

Composting of De-inking Sludge from the Recycled Paper Manufacturing Industry

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Abstract

Composting of two different types of sludge from the recycled paper manufacturing industry was carried out at laboratory scale. Physico-chemical sludge (PCS) from the de-inking process and biological sludge (BS) from the wastewater treatment plant were composted and co-composted with and without addition of a bulking material. Despite its poor initial characteristics (relatively high C/N ratio, low organic content and moisture), PCS showed excellent behaviour in the composting process, reaching and maintaining thermophilic temperatures for more than seven days at laboratory scale, and therefore complete hygienization. Pilot-scale composting of PCS was also studied, and a respiratory quotient of 1.19 was obtained, indicating a full aerobic biological process. Respiration tests showed a complete stabilization of the material, with final values of the static respiration index in the range of $1.1 \text{ mg O}_2 \cdot \text{g TOM}^{-1} \cdot \text{h}^{-1}$. Composting is proposed as a suitable technology for the effective recycling of this type of sludge from the recycled paper manufacturing industry.

Keywords: C/N ratio, Composting, Hygienization, Recycled Paper Manufacturing Sludge, Respiratory Quotient.

Introduction

In recent years, new legislation in the European Union and the United States has promoted the utilization of recycled fibres in newsprint. This fact, together with the implementation of source-separated waste paper collection programs, has changed the raw materials in the paper manufacturing industry. In Spain, some industries are solely accepting waste paper to transform it into recycled paper.

Recycled paper industries remove inks, clay filters and coatings of used paper by a de-inking process and recycle the wood fibres by using physico-chemical treatments. However, some wood fibres are rejected from this process and constitute a sludge with some organic content. Moreover, this type of industry usually generates biological sludge from the biological treatment of wastewater.

Since the majority of sludge from paper manufacturing industries is landfilled or incinerated, alternative methods to treat this waste are being developed. Composting is one of the most promising technologies to treat paper sludge in a more economical way (Das *et al.*, 2002a). It is defined as the biological decomposition and stabilization of organic substrates, under controlled conditions (Haug, 1993). The composting process permits the hygienization of the product by reaching thermophilic temperatures and reducing mass and volume, which makes compost suitable for agricultural applications.

Previous works have studied the feasibility of the composting of sludge from different pulp and paper manufacturing industries. Jokela *et al.* (1997) studied the aerobic and anaerobic digestion of pulp and paper mill sludge, concluding that in the case of de-inking sludge composting some urea addition is necessary to adjust the initial C/N ratio. In fact, C/N ratio appears to be one of the most crucial parameters to adjust in the composting of lignocellulosic wastes. Thus, nitrogen-rich amendments such as chicken broiler floor litter or poultry manure (Charest and Beauchamp, 2002) or

chemicals such as ammonium nitrate (Das *et al.*, 2002b), ammonium sulphate (Paul *et al.*, 1999) or urea (Jokela *et al.*, 1997) are often added to the sludge in order to decrease the initial C/N ratio. However, some recent works have pointed out that in some cases the composting of paper and pulp manufacturing sludge can be successfully carried out at C/N ratios higher than those currently used with other wastes (*e.g.* organic fraction of municipal solid waste or sludge from wastewater treatment plants). Besides, in these cases the amendment with nitrogen-rich wastes may not be necessary (Larsen, 1998; Charest and Beauchamp, 2002).

Other works have focused on particular aspects of paper sludge composting such as the optimization of decomposition rate (Ekinci *et al.*, 2002) or the microbial activities during composting of pulp and paper-mill primary solids, revealing a particular microbial community in the biodegradation of such wastes (Atkinson *et al.*, 1997). The application of composts from paper and pulp manufacturing wastes has also been studied and validated in soil and crops (Hackett *et al.*, 1999; Baziramakenga and Simard, 2001; Rantala and Kuusinen, 2002).

