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BIOGAS DIGESTATE TYPOLOGIES

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SUMMARY: Anaerobic digestion (AD) is a well-known technology for waste treatment and bioenergy production, but digestate management is still a key challenge. Defined simply as the digested residue of AD, digestate refers to a set of heterogeneous matters featuring different biochemical compositions and physical properties inherited from greatly diversified AD processes and feedstocks. This situation induces a blurry scenario for policy-makers, digestate producers, marketers and consumers regarding digestate treatment and valorization.

In this context, the objective of this study was to establish a digestate typology with available data on literature and internal databases. Common fertilizing-value parameters were used in the analysis, which are also present in many policies: dry matter (DM), volatile solids (VS), C/N ratio, C/N_{org} ratio, Total N (TN), Total Ammoniacal Nitrogen (TAN), P and K contents, TAN/TN ratio. Heavy metals contents were also separately assessed. In a first approach, a Principal Component Analysis was performed including raw digestates and mechanical separation fractions. With the selected variables, it was found no statistical difference between raw wet (Wet AD) and liquid fraction and between raw dry (Dry-AD) and solid fraction. Later, Hierarchical Clustering Analysis (HCA) was performed on raw digestates resulting in 8 fertilizing-value groups among a dataset of 91 raw digestates. The groups presented variable nutrients and organic matter contents that could be qualitatively linked to the AD feedstock and to the type of process in terms of moisture (Wet or Dry-AD). HCA was performed separately for liquid and solid fractions after mechanical separation and in both cases, digestates were found to be categorized into two groups depending simultaneously on the separation technique and AD feedstock. With regard to heavy metals content, a typology was found to be similarly grouped by AD feedstock.

1. INTRODUCTION

Anaerobic digestion (AD) of organic waste has been evaluated as one of the most energyefficient and environmentally beneficial technologies for bioenergy production in the frame of the European 2020 renewable energy directive (The Commission of the European Communities, 2009).

AD is mostly regarded as a waste treatment and biogas production technology but the digested residue (digestate) is the main final product in terms of mass. Thus, digestate destination is still a key challenge for the development of AD. Digestate spreading is the most applied solution in Europe but it may lead to several environmental issues. For spreading purposes, digestates may be transported for long distances, notably in the case of centralized plants and nutrient exceedance territories (Dahlin et al., 2015). Due to seasonal agricultural demands, digestate might need to be stored for several months, posing risks of gas emissions and nutrient losses (Lukehurst et al., 2010). Besides, in many countries, there is a lack of specific standards framing for digestate use, forcing it to a waste classification, which results in

expensive legal procedures to allow their recovery.

Valorization technologies are being investigated and industrially applied, with multiple objectives: concentrate valuable components, treat undesired characteristics, reduce quality time-variability, produce pure high-value products, create new markets, certify products, among other (Rehl and Müller, 2011).

The definition of digestate as the digested residue of AD implies a heterogeneous set of products with different biochemical compositions and physical properties inherited from greatly diversified AD processes and feedstocks (Lukehurst et al., 2010). This composition variability has been indicated as one of the major concerns for digestate marketing (Dahlin et al., 2015). The establishment of rational digestate typologies is a strategy towards a smarter digestate management that could secure digestate recovery and enhance the dialogue between digestate suppliers, marketers, consumers and policy makers.

The objective of this study was to establish a digestate typology based on commonly fertilizing-value characterization data found in literature, coupled with internal databases from SUEZ and INRA in order to identify the driving forces of digestate quality, despite the limited available information. As a supplement to the study, other parameters such as heavy metals contents which are considered in most legislations for soil amendment, were taken into account.

