Composting of dewatered wastewater sludge with various ratios of pruning waste used as a bulking agent and monitored by respirometer

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Abstract

The effects of different volumetric ratios of wastewater sludge to bulking agent on the performance of full-scale composting were studied. Volumetric ratios of wastewater sludge to pruning waste, used as a bulking agent, were 1:2 (Pile 1), 1:2.5 (Pile 2) and 1:3 (Pile 3). Experiments were carried out in an uncovered plant using windrow composting with weekly turning. To monitor the evolution of the three composting piles, routine parameters such as temperature and interstitial oxygen level, chemical parameters such as organic matter, moisture and C/N ratio, and biologically related indices such as respiration indices at process temperature ($RI_{process}$) and at 37ºC (RI_{37}) were monitored. Different responses were observed in the three piles; Pile 1 did not accomplish the necessary requirements in terms of sanitation and $RI_{process}$ for a typical composting process; Piles 2 and 3 presented a similar behaviour, reaching thermophilic temperatures for a long period and, due to their high biological activity, high $RI_{process}$,. The quality of the product obtained in the three piles in terms of stability (RI_{37} and the Rottegrade self-heating test) and maturity (germination index) were measured, with compost from Pile 3 the most stable. To achieve satisfactory stability and sanitation for application to land, optimisation of the sludge to bulking agent ratio used to process wastewater sludge into compost appears to be crucial.

Keywords: Bulking Agent, Composting, Respiration Index, Stabilisation, Wastewater Sludge.

Nomenclature

C/N: Carbon to Nitrogen ratio

CBAR: Critical Bulking Agent Requirement

RI: Respiration Index

RI_{37}: Respiration Index at a fixed temperature of 37ºC (mg $[O_2]$ g^{-1} [VS] h^{-1})

$RI_{process}$: Respiration Index at the *in situ* temperature of the pile (mg $[O_2]$ g^{-1} [VS] h^{-1})

VS: Volatile Solids

w/w: Weight to Weight ratio

1. Introduction

In 1998, Spain produced about 0.6-0.8×10^6 Mg of dry wastewater sludge and it is expected that during 2006 the production will reach 1.5×10^6 dry Mg per year (Ministerio de Medio Ambiente, 2001). Catalonia, located in the northeast of Spain, is one of the regions with the highest production of sludge. At present, land application is the main disposal mode used for wastewater sludge, and there are unique legal restrictions for application to soil related to heavy metal content and the presence of potentially toxic compounds. Also, the spreading of sludge onto land must be carried out ensuring the effective elimination of pathogens and the maximisation of agronomic benefits.

Wastewater sludge composting with the use of bulking agents can enhance the stability of organic matter, inactive pathogens and parasites (Larsen et al., 1991; Furhacker and Haberl, 1995; Wei et al., 2001; Wang et al., 2003), and enable the production of a quality product that may be used as a soil conditioner or as an organic fertiliser (Tremier et al., 2005). A long list of waste materials have been proposed as bulking agents although the most widely used materials are wood chips and pruning waste (Atkinson et al., 1996; Jokela et al., 1997; Wong et al., 1997; Larsen and McCartney, 2000). Common volumetric ratios of bulking agent to sludge are approximately 2:1 to adjust C/N ratio to 25 (Wong et al., 1997). The study conducted by Eftoda and McCartney (2004) presented a systematic approach concerning the critical bulking agent requirement in sludge composting, by simulating the compressive load occurring in full-scale composting conditions. Although bulking agents are not believed to degrade significantly under composting conditions, because of their high lignin content, some recent works have reported a certain biodegradability of wood chips (Mason et al., 2004). The optimisation of the sludge-bulking agent mixtures for urban or industrial sludge composting has been extensively studied (Milne et al., 1998; Miner et al., 2001; Gea et al., 2003). The agronomical quality of compost produced is limited mainly by their chemical composition as well as by the stability and maturity of the organic matter (Govi et al., 1993; Sesay et al., 1997; Bernal et al., 1998a; Grigatti et al., 2004). Stability is related to the presence of easily biodegradable compounds in the composting process and can be measured by the respiration index (RI) (Iannotti et al., 1993; Chica et al., 2003). RI is commonly determined at mesophilic temperatures (30-37ºC) and it is an indicator of the biological activity of the composting process (Adani et al., 2003). However, since predominant active microbial populations evolve according to composting temperature profile, if RI is estimated at process temperature it can be used as an indicator of the evolution of the biological activity during composting (Barrena et al., 2005: Barrena et al., 2006a). In fact, recent results have shown weak correlations between respiration indices determined under mesophilic conditions and process temperature in full-scale composting of organic fraction of municipal solid wastes (Barrena et al., 2006b) and sludge composting (Eftoda and McCartney (2004). Other authors have proposed the use of dynamic respiration tests to overcome oxygen transfer limitations, although the cost of this type of test is considerably higher (Barrena et al., 2009). However, there are few references to the use of respiration indices in composting of wastewater sludge. In comparison to stability, maturity is normally defined in relation to compost application and plant growth (Cooperband et al., 2003) and it is usually determined by means of germination experiments (Weppen, 2002; Tang et al., 2006).

Windrow composting is the most common method to produce compost from organic wastes (Avnimelech et al., 2004). In windrow composting systems, the major source of aeration is natural convection, when air moves through interstitial voids (porosity) in the pile supplying oxygen to the microorganisms (Eftoda and McCartney, 2004). However, the need for porosity and, in consequence, the use of bulking agents increases the operational costs for composting plants. The cost of purchasing bulking agents and the cost of transportation may be particularly relevant in regions where there are no available gardens or forest areas, between 20-30 € Mg^{-1} of total cost, according to Spanish prices and providers (Ruggieri et al., 2009). At the same time, bulking agent requirements can result in space limitation as they increase the volume of material to process.

For this reason, when conditioning a high moisture content substrate (such as wastewater sludge) in a full-scale composting process, the minimum amount of bulking agent required to maintain an adequate oxygen level in the pore space of the compost matrix is key to ensuring optimum performance because it saves costs and it reduces the requirement for land. Several recent studies have highlighted the necessity of performing full-scale studies to obtain reliable conclusions about the composting process (Eftoda and McCartney, 2004; Ruggieri et al., 2008). However, such studies conducted at full-scale are rare in the literature.

The objectives of this research were to: i) determine the optimal bulking agent to sludge volumetric ratio on a full-scale composting process of wastewater sludge using mechanical turning; ii) study the overall efficiency of the composting process, in terms of stabilisation of organic matter, iii) monitor the biological activity of the composting process, by using different respiration techniques, and iv) determine the final compost maturity, based on germination indices.

2. Materials and Methods

2.1. Composting materials

Dewatered wastewater sludge was obtained from several urban wastewater treatment plants located in the region of Lleida (Catalonia, Spain); they are usually processed at the composting plant at Alguaire (Lleida). The average composition of wastewater sludge is presented in Table 1. The concentration of other nutrients was (dry weight basis): $N\text{-}NH_4^+$: 0.99 %; Ca: 4.8 (% CaO); Fe: 0.98 %; P: 2.5 (% P_2O_5); Mg: 1.0 (% MgO); K: 1.1 (% K_2O), showing no limitation of the nutrients in the composting process. The heavy metal content was (ppm, dry weight basis): Cd: 1.9, Cu: 130, Cr: 60, Hg: 0.8, Ni: 42, Pb: 54, Zn: 470; well below the international limits for sludge application to soil in Europe (European Commission, 2000). Pruning wastes were used as a bulking agent since this is the typical bulking agent used at the composting plant in Alguaire. Table 1 shows the main characteristics of the wastes composted.

2.2. Composting experiments

Composting experiments were carried out at the uncovered composting plant of Alguaire (Lleida, Spain). The environmental conditions (average daily temperature and precipitation) during the course of the experiments are shown in Figure 1.

