



The production of commercial organic amendments and fertilisers by composting of two-phase olive mill waste (“alperujo”)

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ARTICLE INFO

Article history:

Received 10 October 2011

Received in revised form

28 November 2011

Accepted 8 December 2011

Available online 16 December 2011

Keywords:

Commercial organic fertilisers

Composting

Olive mill wastes

“alperujo”

Poultry and sheep manures

ABSTRACT

Nowadays, the main concern for the Spanish olive oil industry is the disposal of the huge amounts (about 5 Mt in the last olive oil campaign, 2010–2011) of the two-phase olive mill waste or “alperujo” (AL) that it produces. Here, we have studied the technical feasibility of using composting to transform AL into commercial organic amendments and fertilisers. For this, six piles of 20 t each, prepared by mixing AL and either poultry (PM) or sheep manure (SM), were composted. In addition, Fe and P-enriched acidic mineral amendments were added at the beginning of the process in order to reduce alkalisation and nitrogen losses. As composting proceeded, substantial organic matter degradation, progressive humification, increases of pH and germination indices and decreases in fats and water-soluble organic fractions were detected. Also, the addition of the mineral amendments was effective with regard to reducing the pH, water-soluble organic carbon and polyphenols, hemicellulose fraction and nitrogen losses. According to the Spanish legislation for fertiliser production (PRE/630/2011, 2011), the composts obtained were suitable for preparing commercial organic amendments and fertilisers within categories such as *AL Compost*, *Humic Organic Amendment*, *Compost Organic Amendment* and *NPK Organic Fertiliser*. Also, by mixing with organic and/or mineral fertilisers, the composts were optimal substrates for the production of 19 types of *Organo-mineral Fertilisers*, either as solid or liquid formulations.

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1. Introduction

The agrofood industry produces a great variety of organic wastes that potentially can be used as soil fertilisers and amendments due to their high contents of organic matter (OM) and plant nutrients (Martínez-Blanco et al., 2011). A clear example of this issue is the olive oil industry, which has a relevant economic and social importance in Mediterranean countries and produces a huge amount of wastes, depending on the extraction system applied. As the two-phase centrifugation system is the main one used by the Spanish industry, the major concern for this sector is the high production of “alperujo” (AL) and its disposal (Alburquerque et al., 2004). AL is an acidic and very-wet solid olive mill waste, containing phenolic and lipidic fractions which are related to phytotoxic and antimicrobial effects when AL is used as a soil amendment. At present, AL is usually treated with a second centrifugation, or dried and then subjected to chemical extraction,

to extract the residual oil. Finally, the resulting wastes are used to obtain thermal or electrical energy for sale or for use in the olive mill, mainly through combustion of the dried wastes although they are also considered valuable materials for co-firing with coal in power utilities (Gogebakan and Selçuk, 2009).

Alternative methods such as composting, considered an appropriate low-cost technology for organic waste recycling and organic fertiliser production (Arvanitoyannis and Varzakas, 2008; Ruggieri et al., 2009), may well represent an acceptable solution for AL disposal, adding value to this waste and enhancing the sustainability of the olive oil production system (Vlyssides et al., 2004; Salomone and Ioppolo, in press). The AL composting process has been evaluated by studying several factors at both the pilot plant (Madejón et al., 1998; Alburquerque et al., 2006a; Cegarra et al., 2006; Alfano et al., 2008) and industrial scale (Cayuela et al., 2004; Sánchez-Arias et al., 2008). These studies focused mainly on the composting aeration system employed (mechanical turnings, forced ventilation or a combination of both) and the bulking agents added to improve the deficient physical properties of AL. Animal manures, such as those of poultry (PM) and sheep (SM), seem to be appropriate and effective bulking

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agents and also show an inoculant effect and add nutrients, especially available nitrogen compounds (Kelleher et al., 2002; Cayuela et al., 2004; Alfano et al., 2008; Canet et al., 2008). However, some disadvantages related to AL composting and the agricultural use of AL composts should be taken into account. Some examples are: nitrogen losses during the process, alkaline compost pH and an unbalanced nutrient content. In several composting experiments, inorganic amendments rich in different elements were employed, which reduced the pH and ammonia volatilisation significantly (Kithome et al., 1999; Koenig et al., 2005). Also, S^o and Fe-enriched acidic mineral amendments were employed satisfactorily in AL composting (Roig et al., 2004; Canet et al., 2008; Sánchez-Arias et al., 2008). However, the addition of such materials can increase substrate salinity and reduce compost quality.