2. METHODOLOGY

2.1 Data sources

Two different datasets were built: one for a fertilizing-value analysis and one for a heavy metals content analysis. Fertilizing and heavy metals statistics were performed separately because very few studies were found providing a complete digestate characterization (i.e. including both fertilizing value and chemical contamination). Moreover, other crucial information such as biological stability (e.g. respirometric tests, residual biogas potential), phytotoxicity and biological contamination (pathogens) were collected but not sufficiently to be processed by the advanced statistical analysis. For the fertilizing-value clustering analysis, unpublished internal data provided by SUEZ (CIRSEE) and INRA (LBE) was complemented with data collected from 15 peer-reviewed scientific articles (Bachmann et al., 2016; De Moor et al., 2013; Marcato et al., 2008; Massaccesi et al., 2013; Géraldine Maynaud et al., 2017b; Möller et al., 2008; Riva et al., 2016; Schievano et al., 2011; Seppälä et al., 2013; Solé-Bundó et al., 2017; Tambone et al., 2010; Tampio et al., 2015; Teglia et al., 2011; Vaneeckhaute et al., 2013a, 2013b), 1 conference paper (Chiumenti et al., 2010) and from three technical reports (Dabert, 2015; Martin, 2004; Moletta Méthanisation, 2011). For the heavy metal content analysis, data from SUEZ was coupled with literature data collected from 10 peer-reviewed scientific articles (Abubaker et al., 2012; Alburquerque et al., 2012; Bustamante et al., 2013; Carballa et al., 2009; De Moor et al., 2013; Gulyás et al., 2012; Moreira et al., 2008; Stefaniuk et al., 2015; Tampio et al., 2016; Vaneeckhaute et al., 2013a). The referenced datasets are available in Appendix I and II. Data from SUEZ was omitted for confidentiality reasons.

Detailed information on AD configuration was not always available, but the digestates in both datasets came from greatly diversified inputs and AD processes (wet/dry-AD, continuous stirred-tank reactor/plug-flow/batch, meso/thermophilic, single/two-stage). In the fertilizing value dataset, from the 150 digestate data lines (which include liquid and solid fractions obtained after a separation step applied to the digestate), 110 digestates were sampled and analyzed from full-scale digesters, the rest being produced by either pilot or bench scale tests.

In order to illustrate the feedstock variability of the resulting datasets, the three main inputs of each digestate (as presented in Appendix I and II) were counted and summarized in Table 1. Each dataset column represents a sub-dataset used for statistical analysis.

RW: Raw digestate from liquid and wet AD. RD: Raw digestate from dry AD (high solids). P_SF: Raw digestate from dry-AD, when specified as the solids of a percolation system. LF: liquid fraction of digestate after mechanical phase separation. SF: solid fraction of digestate after mechanical phase separation. * Including Energy Crops when specified as silage

Agricultural residues include field and process residues, straw, litter and fodder material.

2.2. Selected variables, unit conversions and other calculation

The chosen parameters were Dry Matter (DM), Volatile Solids (VS), Carbon/Nitrogen ratio (C/N), Carbon/Organic Nitrogen ratio (C/N_{org}), Total Nitrogen (TN), Total Ammoniacal Nitrogen (TAN), N-NH4/TN ratio,Total Phosphorus (TP) and Total Potassium (TK).

Table 2 summarizes the usefulness of each selected parameters. Variables were related to a dry mass basis since it reduces the major effect of the absolute values that are mostly driven by the applied moisture in the AD process. Table 3 summarizes the heavy metals used as variables for the statistical analysis and their maximum limit according to quality criteria from United Kingdom (for digestates), Sweden (for digesates) and France (for organic soil improvers).

Only papers where the units and analytical methods were clear were included. In some cases, presented information was confirmed directly with the authors by either e-mail or research social networks. In order to establish a common dataset, units were uniformized. For example, some papers presented data on a fresh weight basis and other on dry matter basis. These values were converted if the dry matter content (total solids) was available. Many authors presented nutrient content in mineral form equivalents (P_2O_5 and K_2O), especially in the agronomic research field. Those values were converted to total P, K and Mg by their conversion factor based on their molecular composition (0.4364 and 0.8301, respectively), and reconverted when compared to legislation values. The TAN/TN ratio was calculated and added to the dataset when both values were available. Total Kjeldahl Nitrogen was considered as TN since

nitrates and nitrites are negligible in digestates, ranging from 0 to 30 mg/L (De Moor et al., 2013; Haraldsen et al., 2011; Seppälä et al., 2013; Walsh et al., 2012). When N_{org} was not available, it was calculated from the difference of TN (or TKN) and TAN. Since the definition of calculation for C/N was observed to be variable among the literature, C/N and C/N_{ora} were calculated indirectly through the VS content (TOC as 50% of VS, thus allowing a homogenous definition across the database. This definition is the one proposed by some policies as French NFU 44- 051 (AFNOR, 2008).

Table 2: Parameters used for the fertilizing-value clustering analysis.

