

Monitoring the organic matter properties in a combined anaerobic/aerobic full-scale municipal source-separated waste treatment plant

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Abstract

Respiration indices (dynamic and cumulative) and the anaerobic biogasification potential are applied to the quantitative calculation of the biodegradation efficiency in a combined anaerobic/aerobic treatment for the organic fraction of municipal solid waste (OFMSW). They also permit to observe possible deficiencies in some parts of the entire sequence of organic matter decomposition. On the contrary, chemical methods presented a limited utility. Dynamic respiration indices highlighted that anaerobic digestion was the most efficient step to reduce the respiration activity of the waste (61% calculated on a $\text{DRI}_{24\text{h}}$ basis). Respirometric activity of final compost was 93% lower than initial OFMSW confirming the overall efficiency of the plant studied and the stability of the final product ($0.3 \text{ g O}_2 \text{ kg TS}^{-1} \text{ h}^{-1}$). Finally, the use of an advanced methodology such as the Diffuse Reflectance Infrared Fourier Transformed (DRIFT) allows the determination of the main functional groups of organic matter, which significantly change during the biological treatment of organic matter.

Keywords: Anaerobic digestion; Stability; Composting; DRIFT; Organic fraction of municipal solid waste; Full-scale plant.

1. Introduction

New strategies in municipal solid waste (MSW) management, i.e., source-separate collection of the organic fraction (OFMSW) (EU Directive 1999/31/EC), and the need to reduce the biodegradable-MSW fraction allocated in landfills have favoured the development of composting and anaerobic digestion as useful biotechnologies for transforming organic wastes into suitable agricultural products. Nowadays the number of OFMSW treatment plants that use anaerobic digestion technology coupled to a composting process is increasing (Pognani et al., 2009).

The analysis of a waste treatment plant efficiency requires a reliable measure of the biodegradable organic matter content of waste samples and thus, respirometric indices and anaerobic assays as anaerobic biogas potential (ABP) can be useful to indicate the quantity of readily biodegradable organic matter that has really been decomposed during the process. Biological activity measurements have been suggested in the literature as a measure of quality (stability) of the final product (Ianotti et al., 1993; Adani et al., 2004).

Spectroscopic techniques are suitable to distinguish biochemical components in organic materials. In particular DRIFT (Diffuse Reflectance Infrared Fourier Transform) is a simple technique, which is rapidly performed, non-destructive and does not require sample pre-treatment. This spectroscopic technique has been also used to study the transformation of humic substances during composting of various organic wastes and to determine the maturity degree of the products (Tseng et al., 1996; Jenn-Hung and Shang-Lien, 1999). However, to our knowledge, it has never been applied to follow the evolution of the main functional groups of organic matter in a complex waste treatment plant as the one described in this work, which includes anaerobic digestion and composting.

The objectives of this study were: to use the aerobic respiration index and the ABP as a quality index for monitoring the process for the stabilization of the biodegradable organic

matter of the OFMSW and to study if the advanced spectroscopic technique (DRIFT) can be used as a index of stability during the process by following the evolution of the functional groups in organic matter.

2. Materials and methods

2.1. Organic waste treatment plant

In 2008 a complete monitoring of a full-scale plant located in Barcelona (Spain) was done. This plant treats around 25,000 Mg OFMSW yr⁻¹ coming from a street bin source-separated collection system. The plant has two types of rejected materials: pre-treatment and compost rejects. All the rejected materials together correspond to 0.41 Mg Mg⁻¹ OFMSW and they are disposed in a sanitary landfill. The plant uses a small amount of diatomaceous earth waste with vegetal grease from the biodiesel industry as co-substrate to improve the anaerobic digestion performance. This material reduces the moisture of the OFMSW and adds a plus of organic carbon to the anaerobic digestion. Diatomaceous earth waste is added to OFMSW before the pre-treatment at ratio 1:10 w.w. This co-substrate was characterized by a high fat content (409 ± 209 g kg⁻¹ TS) and practically no nitrogen content.

