



Gaseous emissions and process development during composting of pig slurry: the influence of the proportion of cotton gin waste



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ABSTRACT

Composting is a feasible, environmentally-friendly management tool for the treatment of animal manures based on nutrient recovery. However, the liquid character of the pig slurry, with low dry matter, requires previous solid–liquid separation and the mixture of the solid fraction with an adequate bulking agent in the correct ratio. This work studies the influence of the bulking agent proportion on the composting of the solid fraction of pig slurry, focussing on the development of the process and on the greenhouse gas emissions. For this, two mixtures of the solid fraction of pig slurry and cotton gin waste, in different proportions, were prepared (4:3 and 3:4 solid fraction of pig slurry:cotton gin waste, v:v) and composted by the Rutgers static pile system in a pilot plant (2000 kg each pile). The temperature profiles of the composting piles were similar, but the pile with the greater proportion of bulking agent showed higher temperatures and a longer thermophilic phase, with greater aeration demand. The specific heat capacity of cotton is lower than those of other bulking agents used frequently for animal manure composting, which means that this material requires less energy from microbial activity for its temperature to increase. However, the easily degradable organic matter present in the pig slurry explained the faster organic matter mineralisation occurred in the pile with the higher proportion of solid fraction of pig slurry; in pile with higher proportion of bulking agent, the mineralisation process was slower due to the lignocellulosic character of the cotton gin waste. The organic matter mineralisation was closely related to the gaseous emissions (mainly as carbon dioxide): significant methane emissions were detected when the highest organic matter degradation occurred in the pile with the greater proportion of solid fraction of pig slurry, together with the highest nitrogen oxide emissions – indicating the existence of anaerobic pockets within the composting mass. So, it can be concluded that the thermal properties of the bulking agent were responsible for the temperature development and aeration demand, while the gaseous emissions were related to the organic matter degradation process. The composts produced were stable, with a good degree of maturation; the compost with the higher proportion of solid fraction of pig slurry had greater organic matter humification and higher nutrient concentrations.

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1. Introduction

The management of animal manures is one of the main problems that the current intensive farm production systems have to deal with in developed countries. According to Eurostat (2015), Spain is the country with the second-greatest pig production in Europe (after Germany). Within Spain, Murcia is the region with the fourth-greatest pig production (INE, 2015), with about 1.8×10^6

pigs (CARM, 2015). The annual pig slurry production in Murcia exceeds 6.5 million m³, concentrated mainly in two areas: Guadalentin Valley and Fuente Álamo (CARM, 2015), both with declared nitrate-vulnerable zones. This situation – the high concentration of pig farms within nitrate vulnerable areas – has led to the production of pig slurry in excess of the amount which can be applied directly to agricultural soil. Then, in order to avoid possible negative impacts on the environment and to comply with the national legislation, treatment technologies for the management of the pig slurry need to be established (Burton and Turner, 2003). There are different technologies for pig slurry treatment, based either on the

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recovery of plant nutrients or on their elimination (Sommer et al., 2014; Flotats et al., 2009).

Composting is a feasible, environmentally-friendly management tool for the treatment of these residues based on nutrient recovery (Huang et al., 2001), where organic matter (OM) is degraded into a stable and humified final product, free of pathogens and malodours, rich in nutrients, beneficial for plant growth and with agricultural value as a fertiliser (Bernal et al., 2009). Composting can minimise the potential environmental problems that can occur during storage of animal manures and after their direct soil application, such as greenhouse gas (GHG) emissions, the spread of pathogens and nutrient leaching into superficial and ground waters (Brown et al., 2008; Moral et al., 2005; Vinnerås, 2013).

However, pig slurry is a liquid material with low dry matter (<5%) and is rich in N (Moral et al., 2005). Thus, prior to composting, the physical and chemical properties should be improved by solid–liquid separation and the addition of a bulking agent (Nolan et al., 2011); for example, by adjusting the air space of the matrix, reducing the excessive moisture of the solid fraction of pig slurry (SFP) and adjusting the carbon to nitrogen (C/N) ratio, as high values lead to high N losses during composting (Bernal et al., 2009; Georgacakis et al., 1996; Nolan et al., 2011). Due to their availability on farms and ability to provide the optimal conditions for the development of the process, lignocellulosic materials such as barley and rice straws (Qian et al., 2014), maize stalks and other woody materials – sawdust and wood shavings (Huang et al., 2001; Ros et al., 2006) – have been used as bulking agents for pig slurry composting (Bernal et al., 1993; Hang et al., 2004; Nolan et al., 2011). According to Vanotti et al. (2006), the use of wood chips and cotton gin waste helped to reduce the bulk density of the pile and the moisture content, increasing the porosity and C/N ratio. Nolan et al. (2011) found that the addition of a bulking agent (sawdust, green waste or barley straw) to maintain the initial water content below 60% was necessary for composting of SFP at a low C/N ratio; they concluded that wood chips were not a suitable bulking agent due to the presence of recalcitrant lignin and its large surface area. The proportion of bulking agent appropriate for pig slurry composting depends on the initial characteristics of the separated solids, which are determined by the efficiency of the solid–liquid separator. Huang et al. (2004) concluded that increasing the proportion of sawdust in a mixture with pig manure increased the C/N ratio of the mixture, reducing the composting time and the salinity of the compost produced – the latter is mainly responsible for the phytotoxic effects in compost with a low proportion of bulking.

Also, the incorporation of a bulking agent has been reported as one of the most-effective measures to reduce GHG emissions during composting – since it improves the structure of the pile (waste or manure heap) while simultaneously reducing methane (CH₄) and nitrous oxide (N₂O) emissions (53% and 71%, respectively), without increasing substantially the N losses through ammonia (NH₃) volatilisation (Pardo et al., 2015). For reducing ammonia losses, Bernal et al. (1993) highlighted the relevance of the C supplied by the bulking agent and its particle size in the composting of pig manure.

A preliminary laboratory study for bulking agent selection for SFP composting, based on thermal evaluation in a self-heating test, revealed that cotton gin waste was a better bulking agent than cereal straw, maize stalks or pruning wastes, to control the excessive moisture content of the SFP and temperature development (Santos et al., 2014). Also, cotton gin waste has proved to be an adequate material to control the excessive moisture content of certain liquid wastes (Paredes et al., 1996), promoting the development of thermophilic temperatures during the composting process and hence ensuring the sanitation of the final product.

Therefore, the aim of this study was to assess the influence of the proportion of cotton gin waste on the thermal profile, chemical evolution of the solid fraction of pig slurry during composting and the environmental implications regarding GHG emissions.

2. Material and methods

The solid fraction of pig slurry (SFP) was obtained from a fattening pig farm after a solid–liquid separation with a screw press. Cotton gin waste was used as a bulking material. The main characteristics of both initial materials are shown in Table 1.