Table 3: Selected Heavy Metals and their maximum contents required by quality criteria from UK (British Standards Institution, 2010), Sweden (Petersson, 2013) and France (AFNOR, 2006)

*French legislation also includes limits for As and Se.

2.3 Statistical analysis

Statistics were carried out with the software R-studio and R language version 3.3.2. The outcome typology was established by Hierarchical Clustering Analysis (HCA). HCA was applied after a Euclidean distance matrix was calculated with center-scaled variables. The clustering method was the one defined by Ward (1963), applied by the "hclust" algorithm (method Ward.D2) of the "stats" R package version 3.3.2.

Once the dendrogram was constructed, the definition of the number of groups (i.e. the cutting height on the cluster tree) was performed by a heuristic approach. Qualitative information associated with each individual (digestate) was used to justify the resulting clusters. This information consisted on the origin AD feedstock and selected AD operational parameters. The parameters were moisture (Wet/Dry-AD), temperature (mesophilic/thermophilic) and the organic loading rate, but the last two were barely available.

To allow a better understanding of the variables influence in the formation of clusters, the resulting HCA dendrograms were coupled with heatmaps. The resulting typology was then used to group the individuals in Principal Component Analysis (PCA), whose resulting biplots permitted a spatial visualization of the clusters with an evaluation of correlations between the variables.

Finally, in some cases, variables of interest were selected to produce boxplots where absolute values can be observed.

3. RESULTS AND DISCUSSION

3.1 Fertilizing value

3.1.1 Raw digestates, liquid and solid fractions

In many reports, digestate characterization and treatment options are classified separately for raw digestate, liquid and solid fractions after separation process (hereafter mentioned as digestate states). In order to test this *a priori* typology, statistical analyses were performed in the sub-dataset 1.1 (cf. Table 1). A PCA plot of individuals for raw digestates, liquid and solid fractions is presented in Figure 1. In this plot, the dimensions 1 and 2 describe more than 65% of the variance. The individuals are grouped according to the digestate state as informed by the data source. It can be observed that resulting groups are not completely different according to this classification. 95% confidence intervals for RW and LF and for RD and SF are almost completely overlapped. In the other hand, even if the presence of some outliers generated confidence ellipses crossing the y-axis, digestates are almost perfectly separated into dry/solid (on the left area) and wet/liquid (on the right area). This means that the variance among this classification relies on dimension 1, which is positively correlated to TN, TAN, TAN/TN and TK (0.90, 0.87, 0.71, 0.65, p-values < 0.01) and negatively correlated to C/N, DM, C/Norg, VS and TK (-0.86, -0.83, -0.57, -0.41, respectively, p-values > 0.01). Besides the overlapping with raw digestates, LF confidence interval is more skewed to the right compared to RW while SF is more distributed to the left than RD.

For treatment processes, for example, many schemes on literature propose different solutions for raw digestates, liquid and solid fraction, but this result suggests that these definitions are insufficient for digestate classification. Treatment and destination options must be regarded more case-by-case or by categories relying on other criteria. Since digestates, including LF and SF in the definition, were observed to be not completely dissociable with the available parameters, further statistical analysis were conducted separately (according to the sub-datasets described in Table 1) with the focus of discussion being on raw digestates.

Figure 1: PCA of digestates in raw dry (RD), raw wet (RW), Percolate Solid Fraction (P_SF) liquid fraction (LF) and solid fraction (SF) of digestates based on fertilizing-value parameters. Individuals are grouped by state according to the source. Ellipses indicate 95% confidence interval.

3.1.2 Solid fraction

Clustering analysis of sub-dataset 1.2 (cf. Table 1) resulted in four groups. The first consist of two silage+manure digestates from the study from Chiumenti et al. (2010), that are clustered thanks to a particularly high TK content (>25 g/kg DM). The second one is SF of sewage sludge (SS) and SS co-digestion, characterized by low C/N and C/N org (about 0.7, both), lower DM $(20 - 25\%)$ ¹, lower VS $(60 - 73\%)$ ¹, lower TAN/TN $(15 - 25\%)$ ¹, higher TN $(42 - 53$ g/kg DM)¹, higher TP (24 – 30 g/kg DM)¹ and lower TK (<5 g/kg DM). The third group is for SF clustered by a specifically high TAN (>15 g/kg DM) and TAN/TN (>80%), which are not common for SF (Géraldine Maynaud et al., 2017a; Vaneeckhaute et al., 2013a). The last group is for fibrous feedstock material such cattle manure and silage, separated mostly by screw presses. This groups is characterized by higher C/N and C/Norg values (15 – 23 and 20 – 30, respectively)¹, higher DM $(24 - 30\%)$ ¹, higher VS $(75 - 86\%)$ ¹, higher TAN/TN $(25 - 35\%)$, considerably lower TN and TP (20 – 24 and 6 – 10 g/kg DM, respectively)¹ and higher TK (8 – 13 g/kg FM)¹. It is important to notice that DM is higher in the cluster for "low performance" separation but it does not imply a more efficient solids separation since the associated raw digestates normally present a higher initial DM content.