This paper describes an investigation of the possibility of composting and co-composting the most typical wastes produced in the recycled paper manufacturing industry, PCS (physico-chemical sludge) and BS (biological sludge). The work consisted of an initial set of laboratory scale experiments to explore the compostability of different mixtures of paper sludges and a second pilot scale experiment where biological indices were determined for the optimal mixture. This methodology can be generalized for the study of similar organic wastes for which few data are available.

Materials and Methods

Sludge and bulking agent

PCS and BS were collected from a recycled paper manufacturing industry in Spain. PCS was obtained after centrifugation of the liquid fraction of the waste paper de-inking process. BS was obtained after the centrifugation of the biological sludge generated in the wastewater treatment plant of the recycled paper manufacturing industry. In this particular industry, PCS is produced in significantly larger amounts than BS. The main parameters of PCS and BS collected in the industry are presented in Table 1.

Wood chips from a local carpentry were used as bulking agent. The chips consisted of a variable mixture of pine and beech tree wood.

Composting experiments

Laboratory-scale experiments were undertaken using 4.5-L Dewar® vessels conditioned for composting and previously validated in the composting of organic fraction of municipal solid waste and wastewater sludge (Gea *et al.*, 2003). A perforated lid was fitted for temperature monitoring and air supply and a rigid wire net was placed near the bottom of the vessel to separate the composting material from possible leachates.

Pilot tests were undertaken in an old 100-L refrigerator adapted for use as a static composter. The recipient was placed horizontally with a slight inclination to allow its opening from the top and to permit the collection of leachates. A plastic mesh was fitted at the bottom of the recipient to support the material and separate it from possible leachates. Several holes were perforated through the walls of the vessel to permit air movement, leachate removal and the insertion of different probes. Air was supplied to the composter by means of control software to maintain an O₂ concentration over 10%.

Temperature, O₂ and CO₂ monitoring

Laboratory scale: Pt-100 sensors were used for temperature monitoring in the 4.5-L Dewar vessels placed in the material to have a measuring point at 1/2 of the height of the material in the vessel. Temperature sensors were connected to a data acquisition system (DAS-8000, Desin, Spain) which was connected to a standard personal computer. The system allowed, by means of the proper software (Proasis® Das-Win 2.1, Desin, Spain), the continuous on-line monitoring and recording of the temperature. O₂ content was measured with a portable O₂ detector (Oxy-ToxiRAE, RAE) with a frequency of 3-7 times during one day.

Pilot scale: Four Pt-100 sensors (Desin mod. SR-NOH) inserted at different points inside the 100-L tank were used for monitoring the temperature in the pilot scale composting experiments. Temperature was recorded every 30 minutes. Interstitial air was pumped out of the reactor every 10 minutes and sent for O₂ and CO₂ measurement to an oxygen sensor (Sensox, Sensotran, Spain) and a CO₂ infrared detector (Sensortran I.R., Sensotran, Spain) respectively. All sensors were connected to a specially-made data acquisition system. Oxygen was controlled by means of a feedback oxygen control which automatically supplied fresh air to the reactor (flow rate 20 L/min) to maintain an oxygen concentration over 10%. Measures of temperature and O₂ and CO₂ content showed a high level of reproducibility in laboratory and pilot experiments, with a deviation of less than 1%.

Respiratory Quotient (RQ)

RQ was calculated as the quotient of CO₂ produced and O₂ consumed as indicated in Equation 1:

$$RQ = \frac{CO_{2,out}}{20.9 - O_{2,out}} \quad (\text{Eq. 1})$$

where: RQ, respiratory quotient (dimensionless); $CO_{2,out}$, carbon dioxide concentration in the exhaust gases (%); $O_{2,out}$, oxygen concentration in the exhaust gases (%). CO_2 percentage in inlet air was considered negligible and O_2 concentration in inlet air was 20.9%. RQ is presented as an average of 10 values taken over 100 minutes of measurement.

Analytical methods

Water content, total organic matter (TOM), pH, electrical conductivity, total nitrogen (Kjeldahl method), $N-NH_4$ and compost maturity grade (Dewar self-heating test) were determined according to the standard procedures (U.S. Department of Agriculture and U.S. Composting Council, 2001). Cellulose content was determined according to the method proposed by Rivers *et al.* (1983).

