behaviour. Basic digester and biogas engine sizing approach was also considered, based upon the typical feedstock characteristics. Furthermore, at the end of some incubation tests, materials were again characterised in order to evaluate digestate matter quality and more particularly organic nitrogen mineralisation during anaerobic digestion. A simple calculation approach was implemented to establish a well mass-balanced reaction scheme according to the following hypothesis: loss of matter and water in biogas, total nitrogen conservation and initial organic nitrogen partial mineralisation. Incubated materials properties calculated from initial waste and inoculum characteristics were compared to experimental data to determine the nitrogen mineralisation degree.

Characterisation results highlighted a great variability between the different products: pH ranged from 4 (food waste) to 9 (solid manure), DM content from 10 (liquid manure) to 53 % (grease) of raw matter, total nitrogen from 5 (grease) to 75 g/kgDM (food waste) and ammonium nitrogen from 0.4 (grease) to 12 g/kgDM (liquid manure). Anaerobic biodegradabilities also showed important variability as methane potentials ranged from 18 L/kg of raw matter (RM) for liquid manure to 557 L/kgRM for grease. As a consequence, mix sample presented the following medium characteristics: 17% DM, 89% VM, 47 gN/kgDM and a methane potential of 75 L/kgRM. From those different results, we can argue that agricultural materials, while representing 50 % of inputs total raw matter, only contribute to 15 % of overall feedstock methane potential. Industrial wastes contribution is thus essential in such co-digestion activities. Assuming a combined heat and power system functioning 8000 h/year with a 38% electrical yield, an electrical power engine of 35 We/(tRM$_{input}$/year) should be needed for the considered feedstock. Furthermore, from biogas production kinetic observed in batch experiments, a minimum retention time of 35 days could be estimated, to achieve at least 90 % of ultimate methane production reached after 60 days of incubation. This means that minimum reactor volume should be around 0,1 m^3/(tRM$_{input}$/year). From analysis performed at the end of incubation tests, anaerobic digestion mass balance was established. Concerning nitrogen, a 25 % average mineralisation of substrate initial organic nitrogen was observed. The higher mineralisation degrees were observed after food wastes incubations where almost 40% of substrate initial organic nitrogen were converted to ammonium, after 60 days of anaerobic degradation.

To conclude, this study demonstrates the particular interest of agricultural and industrial wastes co-digestion to combine organic wastes treatment, bio energy production and agronomic valorisation.

1 INTRODUCTION

This paper summarizes preliminary research results of CIRSEE team to analyse yields and mass balance of agricultural and industrial wastes co-digestion. This works was realised in partnership with AgroParisTech experimental farm and SITA Ile de France.

1.1 Background

2006 and 2011 French regulations regarding biogas valorisation gave a recent boost to the development of anaerobic digestion activities in France: electricity produced from biogas is currently purchased between 112 and 200€/MWhe (in function of installed electrical power) against slightly more than 50€ before 2006. As a consequence, total installed electrical power from agricultural and industrial wastes co-digestion (<600kWe) had raised from less than 1 MWe before 2006 to more than 5 MWe in 2011 (Ademe, 2011). However, this remains quite low with regard to other European countries such as Germany for instance. As a rough guide, Table 1 gathers some French anaerobic digestion units examples with their main characteristics (tonnage, digester volume and electrical installed power).

TABLE 1 **Agricultural and industrial wastes co-digestion plants French examples**

Plants name	**Constructor**	**Commissioning** (year)	**Feedstock** (t/year)	**Digester** (m^3)	**Power** (kWe)
Treflamm	PlanET Biogas	2011	3720	923	100
Gazéa	AEB	2009	5000	2160	205
SCEA du Bois Brillant	CLAIE	2011	6100	1073	104
GAEC du Roitelet	Agrikomp	2010	7330	1300	250
EARL Guernequay	Evalor	2010	8138	1200	130
BioWatt	Envitech	2010	15162	2570	526
Capik	Naskéo	2011	18000	3200	450
Biogasyl	Schwarting Biosystem	2009	25000	1700	600
Géotexia	NESolutions	2011	75000	6000	1600

1.2 Research objectives

Overall research objective was to contribute to the assessment of such co-digestion market opportunities. More particularly, this preliminary characterisation campaign aimed at analysing two specific aspects of anaerobic biodegradation:

- Firstly, analyse co-digestion effects by comparing anaerobic biodegradability of each considered “substrate” with the biodegradability measured on a specific mix of most of them, corresponding to a typical feedstock with half agricultural and half industrial wastes inputs. By the way, digester and biogas engine basic sizing has been also considered, based upon the typical feedstock characteristics.
- Furthermore, evaluate digestate quality prediction opportunity through a simple calculation approach, based on input wastes characteristics, establishing a well mass-balanced reaction scheme. Special attention concerned organic nitrogen mineralisation during anaerobic degradation. By the way, digestate quality of the typical feedstock and its agronomic valorisation have been also briefly considered.

2 METHODOLOGY

Both agricultural and industrial wastes were identified and sampled. All were characterised for pH, dry and volatile matter (DM and VM), total (TKN) and ammonium ($N\text{-}NH_4$) nitrogen contents and methane potentials (BMP, Biochemical Methane Potential). Same analysis were performed on digestates used as inoculum for BMP tests. The specific mix of substrates corresponding to typical feedstock was also analysed for methane potential. At the end of some incubations (BMP tests), digested materials were characterised again for DM and VM, TKN and $N\text{-}NH_4$ contents to assess the ability of the calculation approach to predict digestate quality from input materials characteristics.

2.1 Materials

Wastes identified and sampled were the following ones:

- Agricultural wastes:

liquid and solid cow manures, solid sheep manure and wastewaters from milking room washing were the principal ones, composing the typical feedstock detailed thereafter. Goat and horse solid manures were also sampled to analyse characteristics differences between solid manures of different origins. Rotten maize silage was sampled as it represents a potential input, along with clean maize silage to analyse effect of silage decay on biodegradability.

- Industrial wastes:

two different types of food wastes were considered, one sampled at a company restaurant where food leftovers are separately collected and the other obtained from industrial unpacking of unsold food articles. Grease from catering grease trap was the third industrial waste considered.

The **typical feedstock** with half agricultural and half industrial wastes was named Mix sample. Its composition was the following one, expressed in % of total raw matter (RM): 30% liquid cow manure, 10% wastewaters, 5% solid cow manure, 5% solid sheep manure, 12% food leftovers, 12% grease and 26% unpacked food unsold articles.

Three digestates were used as inoculum in BMP tests described thereafter. First one (Inoculum 1) was sampled at an industrial co-digestion plant treating both agricultural and industrial wastes. It was used for all BMP tests performed on individual substrates. The second digestate (Inoculum 2) was composed of incubated materials from previous BMP experiments where both industrial and agricultural wastes had been analysed. It was only used for the Mix sample analysis (typical feedstock). A third digestate was used to analyse grease sample again, as first incubation showed a very low biodegradability. This inoculum was used to grease as it was sampled in a wastewater treatment plant anaerobic digester with grease co-digestion. All digestates were kept a few days at mesophilic temperature under agitation before being used for present BMP tests to insure a low residual biodegradable matter content.

2.2 Methods

- **BMP tests**

Precise quantities of substrate(s) were introduced in 1 litre glass bottles. Digestate was added to reach a specific inoculum to substrate (I/S) ratio in each bottle. Almost all tests were realised at I/S ratio of 1, expressed in $gVM_{inoculum}/gVM_{substrate}$, to ensure favourable organic loading conditions (Chynoweth et al., 1993; Gunaseelan, 1997). However, BMP tests performed on food wastes were carried out with a I/S ratio of 2, as previous analysis showed that degradation was often unstable with a ratio of 1 with this type of substrate. If necessary, tap water was added to obtain overall DM content of less than 10% to insure favourable mixing condition (this was only the case of the Mix sample analysis because of the Inoculum 2 higher DM content). Each bottle free air space was then flushed with nitrogen before closing with an airtight rubber septum. Reactors were placed in mesophilic conditions (37°C) with an orbital agitation and monitored during 60 days. Produced biogas quantities were determined by pressure measurements and biogas quality by µGC analysis, both done directly in the bottles through the septum using specific needles. Assays containing only digestate were also monitored to measure biogas production from endogenous respiration and residual organic matter. Inoculum contribution was subtracted from biogas and methane productions obtained within the assays containing substrate(s). All assays were performed in duplicate. At the end of the 60 days of incubation, most of digested materials were analysed for DM, VM, TKN and $N\text{-}NH_4$ contents. As BMP duplicates presented a low deviation between them, duplicates contents were mixed prior to analysis.

- **Chemical characterisations**

DM, VM and TKN characterisations were performed according to European standards, EN 12880, 12879, 13342 respectively. $N\text{-}NH_4$ contents were determined according an internal method.

- **Calculation approach (for digestate quality prediction)**

Final DM and VM masses (after incubations) were calculated by subtracting masses of dry biogas produced (from CH_4 and CO_2 total volumes measured during BMP tests) to initial DM and VM masses (from substrates plus inoculum). Final raw matter (RM) masses were calculated accounting losses through dry biogas (as for DM and VM) and adding water losses in biogas assuming gas produced was vapour saturated at 37°C. Final TKN contents were determined assuming TKN complete conservation during anaerobic degradation and, thus, neglecting NH_3 gas losses in biogas. Final $N\text{-}NH_4$ contents were calculated, in a first step, assuming a 25% mineralisation of total initial organic nitrogen ($\approx TKN - N\text{-}NH_4$). Final DM, VM, TKN and $N\text{-}NH_4$ concentrations were finally calculated and compared to measured final characteristics for almost all substrates incubated.

3 WASTES CHARACTERISTICS, CO-DIGESTION EFFECTS AND BASIC SIZING CONSIDERATIONS

3.1 Wastes characteristics

Table 2 summarizes overall characterisation results of agricultural and industrial wastes sampled.

Concerning **chemical characteristics**, whereas all agricultural wastes present a basic pH, between 7.5 for liquid cow manure to more than 9 for solid ones, all industrial wastes present acid pH values, between 4 to 6. This is the first co-digestion benefit in evidence: co-digestion of those two types of feedstock may help overall digester input to reach a neutral initial pH, more suitable for anaerobic digestion. Wastewaters apart, all wastes show relatively high dry matter contents, from 11% for liquid cow manure to 53% for grease and around 30% for solid manures. VM contents are comprised between 70 and 98%, the lowest values being measured on the different manures, highlighting the important mineral contents of those wastes. TKN contents, expressed in gTKN/kg of raw matter, range between 5 and 10 gTKN/kgRM. Grease present the lowest TKN content among all sampled wastes.

TABLE 2 **Overall analysis results of agricultural and industrial wastes**

Wastes samples	**pH**	**DM** (% of RM)	**VM** (% of DM)	**TKN** (g/kgDM)	**N-NH$_4$** (g/kgDM)	**P CH$_4$** (L/kgVM)		**P CO$_2$** (L/kgVM)	
Liquid cow manure	7,7	11,0	79,8	37,9	11,8	259	(23)	158	(17)
Wastewaters	7,5	1,0	54,6	89,5	50,6	125	(7)	84	(5)
Solid cow manure	9,2	26,9	72,2	18,7	1,4	197	(7)	155	(4)
Sheep manure	9,2	29,2	83,3	34,6	8,7	208	(2)	158	(0)
Goat manure	9,4	23,5	79,4	31,6	7,6	171	(2)	130	(1)
Horse manure	9,4	35,5	85,0	15,7	3,9	240	(11)	194	(6)
Clean maize silage	3,7	32,5	94,2	17,8	1,1	359	(3)	304	(4)
Rotten maize silage	7,8	23,1	95,8	18,1	0,4	314	(12)	267	(6)
Food leftovers	5,1	16,0	91,2	32,5	0,5	511	(20)	353	(3)
Unpacked food articles	3,9	11,4	89,3	74,8	2,8	537	(18)	295	(8)
Grease (1)	5,7	53,3	97,8	5,4	0,4	170	(75)	64	(22)
Grease (2)						1070	(7)	394	(5)

Note: - values in brackets for CH$_4$ and CO$_2$ corresponding to standard variation upon duplicates of BMP tests
- RM: Raw Matter; DM: Dry Matter; VM: Volatile Matter; TKN: Total Kjeldhal Nitrogen

Concerning **anaerobic biodegradability**, Figure 1 illustrates methane productions obtained during BMP tests. The higher yield was recorded on grease, with a methane potential of 1070 L/kgVM. This value, slightly higher than theoretical methane potential of lipid of 1013 L/kgVM, indicates a complete degradation of grease. However, this was only observed within BMP test performed with the inoculum used to grease (value mentioned as grease (2) in Table 2). The other incubation of the same grease sample (mentioned as grease (1) in Table 2), with a methane potential of 170 L/kgVM after 60 days of monitoring, was thus clearly inhibited. The large standard deviation obtained for grease (1) also revealed uncertain equilibrium conditions in this test. Chen *et al.* (2010) had shown a great influence of I/S ratio on grease trap anaerobic degradation in batch experiment, with final methane potentials varying from 550 to 970 L/kgVM for I/S ratios of 1 and 2 respectively. Here, since I/S ratio was the same in both experiments (I/S = 1), results highlight an impact of inoculum quality too. This aspect is discussed again in the next part (co-digestion effects). The two food wastes are the other samples with the higher methane potentials, with values of 511 and 537 L/kgVM, typical for such materials. Chynoweth *et al.* (1993) and Cho *et al.* (1995), for instance, reported values for mixed food wastes of 541 and 472 L/kgVM respectively.

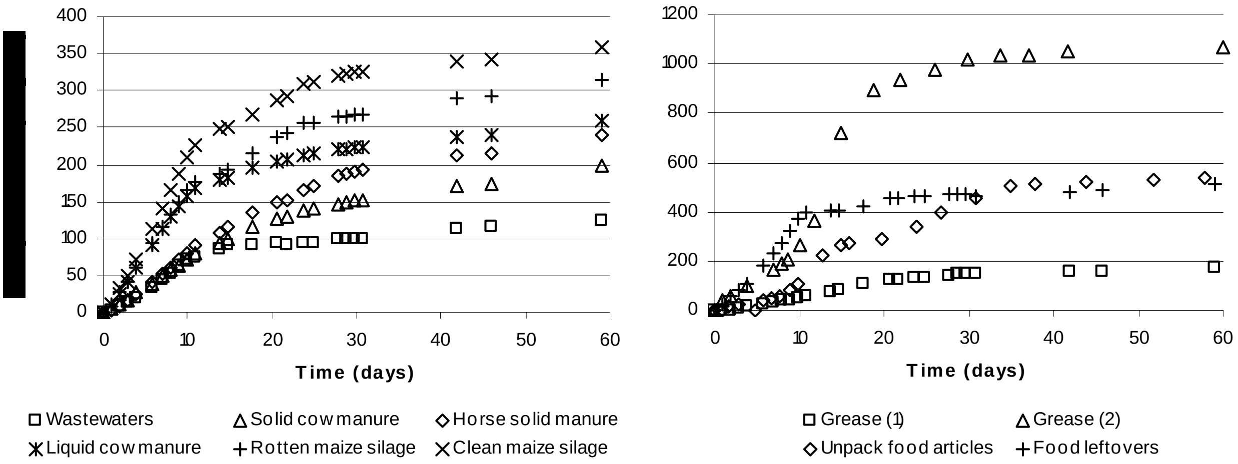

FIGURE 1 **Illustration of methane cumulative productions during BMP tests for most of agricultural wastes tested (at left, not all the solid manures presented) and for industrial ones (at right)**

Agricultural wastes present lower methane yields than industrial ones, between 150 and 350 L/kgVM, most probably because of their higher cellulosic and ligno-cellulosic contents. Furthermore, agricultural wastes are, in a way, materials already digested. Between the different tested manures, solid goat manure present the lower methane potential with 171 L/kgVM and liquid cow manure, the higher one with 259 L/kgVM (see Table 2). All those values are quite similar with the ones usually reported in literature and used for biogas project basic assessment (see Ademe, 2007, with values comprised between 200 and 300 L/kgVM). It is noticeable that the solid manures present a lower degradation kinetic than all others wastes, with significant methane productions still observed after 35 days of incubation (see Figure 1 with cow and horse solid manures methane productions illustrated). This indicates that those types of substrates may require higher retention times. Clean maize silage show the higher methane potential among all agricultural wastes tested, with 359 L/kgVM, value in agreement with the ones reported for instance by Amon *et al.* (2007), where methane potentials of different maize silages ranged from 250 to 365 L/kgVM. Expressed with regard to raw matter, clean maize silage methane potential is the second higher potential recorded in this study (after the one of grease), with 110 L_{CH4}/kgRM. Conjugated with harvest yields that can reach 30 t_{VM}/ha according to Amon *et al.* (2007), this partly explains the keen interest for this substrate in some European countries. The lower methane yield obtained with rotten maize silage (314 L/kgVM) may be the consequence of the aerobic partial degradation of maize silage. In spite of its lower methane yield, rotten maize silage stays a suitable substrate for anaerobic degradation and, especially, a more acceptable one than clean maize silage, particularly in the French context.

3.2 Mix sample analysis and co-digestion effects

Figure 2 illustrates methane production obtained during incubation of the Mix sample, corresponding to a typical feedstock with half agricultural and half industrial wastes (composition detailed before in material part). A methane potential of 480 L/kgVM was reached after 60 days of incubation. Figure 2 also illustrates the calculated methane productions corresponding to this Mix sample, calculations done using methane productions obtained for each component of the Mix sample and weighing them by the proportion of the considered substrate in the Mix. As two antagonist BMP values were obtained for grease (named grease (1) and grease (2), see Table 1), two calculated productions are mentioned. As, in one case, calculated Mix methane production largely under-estimates experimental one (280 L/kgVM) and, in the other, over-estimates it (640 L/kgVM), a third Mix methane production is mentioned, calculated using the average BMP value for grease. This last calculated production fit the experimental one accurately.

Those results raise different remarks and issues:

- Anaerobic degradation of grease as mono-substrate may be highly sensitive, with both effects of micro-organisms quantity with regard to amount of substrate (as pointed out by Chen *et al.* (2010) through different I/S ratios) and of micro-organisms "quality" or adaptation, as pointed out in this study through the results obtained with the same grease sample and same I/S ratio but with different inoculums.

- Co-digestion of grease with other types of substrates may facilitate their anaerobic degradation, maybe thanks to the presence of more (bio) accessible substrates or simply through C/N balancing, as grease analysed here presents a very low nitrogen content
- Calculation of a particular feedstock methane potential from the characteristics of its component can lead to misinterpretations, as pointed out here through the different calculated Mix methane productions with regard to Mix experimental one
- Grease is of major importance in the feedstock considered here: according to the BMP value taken for grease, overall calculated Mix methane yield was more than doubled, from 280 to 640 L_{CH4}/kgVM. As grease trap sample present very high DM and VM contents, while representing only 12% of Mix overall raw matter, grease VM represents more than 40% of Mix total VM. This may be too much, at least in the starting period of a continuous process, with regard to sensitivity of grease anaerobic degradation

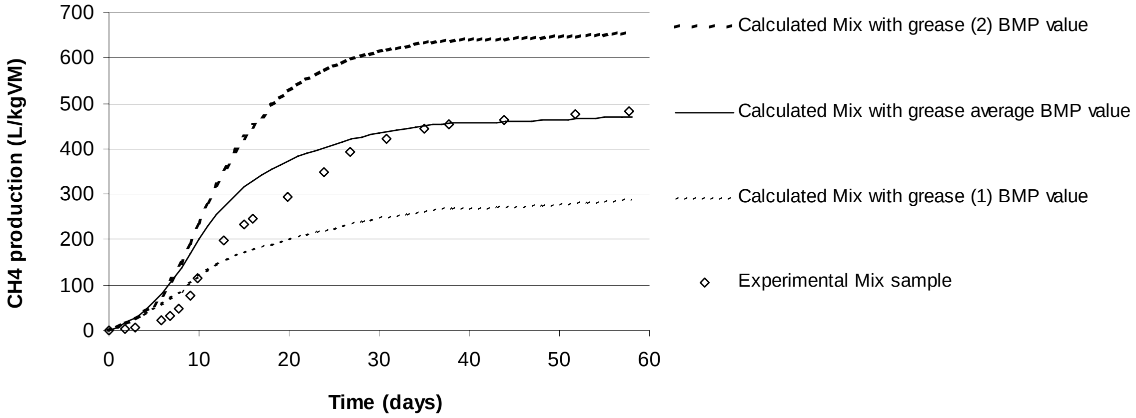

FIGURE 2 **Mix sample BMP : comparison of methane experimental production with calculated productions based upon mix sample components methane productions**

Despite uncertainty linked to grease behaviour in the Mix sample degradation and assuming that grease exhibited in this experiment a methane potential of 620 L/kgVM (average value of grease (1) and grease (2) results), we can argue that agricultural materials, while representing 50 % of Mix sample total raw matter, only contributed to 15 % of the overall feedstock methane potential. Industrial wastes contribution is thus clearly essential in such co-digestion activities.

3.3 Basic sizing considerations

An experimental Mix methane potential of 480 L/kgVM was obtained after 60 days of incubation. Considering DM and VM contents of Mix sample of 17.5 and 88.7% respectively (calculated from DM and VM contents of its components), this is equivalent to 75 m^3_{CH4}/tRM and, thus, approximately to 750 kWh_{tot}/tRM. Assuming a combined heat and power system with a 38% electrical yield, 285 kWh_e/tRM could be recovered. Functioning 8000 h/year, an electrical power engine of 35 We/(tRM_{input}/year) should thus be needed for the considered feedstock.

From methane production kinetic observed in Mix sample BMP test (see Figure 2), we can deduce that, in batch, a minimal duration of 35 days of incubation was necessary to recover slightly more than 90 % of ultimate methane production (reached after 60 days). This means that 35 days should be the minimal retention time in a continuous process to achieve, at most, 90% of batch ultimate methane yield. This implies a minimum reactor volume around 0,1 m^3/(tRM_{input}/year).

Those values are consistent with the ones that can be calculated from co-digestion units examples given in Table 1, where average electrical installed power is 0.026 ± 0.008 We/(tRM_{input}/year) and average reactor volume is 0.174 ± 0.107 m^3/(tRM_{input}/year), with intervals (±) corresponding to standard deviations.

Finally, the maximum organic loading rate (OLR) obtained with the typical feedstock and the sizing values considered here is 4.2 kgVM/m^3/day. This value appears acceptable with regard, for instance, to the ones reported by Braun *et al.* (2006) over 41 representative Austrian biogas plants with crop and co-substrates digestion. Those authors mentioned minimal, median and maximal OLR of respectively 1, 3.6 and 8 kgVM/m^3/day.

4 DIGESTATE QUALITY PREDICTION AND AGRONOMIC VALORISATION

4.1 Digestate quality prediction

Table 3 presents the comparison between calculated and experimental characteristics of digested materials at the end of BMP tests for some of the substrates incubated. Initial characteristics, corresponding to the mixture of inoculum and analysed material (except "Inoculum 1" raws corresponding to the digestate only), are also mentioned as a rough guide. Not all the data are presented as, in all, 12 digested materials were analysed for 4 characteristics. The percentages in last column indicate deviation between calculated and experimental values for each considered characteristic. As illustrates Table 3, deviation was sometimes positive and sometimes negative with regard to different materials and characteristics, indicating that calculation did not systematically deviate in the same way. For instance, final VM calculation under-estimates experimental result by 3.5% for liquid cow manure whereas an over-estimation of 2% is observed for solid sheep manure final VM content. Concerning N-NH_4, the calculation over-estimates final content by 3.8% for solid sheep manure whereas it under-estimates it by 1.5% for food leftovers. In an overall point of view, the average deviation calculated on absolute values of the 48 deviations (12 digested materials x 4 characteristics) is of 3.6% and the maximal deviation, of 10% (on clean maize silage final TKN content, data not shown). Those results indicate that final properties of digested materials can be roughly estimated through the simple calculation approach proposed.

TABLE 3 **Comparison between calculated and experimental characteristics of digested materials at the end of BMP tests for some of the products incubated**

Digested materials	**Characteristics**		**Values** **Initial** from analysis	 **Final** from analysis	 **Final** from calculator	**Deviation** Final calculation / analysis (%)
Liquid cow manure	%DM	(/RM)	6,95	5,80	5,62	-3,1
	%VM	(/DM)	72,68	68,00	65,64	-3,5
	NTK	(g/kgRM)	6,33	6,54	6,44	-1,6
	N-NH_4	(g/kgRM)	3,05	3,66	3,94	7,8
Solid sheep manure	%DM	(/RM)	8,45	6,70	6,87	2,6
	%VM	(/DM)	74,33	66,50	67,80	2,0
	NTK	(g/kgRM)	7,49	8,01	7,64	-4,5
	N-NH_4	(g/kgRM)	3,55	4,46	4,63	3,8
Food leftovers	%DM	(/RM)	6,70	4,84	4,71	-2,8
	%VM	(/DM)	74,08	65,00	62,11	-4,4
	NTK	(g/kgRM)	6,89	7,12	7,07	-0,7
	N-NH_4	(g/kgRM)	3,27	4,35	4,29	-1,5
Inoculum 1	%DM	(/RM)	5,48	4,87	5,14	5,5
	%VM	(/DM)	67,50	61,20	65,19	6,5
	NTK	(g/kgRM)	7,11	7,00	7,15	2,0
	N-NH_4	(g/kgRM)	3,69	4,53	4,57	0,9

Note: RM: Raw Matter; DM: Dry Matter; VM: Volatile Matter; TKN: Total Kjeldhal Nitrogen

Calculation concerning organic nitrogen mineralisation may be refined by considering different mineralisation degrees for substrates and inoculum. From batch experiment containing only digestate, a mineralisation percentage for organic nitrogen of inoculum only could be fitted precisely and, then, a specific mineralisation degree could be calculated for each substrate by analysing experiments containing substrate plus inoculum and considering that inoculum behaviour was always similar. Doing so (data not shown), the maximum organic nitrogen mineralisation is observed on food wastes samples, with a conversion of almost 40% of initial organic nitrogen to N-NH_4 while most of agricultural wastes present mineralisations of less than 10%. Using this approach on Mix sample degradation, an organic nitrogen mineralisation of 20% was calculated.

4.2 Application to the considered feedstock in view of digestate agronomic valorisation

The main characteristics of the digestate resulting from typical feedstock considered here (see composition in part 2.1) may be estimated through the simple calculator, considering methane and carbon dioxide productions equal to 90% of ultimate potential measured experimentally on Mix sample and a 20% conversion to N-NH_4 of initial organic nitrogen.
Table 4 presents the calculated characteristics of the digestate produced from the typical feedstock anaerobic digestion.

TABLE 4 **Anaerobic digestion process input (experimental) and output (calculated) characteristics considering the typical feedstock with half agricultural and half industrial wastes**

Characteristics		**Input** (experimental)	**Output** (calculated)
t RM	(t)	1	0,88
% DM	(/ RM)	17,5	7,1
% VM	(/ DM)	88,7	68
TKN	(kg / tRM)	5,27	5,97
N-NH_4	(kg / tRM)	0,70	1,83

Digestate may present a DM content around 7% and a total nitrogen concentration around 6 kgN/tRM. Considering nitrogen contents as the limiting factors for digestate spreading and a maximal nitrogen load of 170 kgN/ha/year, a spreading of 28 $tRM_{digestate}$/ha/year could be realised. Considering that 1 t of feedstock would led to 0.9 t of digestate (see Table 4), we can argue that 0.036 ha/(tRM_{input}/year) would be necessary for the considered feedstock to insure digestate agronomic recycling. Of course, those hectares must belong to a land spreading plan approved by the local administration because of the current waste status of the liquid digestate.

5 CONCLUSIONS

Characterisation results of the agricultural and industrial wastes considered highlighted great differences between them, particularly regarding anaerobic biodegradability. While methane potentials of agricultural wastes ranged from 171 (for goat solid manure) to 314 L_{CH4}/kgVM (for rotten maize silage), food and grease industrial wastes presented potentials higher than 500 and 1000 L_{CH4}/kgVM respectively. Grease may however need an adapted micro-organisms consortium to achieve such yields, as also very low biodegradability was recorded within some experiments with grease as only substrate. From results obtained on the Mix sample, corresponding to a typical feedstock with half agricultural and half industrial wastes, it appears that a minimal digester volume of 0,1 m^3 per tons of raw matter (RM) treated per year may be needed to achieve a minimal retention time of 35 days and that a biogas engine with an electrical power around 35 We/(tRM_{input}/year) may be considered. The approach developed to determine digestate quality from input wastes properties was able to predict incubated materials characteristics with a 10% maximum deviation from experimental results. It consists in accounting loss of matter using experimental CH_4 and CO_2 yields and assuming total nitrogen conservation and an average 25% conversion of initial organic nitrogen to N-NH_4. Applied to the typical feedstock considered, 0.036 ha/(tRM_{input}/year) may be necessary for digestate agronomic recycling through a land spreading plan.
Some of the research perspectives pointed out are the following ones:

- Assess deeply grease anaerobic (co-) digestion behaviour using different co-substrates and inoculums types, through different I/S initial ratios in batch experiments or with different loading rates in continuous systems.
- Refine the simple calculator presented here to assess more precisely digestate quality for different feedstock of interest and broaden its application to large scale units to verify its consistency.

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(210) RUMEN CONTENT AND ANAEROBIC SLUDGE CO-INOCULATED OF IN CELLULOSE FED ANAEROBIC BATCH DIGESTERS

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EXECUTIVE SUMMARY

Municipal solid waste is a very complex and heterogeneous system made of a wide range of materials. Among these materials, more than 50% are biodegradable and can be valorized through treatments based on anaerobic biological processes to produce biogas, a renewable source of energy. The ligno-cellulosic fraction of waste, mainly constituted by paper and cardboard (25%), plays an essential role in the production of biogas and is responsible for almost 80% of biogas emission (Eleazer, et al., 1997). It is consequently a keystone in the anaerobic biological processes of degradation. However, cellulose is a recalcitrant molecule, and is not hydrolyzed easily in bioprocesses, which limits its anaerobic digestion kinetic (Verstraete, et al., 1996) and efficiency. The rate of biodegradation occurring in animal digestive systems, such as ruminants' guts, is significantly higher despite much lower residence times than in digesters (Weimer, et al., 2009). Consequently, to improve the cellulose degradation and methane production in anaerobic digesters, several studies involving rumen microorganisms in anaerobic digesters have been conducted (Gijzen, et al., 1987, Blasig, et al., 1992, Barnes & Keller, 2003). However, macroscopic observations suggest that ruminal flora may not settle easily in industrial anaerobic processes (O'Sullivan & Burrell, 2007). The potential of cellulolytic ruminal flora properties expression in an anaerobic digester can therefore be questioned.

In this study, cellulose degrading anaerobic batch incubations were set up and run in parallel. They were co-inoculated with MSW digester sludge and ruminal content. ^{13}C labeled cellulose degradation and reaction pathways were followed and described over time with GC-C-IRMS. These results were linked to the identification and the monitoring of the dynamic (Automated Ribosomal Intergenic Spacer Analysis method, ARISA (Fisher & Triplett, 1999)) of microorganisms.

This experiment highlighted the functional characteristics of cellulose degradation digestion in a digester co-inoculated with a natural and an industrial biomass. After 65 days, 50% of the cellulose was converted into biogas, composed of 60% of methane. The dominant intermediates were acetate and propionate. Their partial ^{13}C enrichment suggests a strong degradation of endogenous substrates. The dynamics of functional groups followed by ARISA showed a strong correlation of the appearance and disappearance of bands with the dynamics of Volatile Fatty Acids. However none of the groups found in the reactors were initially present in the rumen. Cellulolytic potentiality of ruminal flora did not expressed during the experiment despite inoculation in large quantities, in a closed system, with a suitable substrate (cellulose) and favorable physico-chemical conditions such as mesophilic temperature or the presence of rumen fluid. Yet the diversity was more important in the batch reactor than in the initial industrial sludge. The co-inoculation probably disordered diversity equilibrium within the anaerobic industrial sludge, and several ecological niches were taken over by species subdominant in the initial inocula. Co-inoculation of an exogenous flora with anaerobic sludge could thus be an environmental lever to modify diversity in full scale reactors.

This study demonstrates that a more precise identification of selection pressures that lead to the stabilization of different types of flora is necessary to use natural flora into industrial systems. Design of processes mimicking animals digestive systems also seems very promising (Godon, et al., 2010, Bayané & Guiot, 2011).

Statement

This work was realized in the Framework of the DANAC research project (ANR-Bioénergies 2009)

1 INTRODUCTION

1.1 Background

Municipal solid waste is a very complex and heterogeneous system made of a wide range of materials. Among these materials, more than 50% are biodegradable and can be valorized through treatments based on anaerobic biological processes to produce biogas, a renewable source of energy. The ligno-cellulosic fraction of waste, mainly constituted by paper and cardboard (25%), plays an essential role in the production of biogas and is responsible for almost 80% of biogas emission (Eleazer, *et al.*, 1997). It is consequently a keystone in the anaerobic biological processes of degradation. However, cellulose is a recalcitrant molecule, and is not hydrolyzed easily in bioprocesses, which limits its anaerobic digestion kinetic (Verstraete, *et al.*, 1996) and efficiency. The rate of biodegradation occurring in animal digestive systems, such as ruminants' guts, is significantly higher despite much lower residence times than in digesters (Weimer, *et al.*, 2009). Consequently, to improve the cellulose degradation and methane production in anaerobic digesters, several studies involving rumen microorganisms in anaerobic digesters have been conducted (Gijzen, *et al.*, 1987, Blasig, *et al.*, 1992, Barnes & Keller, 2003). However, macroscopic observations suggest that ruminal flora may not settle easily in industrial anaerobic processes (O'Sullivan & Burrell, 2007). The potential of cellulolytic ruminal flora properties expression in an anaerobic digester can therefore be questioned.

1.2 Research objectives

In this study, cellulose degrading anaerobic batch incubations were set up and run in parallel. They were co-inoculated with MSW digester sludge and ruminal content. ^{13}C labeled cellulose degradation and reaction pathways were followed and described over time with GC-C-IRMS. These results were linked to the identification and the monitoring of the dynamic (Automated Ribosomal Intergenic Spacer Analysis method, ARISA (Fisher & Triplett, 1999)) of microorganisms.

2 METHODOLOGY

2.1 Batch system digester and inocula

Anaerobic batch digesters (150 ml) were set up using a mix of mesophilic MSW anaerobic digester sludge (0.9 g of volatile matter introduced in each bottle), 20 ml of ruminal content (DOC=4.1 g/l, acetic acid =3.3 g/l, propionic acid =0.8 g/l) containing 0.16 g of volatile matter. The rumen vs sludge ratio was thus fixed at 18% (VM_{rumen}/VM_{sludge}) in each bottle. 80 ml of supernatant originating from the centrifugation (13000 g, 15 minutes) of MSW leachate (from closed landfill of Vert-le-Grand, France was then added as the liquid phase (DOC=1.9 g/l, VFA< 10 mg/L). Mesophilic sludge was recovered from a full scale household waste digester (Varennes-Jarcy, France). It was filtered to remove solids bigger than 3 mm, and incubated in anaerobic condition at 35°C for several days until gas production was over. Ruminal content was obtained by direct sampling of the rumen fluid by passing a strainer and tubing through a Frick speculum placed in the mouth of a living cow. After sampling, the rumen content was strained through two layers of 1 mm nylon mesh. Samples were stored at -80°C until use. Bottles were hermetically sealed with a screw cap and a rubber septum and headspaces were flushed with nitrogen.

9 bioreactors in total were set-up. A first pair of duplicate reactors (bottles 1 and 2) were fed with ^{13}C-labeled cellulose (235 mg, synthesized by *Acetobacter xylinum* (Gagnaire & Taravel, 1980)). In parallel, one control bioreactor (bottle 3) containing unlabelled cellulose (same amount and type) and two negative control bioreactors without cellulose (bottles 4 and 5) were set up. Four controls without rumen co-inoculation (bottles 6, 7, 8 and 9) (only anaerobic sludge as inoculum) were also set-up. Time zero samples were taken and the reactors were incubated under anaerobic conditions at 35°C, without shaking, in the dark. Gas production and composition were measured and analyzed every two days. Liquid samples of 6 ml were regularly collected through the septum and centrifuged at 12 000 g for 10 minutes. Resulting pellets and supernatant were stored separately at -20°C for biomass and chemical parameters analysis, respectively.

2.2 Gas analysis

Biogas accumulation in the headspace was measured using a differential manometer (Digitron 2082P).

Head-space gas analysis was performed with a micro GC (CP4900, Varian) equipped with four parallel chromatographic lines (two molecular sieve 5A, a poraplot Q and a volamine columns) coupled with a thermoconductivity detector.

The poraplot Q column is used for CO_2, N_2O and H_2S (helium as carrier gas, column temperature of 30°C, injector temperature of 50°C, medium sensitivity), the first molecular sieve column is for O_2, N_2 and CH_4 (helium as carrier gas, column temperature of 55°C, injector temperature of 50°C, medium sensitivity), the second molecular sieve is for H_2 (argon as carrier gas, column temperature of 55°C, injector temperature of 50°C) and the volamine column is for NH_3 (helium as carrier gas, column temperature of 55°C, injector temperature of 50°C). The standard calibration was made with gas mixtures containing (v/v) 0.5% H_2S, 3% N_2O, 40% CO_2, 50% CH_4, 5% H_2 and 6.5% N_2.

These data were used to calculate gas production and composition, at standard temperature and pressure, allowing for the volume of gas and liquid samples extracted. Biogas was assimilated to an ideal gas.

2.3 Organic and inorganic carbon analysis

Dissolved organic carbon (DOC) was analyzed in the supernatant of the samples (Bioritech Model 700). The sample is first acidified to release the dissolved inorganic carbon (DIC - mainly carbonates) under CO_2 form, and then oxidized using potassium persulfate at 100°C. A double infrared beam detects and quantifies the CO_2 flow.

Acetic acid and propionic acid concentrations were measured with a Dionex 120 with an IonPAc ICE-AS1 column 9mmx250mm and conductimetric detection.

2.4 Isotopic composition measurement

Gas samples for analysis of percentage of ^{13}C in CH_4 were periodically collected with a syringe and transferred into 7mL vacuumed serum tubes. The analyses were performed using a Trace GC Ultra (Thermo Electron Corporation) attached to a Delta V Plus isotope ratio mass spectrometer via a GC Combustion III (Thermo Electron Corporation). Typical uncertainties, quantified by replicate measurements of different samples, were ±0.2‰.

Isotopic composition of VFA was measured in the supernatant of the samples. Samples were acidified (orthophosphoric acid- ph<2), heated at 90°C and shaked for 9 minutes. 600 µl of headspace gas were injected in a Trace GC Ultra (Thermo Electron Corporation) (splitless mode, 200°C). VFA were separated on a TR-WAX column (30 m X 0.53 mm X 0.1 µm, Thermo Fisher). Isotopic composition was measured on a Delta V Plus isotope ratio mass spectrometer via a GC Combustion III (Thermo Electron Corporation).

2.5 DNA extraction

Samples pellets were kept at -20°C before DNA extraction. Total DNA was extracted with the Power Soil DNA Isolation Kit (Mobio Laboratories Inc. Carlsbad) according to the manufacturer's instructions. Extracted DNA was quantified by Qubit (dsDNA HS Assay Kit, Invitrogen, Eugene) and DNA integrity was checked by electrophoresis on 1% agarose gel and EtBr staining.

2.6 Polymerase chain reaction (PCR) and Automated Ribosomal Intergenic Spacer Analysis (ARISA)

Two different sets of primers were used to perform ARISA on samples. The bacterial set ITSF/ITSReub (5'-GTC GTA ACA AGG TAG CCG TA-3'/5'-GCC AAG GCA TCC ACC-3') (Cardinale, *et al.*, 2004) was used for the amplification of the 16S-23S ITS of *Bacteria*. The set 71R/1389F (5-'TCG GYG CCC GAG CCG AGC CAT CC-3'/5'-CTT GCA CAC ACC GCC CGT-3') was used for the 16S-23S ITS of *Archaea* (Casamayor, *et al.*, 2002, Loy, *et al.*, 2002).

Bacterial 16S-23S ITS DNA was amplified in a 25 µL reaction mixture containing 1X PCR buffer, 0.75U of Thermo-Start Taq DNA polymerase (ABGene Ltd, Epsom, UK), 0.04mM (each) dNTP, 1.5mM of MgCl2, 0.02 µM ITSF and ITSReub (each) and 1µl of extracted DNA (2 to 25 ng). The mixture was held at 94°C for 3 min, followed by 35 cycles of 94°C for 1 min, 55.2°C for 1 min, 72°C for 2 min and a final extension step at 72°C for 10 min. The PCR protocol that was used to amplify the Archaeal 16S-23S ITS DNA region was similar (annealing temperature: 54.5°C).

ARISA (Fisher & Triplett, 1999) was performed using a capillary electrophoresis bioanalyzer for a fast processing of samples. Amplified ITS fragments were separated on an electrophoresis Bioanalyzer (2100 Electrophoresis Bioanalyzer, Agilent Technologies, Santa Clara) using the Agilent DNA 1000 Kit (Agilent Technologies) as described in (Fechner, *et al.*, 2010). Electrophoregrams in ASCII formats were imported into the StatFingerprints R package (Michelland, *et al.*, 2009) using the 2.10.1 version of the R project. ARISA profiles were aligned and the area under each curve was normalized to 1 before statistical comparison (scaled centred principal component analysis, PCA).

3 CELLULOSE DEGRADATION AND INTERMEDIATES ACCUMULATION

3.1 Gas production

Gas production of cellulose degrading batch reactors (bottles 1, 2 and 3) was compared to gas production of negative controls with no cellulose (bottles 4 and 5) to estimate the contribution of the inoculum itself to total gas production.

Contribution of cellulose degradation to gas production was estimated by subtracting mean negative control gas production to mean cellulose degrading reactor gas production. Figure 1A presents the contribution of cellulose degradation to methane and carbon dioxide production. After injection of cellulose into the bioreactors, biogas production starts on day 7. Cumulated production levels off after 65 days. Between day 7 and 20, both CO_2 and CH_4 are produced at the same level. After day 20, only CH_4 is produced. In total, 50 mg of carbon is emitted in biogas: circa 50% of the initial cellulose is converted into biogas. 60% of the biogas is composed of methane. Similar observations were made for cellulose degrading bioreactor not co-inoculated with ruminal content.

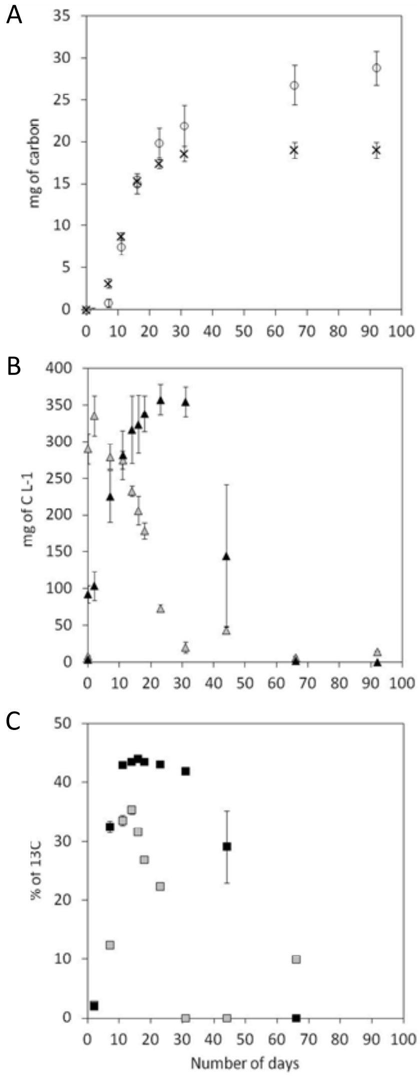

Figure 1: A-Cellulose degradation to methane (circles) and carbone dioxyde (cross) during time production (mean values, error bars represent standard deviation of triplicate bottles 1, 2 and 3) – B – Acetate (grey triangles) and propionate (black triangles) concentrations during cellulose degradation, (mean values, error bars represent standard deviation of triplicate bottles 1, 2 and 3) – C - Isotopic composition of acetate (grey squares) and propionate (black squares) during time (mean values, error bars represent standard deviation of duplicate bottles 1 and 2)

3.2 Intermediate accumulation

DOC, DIC (data not shown) and VFA (figure 1B) concentrations were measured during cellulose degradation (bottles 1, 2 and 3). Respectively 10 mg and 30 mg of carbon were directly provided by propionate and acetate coming from the ruminal content at time zero. Both acetate and propionate were then produced, and acetate was consumed first. Isotopic composition of VFA (Figure 1C) and gas (data not shown) was measured in bottles 1 and 2 to estimate the amount of intermediates coming from cellulose. The intermediates are not 100% ^{13}C enriched, which suggests that they do not come from cellulose degradation only.

Gas isotopic composition was measured on day 16 and 31 (data not shown). Methane is 35% enriched at both dates, which is in good accordance with the values measured for VFA (circa 35% for acetate and 42% for propionate).

3.3 Cellulose degradation

To evaluate the extend of cellulose and ruminal content degradation in our different incubations, mass balance calculations were performed in order to estimate the influence of sampling. Amounts of VFAs coming from cellulose degradation and sampled were deduced from ^{13}C measurements performed on labeled cellulose experiments. These calculations (data not shown) indicated that approx. 8 mg of ^{13}C acetate and 5 mg of ^{13}C propionate were removed with liquid samplings. It was measured that approximately 50 mg of gas were produced during the experiment. Amounts of cellulose and other intermediates sampled cannot be estimated precisely, but it can be concluded that more than 63 mg of the introduced cellulose was catabolized. Moreover, isotopic enrichment of gas and VFAs shows that an important part of ^{12}C was catabolized, demonstrating that a large part of the ruminal content was processed as substrate.

4 CELLULOSE DEGRADING MICROBIAL COMMUNITY DYNAMICS

Total extracted DNAs were analyzed by automated ribosomal spacer analysis (ARISA).

ARISA profiles obtained from the total DNA for *bacteria* and *archaea* are presented on figure 2A and B. The *archaea* ARISA profiles mainly show the increase of the intensity of a band at 400 bp. Archaeal diversity does not evolve a lot. Diversity for *bacteria* is also rather stable over time. Several smooth changes can however be observed in the profiles (Bands A and B). In both cases, bands found in the ruminal content are not observed in these profiles, suggesting that ruminal microorganisms did not survived and settled in the reactors.

A statistical comparison of ARISA profiles of co-inoculated cellulose degrading bioreactor (bottles 1, 2 and 3) and control without co-inoculation of ruminal content (bottles 6, 7, 8 and 9) was realized (total DNA). R package StatFingerprint was used to compute a principal component analysis (PCA) with *bacteria* profiles data (figure 3B). After PCA, two groups of profiles can be clearly distinguished corresponding to co-inoculated and non co-inoculated bioreactors samples. Ruminal content co-inoculation is responsible for a strong modification of bacterial diversity, noticed in ARISA profiles patterns. Same observation is made for *archaea* profiles (figure 3A). Co-inoculation leads to a shift of total diversity equilibrium during cellulose degradation.

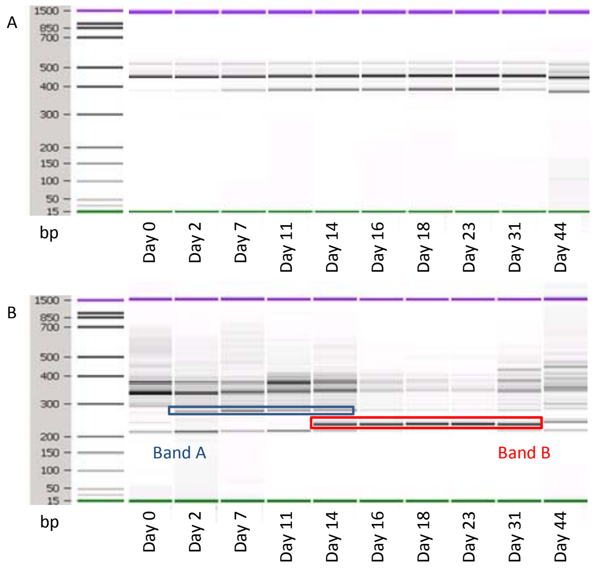

Figure 2: Evolution of archaeal (A) and bacterial (B) ARISA profiles during time for bottle 1. Number of days of incubation is indicated at the bottom of the profiles

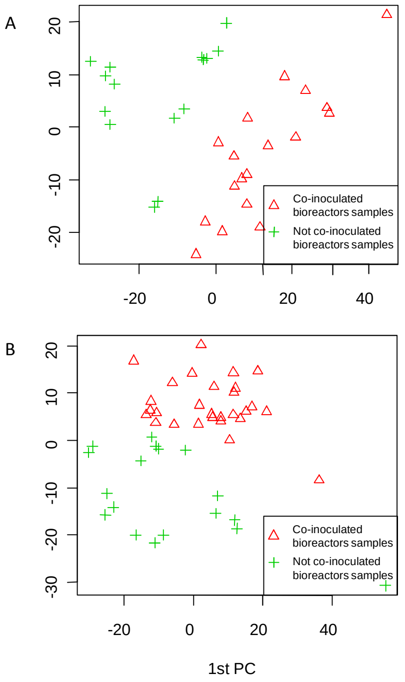

Figure 3: Scaled and centered PCA realized with *Archaea* (A) et *Bacteria* (B) ARISA profiles of samples from co-inoculated (bottles 1, 2 and 3) and not co-inoculated (bottles 6, 7, 8 and 9) cellulose Bioreactors

5 CONCLUSIONS

Even if efficient degradation of cellulose is observed, all the microorganisms initially present in the inocula did not adapt in the microcosms. Both inocula compete for the carrying out of a functional process. Industrial flora is more polyvalent than natural ones and probably resists better to parameters changes. On the contrary, rumen species, highly specialized, did not settle in the microcosms, even if experimental conditions were designed to facilitate implantation of this flora. Other specific parameters are probably needed and should be better taken into account. Yet a clear shift is noticed in ARISA profiles between co-inoculated and non co-inoculated bioreactors samples. Co-inoculation enabled anaerobic industrial digester inoculum diversity modification. The co-inoculation probably disordered diversity equilibrium within the anaerobic industrial sludge, and several ecological niches were taken over by species subdominant in the initial inocula. Co-inoculation of an exogenous flora with anaerobic sludge could thus be an environmental lever to modify diversity in full scale reactors. This study demonstrates that a more precise identification of selection pressures that lead to the stabilization of different types of flora is necessary to use natural flora into industrial systems. Design of processes mimicking animals' digestive systems also seems very promising (Godon, *et al.*, 2010, Bayané & Guiot, 2011).

6 ACKNOWLEDGEMENTS

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Session 31

(180) EVOLUTION OF MICROBIAL POPULATIONS, HUMIC AND FULVIC ACIDS ON COMPOST FROM GRAPE POMACE

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EXECUTIVE SUMMARY

Pisco is a main distillate spirit in Chile and Peru. This product is obtained from the muscatel grape fermentation and it production reach from 30- 50 millions of liters by year, this industry generates a large amount of residues reaching about 12-15 ton/ha, especially in summer and fall. Composting process results a good option to correct disposal of wastes and many microbiological changes provides to final compost, properties to maintain and improve the soil quality in terms of physical, chemical and biological properties.

To evaluate the evolution of microbial populations and enzymatic activities and its relation with the production of humic and fulvic acids, different composting treatments were proposed, using grape pomace (skin and seeds) and bunch raquises, horse and goat manure, and pruning oat residues. There were organized 9 treatments with 3 repetitions each one, using the turned pile system with periodic turning (each 10 days); during a 180-day period at beginning of the temperature stabilization (120 days) the piles were analyzed. It was determinated parameters related with microbial populations (heterotrophic bacteria, fungi and yeast, phosphate solubilizers) and hydrolytic groups: cellulolytic, proteolytic and amilolytic microorganisms. At the same time some enzymatic activity (β glucosidase, phosphatase, and urease), and chemical composition (pH, electrical conductivity, $N\text{-}NH_4/N\text{-}NO_3$ ratio, organic matter, humic acids and fulvic acids) were determined. Main effects and the interactions were evaluated by statistical analysis, comparing means by ANOVA, and correlation were determinated by Pearson's correlation coefficient (p=0,05).

It was possible to quantify the populations of different microorganisms on the compost, which using the organic matter as a complex substrate, produces an important amount of different enzymes that promote the mineralization producing stable forms of carbon as humic substances. Microbial populations as cellulolytic and fungi and yeast reached populations about 8,0 and 6,91 logarithmic units respectively, and presented statistical differences ($p<0.001$) between treatments product of the inherent characteristics of the substrates uses for this co-composting process. After composting process, the organic amendment resulted with 40 -68% OM ($p<0,001$) and grape pomace (91%) and pruning residues/oat straw (5%), which presented high level of organic matter. Most of treatments were slightly alkaline, with pH between 8,0 to 8,5, and others were closer to neutral pH. Results suggest that concentration of humic and fulvic acids at the final of the study could be explained because of the availability of nutrients that incentive the metabolisms microorganisms as fungi and yeast and the production of specific enzymes as β- glucosidase.

Enzymatic activity produced by microorganisms showed a decrease, when populations as heterotrophic bacteria, phosphate solubilizers, cellulolytic microorganisms were lower; in general β - glucosidase and phosphatase decrease on the time, but urease activity tend to increase at the final of the process, it would be explained because of the degradation of organic compounds as proteins on the maturation phase. Statistical analysis, indicated positive and significant correlation between β -glucosidase activity and humic substances production ($r= 0.333$, $p=0.01$), and between fulvic acids and fungi and yeast count ($r=-0.447$, $p<0.001$), heterotrophic bacteria ($r=-0.261$, $p=0.04$), and phosphate solubilizers ($r= -0.396$, $p= 0.04$).