The resulting PCA is presented in Figure 2**Errore. L'origine riferimento non è stata trovata.** as a biplot of variables and individuals grouped by the result of HCA. Total variance description with the two components is greater than 65%. In the PCA, it can be observed two well-defined groups simultaneously linked to AD feedstock and separation technique: techniques known for lower separation performance are on the right, along with more fibrous feedstock and higher performance separation techniques on the left, with less fibrous input

 ¹ Rounded interquartile ranges (0.25 and 0.75).

material.

Some centrifuge on the low-performance cluster can be observed. Those consist of two SF from 100% silage digestion (Bachmann et al., 2016) and one from sewage sludge with food processing waste (Teglia et al., 2011). It must be noticed that within the database, Bachmann et al. (2016) centrifuges are the single ones presenting less than 10% of mass distribution into the \mathbb{Z} liquid fraction, which was found to be typical of screw presses. For centrifuges, this value was observed to be normally greater than 20%. Moreover, the sewage sludge co-digestion digestate from Teglia et al. (2011) has both relative low DM and VS for cluster 4.

The two individuals of group 1 consists of two solid fractions (SF) from the same plant where the digestate was separated by screw press and then the liquid fraction from screw press was centrifuged (Chiumenti et al. , 2010). Even if they were statistically clustered in the same group, it can be observed that they are separated respecting the confidence intervals of groups 2 and 4: the first SF is similar to group 4 (lower performance separation) while the second SF is similar to group 2 (higher performance separation).

The great difference of compositions within the two main groups are probably due to a double effect of feedstock and separation technique (interdependent): in one hand, similar groups were found for raw digestates (section 3.1.4), which are linked to the same types of feedstock: lower DM, N and P-rich but K-poor (sewage sludge, biowaste) and higher DM and Krich (fibrous material). In the other hand, for a same kind of substrate, pressurized filtrations techniques such as screw presses (used for fibrous material) produce solid fractions with higher DM and less retention of N and P than centrifuges (Hiorth et al., 2010). Moreover, since K is associated to the water fraction, less performing separation techniques will produce a solid cake with greater K content (Hjorth et al., 2010).

Figure 2: PCA of solid fraction of digestates based on fertilizing-value parameters. Individuals grouped by HCA. SP: Screw Press, SV: Sieve, C: Centrifugation, CD: CentriDry.

3.1.3 Liquid fraction

Resulting PCA biplot of the analysis performed with liquid fraction of digestates (sub-dataset

1.3) is presented in Figure 3. In this plot, the associated separation techniques are presented when the information was available. The confidence interval was excluded for group 1 since it was greatly distorted by a single outlier (TAN > 250 g/kg DM).

The HCA resulted in three groups. The first one gathers liquid fractions from digestate of feedstock material such as pig slurry, food waste, FAI and OFMSW, which are normally poorly fibrous. This cluster is characterized by lower DM content $(2.1 - 4.9 \%)^1$, lower VS content (<60%), higher TAN/TN (70 - 80%)¹, higher TAN and TN (both >100 g/kg FM). The second group presented three liquid fractions (CS/FW, 95% CM and SepHHW/GW as inputs, the first two from Dry-AD and the last from wet-AD but high solids), presenting particularly high DM content (>10%), all of them separated by screw press. The last group was for fibrous feedstocks (mainly cattle slurry, cattle manure and silage), presenting liquid fraction of digestate characterized by a higher DM content (4.8 – 5.8%)¹ greater VS content (65 - 70%)¹ lower TAN/TN ratio $(41 - 60\%)$ ¹ lower TAN (<50 g/kg FM), lower TN (<100 g/kg FM).