Total weight of the material was monitored on-line using a semi-industrial scale BACSA mod. I200.

Respiration tests

A static respirometer was built according to the original model described by Ianotti *et al.* (1993) and following the modifications and recommendations given by the U.S. Department of Agriculture and U.S. Composting Council (2001). Approximately 250 mL of compost samples were placed in 500 mL Erlenmeyer flasks on a nylon mesh screen that allowed air movement under and through the solid samples. The setup included a water bath to maintain the temperature at 37°C during the respiration test. Prior to the assays, samples were incubated for 18 hours at 37°C. Samples were aerated with previously humidified air at the sample temperature throughout the incubation

period. The drop of oxygen concentration in each flask containing a compost sample was monitored with a dissolved oxygen meter (Lutron 5510, Lutron Co. Ltd., Taiwan) connected to a data logger. The rate of respiration of the compost sample (Oxygen Uptake Rate, OUR or Respiration Index, RI) based on total organic matter content, TOM) was then calculated from the slope of oxygen level decrease according to the standard procedures (U.S. Department of Agriculture and U.S. Composting Council, 2001). Results of the static respiration index referred to total organic matter content are presented as averages of three replicates.

Results and Discussion

Composting of different paper sludges and bulking agent was studied in two steps.

Laboratory scale experiments

The objective of these experiments was to investigate the optimal mixture in paper sludge composting when temperature was selected as process variable. Table 2 presents a summary of the results obtained on composting different mixtures of PCS, BS and bulking agent (wood chips) at laboratory scale (4.5-L). In all the experiments, the moisture content was maintained within the optimal range for composting (40-60%) (Haug, 1993). In Table 2, the maximum temperatures achieved and the times for which temperature was over the thermophilic range threshold ($>45^{\circ}\text{C}$) are presented as average values of at least two experiments resulting in a total number of 18 runs. Maximum temperature is a good indicator of the composting possibilities of each mixture, since it determines if the thermophilic range of temperatures is reached and hence sanitation of the material achieved. From the results obtained in Table 2, it could be stated that:

- PCS by itself showed the best potential for composting, since it reached the highest temperature (65.5°C) and maintained thermophilic temperatures (over 45°C) for the longest period (7 days+14 hours).
- The addition of an inert bulking agent (wood chips) did not improve the composting of PCS at either volumetric ratio tested (1:1 and 2:1). Therefore, the inherent porosity of PCS can be considered as adequate for composting. When an inert highly-porous bulking agent such as wood chips was added to the mixture, temperature values were lower than those obtained in the composting of PCS alone (65.5°C without bulking agent vs. 60.1°C and 52.3°C using increasing ratios of bulking agent).
- The mixtures of PCS and BS at different ratios reached the thermophilic range, however, maximum temperature was lower than that achieved in the PCS composting, and thermophilic conditions were maintained for shorter times. Characteristics of the two sludges (Table 1) seemed to indicate that they were complementary in aspects such as C/N ratio or organic matter content. In practice, however, it was very difficult to mix the two sludges homogeneously, and the final product mainly contained unmixed parts of both sludges.
- The addition of a bulking agent to the mixtures of PCS and BS produced a negative effect in the composting process, and the thermophilic range was not achieved. This effect had been already observed in the composting of PCS alone.

Moreover, as PCS is produced in much larger amounts than BS, PCS composting without the addition of BS or bulking agent was selected for the pilot study.

Composting of PCS at pilot scale

At this point, composting of PCS was studied with the objective of determining the biological indices (RQ and RI) to validate temperature profiles obtained at laboratory scale. Temperature profile is presented in Fig. 1. The thermophilic range of temperature was reached within two days, and was maintained for more than two weeks, which implied a full sanitation of the material. Other values of temperature registered at different points of the composter showed similar profiles (data not shown). The decrease in the temperature at day 8 corresponded to a failure in the aeration system. These results are in agreement with other works undertaken with similar sludges (Charest and Beauchamp, 2002; Das *et al.*, 2002a).