The recycling of organic wastes for agricultural purposes is crucial in order to sustain soil productivity in Mediterranean areas, where the soil OM content is very low (Lasaridi et al., 2006; Ruggieri et al., 2009; Martínez-Blanco et al., 2011). Therefore, composting may act as an appropriate disposal option for biodegradable wastes. However, compost of satisfactory quality is required to establish a fertiliser market, which could promote an efficient recycling strategy for AL. Several countries have developed their own quality criteria for composted materials (ECN-QAS in Europe, STA and CCQC in the USA, RAL in Germany, BSI PAS 100 in the UK, CIC in Italy, etc.). Thus, AL composts with an acceptable nutrient content could be obtained and both the Spanish legislation for fertilisers (PRE/630/2011, 2011) and the 2nd draft of the Biological Treatment of Biowaste (European Commission, 2001) should be considered as the best references for assessment of their quality and potential as commercial products. Their high OM content and the possibility of obtaining the European eco-label (European Commission, 2010) as *Soil Improvers*, due to their low content of heavy metals, make their commercialisation attractive, as mentioned by Canet et al. (2008).

In this paper, we present the results of a technical feasibility study evaluating the production of commercial organic amendments and fertilisers rich in OM and nutrients, through composting of the main by-product of the Spanish olive oil industry.

2. Materials and methods

2.1. Composting methodology

Six trapezoidal piles of about 20 t each (1.5 m wide and 0.6 m high, approximately) were prepared using a backhoe loader, mixing AL with the bulking agents (PM: poultry manure, SM: sheep manure) and the acidic mineral additives in the following proportions, on a fresh weight basis (dry weight basis in brackets):

AL + PM pile: 51% AL + 49% PM (37/63)

AL + PM + Fe pile: 51% AL + 48% PM + 1% Fe (36/62/2)

AL + PM + P pile: 51% AL + 48% PM + 1% P (36/62/2)

AL + SM pile: 65% AL + 35% SM (57/43)

AL + SM + Fe pile: 65% AL + 34% SM + 1% Fe (56/42/2)

AL + SM + P pile: 65% AL + 34% SM + 1% P (56/42/2)

The experiment was conducted using the mechanical turning method recommended by Cayuela et al. (2006) and Cegarra et al. (2006) for AL composting. Seven turnings were applied during the process, according to the temperature evolution. The turnings were more frequent at the beginning, since the organic matter biodegradation was more active, and less frequent when the thermophilic temperature started to decrease. An aspersion system was used to keep the substrate moisture level above 40%.

A minimum of 30 sub-samples per pile were taken, mixed and homogenised. This sample was considered representative of the composting substrates and later it was divided into three fractions

in the laboratory: the first was frozen (−20 °C) and kept for the determination of NH₄⁺ and NO₃⁻, the second was dried in an oven at 105 °C for 24 h, to determine its moisture content, and the third was freeze-dried and ground to less than 0.5 mm prior to analysis. The water-soluble organic carbon (WSC) was determined, using an automatic analyser for liquid samples (Shimadzu TOC Analyser), for 1:20 (w/v) water extracts produced by shaking for 2 h. The NH₄⁺ was determined using a colourimetric method based on Berthelot's reaction (Kempers and Zweers, 1986; Sommers et al., 1992), NO₃⁻ was measured by HPLC after 1:20 (w/v) water extraction and the phytotoxicity was determined using the germination index (GI) test with cress (*Lepidium sativum*) and radish (*Raphanus sativus*) seeds (Zucconi et al., 1981; Satisha and Devarajan, 2007). The macro and micronutrients (P, K, Ca, Mg, Na, S, Fe, Cu, Mn, Zn) and heavy metals were determined by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) after microwave digestion. The humification indices - humification ratio (HR), humification degree (HD) and percentage of humic acids (P_{HA}) - were calculated according to Alburquerque et al. (2009). Other parameters, including electrical conductivity (EC) and pH, OM, total nitrogen (T_N), total organic carbon (T_{OC}), total fat content, water-soluble phenolic substances (WSPH), water-soluble carbohydrates (WSCH), lignin, cellulose and hemicelluloses, were determined according to methods described previously (Alburquerque et al., 2004; Cegarra et al., 2006). Losses of total OM, lignin, cellulose, hemicellulose and T_N were calculated by taking into account the apparent increase in the ash content resulting from the loss of dry weight, in order to reflect better the overall changes (Viel et al., 1987; Paredes et al., 1996).