Three composting piles were built simultaneously by mixing approximately 55-60 Mg of sludge with pruning wastes in three different volumetric ratios (sludge:bulking agent): 1:2 (Pile 1); 1:2.5 (Pile 2) and 1:3 (Pile 3). The initial ratios were selected according to the normal operation of the composting plant, from a low ratio (1:2), in terms of available porosity, and a high ratio (1:3), which was selected as the maximum value because of the availability of bulking agent at the plant. The approximate amounts, volumes and bulk densities of sludge and bulking agent mixtures used for each pile are presented in Table 2 and the main characteristics of the composted mixtures are shown in Table 1. It should be borne in mind that it took approximately one week to build the three piles, so it is probable that some degradation occurred during this period. Initial bulk densities for the mixtures tested can be considered high, as the sludge ash content was also high in comparison with other wastewater sludge (Haug, 1993). The base time for composting experiments was the day when all three piles were fully-formed.

The approximate dimensions of the piles were: base: 4 m; height: 1.5 m; length: 30-40 m, and they were trapezoidal in shape. The piles were built on a slopped concrete floor and they were not sheltered from the rain according to the normal operation of the plant. Sludge and pruning wastes were initially mixed as follows. Sludge was firstly deposited onto a 40-50 mm bed of pruning wastes with a particle size smaller than 30 mm. The remainder of pruning wastes, necessary to reach each of the three selected ratios, was then added on top of the mixture and then mixed using a turner (Backhus Model 15.50, Edewecht, Germany). Once the process was finished, and the wastes were well mixed, three passes of the turner were necessary to build-up each pile to the specified dimensions. The composting experiment was carried out from the 7th November, 2005 to the 31st January, 2006 (i.e. 85 days). All the piles were turned weekly and forced aeration was not provided.

Temperature and oxygen content of the piles were measured *in situ* at 1.00 and 1.50 m depth in 4 points of each pile. The temperature and oxygen values presented are average values and the standard deviation is shown. Temperature was measured with a portable Pt-100 sensor (Delta Ohm HD9214, Caselle di Selvazzano, Italy) and oxygen concentration was measured with a portable O_2 detector (Oxy-ToxiRAE, RAE, San Jose, USA) connected to a portable aspiration pump.

2.3. Analytical methods

Moisture, total organic matter (expressed as volatile solids, VS), organic nitrogen content, carbon/nitrogen ratio (C/N), nutrient content, heavy metal content, pH, electrical conductivity, bulk density, germination indices and the results from the Rottegrade self-heating test were all determined using an integrated sample and using standard procedures (US Department of Agriculture and US Composting Council, 2001). This bulk-integrated sample was obtained from eight different locations of each pile giving a final volume of approximately 24 l. Then, the integrated sample was manually mixed in the laboratory and reduced using the quartering method to several sub-samples of 1.5-2 l (approximately 1-1.5 kg) (Keith, 1996), which were used to carry out the analytical procedures. The sub-samples were stored at 4ºC for less than 24 hours prior to analysis.

Moisture content was determined by drying a moist sample in a forced-air oven set at 105ºC until constant sample weight and expressed on a wet weight basis. Total organic matter was determined by ignition of a dry sample in a muffle furnace at 550ºC for 2 h and expressed on a dry weight basis. Organic nitrogen content was determined by digestion using sulphuric acid at 420ºC for 1.5 h followed by distillation according to Kjeldahl method. Electrical conductivity and pH were determined using a mixture of compost and deionised water blended to a ratio of 1:5 (w/w) (US Department of Agriculture and US Composting Council, 2001). Bulk density was determined using the water displacement method. Results from the Rottegrade self-heating test were determined by measuring with a Pt-100 sensor the maximum temperature produced by a sample of compost incubated for 10 days in a 1.5 l Dewar vessel. Carbon content was based on the total organic matter of the samples and considering that, for biological materials, the carbon content is 55% of the volatile solid fraction (Tiquia et al., 1996). All the analyses mentioned above were triplicated with each replicate coming from a different sub-sample. The standard deviations of the replicated measurements are presented with all experimental data. Where no standard deviation is shown, errors can be considered negligible.

2.4. Respiration tests

Static respiration indices (RI) were determined using a respirometer, which was built to the design of the original model (Iannotti et al., 1993) but including the modifications and recommendations given by the US Department of Agriculture and the US Composting Council (2001). A detailed description of the respirometer and its design was published by Barrena et al., (2005).

The experimental setup also included a water bath to maintain the selected temperature during the respirometric test (total duration was 1 h). Respirometer tests were assayed at the *in situ* temperature of the pile during sampling in order to obtain the real biological activity of the process (i.e. under the same conditions, without incubation to avoid hydrolysis of organic matter). Results of the static respiration related to the stability of the material (carried out during the last sampling) were also measured at a fixed temperature value of 37ºC after an incubation period of 24 hours, as this is the usual temperature and incubation period used for the prediction of compost stability (Iannotti et al., 1993). In addition, in Pile 2 the respiration index at 37ºC was measured for comparison purposes, since previous results with municipal solid wastes have demonstrated that it does not change significantly during composting (Barrena et al., 2005). Our previous results (Barrena et al., 2006b), obtained during full-scale composting of municipal solid wastes, have demonstrated that the use of the actual process temperature to determine the respiration index is necessary to accurately determine the biological activity, whereas mesophilic conditions are only able to predict compost stability.

RI is expressed as the amount of oxygen consumed per unit of total organic matter of the sample per hour (mg [O_2] g $^{-1}$ [VS] h^{-1}). Values of RI are presented as the mean of three replicates. Standard deviation is also presented; usually in the range of 5-10%.

2.5. Phytotoxicity

Aqueous extracts of the three piles final samples were prepared by adding two parts of deionised water per part of sample weight (on a dry matter basis). The phytotoxicity of these extracts (filtered by 0.45 µm) was evaluated by the seed germination technique (Tiquia et al., 1996; Tiquia and Tam, 1998). Cucumber (*Cucumis sativus*) was used for the test. Using 10 seeds of cucumber incubated in the extracts at 25ºC for 5 to 7 days, the seed germination percentage (germination index) and root length of the cucumber seeds were determined. Seed germination percentage and root elongation in distilled water were also measured as a control. The percentages of relative seed germination, relative root elongation and combined germination index were calculated as follows:

$$\text{Relative seed germination (\%)} = \frac{\text{No. of seeds germinated in final compost extracts}}{\text{No. of seeds germinated in control}} \times 100 \quad (1)$$

$$\text{Relative root growth (\%)} = \frac{\text{Mean root length in final compost extracts}}{\text{Mean root length in control}} \times 100 \quad (2)$$

$$\text{Combined germination index} = \frac{(\%\ \text{Relative seed germination}) \quad (\%\ \text{Relative root growth})}{100} \quad (3)$$

All germination indices were calculated using three replications.

3. Results and Discussion

3.1. Evolution of temperature and interstitial oxygen

Weather conditions affect the performance of the composting process since parameters such as interstitial oxygen and moisture content are dependent on the average daily environmental temperature and the average daily precipitation. The profiles of average daily environmental temperature and average daily precipitation are plotted in Fig. 1. Low temperatures and high precipitation were recorded during the composting experiments. Few studies have been published about the influence of cold weather and precipitation conditions on the composting performance but McCartney and Eftoda (2005) found a positive correlation between snowfall and oxygen supply and pile moisture content.

The temperature profiles of the three composting piles are shown in Fig. 2. No significant differences were observed between oxygen and temperature measurements obtained at different depths of the material. The standard deviations for temperature values were in the range of 2-8 ºC, whereas in the case of oxygen they were in the range of 1-4 % (Fig. 3). However, the initial values of temperature had a high degree of variability because the construction of the piles took one week.