On the other hand, oxygen and carbon dioxide concentrations (Fig. 2) showed a typical profile in the composting process. Initially, oxygen was consumed at a high rate (air flow rate up to 1 L/s) and CO₂ was produced in large amounts, reaching extremely high values (over 20%). Oxygen concentration fell below 5% from day 2 to 5, however no evidence of anaerobic conditions was observed (malodours, presence of organic acids, etc.). This period corresponds to the thermophilic phase (Fig. 1).

CO₂ and O₂ can be related by means of the respiratory quotient (RQ). This parameter is defined as the ratio between CO₂ produced and O₂ consumed, and has been routinely used in the biotechnological field (Atkinson and Mavituna, 1983) but, to the authors' knowledge, it is rarely measured in composting processes. Its value is approximately equal to 1 under aerobic conditions, although this obviously depends on the state of oxidation of the organic material. For instance, Smars *et al.* (2001) reported a value of 1.02 in the composting of source-separated household waste. Other authors (Mönnig *et al.*, 2002) reported similar values for the composting of municipal solid wastes. The range of RQ for PCS was between 0.96 and 1.31 (average value 1.19),

which clearly indicated that PCS was composed of organic material with a moderate degree of oxidation. Since RQ is a characteristic value directly related to organic waste composition, RQ can be used in the control and monitoring of the composting process of PCS to predict air requirements and CO₂ production. RQ value can also be used to compare PCS with other wastes.

Other typical parameters of the composting process remained practically steady throughout the experiments. Initial and final values of such parameters are presented in Table 3. For instance, the pH of compost material only increased slightly from 7.6 to 8.0. A similar pattern was observed for the total nitrogen profile, which only decreased from 0.43 to 0.30 % during the composting period. This fact, together with the high organic matter decomposition, implied that C/N ratio decreased significantly to reach a final value of 26.0. Although this C/N ratio value could not be compared to the typical values for stabilized compost of below 15 (Haug, 1993), it could be considered satisfactory since no nitrogen amendments had been used, and it was in accordance with other studies (Das *et al.*, 2002a). Other forms of nitrogen, such as ammonium nitrogen, were not detected during the composting process. Finally, electrical conductivity decreased slightly from an initial value of 1.92 dS/m to a final value of 1.31 dS/m, which has been also observed in the composting of paper residues (Jokela *et al.*, 1997; Das *et al.*, 2002b).

Moisture content and organic matter content profiles are shown in Fig. 3. It is evident from Fig. 3 that moisture and organic matter followed similar profiles. Thus, the presence of easily biodegradable compounds provoked the temperature increase and water evaporation. Once the thermophilic phase was reached, values of both organic matter and moisture content reached plateaus. Overall reductions in total weight, dry matter, moisture and organic matter content are also presented in Table 3. As a

consequence of the rigid aggregated structure of PCS, no considerable compaction was observed during the composting time, and the volume reduction can be considered as negligible. Total nitrogen losses were only 13% (Table 3), which was probably caused by the high C/N ratios observed throughout the composting time. Additionally, ammonia in exhaust gases was not detected. However, as no leachates were collected the only possible fate for this nitrogen is its release as ammonia emissions in exhaust gases. More significant losses were observed for moisture (37.5%) and organic matter (33.5%), which contributed to the observed decrease in C/N ratio. Compared to other de-inking paper sludges, the C/N ratio of PCS is low (Charest *et al.*, 2004). However, the organic matter content of PCS is also very low (33.7%, Table 1). This fact accounts for a low C/N ratio since other paper sludges present a higher organic matter content.