Before its anaerobic digestion, OFMSW plus diatomaceous earth is treated to remove plastic and inorganic materials such as metal, glass and stones, which are rejected. The anaerobic digestion process is based on the DRANCO (DRy ANaerobic Composting, OWS, Belgium) technology. It is a dry process performed at thermophilic temperature (50-55°C). The digester mixing is provided by the recirculation of digested material (digestate). The retention time is set at 22 days and the digester capacity is 1700 m³.

The material coming from the anaerobic digester is mixed with bulking agent (1:4 v/v digestate/bulking agent) and composted in a tunnel composting system (5 tunnels) during 7 days. As it is typical in composting process, the bulking agent is added and mixed before

filling the composting tunnels to improve porosity and reduce moisture of the digestate. Bulking agent consists of shredded wood and vegetal fraction. Semi-composted material is transferred to a maturation area for 1 or 2 weeks (windrow process). The final compost is treated using a trommel screen (10 mm of cut-off) to remove the residual bulking agent and residual impurities before being stocked and commercialized. Leachate (TS of $50 \pm 20 \text{ g kg}^{-1}$ w.w. and VS of $500 \pm 70 \text{ g kg}^{-1}$ TS) is sent to an external wastewater treatment plant for treatment. The production is around 77 L Mg^{-1} .

2.2. Samples collection

Samples were collected from the most significant points of the plant. The samples selected for the study of the plant were: input material (OFMSW), OFMSW mixed with diatomaceous earth, the input to the anaerobic digester, digestate, material from the composting tunnels, material from composting windrow maturation, final compost and the two main rejects (Tables 1 and 2).

2.3. Analytical methods

Analytical methods were carried out on a representative sample (approximately 20 kg) obtained by mixing four sub-samples of about 5 kg each, taken from different points of the bulk material. Samples were grinded to 15 mm particle size to obtain representative samples. The samples were frozen at $-18 \text{ }^{\circ}\text{C}$ within 12 hours after sampling. Before each analysis samples were thawed during 24 hours at room temperature. These representative samples were used to carry out all the analytical tests. pH, Electrical Conductivity (EC), Total Solids (TS), Volatile Solids (VS), Total Organic Carbon (TOC), Total Kjeldahl Nitrogen (TKN), N-NH_4^+ (measured on fresh material), and fat contents were determined according to the standard procedures by The US Composting Council: Test Methods for the Examination of Composting and Compost (TMECC, 2001). All tests were run in triplicate and the results are presented as an average value followed by standard deviation.

2.4. *Respirometric tests*

The procedure established in this study for the determination and calculation of dynamic respiration index (DRI) and cumulative respiration activity after 4 days (AT_4) was based on previous works by Adani et al. (2004; 2006a) and Barrena et al. (2005).

2.5. *Anaerobic biogas potential (ABP)*

The waste samples were mixed at a 1:1 ratio (dry basis) with the inoculum except for fresh materials like: OFMSW, OFMSW plus diatomaceous earth and input of anaerobic digester where the ratio was 1:4 (dry basis) to avoid the inhibition risk for accumulation of volatile fatty acids (VFA). Water was added to correct the TS of the mixture. Inoculum in stable methanogenic activity (methane content > 60% in biogas, v/v) was obtained using the output of a full-scale anaerobic mesophilic digester located in Montcada i Reixac (Barcelona, Spain) and was incubated during two weeks at 37°C before use (Schievano et al., 2008; Pognani et al., 2009). The results obtained at 21 days (ABP_{21}) and at the end of the test (100 days, ABP_{100}), were expressed as biogas volume (L) produced and measured at normal conditions per kg of initial TS. Biogas was analyzed according to Ponsá et al., 2008.