2.1. Experimental design and conditions

Two composting mixtures (M1 and M2) were prepared by mixing SFP with cotton gin waste, considering the moisture content and C/N ratio of both materials: M1, with a ratio of 4:3 (SFP:cotton gin; v/v); and M2, with a ratio of 3:4 (SFP:cotton gin). The experiment was run in a pilot-scale composting plant. The mixtures were set up in trapezoidal piles of 2000 kg each, with a base of 2.7 × 3.7 m and a height of 1 and 1.2 m, for M1 and M2, respectively, and composted using the Rutgers static pile composting system with forced aeration at temperature demand, set to 65 °C. Aeration was programmed, occurring every 120 min for: 1 min if T < 55 °C; 2 min if 55 °C < T < 60 °C; 3 min if 60 °C < T < 65 °C; and continuously if T ≥ 65 °C, until T ≤ 60 °C. The ventilation demand was calculated as the time (hours per day) that the pump was blowing continuously (T > 65 °C). The piles were turned twice throughout the process, when the temperature started to decrease, in order to improve both the homogeneity of the materials and the composting process. The moisture of the piles was controlled weekly by adding the amount of water necessary to keep the moisture content above 40%. The evolution of the temperature was monitored using two temperature probes for each pile, at different places in the pile. The bio-oxidative phase of composting was considered finished when the temperature inside the pile was close to the external temperature and re-heating did not occur after turning. Then, the ventilation was turned off and the composts were left to mature over a period of two months.

During the 167 days that the composting process lasted, ten samples were taken to assess the evolution of the mixtures. The samples were taken by mixing seven sub-samples from seven representative sites of the pile, from the whole profile (from the top to the bottom of the pile). Each representative sample was divided into three fractions in the laboratory: one was dried in a drying-oven at 105 °C for 24 h to determine the moisture content, the second was immediately frozen (−18 °C) and kept for the

Table 1
Characteristics of the initial materials (dry weight basis).

	Cotton gin waste	Solid fraction of pig slurry
pH	6.97	7.31
EC (dS m ⁻¹)	7.15	6.85
Moisture (%) ^a	14	75
OM (%)	87.9	68.6
TOC (g kg ⁻¹)	534	356
C _w (g kg ⁻¹)	n.d.	5.41
TN (g kg ⁻¹)	10.4	32.8
NH ₄ ⁺ -N (g kg ⁻¹)	0.33	10
C/N ratio	51.3	10.9
Lignin (%)	49.9	n.d.

EC: electrical conductivity; OM: organic matter; TOC: total organic carbon; C_w: water-soluble carbon; TN: total nitrogen.

n.d.: not determined.

^a Calculated based on fresh weight.

determination of ammonium nitrogen ($\text{NH}_4^+\text{-N}$) and the third was freeze-dried and ground to less than 0.5 mm for the rest of the analytical determinations.

2.2. Physico-chemical and chemical analysis

The raw materials and the composting samples were analysed according to standard procedures: electrical conductivity (EC) and pH in a water-soluble extract 1:10 (w/v); OM by loss-on ignition at 550 °C for 24 h; total organic carbon (TOC) and total nitrogen (TN) by automatic microanalysis (EuroVector EuroEA 3000 elemental analyser) (Navarro et al., 1991); water-soluble organic carbon (C_w) in a 1:10 (w/v) water extract, extractable carbon (C_{ext}) in a 1:20 (w/v) sodium hydroxide (NaOH) extract and fulvic acid-C (C_{FA}) after precipitation of the humic acid-C (C_{HA}) at pH < 2 using sulphuric acid (H_2SO_4), all analysed by an automatic analyser for liquid samples (TOC-V CSN Analyser, Shimadzu); ammonium nitrogen ($\text{NH}_4^+\text{-N}$) by a colorimetric method based on Berthelot's reaction (Sommer et al., 1992), after potassium chloride (2 M KCl) 1:20 (w/v) extraction; nitrate with a selective electrode in a 1:20 water extract; cation exchange capacity (CEC) with barium chloride (BaCl_2)-triethanolamine (Lax et al., 1986); and macro and micro-nutrients by inductively coupled plasma-optical emission spectrometry (ICP-OES, Thermo Elemental Co. Iris Intrepid II XDL) after nitric acid and hydrogen peroxide ($\text{HNO}_3\text{-H}_2\text{O}_2$) digestion in a microwave oven. The germination index (GI) was determined using seeds of *Lepidium sativum* L., according to Zucconi et al. (1981).

The emission flow of the GHGs (carbon dioxide – CO_2 , methane – CH_4 and nitrous oxide – N_2O) was measured at the surface of the pile using the closed static chamber system (Sommer et al., 2004; Sánchez-Monedero et al., 2010). For this, two chambers (both 0.055 m² and 0.01 m³) were placed at two different points of the pile. The gas samples were taken every 5 min for 30 min with a syringe and the gas sampled was injected into two different containers: 30 ml into a 0.1 M NaOH trap, for CO_2 determination by HCl titration, and 10 ml into a vacuum tube (Vacutainer) for CH_4 and N_2O determination by gas chromatography (HP5890, column HP Innovax with Flame Ionization Detector for CH_4 , and HP 4890D equipped with an electron capture detector – ECD for N_2O). The flow rate emissions ($\text{g m}^{-2} \text{day}^{-1}$) were calculated according to Hao et al. (2001). All the samples were taken after turning off the ventilation system. All the analytical determinations were performed at least in duplicate.

The energy produced by microbial activity during the composting process was calculated according to the Eq. (1) (Santos et al., 2014):

$$q = m \times c \times \Delta T \quad (1)$$

where q is the energy produced (kJ), m is the mass of compost (kg), c is the heat capacity of the compost ($\text{kJ kg}^{-1} \text{K}^{-1}$) and ΔT is the increase in temperature from the room to the internal values of the composting mass.

The OM-loss by mineralisation was calculated, using the initial (X_1) and final (X_2) ash concentrations, with the following the Eq. (2) (Bernal et al., 1996):

$$\text{OM loss (\%)} = 100 - \frac{100[X_1(100 - X_2)]}{[X_2(100 - X_1)]} \quad (2)$$

2.3. Statistical analysis

The standard error of the means was calculated for the chemical and physico-chemical characteristics of the composting samples;

one-way analysis of variance (ANOVA) and least significant difference (LSD) were used for determining changes of the parameters during composting. The normality and homogeneity of the variances were checked using the Shapiro–Wilk and Levene test, respectively, before ANOVA. The IBM SPSS Statistics v.19 software package was used for the statistical analyses. The OM mineralisation (OM-loss) data were fitted to a first-order kinetic model (Eq. (3)) by a non-linear least-square technique (Marquardt–Levenberg algorithm), using the SigmaPlot v. 12.0 Systat Software Inc.:

$$\text{OM}_{\text{min}} = A \times (1 - e^{-kt}) \quad (3)$$

where OM_{min} is the mineralised OM (%) at the composting time t (day), A is the potentially mineralisable OM (%) and k is the rate constant (day^{-1}). The goodness of curve fitting was estimated according to the residual mean square (RMS) and the significance of the F-value of the ANOVA.