The thermophilic range of temperatures was easily reached by Piles 2 and 3 despite the low ambient temperatures (< 0ºC for several weeks) and the poor weather conditions (rain, fog and high humidity). In Pile 3 average temperatures over 55ºC were measured for more than 70 days, which was a positive indication for sanitation of the material. Also, Pile 2 had average temperatures over 55ºC for at least 15 days. Turning of the piles was carried out once a week, in excess of that required by the regulations for sanitation (US Environmental Protection Agency, 1995). Therefore, the material composted in Piles 2 and 3 met the international requirements on compost sanitation, which are based on time-temperature conditions (US Environmental Protection Agency, 1995; European Commission, 2000). However, as shown in Fig. 2, according to international regulations the material in Pile 1 was not sanitised. Nevertheless, a microbial study would be necessary to ensure the absence of pathogen microorganisms in the three piles.

Fig. 3 shows the evolution of interstitial oxygen of the material for each pile. Initially a drop in the oxygen values for the three piles could be observed until a minimum value of 10, 5.5 and 8 % for Piles 1, 2 and 3, respectively. These drops in the values of oxygen content could mostly be attributed to the high initial biological activity of the material in Piles 2 and 3, as it was observed by the rapid temperature rise, which is the typical composting profile for highly biodegradable wastes (Gea et al., 2004). In Pile 1, the reason for oxygen depletion may be related to high moisture content and a resulting low porosity, as it can be observed in Fig. 4, since biological activity was always low for this Pile 1, as will be explained later. Pile 3 followed a typical interstitial oxygen profile; initially a drop followed by an increase as temperature decreases. This indicated a reduction in the biological activity due to the progressive diminution and exhaustion of biodegradable organic matter. Furthermore, it is necessary to point out that the interstitial oxygen level in Pile 1 was always slightly higher, which could be due to the lower biological activity as will be discussed later. During days 20-40, an increase in oxygen content was observed for all three piles. This could be due to a reduction in the moisture content (Fig. 4), which implies an increase in the porosity that allowing better air circulation, as has been reported for other organic wastes (Ruggieri et al., 2008). During the next period (from day 40) a slight decrease of oxygen for Piles 1 and 2 was observed, even when moisture content was stable. A possible explanation of this might be a progressive compaction of the material and a loss of free air space, which is often related to a lack of bulking agent (Miner et al., 2001; Gea et al., 2003; Eftoda and McCartney, 2004).

On the other hand, some values of interstitial oxygen detected during the experimental period can be considered as low. Although these values can be considered to limit the composting process, there has been some evidence of good composting performance when a mechanical system with frequent turning is used (Barrena et al., 2006b; Ruggieri et al., 2008). It appears that having an adequate set of conditions for the start-up of a composting process with wastewater sludge (moisture, porosity, etc.) is not the only requirement necessary to ensure a successful process performance. It is possible that, in order to achieve the desired temperature profiles, there is a minimum level of respiration activity or, in our words, a high oxygen uptake rate, necessary for the first stages of composting at full-scale, as will be discussed later.

3.2. Physico-chemical parameters

Initial and final properties of the three composting mixtures are shown in Tables 1 and 3 respectively. Evolution of moisture content during composting is shown in Fig. 4. Because of the weather conditions, environmental humidity and a total precipitation of 90 mm (Fig. 1), an increase in moisture content during the first week was observed in all three piles. It is probable that the initial mixture of sludge and bulking agent was not water-saturated and consequently some rainwater was retained thereby increasing moisture content. In addition, the temperature of the piles was not high enough to evaporate the excess of water. However, contrary to the results from other authors (McCartney and Eftoda, 2005), no significant correlation ($p<0.05$) was found between precipitation and moisture content. The abnormally low initial moisture content of Pile 3 can be due to a sampling error, as this value is not feasible according to the initial moisture values of sludge and bulking agent. Unfortunately, no sample was available to repeat this analysis. The effect of the increasing moisture, combined with the aerobic biological activity, was reflected in a drop of the interstitial oxygen level for the three piles. However, after the first week, a decreasing tendency was observed with the three experiments reaching final values of 51 % for Pile 3 and 60 % for Piles 1 and 2. Nevertheless, moisture levels were over 40 % during the composting experiments for the three piles, which is considered as the minimum value for optimal composting piles. Thus, composting was not limited by moisture content (Haug, 1993). Dry matter reductions are shown in Table 4, with values in the range of 5.4-16.3% being obtained. However, a significant correlation could not be determined between dry matter reduction and the extent of the composting process.

Organic matter evolution is shown in Fig. 4, and total reductions of organic matter content are presented in Table 4. No conclusions could be drawn from the results for organic matter since the bulking agent (which is mostly organic) was not significantly degraded. Nevertheless organic matter reduction for Piles 2 and 3 were higher than for that of Pile 1 because of thee more active process that occurred in Piles 2 and 3. Other authors have studied the reduction in organic matter in the different sludge composting systems. For instance, a 10% reduction was obtained with a pile-scale aerated static bin (Zhu, 2006), whereas other authors obtained high values of reduction (55%) for a full-scale aerated pile (Baeta-Hall et al., 2005). Recently, other authors have reported reductions in the range of 30-50% depending on the type of bulking agent using the Rutgers system, forced ventilation and mechanical turning (Alburquerque et al., 2006).

The trends for pH and electrical conductivity are presented in Tables 1 and 3 and Fig. 5. All the values obtained had a low standard deviation and were in accordance with typical values for composting processes with a slight decrease of electrical conductivity (Pagans et al., 2006). The trend for pH showed is typical of that related to fatty acid production and ammonia generation and release. Initially, pH was relatively high compared to other organic wastes (e.g. municipal solid wastes) (Gea et al., 2004), which is usual for wastewater sludge because of its high free ammonia content (Weppen, 2002). Then, during the most active part of the composting process (0-20 days) ammonia is stripped as gas and the possible formation of fatty acids provokes a slight decrease in pH (Pagans et al., 2006; Sundberg et al., 2004). After the active phase, the decrease in temperature results in a lower vaporisation of ammonia and the biodegradation of fatty acids, which leads to an increase in pH to an alkaline value. As it can be observed, only slight differences were detected among the three piles, therefore it can be concluded that bulking agent ratio did not have a significant influence on these parameters.

In relation to C/N ratios presented in Tables 1 and 3, the initial values were slightly lower than those recommended for composting (Jokela et al., 1997; Wong et al., 1997; Larsen and

McCartney, 2000). The highest initial C/N ratio was 17.5 for Pile 3, which is very close to the optimal reported values for C/N ratio (Jokela et al., 1997). The C/N ratio increased during the composting process resulting in a relatively high C/N ratio, which may have been caused by high rates of ammonia emission. In fact, low C/N ratios have been proposed as a measure of compost stability (Bernal et al., 1998a; Bernal et al., 1998b). However, it must be pointed that C/N ratios in initial mixtures for composting are usually formulated on a total C/N basis, whilst not all the C is biodegradable (Sánchez, 2007). However, the C/N ratio results demonstrated that none of the piles was nitrogen limited.

3.3. Biological activity indices

Different respiration indices (RI) were measured during the experimental period. Respirometry usually refers to the aerobic biological activity of the material and were determined at two different temperatures: RI at process temperature ($RI_{process}$), which is an indicative parameter of the real process activity at operating conditions, or RI at 37 ºC (RI_{37}), which is related to the material stability in the maturation phase (Barrena et al., 2005). It is also generally considered that values of RI_{37} below 1 mg [O_2] g $^{-1}$ [VS] h^{-1} correspond to a stable compost (California Compost Quality Council, 2001). On the other hand, the Rottegrade self-heating test gives information in form of a stability grade. In Europe, this test is commonly used to characterise the stability of compost, with a range from I (fresh material) to V (mature compost) (US Department of Agriculture and US Composting Council, 2001). The test is based in determining the increase of temperature of a compost sample in an adiabatic Dewar vessel. However, the Rottegrade test is used routinely with municipal solid wastes (Barrena et al., 2005) and no information is available on the performance of this test with wastewater sludge.