2.6. *DRIFT spectroscopy methodology*

Waste samples were analyzed by the Diffuse Reflectance Infrared Fourier Transformed (DRIFT) spectroscopy using an Avatar 370 FT-IR ThermoNicolet Instruments (Madison, WI, USA). Samples (7 mg), previously dried at 65 °C for 48 h, and KBr (700 mg; FT grade, Aldrich Chemical Co, ST Louis, Missouri) were finely ground in a Wig-L-Bug (Specamill-Greseby-Specac, Kent, UK) for 10 minutes, using an agate ball mill. Measures were set up using instrument parameters at the following conditions: scanning 128, resolution 4 cm^{-1} , frequency 400-4000 cm^{-1} and gain 16. As background a spectrum of finely powdered potassium bromide was used. The spectrums were obtained in absorbance units and

successively analyzed by the EZ OMNIC® software (Thermo Nicolet Corporation Madison, WI, USA).

3. Results and discussion

3.1. Chemical properties

The chemical composition of the samples studied is reported in Table 1. The OFMSW is characterized by an acidic pH (5-5.5) as a consequence of the production of volatile fatty acids due to the anaerobic processes occurring in the plastic bags in which the material is stored before its collection (Adani et al., 2006a). After the arrival of the OFMSW, diatomaceous earth is added. This storage time and the addition of diatomaceous earth (VS of $540 \pm 210 \text{ g kg}^{-1}$ TS) caused a drop of VS (13%). This was probably due to a consumption caused by microbiological activity that happened into the plastic bags (Adani et al., 2006a) and a dilution of the VS caused by the addition of diatomaceous earth. The OFMSW with the addition of diatomaceous earth passed through the mechanical pre-treatment process before being introduced in the anaerobic digester. This step did not determine significant changes in respiration activity of the waste material.

The anaerobic digestion step, the decrease of VS content was 28% and corresponded to the highest reduction observed in the points analyzed in this waste treatment plant. Total nitrogen was higher at the output of anaerobic digestion probably due to the concentration effect of ammonia during the digestion process (Schievano et al., 2008).

Bulking agent was added to digestate with the aim to create a porous structure and promoted the air circulation through the biomass (Ruggieri et al., 2009). Composting material temperature reached 55-60°C during three weeks (tunnels and windrow phase) achieving a significant water evaporation, which in the last days was convenient for sieving purposes. During tunnel phase the material also lost a great quantity of ammonia (85%).

3.2. Evolution of respiration indices and ABP

The results of the evolution of the respiration indices and the ABP tests are shown in Table 2. The values presented of $\text{DRI}_{24\text{h}}$, AT_4 and ABP are expressed on a dry matter basis to help to the comparison of values, because the organic matter content of the sample varied significantly as the biodegradation process occurred (Barrena et al., 2005; Ponsá et al., 2008).

Evolution trends indicated a progressive stabilization of the material. Respiration indices found for the OFMSW are high, which is expected since this fraction contains a higher content of labile organic compounds (Ponsá et al., 2008). Anaerobic digestion was the main step to reduce the biodegradable organic matter of the waste. $\text{DRI}_{24\text{h}}$ presented a decrease of 61% (calculated on a dry basis) and a cumulative oxygen consumption (AT_4) decrease of 64% (calculated on dry basis), which is in agreement with the results obtained by Ponsá et al. (2008). These results indicate that there is a significant loss of biodegradable organic matter during the digestion (78% for the OFMSW calculated on a $\text{DRI}_{24\text{h}}$ basis). This is in accordance with previous studies (Ponsá et al., 2008), where reductions of biological respiration near 70% were reported. In the composting step, respirometric activity decreased around 50% during the tunnel phase and 50% during the final maturation process. Final compost showed a respirometric activity higher than maturation step due to the refining process that caused a concentration of organic matter by the separation of non-degraded bulking agent and the residual inorganic impurities. This phenomenon has been observed in other experiences when refining compost from the OFMSW (Ruggieri et al., 2008).