As a conclusion, it was possible to determinate, changes in time, and positive correlations between production of humic acids and heterotrophic bacteria, especially on the co-composting process of grape pomace and goat manure, it suggest that this parameters would be used as quality indicators.

1. INTRODUCTION

1.1. Background

Production of grape represents one of the most important exporter crops in Chile, reaching about to 9.982 ha cultivated on the country; 94% of this production is focused on Coquimbo region, being Muscatel variety the most important with an 86% of the total area cultivated, this grape is processed by the Pisco industry (Bravo, M., 2010).

Composting is a viable option to the treatment of different kind of residues that allow making an approach of the nutritional characteristics and improving the soil quality at the same time. In Chile one of the main environmental problems is related with the Pisco industry, this activity generates large amounts of residues including skin, bunch raquises, and seeds (grape pomace). These residues particularly rich on organic matter (cellulose and lignin), are characterized by a low pH, and electrical conductivity, as well as significant levels of nitrogen, and phosphorus, being the last ones important actors in soil fertility (Bertrán, *et al* 2004).

The compost quality is defined in terms of chemical and physical parameters and presence or absence of human pathogens and fecal indicators. However, also it is possible to include some benefical groups of microorganisms, associated with some hydrolytic activities related with the mineralization of organic matter such cellulose, lignin, protein, or catalytic activity of different enzymes or groups that show the evolution of the compost process in terms of the decomposition of organic matter, nitrogen and phosphorous transformation, and humic substances production. Therefore, final compost is a stabilized, deodorized, safe material for plants and humans, and rich in humic substances (Rallani et al., 2001)

Organic matter, would be classified in non humic substances which include carbohydrates, aromatic and aliphatic amino acids that could be easily degraded by microorganisms, and humic substances (humic acids, fulvic acids and humin) these are more stable compounds, and its degradation depends on the microbial populations present on the composting process, the dynamic of this compound depends on the microbial populations, mediated by microorganisms that play an important role on the soil, they are responsible for many process such as SOM turnover, soil humus formation, cycling nutrients, (Magdoff y Weil, 2004), release plant nutrients from insoluble inorganic forms, improve soil aggregation, aeration and water infiltration.

1.2. Research objectives

The main objective of this research was to evaluate the evolution of microbial populations and enzymatic activities and its relation with the production of humic and fulvic acids

2. METHODOLOGY

The experiment was performed in Ovalle which is located, in the IV region in Chile. Soils are predominantly Alfisols and Inceptisols, with low organic matter (OM) content, and a rainfall regime of approximately 100 mm/year. The material used to carry out the study was obtained from Empresas Bauzá, which generates about 400-600 T of grape pomace for season.

2.1. Composting process

Treatments were proposed depending on the availability of substrates on the region, and were selected based on their nutritional characteristics. Substrates selected included: grape pomace, horse and goat manure, and pruning oat residues, with a total of 9 treatments with 3 repetitions each one (Table 1).

Compost piles (18 m length x 2 m width x 1.5 m height) were composted from June to November 2009 (winter-spring season) and kept in maturity until February 2010 (spring-summer season). The piles were turned mechanically every 10 days, to provide oxygen and to give homogeneity to the system. Moisture content of each pile was adjusted according to manual test, by adding water to maintain optimum composting conditions (approximately 60% moisture). Pile temperatures were measured using thermocouples connected to loggers, and analogue thermometers at top, middle, and bottom locations (data not shown).

Organic matter was incorporated using the turned pile system with periodic turning (each 10 days), during a 180-day period at beginning of the temperature stabilization (120 days), when the temperature declined and stabilization phase started, around 100 days from pile establishment, the materials were analyzed in terms of their chemical composition, physical characteristics as well as microbial activity, every 60 days, until 220 day from establishment.

Table 1. Composition of evaluated treatments

Treat.	HM	GM	GP-Fresh	GP -Aged	PR+OS	Total
	--%--					
1	1	0	89	0	10	100
2	9	7	82	0	2	100
3	0	0	91	0	5	100
4	0	50	50	0	0	100
5	0	63	33	0	4	100
6	22	25	53	0	0	100
7	21	26	21	28	4	100
8	42	20	33	0	5	100
9	0	66	34	0	0	100

Treat: treatment; HM: horse manure; GM: goat manure; GP: grape pomace; YFR: yeast and fermentation residues; PR: pruning residues; OS: oat straw.

2.2. Compost sampling

Samples for microbiological and chemical analysis were collected, when the compost was turned, from three equidistant cross sections to a 60-cm depth from the pile surface, using 2.5 cm diameter PVC tubes (Rodriguez et al., 2007). Ten subsamples were randomly collected and mixed to form a composite sample; three replications per treatment were collected.

2.3. Analytical methodology

Once on the laboratory, the samples were correctly homogenized and divided in two subsamples; one of them was dried at 60°C, for the determination of chemical parameters (pH, electrical conductivity, N-NH_4/N-NO_3) ratio, organic matter, and humic and fulvic percentage), and the other was frozen to microbiological and biochemical parameters.

2.4. Chemical parameters

Electrical conductivity and pH were analyzed in 1:5 (w/v) water-soluble extract (TMECC 04.10; TMECC 04.11), Organic matter was determined by the dry combustion method (Dumas method). NH_4-N/ NO_3-N were determined according with the TMECC 05.02-C method. Fulvic acids were obtained extracting the compost material with 0.5 M NaOH and later after precipitation of humic acids by acidifying the extract below pH 2.0 (Pansu and Gautheyrou, 2006).

2.5. Microbiological parameters

Microbiological populations were quantified by the counting plate method, with serial dilutions in a final volume of 10 ml of sterile saline solution (0.85%), from a previously pre-homogenized mixture of fresh compost and saline solution with a relation of 1:10.

Heterotrophic bacteria populations were determined using the micro drop method on Nutritive Agar (Merck). Fungi and yeast, cellulolytic, amylolytic, proteolytic and phosphate solubilizing bacteria were determined by surface plate count method in specific media: potato dextrose agar (PDA Merck media), cellulose agar, starch agar, milk agar and SMRS1 agar, respectively (Martinez et al., 2010; Caballero et al., 2007); for the cases of the amilolytic and cellulolytic groups, it was necessary to reveal the activity by the addition of lugol (Merck) and congo red 1% (m/v), respectively (Martinez et al., 2010). Microbial colonies were counted as colony forming units per fresh gram (CFU g^{-1}.).

2.6. Biochemical parameters

The ureases were extracted following the method of Kandeler and Gerber (1988) and their activity measured by the indophenol blue method; it was expressed in terms of urease units (UU = µg NH_4 released per gram of dry sample per hour) (García et al., 2003). The acid and alkaline phosphatases were determined using p-nitrophenyl phosphate (Sigma) as substrate, and measuring the product p-nitro phenol to determine the phosphatase units (UP = µg p-nitrophenol released per gram of dry sample per hour) (García et al., 2003). β-glucosidases were also evaluated by measuring the p-nitrophenol released, but in this case, from the substrate p-β-D-glucopyranoside (Sigma), expressing the results in terms of β-glucosidase units (UBG = μg p-nitro phenol released per gram of dry sample per hour) (García et al., 2003).

2.7. Statistical analysis

Data obtained from the study were analyzed by multivariate analysis to establish correlations between different parameters ($p>0.05$), in SAS (SAS Institute, 2000), main effects and interactions were evaluated by analysis of variance and Protected LSD ($p<0.05$).

3. RESULTS AND DISCUSSION

3.1. Chemical parameters

Chemical parameters were compared with NCh 2880/04, which is the national normative that regulates compost quality on Chile. The pH of the treatments in the all cases required the normative; and tends to be alkaline. The differences on pH presented by treatments would have a direct relation with the different substrates used to the process. As some authors have reported, grape residues are characterised by an acidic pH, low electrical conductivity (EC) values and high organic matter (OM) (Bustamante *et al* 1998; Bertrán *et al* 2004). Another factor that influences is the initial pH, Miller *et al* 1993, suggested that a pH between 6-8 as suitable for composting, and the increment in the pH in the material could be attributed to the degradation of acid- type compounds, such as carboxylic groups, and to the mineralisation of proteins, amino acids and peptides to ammonia. In contrast the decrease of this parameter would be due to the generation of acid-type organic compounds of low molecular-weight via the decomposition of the most-easily-degradable OM fraction. (Bustamante 2008). Electrical conductivity increases on the time because of the organic matter degradation due to the production of inorganic compounds and the increasing relative concentration of ions due to the mass loss of the pile, as described by Paredes *et al*., 2002. (Figure 1 A), the treatment 4, 5, 6, 8 and nine does not complain this parameter with NCh 2880/2004, exceeding in some cases more than two units form the allowed (3mS/cm).

According to the Chilean standard NTCh 2880/04, for compost quality, the $N\text{-}NH_4/N\text{-}NO_3$ ratio is used as indicative of compost maturity, establishing the limit on 3. Treatments 6 and seven presented a high $N\text{-}NH_4/N\text{-}NO_3$ ratio; it indicates that these treatments did not reach a maturation phase; it would be explained by an imbalance on the C/N ratio, that generates an excess of N per degradable C, also inorganic N is produced in excess and can be lost by ammonia volatilization or by leaching from the compost (Bernal *et al* 2009). Other treatments as 3, 4, and eight, presented similar values to obtained from analysis made at the laboratory (data not shown), to compost produced with similar characteristics on Chile, which are on the range from 0,108 to 0,402, that complies the normative on Chile. Microorganisms requires an energy source (degradable organic-C) and N for their development and activity, it is well know that an adequate C/N ratio for composting is in the range 25–35, because it is considered that the microorganisms require 30 parts of C per unit of N. As reported by Bernal *et al* in 2009, mineralization of organic compounds especially during the thermophilic phase favors the formation of $N\text{-}NH_4$, is higher than other phases. On the other hand nitrification process occurs when the temperature falls below thermophilic values, and depends directly on the amount of N-NH4 available to the nitrifying bacteria, most of the nitrification occurs during maturation, leading to a low NH4-N/NO3-N ratio in mature compost (Bernal *et al* 2009).

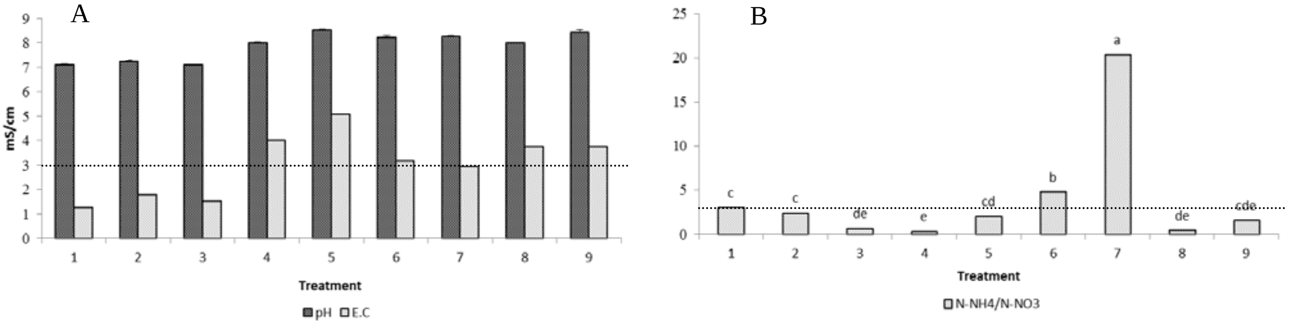

Figure 1. Chemical parameters: (A), pH and electrical conductivity, (B) N-NH4/N-NO3 ratio, at 220 days of the process, dotted line remark standard defined on the NCh 2880/04 to electrical conductivity (A), and N-NH4/N-NO3 ratio (B)

On the other hand, determinations of organic matter, were compared to the Chilean standard, resulting above of the range established for this parameter (<=20, INN, 2004), the treatments presented statistically significant differences, as described on figure 2B. These results could be explained by the availability of nutrients on the pile; when the most labile organic matter constituent as sugar, proteins and hemicelluloses have been degraded, more complex compounds contained on organic matter become available by enzymatic attack product of microbial metabolism, it generates a gradually decrease, the residual organic matter is transformed forming humic-like substances (humification process), a new and stable form of organic matter (Magdoff y Weil, 2004).

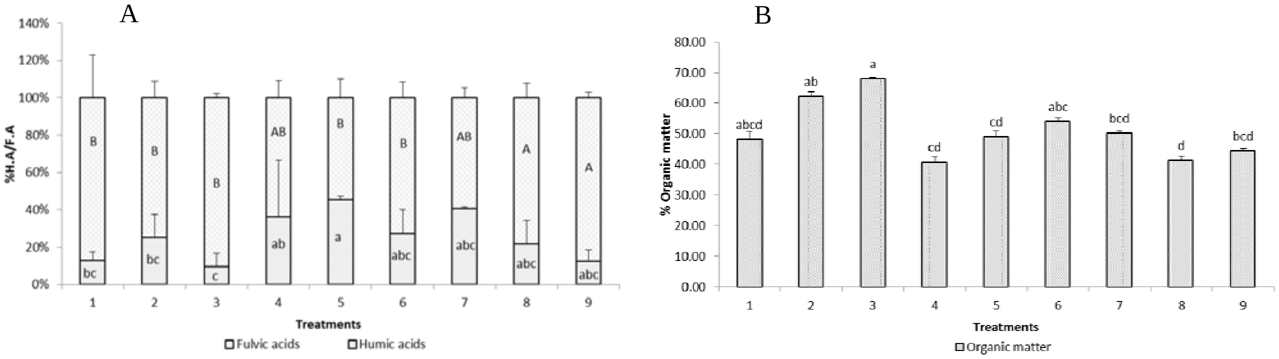

Figure 2. Chemical parameters: (A) Humic and fulvic acids, (B) Organic matter at 220 days of the process.

The concentration of humic acids on the first stage of maturation increased, while fulvic acids tend to decrease, in the second phase fulvic acids increase (Figure 3), it is related with the origin of the substrates; as reported by Bustamante *et al* in 2008, this could be explained due to the different character of the OM contained in the materials used, fresh materials as manure, contains lower concentrations of humic acids but higher contents of fulvic acids. Moral *et al* (2005), determined that manures form horse and goat present higher concentrations of easily-biodegradable organic compounds than other, manures used on composting process. It is expectable that during the maturation phase stabilization and humification of the OM occur, producing humic substances more resistant to microbial degradation, this parameter would useful to determinate compost quality (Bernal *et al* 1998).

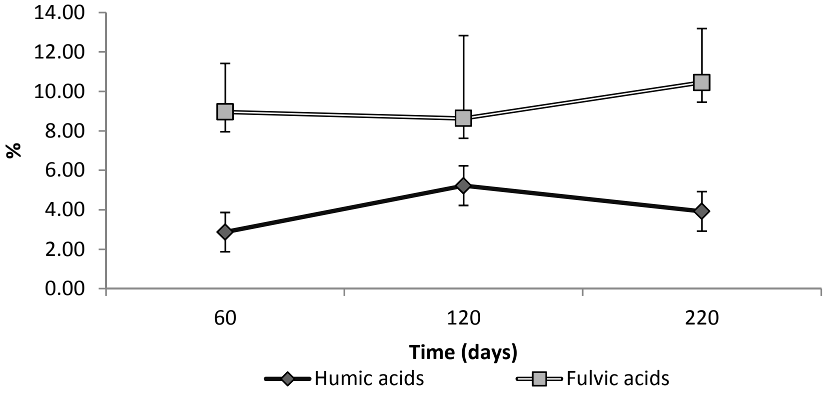

Figure 3. Average of humic and fulvic acids at the 220 days

3.2. Microbiological and biochemical parameters

As described by Bernal *et al* 2009, control of parameters such as nutrient content, C/N ratio, temperature, pH, moisture and oxygen supply have demonstrated to be key for composting optimisation since they determine the optimal conditions for microbial development; favouring the multiplication of beneficial microorganisms which have a potential when applying on soil.

On this study microbial populations were quantified following the methodology previously described, the results obtained at the final time, are specified on the table 2. The changes on microbial populations in compost depends on the different phases involved on its production, it have a direct relation with the availability of nutrients that stimulate the production of specific enzymes, heterotrophic bacteria, fungi and yeast, and cellulolytic microorganisms presents significant differences between treatments, the presence of this beneficial microorganisms would increase the populations in soil that improves the soil quality, promotes cycling nutrients, and have a potential in biological control (Magdoff y Weil, 2004).

Treatments 1 and 3, with high content of grape pomace, increased specific metabolic groups of microorganisms as cellulolytic and fungi and yeast, it would be related with the endogenous characteristics of grape: skin, seeds and bunch raquises are rich on cellulose and lignin; it favors the proliferation of microbial populations that produces specific enzymes for its degradation as fungi and cellulolytic microorganisms. As reported by Sanchez (2009), fungi are more important for cellulose degradation than bacteria, especially when cellulose is encrusted with lignin; the degradation of lignin is primarily accomplished by fungi.

On the other hand, temperature plays an important role on the dynamic of populations, when the temperature of the pile decreases after thermophilic phase, mesophilic microorganisms recolonize the substrate, either originating from surviving spores, or external inoculation. During the maturation phase, the quality of the substrate declines, and microbial populations are altered, usually the proportion of fungi increases, while bacterial numbers decline. (Díaz *et al* 2007).

Table 2. Microbiological characteristics of materials after 180 days of maturation phase.

Treatment	HB	FY	PSM	CM	AM	PM
	--- Log (CFU*g-1)---					
1	5,93±0,04	7,15±0,21 **ab**	5,83±0,18	6,36±0,12 **a**	6,49±0,64	5,93±0,04
2	6,36±0,26	5,24±0,34 **abc**	5,77±0,10	6,80±0,80 **ab**	6,70±0,12	5,89±0,16
3	5,29±0,16	8,07±0,05 **a**	6,15±0,21	5,59±0,83 **ab**	6,22±0,11	6,48±0,67
4	4,73±0,11	6,74±1,04 **abcd**	4,69±0,30	6,91±0,29 **ab**	7,01±0,38	6,15±0,21
5	4,50±0,14	6,15±0,21**cd**	6,42±1,33	5,54±0,09 **b**	5,87±0,12	5,65±0,49
6	4,55±0,21	5,42±0,60 **bcd**	5,09±0,08	5,36±0,08 **b**	5,21±0,19	6,15±0,21
7	5,09±0,04	6,57±0,8 **bcd**	5,00±0,00	5,69±0,12 **b**	5,65±0,47	5,39±0,12
8	5,85±0,21	6,01±1,43 **bcd**	5,30±0,43	6,43±0,07 **ab**	6,30±0,00	4,98±0,71
9	5,00±0,00	5,15±0,21**a**	6,54±0,34	6,23±0,07 **b**	6,35±0,49	6,81±0,47

HB= Heterotrophic bacteria, FY= Fungi and Yeast, PSM= Phosphate solubilizing microorganisms, CM= Cellulolytic microorganisms, AM= Amylolytic microorganisms PM=Proteolytic microorganisms. Different letter means statistical differences. P>0,001

Bacteria are the most populous microbial group in compost and are responsible for most of decomposition; they proliferate faster that other species, and predominate early, because they grow faster than other microorganisms, as fungi, or actinomycetes; Heterotrophic populations, include mesophilic populations and thermophilic populations, which are able to grow on mesophilic conditions too. During the initial stage of decomposition readily available substrates as proteins, sugars and starch are rapidly oxidized, being approached to generate energy and biomass. On the other hand microorganisms as actinomycetes and fungi, are commonly associated with the degradation of cellulose, hemicellulose, and lignin, and predominate during stabilization.

Organic matter provides the principal substrates for the microorganisms, compost pile is characterized by high substrate density and diverse and highly interactive microbial populations that go through a succession of populations dominated by first by mesophilic organisms and then by thermophiles, the number of actinomycetes and fungi rise the high count during the thermophilic phase, bacteria peak in numbers as the compost starts to cool.

Cellulolytic microorganisms showed differences among treatments (p=0,001). This group of microorganisms is related with the availability of cellulose as substrate, in the ending phase of the process cellulose would be associated with another recalcitrant compounds such as lignin, at the end of the composting process the cellulose content is low, it generate a decrease on cellulolytic populations (Díaz *et al* 2007). Other microorganisms associated with the production of specific enzymes as proteolytic, amylolytic does not present differences between treatments, these microorganisms present the highest counts after the thermophilic phase, because of the degradation of more complex substrates.

It was determined high quantities of populations of yeast and fungi on the different treatments, these microorganisms are usually present on grape residues (specifically on grape, bunch and raquises), and its growth would be favors by the low initial pH. On the other hand fungi contributes to the formation of organic matter, upon decomposition by saprophytic fungi, elements such as nitrogen and phosphorous are transformed from available organic compounds to available inorganic nutrient sources (Magdoff y Weil, 2004), and contribute to degradation of complex compounds from substrates.

On the other hand microbial metabolism generates an important transformation of organic matter, by the action of extracellular hydrolytic enzymes as cellulose, β-glucosidase, and as reported by Díaz *et al*., (2007), an increasing levels of organic matter, can enhance the diversity of the microbial populations as well as it biomass, it influence directly the dynamic of microorganisms on soil. Enzymes are produced by a wide range if microorganisms, and are important to depolymerize complex compounds, in this case for every composting treatments, the highest values of acid phosphatase were detected at the beginning of the process. The decrease on β-glucosidase production, at the final of the composting process is justified as a consequence unavailable of organic substrates, as reported by Cunha-Queda 2006 (Figure 4)

Not significance differences were determinated between treatments, for ureases, and β-glucosidase, but phosphatases as alkaline and acid were evident.

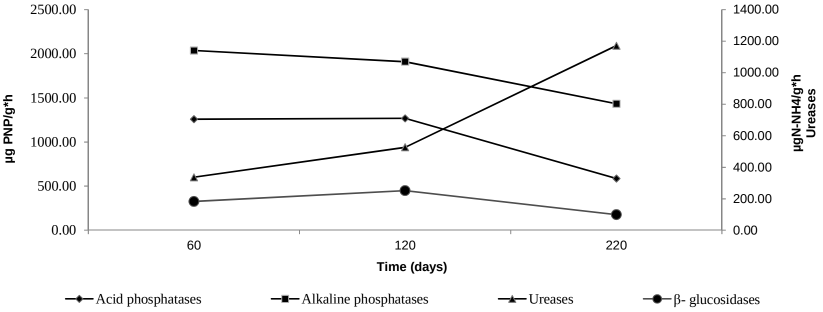

Figure 4. Average of enzymatic activities: β- glucosidase, acid and alkaline phosphatases (µg PNP/g*h), and ureases (µg N-NH4/g*h) at three times.

Data obtained from the study were analyzed to establish correlations ($p>0.05$). Significant positive correlations were determined by β- glucosidase activity on the production of humic acids as described on Table 2. As reported by Knight and Dick in 2004, enzymes as β- glucosidases and phosphatases can be stabilized in humic substances, these immobilized enzymes appear to be protected against denaturation by both proteolytic enzymes and heat, on the other hand extracellular enzymes plays an important role on the degradation of a wide range of substrates. During composting compounds such as protein, cellulose and hemicellulose are utilised by microorganisms as C and N sources, the residual plant organic matter, along with compounds of microbial origin, is transformed by microorganisms to form humic-like substances of increasing complexity (Mondiny *et al* 2004).

Alkaline and acid phosphatases, presented a positive correlation ($r=0.602$, $p>0.001$ Benitez *et al* 1999, they determinated a close relationship between hydrolytic enzyme activities during the vermicomposting of sewage sludge on a laboratory scale. Moral *et al* (2005), reported that concentration of easily- biodegradable organic compound was significantly greater in horse and goat,

The correlations determined by the statistical analysis between heterotrophic bacteria and different functional groups as cellulolytic ($r=0.445$, $p>0.001$), proteolytic ($r=0.514$, $p>0.001$), amilolytic microorganisms ($r=0.491$, $p<0.001$), phosphate solubilizers (r=0.396, p>0.001), would be explained because of the metabolic specificity that presented each functional group. Microorganisms preferentially utilized soluble and easily-degradable carbon sources contained on starting material; however, once the easily-degraded compounds were exhausted, the complex substrates such as lignocellulosic materials started to be degraded, resulting in the increase in lignocellulolytic microbial communities (Dongyang *et al* 2011), it generates a microbial succession that promote the proliferation of new microbial populations on compost (Ishii et al., 2000). Other interesting correlation was found between, HB and the enzymes determined on this study, this group of microorganisms, include not only mesophilic populations but thermophilic too, which are able to grow under mesophilic conditions on the count; some bacterial populations produce a wide range of enzymes related, with the different substrates available on the all process, including early and later stages. Probably as heterotrophic bacteria are predominant on the all process the quantification of these populations would be an indicative of the general enzymatic activity (Table 2),

Characterising and quantifying enzymatic activities during composting can reflect the dynamics of the composting process in terms of the decomposition of organic matter and nitrogen transformations, and may provide information about the maturity of composted products.

Treat	AC P	ALK P	U	β-GLU	HB	FY	PSM	CM	PM	AM	HA	FA	OM
Treat													
AC P	1												
ALK P	**.602**	1											
UREASE	**-.249**	-.156	1										
B-GLU	.222	.234	**-.381**	1									
HB	**.416**	**.271**	**-.566**	**.652**	1								
FY	**.234**	**.251**	-.071	-.197	.133	1							
PSM	.167	**.256**	-.011	.175	**.396**	**.274**	1						
CM	**.450**	**.443**	**-.445**	.232	**.445**	.203	.208	1					
PM	.219	.175	**-.291**	**.334**	**.514**	**.309**	**.469**	**.283**	1				
AM	**.244**	.177	-.032	.182	**.491**	**.368**	**.562**	**.369**	**.414**	1			
HA	-.202	-.194	-.030	**.333**	.082	-.190	.075	-.130	.020	.111	1		
FA	**-.253**	-.084	**.270**	.029	**-.261**	**-.447**	**-.266**	-.132	**-.335**	-.202	.142	1	
OM	.281	.036	.068	-.259	.113	**.387**	.288	-.165	.269	.207	**-.337**	**-.455**	1

Table 2. Significant correlation coefficients are bolded. ACP= Acid phosphatases, ALK P=Alkaline phosphatases, U= Urease, B-GLU=β-glucosidase, HB= Heterotrophic bacteria, FY= Fungi and Yeast, PSM= Phosphate solubilizing microorganisms, CM= Cellulolytic microorganisms, AM= Amylolytic microorganisms PM=Proteolytic microorganisms, HA=Humic acids, FA= Fulvic acids, OM=Organic matter.

4. CONCLUSIONS

It was possible to quantify the populations of different microorganisms on the compost, which using the organic matter as a complex substrate, produces an important amount of different enzymes that promote the mineralization producing stable forms of Carbon as humic substances.

The results suggest that concentration of humic and fulvic acids at the final of the study could be explained because of the availability of nutrients that incentive the metabolisms microorganisms as fungi and yeast and the production of specific enzymes as β glucosidase. Statistical analysis, indicated positive correlation between β- glucosidase activity and humic substances production (r= 0.333, p=0.01), fulvic acids were correlated with fungi and yeast count (r=-0.447, p<0.001), heterotrophic bacteria (r=-0.261, p=0.04), and phosphate solubilizers (r= -0.266, p= 0.04). As a conclusion associated with the composting process, we conclude that goat manure and grape pomace fresh where the residues that presented the highest production of humic acids (8.6%), favoring the production of stables forms of Carbon, product of humification process.

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(197) LONG-TERM PERSISTENCE OF AMINOCYCLOPYRACHLOR AND CLOPYRALID DURING THE COMPOSTING OF YARD TRIMMINGS

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EXECUTIVE SUMMARY

Certain pyridine carboxylic acid herbicides, including clopyralid, aminocyclopyrachlor and, aminopyralid that have recently been introduced, have been found to be recalcitrant in soil and composts. These herbicides are generally used for broadleaf weed control in pastures, lawns and natural areas. Certain types of plants, including tomatoes and beans are very sensitive to residues of these compounds even at very low concentrations. To better understand the impact of the use of these compounds on composts, we determined the persistence of granular and liquid formulations of aminocyclopyrachlor (tradename imprelis), and clopyralid during long-term composting of mixtures of grass and leaves. Herbicide treated turfgrass plots were established that received one of four treatments: no herbicide, aminocyclopyrachlor granular application (84 g AE/ha), aminocyclopyrachlor as liquid spray (84 g AE/ha), or clopyralid (209 g AE/ha) as a liquid spray. Grass clippings were collected from each plot within 7 days after herbicide application. The treated grass samples were mixed with leaves (2:1 L:G ratio) and composted under conditions that simulated commercial windrow composting conditions for 178 days. A system of triplicate 4 liter compost reactors were used each containing 1.2 kg of wet compost. The reactors were incubated at 55ºC (±2 ºC). Aeration was maintained at 100 ml/min and CO_2 concentrations were measured hourly. The grass and leaf samples, plus samples removed from the reactors on days 0, 10, 30, 50, 78, 100 and 178, were analyzed for pH, moisture, ash, volatile solids, total carbon and nitrogen, and aminocyclopyrachlor and clopyralid concentrations. Results showed that 58 and 47 % of the aminocyclopyrachlor was lost after 178 days of composting from the liquid and granular formulations respectively. By comparison, 70% of the clopyralid was lost over the same period. A second experiment in large scale reactors (75 kg compost) with aminocyclopyrachlor at two different concentrations showed similar results. To evaluate their phytotoxicity, each of the final composts were used at 4, 10 and 20% amendment levels in potting media into which seeds of bean and tomato were sown. Composts made from yard trimmings that did not receive a herbicide application and ProMix without compost were used as controls. Results showed that none of the composts had a significant effect on tomato or cucumber seed germination. However at the 10 and 20% levels, bean germination was reduced significantly by more than 50%. The aminocyclopyrachlor containing composts reduced plant height, shoot dry weight and leaf area of both bean and tomato to some degree. Various symptoms of herbicide toxicity were evident on the affected plants. Beans were more sensitive to imprelis while tomatoes were more sensitive to clopyralid. These results indicate that in certain cases, imprelis or clopyralid applied to grass may not degrade to a sufficient extent during long term composting to be used in gardens or in plant growth media.

1 INTRODUCTION

Composting is widely used for the management and recycling of yard trimmings including leaves, grass and brush. Composting generates an organic soil amendment that is used to improve soil structure, provide plant nutrients and improve soil physical, chemical and biological properties. Other feed stocks commonly used for composting include manure, biosolids, and agricultural residues. All of these feed stocks may contain various synthetic organic compounds including herbicides (1, 2, 3, 4). One of the greatest concerns with the use of composts, especially in gardening and nursery media, is the persistence of these compounds.

Most studies have shown that composting normally is very effective at reducing herbicide residues below concentrations that would be harmful to plants or animals. For example it has been reported that the commonly used pesticides diazinon, pendimethalin and 2,4-D are degraded to low levels during the composting of yard trimmings to which they have been recently applied (8, 9, 10). Other studies have reported that although herbicide residues are found in composting feed stocks at commercial facilities, they are degraded to low levels during composting and rarely detected in finished composts (3, 4, 8, 9).

Recently, certain pyridine carboxylic acid herbicide residues have been found in finished composts. These residues include clopyralid and picloram which are used for broadleaf weed control in pastures and lawns (1, 2). Aminopyralid has also been reported to be persistent in composts (11). Certain types of plants, including tomatoes and beans are very sensitive to these residues even at very low concentrations (< 50 ppb). The use of clopyralid has since been limited to licensed, commercial applicators. However, because of the resistance of these herbicide residues to breakdown and their effects on plant growth at low concentrations, it has become important to test the biodegradability and persistence of novel herbicides during the composting process to avoid future environmental and plant health problems.

Aminocyclopyrachlor (6-amino-5-chloro-2-cyclopropylpyrimidine-4-carboxylic acid) is a new herbicide sold as imprelis under development for the control of a wide range of broadleaf weed species and as a potential replacement for clopyralid and aminopyralid. Currently two different application formulations of imprelis are available; a granular formulation and a liquid formulation. Since these compounds likely would be applied to lawns and grass, their fate during grass composting is an important consideration.

1.1 Research objectives

The objective of this study was to evaluate the persistence of aminocyclopyrachlor (imprelis) during long term composting of grass. Treated grass was mixed with leaves and composted for 178 days under conditions that simulate commercial composting conditions for yard trimmings. The degradation of aminocyclopyrachlor during composting was compared to that of clopyralid.

Another objective was to determine the phytoxicity of composts made from grass to which aminocyclopyrachlor and clopyralid has been applied.

2 METHODOLOGY

2.1 Feedstocks

Leaves were collected during fall, from the Ohio Agricultural Research and Development Center’s surroundings at the Ohio State University, then were dried at room temperature (20°C) and ground in a Glen Mills model HM3000 mill using a 9.5 mm screen. They consisted of a mixture of oak, maple, ash and other hardwood species.

2.2 Herbicide application

Experimental plots were established to provide herbicide-treated turf grass clippings for composting. Twelve plots each received one of four treatments: no herbicide (untreated control), aminocyclopyrachlor in a granular form, aminocyclopyrachlor as liquid spray or clopyralid as a liquid spray. The granular form was applied using a rotary spreader calibrated to deliver 84 g ae ha^{-1} of aminocyclopyrachlor traveling 4.5 km h^{-1}. The granular treatment was irrigated into the turf grass within 24 hours after application. Liquid aminocyclopyrachlor and clopyralid were applied using a Solo® backpack sprayer with a constant flow valve and one nozzle. Traveling 4.5 km h-1, this system sprays at 1 kg cm^{-2} to deliver a spray volume of 224 L ha^{-1}. Aminocyclopyrachlor was applied at 84 g ae ha^{-1}, and clopyralid was applied at 209 g ae ha^{-1}.

2.3 Grass clippings collection

Clippings were collected from each plot after herbicide application to allow for maximum absorption into the grass blades. The interval between application and clippings collection was 7 days for the granular application and 1 day for the liquid applications. Clippings from each treatment were collected using separate gas-powered, push-type mowers (53.3 cm cut) with a clippings collection bag. After collection, all clippings samples were refrigerated until delivery to the composting facility the following day. The twelve fresh turf grass samples were transported by car to the OARDC in Wooster as 1 kg chilled samples. The samples consisted of clippings samples from each replicate plot of the four treatments. They consisted of untreated grass (U), grass treated with aminocyclopyrachlor in a granular formulation (G), grass treated with aminocyclopyrachlor in a liquid formulation (L) and grass treated with clopyralid (C).

2.4 Compost mix preparation

Moisture contents of leaves and grass were determined and they were mixed at a ratio of 2:1 (L:G dry-weight basis) to simulate the typical annual mix ratio received by large scale composting facilities (6). Deionized water was added to achieve a moisture content of approximately 65%. There were a total of 12 reactors corresponding to 12 turf grass samples (4 treatments x 3 replicates).

2.5 Reactor conditions

A 4-liter bench scale composting system was used that contained approximately 1.2 kg of wet compost per reactor (5). All the treatments were incubated for a period of 178 days at a temperature of 55ºC (±2 ºC). Aeration was maintained at 100 ml/min to each reactor to ensure aerobic conditions over the duration of the experiment. The airflow rate was controlled using a flow restrictor system and monitored daily. The CO_2 concentrations were measured hourly in each reactor and recorded using a data logger. Temperatures inside the reactors were recorded every 10 minutes using thermocouples and recorded using a data logger (5).

2.6 Mixing and sampling

Compost samples for analysis were removed from reactors on days 0, 10, 30, 50, 78, 100 and 178. The reactors were weighed before and after removing samples at each sampling interval to calculate wet-weight loss during composting. The reactor contents were mixed before taking samples in a plastic bucket at each sampling date. Approximately 15 g of samples were dried in porcelain cups at 70°C for 24 h to determine the moisture and were placed in an oven at 550 °C for 4 h to determine ash content of each mix at each sampling date. Samples at day 0 and 178 were also collected and submitted to the Soil Testing and Research Laboratory, at the OARDC, a fully equipped and certified soil and compost analytical laboratory. Standard analytical procedures were used to determine volatile solids, pH, initial and final phosphorous, potassium, total nitrogen and total carbon contents.

2.7 Data analysis

Means and standard deviations for triplicate treatments were calculated for temperature and CO_2 during composting using data retrieved from the data logger. Means and standard deviations were also calculated for other composting parameters such as moisture, volatile solids, nitrogen content, carbon content, herbicide concentrations.

2.8 Analysis of herbicide concentration.

For the analysis of aminocyclopyrachlor, 50 g samples from reactors containing untreated, granular and liquid application grass, were collected on day 0, 10, 30, 50, 78, 100 and 178. These samples were shipped to MPI Research, Inc. 3048 Research Drive, State College, Pennsylvania, USA where they were analysed for the residues of aminocyclopyrachlor. Likewise, 50 g samples from reactors containing clopyralid treatment mix were collected on day 0, 10, 30, 50, 78, 100 and 178 and were sent to Anatek Labs, Inc., Moscow, Idaho, USA for the analysis of clopyralid.

2.9 Phytotoxicity testing

After composting, the composts were mixed with ProMix growth media at 4, 10 and 20% by dry weight. Into these media were sown tomato and bean. The pots had no drainage holes to prevent herbicide leaching and were incubated in a greenhouse. Seed germination and plant growth (stem length, dry weight, leaf area) and other properties were assessed over 35 days. Five replicate pots were used for each treatment.

3 RESULTS AND DISCUSSION

The moisture contents of the treated grass samples were 76.6 % (±0.9), respectively. The initial C:N ratios of the compost mixes were approximately 25:1 for all treatments and the initial moisture contents were near 68% after adjustment with DI water (data not shown). These levels are near ideal for aerobic composting (7). During composting, moisture levels decreased in final samples. The pH of the initial and final mixes was nearly neutral similar to that typically found during commercial yard trimmings composts (6, 7). The fact that neutral pH levels were maintained indicates that the composts remained aerobic throughout the experiment. Volatile solids and carbon content decreased overtime whereas N, P and K increased in final compost samples as a result of dry matter loss.

The temperatures of the composts rose to 65°C after the first 2 days, but remained within 1-2°C of the incubator set point of 55°C throughout the remainder of the experiment for all reactors. No significant difference was observed among the four treatments in terms of compost mix temperature. The results indicated that all of the composts produced CO_2 at a considerable rate. The CO_2 evolution rates of the mixes were nearly the same for the first 10 days. At the end of the experiment, the mean carbon loss to CO_2 from the composts was 48% for the untreated compost, 53% for the composts with the granular herbicide, 51% for the composts with the liquid herbicide and 48% for the compost treated with clopyralid. These differences were not significant among treatments.

Herbicide concentrations and losses during composting were calculated on a wet, dry and ash basis (Table 1). During composting, moisture content fluctuations can occur which can cause variability in herbicide concentrations on a wet basis. In addition since substantial dry weight loss occurs as a result of carbon loss, herbicide concentrations on a dry weight basis are not a good estimate of the extent of biodegradation. However the concentration of the herbicide on a dry weight basis is important from a compost utilization standpoint. To determine absolute amount of herbicide loss, herbicide concentration on an ash basis was calculated. It was assumed that the total ash amount remained unchanged during the experiment since no leachate losses were allowed to occur. This enabled the estimation of herbicide loss during composting.

On a wet weight basis, the initial concentrations of aminocyclopyrachlor in the composts ranged from 1.3-1.9 mg/kg for the granular form and 3.4-4.2 mg/kg for the liquid form. On a dry weight basis these translated into a range of from 4.5– 6.4 mg/kg for the granular form and 12 to 14.6 mg/kg for the liquid form. The initial clopyralid concentrations ranged from 30-38 mg/kg on a wet weight basis and 101 to 133 mg/kg on a dry weight basis (Table 1). During composting the concentrations of aminocyclopyrachlor and clopyralid increased and/or decreased on a wet or dry weight basis for the reasons mentioned above (Table 1). On a dry weight basis the concentration of aminocyclopyrachlor in the granular form declined by 8%, in the liquid form it declined by 20% and the concentration of clopyralid declined by 49% (Table 1).

On an ash weight basis, results indicated a significant decrease in concentration for both formulations of aminocyclopyrachlor and for clopyralid (Table 1). The average initial concentration of aminocyclopyrachlor was 37 and 85 mg/kg ash for the granular and liquid treatments, respectively. The average final concentrations were 20 and 37 mg/kg ash for granular and liquid applications, respectively. Thus 47% of the granular aminocyclopyrachlor and 57% of the liquid aminocyclopyrachlor were lost after 178 days of composting (Table 1). Initial and final clopyralid concentrations were 661 and 194 mg/kg ash respectively, indicating that 71 % of the original clopyralid was lost after 178 days composting (Table 1).

TABLE 1. Concentration of aminocyclopyrachlor (granular and liquid) and clopyralid during composting.

	Treatments (Values are mg/kg)									
Sample day	Untreated	Granular Herbicide			Liquid Herbicide			Clopyralid		
	wet	wet	dry	ash	wet	dry	ash	wet	dry	ash
Day 0	NT	1.5±0.3	5.2±1.0	37.5 ±8.1	3.8±0.4	13.4±1.2	85.2 ±15.1	34±4	112±14	661 ±54
Day 1 0	NT	2.0±0.4	6.9±1.5	37.8 ±7.7	4.7±0.9	17.1±2.5	82.9 ±19.0	44±5	142±20	637 ±60
Day 30	NT	2.9±0.5	6.9±1.6	34.6 ±8.0	6.5±1.4	16.7±2.2	73.8 ±17.8	47±1	99±2	413 ±13
Day 50	NT	3.0±0.5	6.5±1.6	31.2 ±7.8	6.7±1.4	17.2±2.9	69.7 ±18.5	53±4	104±12	403 ±51
Day 78	<0.01	3.5±0.5	6.3±1.5	28.9 ±6.2	6.6±1.6	16.1±2.0	62.8 ±14.3	49±4	81±16	305 ±56
Day 100	<0.01	2.8±0.5	6.5±1.6	28.7 ±7.5	6.6±1.2	15.3±2.1	59.4 ±13.8	50±4	109±15	395 ±34
Day 178	NT	2.5±0.7	4.8±1.6	20.0 ±6.2	6.9±1.2	10.7±2.2	37.0 ±12.1	31±2	57±3	194 ±16
% Loss		+67%	-8%	-47%	+82%	-20%	-57%	-9%	-49%	-71%

Values are averages of triplicate reactors ± standard deviation. NT = Not tested

Each of the final composts was used at a 4, 10 and 20% amendment rate in a potting media into which seeds of bean and tomato were sown. Composts made from yard trimmings that did not receive a herbicide application (U) were used as controls. The herbicide concentrations in the media overall due to the composts are shown in Table 2.

TABLE 2. Amount of composts added to potting media and calculated residual herbicide concentrations in the potting media.

	Growth Media Concentration (ug/kg dw)		
Compost % (dw)	Aminocyclopyrachlor		Clopyralid
	Granular (G)	*Liquid* (L)	*Liquid* (C)
4%	192	428	2,280
10%	480	1,070	5,700
20%	960	2,140	11,400

Results showed that none of the composts containing residual herbicides (G, L, C) or the controls (U, PM) had a significant effect on tomato germination (Fig. 1). However bean germination was reduced significantly in potting mixes containing 20% of aminocyclopyrachlor contaminated composts (G20 and L20).

Clopyralid residue containing composts (C4 and C10) significantly reduced tomato plant height and leaf area but not dry weight (Fig. 1). However, these composts (C4, C10) had no significant effects on bean (Fig. 2). Composts containing the granular formulation of aminocyclopyrachlor at the 10 and 20% levels (G10 and G20) reduced the dry weight of tomato plants (Fig. 1) and plant height, leaf area and dry weight in beans (Fig. 2). All of the liquid aminocyclopyrachlor containing composts (L4, L10 and L20) significantly reduced bean plant height; leaf area and plant dry weight (Fig. 2). The effects of L4, L10 and L20 composts on tomato plants was similar but somewhat less pronounced (Fig. 1).

Various symptoms of herbicide toxicity were evident on the affected plants such as leaf cupping and loss of apical dominance. Overall, beans were more sensitive to aminocyclopyrachlor while tomatoes were more sensitive to clopyralid.

These results indicate that in certain cases, imprelis or clopyralid applied to grass may not degrade to a sufficient extent during long term composting to be used in gardens or in plant growth media.

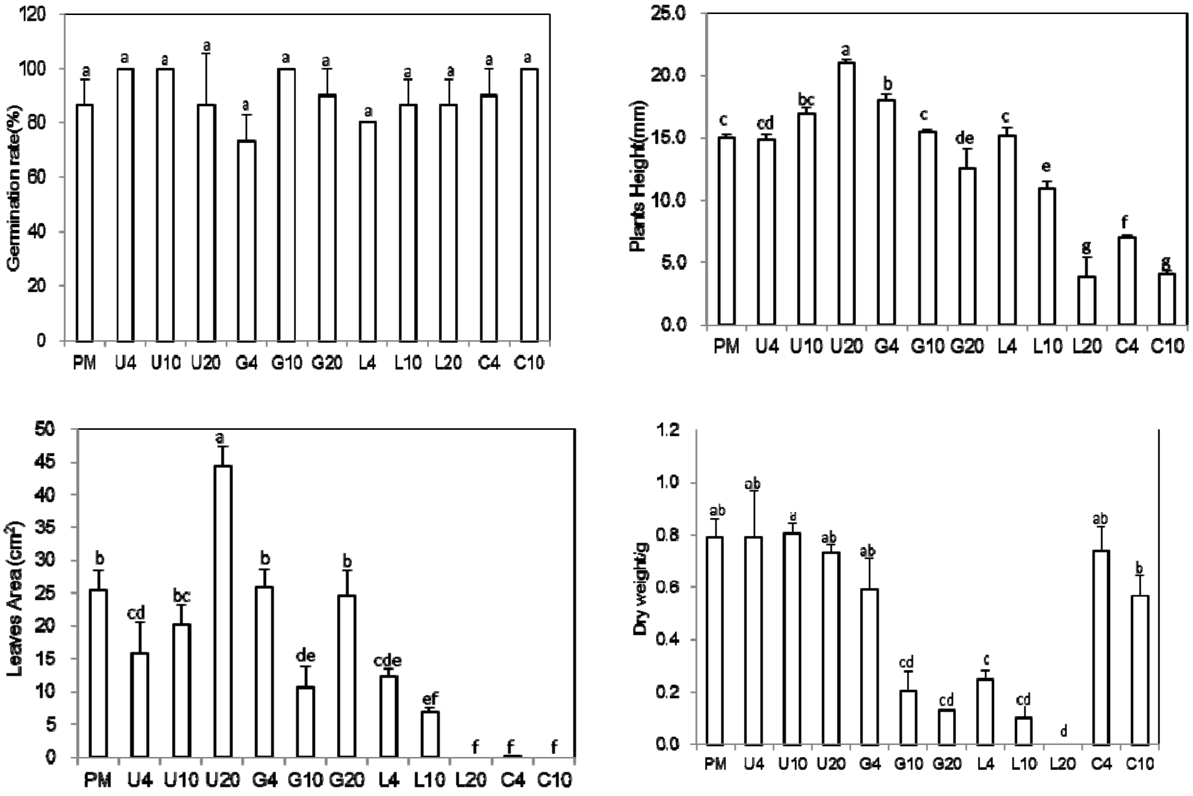

FIGURE 1. Tomato germination rate, plant height, leaf area and shoot dry weight (mean ± standard error) four weeks after germination in a commercial potting medium (ProMix) mixed with 4%, 10% or 20% (by dry weight) of composts made with grass to which granular imprelis (G), clopyralid (C), liquid imprelis (L) or no herbicide (U) had been applied ($P \leq 0.05$). PM = promix control.

4 CONCLUSIONS

- The composting process in the bench scale reactor system closely mimicked commercial scale composting in terms of the feed-stocks, composting temperatures, moisture contents and organic matter and CO_2 losses.
- Herbicides in all three treatments were lost to some extent during composting. On a constant ash basis 47% of the granular aminocyclopyrachlor , 57% of the liquid aminocyclopyrachlor and 71% of the clopyralid was lost.
- Initial aminocyclopyrachlor concentrations were greater in liquid applied than granular applied grass. Grass treated with the granular formulation showed less herbicide loss than grass treated with the liquid formulation during composting. However the final concentrations of aminocyclopyrachlor in the liquid formulation composts were greater, and these composts had a greater impact on bean and tomato growth.
- Clopyralid appeared to be more degradable than either formulation of aminocyclopyrachlor during composting.
- On a wet weight basis the concentrations of both of the aminocyclopyrachlor formulations increased by more than 66% during composting while clopyralid declined by just 9%.
- The final concentrations of all of three of the herbicide treatments of 4.8, 10.7 and 57 ppm on a dry weight basis, respectively, were well above the levels (e.g. 50 ppb for clopyralid) likely to affect sensitive plants.

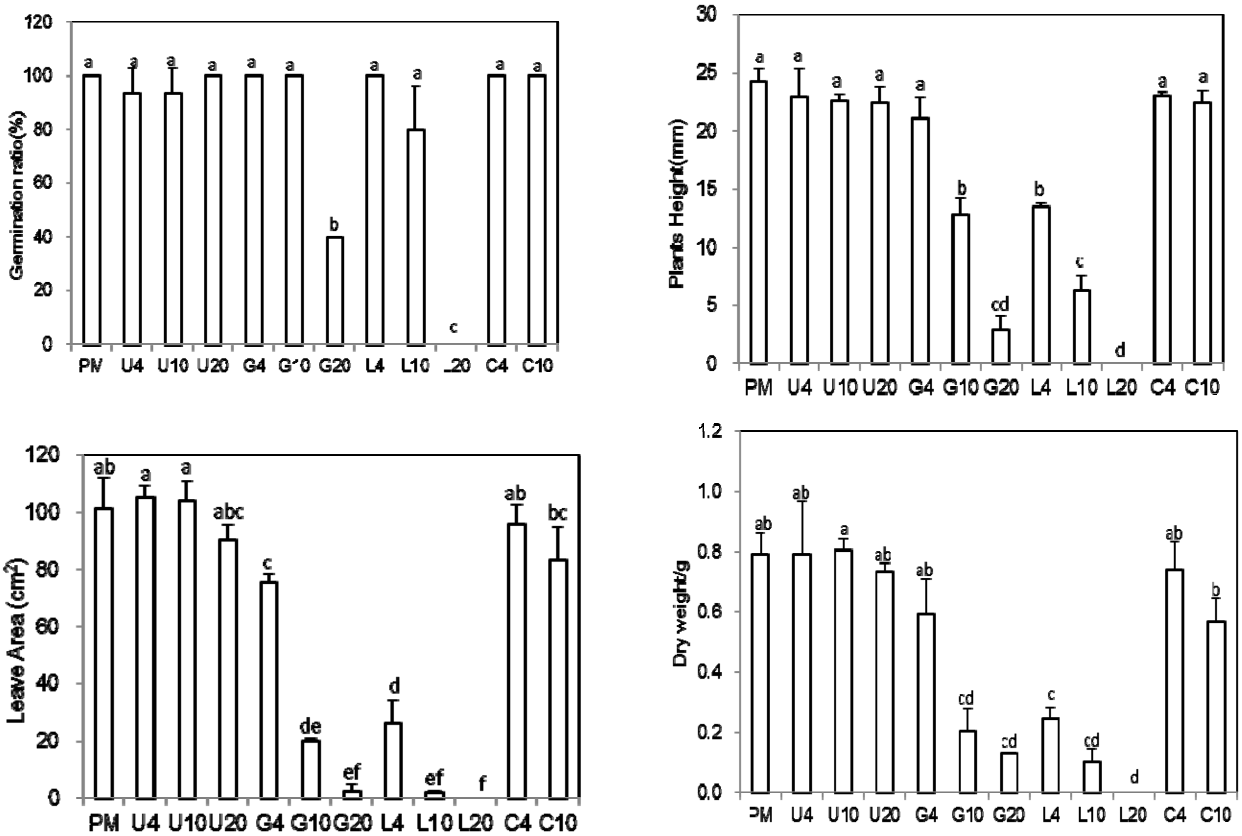

FIGURE 2. Bean germination rate, plant height, leaf area and shoot dry weight (mean ± standard error) four weeks after germination in a commercial potting medium (Pro Mix) amended with 4%, 10% or 20% (by dry weight) of composts made with grass to which granular imprelis (G), clopyralid (C), liquid imprelis (L) or no herbicide (U) had been applied ($P \leq 0.05$). PM = promix control.

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(200) INDUSTRIAL POTATO PEEL COMPOSTING: BLEND FORMULATION USING MIXTURE DESIGN ANALYSIS

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EXECUTIVE SUMMARY

Potato peel (PP) is a surplus material from the industry of processed potato products, like French fries, chips and puree, which create disposal, sanitation, and environmental problems. Given its high content of organic compounds (namely starch, cellulose, hemicelluloses, lignin and fermentable sugars), composting may constitute an interesting option to enhance PP valorisation.

Typically, PP waste is characterized by high interparticle water content which might hinder composting evolution, due to higher oxygen diffusion resistance in pores between particles. Therefore, its valorization may require a previous mixture with other materials, aiming to reach an adequate formulation for composting. Free air space (FAS) is a physical parameter that may play a fundamental role in such processes, in order to maintain aerobic conditions.

This study aims to predict FAS of composting mixtures, when the proportions of the individual materials involved were varied. An augmented simplex-centroid mixture design approach was used to characterize and predict FAS of a mixture involving potato peel (PP), grass clippings (GC) and as bulking agents rice husks (RH) in a set A or sawdust (SD) in a set B.

Mixture design methodology consists in varying the proportions of two or more ingredients of a blend, in order to study the influence of the independent variables (ratios of different components) in a measured response (in this case FAS), which is dependent on the ingredient composition.

FAS was determined experimentally in fourteen different mixtures and these data were modeled by second order Scheffé polynomials. The regression parameters indicated good fit of model to data. Surface and response trace plots allowed to identify antagonistic effects of GC and SD in FAS.

The mathematical model proposed was validated with additional experimental data and very good agreement was achieved. Some theoretical and empirical approaches available in literature for FAS quantification were compared with the predictions obtained through the models provided in our study.