3. Results and discussion

The solid fraction of pig slurry had a slightly alkaline character (Table 1) with an excessive moisture content (75%) for composting without a bulking agent (Bernal et al., 2009), which could cause leaching and anaerobic conditions within the composting mass. The low moisture content of the cotton gin waste (Table 1) can compensate the excess moisture of the SFP. Both materials were rich in OM, but SFP was richer in Cw (easily degradable by micro-organisms) and TN (30.5% as $\text{NH}_4^+\text{-N}$) with a low C/N ratio for composting; all these factors could lead to high ammonia losses by volatilisation if not equilibrated by the bulking agent (Bernal et al., 2009).

3.1. Evaluation of the thermal profiles of the composting piles

The thermal profiles of both composting piles showed a fast increase in temperature during the first week of experiment, when both piles achieved values close to 65 °C (Fig. 1a). Such quick increases indicate fast biodegradation of the materials due to the presence of easily-degradable forms of TOC in the mixtures (Bernal et al., 2009). During the thermophilic phase, both piles showed a similar trend (Fig. 1a), reaching temperatures above 55 °C for more than 35 days, longer than the time required to ensure hygienic conditions in the compost (European Commission, 2001; JRC and IPTS, 2012). However, the thermophilic phase was shorter in M1 than in M2 (50 and 80 days, respectively). The increment of temperature with respect to the external values indicates that the exothermic process was stronger in M2 (1640 °C Mg^{-1}DM) than in M1 (1190 °C Mg^{-1}DM), which may be due to the thermal properties of the cotton gin waste – present in a greater proportion in M2. Similar results were found by Yañes et al. (2009) during composting of sewage sludge with garden prunings; mixtures with a greater proportion of bulking agent showed a longer thermophilic phase. Also, Huang et al. (2001) found that partial substitution of sawdust by garden waste (leaves), in the composting of pig manure, improved composting efficiency by shortening the time required for maturation. However, Ros et al. (2006) found a two-week delay in the temperature development during composting of SFP, with or without wood shavings as a bulking agent. All these findings indicate that the proportion and characteristics of the bulking agent affect the temperature development during composting.

Temperature variations during composting are the result of the thermal balance between the heat generated by the microorganisms and the heat loss through convection, conduction, evaporation and radiation (Haug et al., 1993). Thus, the energy generated by

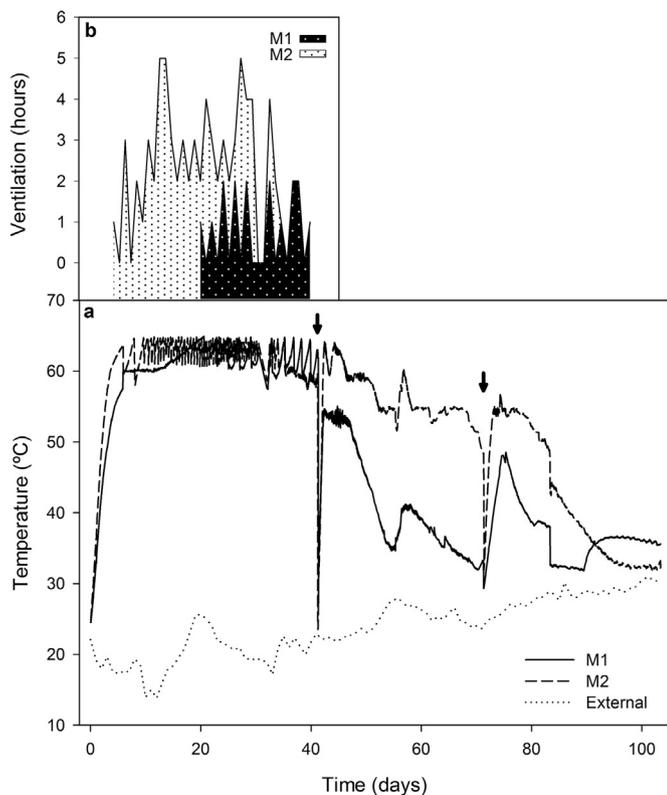


Fig. 1. Thermal profile of the composting piles (a); and ventilation demand to maintain the temperature of the piles below 65 °C during the bio-oxidative phase of the composting process (b). M1: solid fraction of pig slurry + cotton gin waste (4:3, v:v); M2: solid fraction of pig slurry + cotton gin waste (3:4, v:v). Arrows indicate the turnings.

microorganisms and its effect on the temperature of the composting mass depend on the thermal properties of the bulking agent and its proportion. Cotton gin waste is a material with good thermal properties, with a calorific value of 14.7 MJ kg⁻¹ (Zabaniotou et al., 2007), higher than wood chips (12.2 MJ kg⁻¹) (Francescato et al., 2008). Also, the specific heat capacity of cotton (1.34 kJ kg⁻¹ K⁻¹) is comparable to those of several kinds of wood and other biomasses (soybean straw or corn stalks), but is lower than those of wheat straw, silage, oat straw, alfalfa hay and leaves and wood shavings, which are frequently used as bulking agents for animal manure composting (Bernal et al., 2009; Ros et al., 2006), and beef manure or turkey litter (Ahn et al., 2009) – which means that this material requires less energy for its temperature to increase. This property is directly related to the moisture content (Ahn et al., 2009); thus, SFP would have a higher specific heat capacity than cotton gin waste, and the higher the proportion of SFP in the mixture the greater the energy necessary to increase its temperature. The specific heat capacity may also explain the delay in reaching thermophilic temperatures when composting SFP with wood shavings (Ros et al., 2006), showing cotton gin waste to be a more-adequate bulking agent.

The energy produced in the composting mass was higher in M2 than in M1 (8556 and 7837 MJ, respectively; calculated according to Eq. (1)); this is related to the greater length of the thermophilic phase in M2, due to the high microbial activity and the thermal properties of the cotton gin waste. Ventilation demand is a good indicator of microbial activity during the composting process (Haug, 1993). In the present experiment, the high amount of energy produced by the microbial activity and converted into heat meant that the ventilation demand was higher in M2 than in M1 (74 and

16 h of continuous air-blowing, respectively, during the thermophilic phase; Fig. 1b). This was associated with the higher proportion of bulking agent in M2 and the thermal properties of the cotton waste. Then, in M2, the energy generated by microbial activity was efficiently converted into heat within the composting mass, requiring greater ventilation for temperature control. During the extra-ventilation period in M2 a great amount of heat was lost and the oxygen concentration within the mass was enhanced.

