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Organic matter characterization: towards a unified methodology for biological treatments modelling

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Abstract
The present study is an extension of the promising work performed by Jimenez *et al.* (2014). They developed an organic matter characterization methodology based on 3D fluorescence spectroscopy and chemical extractions. The objective was to predict both biodegradability and bioaccessibility to characterize the anaerobic digestion model input variables but only focused on municipal sludge. In this study, the protocol was extended to a larger spectrum of organic wastes and modified to better account for this diversity of organic residues. Accordingly, the ADM1 dynamic model has been modified with new input variables coming from this new methodology.

Keywords
Anaerobic digestion, modelling, biodegradability, bioaccessibility, 3D fluorescence

INTRODUCTION

In a context of environmental biorefinery, there is currently only few studies focused on both optimization of energetic valorization (by anaerobic digestion) and agronomical valorization of organic wastes (after or not composting). One way to achieve both objectives is process modelling. However, input variables from existing models are different (Jimenez et al., 2014; Zhang et al., 2012) and there is a need to find relevant and common input variables to set-up a plant wide model of organic matter of wastes, from the biological treatments to land spreading. In the recent years, a new promising methodology - the three dimensional fluorescence spectroscopy combined with organic matter fractionation by chemical extractions- has been successfully used to predict anaerobic biodegradability and bioaccessibility of municipal sludge (Jimenez *et al*., 2014). This study is an extension of this previous work: the application of this methodology to a wider range of organic wastes and the modification of the anaerobic digestion model input variables for a plant wide organic wastes treatment objective.

MATERIAL AND METHODS

Biochemical methane potential test

To test the potential of the new protocol set up, anaerobic biodegradability of 58 organic wastes samples were measured through the Flash BMP® test, commercialized by Ondalys, and based on an instantaneous optical measurement of Near-Infrared Spectroscopy (NIRS) (Lesteur *et al.*, 2011). The equivalent biochemical methane potential (BMP) is obtained from a Partial Least Square model in Nml CH_4.gVS^{-1}.

Chemical sequential extractions

The new extraction protocol was based on (Jimenez *et al*., 2014) with some modifications such as the addition of the acid extraction from Van Soest protocol (Van Soest, 1963). After drying and grinding (1mm) the sample, sequential extractions (30 mL) are performed on 0.5g of the dried sample. The obtained fractions are: (1) Soluble Extractible Organic Matter (SPOM) (milli-Q water solution containing 10mM of $CaCl_2$, 15 min, 30°C, 300 rpm) (2) Readily Extractible Organic Matter (REOM) (saline basic extraction 10mM, 15min, 30°C, 300 rpm) (3) Slowly Extractible Organic Matter (SEOM) (NaOH 0.1 M, 4 h, 30°C,300 rpm), (4) Poorly Extractible Organic Matter

(PEOM) obtained by 2 sequential acid extractions (25 mL H_2SO_4, 72%, 3 h, 30°C, 300 rpm). Results are expressed in COD ($gO_2.gTS^{-1}$).

Fluorescence spectroscopy analysis

The fluorescence spectrometer used was a Perkin Elmer LS55. Excitation wavelengths varied from 200 to 600 nm with increments of 10 nm. Fluorescence values were recorded every 0.5 nm between 200 and 600 nm. Based Jimenez *et al*. (2014), spectra were decomposed on seven zones corresponding on biochemical families-like fluorescence. The proportion of fluorescence of a zone "i" $P_f(i)$ was calculated from the fluorescence zone volumes.

RESULTS

Accessibility characterization: new protocol

Fractionation protocol and 3D fluorescence spectroscopy were applied on 58 samples of organic wastes of different origins to cover the whole spectrum of applications (municipal wastes:3, municipal sludge:7, green wastes: 9, composts:16, manure: 2; digestate: 12, crop soils:3), . Using 33 variables composed of the 5 fractions (% in COD of SPOM, REOM, SEOM, PEOM and NEOM) and of the 4*7 fluorescence percentages $P_f(i)$ corresponding to the 7 fluorescence zones of each extracts (SPOM, REOM, SEOM and PEOM), a principal component analysis was performed. The first component describes the complexity of the molecules (quality of the extracted COD) whereas the second one describes the accessibility of the molecules (quantity of the extracted COD), the two revealing 5 groups. Results are presented in the Figures A.1 from the supporting information. The variables induced by the combination of fractionation and 3D fluorescence spectroscopy are discriminant enough to classify the different nature of organic waste. The addition of acid extraction allows to extract between 60 and 90% of COD and to characterize not only sludge but also composts, green wastes, biowastes and soils.