In summary, mixture design approach lead to very good results to estimate FAS of industrial PP mixtures intended for composting, avoiding trial and error methods often used to establish the ingredients proportions. By overlapping contour plots that describe the influence of the mixture composition in FAS, it was possible to select a range that complies with requirements in what concerns moisture content, C/N ratio and FAS.

1 INTRODUCTION

1.1 Background

Food industry manages potato peel (PP) waste as a non valuable material, although in its composition there are relevant quantities of starch, cellulose, hemicelluloses, lignin, fermentable sugars and phenolic acids. Therefore it can be used for multiple purposes, namely as dietary fibre for baking products and animal feeding (Djomo, 2008), for biohydrogen and ethanol production (Djomo, 2008; Mars, , 2010; Arapoglou, 2010) and as a source of natural antioxidants (Schieber, 2001; Wijngaard, 2011; Al-Weshahy, 2010). In addition, some laboratorial studies point out the possibility of using PP for wastewater removal of heavy metals from industrial wastewater (Aman, 2008) and for perchlorate bioreduction of water (Okeke, 2005). Given its high content of organic compounds, composting is another interesting option to enhance PP valorisation.

Composting may be defined as the biological decomposition and stabilization of organic subtracts, under aerobic conditions that allow development of thermophilic temperatures as a result of biologically generated heat, to produce a final product that is stable, free of pathogens and plant seeds, and can be beneficially applied to land (Haug, 1993). Indeed, according to some authors (Schaub, 1996), the possibility of PP valorisation by composting can be quite appealing for the food industry, since it can reduce the volume of organic process by-products up to 40%.

Generally, mixture formulation for composting should be based on physical and chemical properties of the wastes involved (Barrena, 2011), namely moisture content, C:N ratio and FAS (free air space). In fact, it is well recognized that for thermophilic composting, carbon to nitrogen ratio (C/N) should range between 25-30:1 (Huang, 2006) and also moisture content might assume values of 40-65% (Agnew, 2003). FAS is defined as the ratio of gas filled pore volume to total compost mix volume and it determines the air movement inside the composting mixture, as well as the intrinsic air content, carbon dioxide, moisture and heat removal from the composting system (Ruggieri, 2009). Commonly, optimum values may be in the range of 60% (Ruggieri, 2009), and values as high as 85 to 90% have been indicated in certain cases

To calculate the mixture composition according to a moisture and C/N ratio target for the composting mixture it is only necessary to know the values of these factors for the individual materials and then using a set of algebraic equations that are solved iteratively, one can obtain the individual amount of each component to achieve the desired mixture moisture content and C/N ratio. However, this procedure is not applicable whenever a final value of FAS is also defined for the mixture, because this parameter is dependent of the structural characteristics of the materials involved such as bulk density and particle size distribution.

1.2 Research objectives

This work aims to use the mixture design approach to understand and predict the influence of each component, singly or combined, on the FAS of a potato peel waste blend intended for composting. In addition rice husk or sawdust were selected as mixing components to act like drying and bulking agents and grass clippings were utilized as nitrogen source.

2 METHODOLOGY

2.1 Composting materials

Potato peel was gathered at a Portuguese industry of potato chips. Bulking agents were supplied by a rice husking factory (for RH) and by a local pine sawmill (for SD). Grass clippings were granted by a garden maintenance service from a national football stadium and were sieved through a 5 cm mesh, in order to obtain a more homogenous material in size and shape.

2.2 Mixture design establishment and validation

The experimental objective of mixture design was to make predictions of the free air space value (desired response) for any blend of the individual components tested by modelling the response surface with regression equations. It was assumed that the measured response depends only on the relative proportions of the ingredients in the mixture, that are considered as independent variables and allowed to range from 0 to 1, but does not depend on the amount of the mixture. The models considered in this study were Scheffé second-order polynomials (Smith, 2005):

$$Y = \sum_{i=1}^{q} \beta_i x_i + \sum_{i<j}^{q-1} \sum_{j}^{q} \beta_{ij} x_i x_j \qquad (1)$$

where x represents the independent variables, and β corresponds to the polynomial coefficients.

A simplex-centroid mixture design augmented was used to define the number of blends necessary to attain the desired response and the composition of each blend. This experimental design was obtained by using Design-Expert® version 8.0.4 software and included namely pure blends (vertices of the triangle), binary blends (midpoints at each side of the triangle), and blends involving three components. Blends in the interior area of the triangle consisted in mixtures of one third of all ingredients (called centroid blend) and three blends located midway between the centroid and each pure blend. Two experimental replicates of pure and centroid blends were performed to detect the existence of lack of fit (to evaluate if the variation of the replicates around their mean values was less than the variation of the design points regarding their predicted values) and pure experimental error.

According to this methodology, 14 experiments were carried out for each set of mixtures under analysis, then referred as set A (PP + GC + RH and set B (PP + GC + SD).

Each blend of 2 kg was prepared by weighting the different components according to the pre-defined proportions and mixing them by hand during 10 min. The analysis of experimental data and the evaluation of the model that best describes the composition effects in the response variables were performed by using Design-Expert ® version 8.0.4.

Some additional mixtures were prepared and analysed to determine if prediction of the response model was adequate. This assessment was achieved by comparing the model prediction and experimental FAS value for a particular mixture composition.

2.3 Analytical methods

FAS was calculated from the wet bulk density and wet particle density of the blends tested for sets A and B, according to the equation used by Adhikari (2009):

$$FAS = \left(1 - \frac{BD}{PD}\right) \times 100\% \qquad (2)$$

where *BD* is the total wet bulk density (kg m^{-3})and *PD* is the wet particle density of the mixture (kg m^{-3}). Wet bulk density was determined in the fresh mixtures prepared, according to a standard procedure described elsewhere (US Department of Agriculture and US Composting Council, 2001). Wet particle density was estimated following a methodology defined by Adhikari (2009).

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3 RESULTS AND DISCUSSION

3.1 Response regression model analysis

To evaluate how a specific blend composition determines FAS for a PP mixture with RH or SD as bulking agents, experimental responses of FAS were obtained using Eq. (2), for each independent variable combination (Table 1), and these data were fitted to Eq. (1) by using Design-Expert ®.

Table 1 - Experimental response values of FAS for set A (with RH) and B (with SD).

Blend order	Independent variables			Experimental response - FAS (%)	
	X_1 (PP)	X_2 (GC)	X_3 (RH or SD)	Set A (RH)	Set B (SD)
1	1.000	0.000	0.000	34.0	23.5
2	0.167	0.167	0.667	90.1	78.2
3	0.000	0.000	1.000	85.3	83.1
4	0.000	0.500	0.500	88.4	77.0
5	1.000	0.000	0.000	35.1	25.2
6	0.000	1.000	0.000	87.1	85.8
7	0.000	1.000	0.000	85.7	86.1
8	0.667	0.167	0.167	67.0	60.0
9	0.333	0.333	0.333	86.0	74.8
10	0.500	0.500	0.000	74.4	71.4
11	0.167	0.667	0.167	85.4	77.6
12	0.000	0.000	1.000	87.9	85.3
13	0.500	0.000	0.500	84.9	72.1
14	0.333	0.333	0.333	82.5	74.3

Regression results for the polynomial model fit are presented in Table 2. Second order polynomial describing FAS response for blends with PP:GC:RH presented a non-linear term (X_2X_3) that is indicated as not significant. Therefore, a reduced model was evaluated, by removing the term with the highest *p-value* until all terms have *p-values* less than 0.05 (Smith, 2005). This procedure was performed in Design-Expert ® and led to the response indicated in Table 3. As a result, parameters R^2_{adj} and R^2_{pred} were slightly improved (from 0.995 and 0.985 to 0.996 and 0.991, respectively).

Table 2 – Regression coefficients of quadratic model for predicting FAS for set A (with RH) and B (with SD).

Set A			Set A (reduced model)		Set B		
Coefficient value	Independent variable	term *p-value**	Coefficient value	Independent variable	Coefficient value	Independent variable	term *p-value**
34.56	X_1		34.52	X_1	24.79	X_1	
86.40	X_2		86.61	X_2	85.74	X_2	
86.77	X_3		86.97	X_3	84.13	X_3	
50.41	X_1X_2	<0.0001	50.81	X_1X_2	61.55	X_1X_2	<0.0001
92.98	X_1X_3	<0.0001	93.38	X_1X_3	68.66	X_1X_3	<0.0001
3.24	X_2X_3	0.5351	-	X_2X_3	-38.85	X_2X_3	0.0002
	R^2_{Adj}=0.995			R^2_{Adj}=0.996		R^2_{Adj}=0.994	
	R^2_{pred}=0.985			R^2_{pred}=0.991		R^2_{pred}=0.985	

* If a term is not significant for the model, then p-value is >0.05 and model reduction should be evaluated.

3.2 Surface and response trace plots analysis

For evaluation of individual ingredients effects on FAS resposnse, trace and surface/contour plots were drawn. Surface plots were generated to obtain a three-dimensional representation of the FAS response surface and in contour plots, mixture compositions that produced the same response were linked by a contour line. For both sets A and B, mixture proportions that return maximum FAS were located towards pure blends of GC and RH/SD. In fact, contour lines in Fig 1a)-b) indicate the existence of a broad range of GC and RH/SD proportions where FAS assume identical values. However this material does not offer any structural resistance to the blend since it will decompose quickly and then tend to compact; thus it will not retain pore spaces necessary for air movement during the composting process (Rynk, 1992).

By observing the response surfaces, Fig.1 a)-b), it can be concluded that the binary mixture of SD with GC acts antagonistically on the FAS response, since it contributes to the negative curvature in the surface shape for the ternary mixture of PP with GC and SD. Mathematically this effect is related to the negative coefficient associated to X_2X_3 of the FAS response model (2). The main reason for this behavior is the great importance of the particle size distribution of the bulking agent to the structural condition of the mixture. In our study, fine amendment SD was used with 86% of the material passing a 2 mm screen opening, which is a high value comparing with the indication of Haug (1993): 50% particles passing a 2.23 mm screen opening. In blends with SD and GC, SD particles are likely to occupy the interstices of GC, and thus reducing FAS, contrary to what is expected from a good bulking agent.

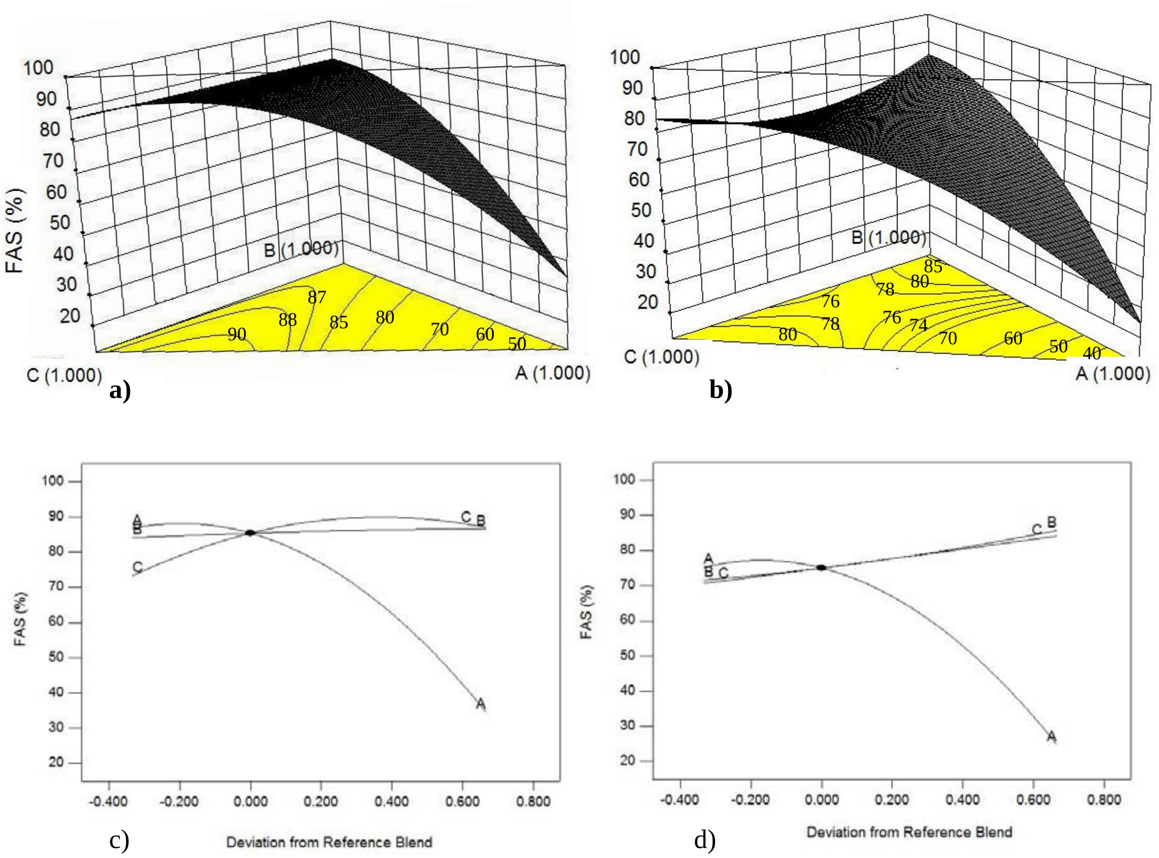

Fig. 1 – Contour plots for FAS: a)- set A (A:PP; B: GC; C: RH); b) set B (A:PP; B: GC; C: SD); Response trace plots for FAS: c)- set A; d)- set B.

Trace plots, Fig.1 c)-d), enable to compare the effects of all components in the design space and determine how sensitive the FAS response is to deviation from the formulation near the reference blend (the centroid blend). The data obtained indicate that, independently of the bulking agent used, PP (ingredient A) has a very strong reducing effect on FAS as the deviation from reference blend is increased. When RH is used, Fig.1-c), the response trace to GC is nearly a horizontal line, meaning that it has a negligible effect on FAS. When SD is used, Fig.1-d), GC and SD response curves are overlapped, meaning that these components have roughly identical linear effect on FAS. This result may be explained by the particle size of the SD used in our study, as previously referred.

3.3 Validation of the response regression model

For validating FAS regression models, Eq (3)-Eq (4), three mixture compositions were selected for each bulking and numerical results are presented in Table 5.

$$FAS_{Set\ A} = 34.52X_1 + 86.61X_2 + 86.97X_3 + 50.81X_1X_2 + 93.38X_1X_3 \quad (3)$$

$$FAS_{Set\ B} = 24.79X_1 + 85.74X_2 + 84.13X_3 + 61.55X_1X_2 + 68.66X_1X_3 - 38.85X_2X_3 \quad (4)$$

Table 3 - Experimental conditions tested and validation results

Mixt. Ref.	X1 (PP)	X2 (GC)	X3	MC (%)	OM(%)	BD (kg m^{-3})	FAS (%) Exp. result	 Model prediction	 Pred. Interval
RH1	0.587	0.192	0.219 (RH)	70.5	86.7	240	75.8	73.9	±4.2
RH2	0.506	0.324	0.170 (RH)	69.6	85.9	229	76.4	76.7	±4.0
RH3	0.660	0.081	0.259 (RH)	64.3	86.2	245	73.9	71.0	±4.5
SD1	0.400	0.400	0.200 (SD)	67.5	90.5	325	71.3	73.3	±5.3
SD2	0.343	0.516	0.141 (SD)	71.4	88.5	268	78.3	76.0	±5.3
SD3	0.600	0.300	0.100 (SD)	70.8	90.0	440	66.9	63.0	±6.5

Experimental results were found to be very close to the models predictions, with relative errors ranging from -2.7 to 5.8%, but within the prediction intervals. Therefore, for the conditions tested, FAS models developed by mixture design approach, Eq.(3) to (4), may be used to properly predict FAS of a ternary composting mixture.

In literature, FAS has been estimated by theoretical or empirical approaches (Alburquerque, 2008). Fig. 2 compares prediction of FAS by the models developed in our study, Eqs. (3)-(4) with the ones obtained by equations proposed by Richard (2004) and Agnew (2003) and indicated in Eq.(5) and (6), respectively.

$$FAS_R = \left(1-\frac{BD}{PD}\times\left(\frac{1-DM}{D_w}+\frac{DM\times OM}{PD_{OM}}+\frac{DM\times\left(1-OM\right)}{PD_{ash}}\right)\right)\times 100\% \tag{5}$$

$$FAS_{Ag} = 100 - 0.0889\times BD \tag{6}$$

where BD is the wet bulk density (kg m^{-3}), DM the fraction of dry matter, OM the fraction of organic matter (volatile solids) in dry basis, Dw the density of water (kg m^{-3}); PD_{OM} the particle density of organic matter (2500 kg m^{-3}) and PD_{ash} the ash particle density (1600 kg m^{-3}).

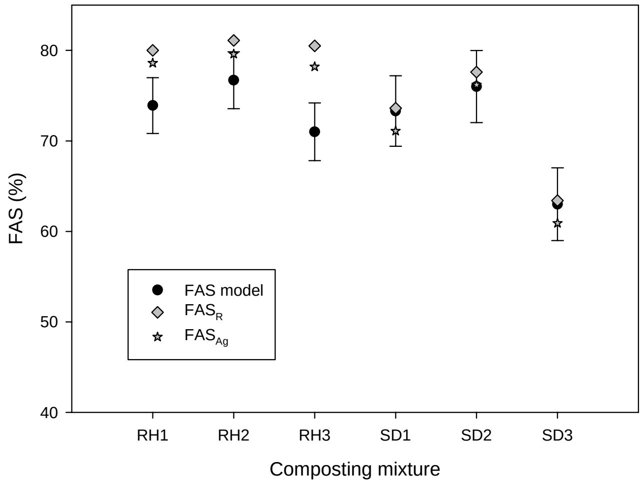

Fig. 2 – Comparison between FAS predictions(error bars indicate prediction intervals with a significance of 95%).

In general for blends involving RH, FAS calculated with Eq.(5)-(6) are slightly higher than the ones predicted by models of our study. However, in the case of composting mixtures where SD is the bulking agent, correlations FAS$_R$ and FAS$_{Ag}$ lead to values inside the prediction interval. These differences might be related to the applicability conditions of theoretical and empirical approaches for FAS quantification. According to Ruggieri (2009), properties such as BD and MC have a strong influence in FAS estimation by these correlations, and boundaries were established to its application. Namely, FAS$_R$, Eq.(6), is limited for materials where BD is superior to 400 kg m^{-3}, while bellow this level FAS$_R$ may lead to misleading values. Also FAS$_{Ag}$, Eq.(5), seems restricted only to high-moisture and/or low-density materials where FAS$_R$ may provide unreliable values. In our study, mixtures with RH have low bulk densities (<245 kg m^{-3}) and FAS$_R$ were the highest ones, over estimating FAS$_{model}$ (relative error ranged from -5.7 to -13.3%).

FAS indicated by FAS_{Ag} were also overestimated but within the relative errors (-3.8 to -10.1%). For mixtures with SD, FAS_R were more consistent with FAS_{model} predictions, especially for mixtures with higher bulk density (SD1 and SD3).

4 CONCLUSIONS

In this study, a mixture design methodology was studied to predict FAS (free air space) of potato peel (PP) mixtures intended for composting. Grass clippings (GC) and rice husks(RH)/sawdust(SD) were used as additional ingredients. The results showed that the mixture design methodology leads to reliable mathematical models for predicting FAS of mixtures involving industrial PP.

In particular, for blends with PP, GC and SD, the FAS model pointed out the antagonistic effect of the binary mixture of GC and SD to the overall blend. Contour plots revealed the occurrence of a wide range of GC and SD (bulking agent) proportions where FAS assume identical values; nonetheless, large proportions of GC should be avoided. As expected, response trace plots clearly indicated that independently of the bulking agent used, the addition of potato peel has a strong effect in reducing the mixture air voids volume.

The models obtained for predicting FAS were compared with theoretical and empirical correlations proposed in literature which consider mixture properties like dry matter, organic matter and bulk density. For blends that incorporate SD, correlations calculation is consistent with our model prediction. When RH was used as bulking agent both literature correlations overestimated the FAS but even so the deviations were low.

In short, the methodology used in this study seems to be adequate to determine regression models for estimating FAS of specific potato peel blends.

5 ACKNOWLEDGEMENTS

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(239) STABILIZATION OF THE SOLID FRACTION OF A DIGESTATE BY COMPOSTING AND VERMICOMPOSTING

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EXECUTIVE SUMMARY

The number of anaerobic digestion facilities has increased remarkably over the last years. Anaerobic digestion constitutes an advantageous process as compared to other waste treatment options because it allows the net withdrawal of energy while treating organic waste. A key step for the success of anaerobic digestion is the correct management of the final digestates produced. Digestates may constitute valuable soil amendments and/or fertilizers due to their high content in organic matter and plant nutrients. Nevertheless many digestates are not completely stabilised and may content human pathogens; therefore they cannot be applied to the soil directly without a previous stabilization treatment. Research into cost-efficient stabilization techniques for digestates is therefore essential for the adequate development and success of anaerobic digestion.

The objective of this work was to study the capacity of composting and vermicomposting to improve stability and to reduce pathogen content on an anaerobic digestate. Material used was the solid fraction of a digestate produced after mesophilic anaerobic digestion of a mixture of effluents from dairy (70%), effluents from canning (20%) and fish fat from canning (10%). Composting assays were carried out in 5 30L vessels. The digestate (9.7 kg) was mixed in 1:2 proportion (vol:vol) with chopped pruning remains. During the composting process temperature and oxygen concentration were monitored continuously. The composting assay was finished at the end of thermophilic phase and final samples were collected. Vermicomposting assays were carried out in 5 mesocosms of 5L. A mix of digestate and manure was used as a bed layer. Over the bed layer 1kg of digestate and 130gr of earthworms were placed. The vermicomposting assay was stopped at the end of active phase, when all the digestate was processed by the earthworms. Vermicompost samples were subsequently collected from each of the mesocosms.

The control treatment consisted in 5 open vessels with 9 kg of digestate that were incubated at room temperature and without management for 17 days. Samples were collected from each experimental unit at the end of the composting, vermicomposting and control treatments and stored at 4°C for subsequent analysis. The main parameters analyzed in the samples were: moisture, organic matter, pH, electrical conductivity, ammonium (N-NH_4^+), nitrate (N-NO_3^-)' respiration rate (AT4), dissolved organic carbon (DOC), dissolved organic nitrogen (DON), total carbon and nitrogen, nitrogen of the microbial biomass (MBN), E. coli and C. perfringens concentration. Data were analyzed through ANOVA and Tukey HSD posthoc test. Statistical analyses were carried out using STATISTICA for Windows.

Results showed an improvement in some stability parameters. The respiration rate (AT4) decreased from 41.3mg O_2/gr dry matter in the initial digestate to 16.4mg O_2/gr dry matter after composting and 22.2 mg O_2/gr dry matter after vermicomposting. Composting and vermicomposting trials had the capacity to reduce significantly ammonium concentrations respect to the control. Besides, vermicomposting increased nitrification of NH_4^+ to NO_3^-, so, the NH_4^+/NO_3^- of fresh vermicompost was lower than 1.

Meanwhile, the high initial levels of nitrogen were probably the reason why DOC/DON ratio in final compost and vermicompost were higher than the suggested limits of stability. The treatments did not reduce the pathogen abundance of the initial waste.

The final material of composting and vermicomposting assays showed better stability values than initial digestate and than control material too. Yet many of these parameters did not meet the quality standards established in the Spanish and European legislations and thus, maturation phase is recommended to adjust NH_4^+, DON/DOC, NH_4^+/NO_3^- ratios to more suitable values for agricultural purposes.

1. INTRODUCTION

Current European legislation on waste materials (1999/31/CE, 2006/12/CE, 2008/98/CE and 2009/28/CE) has the objective of to improve organic matter recycling and to reduce organic matter landfilling. Based on this legislation, many European countries have increased their support to anaerobic digestion facilities. Anaerobic digestion allows generation of energy from wastes, so, increases countries independence from fossil fuels. Furthermore, anaerobic digestion recycles organic matter and recovers nutrients from wastes.

As a result, in European Union the energy production from biogas has risen a 178% in the 2002-2009 period. In 2009 the energy obtained from biogas exceeded 8 M tonnes of oil equivalent (EurObserv'ER, 2009).

In absence of oxygen, anaerobic digestion generates carbon dioxide and biogas from organic matter. The biogas is compound in most part by methane (50-80 vol %), which could be transform in electrical energy or heat (Tani et al., 2006). Moreover, anaerobic digestion takes place in closed reactors which helps to reduce emissions of greenhouse effect gases, such as methane and nitrous oxide (Sørensen and Møller, 2009).

1.1. Digestates and their management

Anaerobic digestion also produces an organic biodegradable subproduct named digestate. These digestates are a mix of water, partly-digested organic matter and microbial biomass (Bernal-Calderón et al., 2011). Thus, the digestate is a valuable source of organic matter and it could be use as fertilizer or as an organic amendment. In this way, some authors support that digestates are as stable as compost and with higher concentrations of N, P and K (Tambone et al., 2010). However chemical characteristics of final digestates are very changeable, because it depends of raw materials and the parameters of the digestion process too. In this way, different researches have showed that digestates produced are not typically suitable for direct land application as they still contain organic matter biodegradable fractions, tend to be odorous, too wet and too high in volatile fatty acid (VFA) concentration, which are phytotoxic), on addition if the digestion is not performed under thermophilic conditions, the solids are not sanitised. (Tambone et al., 2010; Teglia et al., 2011a; 2011b)

Therefore, although anaerobic digestion is a clean technology which allows energy production from wastes, the digestates production could be a problem for the sustainability of anaerobic digestion (Brown et al., 2010). As consequence, the key to anaerobic digestion success is correct management of solid fraction of digestates which allows their use in agriculture (M∅ller et al., 2009). Consequently, a post treatment is required to reduce adverse characteristics of digestates before land application (Poggi-Varaldo et al., 1999). Thus European Commission has been working in future legislation which is going to set up stability and sanitation requirements (European Commission 2001; 2010). Nowadays, digestates does not have a common legislative standard in European Union. Only, UK and Germany have established specific composition requirements (similar as requirements for compost) for digestates in function of their use as fertilizer or as organic amendment (RAL GZ 245; BSI: PAS 110:2010).

1.2. Stability and sanitation parameters

Stability refers to the decomposition rate of organic matter (Chen, 2003). Therefore, stability could be determined based on O_2 consume, CO_2 production or by microbial activity heat production (Iannotti et al., 1993; 1994). One of typical units used to assess stability is accumulated oxygen consumed in 4 days (AT4). Accordingly, several researchers suggest limit values of 5-10 mg O_2/gr dry matter (Cossu and Raga 2008; Ponsa et al., 2008).

Other suggested parameters to determine stability of organic materials are related with the availability of easily metabolizable carbon and nitrogen in substrates, and their ratios. The most accepted maximum for NH_4^+ concentrations for stable organic materials is the value of 400mg/kg (Bernal et al., 1998; Tognetti et al., 2007). Regarding the stability limits for carbon and nitrogen ratios, in the literature there are different values. On one hand, proposed limits for carbon and nitrogen dissolved forms ratio (DOC & DON) is established in DOC/DON <0.7 (Hue and Liu, 1995) or DOC/DON <0.55 (Bernal et al., 1998). At least, values under 1 for NH_4^+/NO_3^- ratio are considerate as indicators of stability (Bernal et al., 1998; Larney, 2000).

In addition to stability, other important parameter to improve before digestates land application is sanitization level. In Table 1, microbial limit levels of current Spanish legislation and limit values suggested for future legislation by the European Commission are presented.

	RD 1310/1990	RD 824/2005	WD 2001*	WD 2010**
Escherichia coli	-	<1000 MPN/gr	-	< 5x10^5 UFC/gr
Salmomella sp	-	25gr	Absent 50gr	Absent 25-50 gr
Clostridium perfringens	-	-	Absent 1gr	-

Table 1: Limit values of microbial concentration of Spanish current legislation and suggested in Working Document 2001 (*) and Working Document 2010 (**).

1.3. Management options

Aerobic processes such as composting and vermicomposting are suitable options for the management of solid fraction of anaerobic digestates. Both processes, composting and vermicomposting, have the capacity of transforming raw wastes in stable organic matter (Zucconi & De Bertoldi, 1986; Domínguez, 2004) and, at the same time, reduce the concentration of pathogens (Imbeah, 1998; Eastman et al., 2001; Edwards y Subler, 2011b). Composting has been already employed in management of anaerobic digestates (Nakasaki et al., 2009). Besides, the working document on biological treatment of biowaste and the working document on sludge and biowaste (European Commission 2001; 2010) specifically recommend composting as a management treatment for digestates. However, the efficiency of vermicomposting has not yet been assayed with this kind of wastes.

1.4. Research objectives

The main objective of this work was to evaluate the efficiency of composting and vermicomposting to improve stability and to reduce pathogens concentration in solid fraction of digestates. To evaluate improvement in stability and sanitization, final data were compared with stability criteria mentioned above and also compared with pathogen limits of legislation. In this way, working document on biological treatment of biowaste (European Commission, 2001), working document on sludge and biowaste (European Commission, 2010), RD 1310/1990 and RD 824/2005 were used as reference.

2. METHODOLOGY

Material used was the solid fraction of an anaerobic digestate produced after mesophilic digestion of a mixture of effluents from dairy (70%), effluents from canning (20%) and fish fat from canning (10%). In this research, two different management options, composting and vermicomposting was carried out.

Composting assays were performed in 5 vessels of 30L. These vessels were thermally isolated and had a false bottom, which allowed air exchange and avoided water logging. Temperature was monitored continuously though two probes installed at 20 and 40 cm depth in the vessels (Figure 1). All the process was controlled by an industrial PLC (Programmable Logical Controller). Thus, oxygen and temperature data was registered each minute. Furthermore, the PLC was programmed to maintain adequate values of oxygen and to avoid overheat (Figure 1).

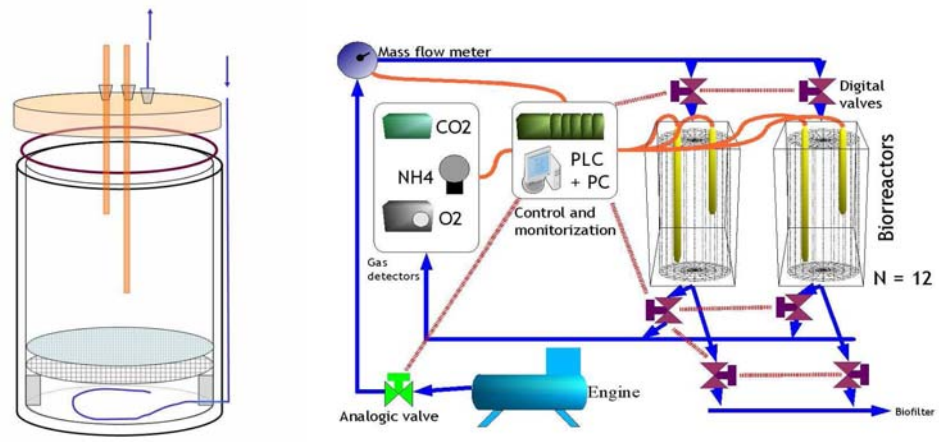

Figure 1: Composting vessel and diagram of control system.

In order to increase its porosity, the digestate was mixed with a bulking agent (chopped pruning remains) in a 1:2 (vol:vol) proportion, the most commonly used in composting facilities.

Composting process followed a homogeneous dynamic in the 5 vessels. Temperature rose until thermophilic values (> 35ºC) within the first two days of the process; thermophilic phase lasted for 9 days (Figure 2). Oxygen values were over 10% during all the process. Once the temperature fell, the composting assay was stopped.

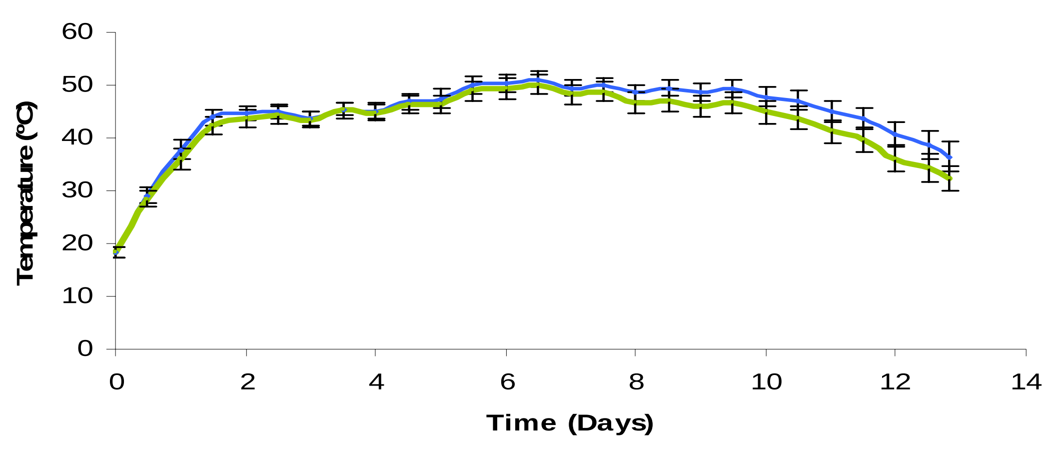

Figure 2: Evolution of temperature during composting assay. Blue line represents data of probe 1 (20cm) and green line shows data of probe 2 (40 cm). Values are averages of the 5 vessels ± standard error.

Vermicomposting assays were carried out in 5 mesocosms of 5 L. The mesocosms also had a false bottom which avoided water logging and allowed the collection of lixiviates. As a bed layer a mix of manure and digestate previously processed by earthworms was used. Over the bed layer 1 kg of digestate and 130 gr of earthworms (*Eisenia sp*.) were placed. The digestate/earthworm ratio employed was the suggested by Eastman (2001) for a correct waste sanitization. The mesocosms were maintained at room temperature, constant humidity and away for light. Vermicomposting process was stopped once the earthworms had processed the digestate supplied (end of active phase of vermicomposting). The length of this active phase was17 days and a processing capacity of 0.45gr of waste /gr of earthworm was observed.

The control treatment consisted in 5 open vessels with 9 kg of digestate, which were incubated at room temperature and without management for 17 days.

At the end of trials each experimental unit was weighted to evaluate the mass balance and lixiviates were measured. Samples were also collected at the end of the composting, vermicomposting and control treatments and stored at 4°C for subsequent analysis. The main parameters analyzed in the samples were: moisture, organic matter, pH, electrical conductivity, ammonium ($N\text{-}NH_4^+$), nitrate ($N\text{-}NO_3^-$), respiration rate (AT4), dissolved organic carbon (DOC), dissolved organic nitrogen (DON), total carbon and nitrogen, nitrogen of the microbial biomass (MBN), E. coli and C. perfringens concentration. Data were analyzed through ANOVA and Tukey HSD posthoc test. Statistical analyses were carried out using STATISTICA for Windows.

3. RESULTS AND DISCUSSION

3.1. Effects on physicochemical parameters

Initial digestate showed an organic matter value 66.97% ± 0.68. The treatments applied had a significant effect over the organic matter of the digestate (F=217.42; p=0.000000). Highest organic matter reduction respect to the control was observed after composting assay. Vermicomposting process had a moderate effect but also significant. In the composting trial a mass reduction of 27% was observed while a mass reduction of 17% was showed in the vermicomposting assay. In control treatment organic matter reduction was not detected (figure 3).

Final values of organic matter in compost and vermicompost were higher than the values established in the Spanish legislation (RD 824/2005) and than levels of 20% suggested in literature (Teglia et al., 2011a). Consequently, further maturation is recommended for these materials.

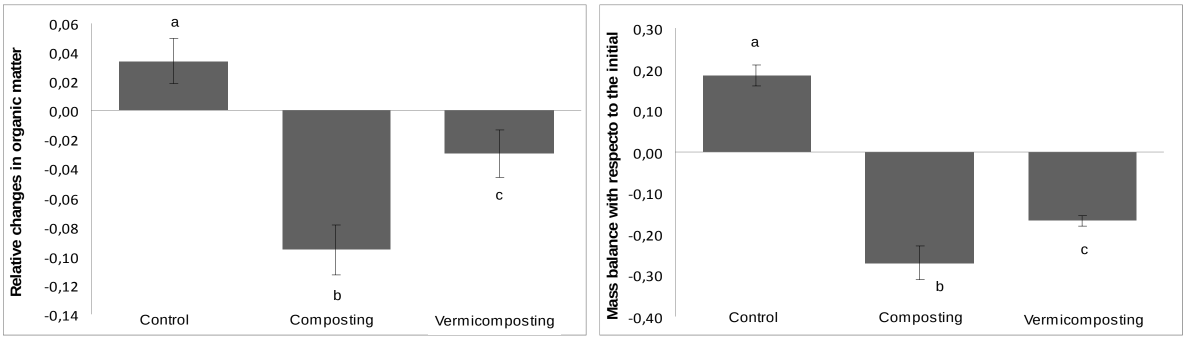

Figure 3: Relative changes in organic matter and mass balance of the digestate after the control composting and vermicomposting treatments (bars are averages of 5 replicates ± standard deviation). Different letters show significant differences (Tukey HSD test at α=0.05)

Ammoniun concentration on initial digestate was 5992 ± 48 mg/kg and the initial concentration of nitrates was 1431 ± 108 mg/kg. Composting and vermicomposting trials had the capacity to reduce significantly ammonium concentrations respect to the control (F= 3745. 02; p=0. 000000) (Figure 4). Nevertheless, final values of ammonium on fresh compost and fresh vermicompost were higher than the recommended limit of 400 mg NH_4^+/kg for stable materials (Bernal et al., 1998; Tognetti et al., 2007). Proposed treatments had also significant effect over nitrate concentrations (F=222. 4484; p=0.000000). During composting process ammonium was presumably lost from the waste material by volatilization. Consequently, NH_4^+/NO_3^- ratio in fresh compost was over the proposed limit of 1 for stable materials (Bernal et al., 1998; Larney, 2000). In contrast vermicomposting process stimulated nitrification, reflected in a significant increase in the concentration of nitrates (figure 4). As a result the NH_4^+/NO_3^- ratio in fresh vermicompost was under the stability limit of 1 suggested for this parameter.

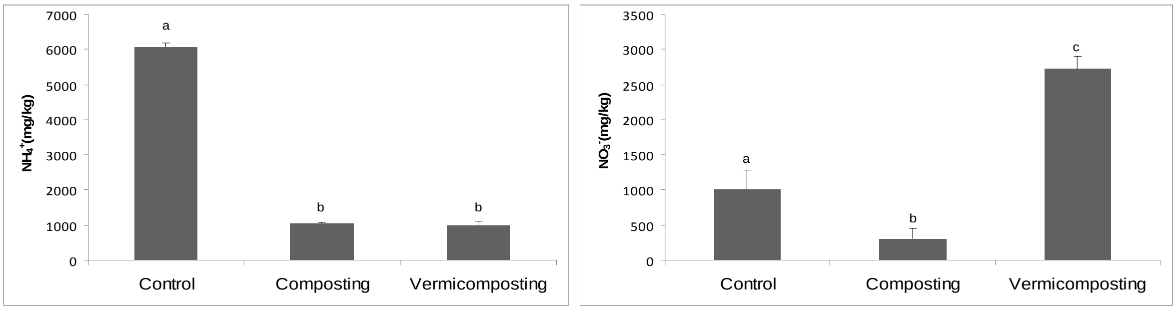

Figure 4: Ammonium (N-NH_4^+) and nitrate (N-NO_3^-) concentrations of the digestate after the control, composting and vermicomposting treatments (bars are averages of 5 replicates ± standard deviation). Different letters show significant differences (Tukey HSD test, α=0.05)

Final values showed in this work for DOC/DON ratio were 1.47 ± 0.42 in digestate, 1.74 ± 0.51 in control, 2.87 ± 0.34 in fresh compost and 1.13 ± 0.75 in fresh vermicompost. These values are higher than the maximum values of 0.7 suggested by Hue and Liu (1995) or 0.55 recommended by Bernal et al., (1998). These final values in DOC/DON ratio were effect of the high levels of nitrogen in the initial digestates, which are consequence of the nitrogen concentration after the anaerobic digestion.

3.2. Effects on biological parameters

Initial values of respiration rate at 4 days (AT4) was 41. 33 ± 4.94mg O_2/gr dry matter for the digestate. Fresh compost (16. 43 ± 1.48mg O_2/grdry matter) and fresh vermicompost (22. 22 ± 1.52mg O_2/gr dry matter) presented an AT4 values significantly lower (F=130.018; p=0.000000) than the control (34.43 ± 1.66mg O_2/ gr dry matter5). Effect of the different treatments was different and composting was showed the highest reduction (figure 5).

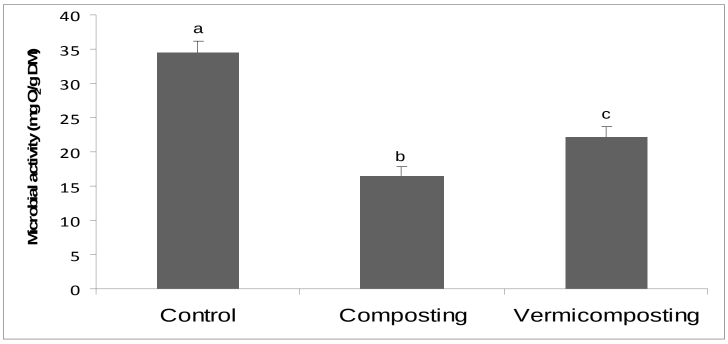

Figure 5: Microbial activity at 4 days of the digestate after the control, composting and vermicomposting treatments (AT4) (bars are averages of 5 replicates ± standard deviation) after the different treatments. Different letters shows significant differences (HSD test of Tuckey, α=0.05)

The values of AT4 obtained after composting and vermicomposting are higher than the limit suggested by working documents of European Commission (European Commission 2001; 2010) (10 mg O_2/gr dry matter). However other classification criteria consider materials with AT4 values between 30 and 20 mgO_2/gr DM can still be considered as stable (Table 2).

O_2 consume (mgO_2/gr DM)	Category of material	Autoheating Test equivalence
≤20	Stable	V
30-20	Stable	IV-III
50-30	Fresh	II-I
80-50	Fresh	I
>80	Raw	I

Table 2: AT4 and autoheating test equivalences. Adapted from Barrena-Gómez and col. 2006

Based on Table 2, AT4 values of initial digestate (41. 33 ± 4.94mg O_2/gr dry matter) and control material (34. 43 ± 1.66mg O_2/gr dry matter) should be classified as fresh material. While fresh compost (16. 43 ± 1.48mg O_2/gr dry matter) and fresh vermicompost (22. 22 ± 1.52mg O_2/gr dry matter) should be classified as stable materials.

Application to land of substrates with high levels of microbial concentrations could produce an immobilization of nutrients (Guerrero et al., 2007) or could generate and excessive consume of O_2 (Butler et al., 2001). Therefore, microbial biomass is a suitable parameter to determine agricultural potential uses of substrates. Initial concentration of nitrogen of microbial biomass (N-MB) was 15456 ± 3388mg/kg. Assayed treatments had a significant effect (F=97.49; p=0.000000) respect of control (26793 ± 2522 mg/kg). Thus composting management reduced N-MB concentrations until 15482 ± 2596 mg/kg. Meanwhile vermicomposting assay showed the highest N-MB reduction (4961 ± 1322 mg/kg). Additionally, only vermicomposting treatment produced a considerable reduction on N-MB respect to initial values.

In order to evaluate the sanitization efficiency of composting and vermicomposting, changes in *Escherichia coli* and *Clostridium perfringens* concentrations was also measured. Initial values in the digestate were 240 ± 102. 23 CFU/gr for *Escherichia coli* and 2120 ± 605.881 CFU/gr for *Clostridium perfringens*. Both processes composting and vermicomposting did not have significant effect over *Escherichia coli* and *Clostridium perfringens* concentrations (figure 5). These results could be consequence of a short thermophilic phase in composting and the low processing capacity of earthworms observed in vermicomposting.

Sanitation efficiency in composting could be improved with high critical mass or through co-composting. While, to enhance sanitation effectiveness in vermicomposting the ratio waste/earthworm must be increased.

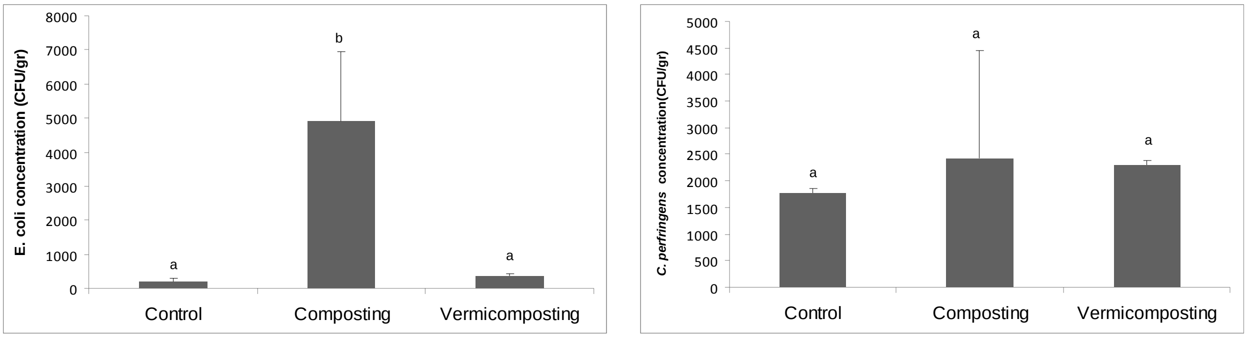

Figure 5: Abundance of *E. coli* and *C. perfringens* in the digestate after control, composting and vermicomposting treatments (bars are averages of 5 replicates ± standard deviation). Different letters mean significant differences (Tukey HSD test, α=0.05)

4. CONCLUSIONS

The two stabilization treatments evaluated in this study (composting and vermicomposting) could be applied to the digestate and had a correct development. The composting process reached thermohilic values and anoxic problems were not detected. On the other hand, mortality of earthworms was not detected in the vermicomposting process.

Composting treatment allowed a reduction in the AT4, NH_4^+ values an in the DON/DOC, NH_4^+/NO_3^- ratios. These values still were higher than limits for stability suggested by scientific literature or by working documents on biowaste. Nevertheless, AT4 and NH_4^+ values should be corrected during maturation phase to most adequate values for land application. The capacity of vermicomposting to increase mineralization of NH_4^+ allows that fresh vermicompost presented NH_4^+ values and DON/DOC, NH_4^+/NO_3^- ratios more close to stability limits than fresh compost. Furthermore, an improvement in AT4 values was observed respect to initial values of the digestate. Although, these values were upper stability limits. To adjust these parameters to more suitable ones for agricultural uses, a maturation phase is recommended.

In a nutshell, composting and vermicomposting treatments allow the recycling of nutrients from digestates which is key for an integral management and sustainability of anaerobic digestion. However more research efforts are necessary to optimize and increase the efficiency of the available alternatives for management of these digestates.

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Session 32

(3) CATALYTIC PYROLYSIS OF BIOMASS FOR OXYGEN REMOVAL FROM BIO-OIL

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EXECUTIVE SUMMARY

The pyrolysis of biomass is a thermal process that converts, at high yield, solid biomass into a liquid product known as bio-oil by using a silica inert material to transfer heat and volatilize the biomass particles. One alternative for the production of a bio-oil of better quality and with lower oxygen content is the use of catalysts in the pyrolysis reactor, rather than an inert, a process called catalytic pyrolysis. The objective of this study was to investigate the effects on product yields and the qualities of products of two different catalysts, one acidic, a commercial fluid catalytic cracking (FCC) catalyst, and a basic one containing hydrotalcite. Inert material, a type of silica, was used as reference. The tests were conducted in a pilot plant with a circulating fluidized bed reactor, specially adapted to perform the catalytic pyrolysis tests, at temperatures of 450°C and 550°C. Due to the relatively high residence times used in the reactor, the process could be classified as an intermediate pyrolysis in terms of this parameter. The results showed that the increase in the residence time of the pyrolysis vapors had a significant impact on products yields, when compared with the profile found in the literature for fast pyrolysis, since the increase in the secondary reactions produced higher yields of coke and water, and lower yields of bio-oil. The FCC catalyst presented higher deoxygenation rates by dehydration, while the hydrotalcite showed greater capacity for decarboxylation. Thus, the use of either the FCC catalyst or hydrotalcite are not suitable for intermediate pyrolysis reactors, since both materials enhance secondary reactions, generating a product with high water content and low content of organic compounds in bio-oil and produce more coke. Analyzing the composition of bio-oils from the standpoint of biofuel production, none of the materials tested produced bio-oils with considerable hydrocarbons yields and presented high amounts of phenolic compounds. In general, silica had the best results in terms of yield and quality of bio-oil. Being an inert material, silica was more suitable for intermediate pyrolysis process conditions, where the contribution of secondary reactions in the gas phase is high due to the residence time in the reactor.

1 INTRODUCTION

The fast pyrolysis of biomass is a thermochemical process that occurs in the absence of oxygen, which main objective is the conversion of lignocellulosic biomass components (cellulose, hemicellulose and lignin) into a black, acid and viscous liquid, called bio-oil. Hot sand is recycled to promote heat transfer from its hot surface to the solid biomass particle. There is also formation of gas (consisting mainly of CO, CO_2 and methane), coal and ash. The use of catalyst instead of sand in the reactor bed also promotes coke formation on the catalyst surface. The yield of each product depends mainly on the biomass composition, operating conditions and type of reactor. The most important reactors used in this process are fluidized bed and circulating fluidized bed, the latter having the advantage of burning the coal, which can be used to generate heat for the process.

According to BRIDGWATER, the following is a list of desirable biomass pyrolysis features to maximize bio-oil, up to 75% dry biomass feed (1):

- Reaction temperature between 450 and 550°C.

- High heat transfer rate to biomass, aiming at complete primary degradation of the biomass.
- In order to minimize secondary reactions of pyrolysis vapors, it is necessary to use a low pyrolysis vapors residence time in the reactor (<2 s) and a rapid condensation of these vapors after the reactor.

The bio-oil is a complex mixture of compounds derived from cellulose, hemicellulose and lignin breakdown with approximately the same elemental composition of the biomass. There is also a large amount of water in the composition of bio-oil, generated from biomass moisture and the pyrolysis reactions. The water content can reach 30% in weight, considering a standard bio-oil (2). A major difference between the bio-oil and oil products is the presence of oxygen in its composition, which represents 45-50% of the bio-oil. Oxygenated compounds are responsible for the bio-oil polar characteristics, making it immiscible with petroleum fractions. The high oxygen content has a negative impact on the heating value of bio-oil, which ranges typically between 14 -18 MJ/kg, representing 40% of the corresponding value for a heavy fuel oil (41-43 MJ/kg). Oxygenated compounds are also responsible for its thermal instability, high corrosiveness and high acidity. Storage of bio-oil is also a critical factor for its commercialization, since the compounds formed are not at thermodynamic equilibrium and many reactions occur modifying the bio-oil characteristics over time (3).

Therefore, properties such as high corrosiveness, thermal instability, incompatibility with fossil fuels and high viscosity are obstacles for using bio-oil as a fuel. In order to reduce the levels of oxygen present in the bio-oil, routes such as hydrodeoxygenation (HDO) and zeolite cracking are considered promising. Blends with diesel for co-processing and steam reforming to produce hydrogen or synthesis gas have also been studied (4).

Another route to reducing bio-oil high oxygen level, called catalytic pyrolysis of biomass, consists of the direct introduction of biomass into the catalytic bed instead of using an inert bed, having the advantage of eliminating the cost of condensation and re-vaporization during the bio-oil production and re-processing, respectively (5). The use of acid catalysts, such as ZSM-5 or conventional FCC catalyst (Y zeolite), has been the focus of most pyrolysis catalytic studies found in literature. The goal of most these studies is to reduce the oxygen content of bio-oil via decarboxylation (CO_2 production), decarbonylation (CO production) and/or dehydration, although there is a significant loss of bio-oil yield. The use of basic catalysts has been little studied and would aim at obtaining high deoxygenation rates associated with a lower loss of bio-oil yield.

2 METHODOLOGY

2.1 Biomass pyrolysis pilot plant

An existing FCC pilot plant was adapted for biomass pyrolysis studies (FIGURE 1). The unit consists of a reactor where a solid (inert or catalyst) is fluidized by nitrogen injection. The reactor is connected to a supply biomass system, which comprises an agitated hopper and a screw conveyor. Another vessel, a fluid bed regenerator, allows the burning of coal and coke produced by the pyrolysis reactions. The coal, produced from the primary degradation of biomass, and coke, arising out from the secondary reactions of pyrolysis vapors in the catalyst pores, could not be distinguished from each other since they are burned together. In the present study, it will be just called "coke". The reactor and the regenerator are interconnected and the control of solid flow is done by screws conveyors located at the bottom of each vessel. The unit has also a two-stage condensation system and a vessel for the retention of solids at the reactor outlet.

The unit operates with continuous biomass feeding and continuous solids circulation. Its product recovery system is provided with vessels to accumulate effluent in test and pretest mode. The operation type "pretest" is intended to ensure that when the test is started, the unit is stable and the liquid product sampled is representative of the period after stabilization.

The regenerated catalyst or sand at 700 °C is transferred continuously by the regenerator screw conveyor to the top of the fluidized bed reactor, where it meets the biomass, which is injected in the reactor into a flow rate range between 700 and 800 g/h. The biomass is then "cracked" by the contact with the fluid bed and the reaction vapors rises through the reactor. The vapors reach the top of reactor and enter into the separator vessel, which has an internal filter to provide the separation between particulates and pyrolysis vapors. The particulates are composed by the catalyst (or sand), coal and ash. At the exit of the reactor and the separator vessel, there is a nitrogen injection to prevent obstruction by the accumulation of solids.