The temperature increased after each turning in both piles, especially for the second turning at day 71, indicating that the piles were still microbiologically active. The reactivation of the composting process by the turnings was also observed during composting of the solid fraction of anaerobically-digested pig slurry or animal manure with different bulking agents (Bustamante et al., 2013; Tortosa et al., 2012). This is due to the incorporation of poorly-degraded material from the external part of the pile into the internal part, where biological activity is greatest. During composting, as the OM becomes more stabilised, the microbial activity and the OM decomposition rate decrease and the temperature gradually decreases to the ambient level (Haug, 1993; Ros et al., 2006; Paredes et al., 2001), indicating the end of the bio-oxidative phase, which in the present experiment occurred at 103 days.

3.2. Evolution of the physico-chemical and chemical parameters and GHG emissions during the composting process

In both piles, most of the physico-chemical and chemical parameters showed a similar pattern along the process (Table 2). The pH slowly decreased during the first 56 days, probably due to the degradation of the OM and NH₃-volatilisation, with a quick decrease after this day until the end of the composting process due to nitrification, an acidification process (Tarre and Green, 2004). Similar results were found when sawdust was composted with raw and digested pig manure (Troy et al., 2012), and are frequently found during composting (Bernal et al., 2009).

The EC values increased during the process (Table 2), as OM mineralisation leads to an increase in inorganic compounds in the compost matrix (Moreno and Moral, 2008). According to Eq. (2), such OM mineralisation gave a reduction in the OM concentration of 64.4 and 50.5% (M1 and M2, respectively) during the process. As a microbial-mediated process, the OM mineralisation can be described by a first-order kinetic model (Paredes et al., 2001; Bernal et al., 1996; Haug, 1993), in which the rate of decomposition depends on the concentration of potentially degradable substrate (OM): $dOM/dt = -K \times OM$.

The integration of the expression from time 0 to “t” gives the usual form of the equation shown in Eq. (3). The use of such a model allows us to discuss the degradation process in kinetic terms. The experimental data of OM degradation in both piles fitted the first-order kinetic model (Eq. (3)) with a high probability level ($p < 0.0001$) (Fig. 2), and the following parameters were obtained:

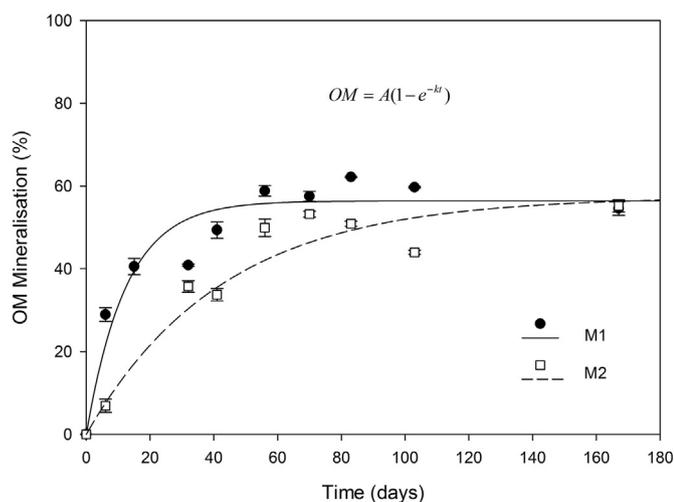
M1: $A = 56.4 \pm 2.5$; $k = 0.0803 \pm 0.0183$; RMS = 33.186, $F = 91.03$, $p < 0.0001$, $R^2_{adj} = 0.909$;

M2: $A = 57.4 \pm 8.0$; $k = 0.0235 \pm 0.0084$; RMS = 71.208, $F = 56.76$, $p < 0.0001$, $R^2_{adj} = 0.861$.

Therefore, an increase in the proportion of bulking agent did not statistically change the concentration of degradable OM (parameter A did not differ statistically between the piles), but led to a significant decrease in the degradation rate (the rate constant, k , decreased from M1 to M2). The product of $A \times k$ indicates that the initial OM mineralisation rate was faster for M1 (4.53% OM d⁻¹), elaborated with a higher proportion of SFP, than for M2 (1.35% OM d⁻¹),

Table 2Evolution of the physico-chemical and chemical parameters of the two piles (M1 and M2) during the composting process (mean \pm standard deviation).

Composting time (days)	pH	EC (dS m ⁻¹)	OM (%)	TOC (g kg ⁻¹)	C _w (g kg ⁻¹)	TOC/TN
<i>M1: SFP + cotton gin waste (4:3, v:v)</i>						
0	7.43 \pm 0.03	5.73 \pm 0.10	79.1 \pm 0.8	363 \pm 3	27.5 \pm 0.7	18.6 \pm 0.6
6	7.32 \pm 0.02	5.64 \pm 0.14	72.3 \pm 1.1	359 \pm 8	14.9 \pm 1.0	15.8 \pm 1.7
15	7.35 \pm 0.03	6.65 \pm 0.05	69.1 \pm 1.0	355 \pm 13	21.3 \pm 1.3	13.8 \pm 1.2
32	7.27 \pm 0.02	7.19 \pm 0.16	68.4 \pm 1.2	355 \pm 5	21.4 \pm 1.2	11.4 \pm 0.6
41	7.28 \pm 0.03	6.80 \pm 0.20	65.5 \pm 0.8	347 \pm 12	20.1 \pm 1.1	11.0 \pm 0.4
56	7.28 \pm 0.03	6.44 \pm 0.15	62.1 \pm 0.8	336 \pm 13	14.9 \pm 0.1	10.9 \pm 1.3
70	7.12 \pm 0.02	6.67 \pm 0.06	61.1 \pm 1.1	335 \pm 7	12.1 \pm 0.8	10.1 \pm 0.2
83	6.92 \pm 0.02	5.80 \pm 0.10	58.3 \pm 1.1	316 \pm 7	9.2 \pm 0.15	10.3 \pm 0.3
103	6.78 \pm 0.02	9.43 \pm 0.14	56.7 \pm 0.6	311 \pm 14	7.8 \pm 0.20	9.3 \pm 0.3
167	6.77 \pm 0.02	10.70 \pm 0.17	57.3 \pm 0.6	303 \pm 10	7.0 \pm 0.10	8.6 \pm 0.3
ANOVA	***	***	***	***	***	***
LSD	0.04	0.23	1.6	17	1.35	1.5
<i>M2: SFP + cotton gin waste (3:4, v:v)</i>						
0	7.81 \pm 0.02	6.23 \pm 0.19	73.3 \pm 0.9	357 \pm 6	24.7 \pm 0.3	16.3 \pm 0.4
6	7.52 \pm 0.02	5.70 \pm 0.19	71.9 \pm 0.3	339 \pm 8	19.3 \pm 1.0	15.6 \pm 0.4
15	7.57 \pm 0.03	6.55 \pm 0.06	70.9 \pm 0.8	333 \pm 5	21.1 \pm 2.0	14.7 \pm 1.2
32	7.57 \pm 0.03	6.64 \pm 0.06	63.9 \pm 1.0	327 \pm 8	19.3 \pm 1.2	11.1 \pm 0.2
41	7.55 \pm 0.04	6.19 \pm 0.09	62.2 \pm 0.8	336 \pm 11	18.9 \pm 1.5	12.2 \pm 0.5
56	7.43 \pm 0.02	5.13 \pm 0.07	56.2 \pm 1.7	308 \pm 7	18.0 \pm 2.0	11.1 \pm 0.5
70	7.36 \pm 0.04	5.76 \pm 0.05	55.2 \pm 1.1	307 \pm 9	12.5 \pm 1.9	10.6 \pm 1.2
83	7.18 \pm 0.03	6.86 \pm 0.17	56.0 \pm 1.7	305 \pm 10	10.3 \pm 0.7	9.7 \pm 0.7
103	6.92 \pm 0.02	7.80 \pm 0.13	57.3 \pm 1.2	298 \pm 11	8.1 \pm 1.0	9.1 \pm 0.4
167	6.85 \pm 0.03	10.06 \pm 0.25	57.2 \pm 1.4	292 \pm 8	7.3 \pm 0.8	9.4 \pm 0.3
ANOVA	***	***	***	***	***	***
LSD	0.05	0.24	2.0	15	2.30	1.1