Trends for $RI_{process}$ for the three piles were determined and monitored during the experimental period. However, RI_{37} was only determined for Pile 2, since this pile was built according to the usual composting pile recipe (i.e. sludge: bulking agent ratio) and was used as a control sample. Furthermore, RI_{37} and Rottegrade self-heating test were determined for the final material of the three piles to measure the final stability of the compost obtained.

Trends for $RI_{process}$ are shown in Fig. 6. Initially, the $RI_{process}$ for the three piles was similar and close to 2 mg $[O_2]$ g^{-1} [VS] h^{-1}. After the first week of composting, the maximum value of $RI_{process}$ was reached in Piles 2 and 3. For large masses, the time necessary to reach the maximum value of respiration activity is because it is necessary to have a homogenous distribution of microbial population and biodegradable organic matter. This phenomenon is not usually observed in laboratory scale composting experiments (Gea et al., 2004; Barrena et al., 2005). A maximum $RI_{process}$ values close to 12 mg $[O_2]$ g^{-1} [VS] $h^{-1}$and close to 5 mg $[O_2]$ g^{-1} [VS] h^{-1} were reached in Piles 3 and 2, respectively. These can be considered as being very high values of RI, and they correspond with values found for other biodegradable organic wastes such as organic fraction of municipal solid wastes and paper sludge (California Compost Quality Council, 2001; Barrena et al., 2006a). However, $RI_{process}$ for Pile 1 did not increase as was expected, given the high values of RI found for wastewater sludge (Table 1) and low values of $RI_{process}$ were obtained. Therefore, it is evident that a high level of porosity is necessary for the composting of high moisture organic wastes such as wastewater sludge at full scale. However, other studies on composting of wastewater sludge at laboratory scale have shown that with a relatively low volumetric bulking agent: sludge ratio (1:1 or 2:1) it is possible to obtain a successful composting process (Gea et al., 2003;

Gea et al., 2004). It is remarkable to note that the optimal bulking agent volumetric ratios found in the present study are very close to those of laboratory composting experiments systematically conducted with the objective of determining critical bulking agent requirement (CBAR) by simulating the compressive load that occurs at full-scale. In this case, the key was to include vertical loadings in the small-scale simulations to obtain results representative of full-scale conditions (McCartney and Chen, 2001; Eftoda and McCartney, 2004). This again reinforces the necessity of carrying out composting experiments at full-scale when the process is to be implemented on an industrial scale. Because of their influence on the composting process, it is also necessary to carry out full-scale experiments under representative weather conditions.

After reaching the maximum value, the $RI_{process}$ for Piles 2 and 3 showed an important decrease during the two following weeks, according to the progressive stabilisation of organic matter; confirming that a significant amount of oxygen was consumed during the first stage. However, during this period no variation in $RI_{process}$ was observed for Pile 1, which confirms the low biological activity found in this pile, even three weeks after the composting pile was built and despite the weekly turnings. This is again evidence of the key role that bulking agent ratio plays in the performance of wastewater sludge composting. It is possible to attribute the initial low activity found in Pile 1 to an excessive moisture content. Nevertheless, after the third week, moisture content in Pile 1 was similar to that in Piles 2 and 3, which showed the maximum $RI_{process}$ values. As a consequence, during the rest of the composting process, it was evident that a low ratio of bulking agent was responsible for the low biological activity observed.

In the final two months of the composting a slight decrease of $RI_{process}$ was observed for

Piles 2 and 3, which could be probably attributed to the degradation of slowly biodegradable compounds. Thus, it can be concluded that a higher level of biological activity takes place during the first phase, when easily biodegradable organic matter is available for the microorganisms. After this, when the pool of organic matter is exhausted, biological activity remains practically constant at low level or with a slightly decreasing tendency because the less-easily biodegradable organic matter requires lower oxygen consumption (Barrena et al., 2006c). After three months, Pile 1 continued showing a constant $RI_{process}$, which possibly implies that basal respiration was maintained during the entire composting period.

Comparing $RI_{process}$ data with pile temperature profiles during the first stage, it can be observed that initially they are well correlated. The pile with the highest biological activity (Pile 3), in terms of $RI_{process}$, reached the highest temperature in a short period. Pile 2, which showed significant biological activity, also reached the thermophilic range of temperature during the first week of process. Pile 1, on the other hand, did not show an increase in biological activity and never reached the thermophilic range.

However, during the maturation phase (from day 30 onwards), biological activity was low and practically constant even when the pile temperature was high and occasionally within the thermophilic range (Piles 2 and 3). This was probably due to the thermal properties of the compost material (low heat transfer rates as a consequence of low thermal conductivity and heat retention) and should not be related to biological activity, as it has also been observed in the maturation phase of other organic wastes (Gea et al., 2005; Barrena et al., 2006c). Therefore, temperature should not be used in the maturation stage to predict compost stability or the stage of organic matter degradation.

Comparing $RI_{process}$ data with interstitial oxygen data, it can be observed that the initial decrease in the level of interstitial oxygen could be due to the increase of moisture content (which implies a loss of porosity) but also to the increase in biological activity (especially in Piles 2 and 3). The level of moisture (and consequently porosity) has been found to be critical for the composting of wastewater sludge in general (Haug, 1993; Gea et al., 2003) and also specific studies (Richard et al., 2002; Eftoda and McCartney, 2004).

The final cumulative oxygen consumption at the end of the process (90 days) was also determined in terms of mass of O_2 consumed per mass of initial VS in sludge for the three composting piles by considering that the bulking agent used was not significantly degraded. In fact, this value can be calculated by means of a numerical integration of the $RI_{process}$ values versus time at any given process time (Fig. 6) using the Simpson method (Yakowitz and Szidarovszky, 1989). When the total process time is considered, this value can be an indicator for good performance of the composting process and a measure of the stability of a final compost product. A very high organic matter degradation would then imply a total oxygen uptake of 23.355 g $[O_2]$ g^{-1} [initial sludge VS] (value obtained in Pile 3). A significant degradation would occur for values of 13.360 g $[O_2]$ g^{-1} [initial sludge VS] (value obtained in Pile 2). Whereas for values below 8.100 g $[O_2]$ g^{-1} [initial sludge VS] (value obtained in Pile 1), the composting process could not be considered finished and the organic matter would not be stabilised. This is, to our knowledge, the first attempt to use the cumulative oxygen consumption to predict compost stability, but the application of this index should be related to the effectiveness of organic matter degradation in a composting process and the extent at which composting occurs. A very interesting feature of the cumulative oxygen consumption is that it can differentiate using initial and final samples, which is not possible using single RI determinations, and this is important because at the initial stage composts show low respiration activity because their biological activity is still starting-up (the typical lag phase of biological processes). The evolution of cumulative oxygen uptake is shown in Fig. 7 for the three piles. It is clear from Fig. 7 that the highest level of oxygen consumption occurred in Pile 3, followed by Pile 2 and Pile 1, which is in agreement with temperature profiles and the discrete respiration index measurements (Fig. 6). From Fig. 7, it is interesting to note that after a stabilisation of organic matter, a basal respiration was observed for all the materials composted. This has been shown with other composting studies dealing with different organic wastes (Barrena et al., 2005; Gea et al., 2005).