Biodegradability characterization: prediction model

In order to go further and to validate the ability of these data to predict the biodegradability, a Partial Least Square (PLS) regression is successfully performed on 25 samples and then 25 samples are used for validation. The X-variables used are the same than in the (Jimenez *et al*., 2014) work. 28 variables are used and are the combination of the four fractions $\boldsymbol{Fract_{zone\ i}} = \sum_{i=1}^{VII} \boldsymbol{P_f(i)} \times \boldsymbol{Fract}$ (%COD) (i.e. SPOM, REOM, SEOM and PEOM) with the 7 fluorescence percentage zones. Y-variable is the BMP value obtained with the FlashBMP® test.

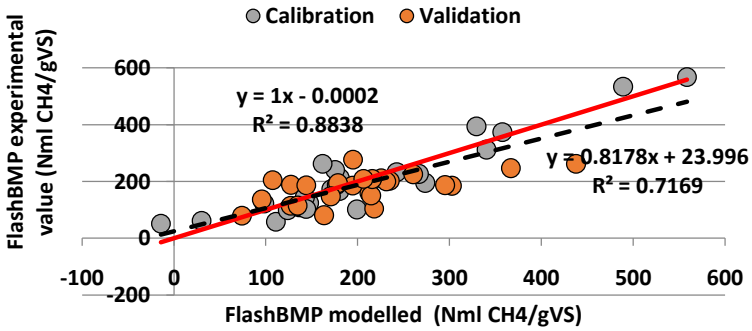

Figure 1. Correlation between modelled FlashBMP® values obtained with PLS and the experimental values

The PLS regression showed that there is a good correlation between the X and Y variables, with a R^2Y value of 0.89 (Figure 1). Only three components are enough to describe 58% of X-variables and 89% of Y-variable. RMSE value was 47 $NmlCH_4.gVS^{-1}$ and RMSEP was equal to 75 $NmlCH_4.gVS^{-1}$. The methodology is successfully modified and is relevant enough to predict anaerobic biodegradability of a wider range of organic wastes (i.e. protein-like, ligno-cellulosic-

like, biowastes, composts).

Modified ADM1: modifications of input variables

Using the modified Anaerobic Digestion model n°1 (ADM1) and data from Jimenez *et al*. (2014), calibration and validation of a lab scale mesophilic continuous reactors are performed. Input variables of the previously modified ADM1 are replaced by the fractionation simulating bioaccessibility (i.e. SPOM, REOM, SEOM as readily hydrolysable and PEOM and NEOM as slowly hydrolysable) and biodegradability is predicted by the PLS model. Acidogenesis biological process is simplified by considering only one soluble product (i.e. dissolved organic matter). Biological parameters are the same than the ones calibrated by Jimenez *et al.* (2014). Modified model can successfully predict anaerobic digestion of municipal sludge during organic matter or load perturbations as shown by the results from the Figure 2. Modifications of the model gave the same simulated results than the previous ADM1. Therefore, model was validated to be used for plant wide modelling.

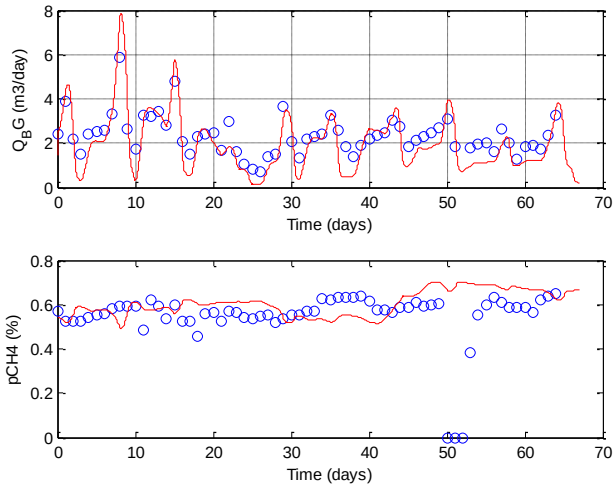

Figure 2. Validation of the new modified model of anaerobic digestion: biogas and methane production (red line: model; blue dot: experimental values)

CONCLUSIONS AND PERSPECTIVES

The new fractionation method developed has been successfully applied on a wider range of organic wastes. It allows characterizing the organic matter and predicts its biodegradability. Besides, the fractions extracted were used as input variables of ADM1 and model was modified for this purpose to obtain the same results than with the previous ADM1. The next step will be the implementation of a composting and soil models with the same input variables to optimize the plant wide treatment according both objectives of energetic and agronomical valorization.

