



# Characterisation of the biodegradability of post-treated digestates via the chemical accessibility and complexity of organic matter



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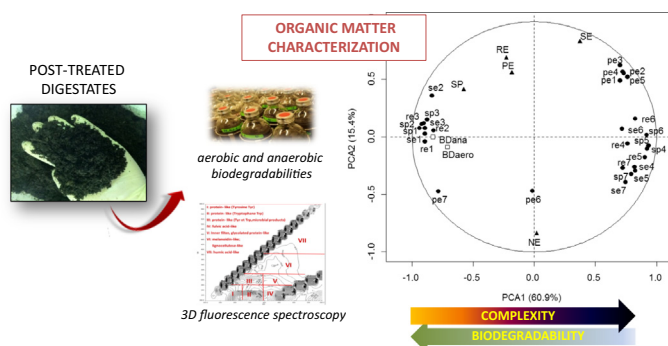
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## HIGHLIGHTS

- Aerobic and anaerobic biodegradabilities are linearly correlated.
- Biodegradability is mostly anti-correlated with the complexity of organic matter.
- Post-treatments have a significant effect on biodegradability and complexity.
- Kinetics of respirometric activity present multi-peaks for certain digestates.

## GRAPHICAL ABSTRACT



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## ABSTRACT

The stability of digestate organic matter is a key parameter for its use in agriculture. Here, the organic matter stability was compared between 14 post-treated digestates and the relationship between organic matter complexity and biodegradability was highlighted. Respirometric activity and CH<sub>4</sub> yields in batch tests showed a positive linear correlation between both types of biodegradability ( $R^2 = 0.8$ ). The accessibility and complexity of organic matter were assessed using chemical extractions combined with fluorescence spectroscopy, and biodegradability was mostly anti-correlated with complexity of organic matter. Post-treatments presented a significant effect on the biodegradability and complexity of organic matter. Biodegradability was low for composted digestates which comprised slowly accessible complex molecules. Inversely, solid fractions obtained after phase separation contained a substantial part of remaining biodegradable organic matter with a significant easily accessible fraction comprising simpler molecules. Understanding the effect of post-treatment on the biodegradability of digestates should help to optimize their valorization.

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**Abbreviations:** AD, anaerobic digestion; AT<sub>4</sub>, cumulative oxygen uptake in four days ( $\text{gO}_2 \text{kg}^{-1} \text{VS}$ ); BDaero, aerobic biodegradability (% CODtot); BDana, anaerobic biodegradability (% CODtot); BMP, biomethane potential ( $\text{NLCH}_4 \text{kg}^{-1} \text{VS}$ ); BOD, biological oxygen demand ( $\text{gO}_2 \text{kg}^{-1} \text{VS}$ ); CO<sub>2</sub>, carbon dioxide; CODtot, total chemical oxygen demand ( $\text{gO}_2 \text{kg}^{-1} \text{VS}$ ); DOM, dissolved organic matter; DRI<sub>24</sub>, average O<sub>2</sub> uptake rate in the 24 h of maximum activity ( $\text{mgO}_2 \text{h}^{-1} \text{kg}^{-1} \text{VS}$ ); MB, easily biodegradable fraction (%BDaero); MH, slowly biodegradable fraction (%BDaero); MSW, municipal solid waste; NEOM, non extractable organic matter (% CODtot); O<sub>2</sub>, oxygen; OFMSW, organic fraction of municipal solid waste; OM, organic matter; OUR, oxygen uptake rate ( $\text{mmO}_2 \text{h}^{-1} \text{kg}^{-1} \text{VS}$ ); PEOM, poorly extractable organic matter (% CODtot); REOM, readily extractable organic matter (% CODtot); RT, retention time; SEOM, slowly extractable organic matter (% CODtot); SPOM, soluble extractable fraction from particular extractable organic matter (% CODtot); TS, total solid; VS, volatile solid.

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## 1. Introduction

Anaerobic digestion (AD) is a biological treatment that converts organic waste into methane (CH<sub>4</sub>). It can thus produce energy, while the digestate is regarded as a potential organic fertilizer. The digestate is a fully fermented nutrient-rich material which can ideally replace inorganic fertilizer (Tambone et al., 2010). Many studies focus on the optimization of CH<sub>4</sub> production from organic waste but rarely on the assessment of the agronomic value of digestate and its valorisation through land application (Teglia et al., 2010). At times, when digestate is not fully stabilized, it can present residual biodegradability and contain complex organic elements such as ligno-cellulosic compounds which the digestion process cannot degrade (Bayard et al., 2015). Consequently, post-treatments such as solid-liquid separation, drying or composting, are generally recommended before land application and the fertilizer produced should also fulfil current standards, *i.e.* efficiency and safety (Teglia et al., 2011).