RI_{37} evolution for Pile 2 is also plotted in Fig. 6. This parameter was only determined for Pile 2 in order to compare it with $RI_{process}$. It showed practically constant values close to 1 mg $[O_2]$ g $^{-1}$ [VS] h^{-1} for the entire period of the experiment. As expected, when respiration indices were determined at 37ºC and at process temperature, differences between both indices were more significant during the first thermophilic phase than in the final maturation phase, when temperature was closer to 37ºC. In fact, RI_{37} values were just slightly different from $RI_{process}$ from day 30 onwards (corresponding to the maturation phase), as shown in Fig. 6. From 0 to 30 days (the active phase of composting) the thermophilic microorganisms only exhibited a limited growth at 37ºC (which implies a low RI) due to the kinetics imposed by low temperatures, whereas the mesophilic population only exhibits a limited respiration activity (Barrena et al., 2005). At process temperature, the respiration index was determined under real operating conditions (i.e. thermophilic range) and the microbial populations present in the material were fully active, resulting in high values of RI. It can be concluded that the $RI_{process}$ can be used for monitoring the biological activity of the composting process;

however, it should be determined at process temperature, whereas determinations at 37ºC (mesophilic temperature) should be exclusively used as a stability parameter in the maturation phase. Similar results have been also obtained in laboratory or pilot scale composting experiments with several organic wastes (Gea et al., 2004; Barrena et al., 2005). At full scale, although some weak correlations between RI, measured at mesophilic conditions, and temperature have been observed in the composting of organic fraction of municipal solid wastes (Barrena et al., 2006b) and sludge composting (Eftoda and McCartney (2004), $RI_{process}$ is a more accurate parameter to show the biological activity of composting mixtures. Another possible approach is the use of dynamic respiration tests, in which the oxygen transfer limitations can be completely overcome, although the cost of these tests can be considerable (Barrena et al., 2009). However, the use of $RI_{process}$ as a measure of biological activity is of special relevance at full-scale facilities (especially in the maturation stage) where temperature is maintained in the thermophilic range because of the limited heat transfer of the compost material (low thermal conductivity) although biological activity is limited (Barrena et al., 2006b). Therefore, in these situations, the RI provides an accurate measure of the biological activity of the compost material.

3.4. Final compost stability

In the final samples, RI_{37} was measured for all three piles. The results are shown in Table 3. Despite all the materials having the same final low RI_{37} value, this was not indicative of similar compost properties, because it is necessary to consider temperature and $RI_{process}$ trends. Consequently, it is possible to affirm that final material in Pile 1 was not significantly composted. Although the three piles had a similar final RI_{37} close to 1 mg [O_2] g^{-1} [VS] h^{-1}, only Pile 3, with a RI_{37} of 0.73 mg $[O_2]$ g $^{-1}$ [VS] h^{-1}, had a value of below the limit established to qualify the compost as stable material (California Compost Quality Council, 2001). These results indicate that fully stable and mature compost from wastewater sludge can be obtained in 60 days in a large-scale facility, if the bulking agent ratio is properly adjusted. In the case of Pile 2 a short curing process could have a positive effect on the stability of the compost.

Rottegrade stability grade for the three composts is also shown in Table 3. All the composts presented a Rottegrade value of V, which corresponds to the maximum stability grade. However, it is necessary to point out these values can lead to wrong conclusions, as no evidence of process development is available using this test. Rottegrade test and RI_{37} values should therefore be considered with care as parameters to predict and determine stability of composts from wastewater sludge, especially if no data from the composting process is available. Parameters based on cumulative oxygen consumption appear to be more reliable in terms of measuring the effectiveness of the composting process and organic matter biodegradation.

3.5. Phytotoxicity analysis

In relation to the phytotoxicity of compost, seed germination tests indicate the presence of significant quantities of phytotoxins (Tiquia et al., 1996; Tiquia and Tam, 1998). The results of phytotoxicity analysis are presented in Table 5. The relative seed germination results were in all the cases 100%, which meant that no phytotoxic compounds were present in compost. Additionally, all the relative root growth results were greater than 100%. It has been suggested that a combined germination index (a product of relative seed germination and relative root elongation) over 80% indicates the absence of phytotoxins in composts (Tiquia et al., 1996). Moreover, a relative root growth over 100% indicated that compost had a positive effect on plant growth. In the case of Pile 1 compost, which also presented a high germination index, a possible explanation is that the original sludge did not contain any important plant growth toxins, and therefore the germination indexes are high even with unfinished compost. Further studies on wastewater sludge composting might consider the determination of germination indices in initial or some intermediate stages of the composting process.

4. Conclusion

The performance of three full-scale composting processes using different bulking agent ratios has been systematically studied. Results revealed that the selection of an appropriate bulking agent ratio is critical for the correct development of the composting process in low-porosity organic wastes such as municipal wastewater sludge. Optimum values of volumetric ratio bulking agent: sludge are within the range 2.5 – 3. Respiration indices are the most suitable parameters to monitor the composting process, as they reflect the biological activity of the composting process. Other physico-chemical measures should be carefully considered and they often need information of the process evolution to be correctly interpreted. According to cumulative respiration values, high organic matter degradation corresponds to 13-23 g [O_2] g^{-1} [initial sludge VS], when high volumetric ratios of bulking agent are used. In fact, cumulative oxygen consumption is able to predict the effectiveness of organic matter degradation in a composting process and the extent at which composting occurs. Finally, by using the adequate volumetric ratio, the compost obtained from wastewater sludge presents a high level of maturity.

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Tables

Table 1: Properties of the initial mixtures for the three composting experiments and characteristics of bulking agent and wastewater sludge.

Material	Moisture content (%)	Organic Matter (db, %)	Organic C (db, %)	Organic N (db, %)	C/N	pH	Electrical Conductivity (mS cm^{-1})	RI_{37} (mg [O_2] g^{-1}[VS] h^{-1})
Sludge	84.7±0.1	75.0±0.1	41.2	5.96	7.0	N/M	N/M	7.3±0.2
Bulking agent	17.3±0.1	58.1±0.3	32.0	0.71	44.9	N/M	N/M	1.3±0.2
Pile 1* (1:2)	62.7±0.9	51.3±0.4	28.5	2.68	10.6	8.04	2.65	2.08±0.04
Pile 2* (1:2.5)	63.9±0.4	55.4±0.4	30.7	2.09	14.7	8.64	2.49	1.7±0.1
Pile 3* (1:3)	43±5	52.0±0.6	28.9	1.65	17.5	8.16	3.10	2.1±0.2

* Properties determined once the pile was built (after one week)

db: dry basis

N/M: not measured

Table 2: Properties of the three piles studied. Sludge and bulking agent weights and total volume of the piles were experimentally determined.

Pile	Bulking agent (ww, Mg)	Sludge (ww, Mg)	Initial percentage of sludge (%, weight basis)	Total Volume (m^3)	Bulk density (Mg m^{-3})
Pile 1	30.2	64.1	68.0	90	1.05±0.2
Pile 2	43.7	74.2	63.0	120	0.98±0.1
Pile 3	42.3	59.8	58.6	105	0.97±0.1

ww: wet weight

Table 3: Properties of final products obtained from the three composting experiments.

Pile	Moisture content (%)	Organic Matter (db, %)	Organic C (db, %)	Organic N (db, %)	C/N	pH	Elec. Cond. (mS cm^{-1})	RI$_{37}$ (mg [O$_2$] g^{-1} [VS] h^{-1})	Stability grade
Pile 1	64.5±0.3	48.6±0.5	26.97	2.22	12.1	8.14	2.14	1.01±0.04	V
Pile 2	60.1±0.4	46.7±0.3	25.96	1.48	17.5	8.27	1.65	1.16±0.09	V
Pile 3	51.4±0.4	47.1±0.5	26.15	0.79	32.8	8.10	1.62	0.7±0.1	V

db: dry basis

Table 4: Dry matter and organic matter reduction for the three composting experiments. Data presented are calculated from an overall mass balance, considering the principle of ash content conservation (Haug, 1993).

Material	Dry matter reduction (%)	Organic Matter reduction (%)
Pile 1	5.5	10.5
Pile 2	16.3	29.5
Pile 3	8.1	15.5

Table 5. Germination indices for the final compost of three piles.