2.2. Raw material characterisation

For the composting experiments, AL was collected from "Aceites Guadalentín S.L.", an olive mill in Jaén (Spain), and the bulking agents (poultry: PM, and sheep manure: SM) from farms near the composting plant. The AL showed high moisture (55.8%), fat and OM contents, the latter composed mainly of lignin (344.7 g kg⁻¹), acidic pH and moderate nitrogen (poor in inorganic forms), P and micronutrient contents (Table 1). With respect to the bulking agents, the PM had a higher OM content than the SM, as well as higher nitrogen content (about 20% as ammonium), while the SM had higher electrical conductivity (EC) and Ca and Fe contents (Table 1). Rice husk, used as animal bedding, constituted an important proportion of the PM. The addition of two acidic mineral additives to the composting mixtures was evaluated also, with regard to their ability to enrich the end-products of composting in essential plant nutrients (Fe and P), reduce the alkalisation resulting from the composting process and improve the agronomic potential of the composts by reducing the nitrogen losses during the process. The Fe-enriched mineral amendment used was a by-product of titanium oxide synthesis (Sánchez-Arias et al., 2008), whereas the P-enriched mineral was a commercial fertiliser (GSSP, Granular Simple Super Phosphate, ICL Fertilisers company).

2.3. Statistical analyses

A descriptive statistical analysis (the mean and the absolute and relative errors) was calculated for each pile. Also, we performed some inferential statistical analyses such as the analysis of variance (ANOVA) within treatments (piles and time), assuming a normal distribution of the data and homoscedasticity. For *post-hoc* analysis, we used the Tukey test ($p < 0.05$) and the Least Significant Difference (LSD) ($p < 0.05$), to determine changes in the analysed parameters for each pile with time. All these analyses were calculated using the SPSS 17.0 program for Windows XP. Another inferential statistical analysis was a regression study of some of the

Table 1
Main characteristics of the raw materials (“alperujo”: AL, poultry manure: PM, and sheep manure: SM) and the mature composts obtained (Fe: iron-enriched mineral amendment and P: phosphorous-enriched mineral amendment).

Parameters ^a	AL	PM	SM	Mature composts					
				AL + PM	AL + PM + Fe	AL + PM + P	AL + SM	AL + SM + Fe	AL + SM + P
pH ^b	5.38	7.50	8.51	8.30	7.97	8.02	8.88	8.52	8.35
EC ^b (dS m ⁻¹)	4.64	8.47	11.33	1.70	1.84	2.44	1.69	1.70	2.03
OM (g kg ⁻¹)	907.9	805.5	456.5	745.6	760.3	746.0	561.2	572.1	588.2
Lignin (g kg ⁻¹)	344.7	129.7	211.4	428.2	467.4	457.2	350.6	375.0	374.1
Cellulose (g kg ⁻¹)	178.7	149.9	113.6	176.6	182.8	178.6	146.1	179.2	188.1
Hemicellulose (g kg ⁻¹)	350.3	307.0	109.7	208.7	152.2	129.1	131.0	92.0	104.0
T _{OC} (g kg ⁻¹)	486.0	398.4	251.6	396.6	403.1	393.2	251.9	272.3	288.4
T _N (g kg ⁻¹)	13.4	32.3	17.7	20.2	20.6	21.1	18.6	19.2	19.1
NH ₄ ⁺ (mg kg ⁻¹)	63	5915	889	141	163	203	46	60	71
NO ₂ ⁻ (mg kg ⁻¹)	16	19	520	23	37	44	6	23	19
T _{OC} /T _N	36.3	12.3	14.3	19.5	19.5	18.7	13.5	14.2	15.0
Fat content (g kg ⁻¹)	99.2	14.9	5.1	2.1	2.1	4.1	2.1	2.1	2.1
WSCH (g kg ⁻¹)	19.7	20.9	4.5	3.2	2.0	4.2	1.5	2.2	1.2
WSPH (g kg ⁻¹)	9.0	9.3	3.3	2.0	2.1	3.4	2.1	1.1	1.3
WSC (g kg ⁻¹)	86.5	67.8	35.4	19.4	13.5	19.3	16.4	10.6	11.7
P (g kg ⁻¹)	0.8	2.2	2.2	2.0	1.9	2.2	1.8	1.8	2.0
K (g kg ⁻¹)	10.4	13.5	16.5	11.0	7.9	6.5	12.8	12.7	10.2
Ca (g kg ⁻¹)	8.0	47.5	100.9	29.4	20.1	18.7	65.0	72.4	65.2
Mg (g kg ⁻¹)	3.1	5.5	18.7	5.7	4.9	4.9	12.7	10.6	10.3
Na (g kg ⁻¹)	0.3	4.1	3.9	8.3	6.0	8.2	7.6	6.2	6.2
S (g kg ⁻¹)	1.1	4.0	13.2	2.5	3.6	4.6	5.2	5.4	5.4
Fe (g kg ⁻¹)	2.4	1.9	4.1	3.9	9.0	4.8	6.5	11.2	6.0
Cu (mg kg ⁻¹)	22	29	51	32	22	27	16	14	19
Mn (mg kg ⁻¹)	56	322	226	183	193	145	167	199	131
Zn (mg kg ⁻¹)	17	79	185	92	54	39	41	55	49
Pb (mg kg ⁻¹)	4	4	12	8	30	37	11	17	24
Cr (mg kg ⁻¹)	19	23	19	14	12	13	24	22	28
Ni (mg kg ⁻¹)	55	49	25	21	29	35	50	42	80
Cd (mg kg ⁻¹)	nd	nd	nd	nd	2	7	nd	1	2