To be used as an organic fertilizer, stability or biodegradability of a digestate has to be fully assessed prior to land application. The biodegradability of organic residues can be evaluated using (i) biological methods including aerobic respirometric tests and anaerobic test methods (Binner and Zach, 1999; Wagland et al., 2009) and (ii) non-biological methods including dry matter, organic matter (OM) and total organic carbon content (Godley et al., 2004). The use of the biomethane potential test (BMP) aims at assessing the biogas potential of both organic waste and digestate. It is the most common method applied for measuring the anaerobic biodegradability of organic residues (Schievano et al., 2008). However, the BMP test protocol can vary from one laboratory to another (Raposo et al., 2011) thus leading to large discrepancies. It is also a time-consuming method (>30 days). For these reasons, alternative approaches such as respirometric tests have been considered (Cossu and Raga, 2008; Scaglia et al., 2010). These aerobic methods are based on the oxygen (O<sub>2</sub>) measurement uptake or carbon dioxide (CO<sub>2</sub>) production which are related to OM biodegradation under controlled conditions. As organic residues can either be directly applied on land or stored prior to their application, they can be exposed to both aerobic and anaerobic environments. An assessment of their biodegradability under aerobic and anaerobic conditions thus provides complementary information on their characteristics. Furthermore, correlations between aerobic and anaerobic tests have been reported by several authors, especially for organic waste before its biological treatment, such as AD processes or composting (Cossu and Raga, 2008; Ponsá et al., 2008; Barrena et al., 2009; Böhm et al., 2010; Liu et al., 2015). However, information is scarce concerning such correlations on digestates.

The objectives of this study are (i) to investigate the biodegradability under both anaerobic and aerobic conditions of 14 post-treated digestates (seven composted or dried solid fractions of digestate and seven solid fractions of digestate obtained after liquid-solid separation) and (ii) to characterize their OM in order to better assess the impact of post-treatments on their characteristics and stability. In this view, a recent method developed to characterize both the accessibility and complexity of organic wastes (Jimenez et al., 2015) has been applied. This method combines successive chemical fractionations to 3D fluorescence spectroscopy. The final goal was correlating biodegradability and OM characterization and describing the effect of post-treatment on the digestate stability. This could help to define better strategies for agricultural re-use of these organic residues.

## 2. Material and methods

### 2.1. Sampling of digestates

Fourteen digestates were sampled from 13 AD sites in France: 7 solid fractions of digestate (SL1S, SL2S, A1S, T1S, T2S, T3S, T4S) obtained following solid-liquid phase separation, 1 dried and composted solid fraction of digestate (A2D) and 6 composted solid fractions of digestate (A3C, A4C, SL1C, O1C, O2C, M1C). The AD sites treated various types of waste, including agricultural waste, sewage sludge from a wastewater treatment plant (WWTP), municipal solid waste (MSW), the organic fraction of MSW (OFMSW), food processing waste (FPW), all according to different digestion processes (wet or dry, mesophilic or thermophilic). The designation of the digestates and their origins are presented in Table 1. All of the samples collected (about 50 kg each) were homogenized for further analyses.

### 2.2. Physico-chemical characterisation of digestates

Representative aliquots of digestate (about 2 kg) were used to perform all the analytical tests. For each sample, the total solid (TS) content was measured by drying 100 g of sample at 105 °C for 48 h. The volatile solid (VS) content was measured after calcination of this dried sample at 550 °C for 3 h. Prior to total chemical oxygen demand (COD<sub>tot</sub>) measurements, the dried sample was ground to a particle size of 500 µm. Then, COD<sub>tot</sub> was determined by titration after digesting 1 g of TS with H<sub>2</sub>SO<sub>4</sub> and K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in accordance with the AFNOR standard NF T90-101. COD<sub>tot</sub> results were expressed in gO<sub>2</sub> kg<sup>-1</sup> VS. All measurements were performed in triplicate.

### 2.3. Residual biodegradability assays

#### 2.3.1. Aerobic biodegradability assay (BDAero)

The aerobic biodegradability was measured on fresh digestates (between 2 and 4 kg according to the bulk density) using a dynamic respirometric method (Berthe et al., 2007). The respirometric device consisted of 10 L hermetically sealed stainless steel cells. These were filled with the studied substrates mixed with plastic Pall rings acting as inert bulking agent. Each cell was maintained at 40 °C by means of a water bath and was supplied with a continuous air flow rate of 70 L h<sup>-1</sup>. The incoming air was pre-heated to the water bath temperature while the aeration system also included a rapid recirculation of part of the exhaust air (360 L h<sup>-1</sup>), thus ensuring homogeneous conditions throughout the substrate. In order to measure the O<sub>2</sub> uptake rate (OUR) during the respirometric experiment, the oxygen concentrations of the inlet and outlet gases were monitored every 2 min using a paramagnetic analyser (Magnos 206, ABB, Zurich, Switzerland). The respirometric test was finally halted when the O<sub>2</sub> uptake rate fell to a low and stable value.

The biological oxygen demand (BOD) of the digestate corresponded to the cumulative O<sub>2</sub> uptake during the test. Two classical aerobic indices were also calculated for each substrate: AT<sub>4</sub> related to the cumulative O<sub>2</sub> uptake in four days and DRI<sub>24</sub> related to the average OUR in the 24 h of maximum activity (Ponsá et al., 2010). Moreover, the aerobic biodegradability (BDAero) could be expressed as the ratio between BOD and COD<sub>tot</sub>, according to Eq. (1) (Liu et al., 2015).

$$\text{BDAero}(\% \text{COD}_{\text{tot}}) = (\text{BOD}(\text{gO}_2 \text{kg}^{-1} \text{VS}) \times 100) / \text{COD}_{\text{tot}}(\text{gO}_2 \text{kg}^{-1} \text{VS}) \quad (1)$$

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