Aerobic and anaerobic indices showed similar evolution profiles. In fact, they can be correlated and the correlation obtained is statistically significant (for instance, R^2 of 0.99 between $\text{DRI}_{24\text{h}}$ and ABP_{21} , $p < 0.01$). These results have been recently confirmed in other MBT plant configurations (Ponsá et al., 2008; Barrena et al., 2009). To confirm this point, Anova test was performed to compare $\text{DRI}_{24\text{h}}$ and ABP_{21} data. Anova test resulted statistically

significant so a multiple comparison test (Tukey test; $\alpha=0.05$) was carried out. Tukey test confirmed that all data related to samples obtained before the anaerobic digestion step were significantly different but not for the composting process (tunnel and windrow phases), which could be related to its short duration.

Respirometric activity of final compost was 93% lower than that of the OFMSW, which means that the studied plant has a great stabilization capacity and that the design is correct. Adani et al. (2004) reported biological stability values for well matured compost of DRI of 1000-500 mg O₂ kg VS⁻¹ h⁻¹. In this study, DRI_{24h} of the final product was 730±332 mg O₂ kg VS⁻¹ h⁻¹, which is within the range of well matured and stable compost.

The anaerobic index used (ABP) indicated that it can be also used for the monitoring of the biodegradation of organic matter in waste treatment plants, since it also reflects the progressive stabilization of organic matter (Table 2). Moreover, if values of ABP₂₁ are compared with ABP₁₀₀, it can be stated that the level of biogas produced at 21 days (samples before anaerobic digestion) is within the range of 85-90% of the overall biogas production at 100 days and for the samples after anaerobic digestion the range was around 65-70%, which is similar to the results previously obtained (Ponsá et al., 2008). These results are of interest because both times related to ABP tests have been proposed as European methods to aid in the evaluation of the diversion of biodegradable MSW from landfill (Wagland et al., 2009).

About the efficiency of the plant, the results suggest that although a good stabilization is achieved in the final compost, the biological activity in the pre-treatment reject was very high (DRI of 3.4 mg O₂ kg TS⁻¹ h⁻¹), indicating that highly biodegradable organic matter is being landfilled (Tables 1 and 2). In general, it can be concluded that biological stability indices in their several forms (dynamic or cumulative, aerobic or anaerobic) are strongly recommended when the overall efficiency of a waste treatment plant is to be evaluated (Cossu

et al., 2008; Ponsá et al., 2008; Barrena et al., 2009). In terms of analysis time, however, aerobic respiration indices are preferable because of the long time needed in ABP.

3.3. DRIFT spectroscopy

DRIFT spectroscopy was used as a qualitative tool to identify the main functional groups in the initial sample and during the treatment process. This approach can provide useful information on the functional groups of these metabolites and their physicochemical features (Carballo et al., 2008). The spectra obtained for samples were well resolved and allowed the various functional groups to be differentiated.

The attribution corresponding to the most significant bands are assigned according to Socrates (1980), Pandey (1998) and Adani et al. (2006b). In this work, only the most significant DRIFT spectra are shown and commented. DRIFT analysis indicated that the OFMSW was mainly composed by bio-macromolecules (Fig. 1 Spectra a): i. polysaccharides: large band between 3600 cm^{-1} and 2400 cm^{-1} and peaking around 3280 cm^{-1} indicated the presence of -OH group of carbohydrates, and the band centred at 1070 cm^{-1} , due to the C-O stretching of polysaccharides; ii. aliphatic molecules: peaks at 2924 cm^{-1} and 2854 cm^{-1} attributed to the C-H stretching and symmetric and asymmetric vibration of $-\text{CH}_2$ and $-\text{CH}_3$ groups indicating the presence of aliphatic acids. O- CH_3 stretching is typical of methyl ether and the peak at 1746 cm^{-1} is due to the stretching of C=O of aliphatic acids (e.g. carboxylic acid); iii. protein-like molecules: the band between 3600 cm^{-1} and 2400 cm^{-1} centred at 3280 cm^{-1} indicated the -NH group of protein structures, the peak at 1655 cm^{-1} attributed to the stretching of C=O of amide (amide I) and the peak at 1539 cm^{-1} indicative of deformation of N-H bond and stretching of C=N in amide (amide II); iv. lignin-like molecules: peak at 2854 cm^{-1} due to O- CH_3 stretching as typical of lignin methoxyl group, whereas the large band at $1300\text{-}1200\text{ cm}^{-1}$ attributed to the interaction between deformations of OH bonds and stretching C-O bonds, indicating the presence of phenols (i.e. lignin like molecules); finally,