EC: electrical conductivity; OM: organic matter; TOC: total organic carbon; C_w: water-soluble carbon; TN: total nitrogen. SFP: solid fraction of pig slurry.***: significant at $p < 0.001$. LSD: least significant difference at $p < 0.05$.**Fig. 2.** Organic matter mineralisation during composting of the two piles, M1: solid fraction of pig slurry + cotton gin waste (4:3, v:v); M2: solid fraction of pig slurry + cotton gin waste (3:4, v:v). The dots are the experimental data and the lines are the curve fittings.

showing quick mineralisation in the first 20 days and reaching its maximum after 40 days of composting. However, M2 showed a slower OM mineralisation rate, stabilising after 60 days. The quicker OM mineralisation in M1 was associated with high microbial activity at the beginning of the process, as observed in the temperature profile (Fig. 1a), a consequence of the high amount of easily-degradable material from the SFP. In M2, the higher amount of recalcitrant compounds, such as lignin, provided by the cotton gin waste can explain the slower OM mineralisation. However, the temperature remained at thermophilic values for a long time, which

may be due to the thermal characteristics of cotton gin waste and also the degradation of polymers (cellulose) from cotton. Similar results were found by Nolan et al. (2011), when composting the solid fraction of pig slurry with several different bulking agents at a low C/N ratio; they observed lower OM mineralisation for the mixtures made with materials rich in lignin, such as sawdust, shredded green waste, chopped straw and wood chips. Also, Doublet et al. (2010) found that increasing the proportion of bulking agent slowed down the OM mineralisation rate. The degradation rates found in M1 were similar to those found by Paredes et al. (2001) and Bernal et al. (1996) (4.48 and 4.00% OM d⁻¹) when co-composting sewage sludge with cotton gin waste and animal manure with sorghum bagasse, respectively.

The OM mineralisation was also manifested in the TOC concentration (Table 2), which was reduced by 16.5 and 18.2% in M1 and M2, respectively. A lesser reduction in the TOC concentration occurred when wood shavings were used as a bulking agent (12%; Ros et al., 2006), reflecting their more-lignified and degradation-resistant character. Taken into account the mass-loss of the piles, the TOC losses by mineralisation at the end of the process averaged 50% (52 and 47% for M1 and M2, respectively), indicating the consumption of the easily-degradable forms of C, due to the high microbial activity during the thermophilic phase. The water-soluble organic compounds (C_w) are the readily-available C source for microorganisms, as their metabolism occurs in the liquid phase. At the beginning of the composting process, the C_w concentration decreased in both piles (Table 2), but formation of soluble organic compounds was detected after 15 days, mainly in M1, indicating the hydrolysis of complex, insoluble organic compounds, such as hemicellulose or cellulose (Charest et al., 2004; Ros et al., 2006). According to Ros et al. (2006), the microorganisms responsible for the composting process use water-soluble compounds as energy sources for their growth and multiplication. This suggests that newly-formed glucose or other forms of C_w may have originated

from microorganisms during this process – giving an alternate decrease and increase in the C_w concentration during the degradation process, as found in M1 and M2 at 15d, related to the synthesis of new carbohydrates of microbial origin. The C_w concentrations were lower in M2 than in M1 during the thermophilic phase, due to the lower proportion of SFP and higher proportion of lignocellulosic cotton gin waste in M2. Tortosa et al. (2012) also found a lower C_w concentration when a higher proportion of bulking agent was used in the composting of two-phase olive mill waste with two different types of animal manure. At the end of the process, the C_w concentration had decreased by 84% (86 and 82% for M1 and M2, respectively), indicating the consumption of readily-degradable OM; this parameter is considered a compost maturity index (Bernal et al., 1998).

The TN concentration increased in both piles during the process (Fig. 3), mainly due to a concentration effect as a result of the weight reduction of the mass, caused by the OM degradation. Huang et al. (2001) and Zhu et al. (2004) also found an increase in the TN concentration along the process, when composting SFP with leaves and rice straw, respectively. The evolution of TOC and TN decreased the TOC/TN ratio during the composting process (Table 2). Fels et al. (2014) explained that the C/N decrease can be attenuated if high losses of N as ammonia occur during the

thermophilic phase. However, at the end of the process, the TN losses were around 20%, lower in M1 (17%) than in M2 (22%). Such values are in the low part of the range reported for the composting of pig slurry or pig manure, as reviewed by Bernal et al. (2009). Similar losses were found by Paredes et al. (1996), when composting poultry manure with cotton gin waste, and by Bustamante et al. (2008), when composting cattle manure with distillery wastes. At the beginning of the process (15 days) the TN loss was lower in M1 (5%) than in M2 (10%), which can be due to the higher ventilation demand in M2, promoting water evaporation and NH_3 -volatilisation. It is well known that high temperatures and aeration, together with alkaline pH and a high NH_4^+ -N concentration in the material during the thermophilic phase, favour NH_3 -volatilisation (Bernal et al., 2009; Tiquia et al., 2002). In fact, the NH_4^+ -N concentration decreased along the composting process in both mixtures (M1 and M2), but the decrease was faster in M2 (Fig. 3).