The pyrolysis vapors leave the top of the separator vessel and proceed to the first stage of condensation system, which consists of a chilled tower, where part of bio-oil is condensed and stored in a vessel (test or pretest).

The second stage consists of two small vessels (test and pretest) inserted into a container with dry ice. At the top of each vessel there is a demister for retention of small particles of bio-oil dispersed in the gas. After the second stage there are still two filters in series. The non-condensable pyrolysis gas is then sent to a wet gas meter (WGM) for measuring flow and calculating the volume of the gas produced. A gas sample from the reaction is collected for injection into a chromatograph in order to obtain its composition.

The catalyst from the reactor, which has a large amount of catalyst deposited on its surface, is continuously transferred from the reactor to the regenerator. The catalyst flow is controlled by the screw conveyor rotation in order to maintain the reactor bed level constant.

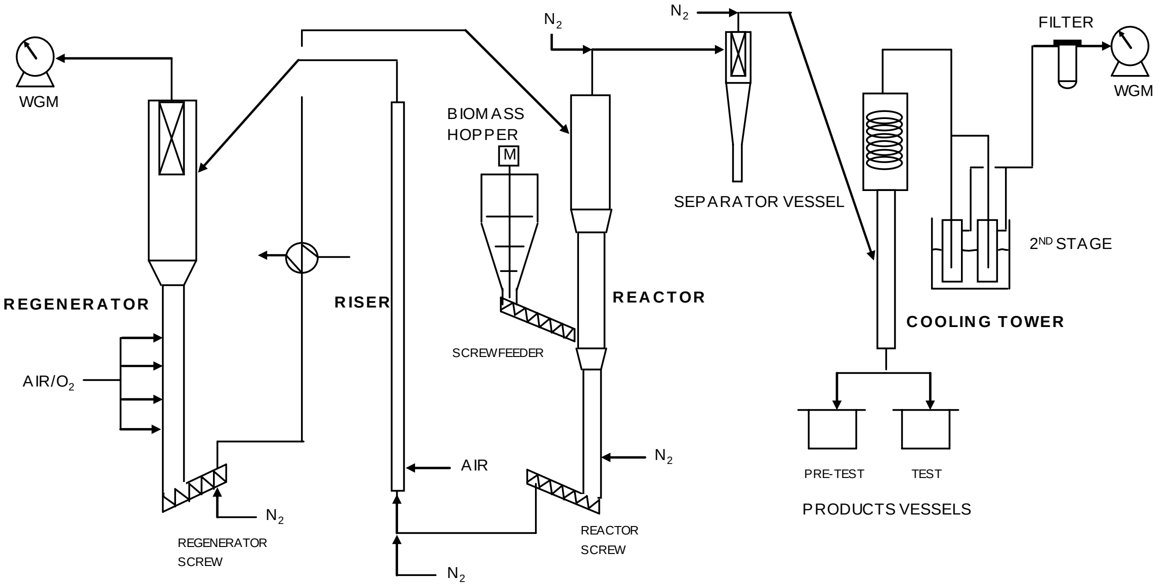

FIGURE 1 **Schematic diagram of the biomass pyrolysis pilot plant**

2.2 Biomass and catalysts

Lignocel BK40-90, supplied by the J. Rettenmaier & Söhne GMBH + CO, was used as biomass feedstock in all tests. The Lignocel is produced from forest timber by means of a process that includes steps of cutting, drying, milling, sieving and final packaging. The final product is supplied in a particle size range between 300 µm and 500 µm. The Lignocel moisture content was 7.4 % wt. and its elemental composition (wt%): C = 46.1%, H = 6.3%, N = 0.3%, O = 46.7% (calculated by difference) and ash = 0.6%.

Two catalysts, with acid and basic properties, were selected to the catalytic biomass pyrolysis experiments. The acid catalyst was a commercial equilibrium FCC catalyst (Ecat) collected directly from a Petrobras refinery. The Ecat comprises Y zeolite exchanged with rare earth and a matrix based on silica and alumina having surface area of 183 m^2/g. The basic material was a hydrotalcite-derived catalyst supplied by Albemarle surface area of 150 m^2/g. Its major component is the magnesium oxide.

A silica/kaolin composite was used to produce the reference bio-oil (non- catalytic pyrolysis). Its preparation consisted essentially of a mixture of 25% silica sol and 75% kaolin (hydrous aluminum silicate), followed by spray drying. After preparation, the surface area was decreased by hydrothermal deactivation at 800°C (5 hours, 100% steam) down to 50 m^2/g.

2.3 Experimental procedures

Biomass pyrolysis experiments were performed at two reaction temperatures: 450 and 550°C, representing the limits for maximization of the liquid effluent in the process of fast pyrolysis (6). Two tests were carried out for each operating condition (material/temperature), totalizing twelve tests. It was established for each test a duration of 1.5 hour, a limitation imposed by the loading capacity of the biomass hopper. A solid amount of 2600g (catalyst or inert) was used in each experiment, which is circulated in the flow range of 3000-4000 g/h. All biomass pyrolysis tests were performed using the following common parameters: screw conveyor rotation: 15%; regenerator temperature of 700 °C, cooling tower temperature of -5 °C; separator vessel temperature of 450 °C; N_2 flow in the reactor of 700 NL/h.

2.4 Product analysis

The bio-oils were submitted to a mass spectrometer (GC/MS), Shimadzu model QP-2010 Plus, with a DB-5 capillary column (methyl silicone 5% phenyl groups) 30 m long, 0.25 mm internal diameter and 0.25 mm thick stationary phase, in order to analyze their chemical composition, specially the oxygenated compounds. The carbon and hydrogen content of the bio-oils were determined using an Elemental Analyser (ASTM D 5291). The oxygen content was calculated by difference. The water content in the bio-oil was determined by Karl Fischer titration (ASTM E 203-01).

The pyrolysis gas components were determined by means of gas chromatography.

The coke yield was calculated through the analysis of the flue gas from the regenerator, which is obtained continuously online by means of a CO_2, CO and O_2 infrared analyzer.

3 RESULTS AND DISCUSSION

3.1 Pyrolysis Yields

Tables 1 and 2 list the yields of the pyrolysis products, gas, coke and liquid, calculated on a wet basis (considering the moisture in the biomass) for each material tested, at reaction temperatures of 450°C and 550°C, respectively. Figure 2 shows graphically the mean values as well as the error bars.

TABLE 1 **Biomass pyrolysis product yields at 450°C (wt% on a wet biomass basis)**

Test	***T.01***	***T.02***	***T.03***	***T.04***	***T.05***	***T.06***
Material	**Silica**	**Silica**	**Ecat**	**Ecat**	**HTC**	**HTC**
Biomass flow rate (g/h)	**618**	**801**	**703**	**578**	**602**	**684**
S/B (wt/wt)	**5**	**4**	**6**	**5**	**6**	**5**
t_{RES} (s)	**23.2**	**22.3**	**21.7**	**21.5**	**23.6**	**22.0**
Coke (wt%)	21.8	23.3	31.7	35.5	35.9	34.5
Liquid (wt%)	40.3	39.9	30.5	34.9	23.9	24.3
Gas (wt%)	18.4	16.3	14.3	17.1	15.5	15.2
$C_1 – C_4$ (wt%)	1.7	1.0	1.0	1.3	1.0	0.7
CO (wt%)	8.5	8.2	7.2	8.6	5.3	5.2
CO_2 (wt%)	6.8	6.9	5.9	6.6	8.9	10.3
Particulates (wt%)	2.6	1.6	2.6	1.4	1.3	6.0
Mass Balance (wt%)	83.0	81.1	79.1	89.1	76.6	80.0
CO_2 / CO (wt/wt)	0.8	0.8	0.8	0.8	1.7	2.0

TABLE 2 **Biomass pyrolysis product yields at 550°C (%wt on a wet biomass basis)**

Teste	***T.07***	***T.08***	***T.09***	***T.10***	***T.11***	***T.12***
Material	**Silica**	**Silica**	**Ecat**	**Ecat**	**HTC**	**HTC**
Biomass flow rate (g/h)	**716**	**790**	**758**	**701**	**765**	**808**
S/B (wt/wt)	**5**	**4**	**4**	**5**	**5**	**4**
t_{res} (s)	**19.2**	**19.0**	**18.6**	**19.2**	**18.1**	**17.8**
Coke (wt%)	17.1	16.2	23.6	22.4	30.7	27.3
Liquid (wt%)	35.3	37.9	32.9	34.4	28.2	23.5
Gas (wt%)	24.8	27.8	25.5	24.2	27.3	25.0
$C_1 – C_4$ (wt%)	3.4	4.0	3.5	3.2	3.5	2.8
CO (wt%)	15.3	14.5	15.0	15.2	12.1	9.0
CO_2 (wt%)	5.7	5.2	6.8	5.5	11.0	12.7
Particulates (wt%)	2.8	0.8	1.2	3.4	1.7	6.6
Mass Balance (wt%)	80.0	79.5	83.2	84.3	87.9	82.4
CO_2 / CO (wt/wt)	0.4	0.4	0.4	0.4	0.9	1.4

HTC – hydrotalcite t_{RES} – residence time of pyrolysis vapors in the reactor. S/B – circulating solid/ biomass flow ratio.

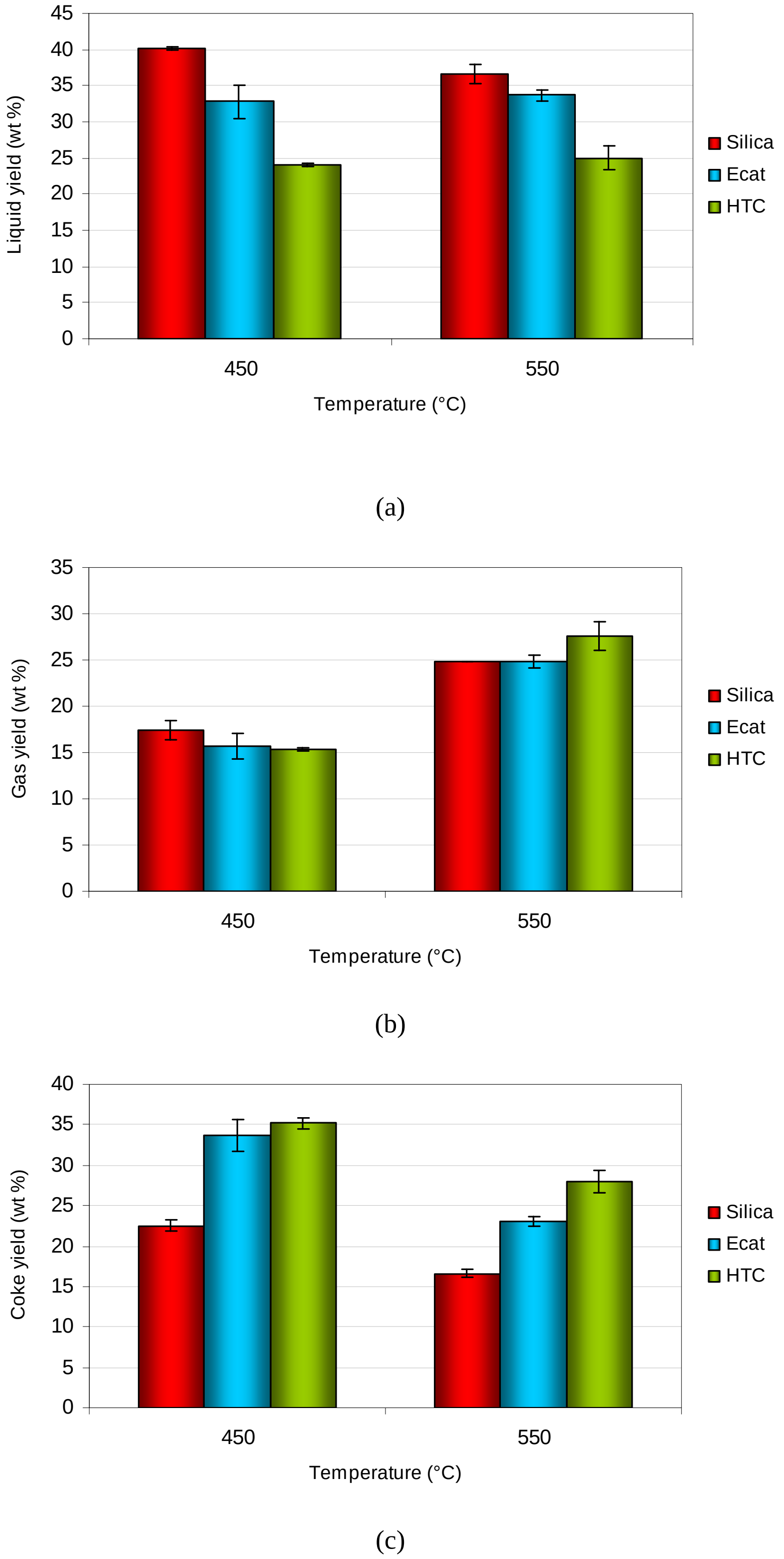

FIGURE 2 **Biomass pyrolysis product yields (wt% on wet biomass basis): (a) Liquid (b) Gas (c) Coke**

The mass balances were between 76 and 89%. This profile is commonly observed in studies of biomass pyrolysis in pilot and bench scale (7,8). The lack in mass balance is attributed to the product recovery system. Using the same rotation of the screw conveyor, the biomass flow rate load was in the range of 715 ± 75 g/h. The circulating solid flow rate to biomass feed rate ratio (S/B) range was 4-6 wt/wt.

As shown in Tables 1 and 2, the ranges found for the liquid yields (36-40%), gas (18-25%) and coke (17-23%) in tests with silica, considering the two temperatures (450 and 550°C) are similar to those found in the intermediate pyrolysis process cited by BRIDGWATER (9), where at temperature close to 500°C and using an inert, total liquid, gas and coke yields were 50%, 25% and 25% (wt% on wet biomass basis), respectively. This profile differs significantly from the one from fast pyrolysis where the yield of liquid may reach 75%, being gas and coal 13% and 12% respectively (1). The distinction between the two processes is given by the residence time of pyrolysis vapors in the reactor. Residence times up to 2 seconds are typical of fast pyrolysis, while a 10-30 seconds residence time range is typical for the intermediate pyrolysis. Further residence times result in an increase in secondary reactions of pyrolysis vapors causing excessive breakdown of the molecules, thus decreasing the yield of liquid and producing more gas and coke. Thus, since this study was carried out at residence times between 17.8 and 23.6 seconds, it can be categorized as intermediate pyrolysis. The tests at 550°C had lower residence time within this range due to the increase in the gas production.

3.2 Effect of the temperature

The effect of reaction temperature was more evident on gas and coke yields, having all materials (both catalysts and silica) the same behavior. As shown in Figures 2 (b) and 2 (c), gas yield increased with the increase of temperature from 450°C to 550°C, while the coke yield decreased, as already observed by other authors (10,11). According to ONAY and KOÇKAR (12), the decrease of coke yield at higher temperatures is related either with a higher primary decomposition of biomass or more secondary decomposition of the coke formed, while a larger secondary cracking of the pyrolysis vapors due to the higher temperature would cause an increase in the gas yield.

3.3 Effect of the catalyst

The influence of the catalyst was evident on coke and liquid yields and less significant for the gas as shown on Figure 2. All three materials showed similar gas yields at both temperatures studied. Since silica has a lower surface area than Ecat and hydrotalcite, this result indicates that the effect of residence time on the production of non-condensable gases in the intermediate pyrolysis prevails over the effect of secondary reactions on the surface of solids.

Compared to silica, hydrotalcite and Ecat resulted in lower yields of liquid and higher yields of coke. This behavior, when using catalytic materials, is described by several authors (7, 8). The loss in liquid yields can be attributed to the partial deoxygenation through water, CO and CO_2 formation. The lowest liquid yield and highest coke yield were obtained using the hydrotalcite. The basic catalysts are known as aldolic condensation reaction promoters (13), where high molecular weight ketones are produced and may be regarded as coke formation precursors. Comparing Ecat with hydrotalcite, differences in yields of liquid are maintained constant for the two temperatures, whereas for the coke this difference was increased at 550°C, indicating that the effect of temperature increase in the yield of coke is more evident with the acid catalyst.

3.4 Pyrolysis gas composition

The yields of the pyrolysis gas components are shown in Figure 3 for each temperature and type of material used. The presence of oxygenated compounds (CO, CO_2) was predominant in the gas composition, while light hydrocarbons (C1-C4) were found in smaller quantities.

As shown in Figure 4, tests with silica and Ecat showed CO yields higher than CO_2 at both temperatures, while the opposite behavior was obtained with hydrotalcite. The results found for hydrotalcite indicate that basic catalysts favor the deoxygenation by decarboxylation, while the acid catalyst and silica favor decarbonylation reactions. The CO_2 production through ketones formation reactions from carboxylic acids catalyzed by basic catalysts is reported in the literature (13). The removal of oxygen as CO_2 is preferred because only one carbon atom is required for the removal of two oxygen atoms, thus reducing the loss of the organic fraction in the bio-oil.

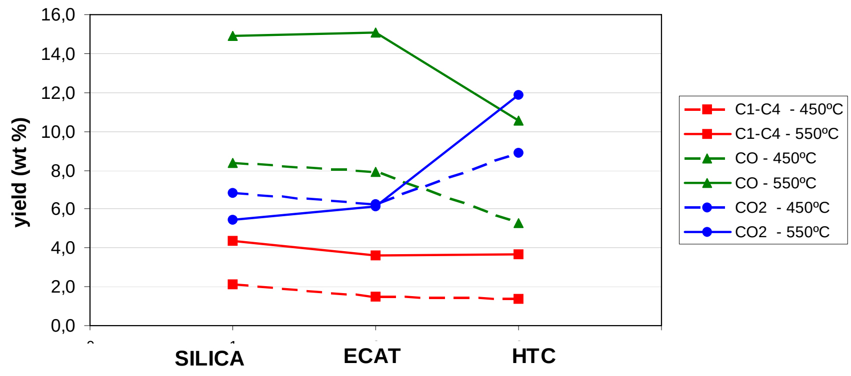

FIGURE 3 **Pyrolysis gas components yields (wt% on wet biomass basis)**

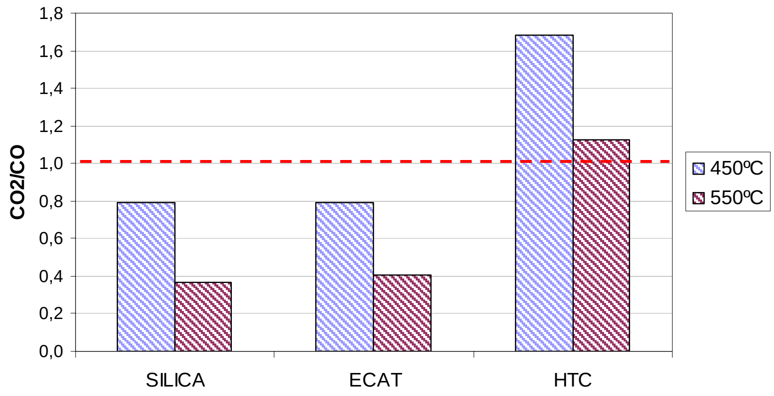

FIGURE 4 **CO to CO_2 ratio**

3.5 Bio-oil quality

A two-phased liquid product was obtained in all tests at 550 °C and tests with silica at 450 °C. These phases were classified as aqueous (water-rich) and organic phase. Tests with Ecat and hydrotalcite at 450°C generated one-phased bio-oils with similar appearance and composition to the aqueous phases of their respective tests at 550°C. According to OASMAA et al. (14), phase separation takes place when the amount of water in the mixture exceeds a certain level, causing the separation of the heavier compounds (derived from lignin) and the hydrophilic polar organic compounds originated from the degradation of cellulose and hemicellulose, such as light carboxylic acids, alcohols and ketones. The production of a two-phased bio-oil is typical of the intermediate pyrolysis process, where long residence times are applied, causing secondary reactions of pyrolysis vapors, thereby producing lower molecular weight components and water.

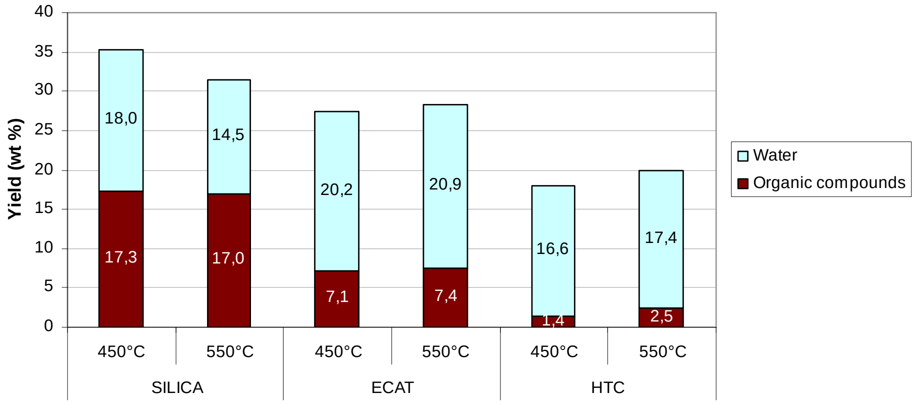

FIGURE 5 **Water and organic compounds yields in bio-oils (wt% on dry biomass basis)**

From the results of elemental analysis and Karl Fischer of the bio-oils, the water and organic compounds yields were calculated on a dry basis, i.e., considering that the biomass feedstock contains 0% of moisture (FIGURE 5). Tests with Ecat and hydrotalcite had lower yields of organic compounds than tests with silica. As expected, Ecat had the highest water yield among the three materials, since the deoxygenation by dehydration is an expected behavior for acid catalysts (7, 15). In the tests with hydrotalcite were found the lowest yields of organic compounds, which can be attributed to its higher coke formation, since the gas yields were similar to those found with the two other materials. However, there was a low production of organic compounds even with silica. The silica low surface area indicates that the secondary reactions which occur in the gas phase due to increased residence time of the pyrolysis vapors contribute strongly to the dehydration and cracking of products.

According to Figure 5 there was no significant influence of reaction temperature on the water yield and organic compounds. Liquid yields, calculated on a dry basis, were about 5% lower than those calculated on a wet basis.

3.6 Deoxygenation extension

The deoxygenation can be understood as the fraction of oxygen present in the biomass that is converted into water, CO and CO_2 (Equation 1). The oxygenated compounds are part of the composition of the bio-oil and their removal, associated with the increase in hydrocarbons production, is the goal of most studies of catalytic pyrolysis. Oxygen can also be present in the composition of the coke, which is largely formed from lignin by condensation and hydrogen transfer reactions. However the oxygen present in the coke was not measured. It is released in the form of CO and CO_2 in the combustion process.

$$\text{\% Deoxygenation} = \frac{O_{H2O} + O_{CO} + O_{CO2}}{O_{feed}} \times 100 \qquad (1)$$

The deoxygenation ratio was obtained from results of elemental analysis and water content, calculated on a dry basis. Tests at temperature of 450°C obtained the lowest deoxygenation values, 55%, 57% and 63% for silica, hydrotalcite and Ecat, respectively. However, the largest losses in the oxygen balance were observed at this temperature. In the tests at 550 °C (74%), the Ecat also obtained the best results, followed by hydrotalcite (71%) and silica (60%).

However, the high water content in bio-oil indicated that most of the oxygen was removed as water. The ratios of oxygen removed as water to the oxygen removed as CO and CO_2 are shown in Figure 5. In the tests at 450 ° C, the effect of dehydration was higher than at 550 ° C for all materials tested, with values above 1. WILLIAMS and NUGRANAD (16) also observed the same behavior, showing that the mechanism for oxygen removal changes when the temperature increases. Ecat promoted greater deoxygenation by dehydration, being the only material that exhibited this behavior at both reaction temperatures. It is worth mentioning that the deoxygenation by decarboxylation or decarbonylation are more attractive when compared to dehydration, since it compromises less the hydrogen content of the liquid product, which allows improving its heating value.

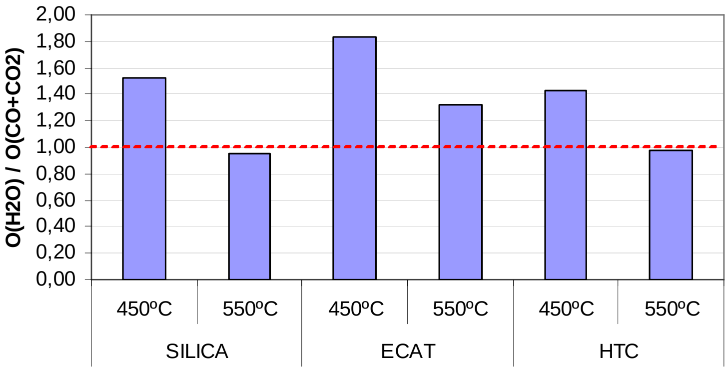

FIGURE 6 **Oxygen removal ratios (ratios of oxygen removed as water to oxygen removed as CO and CO_2)**

3.7 Bio-oil composition

The compounds in the bio-oils analyzed by GC-MS were grouped as ketones, aldehydes, ethers, hydrocarbons, phenols, acids, esters and alcohols. Table 3 shows the most predominant compounds for each group. Figure 7 shows the yields (in weight on dry biomass basis) of each group. The yields of these compounds were calculated by multiplying the GC-

MS area percent in both aqueous and organic phases and each liquid phase yield, assuming that the correction factor for each compound is equal to unity.

The phenolic compounds were the most dominant in the bio-oil. These compounds are largely derived from the breaking chains of lignin. Phenol, methyl-phenol, methyl-benzenediol and benzenediol were the phenolic compounds found at higher concentrations. Figure 7 shows that the temperature increase favors the formation of phenolics. This can be explained by the fact that at the temperature of 450 °C, much of the lignin has been converted into coke, instead of phenols. Only tests using silica (at both temperatures) and Ecat (at 550°C) showed significant yields of phenolics. These yields were comparable to those found in other pyrolysis *in situ* studies with Ecat in literature (LAPPAS et al. (7) and AHO et al. (17)), while the yields obtained from the tests with silica were higher in the present study. Figure 8 shows the distribution of phenolic compounds found in the largest quantities. Tests with silica promoted the formation of higher molecular weight phenols. The comparison between silica and Ecat tests at 550 ° C suggests that the dealkylation of methyl-phenol lead to an increased phenol production with Ecat. Phenolic compounds produced from the catalytic pyrolysis can be a major source of aromatics production after hydrodeoxygenation (18).

As for the ketones, cyclopentanones were found in high quantities. Moreover, a higher temperature leads to a decrease in the ketone yields in the presence of any of the materials studied.

The aldehydes were observed in higher concentration in the tests with silica, being furfuraldehyde the most abundant. In the ethers group, compounds such as benzofurans and methoxybenzene were identified. In the tests with Ecat and hydrotalcite at 450°C, where single-phased bio-oils were obtained, ethers with lower molecular weight were found and dimethyl furan was the predominant compound.

TABLE 3 **Predominant compounds found for each chemical group**

Chemical families	**Compounds**
Phenols	Phenol, methylphenol, benzenediol, methylbenzenediol, methoxyphenol
Ketones	Cyclopentanones
Aldehydes	Furfuraldehyde
Ethers	Benzofurans, methoxybenzene, dimethylfuran
Hydrocarbons	Alkylbenzenes, Alkylnaphthalenes, naphthalene, indenes

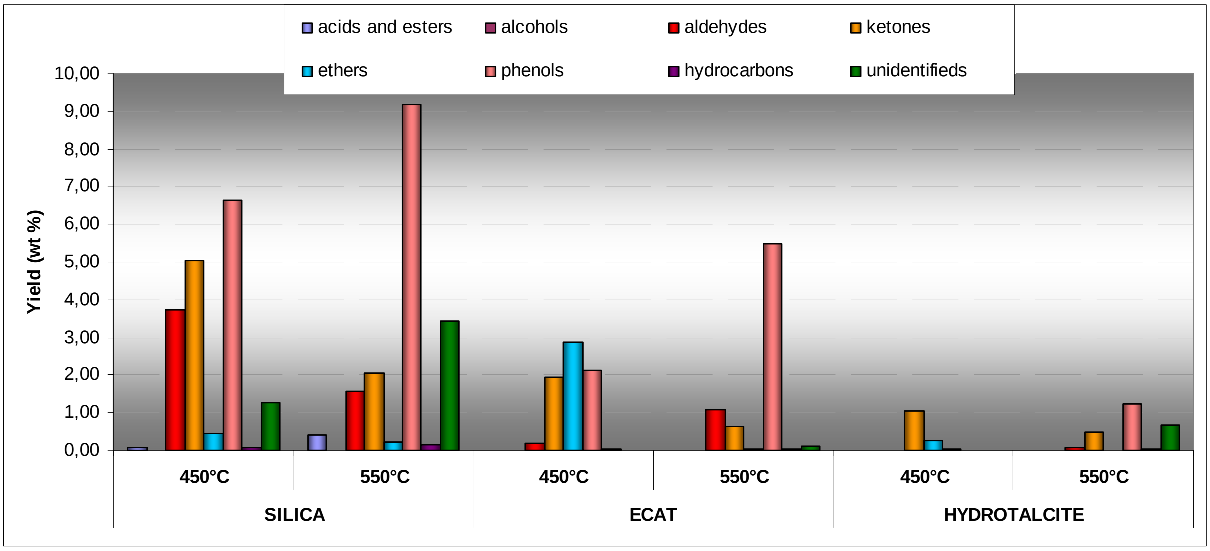

FIGURE 7 **Bio-oil compounds yields (wt% on dry biomass basis)**

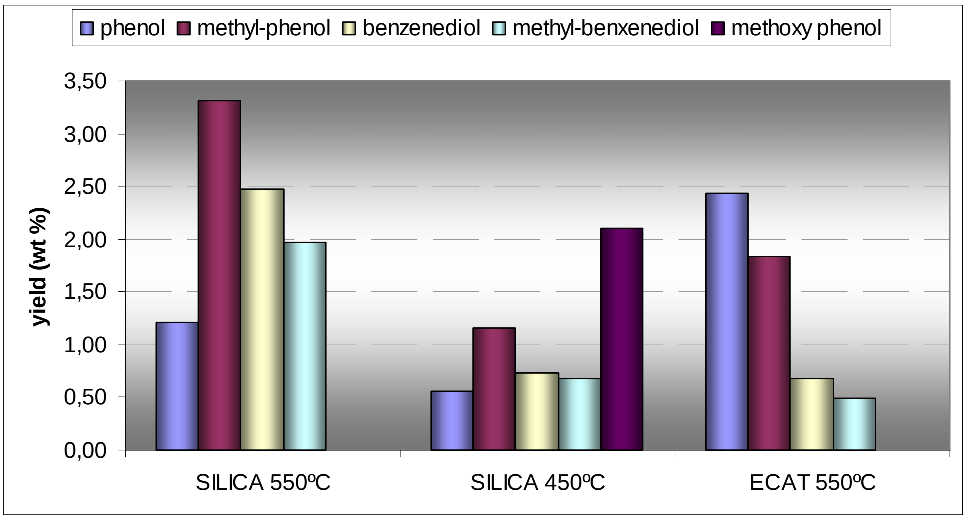

FIGURE 8 **Phenolic compounds yields with Ecat at 550°C and silica at 450°C and 550°C (wt% on dry biomass basis)**

High molecular weight sugars are not volatile enough to be detected by GC-MS and the use of an HPLC is more appropriate. Only levoglucosan, a monosaccharide formed from cellulose, can be detected by GC, and its presence in the aqueous phase is usually reported in studies of biomass fast pyrolysis (4). However, in this study sugars were not found even in the aqueous phases. It is possible that the higher residence times obtained in the intermediate pyrolysis process may degrade sugars into carbon dioxide, aldehydes, ketones and furans.

Hydrocarbons were detected only in the catalytic tests, where aromatic and polyaromatic compounds were found. Alkylbenzenes were identified in tests with Ecat and the most dominating compounds were those derived from naphthalene. In the tests with hydrotalcite, the fractions were equally distributed among alkylbenzenes, alkylnaphthalenes and indenes. The production of aromatic and polyaromatic using both acid and basic catalysts has been observed by several authors (16,19), but in terms of yields, none of the catalysts studied showed significant amounts of hydrocarbons. Lappas et al. (7) also obtained low hydrocarbons yields with an FCC catalyst. The use of ZSM-5 in catalytic pyrolysis has shown a better selectivity towards aromatic hydrocarbons production, but in different reaction systems, as demonstrated by OLAZAR et al. (11), ZHANG et al. (15) and WILLIAMS and NUGRANAD (16). The higher severity of the intermediate pyrolysis process, obtained through higher residence times, can lead to a higher conversion of hydrocarbons into non-condensable gases. The polymerization of aromatic compounds formed during the reaction and the resulting coke formation can also be a factor to loss of hydrocarbons yields.

Acids, esters and alcohols were the least dominant groups. The highest amounts of non-identified peaks were obtained in the tests with hydrotalcite and silica at 550°C as can be observed in Figure 7.

4 CONCLUSIONS

Higher residence times in the pyrolysis of biomass were determinant for yield profiles and chemical composition of bio-oil, which were clearly distinguishable from those found in the literature for the fast pyrolysis. It was also shown in this study that the differences in chemical composition and textural characteristics of an acid catalyst, a basic catalyst and an inert material visibly influenced product yields of the pyrolysis, chemical composition and the appearance of bio-oils.

Tests at temperatures of 550°C are more adequate to produce higher amounts of liquid products and decrease coke yields. Moreover, higher deoxygenation rates and better quality bio-oils are obtained at this temperature.

Analyzing the composition of bio-oils from the perspective of direct biofuels production, without the need for any hydroprocessing, the materials studied here did not result in considerable yields of hydrocarbons. The intermediate pyrolysis conditions provided bio-oils with a phenolic profile.

Among the materials investigated, hydrotalcite was the one that presented the least satisfactory results, producing low yields of organic compounds under the operating conditions used in this study, which are typical of intermediate pyrolysis.

The most acceptable results in terms of yields and bio-oil quality were obtained with silica. Its smaller surface area and its inert characteristics were more suitable for the pyrolysis process carried out at the intermediate severity conditions used in this study, due to the large contribution of secondary reactions in the gas phase as a function of the high residence time in the reactor.

5 ACKNOWLEGMENTS

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(17) SEWAGE SLUDGE GASIFICATION. DOLOMITE PERFORMANCE UNDER DIFFERENT OPERATING CONDITIONS

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EXECUTIVE SUMMARY

Gasification is a technology that can replace traditional management alternatives used up to date to deal with this waste (landfilling, composting and incineration) and which fulfils the social, environmental and legislative requirements. The main products of sewage sludge gasification are permanent gases (useful to generate energy or to be used as raw material in chemical synthesis processes), liquids (tars) and char. One of the main problems to be solved in gasification is tar production. Tars are organic impurities which can condense at relatively high temperatures making impossible to use the produced gases for most applications.

This work deals with the effect of some primary tar removal processes (performed inside the gasifier) on sewage sludge gasification products. For this purpose, analysis of the gas composition, tar production, cold gas efficiency and carbon conversion were carried out. The tests were performed with air in a laboratory scale plant consisting mainly of a bubbling bed gasifier. No catalyzed and catalyzed (10% wt of dolomite in the bed and in the feeding) tests were carried out at different temperatures (750ºC, 800ºC and 850ºC) in order to know the effect of these parameters in the gasification products. As far as tars were concerned, qualitative and quantitative tar composition was determined. In all tests the Equivalence Ratio (ER) was kept at 0.3.

Temperature is one of the most influential variables in sewage sludge gasification. Higher temperatures favoured hydrogen and CO production while CO_2 content decreased, which might be partially explained by the effect of the cracking, Boudouard and CO_2 reforming reactions. At 850ºC, cold gas efficiency and carbon conversion reached 49% and 76%, respectively. The presence of dolomite as catalyst increased the production of H_2 reaching contents of 15.5% by volume at 850 °C. Similar behaviour was found for CO whereas CO_2 and C_nH_m (light hydrocarbons) production decreased. In the presence of dolomite, a tar reduction of up to 51% was reached in comparison with no catalyzed tests, as well as improvements on cold gas efficiency and carbon conversion.

Several assays were developed in order to test catalyst performance under more rough gasification conditions. For this purpose, the throughput value (TR), defined as kg sludge “as received” fed to the gasifier per hour and per m^2 of cross sectional area of the gasifier, was modified. Specifically, the TR values used were 110 (reference value), 215 and 322 $kg/h\cdot m^2$. When TR increased, the H_2, CO and CH_4 production decreased while the CO_2 and the C_nH_m production increased. Tar production increased drastically with TR during no catalysed tests what is related to the lower residence time of the gas inside the reactor. Nevertheless, even at $TR=322\ kg/h\cdot m^2$, tar production decreased by nearly 50% with in-bed use of dolomite in comparison with no catalyzed assays under the same operating conditions.

Regarding relative tar composition, there was an increase in benzene and naphthalene content when temperature increased while the content of the rest of compounds decreased. The dolomite seemed to be effective all over the range of molecular weight studied showing tar removal efficiencies between 35-55% in most cases. High values of the TR caused a significant increase in tar production but a slight effect on tar composition.

1 INTRODUCTION

The European legislation restricts many traditional management options for sewage sludge such as direct use for cultivation and landfilling. Due to the fact that incineration is subject to strong social opposition in countries like Spain, gasification arises like one of the most attractive alternatives for the management of this waste (Seggiani, 2012).

Sewage sludge gasification is the thermal process by which the carbonaceous content of sewage sludge is converted to combustible gas and a solid waste (char) in a net reducing atmosphere.

Unfortunately, the produced gas contains impurities like dust particles, sulphur, nitrogen and chlorine compounds and tars that must be removed to meet the requirements of end-use applications. In fact, one of the main problems to be solved in gasification is tar production. Tars are organic impurities which can condense at relatively high temperatures making impossible to use the produced gas for most applications. The tar content and the gas composition mainly depend on the gasification conditions (temperature, pressure, and residence time), the gasifying agent, the type of gasifier used and the presence of catalysts for tar destruction/reforming (Šulc, 2012).

Regarding gasifying agents, the most widely used are air, oxygen, steam or mixtures thereof (Campoy, 2010; Meng, 2011). As far as the type of gasifier is concerned, most of the gasification plants consist of fixed bed, fluidized bed and entrained flow gasifiers (Hernández, 2010; Seggiani, 2012; Šulc, 2012). The gasification conditions may be very different depending on the system chosen. For instance, temperatures between 800 and 900 ºC are typical in atmospheric air-blown bubbling fluidized bed gasifiers whereas temperatures of up to 1100ºC or even 1500ºC are typical for fixed bed and entrained flow gasifiers, respectively (Gómez-Barea, 2012; van der Drift, 2004). Finally, regardless of their chemical composition, the catalysts used in gasification processes can be classified into primary (when located inside the gasifier) or secondary (when located downstream the gasifier). Although the efficiency of secondary methods for tar removal has been extensively demonstrated (Asadullah, 2004; Huang, 2012) major ongoing research is focused on primary methods because they are less complex and expensive than secondary ones (Devi, 2003). Typical in-bed catalysts are dolomite, olivine and alumina (Corella, 2004; de Andrés, 2011). More effective nickel-based catalysts improve tar reduction in the bed, but the rapid degradation of the catalyst makes this option unfeasible so far (Gómez-Barea, 2011). Thus, taking into account both effectiveness in tar removal and the resistance to deactivation, the dolomite seems to be the most attractive primary catalyst for sewage sludge gasification, according to the present knowledge.

This work deals with the effect of the temperature and the use of dolomite as primary catalyst on sewage sludge gasification products, with special attention to tar production. For this purpose, analysis of the gas composition, tar production (Y_{tar}), cold gas efficiency (CGE) and carbon conversion (X_C) are carried out. Regarding tar production, catalysts performance can be greatly influenced by the throughput (TR, hereinafter), defined as the kilograms of sewage sludge as received fed to the gasifier per hour and per·m^2 of cross sectional area of the gasifier. According to Corella (2008), some studies developed at small scale use very low TR (soft conditions, TR close to 100-150 kg/h•m^2). As a result of that, tar removal efficiencies found may be very different from those obtained at commercial scale, with TR around 750 kg/h•m^2. For this reason, several assays were developed in order to test dolomite performance under TR of 125, 250 and 375 kg/h•m^2.

It is widely accepted that the final use of the produced gas defines the need for tar conversion. However, according to Gómez-Barea (2012), the key parameter for the assessment of the suitability of the gas for a given application is the nature of the tar, not only the tar concentration. This can be explained by the fact that the tar composition determines the dew point of the gas. For this reason, qualitative determinations of tar composition were carried out by gas chromatography-mass spectrometry (GC/MS) to know how the different gasification conditions and the presence of dolomite affect the tar composition.

2 MATERIALS AND METHODS

2.1 Materials

The dried sludge samples were received from a sewage sludge drying plant of Madrid, Spain. The elemental analyses of the dried sewage sludge are shown in Table 1. These data were used to estimate the low heating value (LHV) of the sludge (13.1 MJ/kg) by means of the modified Dulong’s formula (de Andrés, 2011). Silica sand (and catalyst, if necessary) was used as the bed material. The sludge was crushed and sieved to particle size between 300 and 500 µm. The dolomite was supplied by Dolomitas del Norte S.A., Spain.

TABLE 1 Proximate and elemental analysis of sewage sludge (dry basis) from a sewage sludge drying plant

Parameter		**Sludge**	**Analytical method**
Moisture (%)		7.9	UNE-EN 12880-2001
Organic Mat. (%)		55.4	UNE-EN 12879-2001
Ash (%)		44.6	UNE-EN 12879-2001
Carbon (%)		30.7	Elemental micro analyser LECO CHNS-932
Nitrogen (%)		4.3	
Hydrogen (%)		5.0	
Sulphur (%)		1.6	
Oxygen (%)	by difference	13.8	

2.2 Laboratory scale plant

Experiments were carried out in a laboratory scale plant. The reactor used was a stainless steel fluidised bed gasifier with a total height of 700 mm and an inner diameter of 32 mm followed by a freeboard. Both the reactor and the freeboard were heated by an electrical furnace. Inside the gasifier, the bed was held by a distributor plate (0.1- mm pore size). The gasifying agent (air) entering the reactor was electrically preheated.

The sludge was fed into the reactor a few millimetres above the distributor plate by a dosing system consisting of a hopper and two screw feeders (the dosing and launch screw feeders). The bed height was kept at 100 mm by a concentric pipe inserted through the distributor plate.

Downstream of the freeboard, a cyclone and a micronic filter were placed inside a hot box (250 ºC) to prevent condensation of the tars. Tar collection was done following the tar protocol and tar production was determined by weighting after distillation.

Gas production was measured by a mass flow meter. The dry gas composition (N_2, O_2, H_2, CO, CO_2, CH_4, C_2H_6 and C_2H_4) was determined by means of a micro gas chromatograph and the tar composition by gas chromatography/mass spectrometry (GC/MS). More detailed information and a diagram of the plant used can be found in de Andrés (2011).

2.3 Experimental conditions

The tests carried out to know the influence of different parameters on the gasification products can be divided into three main groups:

- Influence of the temperature: a set of tests was conducted at temperatures of 750ºC, 800ºC and 850ºC The equivalence ratio, ER (defined as the ratio between the flow rate of the air introduced into the reactor and the stoichiometric flow rate of the air required for a complete combustion of the sludge), was set at 0.3. It was decided to set the ER to this value because, according to previous experiences, under this conditions tar production was relatively low (6 g/Nm^3) and the LHV of the gases remained within acceptable levels (around 4 MJ/Nm^3).
- Influence of in-bed use of dolomite: different tests were carried out at 750ºC, 800ºC and 850ºC with a constant ER of 0.3 and with a dolomite content in the bed and in the feeding of 10% by weight.
- Influence of the throughput, TR: the performance of the dolomite under different TR (110, 225 and 322 kg/h•m^2) was tested by modifying the flow rate of sludge fed to the gasifier (Table 2). The ER was fixed at 0.3 to be consistent with the before mentioned experiments. For this purpose, the air flow rate introduced in the gasifier was modified and therefore the fluidizing velocity (4, 8 and 12 times the minimum fluidization velocity, u_{mf} (850ºC) = 3.1 cm s^{-1}, respectively).

The conditions and results of the tests carried out are shown in Table 2 and Table 3. Prior to each test 80 g of silica sand (or sand/dolomite mixture in tests with catalyst) were placed in the gasifier. Once the temperature of the test had been reached, the gasifier was continuously fed with sludge and a specific sand-catalyst mixture (20% of the mass rate of fed sludge). The sand-catalyst proportion of the mixture in each test is shown in Table 2 and Table 3 (20-0% or 10-10% depending on the test). The sand introduced in the feeding was used to improve fluidization. The total duration of the tests was 60 min.

To validate each test, it was decided that the experiment closure mass balance should be between 95% and 105%.

3 RESULTS AND DISCUSSION

A summary of the results of the gasification tests carried out is presented in Table 2 and Table 3.

TABLE 2 Results of gasification experiment without catalyst. Effect of temperature and throughput (TR)

Parameter	**Units**	**T_1**	**T_2**	**T_3**	**T_4**	**T_5**	**T_6**	**T_7**	**T_8**	**T_9**
Temperature	ºC	750	800	850	750	800	850	750	800	850
Sludge	g/min	1.2	1.2	1.2	2.5	2.5	2.5	3.7	3.7	3.7
Sand	% fed sludge	20	20	20	20	20	20	20	20	20
TR	kg/h•m^2	110	110	110	215	215	215	322	322	322
u/u_{mf}		3.6	3.8	4.0	7.3	7.6	8.0	10.9	11.5	12.0
Residence time	s	7.8	7.5	7.1	3.9	3.7	3.6	2.6	2.5	2.4
H_2		9.4	11.9	13.4	7.6	9.5	11.2	5.3	8.5	10.6
N_2		63.2	61.0	58.7	65.6	63.6	59.9	67.7	63.3	60.7
CH_4		3.0	2.8	3.0	2.7	2.6	3.2	2.6	3.0	2.9
CO		6.9	7.9	9.28	6.7	7.5	9.2	7.0	8.2	9.4
CO_2		13.4	13.2	12.59	13.6	13.2	12.5	13.5	13.4	12.8
C_2H_6		0.11	0.06	0.03	0.17	0.07	0.03	0.14	0.04	0.04
C_2H_4		1.8	1.8	1.6	2.0	2.0	1.8	2.1	2.1	2.1
LHV gas	MJ/Nm^3	3.1	3.4	3.8	2.7	3.0	3.6	2.5	3.1	3.5
Gas production	Nm^3/kg sludge, daf	2.7	2.8	3.0	2.7	2.8	2.9	2.6	2.7	2.8
Tar concentration	g/Nm^3	7.4	4.1	2.4	12.2	7.7	4.2	15.6	9.8	5.8
Y_{tar}	mg/g sludge, daf	20.3	11.8	7.1	32.4	21.4	12.1	40.8	26.5	16.1
Xc	%	66.9	70.6	76.4	65.0	68.1	75.1	64.1	69.9	73.8
CGE	%	36.8	42.0	49.4	31.8	32.7	46.0	28.3	36.8	42.5
Char	g/kg daf	58.1	42.3	21.1	51.5	43.6	30.4	56.4	48.5	22.0

TABLE 3 Results of gasification experiment with dolomite. Effect of temperature and throughput (TR)

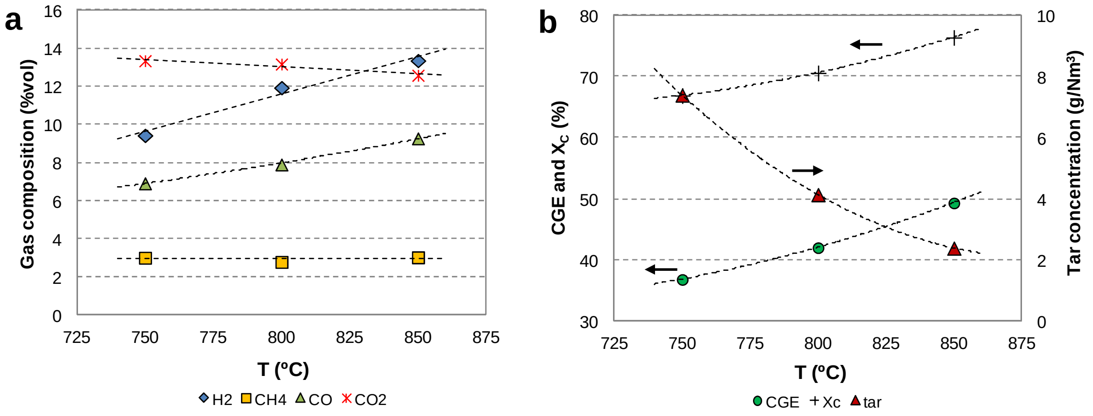
Parameter	**Units**	**T_10**	**T_11**	**T_12**	**T_13**	**T_14**	**T_15**	**T_16**	**T_17**	**T_18**
Temperature	ºC	750	800	850	750	800	850	750	800	850
Sludge	g/min	1.2	1.2	1.2	2.5	2.5	2.5	3.7	3.7	3.7
Sand	% fed sludge	10	10	10	10	10	10	10	10	10
Dolomite	% fed sludge	10	10	10	10	10	10	10	10	10
TR	kg/h•m^2	110	110	110	215	215	215	322	322	322
u/u_{mf}		3.6	3.8	4.0	7.3	7.6	8.0	10.9	11.5	12.0
Residence time	s	7.8	7.5	7.1	3.9	3.7	3.6	2.6	2.5	2.4
H_2		10.8	12.7	15.5	8.5	10.7	13.1	6.5	9.6	12.1
N_2		63.1	58.5	56.7	65.0	62.5	58.8	68.7	63.3	60.1
CH_4		2.8	3.2	2.8	2.8	2.6	2.6	1.9	2.5	2.4
CO		5.3	9.7	11.6	5.7	6.8	10.4	5.4	6.8	9.0
CO_2		14.4	12.6	10.9	13.7	13.9	12.1	14.2	14.2	13.1
C_2H_6		0.11	0.04	0.02	0.18	0.08	0.03	0.12	0.09	0.04
C_2H_4		1.8	1.7	1.0	2.0	1.8	1.5	1.5	1.9	1.8
LHV gas	MJ/Nm^3	2.9	3.9	4.3	2.7	3.1	3.8	2.1	2.9	3.4
Gas production	Nm^3/kg sludge, daf	2.8	2.9	3.1	2.7	2.797	2.914	2.6	2.781	2.8
Tar concentration	g/Nm^3	3.5	2.1	1.3	7.5	4.7	2.4	9.8	6.3	3.3
Y_{tar}	mg/g sludge, daf	9.8	6.0	4.0	19.8	13.1	6.9	25.8	17.4	9.3
Xc	%	66.6	76.7	78.7	62.5	68.5	75.1	58.8	68.7	72.4
CGE	%	36.0	48.8	56.9	31.6	37.1	47.7	24.3	35.0	42.2
Char	g/kg daf	68.7	47.6	23.8	79.3	35.7	21.1	49.3	30.0	15.9

3.1 Effect of the temperature on the gasification products

Fig. 1a shows the effect of the temperature on the gas composition under a TR of 110 kg/h•m^2 (reference TR in this work). The H_2 and CO content increased with the temperature while the CO_2 concentration decreased.

It can be explained by effect of the CO_2 reforming reactions and the Boudouard reaction, especially at the highest temperature (850ºC). As a result of that, the CO/CO_2 ratio increased from 0.51 at 750ºC to 0.74 at 850ºC. Slight differences were found for the CH_4 content whereas the concentration of C_nH_m (C_2H_6 and C_2H_4) decreased as the temperature increased (Table 2). Regarding tar production, reductions of up to 67% were found by increasing the temperature from 750ºC to 850ºC (Fig. 1b). Although not shown, both the LHV of the produced gas and the gas production (Y_{gas}) increased with the temperature. Higher temperatures favoured the production of combustible gases as well as a more intense volatilisation of the sludge, the decomposition of the tars and the conversion of the char (Campoy, 2009). The cold gas efficiency (CGE) varied between 37% (at 750 °C) and 49% (at 850 °C) while carbon conversion (X_C) varied between 67% (at 750 °C) and 76% (at 850 °C).

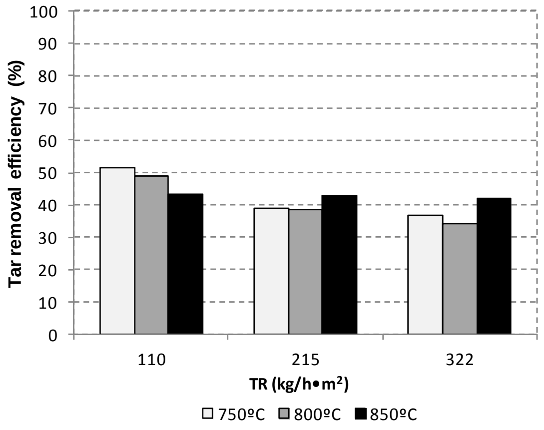
FIGURE 1 Effect of the temperature on gasification products (TR=110 kg/h•m^2). (a) effect on gas composition; (b) effect on cold gas efficiency (CGE), carbon conversion (X_C) and tar concentration (tar)



3.2 Influence of in-bed use of dolomite

The presence of dolomite has a clear effect on the gasification products. As far as gas composition was concerned, the dolomite increased the H_2 and CO content in comparison to tests without catalyst. However, the production of CO_2 and C_nH_m decreased. These results can be explained by the prevalence of the cracking and the CO_2 reforming reactions. No relevant differences were found regarding CH_4 content. Fig. 2 shows the tar removal efficiency obtained with the dolomite. As it can be seen, the reductions found slightly varied depending on the temperature. Specifically, under the reference TR of 110 kg/h•m^2, the tar removal efficiency ranged between 51%, at 750ºC, and 44%, at 850ºC.