Among all the organic compounds present in PCS, cellulose was expected to be one of the major components involved in material decomposition. Figure 3 shows the evolution of cellulose content during PCS composting at pilot scale. Cellulose and total organic content profiles were very similar, presenting a high initial decomposition rate during the thermophilic phase and a plateau during the final mesophilic phase. Cellulose content decreased from an initial value of 37.2% to a final value of 18.1% (both expressed as a percentage of total organic matter) resulting in a cellulose reduction of 67.7% (Table 3). When this value was compared to total organic material reduction (Table 3), it could be concluded that cellulose corresponded to the 75% of the total organic matter decomposed. This fact confirmed that cellulose was the main organic material degraded during composting of PCS. Other studies reported similar results in the composting of de-inking paper sludge, showing that the cellulose breakdown is more rapid than that of the hemicellulose, whereas lignin fractions can be considered as resistant to biodegradation (Charest and Beauchamp, 2002).

Finally, the results obtained from the respiration tests (Fig. 4) indicated that a real stabilisation of the organic matter occurred for PCS. Final values of the respiration index were in the range of $1.1 \text{ mg O}_2 \cdot \text{g TOM}^{-1} \cdot \text{h}^{-1}$, which are in the range of stable compost according to the international standards (California Compost Quality Council web site, 2001). Besides, other maturity tests such as the Dewar self-heating test resulted in the maximum maturity grade (V).

These results confirm that PCS can be successfully composted with a high biological activity to obtain a stabilized organic material.

Conclusions

Composting of two types of sludge from the recycled paper manufacturing industry was studied at laboratory and pilot scale. Biological sludge (BS) composted similarly to other biosolids from wastewater treatment, although no bulking agent was necessary when it was co-composted with PCS. Physico-chemical sludge (PCS) from the de-inking process, which is the major waste produced in this type of industry, was successfully composted without the addition of amendments or bulking agents, which implied an important cost reduction. Although the moisture and organic matter content in PCS were low, the composting material reached a fully thermophilic temperature that permitted its sanitation. Oxygen and carbon dioxide profiles, together with respiratory quotient, indicated a complete decomposition of the material. In addition, respiration index determination showed a high level of organic matter stabilization, which is a key factor in the application of composts from such sludges. The methodology used in this work can be generalized to the study of similar organic wastes for which few data are available.

The composting of this type of sludge, which is predicted to be produced in increasing amounts in the following years, is an innovative sustainable technology for the recycling of paper manufacturing wastes, which are currently landfilled or incinerated.

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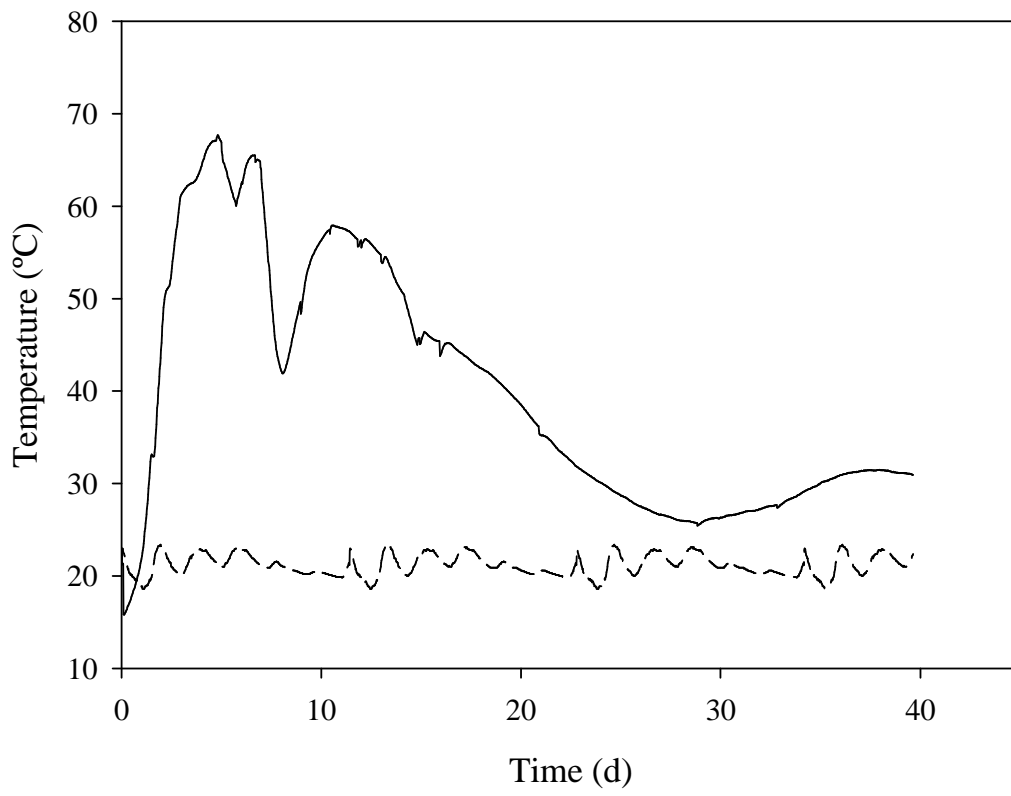
Captions to Figures