the broad band at 1500 and 1400 cm^{-1} was attributed to C-H deformations of polysaccharides but also to lignin-like molecules.

In relation to the process plant, the first fact to mention is that the addition of diatomaceous earth to the OFMSW did not determine modifications of the spectra (corresponding spectra is not shown). However, sample before the anaerobic digestion process led to an increase of the aliphatic molecules contents (see peaks 2925 cm^{-1} and 2854 cm^{-1} associated at 1744 cm^{-1} peak) as consequence of the hydrolysis of the substrate and of the successive fermentation to produce fatty acids (e.g. acetic acid) (Fig. 1, Spectra b).

Anaerobic digestion produced a strong reduction of the aliphatic fraction contents (see the reduction of the peaks at 2925 and 2852 cm^{-1} and 1744 cm^{-1}). This is in agreement with the fact that anaerobic digestion proceeded by transforming volatile fatty acids into CH_4 and CO_2 . On the other hand, peaks of polysaccharides became more evident (see broad band at 1200-900 cm^{-1} picking at 1044 attributed to C-O stretching of polysaccharide). Aromatic compounds until this point can be identified at the broad band at 1500 and 1400 cm^{-1} that was attributed to C-H deformations of lignin-like molecules combined at the peak at 877 cm^{-1} (C-H stretching of aromatics rings) (Fig. 1 Spectra c).

The successive composting process (tunnel phase) determined a further reduction of the aliphatic molecules content (peaks at 2925 cm^{-1}) (Fig. 1 Spectra d). Moreover, the addition of a lignocellulosic material as a bulking agent to the digested material to prepare the composting mixture led to an increase of the lignocellulosic material (bands 1200-900 cm^{-1}). Finally, the spectra of composted mixture sampled during the maturation (windrow phase) did not show significant differences (corresponding spectra is not shown). Also, the successive sieving of compost did not change significantly the compost spectra (Fig. 1 Spectra e).

The results obtained with DRIFT method show the expected degradation of the organic matter and the change to other structures more stable. Although other authors have

applied the DRIFT tool to characterize compost from several wastes (Mamiro et al., 2008; Som et al., 2009), this is, to our knowledge, the first time in which is reported for the source-separated OFMSW and its evolution along a complete waste treatment process.

4. Conclusions

The study presented has demonstrated that both aerobic and anaerobic indices can be used to estimate the stabilization of organic matter during all the treatment sequence for the OFMSW. From this sequence, it can be concluded that anaerobic digestion is the main step to reduce the biological activity of the waste sample. Finally, DRIFT spectra can be a good opportunity to investigate how the organic matter changes after every process step by following the changes of the functional groups.

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Tables

Table 1: Chemical characterization of the waste samples. Different letters indicate significant differences according to ANOVA and Tukey tests ($\alpha=0.05$).