Losses of N can be due to NH_3 -volatilisation, denitrification, nitrification and NO_3^- leaching; microbial N-immobilisation could also occur, mainly at the beginning of the composting process, when NH_4^+ -N-rich materials are co-composted with a C-rich material (Bernal et al., 1996; Ros et al., 2006). The composting conditions – the number of turnings, aeration of the piles, temperatures obtained during the thermophilic phase, initial NH_4^+ -N concentration and bacterial nitrification activity – affect N-losses (Bernal et al., 2009). According to Sánchez-Monedero et al. (1996), the Rutgers static system can reduce N-losses by NH_3 -volatilisation, with respect to the turning systems, due to the direct contact of the internal gaseous phase with the atmosphere during turning. However, recent meta-analysis studies revealed the existence of high NH_3 losses during static pile composting (Pardo et al., 2015). Also, no or little NO_3^- -N leaching could have occurred in the present experiment, and at the beginning of the process the NO_3^- -N concentration remained very low, because high temperatures inhibit the activity of nitrifiers (Tiquia et al., 2002). Hence, NH_3 -volatilisation should be the main factor responsible for N-losses at the beginning of the process. Further losses can be due to denitrification, which can occur in anaerobic microsites within the material, although the aeration programme applied, dependent on the temperatures, should have ensured adequate aerobic conditions.

The NO_3^- -N concentration increased during the composting process for both mixtures (Fig. 3), when the internal temperature of the piles started to decrease from thermophilic values (Tiquia et al., 2002; Ros et al., 2006; Bernal et al., 2009). Nitrification occurs when temperatures are $<40^\circ\text{C}$ and the aeration is favourable (Sánchez-Monedero et al., 2001), consistent with the static composting system and the observed formation of nitrate after the drop in temperature. Similar results were found by other authors when co-composting SFP (Zhu et al., 2004; Ros et al., 2006; Bernal et al., 2009) or sewage sludge (Fels et al., 2014) with several bulking agents.

The mass balances in both piles indicate that a high proportion of the initial inorganic N (up to 80%) was lost during the thermophilic phase, but formation of inorganic-N (as nitrate) was detected as the temperature went down, especially in M1 during the maturation phase, giving net organic-N mineralisation. As for TN, there was a balanced result for the inorganic-N during the process in M1: although, initially, a high proportion of inorganic-N (pre-existing and formed by mineralisation) was lost, the later formation gave a similar proportion of TN as of inorganic-N in the mature compost, with respect to the initial mixture. However, the mass balance for M2 indicates an overall loss of inorganic-N in the mature compost, equivalent to 27% of that initially present, with scarce organic-N mineralisation during maturation. So, the proportion of TN present as inorganic-N was reduced from 18.8% in the initial mixture to 14% in the mature compost (Table 2). These results

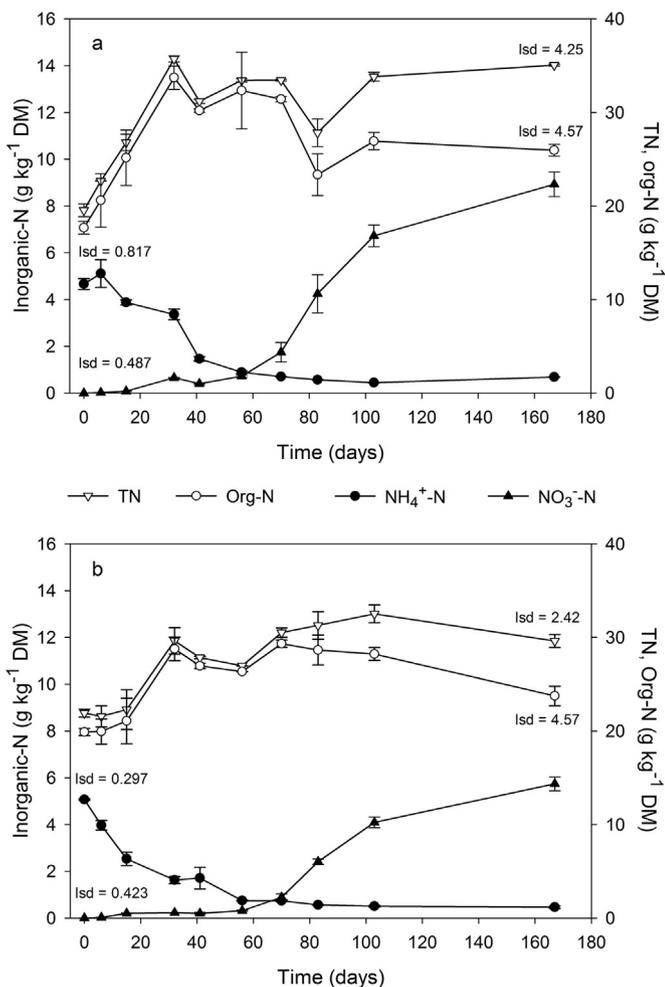


Fig. 3. Evolution of the N-forms during the composting process in M1 [solid fraction of pig slurry + cotton gin waste (4:3, v:v)] (a) and M2 [solid fraction of pig slurry + cotton gin waste (3:4, v:v)] (b). Right-hand axes for TN and organic-N, left-hand axes for inorganic-N (NH_4^+ -N, NO_3^- -N).

Table 3
Flow rates of the gaseous emissions from piles M1 and M2 during the thermophilic phase of composting.

Composting time (days)	CO ₂ -C (g m ⁻² d ⁻¹)	CH ₄ -C (g m ⁻² d ⁻¹)	N ₂ O-N (g m ⁻² d ⁻¹)
<i>M1: SFP + cotton gin waste (4:3, v:v)</i>			
0	104	—	—
6	131	<0.01	—
15	253	0.46	5.99
32	459	11.6	11.3
41	245	0.67	4.69
56	22	<0.01	0.08
<i>M2: SFP + cotton gin waste (3:4, v:v)</i>			
0	—	—	—
6	88	0.30	1.6
15	167	—	0.97
32	141	2.44	4.02
41	147	4.20	6.04
56	27	<0.01	0.05

can be very relevant for the agronomic value of the composts as fertilisers.

Gaseous emissions of anthropogenic CO₂ and GHGs (N₂O and CH₄) were measured during the thermophilic phase (Table 3), before the second turning (active phase). The main gases generated by the microbial degradation of OM during composting are CO₂ and CH₄, and N₂O is formed through both nitrification and denitrification of the NH₄⁺-N and NO₃⁻-N contained in the composting mixtures, when oxygen is limited (Sánchez-Monedero et al., 2010; Szanto et al., 2007; Kampschreur et al., 2009). Carbon dioxide emissions increased during the first 32 days in M1, which showed higher emissions than M2-associated with the already-discussed intense OM mineralisation caused by the high amount of SFP in M1 (Table 3). The values decreased after 56 days, in both piles. The results are well related to the OM mineralisation (losses of TOC and C_w) and the kinetic parameters of the process (Fig. 2), which indicate maximum OM mineralisation in M1 after 40 days (with the maximum CO₂ emission) and slower OM mineralisation in M2. Similar results were reported by Hao et al. (2004) when composting cattle manure, but higher values were found during sheep manure composting (Sánchez-Monedero et al., 2010) and when SFP was composted without any bulking agent (Pampuro et al., 2014), using the turning system.