Analogously to what was observed with solid fractions, the groups and their nutrient composition are simultaneously depending on feedstock and separation performance. Groups 2 and 3, both containing LF from fibrous material and lower separation technique are in the negative part of the x-axis (left area) of the biplot presented in Figure 3 (i.e., higher DM and higher C/N). The only two liquid fractions from centrifuge in the whole left area (supposedly lower performance) are from the study of Bachmann et al. (2016). This centrifuges are the same that were already discussed in the section about solid fractions. However, it is difficult to state that group 1 is for LF from higher separation performance equipment. This is due to the fact that there is an important number of samples from drainage techniques such as belt filters, drum filters and rotating screens. These techniques retain particles not only on the mesh/screen but on the solid cake during the filtration process, which results in performances that are highly depending on retention time (Hjorth et al., 2010). A liquid fraction from screw press is also present in group 1, presenting a DM content (5.6%) that is in the range of group 3 $(4.8 - 5.8\%)^1$ rather than its own $(2.2 - 4.9\%)^1$.

 $¹$ Rounded interquartile ranges (0.25 and 0.75).</sup>

Figure 3: PCA of liquid fraction of digestates based on fertilizing-value parameters. Individuals grouped by HCA. SP: Screw Press, SV: Sieve, C: Centrifugation, BF: Belt filter, DF: Drum filter, RS: Rotary screen. Lab.: from laboratory scale.

3.1.4 Raw digestate only

For raw digestates, HCA result is presented as a heatmap (Figure 4), where despite the fact that variables are center-scaled, there is no loss of information. From the heatmap, it can be observed that digestates from wet and dry-AD were almost perfectly separated into two first clusters, the driving factors being C/N, C/Norg and DM. Raw dry digestates present DM values ranging from 10 to 45%¹, while raw wet/liquid digestates presented DM content from about 2 to 10%. C/N and C/Norg in RD are greater than 10 and 20 respectively and the opposite for RW.

The 8 resulting clusters (cutting height based on a heuristic approach) consist of 4 RD and 4 RW digestate groups. **Group 1** is for the experimental dry digestate from a percolate system proposed by Massaccesi et al. (2013), which presents a particularly high DM content (35-50%). **Group 2** is for manure/silage and FAI co-digestion (three digestates from De Moor et al. (2013) and one from (Maynaud et al., 2017) that resulted on very high C/Norg (about 60), high TAN/TN (about 75%) and poor N and P but average/high TK of about 35 g/kg DM , probably due to an dilution effect of TK from cattle manure. **Group 3** is for dry-AD of OFMSW and/or sourceseparated biowaste (SepBW), being characterized by a global poor nutrient content (TAN 5 – 11 g/kg DM, TN 16 - 24 g/kg DM, TK^1 0.6 - 13 g/kg DM) and a VS content of about 40-50% while most of the digestates present VS greater than 50%. **Group 4** is for dry-AD of fibrous material such as cattle manure, silage and green waste. This groups present the highest VS content (72 to 82%², reaching up to 90%) and a general poor nutrient content (TAN 1 – 18 g/kg DM, TN 13 - 45 g/kgDM, TP 0.2 - 8 g/kg DM, TK¹ 3 to 17 g/kg DM). Entering in the RW branch, group 5 gathers digestate from Wet-AD OFMSW and SepBW, but also with a few digestate from animal slurry co-digestion. This group is characterized by high TN (115 – 145 g/kg DM), TAN

 1 Values greter than 35% were from an experimental percolate system ((Massaccesi et al., 2013)

 2 Rounded interquartile ranges (0.25 and 0.75).

 $(87 - 196 \text{ g/kg DM})$ and TAN/TN values $(63 - 85%)$ and also relatively K-rich $(40 - 95 \text{ g/kg DM})$. **Group 6** is for wet-AD of fibrous material such as manure and silage, being characterized by a high VS of 69 to 81% excluding two outliers of about 65% that are also digestates from cattle manure/silage. This group presents a relative high TK content (50 – 70 g/kg DM) ¹. Group 7 is for SS and SS co-digestion, being characterized by low VS, low TAN/TN and low TK. This cluster also includes the microalgae (MA) and 1ry-SS + MA digestates from Solé-Bundó et al. (2017), that are sub-clustered into a P-poor group $(3 - 4)$ g/kg DM) while the other SS digestate from the group are P-rich (27 - 40 g/kg DM). In this paper, MA has been sampled from a pilot scale raceway pond treating municipal wastewater, which may explain the similarity to other digestate from sewage sludge and SS co-digestion. Finally, **group 8** is for a diversified range of inputs. Among the feedstock composition, 22 out of 27 are from mixtures of SepBW, FAI and animal slurry. The group also include a few SS and SS co-digestion individuals (5/27). Group 8 is characterized by relatively high TAN (31 – 66 g/kg DM) and TN contents (75-100 g/kg DM)¹. One can notice in the heatmap that digestates from group 5 and 8 are similar in terms of composition, being separated mostly by the considerably higher TAN, TN and TAN/TN values of group 5.