Material	Relative seed germination (%)		Relative root growth (%)		Combined germination index (%)	
	5 days	7 days	5 days	7 days	5 days	7 days
Pile 1	100	100	128.3	137.3	128.3	137.3
Pile 2	100	100	123.2	149.3	123.2	149.3
Pile 3	100	100	118.2	130.1	118.2	130.1

Figure Legends

Figure 1: Average daily environmental temperature and average daily precipitation during the course of the experiments in the composting plant of Alguaire.

Figure 2: Temperature profiles (average values) during the course of the experiment for Pile 1, Pile 2 and Pile 3. Error bars show the standard deviation.

Figure 3: Oxygen content profiles (average values) during the course of the experiment for Pile 1, Pile 2 and Pile 3. Error bars correspond to standard deviation.

Figure 4: Evolution of moisture on wet weight basis (solid symbols) and organic matter on dry matter basis (open symbols) content during the course of the experiment for Pile 1, Pile 2 and Pile 3. Error bars show the standard deviation.

Figure 5: Evolution of pH (solid symbols) and electrical conductivity (open symbols) during the course of the experiment for Pile 1, Pile 2 and Pile 3.

Figure 6: Respiration index at process temperature for Pile 1, Pile 2 and Pile 3 jointly with the temperature of each sample. Respiration index at 37ºC for Pile 2 (open circle) during the course of the experiment. Error bars show the standard deviation.

Figure 7: Cumulative oxygen consumption for Pile 1, Pile 2 and Pile 3 during the course of the experiment.

Fig. 1:

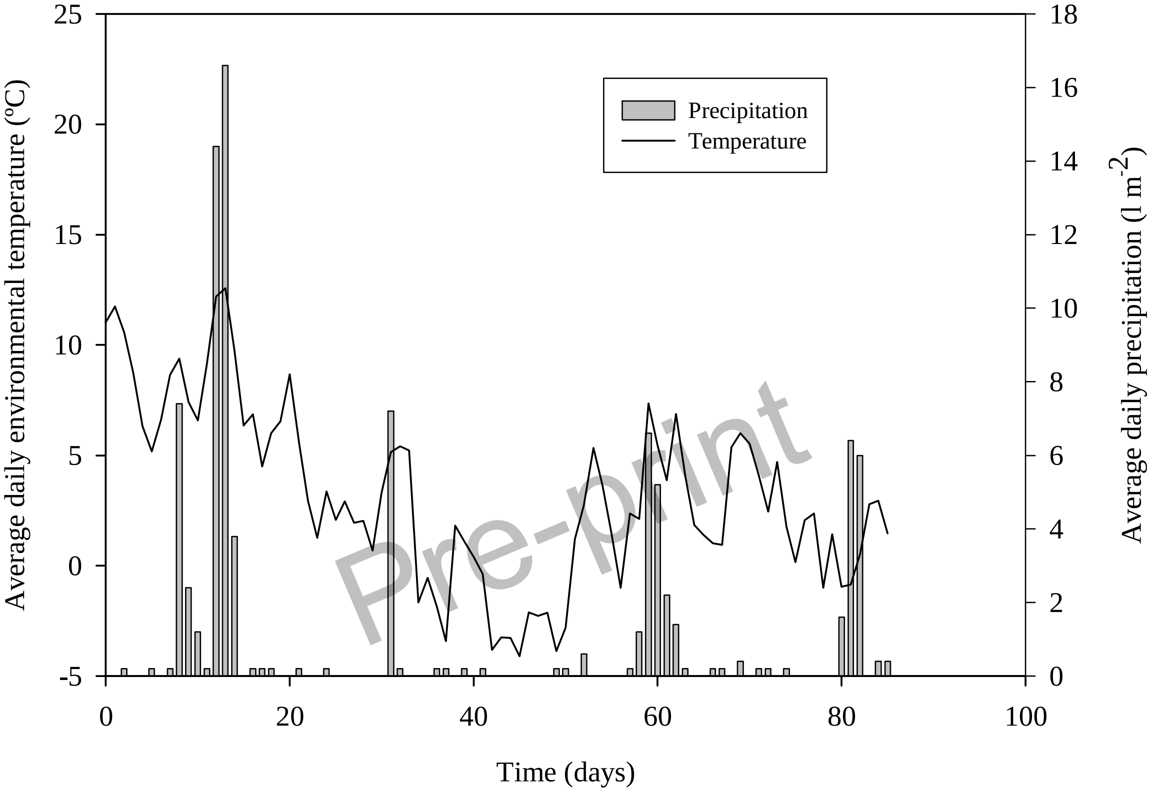

Precipitation
Temperature
Average daily environmental temperature (ºC)
25
20
15
10
5
0
-5
Average daily precipitation (l m^{-2})
18
16
14
12
10
8
6
4
2
0
0
20
40
60
80
100
Time (days)

Fig. 2:

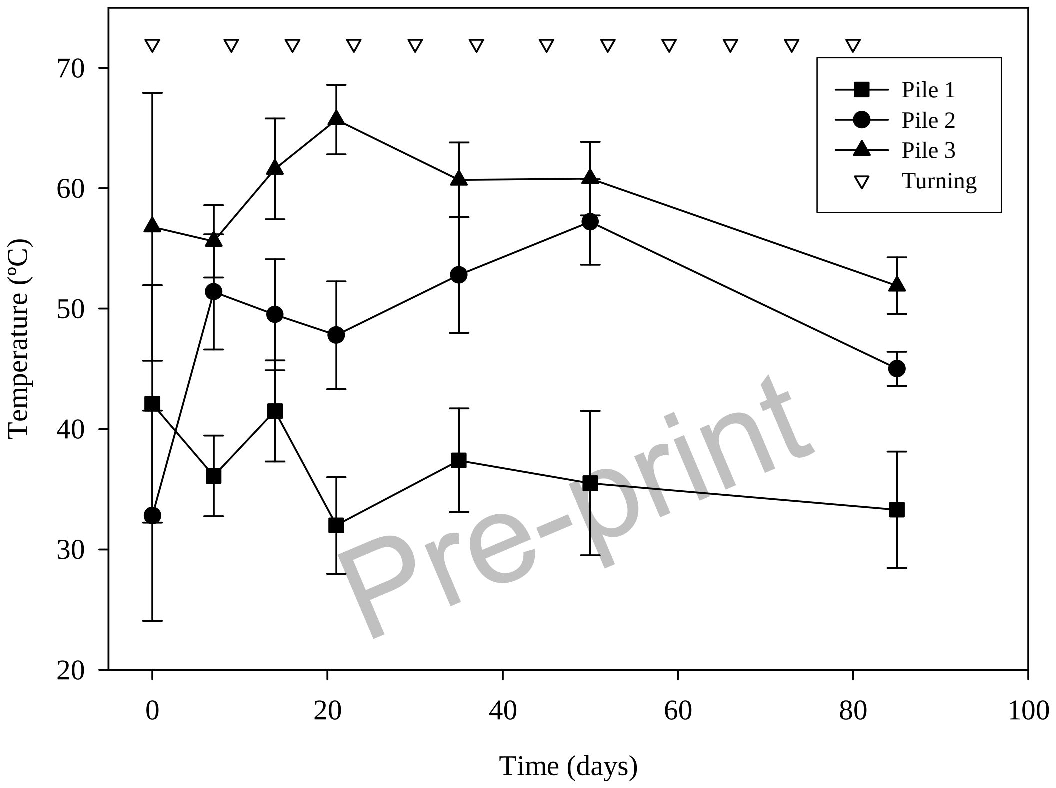

Temperature (°C)
20
30
40
50
60
70
Time (days)
0
20
40
60
80
100
Pile 1
Pile 2
Pile 3
Turning

Fig. 3.