Methane is formed in anaerobic microsites that occur inside the piles due to insufficient aeration associated with intense microbial activity and O₂ consumption and/or the compaction of the pile due to its excessive height (Fukumoto et al., 2003; Beck-Friis et al., 2000; Sommer and Moller, 2000). The CH₄ emissions were greatest at 32 days in M1 (Table 3), when microbial activity was intense and the ventilation demand was also high in this pile, indicating high oxygen demand for microbial degradation of the OM. However, the values of CH₄-C were very low in comparison with CO₂-C, indicating that the degradation of the OM occurred in a mainly aerobic environment. The CH₄-C emission values were lower than those found by Hao et al. (2004) and Sánchez-Monedero et al. (2010), during composting of animal manures, and by Pampuro et al. (2014) when SFP was composted without bulking agent using the turning pile system. Only the result on day 32 in M1 was close to the results found by Pampuro et al. (2014). It has been proved that CH₄ is oxidised to CO₂ when air passes through the pile, especially in the Rutgers static composting system – where air is blown from the bottom of the pile (Hao et al., 2001). In the turning system, the CH₄ emission rate decreases immediately after each turning operation (Pampuro et al., 2014), as most of the emissions occur during the turnings. Brown et al. (2008) suggested that the

gases present in the interior of the piles were released as the material from the interior was brought to the surface during turning; but, in static systems, diffusion of these gases to the atmosphere through the pile was limited by decomposition of the compounds in the aerated surface section of the pile. In M2, the CO₂-C and CH₄-C values followed a similar pattern but were lower than in M1, although M2 required longer extra ventilation periods than M1. As previously discussed, OM mineralisation was slower in M2 (with a higher proportion of bulking agent) than in M1, indicating that the thermal characteristics of the cotton gin waste conditioned the temperature reached during the process and the heat diffusion by air ventilation (Keener et al., 1993).

The highest CH₄ and N₂O emission rates were found before the first turning in M1 (Table 3), indicating the presence of anaerobic microsites – probably due to pile settlement (in M1, with less bulking agent) that reduced the pore spaces and consequent O₂ availability, although the extra ventilation by temperature demand should have provided an O₂ concentration sufficient to guarantee aerobic conditions. During the composting of the solid fraction of pig slurry with wood chips, Vázquez et al. (2015) found that a low proportion of bulking agent reduced the O₂ concentration in the pile, which could have created anaerobic conditions. The proportion of bulking agent in the mixture and the turning frequency of the pile in a turned windrow system were key factors determining the O₂ concentration inside the composting pile (Vázquez et al., 2015). Substantial CH₄ emissions after the first turning event, during composting of dairy manure, were also reported by Ahn et al. (2011).

Also, as mentioned before, N₂O can be formed through both nitrification and denitrification of NH₄⁺-N and NO₃⁻-N in the material, when the O₂ concentration is limited. The N₂O emissions found before the first turning were much higher than those found by other authors (Hao et al., 2004; Sánchez-Monedero et al., 2010), perhaps due to the high NH₄⁺-N concentration provided by the SFP. The most-probable mechanism of N₂O formation was through nitrification, since the nitrate concentration during the bioactive phase was low and NH₄⁺-N was the predominant form of inorganic-N. It seems that N₂O emissions were responsible for a relevant fraction of the TN losses. After 41 days, the emissions were reduced drastically, indicating that the turning event supplied the O₂ needed to eliminate anaerobic microsites (emissions of CH₄ and N₂O were not detectable).

3.3. Compost maturity and quality

Compost maturity is considered a compost quality indicator (Bernal et al., 1998). In this sense, several parameters have been established to define compost maturity (Bernal et al., 1998, 2009; Itavaara et al., 2002; Benito et al., 2003; Chang and Chen, 2010). Both composts reached adequate values for the maturity indices (Table 4): NH₄⁺-N/NO₃⁻-N ratio < 0.16, C_w concentration < 10 g kg⁻¹, C_w/TN ratio < 0.7, water-soluble C/N < 16, CEC > 67 meq 100 g⁻¹ OM, CEC/TOC ratio > 1.7 and GI > 50%, all indicative of a good degree of maturation. The only exception was the NH₄⁺-N concentration (higher in M1 than in M2), which was slightly higher than the limit value (<0.4 g kg⁻¹) established (Zucconi and de Bertoldi, 1987; Bernal et al., 1998). Hence, M1 seems to have required a maturation period longer than two months, due to the high concentration of inorganic-N of the SFP.

Maturation implies the formation of humic-like substances, and the degree of OM humification (HR, HI, PHA and PI) is commonly used as a criterion for compost maturity (Bernal et al., 2009). However, most of the limits established for these parameters were not applicable for all materials, as some authors found that such limits can be reached in early stages of animal manure composting

Table 4
Characterisation of the mature composts obtained from piles M1 and M2 (dry weight basis).