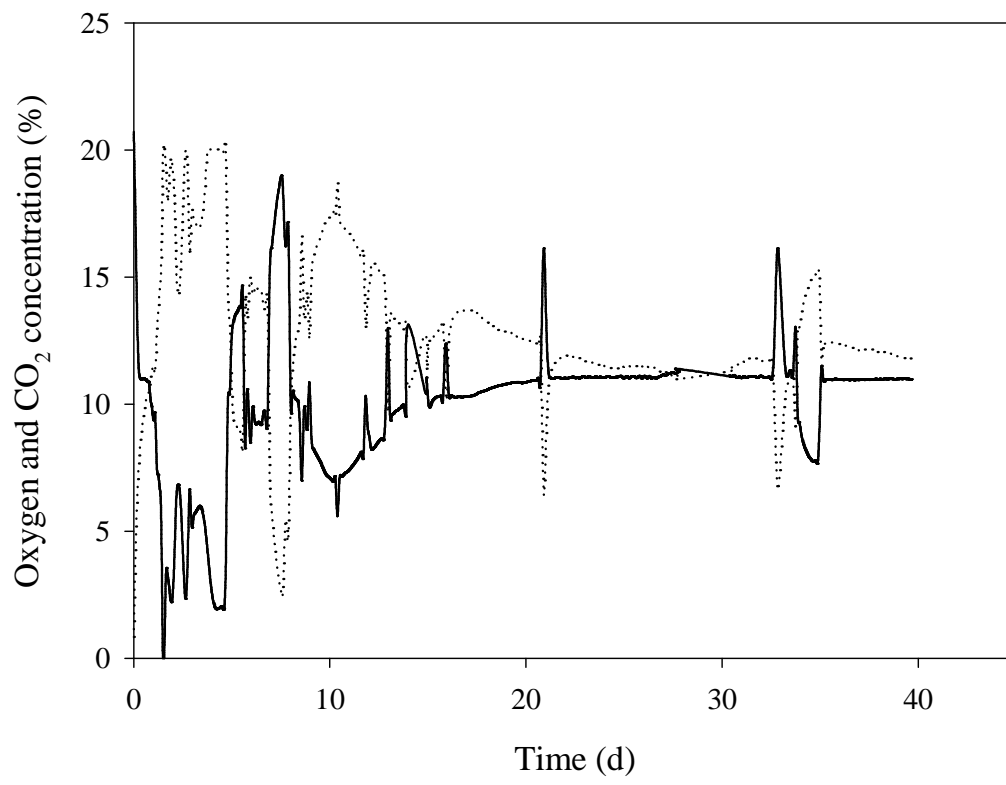
Figure 1: Temperature profiles in the composting of PCS at pilot scale. Composting temperature (central probe, solid line) and room temperature (dotted line).