FIGURE 2 Tar removal efficiency of the dolomite as a function of the throughput (TR) and the temperature

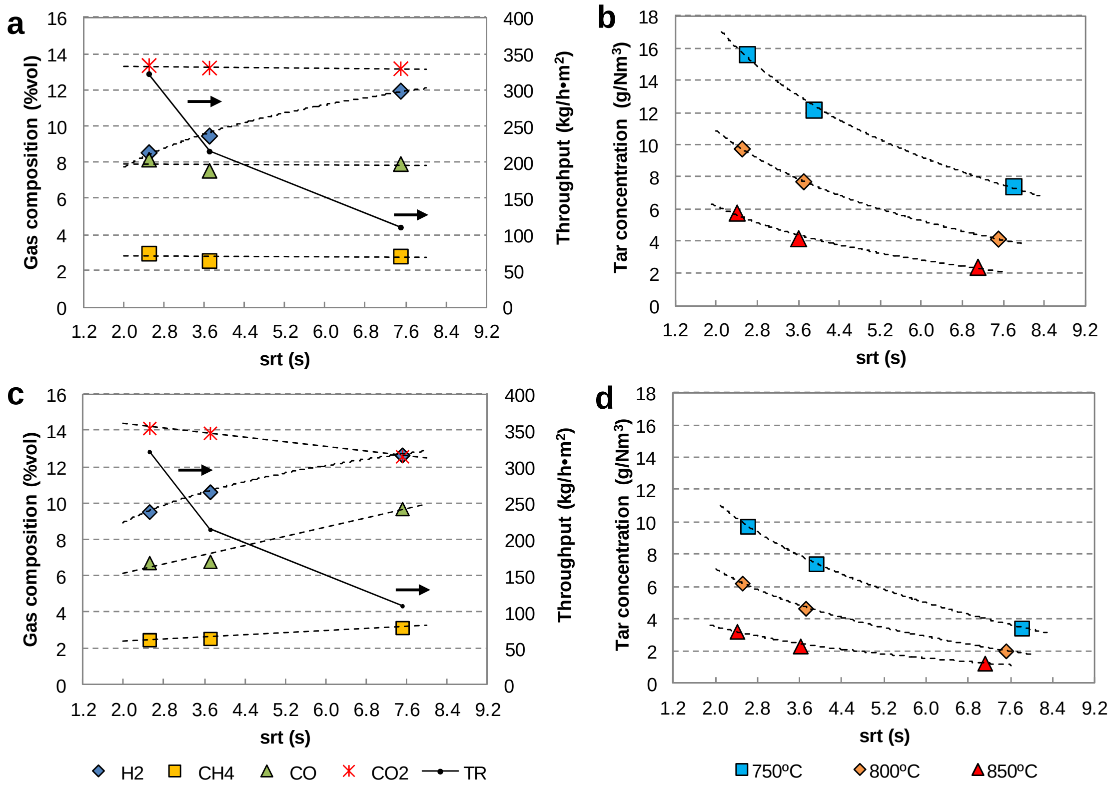


The changes in gas composition (and hence in their LHV) and the slight increase in gas production related to the conversion of tars into permanent gases, modified the carbon conversion and the cold gas efficiency. On average, under the reference TR, the carbon conversion increased 4% and the cold gas efficiency 10% when dolomite was used.

3.3 Influence of the throughput, TR

Fig. 3 shows the effect of the throughput (TR) on the produced gas composition and the tar concentration. Actually, the data are presented as a function of the space residence time (srt) of the gas in the gasifier (*srt*, is defined as the gasifier volume divided by the air volumetric flow rate). As previously explained in Seccion 2, the TR was modified between 110, 225 and 322 kg/h•m^2 by increasing the flow rate of sludge fed to the gasifier. In order to keep the ER at 0.3, the air flow rate introduced in the gasifier and thus, the *srt*, had to be modified (Table 2). The trends shown in Fig. 3a for 800ºC were similar to those found for the other temperatures in tests without catalyst. There was an increase in H_2 content with the *srt* while the CH_4, CO and CO_2 production slightly changed. According to Chen (2003), the longer is the residence time the higher is the cracking reaction, which would partially explain the increase in H_2 content and the decrease in C_nH_m production obtained by increasing the *srt* (Table 2). Different results were found in tests with dolomite (Fig. 3c). It can be seen that the production of H_2, CO and CH_4 increased by increasing the *srt* while the CO_2 content decreased. These results are in agreement with Hernández (2010), who stated that an increase of the *srt* increases the concentration of the combustible species in the producer gas as a result of the closer approach to equilibrium values.

FIGURE 3 Effect of the throughput (TR) on the gasification products. (a) effect on gas composition at 800ºC in tests without catalyst; (b) effect on tar concentration at 750ºC, 800ºC and 850ºC in tests without catalyst; (c) effect on gas composition at 800ºC in tests with dolomite; (d) effect on tar concentration at 750ºC, 800ºC and 850ºC in tests with dolomite



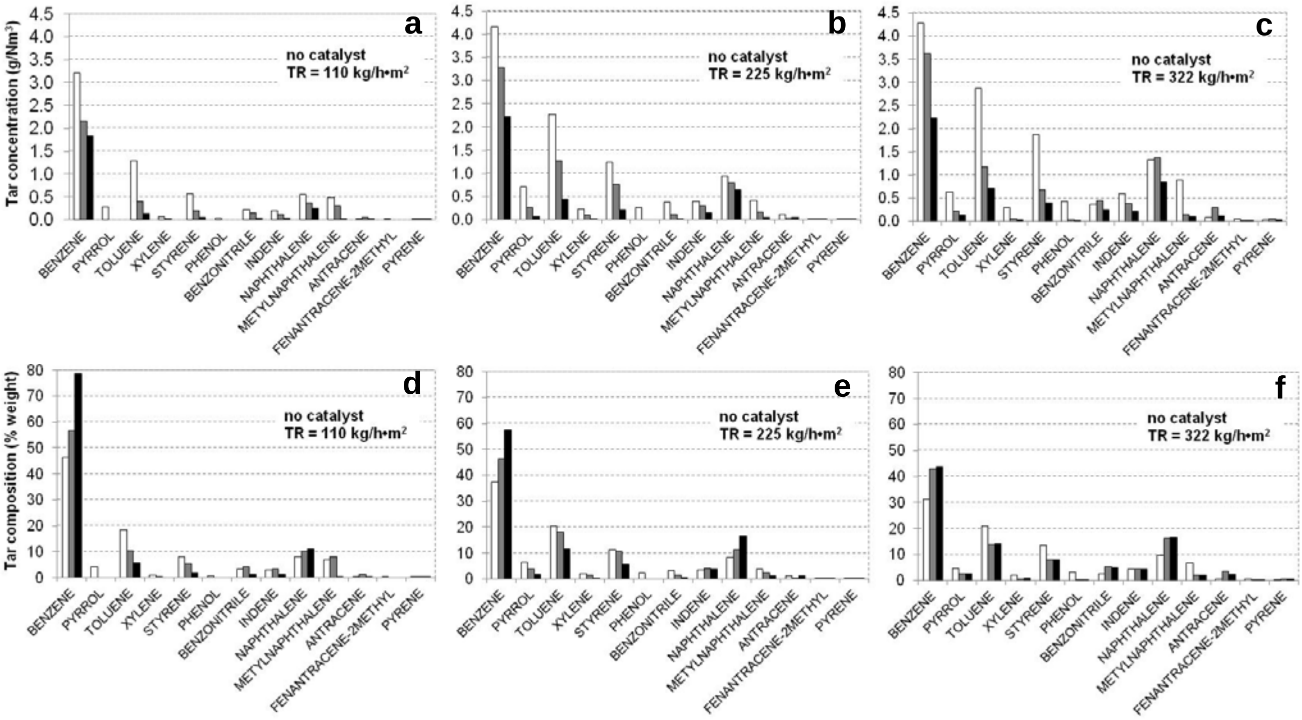
Regarding tar production, there was an increase in tar concentration when *srt* decreased (higher TR) under all the temperatures tested (Fig. 3b and Fig. 3d). Low *srt* reduce the effect of the cracking and reforming reactions on the tar removal. As it can be seen in Fig 2, even under the highest TR, the tar removal efficiency of the dolomite kept almost constant and always above 35%. In fact, at 850ºC, no relevant differences in dolomite performance were found in the range of TR studied. As far as CGE and X_C were concerned, both parameters increased with the *srt* as a result of slight increases of the LHV and the Y_{gas} (Table 2).

3.4 Effect of gasification conditions on tar composition

Even though the GC/MS detected a lot of peaks, most of them were considered negligible. From these peaks, 13 compounds (summing up more than 95% of the total tar weight) were selected as major components of the tar in order to simplify the analysis presented (Fig. 4).

Benzene, toluene, styrene and naphthalene were the most important components of the tar produced. As shown in Fig. 4a,b,c, the yield of all the components evaluated decreased when the temperature increased over the range of TR studied. This can be explained by the effect of the thermal cracking and the reforming reactions (Phuphuakrat, 2010). Regarding the effect of the temperature in the relative composition of the tar, different trends were found depending on the component observed Fig. 4d,e,f. Even though the relative production of most of tar components decreased, the benzene and naphthalene behaved in a different way and their relative yield increased by increasing the temperature. These findings agree with those reported by Kinoshita (1994). According to these authors, lower temperatures favour the formation of aromatic tar species with diversified substituent groups (such as phenol, xylene and toluene) while higher temperatures favour the formation of more stable aromatic tar species without substituent groups such as benzene and naphthalene. For example, with rising temperature, phenols are decarbonylated into radicals of the cyclopentadiene type, supposed to be intermediates in the formation of indene and naphthalene (Brage, 1996).

FIGURE 4 Effect of the TR and temperature on the tar yield and composition (□ 750ºC; ■ 800ºC; ■ 850ºC).



The yield of all the components increased with the TR because higher TR are related to lower *srt* and, therefore, the time available for the tar removal reactions is lower. As for changes in tar composition, unlike the rest of components, the relative production of benzene decreased as TR increased.

Regarding the effect of the dolomite in the tar yield and tar composition, the use of the catalyst decreased the yield of all the components studied. Although not shown, no relevant differences were found in the relative compositions of the tar in the presence of dolomite and the bar diagrams were similar to those presented in Fig. 4d,e,f.

4 CONCLUSIONS

This work deals with the effect of the temperature, the throughput (TR) and the use of dolomite as primary catalyst on the sewage sludge gasification products.

- The production of combustible gases (H_2, CO and CH_4) increased by increasing the temperature, making the produced gases more suitable for thermal applications. Over the range of temperatures studied (750-850ºC), reductions of tar yield of up to 67% were found. The changes in gas composition and the increase in gas production detected at higher temperatures increased both the carbon conversion and the cold gas efficiency.
- The use of dolomite as primary catalyst resulted in an additional increase of H_2 and CO content in the gas produced. However, the main advantage of the use of this catalyst is the decrease in tar yield. Under the gasification conditions studied, the tar removal efficiency ranged between 35% and 51%. Even under the highest TR, the tar removal efficiency of the dolomite kept almost constant, especially at 850ºC.

- Lower TR (higher gas residence time) resulted in an increase in H_2, CO and CH_4 content and a decrease in CO_2 production as a result of the closer approach to equilibrium values. On the other hand, higher TR increased tar production because of the reduction of the effect of the cracking and reforming reactions on the tar removal.
- Benzene, toluene, styrene and naphthalene were the most important components of the tar produced. Higher temperatures decreased tar production but increased the relative yield of benzene and naphthalene. The use of dolomite reduced the tar yield but did not produce relevant changes in its relative composition.

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(39) USE OF HEADSPACE AND SUBSTRATE COMPOSITION TO STEER FERMENTATION PROCESSES

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EXECUTIVE SUMMARY

Currently, industrial chemicals such as acids and solvents are produced via fossil fuel resources. However, because of increasing environmental concerns, the new trend is towards bio-based production of these chemicals. Fermentation, which is an intermediate step in the anaerobic digestion process, can generate acids and alcohols. In other words, fermentation processes can be used for the production of bulk chemicals from organic biomass sources. Among the variety of biomass sources, organic wastes are the most interesting ones because they are abundantly available, cheap and they do not compete with food (van Wyk, 2001). Organic waste materials consist of carbohydrates, proteins and lipids. Because of this complexity, only mixed culture fermentations can provide the reduction of mixed sources for desired products. The benefit of using a mixed culture is the elimination of the costly sterilization step in the fermentation process. The mixed culture environment, however, creates a final fermentation broth in which the produced bulk chemicals are diluted and occur as a mixture. As a result, to recover these chemicals more energy is needed in the further downstream processing steps. It is, therefore, desired to steer the fermentation process towards a single compound in sufficiently high concentrations in the final fermentation liquor.

This study investigated the thermodynamics of the reactions and assessed how selected environmental parameters influence the feasibility of the reactions. The Gibbs' free energy values from the model substrates towards volatile fatty acids and alcohols were calculated. Selected model substrates were glucose, glutamate, glycine and proline as they represent carbohydrate and protein substrates in their simplest forms. The change in product formation from model compounds under a range of proton concentrations and hydrogen partial pressures was calculated. The first calculations showed that nearly all reactions are under the thermodynamically required limit of -20 kJ/mol, independent of the conditions chosen. There was a clear and variable impact of the studied operational parameters on the Gibb's free energy levels. However, the effects were often similar for different reactions.

After the thermodynamic study, mixed culture fermentations were run with real waste streams. A batch reactor headspace was modified by adding 2 bar of hydrogen and was run for a 30 d incubation period. A control reactor was flushed with N_2 at the beginning of the experiment. To represent the selected model substrates in the thermodynamic study, organic waste streams different in carbohydrate and protein composition were collected. The pH of the batch reactors with carbohydrate rich waste was adjusted to 5 at the beginning of experiments and was not controlled. In the test with protein rich waste, pH was controlled at 5 to avoid pH increase by ammonia release from proteins. Reactors were kept at 30°C.

The batch experiments showed that acetate and butyrate were the main fermentation products under hydrogen modified headspace. Fractions and concentrations of acetate and butyrate varied with time. With the carbohydrate rich waste stream, the butyrate concentration was slightly higher than the acetate concentration over a 1 month incubation period. However within 1 week of incubation, butyrate concentration was much higher than acetate and its fraction reached 51% while it was only 17% in the control. In the control reactor, acetate and propionate were the major products. When the protein rich waste was used as substrate, acetate was the only major compound under all tested conditions.

Thermodynamic calculations showed that all fermentation products can in principle be formed under hydrogen headspace pressures up to 5 bar. However, batch experiments with 2 bar of hydrogen in the headspace showed the production of only two main products, namely acetate and butyrate. Other products like alcohols or higher carbon carboxylates were not found. This indicates that other than thermodynamic parameters such as kinetics are playing a major role in controling the fermentation product spectrum.

1 INTRODUCTION

1.1 Background

In the desire to reduce society's dependence on petroleum based chemicals, recent progress in the industrial area has focused on white biotechnology or more specifically anaerobic fermentation technology. By using organic waste streams as feedstock, bulk chemical production has become an attractive concept and further progress is expected to increase the yields of the process.

Organic waste fermentation has potential to generate many bulk chemicals. Evidently, not all bio-based materials have the same market potential. For example, lactic acid, C2 and C4 alcohols are on the list of the top chemicals produced worldwide (Danner and Braun, 1999; Rogers et al, 2006). Secondly, final concentrations of the chemicals are in diluted form due to the high water content of the organic waste stream which requires costly recovery and further concentration in downstream processes. As most of the target compounds of the fermentation process are Volatile Fatty Acids (VFAs) and alcohols and both groups will require different types of recovery techniques, optimization of the fermentation process towards a single product at maximal concentrations is needed to reduce high energy requirements for the downstream process. Only then, the production cost of target compounds can be minimized and chemicals produced via biological ways can compete with oil-based ones in the market.

In the first step, *hydrolysis*, of anaerobic fermentation of organic waste, particulate materials are converted to soluble organic compounds that can then be hydrolyzed further to simple monomers such as sugars, amino acids, long chain fatty acids and glycerol. The second step is called *acidogenesis* in which the products of the hydrolysis step are degraded mainly to VFAs such as acetic, propionic and butyric acid. Other fermentation products include alcohols such as ethanol and butanol, lactate and gaseous products such as carbon dioxide and hydrogen. In the final step, *acetogenesis*, the monomeric compounds from the first step and the VFAs are converted to acetate, carbon dioxide and hydrogen. After fermentation, *methanogenesis* can occur, in which methane gas is formed by the reduction of carbon dioxide and hydrogen and/or by acetate decarboxylation (Metcalf and Eddy, 2003). For maximal recovery of soluble intermediates, acid or alcohol consuming reactions during the methanogenesis step will have to be prevented.

Because anaerobic fermentation is carried out under a mixed culture environment, thermodynamics, kinetics, regulation, bacterial community structure, or a combination cause the bacteria to invest carbon source into a mixture of fermentation products rather than a single chemical. Among them, perhaps the most fundamental is thermodynamics (Lee et al., 2008). For fermentation reactions to occur, thermodynamics have to be favourable, i.e. reactions have to be exergonic or their Gibb's free energy should be negative. Reactions, which are exergonic or spontaneous, tend to reach a thermodynamic equilibrium state. The equilibrium state conditions can be shifted by manipulating the amounts of molecules involved in the fermentation reactions. Gas molecules such as hydrogen and carbon dioxide are produced during fermentation and a certain partial pressures of these two gases can shift the thermodynamic equilibrium from one reaction to another. Therefore, fermentation processes can probably be directed towards a desired target product by controlling the levels of these gases in headspace.

1.2 Research objectives

The objectives of this study were to evaluate the boundaries of mixed culture fermentation processes from a thermodynamic point of view, to assess how selected environmental parameters influence the feasibility of the reactions towards selected fermentation products and to apply the most favourable conditions in a batch-wise fermentation process to drive the system towards one major product at sufficiently high concentrations. Accordingly, the electron equivalents of numerous fermentation reactions were calculated by using the principles of thermodynamics. The effect of selected environmental conditions on the Gibbs' free energy values of production reactions of liquid fermentation products from the model substrates were investigated. After the thermodynamic evaluation, these reactions were investigated under laboratory conditions. The effect of 2 bar of hydrogen pressure on fermentation product concentration and distribution was evaluated for 2 different substrate types

2 METHODOLOGY

2.1 Thermodynamic approach

The reaction Gibbs free energy values ($\Delta G'_r$) under the actual environmental conditions were calculated by Eq. (1).

$$\Delta G'_r = \Delta G^{\circ}_r + R.T.\ \Sigma_i\,(v_i \ln c_i)$$

where v_i is the stoichiometric coefficient and c_i is the concentration for each reactant and product. Standard Gibbs free energy values of formation of compounds, ΔG°_f , were taken from Amend and Shock (2001). $\Delta G'_r$ values from the model monomers glucose, glutamate, glycine and proline towards acetate, propionate, butyrate, valerate, caproate, lactate, ethanol, butanol, propanol, pentanol, hexanol and methanol were calculated from the ΔG°_f values. It was assumed that the reactions reached thermodynamic equilibrium. The substrate and product concentrations were set to 1M, hydrogen and carbon dioxide pressure at 1bar, pH 7 and temperature 25°C. The calculations were set up assuming that they take place under unlimited hydrogen supply and with the substrate as sole carbon source. Microbial diversity was neglected.

2.2 Batch experiments

2.2.1 Inocula and organic waste streams

Inoculum was anaerobic granular sludge collected from a potato processing company (Opure, Ede, The Netherlands). Granules were washed with potassium phosphate buffer at pH 5 for 3 times. After the 3rd washing, they were boiled for 15 min to inhibit methanogens. Organic waste streams were collected from potato and meat processing industry.

2.2.2 Batch reactors

The experiments were conducted in 5-L water jacketed reactors (Figure 1). Reactors were filled with 750 ml basal medium which was prepared in the same way as described by Phillips et al. (1993) without sulphate addition. pH of the medium was adjusted to 5 with potassium phosphate buffer. Inoculum was added to the reactors to reach a final wet weight of 1%. Substrate concentration was added to create 8 g COD/l at the start-up of experiment. After addition of inocula and waste material, pH was corrected to 5 again with 2 M HCl and NaOH solutions. In the experiment with the waste of a meat processing company, pH was controlled at 5 to avoid a pH raise by ammonia release from proteins. The headspace of the reactors was filled with hydrogen to a final 2 bar pressure. Pressure of the reactors was monitored on-line by a manometer. In case a pressure decrease was observed during the experiment, it was adjusted back to 2 bar. A control reactor was prepared under the same conditions without headspace manipulation. It was flushed with nitrogen at the beginning of the experiment. Throughout the test, the headspace of the control reactor was connected to a milligas counter under atmospheric pressure to observe the gas production. Reactors were continuously stirred at 100 rpm and kept at 30°C. The experiments lasted 30 days with weekly gas and liquid sampling. Sampling amount was calculated in a way that liquid to headspace ratio was not disturbed or no underpressure occurede.

2.3 Analysis

To determine the composition of the gas produced, biogas in the headspace of bioreactors was sampled using a gas-tight syringe and analyzed in two separate gas chromatographs (Fisons GC 8000) equipped with thermal conductivity detectors as described by Calli et al. (2009). Liquid samples taken from the completely stirred bioreactors were centrifuged for 5 min at 10000 rpm before being analyzed for NH_4-N, VFAs, lactate and alcohols. For analysis of VFAs, filtrated samples were acidified with 1:1 (v/v) H_2SO_4 solution and then extracted with diethyl ether. VFAs in the diethyl ether phase were analyzed in a GC (CE Intruments-Thermoquest) equipped with a flame ionization detector (FID) and a 15 m AT-1000 filled capillary column (0.53 mm x 1.2 µm). Injection and detector temperatures were both adjusted to 250°C. The oven temperature varied between 110 and 150°C. Helium was used as the carrier gas at a constant flow of 6 ml/min. Acetone, ethanol, propanol and butanol were analyzed in the headspace of the samples at 60°C with the same GC fitted with an AT-WAX capillary column (60m x 0.32mm x 1.00µm). A variable temperature program from 40 to 100°C was used in the oven. The injection and detector temperatures were 145 and 200°C, respectively. The carrier gas was helium and its flow rate was 1.6 ml/min. Lactate was analyzed with a GC (Interscience, CE Instruments-Trace GC) equipped with FID detector, split/splitless injector and AT-1 capillary column (30m x 0.53mm x 5µm column). Samples were treated with $Ce(SO_4)_2$ and warmed up at 60°C for 10 min to convert the lactic acid into acetaldehyde which was then analyzed in the headspace.

Soluble chemical oxygen demand (CODs) was measured with Hach Lange cuvettes after centrifuged samples were filtered through 0.45 µm filters. Total chemical oxygen demand (CODt) was measured only at the beginning and at the end of the test with Hach Lange cuvettes.

Glucose content of the organic waste streams was analyzed with the Phenol-sulfuric method (Dubois et al., 1956), protein content with the Total Kjeldahl Nitrogen (TKN) method, and lipid content with the Soxhlet extraction method (Pomeranz and Meloan, 1994). Total and volatile solid contents were analyzed according to the Standard Methods (APHA, 1995).

3 RESULTS AND DISCUSSION

In this section, first the outcome of the thermodynamic study is evaluated. Later on, results of the experimental work are presented and discussed. The results of the experimental study are divided into two sections: (1) the effect of substrate and (2) the effect of hydrogen on product type and concentration.

4 THERMODYNAMIC STUDY ON FERMENTATION OF WASTE MATERIALS BY MIXED CULTURES

The calculated $\Delta G'_r$ values from the selected monomers glucose, glutamate, glycine and proline towards VFAs and alcohols were calculated. To observe the change in energy quantity of the reactions, the pH was varied in the range of 4.5 to 7 and hydrogen partial pressures were altered from 0.5 to 5 bars.

The calculations showed that nearly all reactions are under the thermodynamically required limit of -20 kJ/mol, independent of the conditions chosen. Figure 2 shows examples of the calculated $\Delta G'_r$ values of reactions from glucose and glutamate to acetate, butyrate, and propionate at 0.5 and 5 bar hydrogen partial pressure as a function of pH. There was a clear and variable impact of the studied operational parameters on the $\Delta G'_r$ levels.

It was observed that pH has more effect than hydrogen partial pressure. In general, $\Delta G'_r$ values of all carboxylates from glucose decreased with increasing pH (Figure 2). Therefore thermodynamically, the effect of pH is not specific to a single product when glucose is used as a substrate. The effect of pH was however different with different products from amino acids. For example, acetate production from glutamate is more favourable with increasing pH, while the opposite was true for butyrate production from glutamate (Figure 2). This finding shows that for protein rich substrates, according to the amino acid source, product type can be affected with pH control. However among the studied amino acid types, it was observed that the pH range for a target product differs for each amino acid. This makes it difficult to draw a pH boundary for single product formation in a mixture of substrates with various amino acids and glucose.

Thermodynamic calculations showed that the effect of hydrogen partial pressure on fermentation reactions varied depending on the substrate and product type (Figure 2). For instance, acetate production from glucose (Eq. 1) and proline (Eq. 2) is accompanied with hydrogen production whereas glutamate (Eq. 3) and glycine (Eq. 4) reactions consume hydrogen. From this, it can be concluded that keeping the hydrogen partial pressure as low as possible favours acetate production from glucose or proline but not from glutamate or glycine. Therefore, controlling the headspace composition for substrates having a mixture of carbohydrate and protein type polymers or even variable proteins sources cannot direct the fermentation towards a single product. From variable reactions investigated, it was observed that this conclusion is true for all carboxylates formed from monomers of carbohydrates and proteins.

$C_6H_{12}O_6 + 0.5H_2O \rightarrow 2.8\ Acetate^- + 0.5CO_2 + 0.9H_2 + 2.8H^+$ $\Delta G'_r = -289.7$ Eq. 1

$C_5H_9NO_2^- + 2.5H_2O + 0.5CO_2 \rightarrow 2.7\ Acetate^- + NH_4^+ + 0.1H_2 + 1.7H^+$ $\Delta G'_r = -41.8$ Eq. 2

$C_5H_8NO_4^- + 1.4H_2O + 0.2H_2 \rightarrow 2.3\ Acetate^- + NH_4^+ + 0.4CO_2 + 0.3H^+$ $\Delta G'_r = -68.5$ Eq. 3

$C_2H_5NO_2^- + 0.4H_2O + 0.2H_2 + 0.2H^+ \rightarrow 0.8\ Acetate^- + NH_4^+ + 0.4CO_2$ $\Delta G'_r = -50.2$ Eq. 4

Calculations showed that from glucose and the other selected amino acids, except proline, all alcohol products were thermodynamically feasible. Alcohol production reactions from glucose were thermodynamically independent of pH values. However, with selected amino acids, different pH conditions favour the different reactions. It was observed that $\Delta G'_r$ values of alcohols became favourable with elevated hydrogen partial pressure in the headspace. The effect of hydrogen on $\Delta G'_r$ values of the carboxylate reactions were again limited.

5 EFFECT OF POLYMER TYPE IN ORGANIC WASTE ON FERMENTATION PRODUCT DISTRIBUTION

The effect of carbohydrate and protein type of polymers on fermentation product distribution and concentration was investigated in batch experiments. The characterization results of the collected waste streams are presented in Table 1. Conform expectations, the waste stream collected from a potato processing industry is high in carbohydrate, from a meat processing industry in proteins. As they represent 2 main organic polymers, they were considered suitable to use in the batch experiments.

From thermodynamic calculations, the $\Delta G'_r$ levels for carboxylate production were lower than for alcohols with the carbohydrate glucose. Confirm the expectations, reduction of the carbohydrate type of waste stream was more towards carboxylates than alcohols.

The total carboxylate production was 4.9 g COD/l with this waste stream while alcohol production was only 1.1 g COD/l. Thermodynamically, the most favourable product from reduction of glucose was butyrate since the $\Delta G'_r$ value was the lowest (Eq. 5). The other favourable products were valerate and propionate (Eq. 6 and 7). This did not appear from our batch test results, where the main products were found to be acetate and propionate (Figure 3). Acetate reached 2.2 g COD/l with a fraction of 43% and propionate 1.7 g COD/l with a fraction of 33%. Butyrate concentration remained as low as 0.7 g COD/l. Instead of butyrate, acetate thus became the main product though its $\Delta G'_r$ production value was the highest among the carboxylates. Valerate fraction even remained lower than 5% of the total carboxylate production.

$C_6H_{12}O_6 \rightarrow 1.2\text{Butyrate}^- + 1.3CO_2 + 0.3H_2 + H_2O + 1.2H^+$ $\Delta G'_r = -307.2$ Eq. 5
$C_6H_{12}O_6 \rightarrow 0.9\text{Valerate}^- + 1.5CO_2 + 0.3H_2 + 1.2H_2O + 0.9H^+$ $\Delta G'_r = -306.6$ Eq. 6
$C_6H_{12}O_6 \rightarrow 1.7\text{Propionate}^- + CO_2 + 0.4H_2 + 0.6H_2O + 1.7H^+$ $\Delta G'_r = -306.2$ Eq. 7

Thermodynamic calculations showed that the most favourable reaction among the selected amino acids is the reduction to propionate (Eq 8, 9 and 10). However with the protein rich waste stream, propionate remained below 10% in the control reactor. Acetate was the main product with a concentration of 1.3 g COD/l at a fraction of 67% (Figure 3). In fact, the acetate $\Delta G'_r$ value from all selected amino acids was the lowest among all carboxylates.

$C_5H_9NO_2^- + 1.4H_2O + 0.5H_2 \rightarrow 1.6\ \text{Propionate}^- + NH_4^+ + 0.1CO_2 + 0.6H^+$ $\Delta G'_r = -93.4$ Eq. 8
$C_5H_8NO_4^- + 0.5H_2O + 0.6H_2 + 0.6H^+ \rightarrow 1.4\ \text{Propionate}^- + NH_4^+ + 0.9CO_2$ $\Delta G'_r = -88.2$ Eq. 9
$C_2H_5NO_2^- + 0.1H_2O + 0.3H_2 + 0.5H^+ \rightarrow 0.5\ \text{Propionate}^- + NH_4^+ + 0.6CO_2$ $\Delta G'_r = -50.2$ Eq. 10

The pH of the carbohydrate rich waste stream dropped to 4.2 in the control reactor during the 1st week incubation period. At this pH, the most favourable products were higher carboxylates like valerate and caproate and the least favourable one was acetate. This was not observed in the control reactor. In the protein rich waste experiment, pH was controlled at 5.0, and in this pH region the most favourable products were valerate, caproate and butyrate.
When $\Delta G'_r$ values are below the required limit of -20 kJ/mol, thermodynamics are thus not helpful to predict which fermentation product will preferentially be formed.

6 EFFECT OF HYDROGEN ON CARBOXYLATE PRODUCTION IN FERMENTATION

From thermodynamic calculations, it was anticipated that by applying high levels of hydrogen in the headspace, fermentation reactions can be directed towards certain products. For example, ethanol production from glucose consumes hydrogen (Eq. 11) whereas carboxylate production from glucose produces hydrogen (Eq. 1, Eq. 5, Eq. 6 and Eq. 7). Therefore, by adding hydrogen to the headspace, ethanol production from glucose might become thermodynamically favourable while carboxylate production might become limited.

$C_6H_{12}O_6 + 0.2H_2 \rightarrow 2\ \text{EtOH} + 1.9CO_2 + 0.1H_2O$ $\Delta G'_r = -238.8$ Eq. 11

Accordingly, 2 bar of hydrogen was added to the reactors with carbohydrate and protein rich waste streams to investigate the product distribution.
In the 2 bar hydrogen headspace reactors, carboxylate concentrations were higher than alcohol concentrations with carbohydrate rich waste stream. The total carboxylate concentration was 7.2 g COD/l. Alcohol concentration remained below 0.6 g COD/l. This finding showed that carboxylate production still can continue under 2 bar of hydrogen partial pressure. Although $\Delta G'_r$ values for carboxylate production reactions tended to decrease with increasing hydrogen partial pressure, production can be even higher under hydrogen headspace conditions. Alcohol production cannot be favoured with increasing hydrogen in headspace.
Figure 3 shows the concentrations of carboxylates produced in the carbohydrate and protein rich experiments. Acetate and butyrate were the main 2 products in the hydrogen manipulated headspace condition with the carbohydrate rich waste stream whereas in the reactor without headspace manipulation propionate appeared together with acetate. Propionate fraction was 33% in the control reactor whereas it was 3% in the hydrogen reactor. In fact, with carbohydrate type of substrates propionate and butyrate were more favourable compounds under elevated hydrogen partial pressures than acetate (Figure 2). However, on the carbohydrate rich waste stream, acetate fraction was 33% and butyrate 41% and their concentrations were stable after the 2nd week of incubation (Figure 2).

After the 2nd week, 0.5 bar hydrogen consumption was observed in the headspace until the end of experiment. While hydrogen was consumed, caproate production started and its fraction reached 11% albeit its concentration was lower than 1 g COD/l. This reaction was actually thermodynamically the most favourable one, only at a pH around 7 (data not shown), while in the experiments, the pH of the reactor was near 4.0.

Thermodynamically, almost all carboxylates and alcohols are produced from the selected amino acids with consumption of hydrogen and the carboxylate production $\Delta G'_r$ values were higher than for alcohols in all conditions (data not shown). Therefore, the expectation for the protein rich waste stream would be that the carboxylate production would be more favourable. Conform expectations, total carboxylates reached 2.2 g COD/l while alcohol concentration was negligible. Acetate and butyrate were the two main products under the hydrogen modified headspace with the protein rich waste material. Acetate concentration was 0.9 g COD/l and butyrate was 0.7 g COD/l (Figure 3). In fact, thermodynamic calculations showed that at pH 5.0, under 2 bar hydrogen partial pressure, propionate and valerate had the lowest $\Delta G'_r$ values with the selected amino acids, while it was never observed with the experiments with real waste streams.

7 CONCLUSIONS

This study aimed to achieve the production of one compound in sufficiently high concentrations by mixed fermentation process. In thermodynamic calculations, selected environmental parameters were investigated to assess the feasibility of the fermentation reactions and the influence on the boundaries of these reactions. Based on the thermodynamic study, batch-wise fermentation experiments were conducted.

From thermodynamic calculations, it could be concluded that a change in pH and hydrogen headspace pressure affects the free energy levels, but is not sufficient to direct mixed culture fermentation on a mixed substrate towards a single product, which was also confirmed in our experiments. Therefore, other than thermodynamic parameters such as kinetics and selective extraction of products will be crucial to control the fermentation product spectrum.

Our results indicated that in mixed culture processes organisms can choose multiple routes to gain energy, all ending with different products. When these routes show energy levels well below the thermodynamic limit of -20 kJ/mol, other parameters will determine which pathways are somehow favoured. Our experimental work showed that adding 2 bar hydrogen to the headspace can affect the product distribution and concentration but not in the way that was thermodynamically expected. The advantage of supplying 2 bar hydrogen to the headspace was that the total carboxylate concentration could be increased almost twice compared to control reactor.

8 ACKNOWLEDGEMENTS

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Figures

FIGURE 1 **Schematic view of bio-reactor (5 L) with modified headspace. (1) magnetic stirrer, (2) sampling port, (3) gas inlet, (4) pH electrode, (5) manometer, (6) water bath at 30°C, (7) outflow port.**

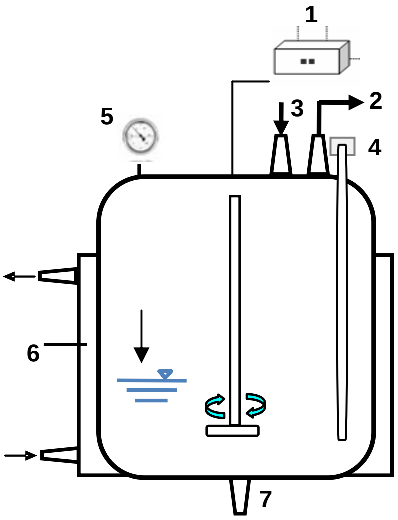
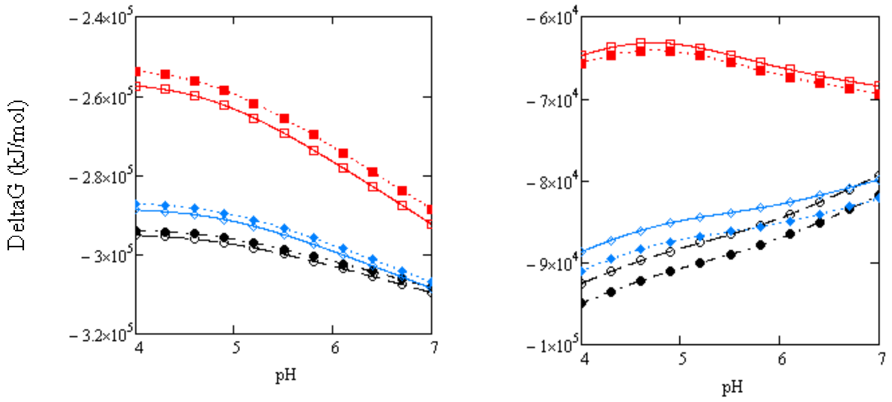
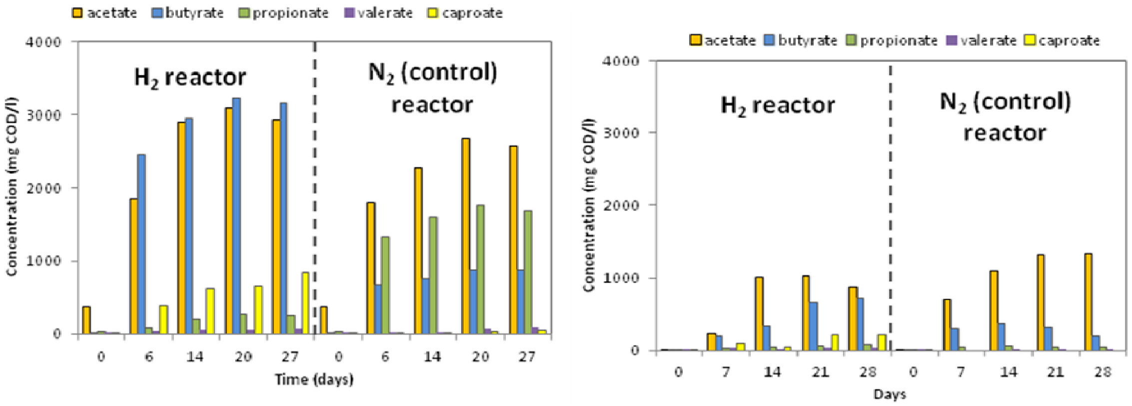


FIGURE 2 **Change in Gibbs free energy of reactions from (a) glucose and (b) glutamate reduction to acetate at 0.5 bar (⊟⊟⊟) bar, at 5 bar (■■■), to propionate at 0.5 bar (◇◇◇), at 5 bar (◆◆◆) at 5 bar, to butyrate at 0.5 bar (○○○) bar, at 5 bar (●●●). Scales are different.**



(a) (b)

FIGURE 3 **Carboxylate concentrations in reactors with 2 bars hydrogen and N_2 (control) over 4 weeks incubation time with (a) carbohydrate rich waste stream and (b) protein rich waste stream.**



(a) (b)

TABLE 1 **Characterization of organic waste streams. Concentrations are mean values. Results are presented on wet weight basis.**

Organic waste streams	Total Solids (mg/kg)	Volatile Solids (mg/kg)	Carbohydrate (g/kg)	Protein (g/kg)	Lipid (g/kg)	CODt (g/kg)
Potato	37	31	29	6	0,2	26
Meat	70	65	1.8	51	13	112

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(68) OPTIMIZATION OF BATCH BIO-H_2 AND METHANE PRODUCTION FROM RAW CHEESE WHEY

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EXECUTIVE SUMMARY

Due to the renewed interest of finding new sources of green energy, biologically produced hydrogen (bio-H_2) from biomass and mixed cultures is a promising alternative. Under anaerobic conditions, a wide range of food wastes can be used as substrates to generate bio-H_2.

Raw Cheese Whey (RCW) represents an important waste for dairy industries due its high organic content and production rate (7-9 litres kg^{-1} of final product). Up to day, the options for whey valorisation as a food additive or for animal feed are not always feasible or economically viable. Thus, biogas production emerges as an alternative for its energetic valorisation. Nevertheless, the utilization of cheese whey as a unique substrate results in acidification problems and consequently, low methane production rates (90-180 NL CH_4 kg^{-1} volatile solid-VS), even with pH adjustment. The combination of two-step anaerobic digestion process with separated bio-H_2 and methane production represents one of the most promising alternatives to overcome this problem.

This study primarily focused on optimising the main operational parameters (initial pHs, and substrate:inoculum ratios) for bio-H_2 production (1st phase) by digestion of RCW, utilizing a digested Cow Manure Inoculum (CMI) in batch conditions. In this phase, all tests were performed without pH controlling or adjusting the pHs initially to different values (5.5, 6.0, 6.5 and 7.0). Alkalinity addition effect and different RCW:CMI ratios (50:50, 60:40, 65:35, 70:30, 80:20) were also evaluated.

Secondly, the study tested the potential of the obtained digestates in the first phase for methane production in a 2nd phase. In this phase, the digestates were inoculated with fresh and pre-filtered digested CMI in 50:50 ratio in Volatile Solids basis to assure the methanogenic bacteria population, without adjusting the initial pH or adding alkalinity.

All the tests were performed in stirred 500 mL bottles, by triplicate and under mesophilic conditions (37±3 °C) with continuous biogas production registration. Both bio-H_2 and methane percentages in the biogas were analysed by Infra-Red equipment.

The highest Specific Bio-H_2 (SHP) and Methane (SMP) Production rates were obtained for an initial pH and RCW:CMI ratio of 6.5 and 65:35 respectively. Those values were 85.8 NL bio-H_2 kg SV^{-1} and 166.9±2.3 NL CH_4 kg VS^{-1}.

Cumulative bio-H_2 data were also fitted to the Gompertz equation with a r^2~0.99. The lag phase before bio-H_2 exponential production for the highest bio-H_2 production (65:35, pH =6.5) was 3.8±0.1 hours.

These findings could be on significance for further two-stage full-scale continuous systems design, in order to improve the viability of conventional anaerobic digestion plants and to offer a sustainable valorisation option for cheese whey effluents.

1 INTRODUCTION

The environmental concern over the greenhouse effect, caused by the enormous combustion of fossil fuels is the main driver towards the bio-H_2 based economy. Bio-H_2 is a clean alternative energy vector as water and is the only by-product after its combustion. The traditional hydrogen manufacturing methods require high energy consumes. Within this framework, biological processes are emerging as alternative sources of bio-H_2. Photo and dark fermentation, both in single or combined processes, are considered the most promising technologies (Venetsaneas et al., 2009). However, several obstacles must be overcome to produce bio-H_2 effectively from biological processes. Under normal anaerobic conditions the majority of bio-H_2 produced is consumed by methanogens. Therefore, these bacteria must be inhibited by pre-treating the seed. Another major issue is the pH control. pH was found to have performed effect on both bio-H_2

production potential from RCW (Ferchichi et al., 2005).These issues along with substrate and inoculum composition and concentration are important parameters that need to be understood and controlled.

Different studies have tested several types of substrates for biological production of bio-H_2. The major criteria for substrate selection are availability, cost, carbohydrate content and biodegradability. Wastewaters from agricultural and food processing industries, which are generally rich in carbohydrates can provide the essential nutrients required for bio-H_2 production and reduce treatment and disposal costs currently needed for these particular waste stream. Concerning to microbial populations there are several reports that have used pure and mixed cultures (Karapinar et al., 2006; Reith et al., 2003).

Cheese manufacturing industry generates large amounts of whey characterized by high organic (up to 70 kg m^{-3}) and low alkalinity content (2.5 kg m^{-3}) which makes difficult to treat it by anaerobic digestion due to acidification problems. Although RCW represents a key substrate in terms of availability and high biodegradability, only a few reports in bio-H_2 production from these substrates exist (Venetsaneas et al., 2009, Reith et al., 2003). Ferchichi et al. (2005) used diluted cheese whey (ca. 41.1 g lactose L^{-1}) to study the influence of initial pH on bio-H_2 production in batch experiments with pure *Clostridium* culture. Another study with a pure culture reported the use of lactose (10 g L^{-1}) in a continuous regime at different pH and dilution rates (Collet, 2004). The use of mixed anaerobic cultures for continuous bio-H_2 production from cheese whey was first reported by Yang et al., 2007. There are several authors that have tested bio-H_2 gas yield for different cheese whey concentrations in batch mode (Azbar et al., 2009; Dávila-Vazquez et al., 2009).

Operating in a Single Phase Anaerobic Digestion (SPAD) process results in fragile balance between acidogenic and methanogenic bacteria. Both groups differ extensively in in terms of physiology, nutritional needs, growth kinetics, and sensitivity to environmental conditions (Thompsom, 2008). Antonopoulou et al. (2008a) suggested that separation of the acidogenesis and methanogenesis steps in a two-stage anaerobic process could be an alternative solution for treating cheese whey.

The two stage processes have been traditionally used for methane production to increase biogas yields (Ghaly et al., 1996), specially when treating substrates with high rates of acidification. In this study, a 155 L digester with acidic cheese whey, under mesophilic conditions and at Hydraulic Retention Time (HRT) of 20 days produced 8 L CH_4 d^{-1} without pH control, while the productivity reached 87.5 L CH_4 d^{-1} with pH control.

The low pH and rapid acidification of the RCW represents an advantage as the optimum pH for bio-H_2 production occurs at pH between 5.5 and 7.0 depending on the type of substrate (Ferchichi et al., 2005). This fact in conjunction with the current interest in obtaining a high energetic fuel as bio-H_2 (120 MJ kg^{-1}) in a first stage, and methane in a second one, make these processes very attractive from an energetic point of view.

This study primarily was focused on optimising the main operational parameters (initial pH, and RCW:CMI ratios) for bio-H_2 production (1st phase) by digestion of RCW in batch conditions. Secondly, the study has assessed the potential of the obtained digestate for methane production (2nd phase). The overall energy yield and digestion properties of the Two-Phase Anaerobic Digestion (TPAD) processes comparing to SPAD processes were evaluated. Finally, a study of bio-H_2 production kinetics was also performed.

2 METHODOLOGY

Analytical methods

Determination of Total Chemical Oxygen Demand (TCOD), Total Solids (TS), Volatile Solids (VS), Total Alkalinity (TA), Total Kjeldahl Nitrogen (TKN) and ammonia nitrogen ($N\text{-}NH_3$) were carried out according to standard methods (APHA et al. 1992). Fats were determined by Blyer-Dyer method. The concentrations of VFAs (acetic, propanoic, butyric, iso-butyric, valeric, iso-valeric) were determined by GC-FID fitted with a INNOWAX 1909 1N-113 column (30 m x 0.32 mm x 0.25 μm film thickness). The operation temperatures for the injection port and flame injector detector were 260 °C and 280 °C respectively.

The bio-H_2 was quantified by modified Infra-Red equipment provided by H_2 Scan (Valencia, USA). The methane was also determined by Infra-Red equipment provided by Geothermical Instruments (Munich, Germany). Lactose was determined using an enzymatic reagent kit (lactose and D-Galactose, *Boehringer Mannheim Enzymatic BioAnalysis and Food Analysis*). The measurement of the pH was done using a Hanna (pH 211) pH meter and a Hanna (HI 1230) electrode.

Inoculum preparation

The seed sludge for the experiments was provided by a cow manure digestion plant located in Iraizotz (Navarre). The raw seed sludge was filtered through a screen (pore size: 3 mm) to remove fiber-like undigested materials before using. Before seeding, the filtered raw sludge was heated at 80 °C for 30 min to inhibit the activity of hydrogen trophic non-spore formers present in the anaerobic sludge. No micro-nutrients solutions were added to the medium.

Substrate preparation

The cheese whey was obtained from a local dairy factory located in Zamudio, Spain. The whey samples were collected every two months and stored at -5 °C up to 1 month.

TABLE 1 **Average characteristics of Raw Cheese Whey (RCW) and Cow Manure Inoculum (CMI) from different batches used in this study (n=2)**

Characteristic	Raw cheese whey (RCW)	Cow manure inoculum (CMI)
Ph	6.8±0.5	7.8±0.2
TS (%)	7.6±0.1	4.2±0.4
VS (%)	7.35±0.1	4.1±0.3
TCOD (g L^{-1})	79.9±1.20	43.1±10.4
TKN (g L^{-1})	1.75±0.05	2.4±0.14
N-NH_3 (g L^{-1})	0.1±0.05	2.01±0.03
Fats and oils (g L^{-1})	2.8±0.1	<0.4
TA (g $CaCO_3$ L^{-1})	2.0±0.2	17.9±3.3
Lactose (g L^{-1})	5.4±0.6	-

Experimental Setup and Operation

Batch experiments were conducted in 500 mL bottles with a working volume of 400 mL. All the experiments were carried out at mesophilic temperature (37 °C). Gas production was continuously monitored using an Automatic Methane Potential Test System (AMPTS) provided by Bioprocess Control AB (Lund, Sweden). All gas volumes are reported at 1 atm and 273 K.

In the first stage, bio-H_2 producing batch reactors were inoculated with undiluted RCW and heat pre-treated (80 ºC, 30 min) and filtered (pore size: 3 mm) CMI. pH trials were performed to determine the optimal pH within a range 5.5-7.0 and determine the quality and the quantity of biogas production. The pH was initially adjusted to 5.5, 6.0, 6.5 and 7.0 by adding HCl 5M. Bio-H_2 producing fermentations were also conducted without pH control and with sodium bicarbonate addition (8 g L^{-1}) in order to increase the alkalinity of the medium. Trials for different RCW:CMI ratios were performed at 60:40, 65:35, 70:30, 75:25, 80:20 in terms of w/w VS at a defined pH. All trials were realized in duplicate and were previously sparged with nitrogen gas for 5 minutes prior to operation. For the second stage, the effluents from the bio-H_2 trials (acidogenic phase) were collected and mixed with non-pre-treated and filtered (pore size: 3 mm) CMI (50:50, w/w VS) to assure the methanogenic bacteria population, without performing any pH control. As in the first stage, the reactors were also sparged with nitrogen gas for 5 minutes before running each trial.

3 RESULTS AND DISCUSSION

Each trial was operated over 2.5 days (60 hours) for the 1st phase and 14 days for the 2nd phase. Data from these trials were collected and analyzed to check if a TPAD system could be advantageous in comparison with a SPAD process.

Trial 1: Effect of different initial pHs over the bio-H_2 and methane production

The aim of this trial was to study the effects over bio-H_2 and the subsequent methane productions when adjusting the initial pHs at different values within a range between 5.5 and 7.0, or when adding alkalinity and without controlling the pH. The RCW:CMI ratio was fixed at 60:40 w/w SV based on preliminary trials.

1st Phase: Bio-H_2 production for different initial pHs

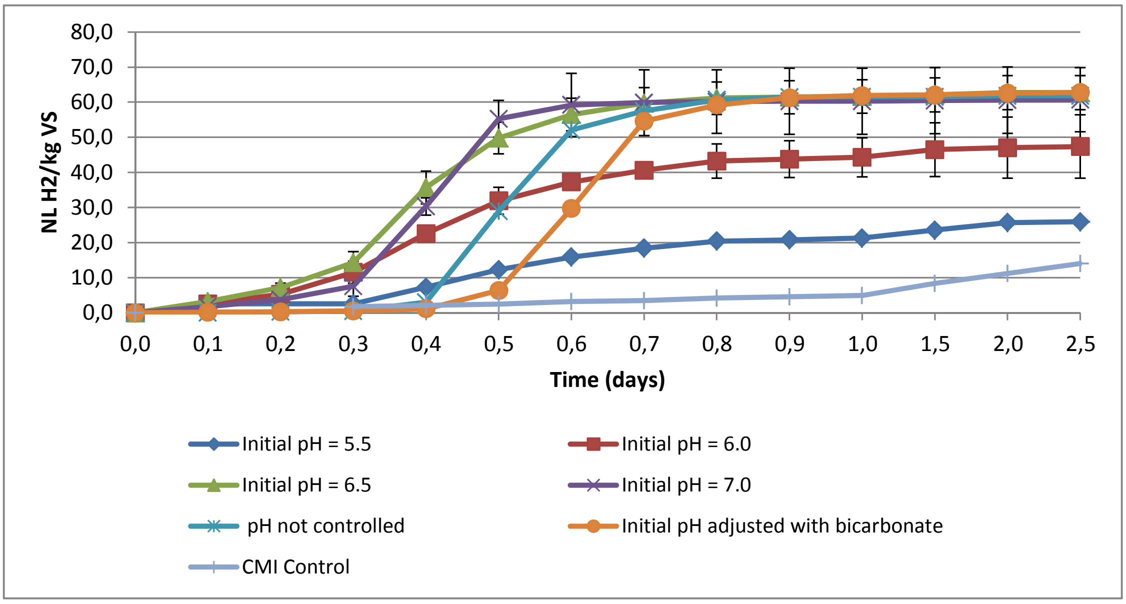

FIGURE 1 **Cumulative bio-H_2 productions for different initial pH adjustments or alkalinity addition**

2nd Phase: Methane production

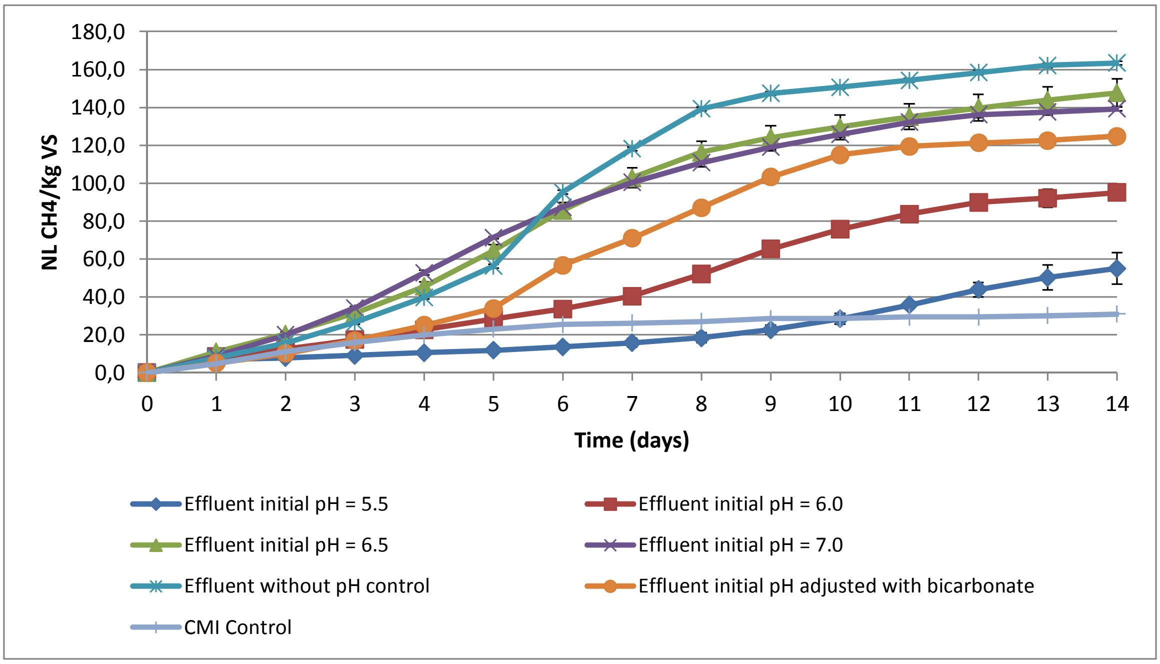

FIGURE 2 **Cumulative CH_4 production for different initial pH or alkalinity addition**

Table 2 shows the Specific Biogas Production SBP, % H_2 into the enriched bio-H_2 biogas, the SHP and the SMP obtained in the 2nd phase.