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SUPPORTING INFORMATION

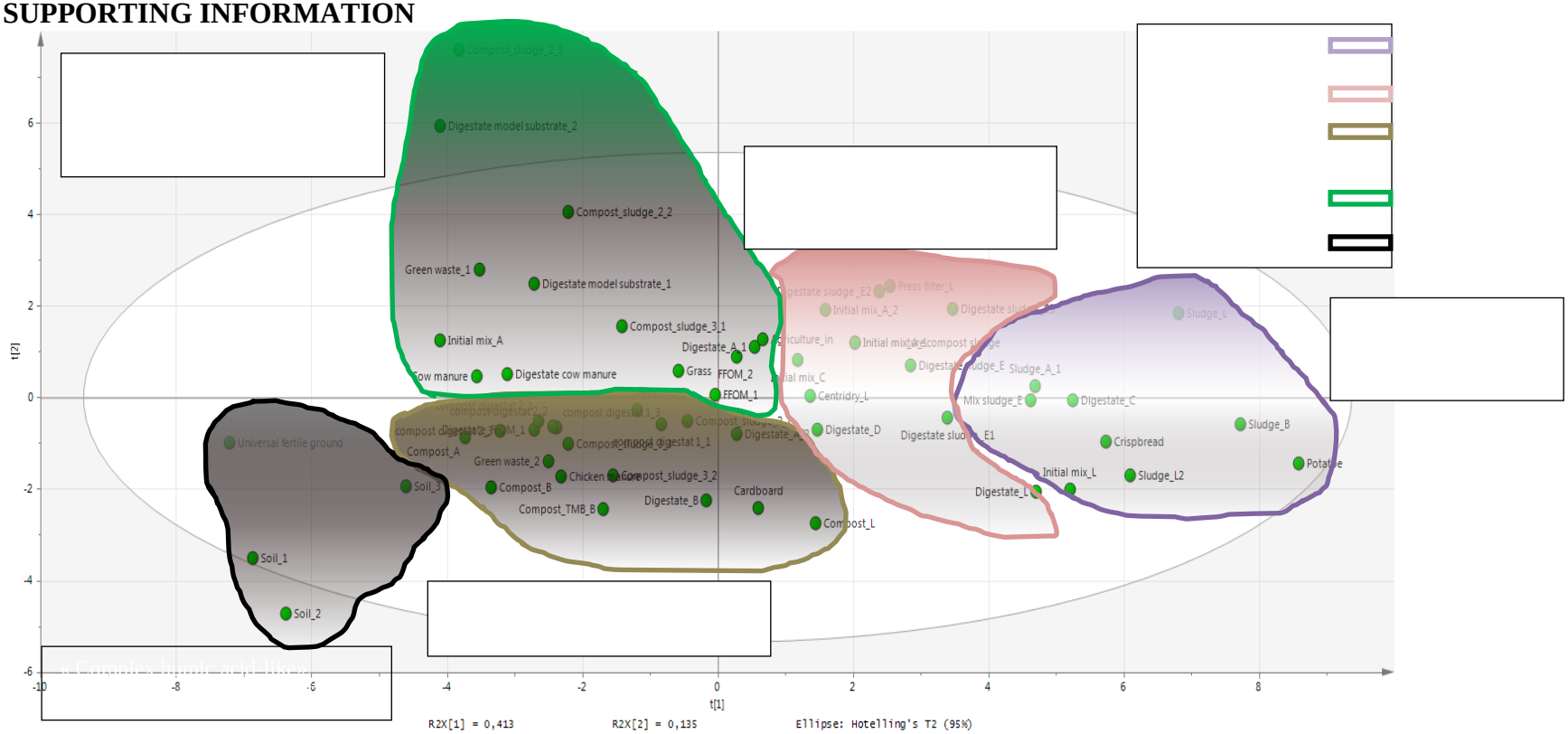

Figure A.1 : Correlation circles (scores) obtained from PCA analysis of all the samples studied for the 58 samples of organic wastes.