Sample*	pH	EC (mS cm ⁻¹)	TS (g kg ww ⁻¹)	VS (g kg TS ⁻¹)	TOC (g kg TS ⁻¹)	TKN (g kg TS ⁻¹)	N-NH ₄ ⁺ (g kg ⁻¹ TS)	N _{tot} (g kg TS ⁻¹)
OFMSW (as received)	5.3±0.6a	5.6±0.3a	280±50a	780±110a	60±22a	26±2a	0.6±0.2a	27±2a
Diatomaceous earth	4.3±1.3b	8.4±0.3b	810±80b	540±210b	48±14b	0b	0b	0b
OFMSW + diatomaceous earth	5.8±0.8a	3.3±0.3c	330±30a	680±140c	44±03b	14±7c	0.3±0.1c	15±7c
Pretreatment reject	5.7±0.6a	5.0±0.1d	390±100a	510±20d	56±02b	13±7c	0.4±0.1c	14±8c
Anaerobic digestion input	5.5±0.4a	5.0±0.4d	430±60a	580±30d	50±10b	16±3c	1.4±0.3d	18±3c
Anaerobic digestion output	8.2±0.2c	5.4±0.8d	320±40a	420±10d	50±10b	17±6c	11±1.0e	27±9d
Composting (tunnels)	8.6±0.1c	4.1±0.3d	540±30c	440±30d	42±8b	12±4c	1.7±0.3f	15±6e
Maturation (windrows)	8.8±0.2c	3.9±0.8d	590±30c	400±10d	40±4b	13±5c	1.1±0.1f	15±4e
Final Compost	8.6±0.1c	5.0±0.4d	600±10c	400±20d	50±10b	15±3c	1.5±0.5f	17±4e
Compost Reject	8.7±0.1c	4.5±0.1d	620±10c	350±20d	43±6b	11±2c	2.5±0.9f	14±1e

*Abbreviations: OFMSW: Organic Fraction of Municipal Solid Waste; EC: Electrical Conductivity; TS: Total Solids; VS: Volatile Solids; TOC: Total Organic Carbon; TKN: Total Kjeldahl Nitrogen; N_{tot}: Total Nitrogen.

Table 2: Respiration indices and anaerobic biogas potential of the waste samples. Different letters indicate significant differences according to ANOVA and Tukey tests ($\alpha=0.05$). Columns five and seven indicate the average methane content during the ABP₂₁ and ABP₁₀₀, respectively.

Sample*	DRI _{24h} (g O ₂ kg TS ⁻¹ h ⁻¹)	AT ₄ (g O ₂ kg TS ⁻¹)	ABP ₂₁ (NL kg TS ⁻¹)	CH ₄ (%)	ABP ₁₀₀ (NL kg TS ⁻¹)	CH ₄ (%)
OFMSW (as received)	4.2±1.2a	298±114a	393±7a	66±5a	441±36a	66±4a
Diatomaceous earth	0.14±0.1b	10.6±0.5b	614±72b	72±8b	724±112b	70±5a
OFMSW + diatomaceous earth	3.5±1.4a	235±99a	303±22a	62±1a	338±36a	62±1a
Pretreatment reject	3.4±1.1a	261±44a	343±67a	65±6a	391±70a	65±5a
Anaerobic digestion input	2.3±0.8a	179±61a	384±28a	65±5a	434±26a	64±4a
Anaerobic digestion output	0.9±0.1c	61±2c	56±15c	67±4a	81±20c	66±4a
Composting (tunnels)	0.4±0.2d	20±8d	20±6d	66±3a	35±8d	66±3a
Maturation (windrows)	0.2±0.1d	16±6d	22±7d	67±4a	34±10d	63±4a
Final Compost	0.3±0.1d	21±10d	24±9d	66±3a	41±14d	65±3a
Compost reject	0.2±0.1d	12±4d	21±3d	70±1a	33±5d	69±1a

*Abbreviations: OFMSW: Organic Fraction of Municipal Solid Waste; DRI: Dynamic Respiration Index; AT₄: Cumulative Respiration Index (4 days); ABP₂₁: Anaerobic Biogasification Potential (21 days); ABP₁₀₀: Anaerobic Biogasification Potential (100 days).

Figure 1: DRIFT analysis of the waste samples. Spectra a: OFMSW; spectra b: anaerobic digestion input; spectra c: anaerobic digestion output; spectra d: composting process (tunnel phase); spectra e: final compost.