TABLE 2 **SBP, % H_2, SHP and SMP for different initial pHs. No methane or traces were produced during the regular fermentation time in the first stage (<0.5 % CH_4)**

		1st Phase			2nd Phase
Trial	**Conditions**	**SBP (NL biogas kg SV^{-1})**	**% H_2**	**SHP (NL H_2 kg SV^{-1})**	**SMP (NL CH_4 kg SV^{-1})**
TPAD-MP RCW:CMI 60:40	Initial pH = 5.5	68.9±1.7	37.6	25.9	55.1±8.3
	Initial pH = 6.0	112.8±9.0	42.0	47.4	95.0±4.1
	Initial pH = 6.5	166.7±13.0	37.6	62.7	155.5±11.0
	Initial pH = 7.0	154.1±9.1	39.4	60.7	139.3±1.2
	pH controlled with alkalinity addition (pH=8.0±0.1)	206.4±11.2	29.5	60.9	124.7±5.0
	pH not controlled (pH =7.98±0.01)	212.7±10.6	30.0	63.8	163.5±2.3
SPAD RCW:CMI	RCW:CMI 50:50	-	-	-	114±5.6

From the fig. 1, it can be concluded that the majority of bio-H_2 is produced within the first 18-20 hours. There are not substantial differences in SHP rates for initial pHs of 6.5, 7.0 and either by adding alkalinity to the medium or without controlling the pH. Regarding to lag phase, and as it was expected, the lower the pHs were, the shorter the lag phase were as seen in fig. 1. The alkalinity addition delayed the lag phase from 5.0 to 7.0 hours, while fixing the initial pH at 6.5, it shortened from 5.0 to 4.3 hours (Table 5). The lag phase reference value (5.0 h) was calculated from the data obtained without carrying out the pH control with acid or alkalinity addition. The results suggest that the duration of the lag phase was shorter when adding acid to fermentation medium. However, a shorter lag phase did not imply an increase over the bio-H_2 potential. This is in agreement with other studies that have shown that the addition of acid beyond to certain levels to fermentation medium results in the protonation of undissociated weak acids in the medium, which may pass freely through the cell´s membrane into cytoplasm (O'Sullivan et al., 1999). This new internal condition could result in DNA, structural and protein damages which could down the growth) of hydrogen producer bacteria. (Ferchichi et al., 2005).

The SHP rates ranged from 60.7 to 63.8 NL H_2 kg SV^{-1} (209.1 and 210.3 mmol H_2 L $substrate^{-1}$ respectively) for initial pHs above 6.5. There were not significant differences over SHP rates between performing an initial pH adjustment at 6.5 and 7.0, and either when adding alkalinity or controlling the pH. All values reported for these conditions are similar to those reported by Wang et al. (2009). In this study a pilot scale continuously operated unit was used for testing a rotating drum for H_2 production followed of a methane fermentation reactor (200 and 800 L of working volume respectively). The H_2 yields ranged among 49-65 L H_2 kg SV^{-1} for an organic loading rate of 22.7-37.8 kg SV m^{-3} d^{-1}. The process was studied under mesophilic anaerobic conditions using as inoculum indigenous mixed microbial culture contained in food waste.

Concerning to the effects of the effluents of the acidogenic phase over the methane production in the second phase, only when mixing effluents with initial pHs of 5.5 and 6.0, lower volumes of methane were produced in comparison with a SPAD process (Table 3). This fact could have led to high VFA_S concentrations, reducing the initial buffer capacity of the fresh inoculum. Consequently, the initial pHs could decrease below 7.5-8.0, which is considered the optimum pH range for methanogenic bacteria. In this study, the initial pHs of the mixtures for methane production were 7.1 to 7.3 when mixing the effluents with initial pH adjustments of 5.5 and 6.0 with fresh CMI respectively (50:50 VS). Therefore, these values are below the optimum pH range for methane production. For the rest of mixtures, the initial pH values were among 7.5-8.0.

Trial 2: Effects of different susbstrate:inoculum ratio over the bio-H_2 and methane production (TPAD-MP)

The aim of this trial was to study the effects of different RCW:CMI ratios over the bio-H_2 and the subsequent methane potential productions. The pH was fixed based on the optimum value of 6.5 obtained in the trial 1.

1st Phase: Bio-H_2 production

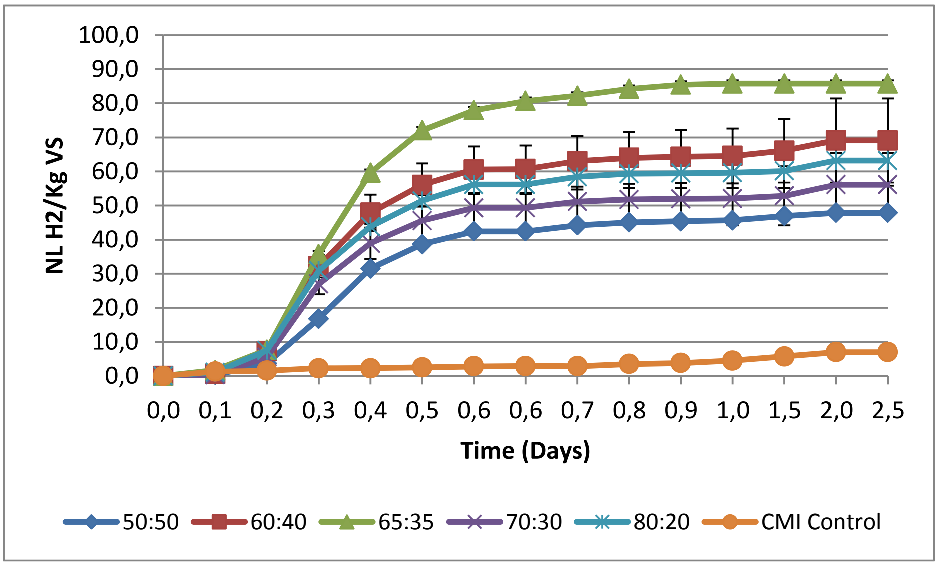

FIGURE 3 **Cumulative bio-H_2 production for different RCW:CMI ratios**

2nd Phase: Methane production

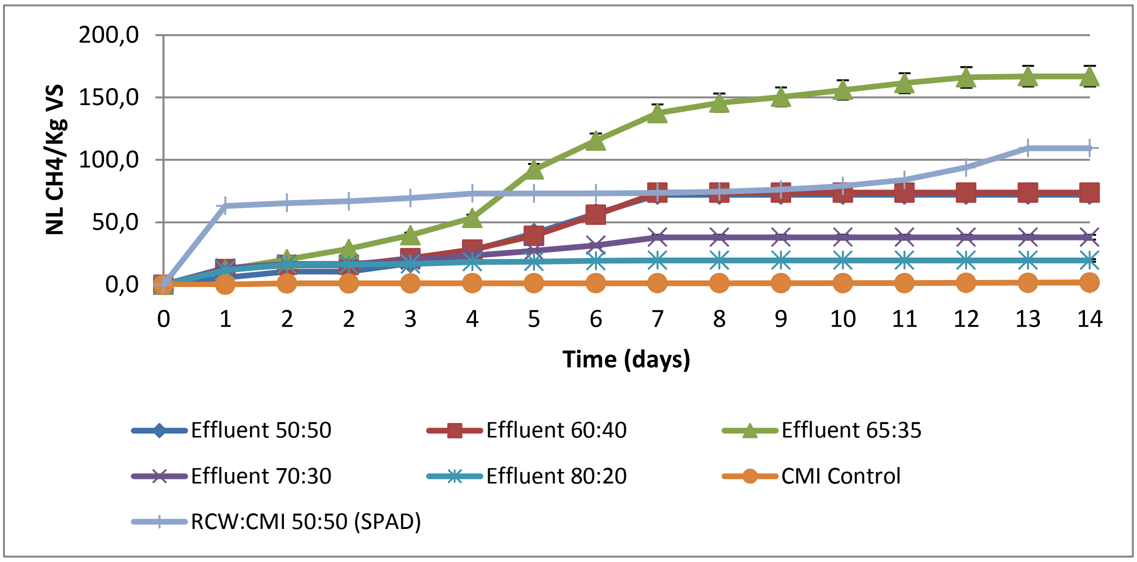

FIGURE 4 **Cumulative methane production for different RCW:CMI ratios**

Table 3 shows the SBP, % H_2 into the enriched bio-H_2 biogas, the SHP and the SMP obtained in the 2nd phase for different RCW:CMI ratios.

TABLE 3 **SBP, % H_2, SHP and SMP for different RCW:CMI ratios. No methane or traces were produced during the regular fermentation time in the first stage (<0.5 % CH_4)**

		1st Phase			2nd Phase
Trial	**Description**	**SBP (NL biogas kg SV^{-1})**	**% H_2**	**SHP (NL H_2 kg SV^{-1})**	**SMP (NL CH_4 kg SV^{-1})**
TPAD **pH = 6.5**	RCW:CMI 50:50	113.5±21.5	42.2	47.9	72.0±2.1
	RCW:CMI 60:40	147.9±6.6	46.7	69.1	73.7±11.6
	RCW:CMI 65:35	190.4±11.0	45.0	85.8	166.9±2.3
	RCW:CMI 70:30	127.5±19.5	44.0	56.1	37.9±3.1
	RCW:CMI 80:20	149.4±17.5	42.3	63.2	19.5±2.0
SPAD		-	-	-	114±5.6

The higher SHP (85.8 NL H_2 kg SV^{-1}) was obtained for a RCW:CMI ratio of 65:35. The values shown in table 3 indicate that higher concentrations of RCW does not mean higher SHPs, as it decreased for RCW:CMI ratios of 70:30 and 80:20. The lowest VFAs concentration was for a RCW:CMI ratio of 65:35 (5114 mg kg^{-1}), while the highest was for the RCW:CMI ratio of 50:50 (8903 mg kg^{-1}). Argun et al. (2009) reported a similar phenomenon using as substrate ground wheat. According to this study, further increases in the initial concentration of ground wheat lead to a lower hydrogen yields due to formation of inhibitory levels of VFAs (>3 g L^{-1}). Therefore, it is concluded that the concentration of RCW can only be increased up to a certain level. An excessive concentration of RCW can cause a build-up of VFAs in the system leading to a decline of pH in the reactor and the subsequent inhibition of hydrogen producer bacteria as stated by Fang et al., 2006.

Regarding to SMPs, only with the exception of RCW:CMI ratio of 65:35, lower volumes of methane were obtained comparing to a SPAD process. This could be a consequence of the higher SHP rates obtained in the 1st phase comparing to trial 1. This fact could have led to higher VFAs concentrations, which would reduce the pH below the optimum values for methane production in the 2nd phase as seen in trial 1.

Energy yields

In order to have a better insight about the advantages of using a TPAD against a SPAD process, the maximum total net energy was calculated (Table 4).

TABLE 4 **Energy production of all trials. The energy yield uses a density of 8.3E-05 g $cm^{-3}$and 122 kJ g^{-1} for bio-H_2 and 7.175E-04 g cm^{-3} and 30.7 KJ g^{-1} for methane (Thompson, 2008).**

		1st Phase	2nd Phase	
Trial	**Conditions**	**Energy produced (KJ kg SV^{-1})**	**Energy produced (KJ kg SV^{-1})**	**Total Energy produced (KJ kg SV^{-1})**
TPAD **RCW:CMI** **60:40**	pH = 5.5	282.3	1129.6	1411.9
	pH = 6.0	516.7	1947.5	2464.2
	pH = 6.5	683.4	3187.8	3871.2
	pH = 7.0	661.6	2855.7	3517.3
	pH controlled with alkalinity addition (pH=8.0±0.1)	663.8	2556.4	3220.2
	pH not controlled (pH =7.98±0.1)	808.8	3351.8	4160.5
TPAD **pH = 6.5**	RCW:CMI 50:50	522.1	1476.0	1998.1
	RCW:CMI 60:40	753.2	1510.9	2264.0
	RCW:CMI 65:35	935.2	3421.5	4356.7
	RCW:CMI 70:30	611.5	777.0	1388.4
	RCW:CMI 80:20	688.9	399.8	1088.6
SPAD **RCW:CMI**	RCW:CMI 50:50	-	2337.0	2337.0

Table 4 shows that in four cases (RCW:CMI 60:40, Initial pH = 5.5 / RCW:CMI 50:50, Initial pH = 6.5 / RCW:CMI 70:30, Initial pH = 6.5 / RCW:CMI 80:20, Initial pH = 6.5) a TPAD process did not result energetically advantageous comparing to a SPAD process. The highest SHP, SMP and subsequently, the highest energy yield was obtained for a RCW:CMI ratio of 65:35 with an initial pH adjustment of 6.5. However, there is not a significant difference in terms of energy yields comparing to not performing the pH control.

In terms of hydrogen and methane yields, for the optimum susbstrate:inoculum and pH conditions, the SHP and SMP were 85.8 NL bio-H_2 kg SV^{-1} (2.33 mmol H_2 g $TCOD^{-1}$) and 166.9 NL CH_4 kg SV^{-1} (4.95 mmol CH_4 g $TCOD^{-1}$, 0.225 NL CH_4 g $TCOD^{-1}$) with a percentage of 69 % of CH_4 within the biogas. The SHP is slightly higher to the value reported by Yang et al. 2007 (1.98 mmol H_2 g $TCOD^{-1}$) for the same conditions, using an anaerobic sludge from a wastewater treatment plant instead of cow manure as inoculum. The methane yield and CH_4 percentage is very similar to those values reported by Göblös et al., 2007 when treated untreated cheese whey in a TPAD process to produce only methane under mesophilic conditions. In this study, the methane percentage within the biogas and the yield were 70 % and 0.236 NL CH_4 g $TCOD^{-1}$ respectively.

Total Chemical Oxygen Demand (TCOD), Total Solids (TS) and Volatile Solids (VS)

TCOD and solids tests were performed for the influent and effluent of each trial. Both parameters are essential factors in determining the efficiency of waste treatment.

The SPAD had a total solids removal of 33.6 % and a volatile solids removal of 32.9 %. The TPAD trials reported removal percentages among 28.8 and 65.8 of TS and between 28.6 and 64.6 of VS for the first phase (acidogenic phase). The hydrogen production trials reported a higher variation in TS, VS and TCOD removal degrees for each of the samples analyzed. It means that there was a greater TCOD removal with less total solids removal. This indicates that the solids in the RCW that were removed were relatively high in TCOD. At lower cheese whey concentrations, lower TCOD was observed, while the total solids removal was higher. One explanation of this was the higher solids content within the CMI and higher TCOD within the RCW. By increasing the RCW concentration, the TCOD removal also increased, but the solids removal decreased due to less CMI. These results are in agreement with those reported by Thompson (2008). For the 2nd phase (methanogenic phase), TS and VS removal percentages ranged from 33.2 up to 46.0 and from 33.1 to 45.7 % respectively. TCOD removal percentage was 44 %. These values are similar to those typically found in the literature for this type of processes (Thompson, 2008; Venetsaneas et al., 2009; Ghaly et al., 1996). For the optimum pH and RCW:CMI ratio (pH =6.5, 65:35 w/w VS basis), TS, VS and TCOD removal percentages were 42.2, 42.0 and 26.6 % for the acidogenic phase. For the methanogenic phase (second phase), those values were 42.5, 42.2 and 46.9 % respectively.

Inhibitors of AD processes

Ammonium nitrogen is a well-known inhibitor of anaerobic digestion processes. In this study, it was measured at the influents and the effluents of all trials for both acidogenesis and methanogenesis phases. All the measurements (ranging from 0.6 to 1.3 g L^{-1}) remained below the concentration considered as moderately inhibitory for the process (> 3 g L^{-1}) (Ghaly et al., 1996).

Kinetics analysis of bio-H_2 production with cheese whey

To determine the effect of the initial pH on the bio-H_2 potential, H_{max} (NL bio-H_2 $kgVS^{-1}$), a modified Gompertz equation was used to fit the cumulative bio-H_2 production obtained from each batch experiment. This equation has been widely used to model the production data (Khanal et al., 2004):

$$H(t) = H_{\max} \exp\left\{ -\exp\left[\frac{R_{\max} e}{H_{\max}} (\lambda - t) + 1 \right] \right\} \qquad (1)$$

where H(t) (NL NL bio-H_2 $kgVS^{-1}$) is the total amount of bio-H_2 produced at culture time t (h); H_{max} (NL bio-H_2 $kgVS^{-1}$) is the maximal amount of biogas produced . R_{max} (NL bio-H_2 $kgVS^{-1}$ h^{-1}) is the maximum bio-H_2 production rate, (h) is the lag time before exponential bio-H_2 production and e constant is 2.71828. The cumulative production curves were fit using Microsoft Excel's Solver tool by minimizing the Sum of Square Error (SSE). Initial estimates for the parameters were selected based on visual inspection. (H_{max}, R_{max} and λ).

TABLE 5 **Adjusted H_{max}, R_{max} and lag phase (λ) parameters for bio-H_2 production**

Trial	Conditions	H_{max} (NL bio-H_2 kg VS^{-1})	R_{max} (NL bio-H_2 kgVS^{-1} h^{-1})	λ (h)	R^2
TPAD RCW:CMI 60:40	Initial pH = 5.5	24.6±0.6	1.7±0.0	5.2±0.3	0.993
	Initial pH = 6.0	46.5±3.4	4.1±0.3	4.0±0.6	0.998
	Initial pH = 6.5	63.8±6.7	6.3±1.8	4.3±0.5	0.913
	Initial pH = 7.0	60.7±3.7	12.9±1.0	7.0±0.3	0.997
	Alkalinity addition (pH=8.0±0.1)	62.5±0.4	12.7±0.2	7.1±0.0	0.998
	pH not controlled (pH =7.98±0.01)	68.2±1.2	15.1±0.7	5.0±0.1	0.999
TPAD Initial pH=6.5	RCW:CMI 50:50	46.4±20.0	5.9±3.9	2.7±1.6	0.957
	RCW:CMI 60:40	65.7±9.4	8.7±0.2	3.8±0.2	0.994
	RCW:CMI 65:35	85.3±10.5	12.4±1.0	3.8±0.1	0.998
	RCW:CMI 70:30	53.0±8.3	7.4±0.2	3.9±0.2	0.993
	RCW:CMI 80:20	60.6±5.5	7.9±0.6	3.7±0.1	0.995

For all trials Eq. (1) adequately described bio-H_2 production as shown by high values of regression coefficients (R^2).

Data fitting to the modified Gompertz equation confirms that the bio-H_2 potential (NL bio-H_2 kg VS^{-1}), the maximum bio-H_2 production rate and the duration of lag phase are all pH-dependent. When adjusting the experimental data to Eq. (1), the bio-H_2 potential, H_{max}, (63.8±6.7 NL bio-H_2 kg SV^{-1}) peaked at pH 6.5, and then decreased below and over this value. However, data fitting in contrast with experimental results, gives a slightly higher value (68.2 vs. 63.8 NL bio-H_2 kg SV^{-1}) for the case when pH is not controlled with acid or alkalinity addition. Fig. 5 shows that there was a reasonable high correlation (R^2=0.9066) between the bio-H_2 potential (H_{max}) and production rate (R_{max}).

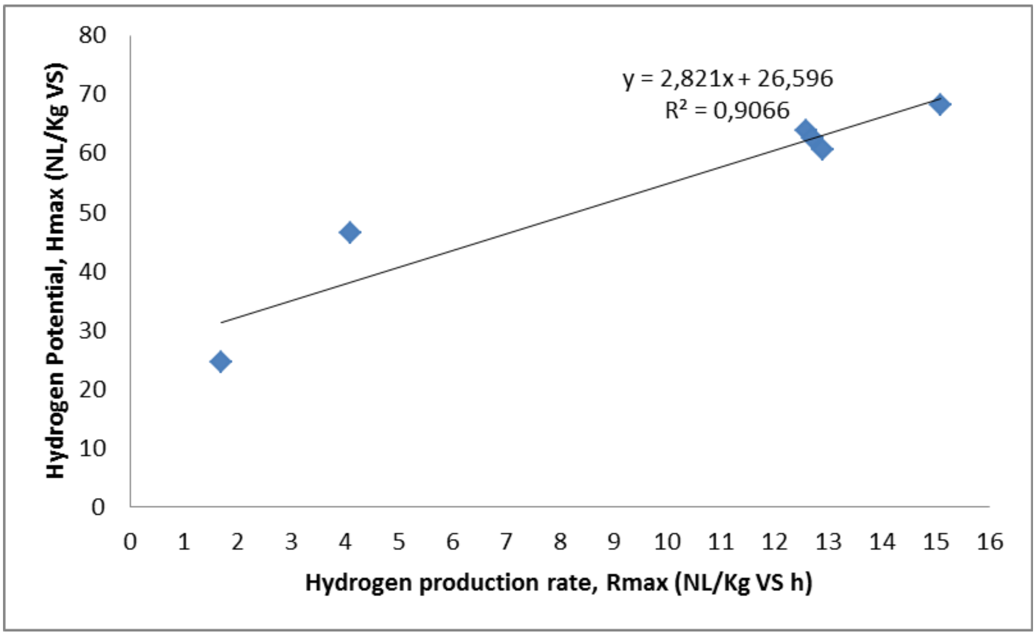

FIGURE 5 **Correlation between the bio-H_2 potential and the maximum bio-H_2 production**

The R^2 = 0.9066 coefficient is similar than the value reported *by* Ferchichi et al. (2005) when using a pure bacterial culture *(Clostridium saccharoperbutylacetonicum).* Therefore, it is concluded that modified Gompertz equation fits accurately the kinetic data when using mixed cultures.

4 CONCLUSIONS

The conclusions of this study are:

- The initial pH and substrate concentrations had several effects over SHPs, SMPs and over lag phase. The addition of acid to the medium up to an initial pH of 6.5 reduces the lag phase from 5.0 hours up to 4.3 without decreasing SHPs for a RCW:CMI ratio of 65:35. The SHP and SMP for those initial conditions were 85.8 NL bio-H_2 kg SV^{-1} (2.33 mmol H_2/g TCOD) and 166.9 NL CH4 kg SV^{-1} (4.95 mmol CH_4 g $TCOD^{-1}$).

- Apart from increasing the total energy produced by producing bio-H_2, TPAD processes removed higher amounts of TS, VS and TCOD. The removal percentages in TPAD processes comparing to SPAD processes increased 50 %, 47 % and 20.5 % for TS, VS and TCOD respectively.

- The energy produced by bio-H_2 represents around 27 % of the total energy produced in both phases. Considering the total energy produced in the SPAD (2337 KJ kg SV^{-1}), it can be concluded that the implementation of a TPAD process increases up to 86 % (4357 KJ kg SV^{-1}) the total amount of energy produced in a SPAD for the optimum pH and RCW:CMI ratio (pH = 6.5, RCW:CMI 65:35).

- Consequently, RCW is a suitable substrate for hydrogen and methane production in TPAD processes as higher yields of energy are produced as well as improving the removal of the organic load comparing to SPAD processes.

- Ammonium nitrogen did not inhibit TPAD processes as their concentrations (0.6-1.3 g L^{-1}) remained below to those considered as moderately inhibitory for AD processes (> 3 g L^{-1}).

- A modified Gompertz indicated a strong correlation with the experimental data as shown by the regression coefficients R^2 =0.999-0.913 values and the high correlation between the bio-H_2 potential, H_{max}, and the maximum bio-H_2 production R_{max}.

Recommendations for further study are as follows:

1. Research of the use of a TPAD processes utilizing a continuous effluent flow from bio-H_2 fermentation is needed along with studies about the use of different substrate concentrations in the methanogenic reactor.
2. Investigation of heat pre-treating cheese whey is needed in order to know the effect of whey indigenous bacteria removal on bio-H_2 production and the subsequent methane production.
3. An economic analysis of the entire two stages process utilizing cheese whey will give greater understanding of the overall efficiency of the process and the payback possible.

5 ACKNOWLEDGEMENTS

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(82) KEY EUROPEAN PLAYERS AND POTENTIAL APPLICATION OF MICROALGAE IN WWTPs

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EXECUTIVE SUMMARY

With the aim of studying the potential to integrate microalgae into wastewater treatment for bioremediation and energy production purposes, different actors, projects and technologies on the microalgae sector have been studied, mainly in Europe. Information on microalgae for wastewater treatment is mainly based on research activities and so the most relevant information has been published by means of scientific papers.

Different technologies from a number of companies can be found in Europe. Some of these companies and their technologies are the following: Algasol Renewables (Spain) with Alga4 PBR, Fotosintetica&Microbiologica SRL (Italy) with Green Wall Panel, Microphyt (France) with its analogous name photobioreactor, Ecoduna (Austria) with PhoBIOR and Proviron (Belgium) with ProviAPT. Regarding microalgae technologies for wastewater treatment, one relevant technology has been found in Europe, namely ABR (AlgaeBioReactor) from the company Ingrepro Renewables in The Netherlands.

Considering harvesting and dewatering technologies, some recent developments in Europe have been documented. GEA Westfalia (Germany) has a centrifuge that can be used to separate microalgae from water, Vito (The Netherlands) is testing integrated permeate channel (IPC) membranes to separate microalgae, and Evodos BV (The Netherlands) has a type of centrifuge with spiral plate technology (SPT), among others. Additionally, the University of Ghent (Belgium) is studying a cheap harvesting method called MaB-flocs (micro-algae and bacteria) which consists of a combination of microalgae and bacteria to induce natural settling (Van Den Hende, 2011).

Even though different microalgae end-uses exist, the most relevant applications for wastewater treatment plants, apart from the treatment itself, concerns anaerobic digestion (AD) of microalgae to produce biogas, mitigation of CO_2 emissions from exhaust gases and CO_2 and H_2S content reduction from biogas. Some experiences of the University of Valladolid (Bahr, 2010) and the University of Rostock (Mann, 2009) seem to be very promising when dealing with the biogas conditioning. In the field of biogas production from microalgae, the project Symbiose has been carried out with the ambition to develop an integrated system designed to produce methane by AD using a source of industrial CO_2, a source of organic waste and solar energy.

In the recent years, the European Comission (EC) has also founded a number of projects under the FP7 Energy program: BIOFAT, All-Gas-Oil, InteSusAl and ALGADISK. Other initiatives have also been founded by the EC under the Life+ program of 2010 and are the following: Agical+, CO2ALGAEFIX and ALGAE-GHG. Moreover, other initiatives are being developed around Europe, mainly at the aim of cultivating microalgae for commercial purposes. Some of these initiatives are the AQUAfuels project, the Algae PARC, the BTM facilities, the ALGAENERGY project, the MAMBO project or the ALCHEMIS project.

In conclusion, microalgae are an important bioremediation agent that may be used in wastewater treatment to solve two key problems: the use of chemicals and high energy costs. Therefore microalgae-based wastewater treatment could become a sustainable alternative to conventional treatments. Moreover, the biomass issued from these microalgae-based treatments is suitable for different applications such as energy production (biogas, biofuels, biohydrogen, etc.) and providing a valorisation way for the waste generated during the treatment, while providing a new source for energy production. In addition, these systems can also be used to mitigate the CO_2 emissions from different sources (e.g. biogas, exhaust gases).

1 INTRODUCTION

The term microalgae refers to a diverse group of organisms, which are generally capable of photosynthesis, and which can be classified into four major categories: (1) photoautotrophic, (2) photoheterotrophic, (3) chemoheterotrophic (not photosynthetic) and (4) mixotrophic (either light or chemicals for energy). Those which are capable of doing photosynthesis, convert solar energy to chemical energy with oxygen (O_2) as a by-product, and in a second step, the chemical energy is used to assimilate carbon dioxide (CO_2) and convert it to sugars (Larsdotter, 2006), according to the following overall stoichiometric formula for photosynthesis: $6\ H_2O + 6\ CO_2 + \text{light} \rightarrow C_6H_{12}O_6 + 6O_2$. The inorganic species normally used by microalgae are CO_2 and HCO_3^-, but some algal species are also able to use organic carbon sources, such as organic acids, sugars, acetate or glycerol. According to algae chemical formula ($C_{106}H_{263}O_{110}N_{16}P$), nitrogen and phosphorus are also important nutrients for microalgae growth, besides carbon. The most common nitrogen compounds assimilated by algae are ammonium (NH_4^+) and nitrate (NO_3^-). In addition, other micro and macro-nutrients are also necessary for microalgae growth but in smaller amounts.

Microalgal cultivation is affected by abiotic factors (temperature, light, nutrients, carbon dioxide and pH), biotic factors (pathogens and bacterial competition) and operating parameters (mixing, dilution rate, depth and harvesting frequency) (Mata et al., 2010).

2 MICROALGAE LIFE CYCLE

Microalgae systems involve three main steps: first the microalgae cultivation step, second the harvesting step in order to obtain the algae cake, and finally the end-use step which consists of processing the algae cake to obtain a valuable product, e.g. "bioenergy"; as presented in Figure 1.

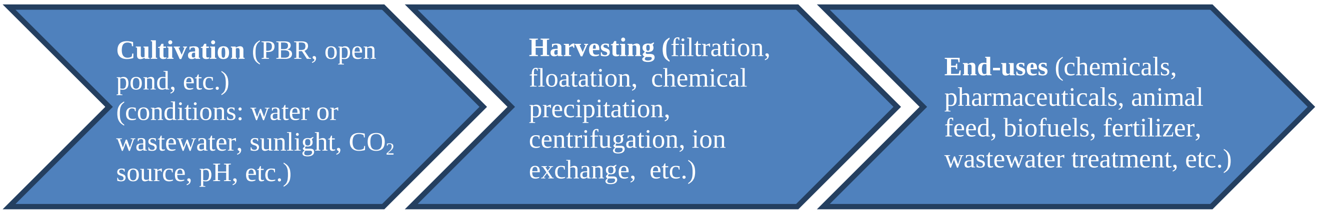

FIGURE 1 **Steps in microalgae life cycle**

2.1 Cultivation technologies

Cultivation technologies can be categorised into three main types: open ponds, photobioreactors (PBRs) and attached growth systems. Open ponds are reactors characterised by being no more than thirty centimetres deep and are mixed via a paddle wheel that circulates water with nutrients and microalgae. Sunlight falls on the reactor surface and is absorbed by the culture, implicating that photon flux decreases with increasing depth.

Regarding PBR systems, they are enclosed transparent reactors for the cultivation of microalgae. In Europe, different companies play an important role, such as Algasol Renewables (Spain) with Alga4 PBR, Fotosintetica&Microbiologica SRL (Italy) with Green Wall Panel, Microphyt (France) with its analogous name photobioreactor, Ecoduna (Austria) with PhoBIOR and Proviron (Belgium) with ProviAPT.

Finally, attached growth systems consist of either immobilizing algae on a surface or encapsulating algae within a porous medium. Ingrepro Renewables (The Netherlands) has begun the process of commercializing the algae encapsulating concept using an innovative wastewater treatment technology called the AlgaeBioReactor (ABR). Nonetheless, past research has considered this technology cost prohibited by the expense of the polymeric encapsulation process (Laliberté et al., 1994). In addition, some research on other immobilized algae-based systems is underway in some universities (e.g. Wageningen WETSUS project, Universite Catolique de Louvain, University of Valladolid).

2.2 Harvesting technologies

Regarding the harvesting step to separate the algal cells from the liquid stream, it has been considered one the major limitations for microalgae-systems to be economically viable for certain applications. Harvesting is particularly difficult since algal cells are negatively charged, present at relatively low concentrations, and often, characterized by small particle diameters. Additionally, algal biomass has a high moisture content, typically greater than 99% by mass (Wiley et al., 2011).

A wide range of harvesting techniques exist including filtration, floatation, centrifugation, chemical precipitation, and ultrasonic vibration, among others. However, similar to solids handling facilities at municipal wastewater treatment plants, Brennan and Owende (2010) suggest that harvesting of microalgae typically follows two steps: first bulk harvesting wherein the algae are separated from the bulk liquid in order to achieve a solids concentrations of 2-7% and a thickening step wherein the harvested algae are concentrated to solids concentrations ranging from 10-20%.

Considering the harvesting and dewatering technologies existing in the EU, some developments have been occurred in recent years. GEA Westfalia (Germany) has a centrifuge that can be used to separate microalgae from water, Vito (The Netherlands) is testing integrated permeate channel (IPC) membranes to separate microalgae, and Evodos BV (The Netherlands) has a type of centrifuge with spiral plate technology (SPT). Additionally, the University of Ghent (Belgium) is studying a cheap harvesting method called MaB-flocs which consists on a combination of microalgae and bacteria to induce natural settling (Van Den Hende, 2011).

2.3 End-uses

Regarding the final processing of microalgae, different applications could be applied to microalgae biomass for a better management of this waste. Therefore, microalgae biomass can be recycled into different products, such us biofuels, biogas, “green” chemicals, cosmetics and pharmaceutical products or animal food, among others. In addition, microalgae can be applied for wastewater remediation while obtaining a biomass with an added-value. In the case of photoautotrophic algae, they can also be applied for fixing CO_2 and thus facilitating the reduction of increasing atmospheric CO_2 levels.

Therefore, microalgae cultivation can have applied for a vast range of different applications, which can be applied both independently or combined. For example microalgae can be cultivated in wastewater while mitigating CO_2 emissions from a biogas CHP engine, and the microalgae biomass can be anaerobically digested to produce biogas or used to produce biofuels if the adequate microalgae strain is cultivated.

3 MICROALGAE APPLICATIONS IN WWTP

3.1 Treatment of different waste and wastewater streams

Although in the United States the research around microalgae has been developed for many years (Oswald et al., 1957), in Europe a growing interest has started in the recent years, and companies and universities have been developing some research on microalgae-based wastewater treatment systems. Therefore, information on microalgae for wastewater treatment is mainly based on research activities. Different wastewater streams can be applied as nutrients sources for microalgae cultivation depending on its origin (urban wastewater, sidestreams from AD, agro-industrial wastewater, piggery wastewater, swine slurry, etc.).

Although microalgae treatment of urban wastewater has potential benefits, the application of microalgae for wastewater remediation has not been implemented in full-scale facilities. In this context some pilot projects involving microalgae cultivation in WWTPs have been launched in the recent years in Spain (WWTPs of Chiclana and Arcos de la Frontera in Cadiz, El Bobar WWTP in Almeria and Vigo WWTP). In addition, research involving the use of microalgae for tertiary treatment, has been conducted by companies and universities such as Ingrepro (The Netherlands), Polytechnic University of Catalonia (Spain), Cadiz University (Spain), Ecoduna (Austria),Wageningen University (The Netherlands), Vito (The Netherlands), Proviron (The Netherlands), Ghent University (Belgium), Fotosintetica&Microbiologica (a spin-off from the University of Florence, Italy), The University of Valladolid Spain), and many more. Regarding microalgae technologies for wastewater treatment, only one relevant technology under development has been found, namely ABR (AlgaeBioReactor) from the company Ingrepro. In addition, the Slovenian company Algen is working on the concept of using anaerobic digestion sidestreams for growing microalgae with the aim of producing biofuels.

Ingrepro Renewables has developed a totally novel and innovative technology, the ABR reactor, which is a compact reactor dedicated to the removal of nitrogen and phosphorus. In this reactor, wastewater is being led alongside encapsulated algae beads which can efficiently absorb the nutrients from wastewater. In due course, the beads become nutrient-saturated and need to be replaced. After filling the system with fresh algae beads, the process starts all over again. The ABR technology is primarily developed for tertiary effluent polishing in order to reach residual nutrient concentrations lower than the guidelines of the European Water Framework Directive; nonetheless, it has also been tested with anaerobic digestion supernatant.

The Slovenian company, AlgEn, is focusing on using biogas anaerobic digestion sidestreams for growing microalgae, which is very interesting option for valorising the “waste” produced in a WWTP without changing the current configuration. In their studies, anaerobic digestion sidestreams is used as nutrient source and the algal biomass produced can be used to obtain different algal products. The remaining biomass is re-introduced to the anaerobic digester as an organic feedstock, and therefore closes the material loop while providing potential energy in the form of biogas.

Regarding industrial wastewater streams (or high loaded streams), some research has been conducted on the treatment on industrial effluents, such as swine slurry, agro-industrial wastewaters, piggery wastewater, livestock wastewater, etc. (Muñoz, 2009; Molinonuevo-Salces, 2010). One aim of applying microalgae-based treatment to industrial streams is to find an alternative, cost-effective solution to traditional land disposal methods, which have overloaded the natural capacity of farms to cope with piggery wastewaters.

In summary, microalgae enhance the removal of nutrients, organic contaminants, heavy metals and furnish an interesting raw material for the production of high-value biochemicals (algae metabolites) or biogas.

3.2 CO_2 capture

Conventional CO_2 capture methods are highly power-intensive which results in high costs. In this context, utilizing microalgae to photosynthetically sequester CO_2 without the need for external energy supply seems to provide a promising solution. Different CO_2 sources can be used to supply inorganic carbon to microalgae, ranging from ambient CO_2 to power plant flue gases to biogas produced in anaerobic digestion. In this last case, microalgae would also be contributing not only to the reduction of biogas CO_2 content but also to the reduction of H_2S biogas content.

In the European context, some CO_2 projects involving microalgae have developed in recent years. In 2007, Researchers at EniTecnologie (Italy) conducted a field experiment of CO_2 uptake, in which the strains of algae were cultivated in a raceway pond supplied with CO_2 from natural gas turbines. The biomass was then harvested and fermented by anaerobic digestion to produce methane, which eventually replaced a fraction of the natural gas used to power the turbines. The residual sludge, containing most of the N, P and other nutrients was recycled back to the cultivation ponds.

E.ON Hanse (Germany) has launched a research project in Hamburg, were they have established a microalgae pilot plant. It uses marine microalgae as a natural CO_2 sink for the flue gases of a 350-MW coal-fired power station in the Bremen precinct of Farge. The aim of the project is to capture a percentage of the power plant’s total emissions, and cultivate two different strains of algae: one strain for utilization as an animal feedstock and the other for biofuel production.

RWE (Germany) is an electric power and natural gas public utility company based in Essen. Through its various subsidiaries, it is active in the generation, trading, transmission and supply of electricity and gas. The company’s declared aim is to expand the electricity and gas business while reducing its own CO_2 emissions. Pillars of growth will form the basis for RWE's future. RWE, together with partners, has launched a project on flue gases from the Niederaussem power station. The gases are fed into an algae production plant in the vicinity of the station to convert the CO_2 from the flue gas into algae biomass.

Regarding biogas used as CO_2 source for microalgae cultivation, two main groups of reference have been found in Europe: the University of Valladolid (Spain) and the University of Rostock (Germany) along with the Lithuanian University of Agriculture.

The University of Valladolid has developed some research for the removal of H_2S and CO_2 from biogas by means of algal-bacterial systems, which constitute one of the few processes capable to cope with both pollutants at minimal energy costs. In these systems, photosynthetic microorganisms use solar energy to fix CO_2 while concomitantly producing oxygen that is subsequently used by sulphur oxidizing bacteria to oxidize H_2S to SO_4^+ (Bahr, 2010).This research aims to design and optimize a pilot plant based on microalgal-bacterial consortia for the simultaneous elimination of H_2S and CO_2 with an experimental setup which consists of a 200 L, artificially illuminated high rate algae pond (HRAP) for biomass growth with an absorption system for CO_2 and H_2S from biogas (Bahr, 2010).

The University of Rostock along with the Lithuanian University of Agriculture have performed a study that applies microalgae as a low-cost method for biogas conditioning. Their first results showed that the biogas components H_2S and CO_2 could be reduced up to 97,07% and 100%, respectively. In this investigation a distinct positive synergistic effect between simultaneous biogas-conditioning and algae biomass production was determined. It has been also claimed that the cultivation of microalgae (Chorella Vulgaris) with biogas as carbon source is regarded as a feasible biogas-conditioning method (Mann, 2009).

3.3 Energy production

Regarding energy production from microalgae, different conversion options exist and they can be separated into two basic categories, namely thermochemical and biochemical conversion, as presented in Figure 2.

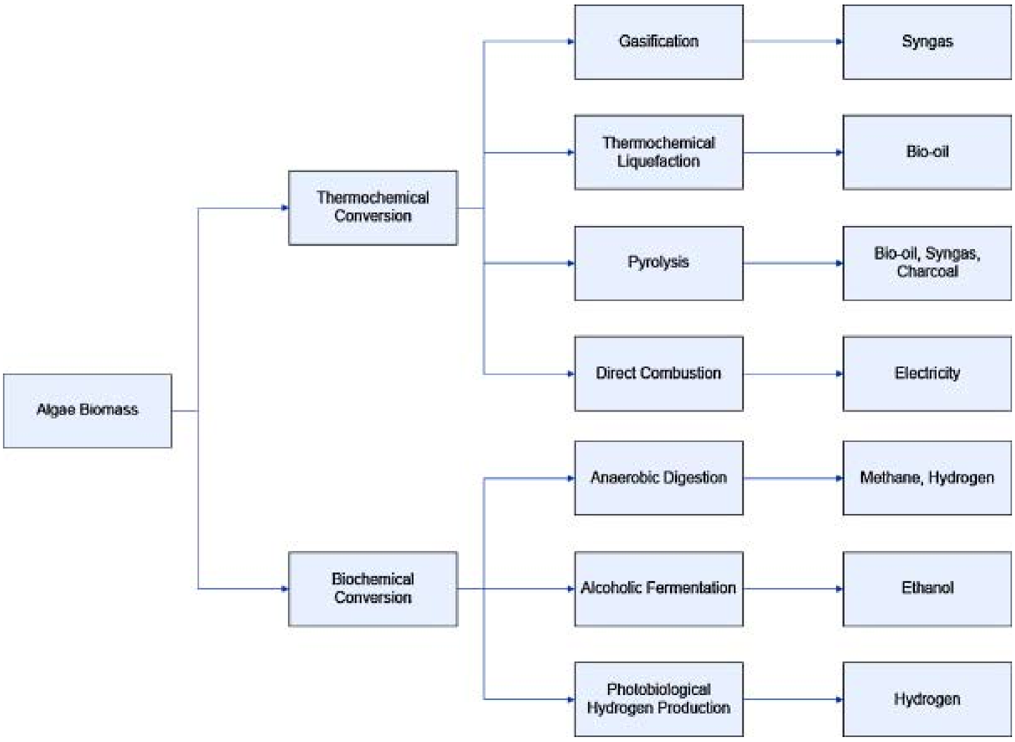

FIGURE 2 **Comparison of various energy-based end-uses for algal biomass (Tsukahara, 2005)**

Thermochemical conversion covers the thermal decomposition of organic components in biomass to yield fuel products and is achievable by different processes such as direct combustion, gasification, thermochemical liquefaction, and pyrolysis. The biological process of energy conversion of biomass into other fuels includes anaerobic digestion, alcoholic fermentation and photobiological hydrogen production. Factors that influence choice of conversion process include: the type and quantity of biomass feedstock, the desired form of the energy, economic considerations, project specificities and the desired end form of the product. (Brennan et al., 2010)

Within these different conversions paths for microalgae energy-conversion, the anaerobic digestion of microalgae represents an important energetic valorization strategy for microalgae to be directly linked with WWTPs but also with biogas plants.

In this way, the Symbiose research project (France, 2009-2011) was launched with the ambition to develop an integrated system designed to produce methane using a source of industrial CO_2, a source of organic waste and solar energy. New fields of research were also investigated, such as co-digestion of an organic substrate with algal biomass and recycling mineral nutrients into the microalgal growth unit, ecosystems involving algae-bacteria symbiosis, and modeling and ecodesign of the integrated process. During the project it has also been seen that coupling a microalgal pond with anaerobic digestion is a promising alternative for sustainable energy production by transforming CO_2 into methane using solar energy. The Symbiose project also demonstrated the ability of the original Anaerobic Digester Model (ADM1) and a modified version (based on Contois kinetics for the hydrolysis steps) to represent microalgae anaerobic digestion. Simulations were compared to experimental data of an anaerobic digester fed with Chlorella vulgaris. The results from the modified ADM1 simultations adequately fit the project’s experimental data, which encompass a variety of influent load and flow rates. It turns out to be a reliable predictive tool for optimising the coupling of microalgae with anaerobic digestion processes (Mairet, 2011).

4 OTHER RELEVANT INITIATIVES IN THE EUROPEAN CONTEXT

The European Commission is funding a number of projects under the FP7 Energy program: BIOFAT, All-Gas-Oil, InteSusAl and ALGADISK. These projects are aimed at large scale demonstration of biofuels production from algae with the following several targets: minimum plantation area of 10 hectares (24.7 acres), minimum productivity of 90 dry solid tons per hectare per year and supply of CO_2 from renewable applications, so excluding CO_2 from fossil fuels. Under the Life+ program of 2010, some relevant projects that are being founded are the following: Agical+, CO2ALGAEFIX and ALGAE-GHG. Moreover, other initiatives are being developed around Europe, mainly at the aim of cultivating microalgae for commercial purposes. Some of these initiatives are the AQUAfuels project, the Algae PARC, the BTM facilities, the ALGAENERGY project or the ALCHEMIS project.

All these iniatives involve an integration of different microalgae applications, such CO_2 emissions mitigation and biofuels production in the case of the FP7 Energy program; or CO_2 sequestration and the production of value-added bioproducts in the case of the project ALGAE-GHG.

The AQUAfuels project aimed at establishing the state-of-the-art on research, technological development and demonstrative activities relevant to the production of 2nd generation biofuels from non-food aquatic biomass. It also aims to draw the detailed, comprehensive and concrete picture of the current status of Europe and international initiatives on algae biofuels; and based on this information, elaborate an overall assessment on the technology and identify major research and industrial needs.

TheAlgae PARC carried out by the Wageningen University and Research Centre in the Netherlands, and it aims at bridging the gap between laboratory-based research and the industrial production of algae. The main algae cultivation systems will be measured against costs, efficiency and sustainability. The industrial partners in the AlgaePARC research programmeare BASF, DSM, Drie Wilgen Development BV, ExxonMobil, GEA, Heliae, Neste Oil, Nijhuis Water Technology, Paques, PDX, Proviron, Roquette, Sabic, Simris Alg, Staatsolie, Synthetic Genomics, TOTAL and Unilever.

Similar facilities to the Algae Parc have been built in Spain. One is the plant of ALGAENERGY in Madrid, co-financed by two large Spanish energy companies (REPSOL and ACCIONA). ALGAENERGY has developed a Technological Platform for Experimentation with Microalgae (TPEM) for several new types of photo-bioreactors (PBRs) in different

configurations. The plant will be entirely automated and controlled by specially-designed software which controls all the cultivation parameters, with a particular emphasis on research into the production of biofuels.
The other important microalgae cultivation facility in Spain is the BTM facility in Cadiz (Southern Spain), which belongs to the AURANTIA Group. BTM facilities, located in Cadiz (Southern Spain) comprise a culture collection of unique species and varieties with high commercial value.

In Belgium, the ALCHEMIS project examines the productionn of algae based on flue gas and wastewater. Besides the production of algae, harvesting of algae is examined and an assessment of the market for algae will be made. An algae farm with a production surface of approximately 500 m^2 has been set up. Alchemis gets support from the government agency for Innovation by Science and Technology (IWT). Partners include, among others, Proviron, VITO, Orineo and the University of Ghent.

The project Agical+ (Belgium) has the aim tovalidate an environmentally friendly system, combining carbon dioxide capture and biofuel production based on algae culture for industrial exhaust application. It also proposes to implement an innovative solution, based on algae culture and biomass production, which will allow for the CO_2 capture of lime or glass furnace fumes and the production of biofuel that can be used within the furnaces during the production process.

The project ALGAE-GHG (Romania) has the overall objective to develop a demonstration integrated photosynthetic system based on the sequestration of greenhouse gases in algal biomass used as raw materials for value-added bioproducts for use in various applications including lipids as an alternative source for biofuels and horticulture oils.

The project CO2ALGAEFIX (Spain) has established the objective to demonstrate, at a one hectare surface scale, an efficient way to capture carbon dioxide from stationary sources (in this case, a power plant that uses natural gas). It also aims to demonstrate that carbon dioxide emissions can be used as a substrate for biomass algae production. Finally it will also evaluate possible uses of the microalgae, for example, in the production of energy.

5 CONCLUSIONS

Microalgae-based systems appear to provide a sustainable alternative solution for the treatment of industrial and municipal wastewater, a critical problem faced by industries and municipalities worldwide. As well as treating the water, these systems, are able to produce a valuable waste that could be transformed into a different number of products, such as bioenergy. In addition they are capable to cope with the CO_2 emissions from different sources, such as flue gases or biogas from AD plants. Therefore, both WWTPs and biogas plants are claimed to be an important source of nutrients for microalgae cultivation, and with higher potential for energy production.

In Europe, a growing interest has started in the recent years for different purposes and many actors are becoming involved in different microalgae projects. In this paper, some relevant references have been briefly commented, however other research and development actions, projects and actors exist within the topics commented.

Currently the main efforts of microalgae cultivation in Europe are devoted to the CO_2 emissions mitigation and the production of energy, mainly by means of biofuels, or the production of value-added products. However, due to the constraints in terms of legislation for the re-use of treated wastewater for human applications, the research and development on using wastewater as nutrient source has been focused on energy production.

6 ACKNOWLEDGEMENTS

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Session 33

(35) EVALUATION FRAMEWORK TO COMPARE THE SUSTAINABILITY OF DIFFERENT APPLICATIONS OF BIOMASS WASTE

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EXECUTIVE SUMMARY

In recent years the demand for biomass as a renewable energy source has increased spectacularly. Biomass has started to play an important role in the production of heat, electricity and bio-fuels, contributing significantly to the achievement of the EU renewable energy objectives. Apart from energy applications, biomass is a versatile resource for food, feed, fibre, construction material or raw material for the chemical industry. As the amount of biomass that can be produced and mobilized in a sustainable way is limited, questions arise on the most optimal and sustainable way to use the available biomass streams. It seems obvious to direct the available biomass towards those applications which contribute most towards societal sustainability objectives while avoiding the disturbance of traditional biomass-dependent sectors or unwanted side-effects from dedicated support mechanisms.

In a recent study VITO developed an evaluation framework which allows policy makers to compare the sustainability of different applications starting from a specific biomass stream, in terms of environmental, economic and social impacts. This means that the performance of different applications in very different sectors needs to be compared based on multiple criteria. For this purpose, a set of eleven indicators is constructed to assess the main sustainability aspects of each application, taking into account the variety of applications for a specific biomass stream. The methodology that is used to do the evaluation is based on the life-cycle thinking (LCT) approach. For each biomass application it is determined which traditional alternative is replaced by the use of biomass as a renewable resource. By measuring or calculating the indicators an evaluation is made of the environmental, economical and social impacts that are avoided or created by replacing the usual alternative with the biomass stream under consideration. Applications in different sectors are compared by evaluating the improvement that can be obtained by dedicating the biomass resource to one or the other application.

In order to show its merits and its limitations, the developed methodology and indicator set is applied on the cases of post-consumer wood and rendered animal fat. The most challenging part of applying the methodology is the development of verifiers for every indicator. Some indicators are relatively easy to quantify, while others are not, due to lack of data or measuring methods. Especially the non-environmental indicators (economic and social) have proven to be difficult to quantify. The choice of the avoided alternative processes turned out to be a crucial element in the results. The differences in net impact between the studied applications of biomass waste are largely dominated by the choice of the alternative processes or primary materials that are avoided by the use of the biomass stream, stressing the importance of a sound market analysis to identify the marginal alternative process that will be replaced. In practice, the alternative process that is replaced depends on existing policies concerning renewable energy and the local market forces of demand and supply.

The methodology can be used as a tool for policy makers to direct biomass waste streams into a direction that serves society best. However, the results do not indicate a “best scenario”, but show a shift of impacts when switching from one application to the other. This means policy has an important role in setting priorities. In order to include sustainability criteria in policies, a good understanding is needed of the level and scale on which different indicators play. While impacts from waste treatment and waste processing are generated on a local level, the avoided impacts often occur abroad. Secondly, indicators differ on their level of action: some can be calculated and influenced on a company or national level, while others play globally.