Global categories could be found and linked to feedstock composition and process moisture. However this was not completely sufficient to understand the variability of digestates and the formation of clusters, mainly due to the lack of precision in the data sources. For instance, some generic inputs categories such as FAI, animal waste, biowaste, among other, are composed of diversified waste streams in terms of nutrients and organic matter composition. Moreover, there were no sufficient elements in the database to further assess the impact of AD process configuration such as temperature, retention time and loading rate since many of them were not available in the sources of data.

Figure 5 shows a set of boxplots for nutrients composition expressed in fresh matter and mineral equivalents. Other information not included in the statistical analysis, but available in the database, are presented: MgO content, CaO content and residual biogas production. When possible, the variables are compared to the limit values set by the French standard for organic soil amendments (AFNOR, 2006) and to the recommended criteria from the End-of-Waste report proposed by the European Commission Joint Research Centre's Institute for Prospective Technological Studies (JRC-IPTS) (Saveyn and Eder, 2014).

Despite the fact that digestates have been extensively demonstrated as effective fertilizers and soil improvers (Abubaker et al., 2012; Alburquerque et al., 2012; Haraldsen et al., 2011; Riva et al., 2016; Tampio et al., 2015), it can be observed that no raw digestate fulfils the French criteria for organic soil improvers (AFNOR, 2006). This result highlights the importance of updating policies conceived for composts and animal manures with high dry matter content.

Regarding the End-of-Waste proposal criteria, some digestate would not be considered stable in terms of residual biodegradability (0.25 NL biogas/kg VS ceiling). The highest residual biogas production in the dataset (about 0.4 NL biogas/kg VS) are from digestates from Tambone et al. (2010). It is interesting to highlight that in this study, the digestates (from OFMSW + Pig slurry) presented residual biogas production values smaller than those of composts from lignocellulosic material (some of them co-composted with OFMSW). Furthermore, organic matter conversion thus organic stabilization of digestates can be driven by AD retention time, loading rate and temperature (Cavinato et al., 2013; De Moor et al., 2013). For instance, in the full-scale plant studied by Pognani et al. (2009), the residual digestate biogas production was decreased from over 0.3 NL/kg VS on raw digestate (over the proposed limit) to 0.1 NL/kg VS (below the limit) after a post-digestion step of 10 days.

Figure 4: Heatmap with clustered individuals. All variables are center-scaled. P_SF: Solid digestate from percolate system (Dry-AD). RD: Raw dry digestate (Dry-AD).

Figure 5: Boxplot for absolute values and nutrient composition expressed in fresh matter and mineral equivalents.

3.2 Heavy metals: Raw digestates, liquid and solid fractions

PCA result for dataset 2 (c.f. Table 1) is presented by individuals/variables biplots in Figure 6 and Figure 7. Digestates are grouped by state classification (according to the source) in Figure 6 and by HCA groups in Figure 7. With two components, this PCA described more than 70% of the variance. Component 1 is basically indicating a high content of all Heavy Metals (HM) except Cd, indicated by component 2. Component 2 also indicate Zn content in the negative part of the y-axis.

As observed in the fertilizing-value analysis, there was no inherent statistical difference within a classification based on digestate state for the heavy metals content (Figure 6).