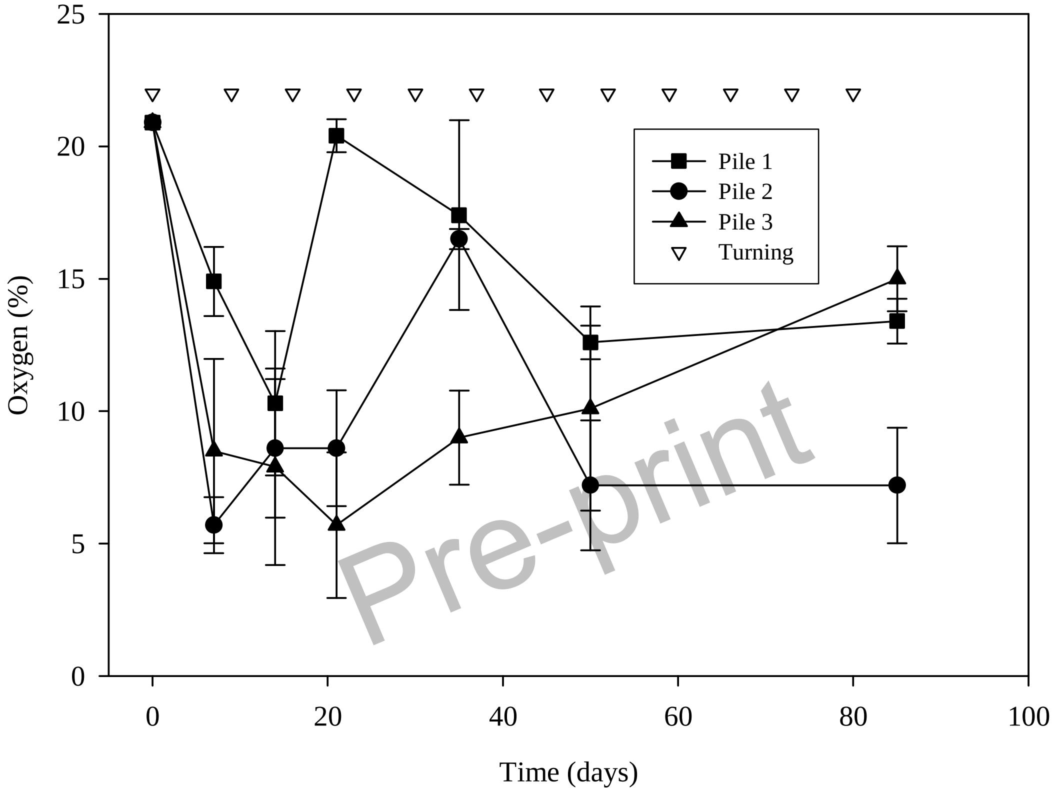
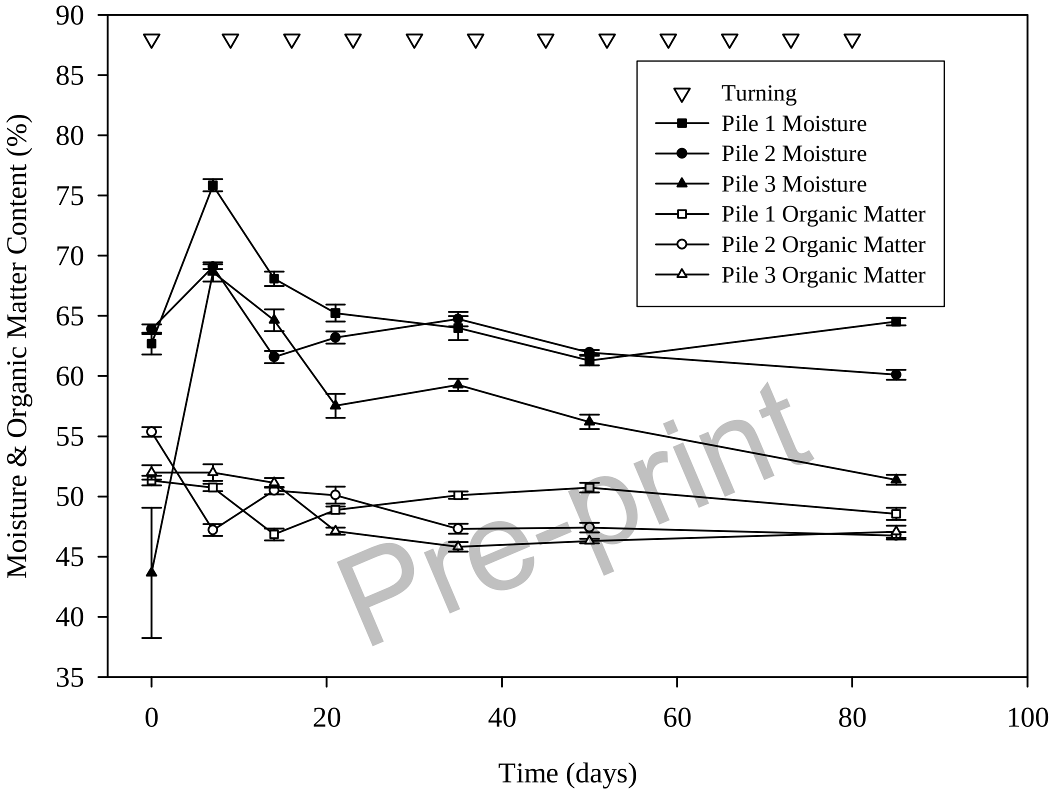

25
20
15
10
5
0
Oxygen (%)
0
20
40
60
80
100
Time (days)
Pile 1
Pile 2
Pile 3
Turning

Fig. 4..



Moisture & Organic Matter Content (%)
90
85
80
75
70
65
60
55
50
45
40
35
0
20
40
60
80
100
Time (days)
▽ Turning
Pile 1 Moisture
Pile 2 Moisture
Pile 3 Moisture
Pile 1 Organic Matter
Pile 2 Organic Matter
Pile 3 Organic Matter

Fig. 5.