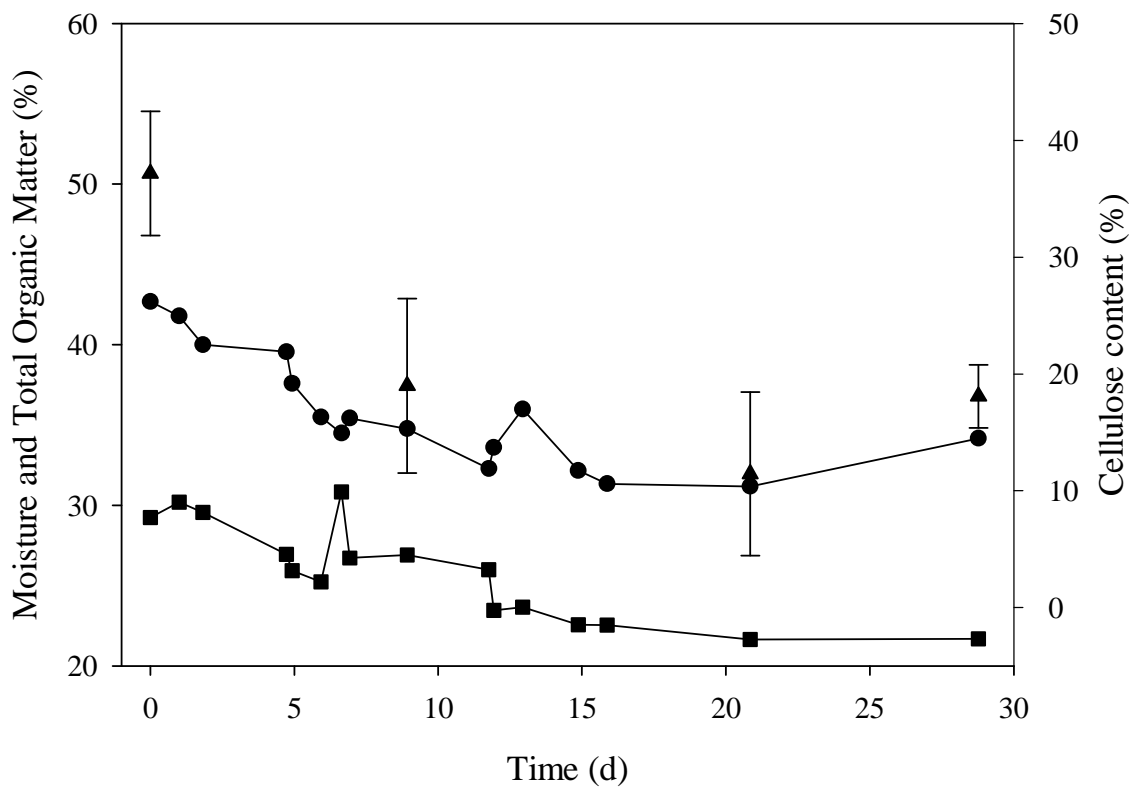
Figure 2: Oxygen (solid line) and carbon dioxide (dotted line) profiles in the composting of PCS at pilot scale.