With the clustering analysis, groups could be successfully associated to AD feedstock (Figure 7). In the collected database, sewage sludge showed higher global heavy metals concentration. High Cd content was observed in 4 digestates containing maize silage as the

main input, all of them from Stefaniuk et al. (2015). Cd in crops is associated with soil contamination due to the application of P fertilizers from phosphate rocks, sewage sludge spreading and atmospheric transport of mining dust (Robson et al., 2014; Van Bruwaene et al., 1984). High Zn and Cu contents were observed in two digestates from animal waste. One is a solid fraction of digested pig slurry (Bustamante et al., 2013) and the other is a raw cattle slurry digestate (Alburquerque et al., 2012). Cu and Zn are widely applied in animal feed due to antimicrobial and growth-stimulating effects (Poulsen, 1998). In addition, copper sulfate is used on dairy disease-preventing footbaths. In the study of Bustamante et al. (2013), the presence of such heavy metals contents in digestate arising from the pig slurry led to a non-compliant final compost regarding the Spanish legislation (BOE, 2005).

After the clusters were established with center-scaled variables, absolute values were regathered and plotted in Figure 8. It can be observed that no digestate cluster fulfills all the limits for the three reference legislations/quality criteria. However, it must be noticed that there are 9 digestates from the 44 that are individually below all limits of the three standards. Moreover, 15 digestates are below all limits for at least one of the three legislations and 11 digestates are below all limits but with absent values (not considering As and Se for NFU 44- 051).

Even if heavy metal content could be associated with certain types of feedstock, this observation must not be taken as a rule. For example, 3 digestates from sewage sludge (2 codigestion with other material) are below all limits. Also, in the low heavy metal content cluster (group 3), digestates containing pig manure (such as those from De Moor et al. (2013)) and maize silage (including digestates from Stefaniuk et al. (2015)) can be found respecting all legislation standards.

Broadly, even if statistical analysis from heavy metals suggests that HM presence is associated to some specific feedstock, it must be regarded more specifically by cases since many other digestates from the same or similar feedstock comply with one or more of the three reference criteria. Unfortunately, it was not possible to clearly identify elements within the sources to explain the causes of high presence of HM other than the AD inputs.

Figure 6: PCA of digestates in raw dry (RD), raw wet (RW), liquid fraction (LF) and solid fractions (SF) of digestates based on heavy metals content. Individuals grouped by state.

Figure 7: PCA of Heavy metals in raw dry and wet (RD and RW) digestates, liquid and solid fractions (LF and SF). Individuals grouped by HCA.

Figure 8: Boxplots of HCA groups from Heavy Metals dataset. Values expressed in mg.kgDM⁻¹. SS: Sewage sludge, OFMSW: Organic Fraction of Municipal Solid Waste. HHW: Sourceseparated household waste.

4. CONCLUSIONS

In this work, a raw digestate typology was statistically established based on common fertilizing-value parameters: groups could be qualitatively linked to the AD feedstock and to the AD process in terms of moisture.

For liquid and solid fractions after mechanical separation, digestates were found to be categorized into two groups depending simultaneously on the separation technique and on the feedstock.

In terms of heavy metals content, a typology was found to be similarly grouped by AD feedstock.

For both fertilizing values and heavy metals contents, no statistical difference was found if digestates, including separation fractions, were arbitrarily classified according to their state: raw and liquid or solid fraction after mechanical phase separation.

The objective determination of digestate typologies can be a tool for policy makers and marketers to settle classifications matching the reality of digestate producers with the needs of digestate consumers. Moreover, the established typology opens a possibility of orientating the final destination of digestate and sub-products by considering the input feedstock and by applying adequate processes to reach market and regulatory specifications. In any case, a deeper characterization and a more detailed typology are necessary, including, for instance, more detailed information on inputs quality and AD parameters and a deeper characterization of the organic matter.

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ABBREVIATION

APPENDIX I: DATASET 1 (FERTILIZING VALUE)

30

a. Calculated from VS and TN (C/N=0,5 x VS / TN)

b. Calculated from VS and N_{org} (C/N=0,5 x VS / N_{org})

APPENDIC II: DATASET 2 (HEAVY METALS)

30

NA: Not available. ND: Not deteced.

a. Converted from Dry matter basis to Fresh matters basis with available DM content.

APPENDIX III – PCA LOADING SCORES

Table 4: Loading matrix for fertilizing-value PCA (sub-dataset 1.1)

* dry matter basis

Table 5: Loading matrix for LF fertilizing-value PCA (sub-dataset 1.3).

* dry matter basis

Table 6: Loading matrix for SF fertilizing-value PCA (sub-dataset 1.4).

dry matter basis

Table 7: Loading matrix for Heavy metals content PCA.