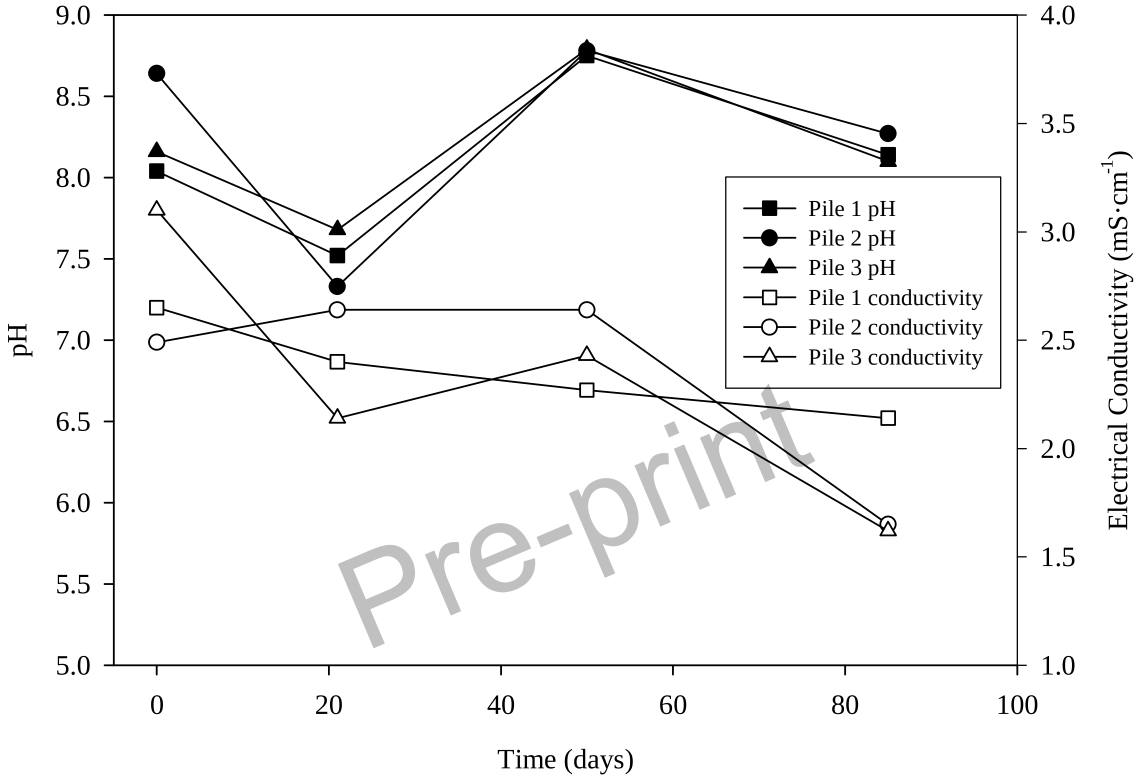

9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
pH
4.0
3.5
3.0
2.5
2.0
1.5
1.0
Electrical Conductivity (mS·cm^{-1})
0
20
40
60
80
100
Time (days)
Pile 1 pH
Pile 2 pH
Pile 3 pH
Pile 1 conductivity
Pile 2 conductivity
Pile 3 conductivity

Fig. 6

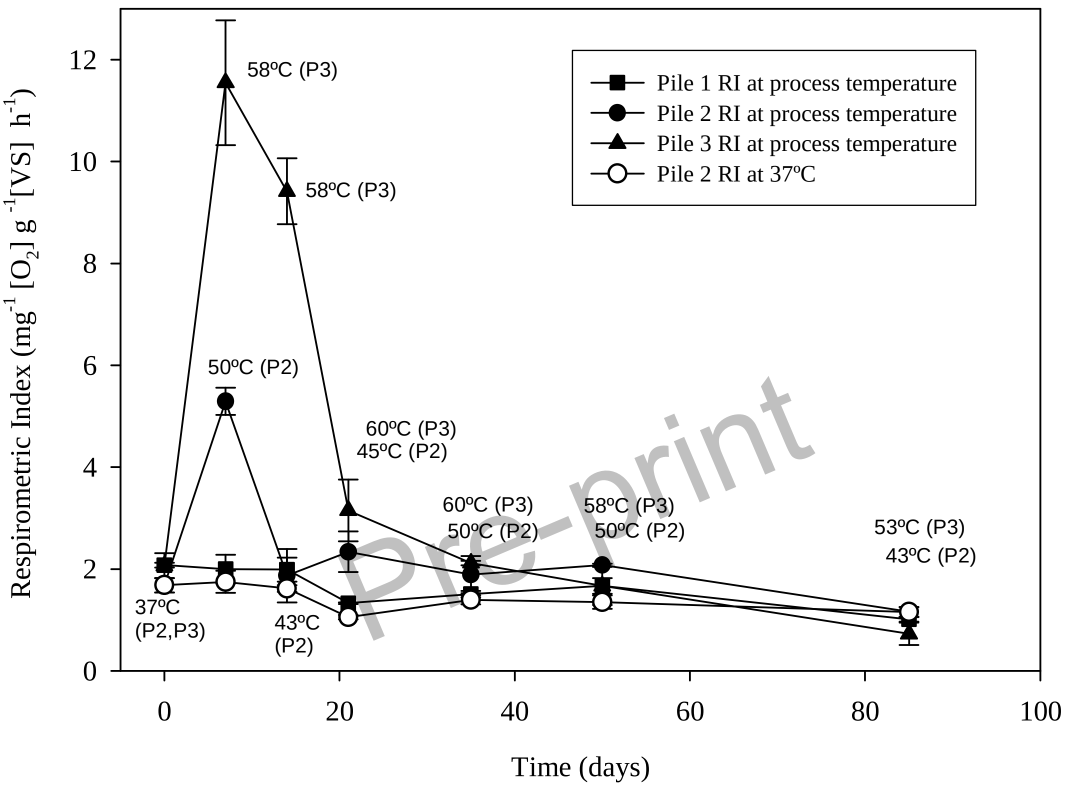
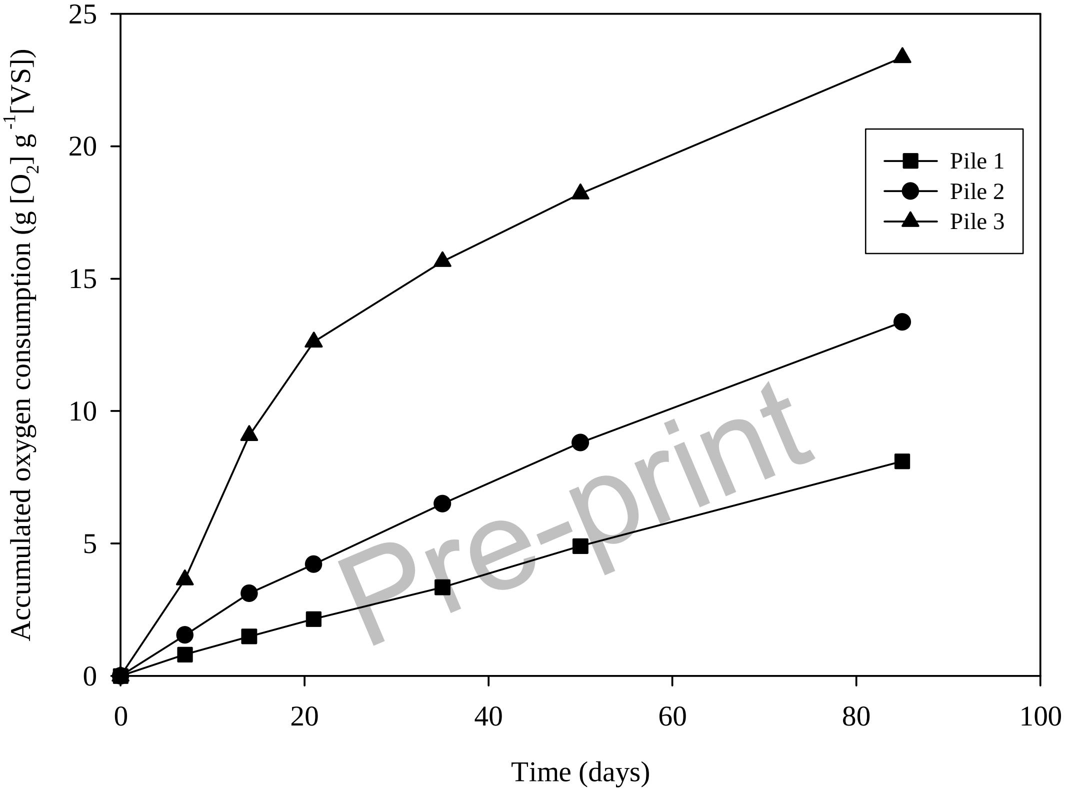

Respirometric Index (mg^{-1} [O$_2$] g^{-1}[VS] h^{-1})
Time (days)
0
2
4
6
8
10
12
0
20
40
60
80
100
Pile 1 RI at process temperature
Pile 2 RI at process temperature
Pile 3 RI at process temperature
Pile 2 RI at 37ºC
58ºC (P3)
58ºC (P3)
50ºC (P2)
60ºC (P3)
45ºC (P2)
60ºC (P3)
50ºC (P2)
58ºC (P3)
50ºC (P2)
53ºC (P3)
43ºC (P2)
37ºC
(P2,P3)
43ºC
(P2)

Fig.7


Accumulated oxygen consumption (g [O$_2$] g^{-1}[VS])
25
20
15
10
5
0
0
20
40
60
80
100
Time (days)
Pile 1
Pile 2
Pile 3