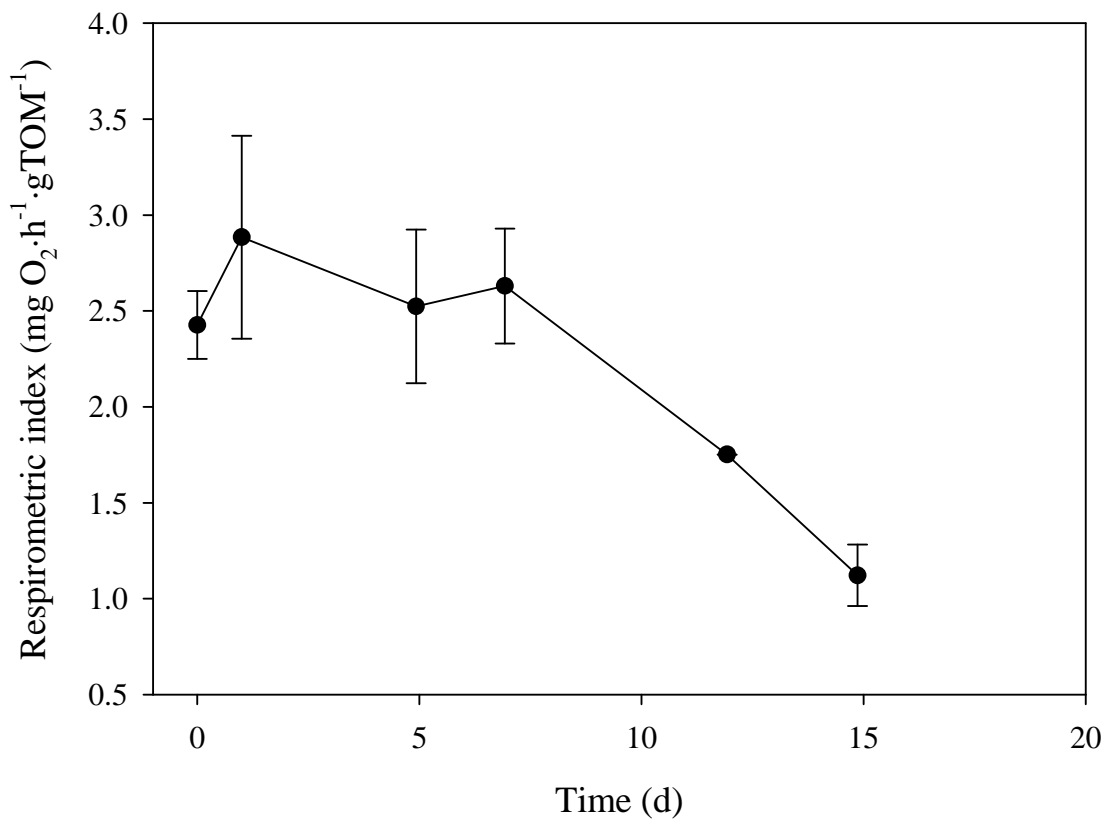
Figure 3: Moisture (circles), total organic matter (squares) and cellulose (triangles) profiles in the composting of PCS at pilot scale.

Figure 4: Static respiration index profile in the composting of PCS at pilot scale.









Tables

Table 1: Main characteristics of PCS and BS.

Parameter	PCS	BS
Dry matter (%)	63.3	47.3
Water content (%)	36.7	52.7
Total organic matter (% , dry matter basis)	33.7	58.8
pH (water extract 1:5)	7.50	6.80
Electrical conductivity (dS/m, water extract 1:5)	1.92	3.60
Total N Kjeldahl (% , dry matter basis)	0.43	1.07
C/N ratio	34.0	23.7
N-NH ₄ (% , fresh matter basis)	0.08	0.17
Total P (% , dry matter basis)	<0.10	0.37
Total K (% , dry matter basis)	<0.10	0.13

Table 2: Summary of the results obtained in the composting and co-composting of different sludges and mixtures.

Sludge volumetric ratio			Average maximum temperature (°C)	Thermophilic time (d+h)
PCS	BS	Bulking agent		
1	0	0	65.5	7d+14h
1	0	1	60.1	1d+17h
1	0	2	52.3	1d+15h
2	1	0	55.0	5d+1h
1	1	0	49.8	1d+23h
2	2	1	34.0	-
2	1	1	34.9	-
1	1	2	38.6	-

Table 3: Reduction of different parameters in the composting of PCS at pilot scale.

Parameter	Initial value	Final value	Reduction (%)
Total weight (kg)	73.0	57.0	22.0
Dry matter content (%)	57.3	65.8	10.3
Moisture content (%)	42.7	34.2	37.5
Organic matter (% , dry matter basis)	29.3	21.7	33.5
pH (water extract 1:5)	7.6	8.0	-
Elec. Cond. (dS/m, water extract 1:5)	1.92	1.31	-
C/N ratio	34	26	-
Total nitrogen (% , dry matter basis)	0.43	0.30	13.0
Cellulose (% , organic matter basis)	37.2	18.1	67.7