Maturity indices	M1	M2	Agronomic criteria	M1	M2
GI (%)	81.5 ± 4.7	85.0 ± 9.7	pH	6.77 ± 0.02	6.85 ± 0.03
NH ₄ ⁺ -N (mg kg ⁻¹)	679 ± 8	466 ± 6	EC (dS m ⁻¹)	10.70 ± 0.17	10.06 ± 0.25
NH ₄ ⁺ -N/NO ₃ ⁻ -N	0.08 ± 0.01	0.08 ± 0.01	OM (%)	57.3 ± 0.6	57.2 ± 1.4
C _w (g kg ⁻¹)	7.0 ± 0.1	7.3 ± 0.8	TOC (g kg ⁻¹)	303 ± 10	292 ± 8
C _w /TN	0.19 ± 0.01	0.22 ± 0.03	TN (g kg ⁻¹)	35.4 ± 0.5	31.1 ± 1.1
C _w /N _w	1.05 ± 0.03	1.28 ± 0.13	NH ₄ ⁺ -N (mg kg ⁻¹)	679 ± 8.1	466 ± 6.2
C _{ex}	33.8 ± 5.1	24.8 ± 0.8	NO ₃ ⁻ -N (mg kg ⁻¹)	8932 ± 528	5740 ± 294
C _{ex} /C _w	4.90 ± 0.24	3.20 ± 0.25	C/N ratio	8.56 ± 0.63	9.40 ± 0.14
C _{FA} (g kg ⁻¹)	16.3 ± 0.4	15.1 ± 0.3	Ca (g kg ⁻¹)	51 ± 1.0	46 ± 5.3
C _{HA} (g kg ⁻¹)	17.5 ± 2.5	9.7 ± 1.2	K (g kg ⁻¹)	22 ± 0.2	22 ± 2.3
CEC (meq 100 g ⁻¹ OM)	90 ± 1	100 ± 5	Mg (g kg ⁻¹)	9.32 ± 0.72	7.47 ± 0.99
CEC/TOC ratio	1.89 ± 0.04	1.95 ± 0.02	Na (g kg ⁻¹)	2.19 ± 0.02	1.88 ± 0.24
HI (%)	5.77 ± 0.38	3.32 ± 0.36	P (g kg ⁻¹)	15 ± 0.1	11 ± 0.4
HR (%)	11.1 ± 0.1	8.49 ± 0.42	Cd (mg kg ⁻¹)	0.35 ± 0.01	0.18 ± 0.01
PHA (%)	51.8 ± 2.9	39.0 ± 2.3	Cr (mg kg ⁻¹)	14 ± 0.1	35 ± 1.8
PI	1.08 ± 0.13	0.64 ± 0.06	Cu (mg kg ⁻¹)	326 ± 1	262 ± 28
			Fe (mg kg ⁻¹)	2304 ± 10	2506 ± 466
			Mn (mg kg ⁻¹)	358 ± 1	292 ± 35
			Pb (mg kg ⁻¹)	10 ± 1.0	8.5 ± 0.01
			Zn (mg kg ⁻¹)	947 ± 5	720 ± 67

M1: Solid fraction of pig slurry + cotton gin waste (4:3, v:v); M2: Solid fraction of pig slurry + cotton gin waste (3:4, v:v).

GI: germination index; N_w: water-soluble N; C_{ex}: extractable organic C; C_{HA}: humic acid-like C; C_{FA}: fulvic acid-like C; CEC: cation exchange capacity; HI: humification index; HR: humification ratio; PHA: percentage of humic acids; PI: polymerisation index.

(Bernal et al., 1996, 1998; Paredes et al., 2001). For instance, the C_{HA}:C_{FA} ratio or polymerisation index (PI > 1) was established for city refuse and sewage sludge composting, while the humification index (HI > 30) was established for agro-industrial and farm wastes. The values obtained in M1 were similar to those found by Bustamante et al. (2008) when composting distillery wastes with animal manures, except for the higher HR in M1. In general, the results indicate a good degree of humification of the OM in both composts.

The agronomic quality of the composts was evaluated using the values of chemical parameters relevant to agricultural use. The composts obtained presented pH values close to neutrality, relatively-high concentrations of TOC and TN and high concentrations of macro and micronutrients, providing the essential elements for plant nutrition. Similar results were found for other composts produced from manures (Zhu et al., 2004; Huang et al., 2004) and sewage sludge (Paredes et al., 2001; Fels et al., 2014). The C/N ratio values met the requirements established by the Spanish legislation for composts (<20) (BOE, 2013) and the value considered as a limit for mature composts by several authors (<12; Bernal et al., 2009). However, slightly-high values of moisture content were found, which can be easily reduced during maturation and storage. High values were found for the EC and Cu and Zn concentrations (higher in M1 than in M2 due to the greater proportion of SFP in the former), limiting the quality of the compost (Table 4). The concentrations of Zn and Cu found in mature compost produced from the solid phase of anaerobically-digested pig slurry by Bustamante et al. (2013) were higher than in the present experiment, due to the occurrence of these elements in the pig slurry. No limit has been established for the EC (soluble salts) in compost to be used as fertiliser, because salts can be easily washed away by rainfall after compost application to soil. The Cu and Zn concentrations were above the values suggested for the End-of-waste criteria in Europe (100 and 400 mg kg⁻¹, respectively; JRC and IPTS, 2012), and both composts can be classified as class C (Cu and Zn < 400 and 1000 mg kg⁻¹, respectively) according to the Spanish legislation for fertiliser products (BOE, 2013), which limits their agricultural use to 5 Mg ha⁻¹ per year. However, the germination index (GI) was high > 80% – indicating the absence of phytotoxicity. Considering an application limited to 5 t/ha, the

annual input of Cu and Zn to the soil would be, respectively, 1.63 and 4.75 kg ha⁻¹ with compost M1 and 1.31 and 3.6 kg ha⁻¹ with M2. These amounts are well below the limit values for the quantities of metals introduced annually into the soil (12 and 30 kg ha⁻¹ of Cu and Zn, respectively) established by the European Union for the protection of the environment, and in particular of the soil, when sewage sludge is used in agriculture (Directive 86/278/CEE; CEE, 1986). These elements are supplied to pigs to prevent several diseases and to improve growth (Zhang et al., 2012), and are later recovered in the pig slurry and concentrated in the solid phase after solid–liquid separation. Therefore, to ensure a compost of acceptable agricultural quality derived from pig slurry, the first action required is to reduce the amount of Cu and Zn at the origin – the amount supplied to the animals. Then, a management strategy at the farm/production system level – based on selection of the SFP with low metal contents for composting – should be implemented, using other recycling treatments for the metal-rich pig slurry.

4. Conclusions

The temperature evolution during the SFP composting process has been shown to rely on the thermal properties of the bulking agent, while the gaseous emissions were very well related to the OM degradation process. The use of cotton gin waste as bulking agent balanced the excessive moisture of the SFP, favouring good development of the composting process, as shown by the thermal profiles. An increase in the proportion of cotton gin waste led to higher temperatures and ventilation demand, prolonging the thermophilic phase, due to the thermal properties of the cotton gin waste. With the higher proportion of bulking agent, the energy generated by the microbial activity was quickly and efficiently converted into heat in the composting mass, requiring high aeration for thermal diffusion and therefore a high energy cost for management. The higher proportion of bulking agent reduced gaseous emissions during composting, especially of CO₂, CH₄ and N₂O. However, total-N losses were greater in the pile with the higher proportion of bulking agent, since the intense aeration required to maintain the ceiling temperature favoured ammonia emissions. Based on the process performance, the 4:3 proportion of SFP:bulking agent (v/v) was the most adequate for composting the

SFP, giving a compost with high OM humification and nutrient concentrations. However, the production of quality compost at the industrial or farm level will be conditioned by the concentration of Cu and Zn in the pig slurry. Increasing the proportion of bulking agent has a dilution effect on such elements in the compost, but the process will have a greater energy cost for aeration of the composting mass. The use of SFP with low Cu and Zn concentrations will be the best way to ensure the production of high-quality compost appropriate for commercial use.

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