1 INTRODUCTION

1.1 Background

In recent years the demand for biomass as a renewable energy source has increased spectacularly. Biomass has started to play an important role in the production of heat, electricity and bio-fuels, contributing significantly to the achievement of the EU renewable energy objectives. The main drivers for this evolution are the exhaustibility of fossil fuels, the determination to reduce greenhouse gas emissions and the desire to reduce our dependency on import from oil-producing regions. Apart from energy applications, biomass is a versatile resource for food, feed, fibre, construction material or raw material for the chemical industry. The amount of biomass that can be produced and mobilized in a sustainable way is limited. As the demand for biomass increases faster than the supply questions arise on the most optimal and sustainable way to use the available biomass streams. It seems obvious to direct the available biomass streams towards those applications which contribute most towards societal sustainability objectives, while avoiding disturbing traditional biomass-dependent sectors or unwanted side-effects from dedicated support mechanisms.

1.2 Research objectives

The aim of this study is to develop an evaluation framework which allows policy makers to compare the sustainability of different applications for a specific biomass stream, in terms of environmental, economic and social impacts. This means that the performance of different applications in very different sectors needs to be compared based on multiple criteria. The innovative and challenging character of the study lies in the fact that different types of applications are considered in the comparison, while most existing methodologies focus on one particular application, usually energy production. When comparing applications we start from the available biomass stream. Therefore, the sustainable production of biomass is considered as a intrinsic precondition and lies out of the scope of this study. The evaluation framework compares the impact of different applications of biomass, but does not evaluate the effectiveness of these applications as such. As a basic principle it is assumed that all applications are needed or wanted by society and have to be produced, using either biomass or an alternative resource.

1.3 Principles, criteria, indicators and verifiers

A methodology is based on a set of principles that define the fundamental framework. The principles of sustainable development are defined by the Global Reporting Initiative as the triple-P principles (People, Planet and Profit). These principles have to be translated into criteria, whose status has to be determined by unambiguous indicators. In a last phase, an indicator is made measurable by developing verifiers that can give a qualitative or quantitative value which makes it possible to compare different results. This study is aimed at translating the Triple-P principles into criteria, indicators and verifiers that are suitable to compare different applications of a biomass stream.

In recent years a broad range of initiatives were taken to build indicator sets for sustainability in general, and for biomass use and bio-energy in particular. The European Directive on the promotion of the use of energy from renewable sources (2009/28/EC) has been the first regulation in which sustainability criteria were introduced for bio-fuels and liquid biomass. According to the directive, every producer of bio-fuels is obliged to demonstrate that 35% CO_2 reduction has been achieved in comparison with fossil fuels, that the biomass used is not originating from land with a high biodiversity or carbon storage and that good agricultural practices are followed. In addition to these four criteria, the EC has the intention to report on seven additional issues that are not quantifiable at this moment. The Global Bio-energy partnership launched in 2011 a report that proposes 24 sustainability indicators for bio-energy, according to the Triple-P principles (GBEP, 2011). The Dutch commissions Cramer and Corbey developed a framework based on six themes (Cramer, 2007): greenhouse gas reduction, environment, biodiversity, competition with other applications, prosperity and well-being. These themes have been elaborated further in the certifying standard NTA8080. Van Dam (2010) has presented an overview of 67 sustainability frameworks, each applicable for a certain biomass use, e.g. bio-energy in general, electricity, bio-fuels, agricultural products, organic farming, forestry and social labelling such as fair-trade products. The Eco-pyramid principle (Derksen, 2008) ranks biomass applications according to volume, value, exergy and environmental impact and aims at optimisation of production chains and loop closure, with energy production as final option. The International Energy Agency (IEA) focuses on the bio-refinery concept as key towards sustainability of biomass use, with attention to competition with food, water use and quality, land-use, soil-carbon and fertility, greenhouse gas balance, biodiversity, toxicological risks and energy efficiency, while maximising the total value derived from biomass feedstock (IEA, 2010).

2 METHODOLOGY

To evaluate the more sustainable application of a biomass waste stream implies that a choice has to be made between different application possibilities in very different sectors. This requires that the performance of the biomass in the different applications can be determined, measured or calculated and finally compared based on multiple indicators. The methodology that is used for this challenge is based on the life-cycle thinking approach and is also sketched by the study commissioned by Platform Groene Grondstoffen in The Netherlands (Ecofys, 2009).

2.1 Comparison of an application with its alternative

For each possible application of a biomass waste stream, the alternative product or production process is identified. In this regard the question is raised which alternative has to be taken into account, as often several alternatives are possible (Ecofys, 2009):

- the fossil alternative: the traditional production process based on fossil resources, if applicable;
- the average alternative: the impact of the alternative process is calculated as the average impact of the mix of available alternative processes;
- the marginal replaced product/process: the alternative process which will be replaced first by the new process, due to practical or economic reasons;
- the ‘best’ alternative: based on the ‘best available technology’ at the moment of research.

In a second step, the impacts that are avoided by using biomass waste instead of the alternative production route are determined. This evaluation is done using the life cycle thinking approach (JRC, 2010).

2.2 Life cycle thinking

Products (goods and services) create several environmental impacts during their life cycle. Life cycle thinking (LCT) is an integrated approach that seeks to lower environmental impacts and reduce the use of resources across all life cycle phases of a product or system (JRC, 2010). Instead of limiting the environmental impact assessment to certain core production processes, it considers the entire product chain in the evaluation by including the indirect impacts of extraction of raw materials, supply chains, product use, re-use, recycling and disposal. The key aim of LCT is to avoid shifting of burdens: minimising impacts at one stage of the life cycle, while taking care not to cause increases in another life cycle stage, another region or another impact category.

When applying the LCT approach on a production process that uses waste as a resource material, the environmental impacts associated with the creation of the waste material are not included in the impact assessment. The historic events associated with the waste production are not relevant for the further evaluation, only the impacts associated with the further treatment or use of the waste are assessed.

2.3 Comparison of different applications

When the impacts of different applications of biomass waste have to be compared using LCT, a common ground for comparison is needed. The products, processes or systems that are to be compared need to fulfil the same function. To create equivalent systems with the same function, the method of system expansion is used. Suppose a certain biomass waste W needs to be processed and has two potential applications: process A producing product a, or process B producing product b. Both products a and b are in demand by society. In order to compare both biomass process routes, the system needs to be expanded in order to fulfil the same function (figure 1). If W is used to produce product a, product b needs to be produced in an alternative way (b’) e.g. by using a traditional (often fossil-based) process, because society demands both a and b. Similarly, if W is used to produce product b, product a needs to be made using an alternative process (a’). An alternative but equivalent way of creating equivalent systems is the method of system substitution. In this method, the production of product a from W avoids the production of a’ using the alternative or traditional process. In this case, the avoided impacts of the traditional production of a’ are subtracted from the impacts of producing a from W.

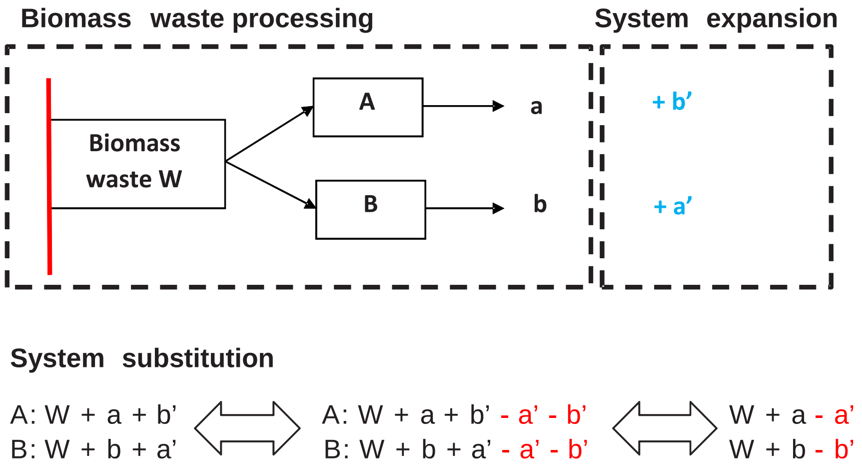

FIGURE 1 **Principle of system expansion and substitution to create equivalent systems**

By using the substitution approach, products a and b are not compared as such (which is problematic as they have different functions), but the environmental, social and economic impacts and benefits of producing a (from waste) instead of a' (traditional process) are compared with the impacts and benefits of producing b instead of b'. This comparison allows to determine the application in which the biomass waste can be used the most efficiently. However, the comparison does not take into account potential process optimisations within a certain application, neither is a value judgement imposed on the usefulness, necessity or desirability of the application.

3 SELECTION OF AN INDICATOR SET FOR SUSTAINABLE USE OF BIOMASS

An important aspect of the methodology is the choice of representative indicators that will be used for the comparison of different biomass applications. To keep the methodology practically feasible in a policy context, a concise set of indicators needs to be constructed, that considers the relevant environmental, social and economic impacts. Based on extensive literature review, the following eleven indicators were selected (figure 2).

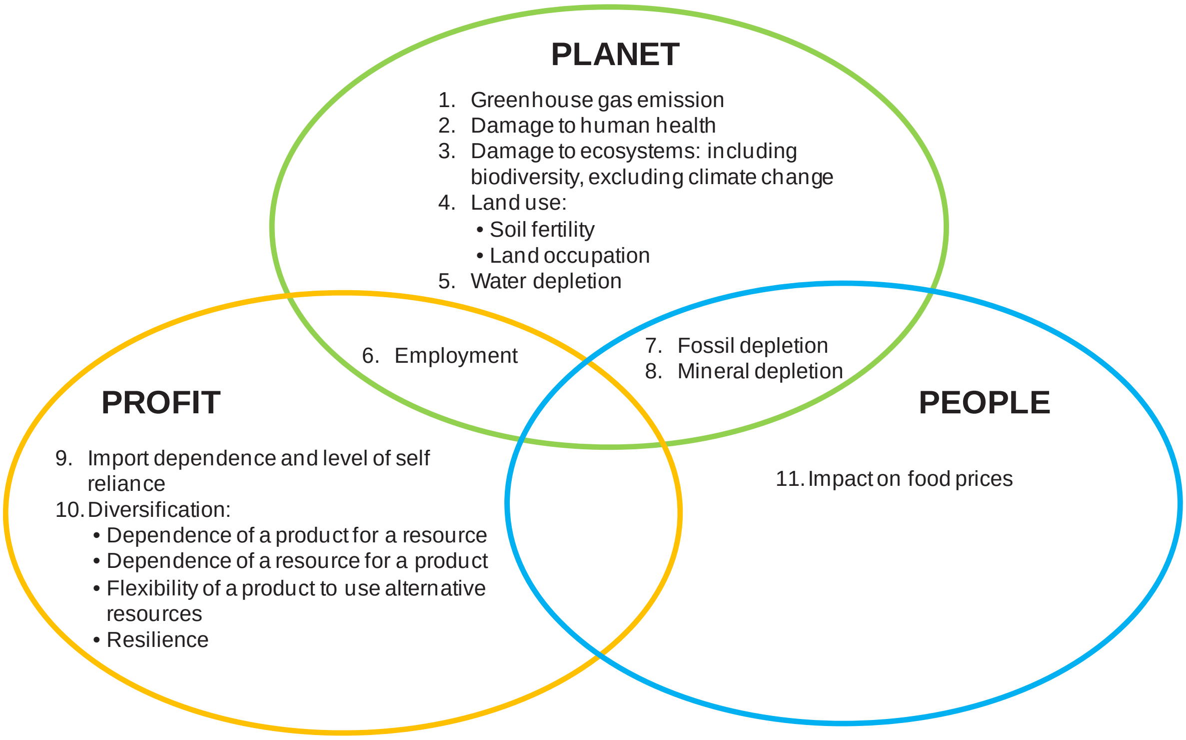

FIGURE 2 **Selection of sustainability indicators**

4 LESSONS LEARNT

The developed methodology and indicator set was applied on the case studies of post-consumer wood and rendered animal fat. Alternative application options were compared. In the case of post-consumer wood the studied applications were particle board production, electricity, heat production and bio-based chemistry. In the case of rendered animal fat, the studied applications were electricity and biodiesel production. The lessons learnt from these exercises are presented in the following paragraphs.

4.1 Practical issues and challenges

The most challenging part of the methodology is the development of verifiers for every indicator. Some indicators are relatively easy to quantify, while others are not, due to lack of data or measuring methods. For the calculation of the environmental indicators life cycle impact assessment (LCA) methods like Eco-Indicator 99 (Goedkoop, 2001) or ReCiPe (Goedkoop, 2009) are often suitable, providing the necessary input data are available and adequate assumptions can be made. However, for the quantification of the indicator ‘soil fertility’ a workable measurement method is lacking. Additionally, also the non-environmental indicators (economic and social) have proven to be difficult to quantify, both for reasons of deficiency of specific data and lack of measurement methods.

A second challenge is the geographic aspect of impacts. An indicator like greenhouse gas avoidance has an impact on a global scale, while the indicator water depletion has an important local component. Apart from absolute water usage, the susceptibility of the extraction region for water shortages is very important. However, this type of detailed geographical data is lacking in most cases. Additionally, the generated and avoided impacts are not necessarily taking place in the same geographic area. Impacts from waste treatment and waste processing are generated on a local level, while the avoided impacts from the avoided processes often occur abroad. For example, the EU typically imports rapeseed for biodiesel production from Russia, Kazakhstan and Australia (Agrarisch Dagblad, 2010). This implies that, when biodiesel is produced using rendered animal fat instead of rape seed, its process impacts will be local, while the avoided impacts due to avoided rapeseed cultivation will take place abroad.

4.2 Importance of the chosen alternative

The differences in net impact between the studied applications of biomass waste are largely dominated by the choice of the alternative processes or primary materials that are avoided by the use of the biomass. When the avoided process for one of the applications has a large impact on certain indicators, this results in a shift of burdens/benefits towards this application. Figure 3 shows an example of this phenomenon for the case on rendered animal fats. In this figure two potential applications of rendered fat are compared: electricity production and biodiesel production. In the case of the electricity production scenario, two different avoided alternative processes are considered: traditional electricity production using the average Belgian fossil mix (natural gas, coal, fuel oil) and the average Belgian total mix, which also includes an important share of nuclear energy. The result shows that the net benefits of the electricity production scenario depend largely on the avoided process used. Moreover, there are large differences between the different indicators.

Natural gas and oil are relatively scarce and using them for electricity production results in a large impact for the indicator ‘fossil depletion’ and, due to CO_2 emissions, ‘greenhouse gas emission’, which means that avoiding them by using rendered fat results in a large environmental benefit for both indicators. When (low emission) nuclear energy production is included in the avoided process, these benefits are significantly lower, while a larger benefit is found on the indicator ‘mineral depletion’. In a similar way, the benefits of the production of biodiesel depend to a large extent on the assumptions concerning the avoided products: biodiesel from rapeseed oil or fossil diesel. As rapeseed has to be cultivated on agricultural land, using rendered fat instead yields a significant benefit for the indicator ‘land use’. This results shows the importance of well evaluating the alternative process that is most likely to be replaced by the new biomass application. In practice, the alternative process that is replaced depends on existing policies concerning renewable energy and the local market. If policy objectives for renewable electricity and bio-fuels have been reached, one renewable fuel (e.g. rendered animal fat) could replace another (rapeseed oil, palm oil, …). In a situation where the policy objectives are not yet reached and the market is evolving towards an increasing use of renewable fuels, the new renewable fuel will replace fossil fuels. In addition to this policy effect, a limited supply of waste materials on the local market may also play a role. Different applications may have to compete with one another. A market study should be performed to identify the marginal alternative process that will be replaced.

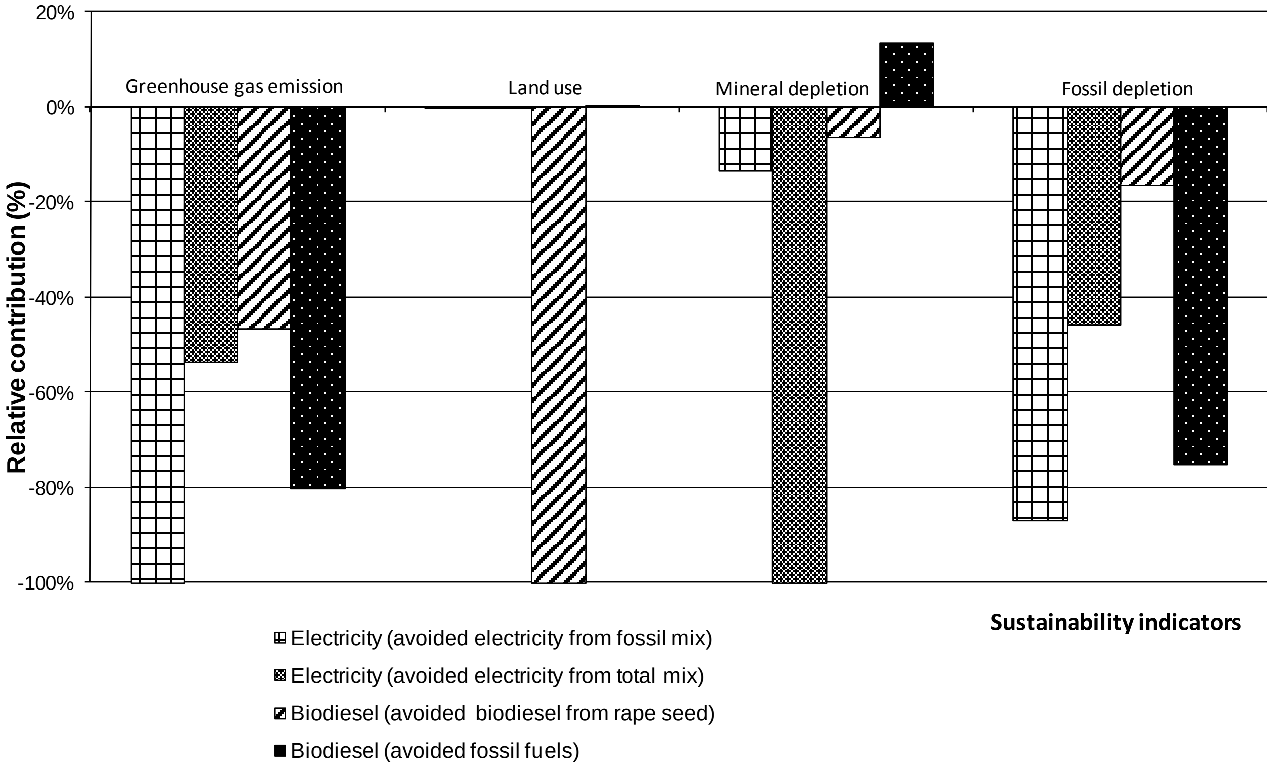

FIGURE 3 **Importance of the avoided alternative in the case of electricity and biodiesel production from animal fat**

4.3 Priority indicators

Figure 3 shows that in the case of rendered fat none of the evaluated scenarios (electricity or biodiesel) is globally better than the other for the indicators under consideration. A similar conclusion could be drawn from the case on postconsumer wood waste. The results do not indicate a “best scenario”, but show a shift of impacts when switching from one application to the other. In the case of rendered fat electricity production shows higher benefits for the indicators ‘greenhouse gas emission’, ‘mineral depletion’ and ‘fossil depletion’, providing the average Belgian fossil mix is taken as the avoided process. Biodiesel production results in a large benefit on the indicator ‘land use’, assuming biodiesel from rapeseed is the marginally avoided process. When the proposed methodology is used in order to make an evaluated choice between different biomass application options, this implies that priorities have to be set among the different indicators, e.g. by attributing weight factors. These weight factors have to be determined in accordance with local or national priorities and included in policy.

4.4 Organisational level of impacts

A third relevant aspect is the organisational level on which the indicators play. Some indicators, like ‘green house gas emission’, can be calculated on a project or company level or on a national level. In this case national policy can regulate these impacts by setting goals or norms. However, other indicators, such as ‘impact on food prices’ play on a global level. In this case, the impact on this indicator of a company level decision cannot be calculated. Local and national policy makers need to be aware of these limitations when designing policy.

5 CONCLUSIONS

The development of an evaluation framework to compare the sustainability of different applications of biomass waste is pioneering work. Even in a broad European context it has proven to be difficult to develop quantifiable indicators that are applicable to different applications of biomass. For the moment, the international community focuses on a further and better interpretation of sustainability indicators for one specific application or only for energy-related applications, such as the recent report by the Global Bio-energy Partnership that seeks to quantify 24 sustainability indicators (GBEP, 2011). Several questions remain unsolved and keep the discussions ongoing. Will the EU implement sustainability criteria for solid biomass? If so, which ones have to be included? How to estimate and quantify indirect land use changes (EC, 2010)? Especially the development of verifiers has proven to be problematic. Moreover, time is an important factor in these discussions: insights grow and priorities change, which will urge the indicators to evolve simultaneously. The methodology for comparison that is presented in this paper has been tested on two cases, showing its merits and its limitations. The availability of data and lack of adequate measurement methods proved to be problematic for some indicators. The choice of the avoided alternative processes turned out to be a crucial element in the results, stressing the importance of a sound market analysis. The methodology can be used as a tool for policy makers to direct biomass waste streams into a direction that serves society best. However, priority setting is needed, as well as a good understanding of the level and scale on which different indicators play.

6 ACKNOWLEDGEMENTS

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Session 34

(16) AUTOMATIC PROCESS CONTROL FOR ANAEROBIC DIGESTION

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EXECUTIVE SUMMARY

The aim of research was to develop a simple and cost-efficient control strategy, which besides secure maintenance of a stable process allows to automatically adjust to user-defined operation states. By these means the operation of the process can be simplified. A well-directed adjustment of the operation state allows the optimization of plant operation.

Considering model-based development as an appropriate method for the development of control strategies for the anaerobic digestion process, the Anaerobic Digestion Model No.1 (ADM1 – see Batstone et al. (2002)) was used to simulate the process. The calibrated ADM1 serves to theoretically calculate the behaviour of different control strategies and to adjust the control settings to specific plant conditions. It is intended to estimate the capability under practical conditions by including different disturbances (e.g. measurement errors). To finally practically verify and assess the previous development steps the whole procedure is completed by experimental operations of control strategies.

The developed control strategy is based on the utilization of produced amounts of methane as intended final product of the complex anaerobic degradation paths. It is assumed that the methane production under undisturbed process operation also reflects the load level or the operation state of the process. Consequently an operator-defined set-point to a value of methane production to which the system finally should be adjusted, is required. To adjust the amount of input of a fix substrate mixture according to the daily measured methane production proportional-integral controllers (PI controllers) or proportional-integral-differential controllers (PID controllers) with a limited differential part, are used. At the beginning of control actions the controller set-point is set to a value close to the actual methane production. The state of the process hast to be assessed reliably by a reasonable combination of additional and on-line ascertainable process parameters.

Besides common measurements like pH, the control concept is based on the direct availability of further process parameters like concentrations of single volatile fatty acids, buffer capacity or dry matter content. Within a research project at the University of Hohenheim the online determination of concentrations of single volatile fatty acids with near-infrared-reflectance spectroscopy (NIRS) is investigated (see Stockl et al. (2011)). The intentions of measuring a sufficient number of process parameters are to characterize and to constantly monitor the state of the process. If alarming values are exceeded, the control system requests manual inspection of the plant. Under normal operation the state characterization of the process and the operator-defined set-point are used to generate an automatic decision about changes of the controller set-point, for example hebdomadally. In the case of trusted process stability and an operator-defined set-point above the controller set-point this would lead to raising the controller set-point by a fix value. By these means the plant is, always according to its performance capacity, slowly conducted towards the operator-defined operation state. This whole knowledge-based procedure is subsumed under the term “adaptation matrix”.

The described PI(D)-controls were successfully operated on experimental digesters with volumes of approximately 450 and nearly 40 litres. Natural variations in process operation, substrate quality, accuracy of feeding and measurement, as well as potential influence of the stirring, led to a fluctuating approach to the particular controller set-points. If the necessary requirements regarding operation, measurements, process, substrate mixture etc. are followed, it can be stated that the PI(D)-control generally works. Further experiments, especially for full-scale and long-term operation completely equipped with the necessary automatic measuring instrumentation, will be needed to confirm the obtained results. Detailed information about the research topic, its background and the findings and conclusions is provided in Löffler (2012).

1 INTRODUCTION

Within the last decade, the number of biogas plants in Germany has increased significantly (see FvB (2011) and FNR (2011)), at which most of the development was and is still taking place in the sector of agricultural biogas plants. The main driver of this development, the Renewable Energies Sources Act (Erneuerbare-Energien-Gesetz, EEG) as already indicated by its name, focuses on energy production. Considering the limit of a maximum biogas production potential which can be used in a sustainable manner, the current utilization rate in relation to this biogas potential consequently also increases. Accompanied by this effect, more attention is paid to sustainable future development scenarios, the related infrastructure and to further development of the technology and the consequences of its utilization. Advances to improve the efficient utilization of the existing potentials are expected within the fields of biomass production, plant operation and design, process optimization or biogas utilization. Within this context and because no automatic control for the process has established as state of the art yet, the approach followed here is the development of a control strategy for the anaerobic digestion process.

The research objective was to develop a simple-to-implement and cost-efficient automatic control strategy, which is capable to maintain the process stable, while optimizing it according to user-defined settings. With the control strategy it is intended to enable easy handling, continuous monitoring of the process and comfortable automation, to avoid operational errors, to achieve better resource efficiency and to reliably and securely maintain process stability.

2 METHODOLOGY

The chosen methodology for the development and evaluation of an appropriate control strategy is based on the simulation of the anaerobic digestion process using the Anaerobic Digestion Model No.1 (ADM1) (see Batstone et al. (2002)). The ADM1 is used here in a Matlab® implementation as described in Cimatoribus (2009). By calibrating the ADM1 with operational data to existing digesters, virtual digesters imaging the real ones are obtained. Control strategies can then be developed taking essential process characteristics and possible control mechanisms into consideration.

Hence, the calibrated ADM1 can be used as a virtual experimental platform to adjust, test and compare developed control strategies on the computer. By tentatively running different control and disturbance scenarios the behaviour of the control strategy can be evaluated preliminarily and it can be examined for its performance limits. Model-based development shows the advantage that time- and cost-intensive real experiments can be avoided. Nevertheless operational experiments have to be the consequently next steps within the development procedure. These experiments from laboratory scale to full scale finally assure that the system or specific parts of it are working. By these means the control strategy can ultimately be proved or in case of contrary findings previous step within this iterative process have to be revised. If the experiments are not exclusively meant to examine specific parts of the control strategy, they have to be conducted as close to practical conditions as possible.

3 RESULTS AND DISCUSSION

3.1 The control strategy

Following a hierarchical design as proposed for example in Konstantinov et al. (1993), the developed control strategy consists of two levels. A “conventional”, time discrete, digital Proportional-Integral control (PI control) or a Proportional-Integral-Derivative control (PID control) as in Löffler et al. (2011) constitutes the part of the control strategy operated on the basic level. A knowledge-based part is employed to guide the “conventional” control and to monitor the state of the process. A detailed description of the control strategy is provided in Löffler (2012) and Kranert et al. (2011).

The high energy content of methane together with the main focus of most biogas plants on energy production, explains why methane production can be denominated as the most relevant product of the anaerobic digestion process, especially under economic aspects. As the methane production also constitutes one of the final products of the whole anaerobic reaction chain, it can be stated that it also indicates, within certain boundaries, the state and the loading rate of the process. Generally speaking and assuming that no disturbances, inhibition, overloading or similar impede this relationship, it can be observed that the more organic matter is fed to the process, the more biogas and methane are produced. Therefore the methane production was chosen as the control variable. The manipulated variable is here represented by the fix mixture of the two substrates cow manure and corn silage.

This means that the measured daily methane production is used to adjust the amount of input of the fix feeding mixture to finally reach the methane production which is defined as the set-point of the controller. The time steps of the digital control were set to 1d, meaning that the control calculations and actions take place once a day.

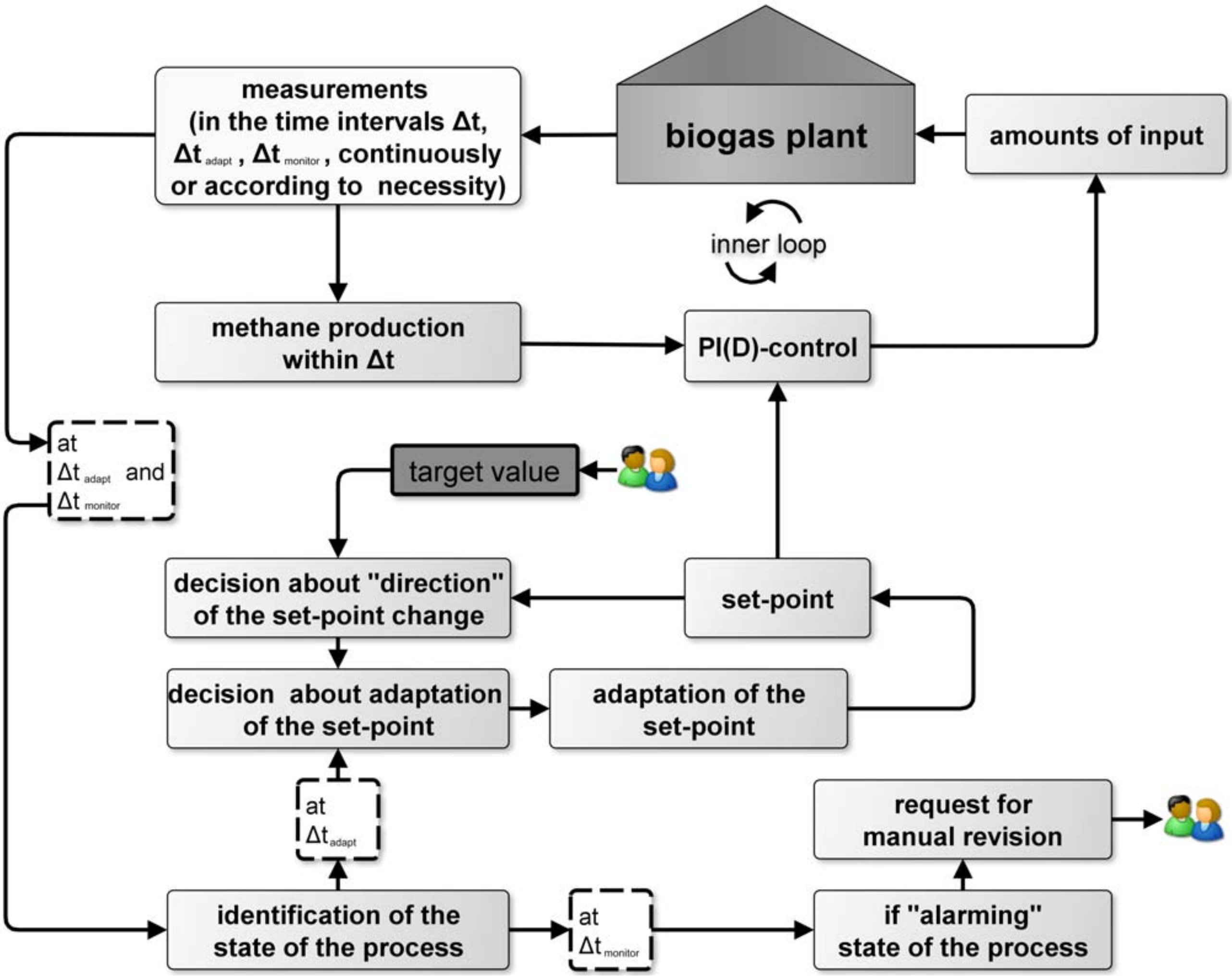

FIGURE 1 **Schematic draft of the control strategy (adapted from Kranert et al. (2011))**

As the process is highly complex and nonlinear, the methane production alone as exclusive parameter cannot provide enough information about the state of the process to enable such a control system to deal with all incidents. In particular, changes in the process, delayed reactions of the process, process disturbances and limitations can better and earlier be covered with sets of other process parameters. Therefore a knowledge-based part was constructed to guide and control the “conventional” control and to monitor the state of the process. Thus a combination of values of different process parameters was set up to classify the state of the process as “securely stable”, acceptable”, “critical” or “alarming”. Besides the commonly used parameter pH, which according to the state of the art can be measured online, the categorization of the state of the process is mainly based on information about the volatile fatty acids (VFA) as an intermediate product in the anaerobic reaction chain. In this context the relation between single acid concentrations as well as the concentrations or changes of the concentrations of single VFA or the sum of VFA are included. Within a research project at the University of Hohenheim the online determination of concentrations of single volatile fatty acids with near-Infrared-reflectance spectroscopy (NIRS) is investigated (see Stockl et al. (2011)). Resulting from the close cooperation to the research at the University of Hohenheim, the development of the control strategy was based on the assumption of direct availability of the concentrations of the VFA.

Additionally to the classification of the state of the process a target value for the methane production has to be defined by the operator.

Based on the information about the state of the process and the target value in relation to the actual set-point of the "conventional" control the knowledge-based system takes an automatic decision about raising, maintaining or lowering the set-point. The interval in which this adaptation mechanism for the set-point is executed needs to be long enough to allow the PI control to reach the set-point with sufficient accuracy. This time interval Δt_{adapt} and the step size of the set-point changes (Δset-point) are both set to fix values during the adjustment of the whole control strategy. For a secure monitoring the described categorization of the state of the process should take place as continuous as possible ($\Delta t_{monitor}$) in order to securely identify "alarming" states of the process. If this occurs the control system will emit an alarm, requesting manual revision. FIGURE 1 schematically shows the whole set-up of the control strategy.

3.2 Example of the simulation and practical operation of the control strategy

After the control strategy was tested and adjusted using the process simulations, operational experiments were conducted on 3 digesters. In the following one of these experiments will be explained. The experimental operation was realized on a laboratory-scale digester with a total volume of approximately 452 litres operated at a process temperature of 41 °C. The digester was fed daily with a fix mixture of 30 weight-% of cow manure and 70 weight-% of corn silage. The time interval Δt_{adapt} was set to 7 days, and the step size of Δset-point was adjusted to 50 l_N CH_4/d. The operation started at an initial organic loading rate of approximately 3 kg ODM/(m³*d), while the target value was set to 700 l_N CH_4/d. A digital PI control for the daily calculations (Δt = 1d) was used according to equations (1) and (2):

$$\varepsilon(t) = \text{set-point}(t) - \text{methane production}(t) \qquad \text{equation (1)}$$

$$u(t) = 2 * 6.9 / 3 * 10^{-6} * \varepsilon(t) + 12 * 6{,}9 / 3 * 10^{-6} * \Delta t * \sum_{i=1}^{t} \varepsilon(t) \qquad \text{equation (2)}$$

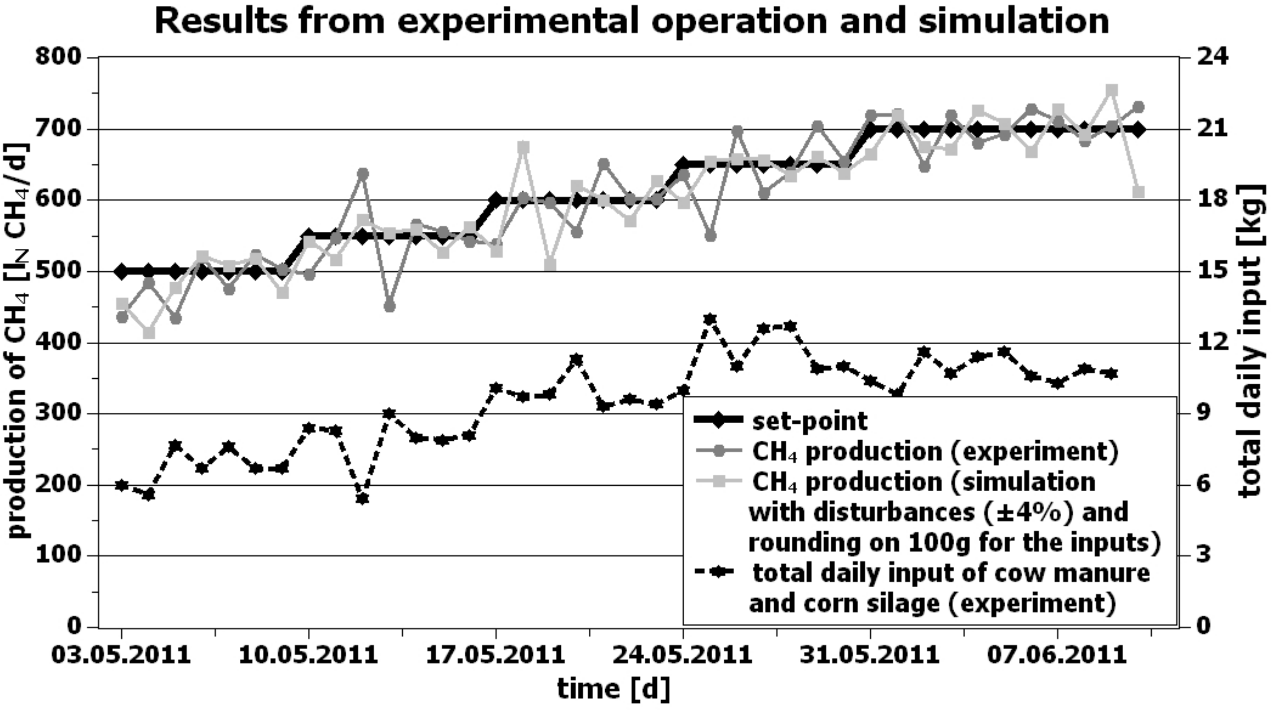

FIGURE 2 **Example of results from experimental operation and simulation (adapted from Löffler (2012))**

All the necessary process parameters for the identification of the state of the process were at least recorded once a week, some in smaller time intervals. This data was then used to classify the state of the process every week on the same day to correctly take a decision about raising, maintaining or lowering the actual set-point. Within the operation period of more than five weeks, the state of the process remained within the category "securely stable", except for the last week where it could only be classified as "acceptable". This means that at this point the set-point would not have been raised again. As the target value of 700 l_N CH_4/d was already reached by then, there was no intention to raise the set-point either.

FIGURE 2 shows the results of the experimental operation of the control strategy. To show the coherence with the simulations, the figure also includes the result of an analogous simulation. This simulation is additionally charged with randomly distributed errors, i.e. disturbances, within a range of 4 % around the original value for substrate characteristics, the methane production and the amount of feeding. Furthermore the amounts of input are rounded to an accuracy of 100 g in this simulation. As can be seen in FIGURE 2 the results of the simulation and the experimental operation are similarly located around the set-point. Consequently the simulation which includes practical conditions by the added errors leads to comparable results as the operation on the real digester. FIGURE 2 also shows that the methane production follows the set-point within a certain range of fluctuation, supposedly resulting from normal disturbances in practical operation. By raising the set-point according to the state of the process, the target value for the methane production was finally reached and kept. Additional information about the process simulations including disturbances and errors similar to real operation, as well as more about other experiments, the control strategy and its adjustment, especially concerning the adaptation matrix, can be found in Löffler (2012) and Kranert et al. (2011).

4 CONCLUSIONS

Different simulations and some operational experiments showed that the process can be conducted to a user-defined methane production by the developed control strategy. More information about the simulations, experiments and the interpretation of both is given in Löffler (2012) and Kranert et al. (2011). A detailed explanation and values for the adaptation matrix, which is only marginally described here, are also presented in Löffler (2012). With reasonable adjustments the control strategy finally proved its capability of working successfully, while disturbances within ranges commonly occurring in practical operation are tolerated. The experiments and simulations showed good results for an adjustment of Δset-point (in l_N CH_4/d) to approximately 10 % of the digester volume (in litres) and of Δt_{adapt} to 7 d. This accounts also for the simplification of the adjustment of the PI-control to only one adjustment factor (f_a) as shown in equation (3).

$$u(t) = 2 * f_a * \varepsilon(t) + 12 * f_a * \Delta t * \sum_{i=1}^{t} \varepsilon(t) \qquad \text{equation (3)}$$

Consequently the control strategy can be used to reach the goals mentioned in the introduction. Therefore it can for example be implemented to simplify and automate the operation of biogas plants or to adjust the methane production according to seasonal demands of energy.

Nevertheless the field of possible applications is limited to applications which comply with required boundary conditions. Due to the set-up of the control strategy the availability of feeding substrates necessarily has to be “free”. This means that the possibility to feed the process according to the demands of the control is essential for the control to properly work and to reach the defined target value. Further assumptions the development is based on are for example a constant process temperature or sufficient stirring. As the research was limited to using cow manure and corn silage as substrates so far, no adjustments for other substrates can be provided yet. Single changes of the feeding mixture (other substrate other feeding composition) are only tolerated to a certain degree. However the control strategy offers many possibilities for modifications and adaptations to adjust the original set-up to specific cases or different practical conditions.

Experiences of long-term and full-scale operation are still required to strengthen the drawn conclusions and to permit further improvement of the control strategy. Further research and experiences about measurement instrumentation, providing necessary data online-available, will be of importance for the development and implementation of automatic controls on biogas plants.

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(87) OPERATION STRATEGY OF GREASE AND MUNICIPAL SEWAGE SLUDGE CO-DIGESTION: RESULTS OF CONTINUOUS TRIALS

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EXECUTIVE SUMMARY

To this day, biogas production on wastewater treatment plant (WWTP) and use has not been optimised, as in many cases it was operated as a means of stabilising sludge, and not recovering energy. Furthermore, many sectors of activity lead to the production of different types of organic wastes. For these reasons, co-digestion strategies are considered a good way to enhance anaerobic digestion performances and the production of biogas (Mata-Álvarez et al., 2000). Grease is a waste produced on WWTP and usually disposed of. Furthermore, this waste is also produced by many other industries (biofuel, restaurants...) and therefore it is an interesting market opportunity. The availability on site, its high production, its biodegradability potential and previous works that have shown interesting results using this waste for sludge co-digestion (Bouchy et al., 2012) are the main reasons to select grease as a promising co-substrate. Several assays in batch mode have been carried out with sewage sludge and grease as co-substrate, showing that grease waste is an adequate co-substrate for anaerobic digestion of sewage sludge. However trials in continuous operation are necessary in order to verify biogas yields, the impact on the WWTP and dewatering performance. The aim of this study is to evaluate the co-digestion of sewage sludge and grease from WWTP in a continuous reactor operation.

The continuous experiments were carried out in two reactors of 20L of useful capacity and 10L of gas chamber. Both reactors were operated at mesophilic temperature range (35ºC), with a hydraulic retention time (HRT) of 20 days, and was conducted during 470 days. Biogas production was measured by a pulse electrical system and analyzed by gas chromatography. The stability of the operation was controlled carrying out periodical analysis of several parameters of the digestates such as solids content, total and soluble chemical oxygen demand (COD), volatile fatty acids, alkalinity, pH and ammonium. Initially the reactor start-up was carried out using WWTP digested sludge as inoculum. Then, the reactors were fed with primary and secondary thickened sludge, mixed in the same proportion in weight. Once the desired feed of sludge was reached, grease from the dissolved air flotation (DAF) unit of a WWTP was mixed with the mixed sludge to feed one of the reactors in a weight ratio of 15/85% respectively (33/66% COD ratio). This ratio was selected based on previous results carried out in batch mode (Bouchy et al., 2012). The operation of two reactors made possible the comparison between the effects of the co-digestion versus a digestion of mixed sludge alone.

The start-up of both reactors was carried out during 4 months to achieve the desired HRT (20 days) and was finished when all the parameters were stabilized and the desired organic loading rate, 2kgVS/m^{3}d, was reached. The average value for the methane potential of the mixed sludge was 220 NmLCH$_4$/kgVS$_{added}$. In the behaviour of the methane productivity curves once the digester is fed with the mixture grease/sludge it can be observed that the methane production increases with the addition of the co-substrate (grease), obtaining an increase in biogas production of almost 100%, reaching a final methane yield of 400 NmLCH$_4$/gVS$_{added}$. The better the acclimation of the biomass, the higher is the biogas production. Additional experiments were carried out in a second phase in order to study the limits of the process. With this objective, the Organic Loading Rate (OLR) was increased until reaching the operation limit of the digester. The HRT was highly decreased during these assays, reaching values lower than 10 days without significant operational problems. The process has shown a great robustness since it accepts high OLR (7 kgVS/m^{3}d) without significant perturbation, even at HRT lower than 9 days.

Therefore, a valuable waste such as grease offers interesting future perspectives for its application in real scale plants digesters, dealing with a problematic waste by a high energy recovery process, at the same time that waste managing costs are reduced.

1 INTRODUCTION

Anaerobic co-digestion process with sewage sludge is a non optimized procedure as means of biogas production and energy recovery. The increasing production of organic wastes and the need for technologies which enhance renewable energy and methane production have shown great interest for biological treatments, particularly anaerobic digestion since it has been established as the most cost-effective technology (Mata-Álvarez et al., 2000).

1.1 Background

More than 10 million tons dry matter of sewage sludge were produced in 2006 in the European Union (Laturnus et al., 2007), representing about 58 kg dry matter per inhabitant-equivalent and year (Silvestre et al., 2011). Sewage Sludge is typically composed of primary sludge and secondary sludge (waste activated sludge), of which the latest is more difficult to be digested (Wan et al., 2011). Anaerobic digestion of sewage sludge has been applied at wastewater treatments plants (WWTP) for decades. It is a well-known, efficient and environmentally sustainable technology which enables energy production as heat, electricity or vehicle fuel, as well as stabilization and volume reduction of sludge. Anaerobic co-digestion of sewage sludge and other organic wastes could enhance biogas production and organic matter degradation due to benefits such as diluting inhibitory compounds, increasing degradation of the treated material due to synergistic effects, optimal moisture and nutrient content, a more balanced carbon to nitrogen ratio, as well as increasing biogas production (Luostarinen et al., 2009; Mata-Álvarez et al., 2000).

The associated transport costs of co-substrates and the addition of new external wastes to the WWTP are two of the limiting factors for co-digestion. One possible option is to use intermediate waste generated in the WWTP, such as the grease waste trapped in the dissolved air flotation (DAF) unit. This would lead to an optimization of the entire plant, since the cost of managing the grease waste to landfill will be reduced, and its high fat content will increase biogas yield (Silvestre et al, 2011). Lipid-rich materials are known to have high methane potentials despite the inhibition that could be produced by their degradation products (long chain fatty acids LCFA). This effect has been proved reversible thus, the increasing consumption of acetate and butyrate will indicate the recovery of the biological system (Luostarinen et al., 2009). Also operational problems such as clogging, scum formation, and sludge flotation could be caused by the coating of the lipid layer around the sludge of biomass surface. Moreover, due to the adsorption of LCFAs to microbial surfaces, inhibition could be caused by the limitations of nutrient transport to cells (Wan et al., 2011).

Despite the likely inhibitory problems of grease, its availability on site and high productions, its high biodegradability potential and previous works that have shown interesting results using this waste for sludge co-digestion (Bouchy et al., 2012; Luostarinen et al., 2009) are the main reasons to select the grease as a promising co-substrate.

1.2 Research objectives

The aim of this paper is to evaluate the influence of co-digestion of sewage sludge and grease from WWTP in a continuous reactor operation, evaluating the increase in methane yield which is produced by the action of grease, optimizing the operational conditions for a co-digested mixture and testing the limit for a regular continuous operation without inhibition problems.

2 METHODOLOGY

2.1 Materials

Thickened primary and biological sludges were sampled from a municipal WWTP in Spain every week and kept refrigerated at 4ºC. Grease comes from the dissolved air flotation (DAF) unit of another WWTP located in Spain. The characterization of the substrates is shown in *Table 1.*

Co-digested mixtures of grease and sewage sludge have been previously studied (Silvestre et al., 2009) considering different volatile solids (VS) ratios of mixture: 4%, 23% and 37%. For the sewage sludge, thickened primary and biological sludges were mixed in a proper ratio (in that case 1:1 according to the common ratio in the WWTP) and then grease was added considering the selected mixture ratio based on VS. The results of the trials showed an increment of volumetric methane productivity for 4% and 23%VS inlet grease addition. Further increment of grease addition (37%VS) did not report an increase in volumetric methane production, then, the higher rate of VS inlet was selected for the present research with the objective of overcoming this limit. So, for this piece of work, the final grease waste content in the mixtures to be co-digested was established at 50%COD, which is equivalent to 40%VS and 15% weight basis.

TABLE 1 **Substrates characterisation**

Parameter	TS	VS	CODt	CODs	TKN	Fat content	VS/TS
Units	g/kg	g/kg	g/kg	g/kg	g/kg	% dry basis	%
Primary Sludge	186.4	106.3	209.3	3.62	4.58	-	57
Secondary Sludge	78.3	57.9	76.9	1.20	4.87	-	74
Grease	447.0	414.8	513.1	-	4.17	21.1	93

Nomenclature: TS/VS – total and volatile solids
CODt/CODs – total and soluble chemical oxygen demand
TKN – total Kjendhal nitrogen

2.2 Continuous digesters

The continuous co-digestion experiments were carried out in two cylindrical reactors of 20L of useful capacity and 10L of gas chamber. Both reactors were operated at mesophilic temperature range (35ºC±1ºC) and were conducted during 470 days. The biogas production was continuously measured by a pulse electrical system and analyzed by gas chromatography (Varian CP-3800). Biogas internal recycle assured a correct mixing in the reactor, avoiding stratification problems. Feeding was carried out once per day, setting a volume of feed according to the desired HRT. The stability of the operation was controlled carrying out periodical analysis (twice per week) of several parameters from the digestates such as solids content, total and soluble chemical oxygen demand (COD), volatile fatty acids, alkalinity, pH, and ammonium. *Figure 1* illustrates the reactors and the experimental set-up.

FIGURE 1 **Description of the experimental set-up used for the co-digestion studies**

2.3 Operation stages

Different phases were carried out in both reactors to evaluate and compare the influence of the co-digested mixture and OLR increases. *Table 2* shows all the stages and their characteristics. Both reactors were feed in a first phase with sewage sludge composed of 50% primary sludge and 50% secondary sludge. The volume of feed that was introduced in the reactor was increased progressively to achieve 20 days HRT which means 1L feed/day and an ORL of 2kgVS/m^{3}d. This phase was carried out during four months (Stage 0). In the next six months grease was introduced in Reactor 1, the proportion of grease was gradually increased to achieve the desired weight ratio of 15% grease. This increases the ORL Reactor 1 to 3kgVS/m^{3}d while Reactor 2 continues working only with sewage sludge maintaining the previous ORL (2kgVS/m^{3}d) (Stage 1). Next, Reactor 2 began to work with grease following the same standard than Reactor 1 in the previous phase to achieve gradually almost the same OLR (3.2kgVS/m^{3}d). Once ORL in both reactors were equal, the feed volume for Reactor 1 was increased to the limit, achieving an ORL of 7.5kgVS/m^{3}d with a final HRT of 9 days (Stage 2). The last 40 days the reactors were subjected to limits operational conditions. OLR continues to increase in

Reactor 1 until 9.3kgVS/m^{3}d for an HRT of 6 days, while Reactor 2 was suddenly cut of grease feeding, maintaining the same ORL with sewage sludge (3.2kgVS/m^{3}d) (Stage 3).

TABLE 2 **Operational stages of both reactors and feed characterisation**

STAGE	**REACTOR**	**Days**	**Co-digestion**		**CHARACTERISATION**			**HRT**	**OLR**
			Substrate	**% grease (w)**	**COD** g/L	**TS** g/L	**VS** g/L	days	kgVS/m^{3}d
0	R1 R2	150	Mixed sludge	0	70	65	40	200-20	0/2
1	R1	180	MSl. + Grease	15	100	75	60	20	3
	R2		Mixed sludge	0	70	65	40	20	2
2	R1	100	MSl. + Grease	15	100	85	65	20-9	3.5/7.5
	R2		MSl. + Grease	15	100	85	65	20	3.2
3	R1	40	MSl. + Grease	15	100	85	65	9-6	7.5/9.3
	R2		Mixed sludge	0	90	85	50	20	3.2

MSl – Mixed Sludge

2.4 Analytical methods

Standard methods (Apha, 2005) for reactors operation characterization were applied to determine the next parameters: TS/VS total and volatile solids; COD chemical oxygen demand; NH_4^+ ammonium; VFA volatile fatty acids; pH; alkalinity and alkalinity ratio. Biogas composition was analysed by gas chromatography (Varian CP-3800).

While TS/VS, COD and biogas yield parameters monitor digesters performance; pH, alkalinity, VFA and NH_4^+ warn of the reactor instability. pH values should not vary over 8 or below 7 to preserve the biological community in the digesters, while VFA and NH_4^+ concentration should maintain values not higher than 1000 mg/L (Kayhanian et al., 1994). If VFA level increases will mean a rise of the acids compounds (acetate, butyrate, propionate…). It is also important to maintain enough alkalinity to buffer the effects of the acids, according to which alkalinity should not fall below 1000 mgCaCO$_3$/L (Ağdağ et al., 2005). If the acids increase and drive the alkalinity level out of normal range, the digester may become distress.

3 RESULTS AND DISCUSSION

3.1 Start up

The start-up of both reactors was carried out during four months to achieve the desired HRT (20 days) and was finished when all the parameters were stabilized and the desired organic loading rate (2kgVS/m^{3}d) was reached in both reactors. During this period the feed volume of mixed sludge was gradually increased until 1L per day, which is the corresponding feed to set 20 days HRT and 2kgVS/m^{3}d OLR. This increase was planned to be slowly achieved in order to assure a correct and robust operation of the biological system. The monitoring parameters that have specially been taken into account were VFA levels, pH, and ammonium concentrations to avoid acidification of the media during this phase caused by an overload. Once a stationary behaviour was reached, the average value for the methane potential of the mixed sludge was 220 NmLCH$_4$/kgVS$_{added}$ and a biogas concentration of 60% CH$_4$.

3.2 Stage 1: co-digestion in R1

When Reactor 1 was then fed with the co-digested mixture of grease and mixed sludge, the next phase of the study got started. After some days as acclimation time, the methane productivity increased in Reactor 1, while Reactor 2, which was normally fed with mixed sludge, maintained the previous methane productivity: 220 NmLCH$_4$/gVS$_{added}$. Organic Loading Rates (OLR) were maintained in constant values; in Reactor 1 the OLR increased to 3 kgVS/m^{3}d by the addition of grease and Reactor 2 kept the same OLR as in previous stage (2 kgVS/m^{3}d). Nevertheless, methane productivities were determined per VS added unit, so this does not make any significant influence at the time to compare both behaviours. Methane productivity of Reactor 1 increased considerably after 100 days of operation with co-digestion, which means that the longer the acclimation of the biomass, the higher the biogas production. During the last phase of the study, the methane production even reached values over 400 NmLCH$_4$/gVS$_{added}$, which is almost double than the conventional digestion production. The results corresponding to this stage can be observed in *Figure 2.*

Biogas composition analysis showed an increase in methane concentration of Reactor 1 during the first 50 days 5% higher than Reactor 2 that was firstly attributed to the grease addition in the feed. However, after this period, both compositions became equal and methane percentage in biogas remained constant around 65%.

While grease was added to Reactor 1 feed, VS removal gradually decreased leading to an unstable operational phase. An ammonium overload took place reaching concentrations above 1100 mgNH$_4^+$/L, while, during the normal operation, ammonium levels oscillate between 600-800 mg/L. On the other hand, COD removal was maintained in 60% in both reactors during the whole phase of operation, which does not fit with the previous observations (see *Figure 2*).

During this stage, pH and VFA levels remained stable in both reactors: pH values stayed close to neutrality, without surpassing 8; and VFA concentrations remained under 1000 mg/L. This data confirm that acidification was not reached during the operation. Moreover, alkalinity content was high enough during all this period (over 3500 mgCaCO$_3$/L) to assure a buffer capacity of the media.

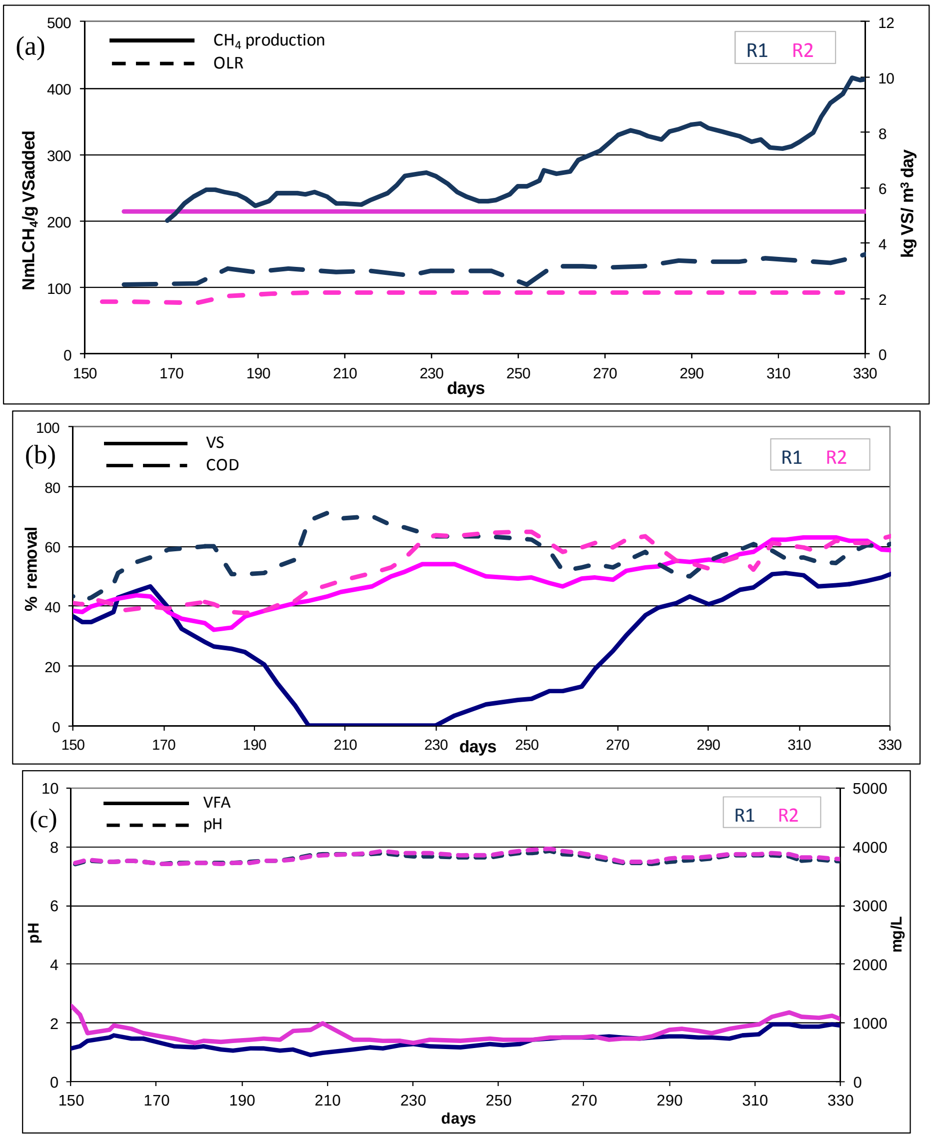

FIGURE 2 **Results from stage 1 (co-digestion in R1): (a) methane production and organic loading rate, (b) VS and COD removal ratio, (c) pH and VFA levels.**

Similar studies where grease and sewage sludge co-digestion was performed in continuous trials have obtained results of the same order (Davidsson et al., 2008), worked with a mixture of 30% (VS basis) grease trap sludge with sewage sludge at 13 days HRT and 2.4 kgVS/m^{3}d OLR in a 35L reactor and reached a methane yield of 344 NmLCH$_4$/gVS$_{added}$, which represented just a 27% increase. This methane yield is very similar to the one obtained in Reactor 1, but the increase regarding the conventional sludge digestion is much lower than the one of this study. The reason is probably due to the different biomethane potential of the substrates that are co-digested since their characterisation differs. Other studies (Davidsson et al., 2008; Luostarinen et al., 2009; Silvestre et al., 2011) obtained very similar values than the previous one, as summarized in *Table 3*.

TABLE 3 **Comparison on sewage sludge co-digestion studies**

Reference	**%grease (%VS)**	**HRT (days)**	**OLR ($kgVS/m^3d$)**	**Reactor volume(L)**	**Methane yield $NmlCH_4/gVS_{added}$**
Davidsson et al., 2008	30	13	2.4	35	344
Luostarinen et al., 2009	46	20	3.46	4	320
Silvestre et al., 2011	23	20	1.6	7	370

So the methane yield reached in the present study (400 $NmLCH_4/gVS_{added}$) can be considered a high value in comparison with other studies, which could be achieved thanks to the high BMP of the grease used (710 $NmLCH_4/gVS_{added}$ in batch tests) (Bouchy et al., 2012).

From all these results, it is deduced that a correct and stable operation in both reactors was carried out despite some parameters such as VS removal and ammonium concentration have shown unexpected behaviours during a period of time. The addition of grease as a co-substrate with mixed sludge has significantly improved the methane production in a continuous operation in laboratory scale; therefore it has interesting perspectives for a future application in larger scale experiments. However, further research needs to be done to assess the validation of this operation under these conditions. Then, in next stages, different modifications to this operation will be implemented to check the robustness of the co-digestion process.

3.3 Stages 2 and 3: load increase in Reactor 1 and sudden cut of grease in Reactor 2

Additional experiments were carried out in a second phase in order to study the limits of the process. With this objective, in a first phase the Organic Loading Rate (OLR) was increased continuously until reaching the operation limit of the digester Reactor 1 (during 100 days), while in Reactor 2 conventional co-digestion was carried out. In a second phase, a sudden cut of grease in Reactor 2 took place to evaluate the possible effects that a lack of co-substrate supply could have in a continuous operation.

In order to increase Reactor 1 OLR, the HRT was gradually decreased, reaching lower values than 10 days, by a higher addition of feed volume. No significant operational problems were observed during the first 70 days (until day 400), corresponding to an OLR below 7 $kgSV/m^3/d$ and a HRT over 9 days. During this phase, very similar values were obtained as in Stage 1 for Reactor 1 when co-digestion was carried out. VS and COD removal percentages remained around 50% and 60% respectively, pH about 7.5 and ammonium and VFA levels under 1000 mg/L. The results from these stages are represented in *Figure 3.*

A progressively higher overload in Reactor 1 (up to 9 $kgSV/m^3/d$, 6 days HRT) was then the cause of an unstable period from the day 400, showing an impact in several aspects of the operation that are subsequently described. Therefore, it can be said that the operational limits of the reactor was reached steadily, obtaining a critical OLR of 7 $kgSV/m^3d$ that cannot be exceeded if a stable operation is desired. The first symptom of instability was the rapid increase of VFA concentration in the reactor, reaching 3000 mg/L in less than 10 days, followed by a slight pH drop down to 7. Moreover, VS and COD removal percentages were affected, especially the former falling under 20%. Alkalinity values in Reactor 1 were maintained over 3000 $mgCaCO_3/L$ despite the acidification of the media, what could supply the neutralisation effect and avoid a pH drop. However, the alkalinity ratio, which indicates the relation between partial and total alkalinity, oscillated around 0.2 in previous stages and reached values close to 0.4, reflecting the perturbation effect of the overload.

Concerning the methane productivities, both curves presented similar but irregular behaviours (see *Figure 3*), following a decreasing tendency since the beginning of the second stage, possibly due to the feed characteristics. A progressive deviation of the curves of both reactors was then observed when OLR reached the critical value of 7 $kgSV/m^3/d$: while Reactor 2 was stabilized with a methane production of 300 $NmLCH_4/gVS_{added}$, Reactor 1's production remained in a lower value of 200 $NmLCH_4/gVS_{added}$. Although the methane production per unit of added VS is lower in Reactor 1 than in Reactor 2, the overall gross methane production is 50% higher in Reactor 1 (30 $NLCH_4/d$ versus 20 $NLCH_4/d$), which indicates that the overloaded process is less effective in terms of substrate elimination but the output to obtain per unit of reactor volume is higher.

Comparing with other studies where an increase of the organic load (or decrease of HRT) was tested, similar behaviours were observed, obtaining lower methane yields. (Luostarinen et al., 2009) decreased the HRT just by 4 days (from 20 to 16 days) and detected a decrease of the methane yield of 13% (from 320 to 278 $NmLCH_4/gVS_{added}$). Furthermore, (Silvestre et al., 2011) increased the OLR from 1.6 to 1.7 $kgSV/m^3/d$ and found a methane yield 11% lower (from 370 to 331 $NmLCH_4/gVS_{added}$). However, it has to be highlighted that in the present study the overload in Reactor 1 has been much more accentuated since OLR has been increased by 210% and HRT dropped till 6 days, then the methane yield reduction was stronger (33%).

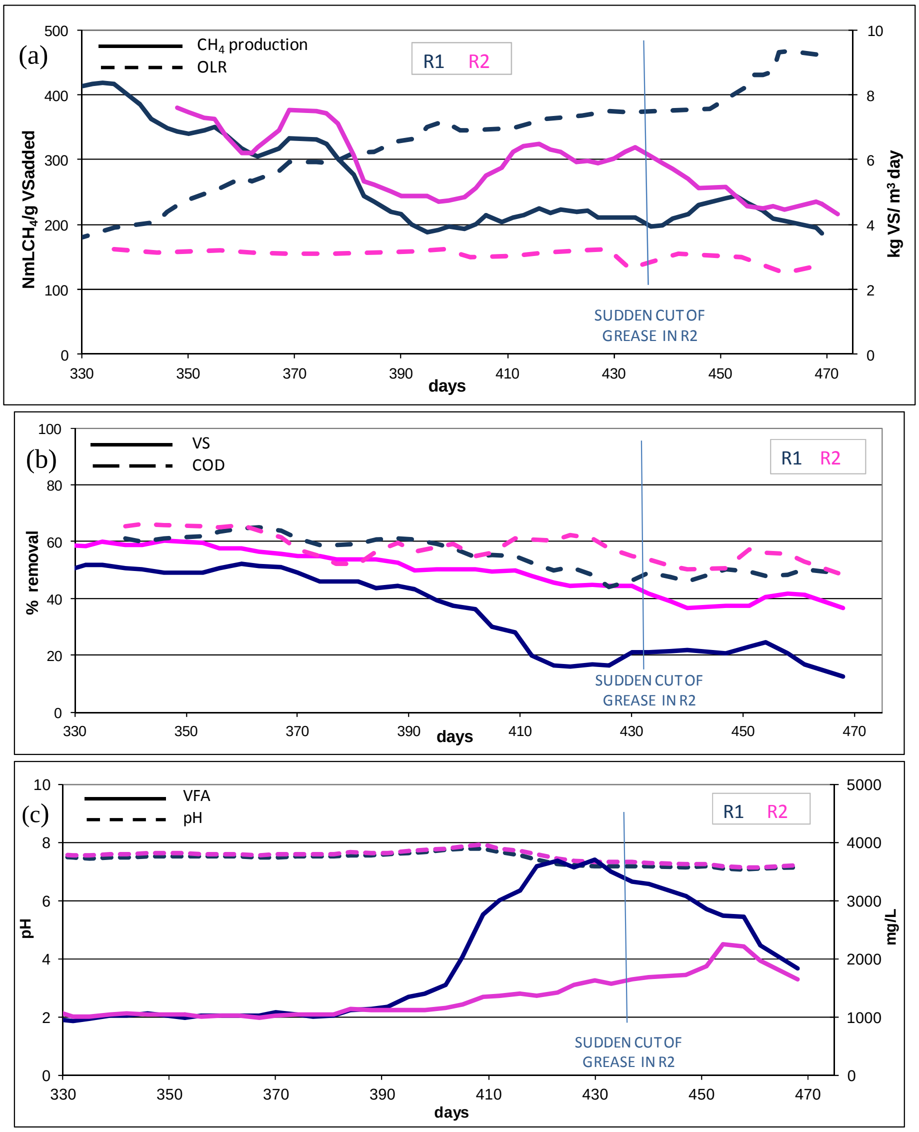

FIGURE 3 **Results from stages 2 and 3 (overloading in R1, cut of grease in R2): (a) methane production and organic loading rate, (b) VS and COD removal ratio, (c) pH and VFA levels**

In a second phase, from the day 435, a sudden cut of grease co-substrate in Reactor 2's feed was evaluated during the last 40 days of experience, maintaining the same OLR but only with mixed sludge (see *Figure 3*). Reactor 2 presented a stable and normal co-digestion operation with an adapted media, and since the grease cut was done, the methane production dropped suddenly from 300 $NmLCH_4/gVS_{added}$ until values close to 200 $NmLCH_4/gVS_{added}$. This fact indicated that the effect of grease as co-substrate in the digestion process is a key factor to achieve an extra methane production yield. Furthermore, other parameters were affected by this change: ammonium levels rose up to 1400 mg/L; the biogas composition decreased its methane content close to 60%, which can be explained by the fact that substrates rich in fat provide higher methane content in the biogas. Therefore, it could be appreciate that a traditional digestion of mixed sludge is less effective concerning methane yields than the co-digestion of grease and sludge, at the same time as it deals with a waste problem disposal in WWTP. Moreover, it can be also deduced that a sudden lack of grease co-substrate does not cause any unstability operation in the anaerobic reactor.

4 CONCLUSIONS

Anaerobic co-digestion of grease and mixed sludge, both from WWTPs, has shown interesting results for continuous operation in a laboratory pilot scale plant. An increase in biogas production (almost 100%) was firstly achieved by the addition of grease co-substrate in a conventional sludge digester, reaching a final methane yield of 400 $NmLCH_4/gVS_{added}$. Furthermore, the process has shown a great robustness since it accepts high OLR (7 $kgVS/m^3d$) without significant perturbation, what means HRT lower than 9 days, thus a reactor volume much more optimised. Therefore, a valuable waste such as grease offers interesting future perspectives for its application in real scale plants digesters, dealing with a problematic waste by a high energy recovery process, at the same time that waste managing costs are reduced.

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(154) EFFECT OF AMMONIACAL NITROGEN ON METHANOGENIC METABOLIC PATHWAYS DURING MSW ANAEROBIC DIGESTION

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EXECUTIVE SUMMARY

Municipal solid waste (MSW) represents an important renewable energy sources and the sustainable management of organic wastes is a major environmental and economic issue. The anaerobic digestion of MSW, which occurs in landfills and methanization treatment plants, is a very complex process. The final step of the waste degradation, *i.e.* methanogenesis, which produces methane, leads to the production of a biogas that could be transformed into heat, electric power and fuel gas (renewable energy). In order to produce more efficiently this renewable energy and to enhance the degradation of the organic fraction of waste, bioreactors landfill and methanization treatment plants are two promising management strategies. However, these new strategies induce new processes failure. One of those is the accumulation of ammonia produced from nitrogeneous compounds. The reason is that there is no efficient ammonia elimination process under anaerobic conditions.

An acetate-adapted landfill leachate microbial community was incubated anaerobically under mesophilic conditions (35°C) with acetate (3.3 $g.L^{-1}$) at four different ammonia concentrations (0.180 $g.L^{-1}$ to 5.4 $g.L^{-1}$). Standard physicochemical parameters were monitored together with CH_4 and CO_2 stable carbon isotopic signatures. As methanogenesis from H_2/CO_2 results in a larger fractionation against ^{13}C than acetoclastic methanogenesis, the use of stable isotopic signature of CH_4 ($\delta^{13}CH_4$) and CO_2 ($\delta^{13}CO_2$) can then allow to determine by which methanogenic pathways methane is produced. The objective of this study is to monitor methanogenic metabolic changes during methanization of acetate at various ammonia concentrations using the natural stable carbon isotopic signature of methane and carbon dioxide.

Using this new tool, combined with standard physico-chemical monitoring, the results obtained in this work show that a sudden increase of ammoniacal nitrogen concentration significantly influenced the methanogenic process of an acetate-adapted-landfill inoculum. Indeed, ammonia concentration increase leads to various effects. CH_4 stable carbon isotope composition evolution indicates a shift from the acetoclastic to hydrogenotrophic methanogenic pathway in function of the ammonia concentration. These results demonstrate that at high ammonia concentration acetate is consumed first by acetate oxidation followed by hydrogenotrophic methanogenesis (this reaction is then called syntrophic acetate oxidation). Ammonia concentration could then induce inhibition of acetoclastic methanogenesis. Even if the quantity of methane produced was the same at the end of all incubations, reaction rates of methane production were lower when acetate was oxidized first.

This work underlines the potential of the approach using carbon stable isotope measurement to study methanogenic processes. Indeed, isotopic approach could be a very useful tool to study AD process at different studies scales (i.e. laboratory and operational) as it allows a rapid and accurate monitoring of metabolic pathways involved during methanogenesis. A combination with microbial community structure analysis (using molecular microbiology techniques) could be very powerful to obtain a better understanding of methanogenic reactions.

1 INTRODUCTION

The final step of the waste degradation, *i.e.* methanogenesis, which produces methane, leads to the production of a biogas that could be transformed into heat, electric power and fuel gas (renewable energy). In order to produce more efficiently this renewable energy from the MSW and to enhance the degradation of the organic fraction of waste, bioreactors landfill and methanization treatment plants are two promising management strategies. However, these two processes can lead to various operating failures. One of those is the accumulation of ammonia produced from nitrogeneous compounds majoritarly in the form of proteins and urea (Kayhanian 1999).

The reason is that there is no efficient ammonia elimination process under anaerobic conditions. High Ammoniacal nitrogen concentration could then induce methanogenic inhibition (Kayhanian 1994; Wens, Vercauteren et al. 2001). Indeed, ammoniacal nitrogen concentration can reach high level in landfill leachate (3421 mg NH_3+NH_4^+/L) (Wens, Vercauteren et al. 2001)) and in digesters (4 g/kg) (Illmer and Gstraunthaler 2009).

The anaerobic digestion of MSW is a very complex process which involves numerous reactions and communities of microorganisms. MSW degradation is divided in four main successive steps, i.e. hydrolysis, acidogenesis, acetogenesis and methanogenesis. During this final step, fermentation products (especially acetate, hydrogen and carbon dioxide) are the main precursors of methane (Welander and Metcalf 2005) that can be converted directly to methane according to the following reactions (reactions 1 and 2) in TABLE 1 (Thauer, Jungermann et al. 1977; Hattori 2008). But, in reality, methanogenesis can be more complicated as reaction such as homoacetogenesis (reaction 3) and acetate oxidation (reaction 4) can occur. The first one consumes hydrogen and carbon dioxide to produce acetate and the second one consumes acetate to produce hydrogen and carbon dioxide.

TABLE 1 Methanogenic reactions.

Reactants	Products	ΔG^0 (kJ/mol)	Process
(1) $4H_2 + HCO^-_3 + H^+$	$CH_4 + 3H_2O$	-135.6	Hydrogenotrophic methanogenesis
(2) $CH_3COO^- + H_2O$	$CH_4 + HCO^-_3$	-31	Aceticlastic methanogenesis
(3) $4H_2 + 2HCO^-_3 + H^+$	$CH_3COO^- + 4H_2O$	-104.6	Homoacetogenesis
(4) $CH_3COO^- + 4H_2O$	$4H_2 + HCO^-_3 + HCO^-_3 + H^+$	104.6	Acetate oxidation
(5) $CH_3COO^- + H_2O$	$CH_4 + HCO^-_3$	-31	Syntrophic acetate oxidation [Sum (4) + (1)]

As underligned previously, reaction of acetate oxidation can take place in anaerobic environment, depending on environmental conditions. This reaction is called syntrophic acetate oxidation (SAO) as it has to be coupled with hydrogenotrophic methanogenesis to be energetically possible (Shigematsu, Tang et al. 2004). This reaction has been reported during methane formation in biogas reactors, particularly when sludges containing high levels of salts (mainly ammonium) were degraded in mesophilic conditions (Schnurer, Houwen et al. 1994; Schnurer, Zellner et al. 1999; Karakashev, Batstone et al. 2006; Schnurer and Nordberg 2008) and also in thermophilic conditions (Petersen and Ahring 1991).

During the anaerobic digestion of MSW, ammoniacal nitrogen is produced (proteins and urea). Free ammonia (NH_3) and ammonium ion (NH_4^+) are the two principal forms of inorganic nitrogen in aqueous solution. Methanogens are known to be sensitive to inhibition by ammonia (Kayhanian 1994) but it is still not clearly establish if it is acetoclastic or hydrogenotrophic methanogens that are the most affected.

Methanogenesis from H_2/CO_2 results in a larger fractionation against ^{13}C, and thus in lower CH_4 stable isotopic signature than acetoclastic methanogenesis. The use of stable isotopic signature of CH_4 ($\delta^{13}CH_4$) and CO_2 ($\delta^{13}CO_2$) can then allow to determine by which methanogenic pathways methane is produced (Conrad 2005). This isotopic approach has already been used to investigate methane generation pathways in complex environmental systems such as soil, rice field soil and lake sediment (Chidthaisong, Chin et al. 2002; Fey, Claus et al. 2004; Chan, Claus et al. 2005) and recently during anaerobic digestion of MSW (Qu, Mazeas et al. 2008).

The objective of this study is then to monitor methanogenic metabolic pathways modifications during methanization of acetate at various ammoniacal nitrogen concentrations using the natural stable carbon isotopic signature of methane and carbon dioxide generated.

2 MATERIALS AND METHODS

2.1 Experimental system

In order to obtain an acetate acclimated inoculum a first series of incubation was launched in anaerobic bioreactors. Anaerobic bioreactors (1.1 L graduated glass bottles) were hermetically closed with a screw cap and a septum (Fischer Scientific Bioblock, Illkrich, France) enabling leachate and biogas sampling with syringe and needle. In those incubations (named LVG A1 to A3), pellets obtained after centrifugation (5,000*g under 4°C during fifteen minutes) of 500 mL of leachate originating from a French municipal solid waste landfill (Vert-le-Grand, France) was added in the reactors as inoculum. They were incubated in 1.1 L glass bottles with 500 mL of liquid medium. To prevent the acidification due to the addition of acetic acid, the liquid medium was constituted of a buffer solution ($NaHCO_3$, 0.14 $mol.L^{-1}$) supplemented with a salt and vitamin nutrient solution adapted to anaerobic methanogenic population growth (Afnor 1998). The entire composition of the liquid medium is given in TABLE 2. Glacial acetic acid (CH_3COOH, 99%, Sigma Aldrich) was then added to the liquid medium to obtain a concentration of 3.0 $g.L^{-1}$ of CH_3COOH. At the beginning of each incubation, the headspace was purged with a neutral gas (N_2) to obtain less than 0.3% of O_2 and the reactors were incubated in the dark at 35°C ± 2°C. This experiment was performed in triplicate. Control reactors (named LVG T1 to T3) containing only the inocula and the liquid medium were launched to measure the background methane production.

TABLE 2 Composition of the liquid medium.

Liquid medium	
Anhydrous potassium dihydrogenphosphate (KH_2PO_4)	0.27 g
Disodium hydrogenphosphate dodecahydrate ($Na_2HPO_4.12H_2O$)	1.12g
Ammonium chloride (NH_4Cl)	0.53 g
Calcium chloride dihydrate ($CaCl_2.2H_2O$)	0.075 g
Magnesium chloride hexahydrate ($MgCl_2.6H_2O$)	0.10 g
Iron(II) chloride tetrahydrate ($FeCl_2.4H_2O$)	0.02 g
Sodium sulfide nonahydrate ($Na_2S.9H_2O$)	0.1 g
Stock solution of trace elements	10 mL
Water (Distilled water)	to 1 L

Stock solution of trace elements	
Manganese chloride tetrahydrate ($MnCl_2.4H_2O$)	0.05 g
Boric acid (H_3BO_3)	0.005 g
Zinc chloride ($ZnCl_2$)	0.005 g
Copper(II) chloride ($CuCl_2$)	0.003 g
Disodium molybdate dihydrate ($Na_2MoO_4.2H_2O$)	0.001 g
Cobalt chloride hexahydrate ($CoCl_2.6H_2O$)	0.1 g
Nickel chloride hexahydrate (NiCl2.6H2O)	0.01 g
Disodium selenite (Na_2SeO_3)	0.005 g
Water (Distilled water)	to 1 L

When acetate has been consumed, liquid mediums of the triplicates containing inoculum (adapted to consumption of acetic acid) were mixed together and shaked. Then liquid phase was separated in four equal's parts for adjustment of NH_4^+ concentration (C1: 0.180 $g.L^{-1}$ the same concentration as previously in LVG-A; C2: 1.44 $g.L^{-1}$; C3: 3.6 $g.L^{-1}$; C4: 5.4 $g.L^{-1}$) with ammonium chloride (NH_4Cl). For each concentrations of NH_4^+, 100 mL of liquid medium were introduced in 3 glass bottles of 330 mL. In each triplicates, acetate concentration was adjusted to 3.3 g/L. Then the headspace was purged with a neutral gas (N_2) to obtain less than 0.3% of O_2 and reactors were incubated in the dark at 35°C ± 2°C. The name and incubation conditions of all reactors are synthesized in TABLE 3.

TABLE 3 Initial incubations conditions.

Incubations	Substrate	[Substrate]	Initial volume of incubation	[NH_4^+]
LVG-T (1,2 & 3)	Ø	Ø	500 mL	0.180 g/L
LVG-A (1,2 & 3)	Acetate	3.0 g/L	500 mL	0.180 g/L
C1 (1,2 & 3)	Acetate	3.3 g/L	100 mL	0.180 g/L
C2 (1,2 & 3)	Acetate	3.3 g/L	100 mL	1.440 g/L
C3 (1,2 & 3)	Acetate	3.3 g/L	100 mL	3.600 g/L
C4 (1,2 & 3)	Acetate	3.3 g/L	100 mL	5.400 g/L

2.2 Analytical methods

2.2.1 Gas production

Bioreactors pressure was monitored with a differential manometer (2082P, Digitron). When the pressure reached more than 300 mbar, the gas composition in the headspace of the bioreactors was analysed by a gas chromatograph (µGC CP4900, Varian) connected with a needle through the septum of the bottle. The gas chromatograph was equipped with four parallel chromatographic columns connected with thermal conductivity detectors. The detection limit for all gases was < 0.1%. The measurement conditions were the same as those described previously (Vigneron, Ponthieu et al. 2007). To measure gas volume, gas headspace was equilibrated at atmospheric pressure and ambient temperature with a gas syringe (30 mL ± 1 mL or 100 mL ± 4 mL).

2.2.2 Stable carbon isotope signature

Gas samples for analysis of $\delta^{13}C$ of CH_4 and $\delta^{13}C$ of CO_2 were collected by a 10 mL gas syringe (SGE Analytical Science), transferred into 5-mL vacuum serum tubes and stored for later isotopic analysis. The analyses were performed using Trace GC Ultra (Thermo Electron Corporation) attached to a Delta V plus isotope ratio mass spectrometer via a Finnigan GC combustion III (Thermo Electron Corporation). Typical uncertainties, quantified by replicate measurements of different samples, for $\delta^{13}C$ of CH_4 and $\delta^{13}C$ of CO_2 were ± 0.2‰.

2.2.3 Leachate characteristics

Periodically, leachate samples were collected through the septum of the bioreactors with a syringe fitted with a 0.7-mm needle. Raw samples were centrifuged at 13400 rpm for 10 min. The supernatants and pellets were separated and stored at -20°C. From supernatants stored at -20°C, acetic acid concentration were analysed by ion chromatography (DIONEX DX-120).

2.2.4 Calculations

To measure gas production, the biogas is assimilated to an ideal gas. Cumulated gas productions were calculated according to the difference of each gas amount in both the discharged gas and the gas in the headspace of the bioreactor between two sampling dates.

The apparent carbon isotope fractionation (α_C) was calculated for all the reactors according to the following formula proposed by Whiticar et al. (Whiticar, Faber et al. 1986).

$$\alpha_C = {\delta^{13}CO_2 + 1000} \Big/ {\delta^{13}CH_4 + 1000} \qquad (1)$$

When α_C is higher than 1.065, it is generally concluded that methane is produced from H_2 and CO_2. When α_C is lower than 1.055, methane is produced through acetoclastic metabolism. And between, methane is produced by both metabolisms.

2.2.5 Characterizing cumulated methane production

Cumulated methane production curves are modelled using a modified Gompertz growth equation (Zwietering, Jongenburger et al. 1990; Lay, Li et al. 1998; Lay, Li et al. 1998; El Hadj, Astals et al. 2009; Zhu, Gikas et al. 2009).

$$Pcm(t) = Pcm(\infty) \times \exp\left(-\exp\left(\frac{Rm \times e}{Pcm(\infty)} \times (\lambda - t) \right) + 1 \right) \qquad (2)$$

Where Pcm(∞) corresponds to the cumulated methane production at the end of the experiment (mL.g^{-1} STP); Rm, maximum rate of methane production (mL.g^{-1} STP.day^{-1}) ; e, exponential of 1 ; λ, latency (day). Parameters were fitted using Excel solver. The parameters λ, Vm et Pcm(∞) are thus used to characterize and compare the different curves.

3. RESULTS

All the results below are given under Standard Temperature and Pressure (STP) conditions. No methane production was recorded after more than 100 days in control reactors (LVG T) (containing only the inoculated liquid medium).

3.1 Acclimatation of the inoculum to acetate consumption

The landfill leachate inoculum used in this study has been first acclimatized in order to enhance its ability to degrade acetate. Active methane production started after an important lag phase of 65 days (TABLE 4, LGV), during which methanogens originating from a French municipal solid waste landfill leachate had to adapt itselves to the bioreactor environment (new liquid medium,…) and the initial acetic acid concentration (3.0 g.L^{-1}). The active methane production proceeded for about 15 days with an approximately maximum rate of methane production of 30 mL of CH_4 .g^{-1} of CH_3COOH .day^{-1} (TABLE 4).

The depletion of acetic acid induced the decrease of CH_4 production rate to a residual level. At the end of the experimental period, the cumulated methane production was about 360 mL per gram of acetic acid (TABLE 4) which represents 96 % of the expected production (373.5 mL). Analyses of stable isotopic signature evolutions (unpublished data) lead to a production of methane mainly by acetoclastic methanogenesis. This acetate adapted landfill leachate was used to study the effect of ammonia on methanogenic metabolic pathways.

TABLE 4 Gompertz parameters.
Pcm(∞): cumulated methane production at the end of the experiment (mL.g^{-1} STP); Rm: maximum rate of methane production (mL.g^{-1} STP.day^{-1}); λ: latency (day). See equation 2.

Incubations	P			Rm			λ		
	(mL of CH_4.g^{-1} of CH_3COOH)			*(mL of CH_4 .g^{-1} of CH_3COOH .day^{-1})*			*(days)*		
LVG	360,0	±	1,4	30,1	±	0,6	65,3	±	0,4
C1	337,4	±	1,6	54,9	±	3,0	5,5	±	0,1
C2	339,6	±	6,3	28,1	±	3,0	8,0	±	0,2
C3	320,6	±	5,6	7,5	±	0,2	42,3	±	3,3
C4	328,7	±	6,5	18,9	±	2,4	75,3	±	0,9

3.2 Effect of ammonia concentration on acetate methanization

3.2.1 Effect on methane production kinetic

During acetate incubations (initial concentration 3 g.L^{-1}) with the acetate adapted inoculum obtained at the end of the previous experiments the evolutions of cumulated methane production and acetate concentration were significantly different in function of the ammoniacal nitrogen concentration introduced.

For the two lowest ammoniacal nitrogen concentrations (C1 (0.180 g.L^{-1}) and C2 (1.44 g.L^{-1})), active methane production started before day 10 and reach 340 mL of CH_4 .g^{-1} of CH_3COOH rapidly (TABLE 4, C1 and C2)). Curves of acetate consumption were inversely correlated to cumulated methane production curves. Some differences could be observed between C1 and C2 bioreactors. Latency is a little shorter and maximum rate of methane production was approximately 2 times higher for C1 than for C2. Methane production for the third ammoniacal nitrogen concentration started after an important lag phase of 42 days (TABLE 4, C3). The active methane production proceeded for about 50 days with a low maximum rate of methane production of 7.5 mL of CH_4 .g^{-1} of CH_3COOH .day^{-1}. At the end of the experimental period, the cumulated methane production was about 320 mL per gram of acetic acid. Finally, methane production for the highest ammoniacal nitrogen concentration (C4 (5.4 g.L^{-1})) started after the most important lag phase (75 days (TABLE 4, C4)). The active methane production proceeded for about 20 days with a maximum rate of methane production of 18.9 mL of CH_4 .g^{-1} of CH_3COOH .day^{-1}. The depletion of acetic acid induced the decrease of CH_4 production to a residual level. At the end of the experimental period, the cumulated methane production was about 328 mL per g of acetic acid (TABLE 4). Theoretically, the cumulated methane production was 373.5 mL at STP conditions. The difference between experimental and theoretical values is probably due to the fact that some acetate was removed from the incubations each time liquid sampling was performed.

3.2.2 Effect on methanogenic metabolic pathways

For the two lowest ammoniacal nitrogen concentrations, the apparent fractionation factor α_C (calculated using equation 1) initially started around 1.025 and remained at this level during all the production of methane (FIG 1). Methane appears then to be produced by the acetoclastic pathway in those incubations. With the third concentration of ammoniacal nitrogen α_C was first at 1.030 and then increased to 1.055 suggesting a shift from the acetoclastic to the hydrogenothrophic pathway during CH_4 production (FIG 1). And for the reactors incubated with the highest ammonial nitrogen concentration, α_C initially start around a value of 1.065 and remained around this level during all the production of methane (FIG 1). Methane appears to be produced mainly by the hydrogenotrophic pathway in those incubations.

4. DISCUSSION

The results obtained in this work clearly show that ammoniacal nitrogen concentration significantly influences the methanogenic process (i.e. latency and rate of methane production).

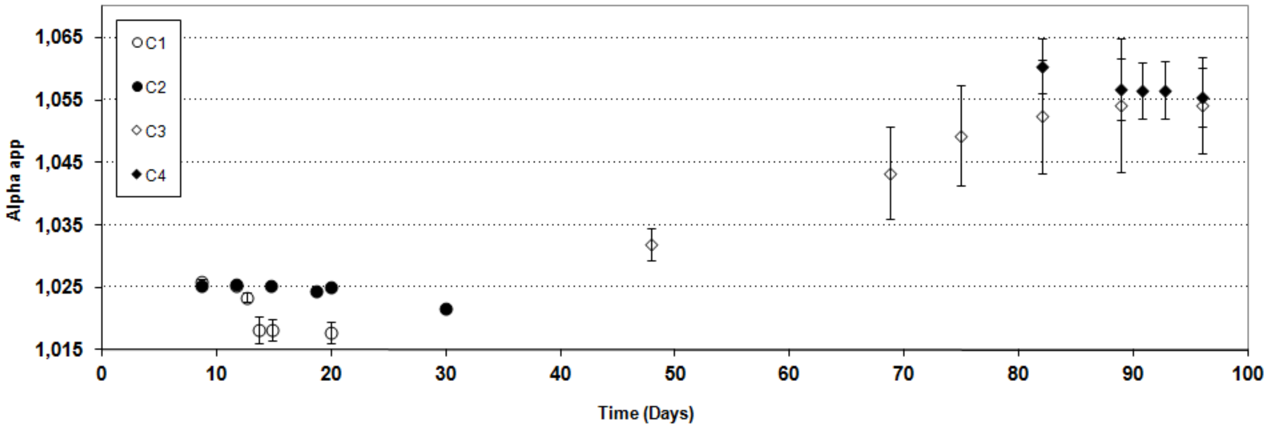

FIGURE 1 **Evolution of apparent fractionation factor (α_C) of C1 to C4 bioreactors. Vertical lines indicate standard error of the mean for 2 bioreactors (bioreactors 1 and 2).**

It is first to note that latency time increases dramatically with increasing ammonia concentration (5.5 to 75.3 days). The isotopic approach applied in this study gives some new informations allowing to explain this inhibitory effect of ammoniacal nitrogen. Indeed for the two lowest ammoniacal nitrogen concentrations methane is produced by acetoclastic metabolism as it was during the acclimatisation experiments. Nearly no latency is then observed. For the highest concentration it appears that a metabolic shift towards the hydrogenotrophic pathway occurred. An important latency period was then observed. During this latency period, the microbial community originating from the acclimated inoculum had to change its metabolism to adapt to the initial higher ammoniacal nitrogen concentration. Secondly, maximum rate of methane production decrease with increasing ammonia concentration (54.9 to 18.9 mL of $CH_4.g^{-1}$ of $CH_3COOH.day^{-1}$). We have to notice that the maximum rate of methane production (7.5 mL of $CH_4.g^{-1}$ of $CH_3COOH.day^{-1}$) for the third concentration (3.6 $g.L^{-1}$) was inferior than the one determined for the highest one (5.4 $g.L^{-1}$) (18.9 mL of $CH_4.g^{-1}$ of $CH_3COOH.day^{-1}$). This phenomenon can be explained by the fact that the metabolic shift (from acetoclastic to hydrogenotrophic) occurred during the incubation for the third concentration when for the highest concentration the methane production start after a long latency period directly through the hydrogenotrophic metabolism. Finally, it is interesting to note that no ammoniacal nitrogen effect was measured on the total methane production which was the same at all concentrations (approximately 330 mL of $CH_4.g^{-1}$ of CH_3COOH).
Using the isotopic approach we then get some new insights on the effect of ammoniacal nitrogen on methanogenesis. It clearly appears that acetoclastic methanogenic pathway was inhibited by increasing ammoniacal nitrogen concentration. At high ammoniacal nitrogen concentration, the hydrogenotrophic pathway became the dominant one. These results are in agreement with literature which underlines the sensitivity of acetoclastic methanogens to ammonia (Chen, Cheng et al. 2008). The consumption of acetate at high ammoniacal nitrogen concentration occurred then through synthrophic acetate oxidation. This reaction takes place in two steps. First acetate is oxidized by bacteria to CO_2 and H_2 and then methane is produced by archaea methanogens through the hydrogenotrophic pathway. The fact that this reaction requires a synthrophic relationship between some bacteria and archaea can explain why some important latency periods may appear when ammoniacal nitrogen concentration suddenly increases.

5 CONCLUSION

Using this new isotopic tool, combined with standard physico-chemical monitoring, the results obtained in this work showed that a sudden increase of ammonia concentration significantly influenced the methanogenic process of an acetate-adapted-landfill inoculum. Indeed, ammonia concentration increase leads to:

- an increase of the latency period before start of the methane production ;
- a decrease of the maximum methane production rate ;
- no change in methane yield at the end of incubation ;

Those effects appear to be due to some modifications of metabolic pathways (shift to a reaction of acetate oxidation followed by a CO_2-reduction at high ammonia concentrations).

This work underlined the potential of the approach using carbon stable isotope measurement to study methanogenic systems. Indeed, this method gives new insights on the dynamic of the metabolic pathways involved during methanogenic reactions. Isotopic approach could be a very useful tool to study AD process at different studies scales (i.e. laboratory and operational) as it allows a rapid and accurate monitoring of evolution of metabolic pathways involved during methanogenesis. A combination with analysis of microbial community structure (using molecular microbiology techniques) could be very powerful to obtain a better understanding of methanogenesis reaction.

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(181) REMOVAL OF AZO DYE FROM TEXTILE WASTEWATERS USING ANAEROBIC BIOMASS: OPTIMIZATION AND EXPERIMENTAL DESIGN

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EXECUTIVE SUMMARY

Textile industry includes highly coloured wastewaters. Common groups of dyes in textile industry, azo dyes (60-70%), are characterized by their typical -N=N- nature. In general, dyes are not easily degradable and are not easily removed from wastewaters by conventional wastewater treatment systems. Therefore, physico-chemical methods such as adsorption, coagulation-flocculation, membrane filtration and advanced oxidation, can be used effectively for color removal from wastewaters. Recently, biosorption process is one of the effective and attractive processes for treatment of dye-bearing wastewaters. Therefore, low-cost biosorbents such as yeast, algae, bacteria and biowaste materials were used for dye removal from wastewaters.

In this study, batch experiments were optimized as a function of pH, initial dye concentration (Co: mg/l), contact time (tc: min.) and biosorbent amount (m: g/l)) using a four factor, three level experimental design combining with response surface modelling (RSM) for MRGRL azo dye removal from aqueous solutions by using dried waste anaerobic sludge (AS) taken from anaerobic digester.

The optimum experimental conditions were found to be pH= 8, Co= 112.5 mg/l, tc=95 min and m=15 g/l and corresponding MRGRL removal was determined as about 97%. Pseudo-second order model was suitable for biosorption kinetics.

Waste anaerobic sludge, one of the low-cost biosorbent, can be used for azo dye removal from textile wastewaters.

1. INTRODUCTION

Effluents from the textile industry contain various kinds of synthetic dyes and textile industry is the largest user of synthetic dyes (Murugesan et al., 2007). Azo dyes which are mostly used in textile industry and based on the azo chromogen (–N=N–) are presently the most important compounds constituting about 20–40% of the total dyes used for coloring (Beydilli et al., 2000; Sharma et al., 2009) Azo dyes are readily decolorized by degradation under anaerobic conditions before aerobic methods (Sarioglu et al., 2007).

Some dyes and their breakdown products may be toxic and even carcinogenic towards aquatic living organisms. If discharged in environment without any treatment, they are highly harmful. Because of chemical stability and complexity of them conventional wastewater treatment technologies have shown to be mostly ineffective to remove color from wastewater (Forgacs et al., 2004). In general, the treatment of dye-bearing effluents is being undertaken by various methods, including biological processes, combined chemical and biochemical processes, chemical oxidation, adsorption, coagulation and membrane treatments. Among them, adsorption is rapidly becoming an effective and promising removal method of treating aqueous effluents because of its simplicity. The color removal from textile wastewater is considered as an important application of the adsorption process using low-cost adsorbents such as shale oil ash (Al-Qodah, 2000), chitosan (Juang et al., 1997), sunflower stalk (Sun and Xu, 1997), natural clay (Rytwo et al., 2000; Meshko et al., 2001) against expensive adsorbents such as activated carbon. Recently, biosorption process is one of the effective and attractive processes for treatment of dye-bearing wastewaters.

Therefore, low-cost biosorbents such as yeast, algae, bacteria and biowaste materials were used for dye removal from wastewaters (Aksu, 2005; Wang and Chen, 2006). Waste anaerobic sludge can be used for biosorption process in order to minimize solid waste problem.Experimental based optimization methodology such as response surface methodology (RSM) is helpful approach to identify the optimum conditions for composite systems (Yetilmezsoy et al, 2009).

In this study, decolorization of Maxilon Red GRL (MRGRL)(Basic Red 46) was studied using dried anaerobic sludge (AS) by optimization of pH, initial dye concentration(Co), contact time(tc) and biosorbent amount (m) using a four factor, three level experimental design combining with response surface modelling (RSM) for MRGRL azo dye removal from aqueous solutions. The model provided a good explanation of the relationship among the independent variables and response.

2. METHODOLOGY

2.1 Dried anaerobic sludge (AS) preparation

Waste anaerobic sludge (AS) was taken from anaerobic digester of Municipal Wastewater Treatment Plant (WWTP) in Turkey. The sludge sample was ground and sieved to 0.063–0.125 mm particle size and then washed with distilled water to remove any nonadhesive impurities and small particles, and then dried at 103°C for 24 h to remove moisture. The sample is referred to as dried anaerobic sludge (AS) in the following sections.

2.2 Dye solution

The dye was supplied from a textile factory in Bursa, Turkey and was of commercial quality. Figure 1 and Table 1 indicate the structures and properties of Maxilon Red GRL (MRGRL). λ_{max} of MRGRL was determined in an aqueous medium by using a scanning UV-vis spectrophotometer.

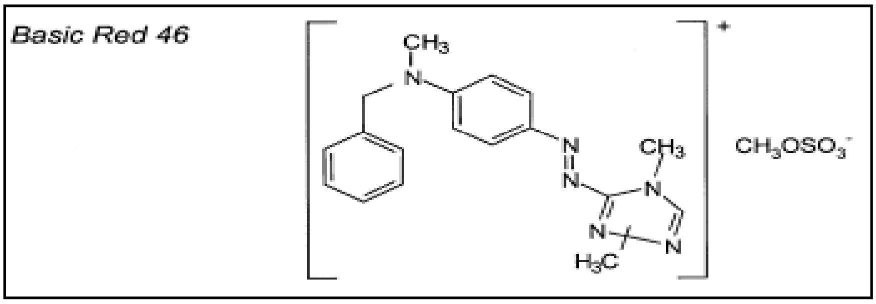

FIGURE 1 **Molecular structures of MRGRL (Basic Red 46)**

TABLE 1 **Properities of MRGRL**

Color index	**MRGRL**
Type	Cationic
Sulphonic group	None
Azo group	1
λ_{max}	530
pH	2-12
*Molecular weight (g/mol)	322
*With (nm)	1.3
*Depth (nm)	0.74
*Thickness (nm)	0.63

* Associated counter ions are not included.

2.2.1 Methods

WTW (Inolab) pH meter was used for pH measurement. Color measurements were carried out in 5 ml samples removed from supernatants of erlenmeyers during the experimental study. The samples were centrifuged at 5000 rpm for 20 min. A Pharmacia Nova Model spectrophotometer was used at 530 nm for MRGRL to measure decolorization.

2.2.2 Response Surface Methodology (RSM)

RSM is an empirical modeling technique used to evaluate the relationship between a set of controllable experimental factors and observed results. (Murugesan et al., 2007)

In this study, the design program was applied at three levels by using a four factor for MRGRL azo dye removal from aqueous solutions by dried anaerobic sludge (AS). Four independent variable input parameters (pH: 2 to 8; Co: 25 to 200 mg/l; tc: 10-180 min., biosolid amount (m) 1-15 g/l were labeled A, B, C and D at three levels as -1 (low),0 (central point or middle) and 1 (high). Three different concentrations of azo dye (25, 112.5, 200), pH (2, 5, 8), tc (10, 95, 180) and m (1, 8, 15 g/l) were chosen as the critical variables and designated as A, B, C, and D respectively.

Total number of 29 experiments was carried out to estimate the 15 coefficients for the removal of MRGRL. Data were analyzed using design program including ANOVA to find out the interaction between the variables and response.

2.2.3 Modelling Results

RSM has been sucessfully applied for optimizing conditions for dye removal using AS. RSM methodology was chosen in this study. The experimental results were analyzed through RSM to obtain an empirical model for the best response. The quadratic model was used to explain the mathematical relationship between the independent variables and dependent responses. The result of analysis of variance (ANOVA) shows that values of probability (P) >F less than 0.05 indicate model terms are significant. Values greater than 0.1000 indicate the model terms are not significant. The model is highly significant, as is evident from the model F-value and a very low probability value (P-value < 0.0001). The R^2 value of 0.90 for percentage dye removal (%), represents the accuracy of the model. The closer the values of R^2 to 1, the better the correlation between the experimental and predicted values and the better the model predicts the response.

Values of Prob> F less than 0.05 indicate model terms are significant.

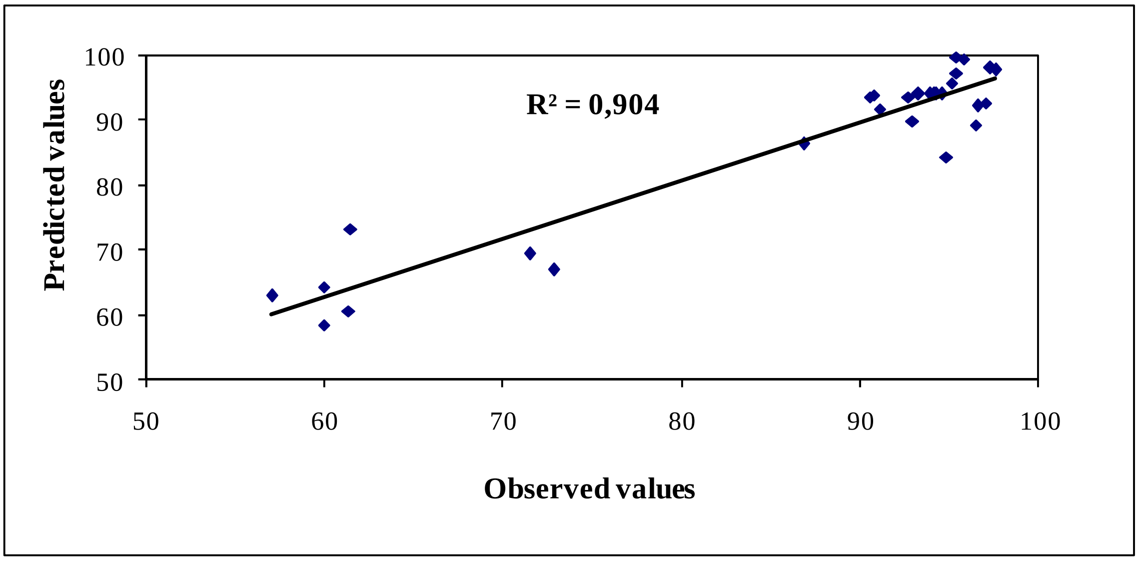

FIGURE 2 **The correlation between the experimental and predicted values**

The value of R^2 >0.904 for the mathematical model shows that the high correlation between observed and predicted values (Fig. 2).

The 3D response surfaces demonstrated that there were considerable interactions between each of the independent variables and MRGRL removal.

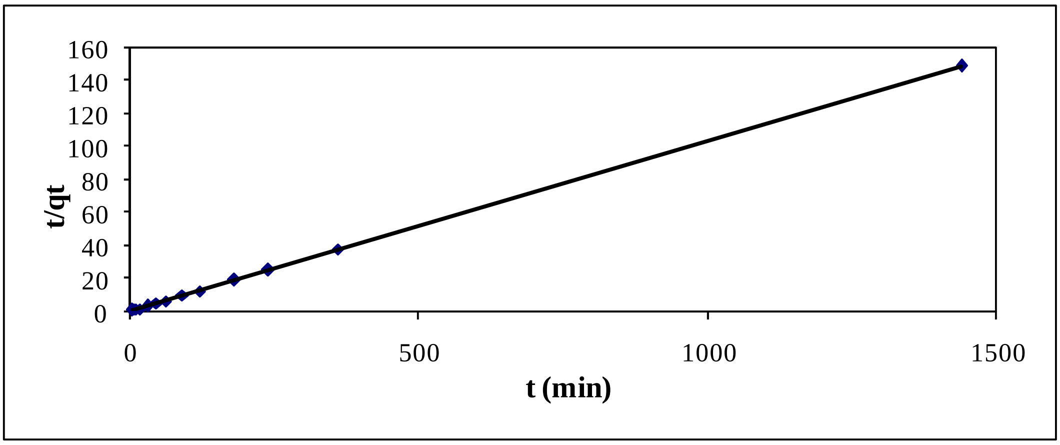

FIGURE 3 **Pseudo-second-order kinetic plot for the biosorption of MRGRL onto AS biomass**

Pseudo-second-order kinetic model was used to investigate the biosorption mechanism. The calculated qe value from the second-order kinetic model agreed with the experimental value of maximum biosorption amount (qexp). Therefore, pseudo-second-order kinetic model was suitable for biosorption kinetics for BR46 removal using AS *(*Fig.3 and Table 2).

TABLE 2 **Pseudo-second-order kinetic constants for the biosorption of MRGRL onto AS biomass**

	$qe_{,exp}$ (mg/g)	**$k_{2,ad}$**	**$qe_{,cal}$mg/g)**	**h (mg/g.dk)**	**R^2**
MRGRL	9.709	0.0862	9.62	8.13	1.000

3. CONCLUSIONS

The optimum experimental conditions were found to be pH= 8, Co= 112.5 mg/l, tc=95 min and m=15 g/l and corresponding MRGRL removal was determined as about 97%. Pseudo-second order was suitable for biosorption kinetics.

AS without any pretreament, one of the low-cost biosorbent can be used for removal of azo dye from wastewaters and it may be an alternative to more costly adsorbent materials.

4. ACKNOWLEDGEMENTS

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