Note: Data were shown as the mean value of four replicates with less than 5% of relative error.

^a Data based on dry weight.

^b Water extract 1:10. EC: electrical conductivity, OM: total organic matter, T_{OC}: total organic carbon, T_N: total nitrogen, WSCH: water-soluble carbohydrates, WSPH: water-soluble phenols, WSC: water-soluble organic carbon, and nd: not detected.

experimental data, especially OM losses. We fitted them to two models: zero-order and first-order kinetic models, with important correlation coefficients. This analysis was performed with Sigma-Plot 2000 software for Windows, Version 10.0.

3. Results and discussion

3.1. Composting process evolution

The addition of the bulking agent and the mechanical turning favoured a rapid increase of temperature in all piles to values typical of the thermophilic phase. In the AL + PM piles, the thermophilic phase lasted about 26 weeks, six more than for the AL + SM piles (Fig. 1). This behaviour could be explained by the differing proportion of the bulking agent used as well as the high OM content of the PM (805.5 g kg⁻¹ compared to 456.5 g kg⁻¹ for SM). Also, the PM had a high content of rice husk, a material rich in cellulose and silica compounds (highly resistant to biodegradation), which could have delayed the degradation process and extended the thermophilic phase (Low and Lee, 1997; Champagne, 2004; Chaudhary and Jollands, 2004). In general, a long thermophilic period characterises AL composting, depending on the process configuration and aeration system employed: from 13 weeks, with an industrial turning machine (Alfano et al., 2008), to 40 weeks with mechanical turning (Cayuela et al., 2004, 2006).

Fig. 1 shows acidic pH values at the beginning of the process, which increased during the thermophilic phase and reached their highest values at the end of the 20th week (8.9 and 9.5 for the AL + PM and AL + SM piles, respectively). Later, all piles showed a slight decrease until the end of composting, that led to final average values of 8.1 and 8.6 for the AL + PM and AL + SM piles,

respectively. This behaviour is observed commonly in AL composting (Cayuela et al., 2006; Cegarra et al., 2006), with end-material pH values higher than 8.5.

The addition of the acidic amendments led to decreases in pH along the composting process (around 0.5 pH units, Fig. 1). Some amendments have been employed successfully to control the evolution of pH in AL composting. Roig et al. (2004) found that the pH decreased by 1.1 units when S^o was added during the curing period of the composting process, while Mari et al. (2005) obtained similar results by adding the same amendment at the end of the thermophilic phase. Finally, Sánchez-Arias et al. (2008) strongly decreased the pH values of AL composts by adding 20% (dry weight basis) of the same Fe mineral amendment used in our experiment; this reduced the biological influence on the organic matter oxidation and increased the chemical oxidation.

The T_N content of all piles increased during composting (Table 2), from initial values of 18.4 and 15.5 g kg⁻¹ to final ones of 20.6 and 18.9 g kg⁻¹ for the AL + PM and AL + SM piles, respectively. In the former, the T_N content was higher, due to the higher nitrogen content of the PM (32.3 g kg⁻¹, compared to 17.7 g kg⁻¹ for SM). During the thermophilic phase, the AL + PM piles showed an initial decrease in T_N content. This could be explained by the initial NH₄⁺ content in the AL + PM piles: 1742 mg kg⁻¹ (much higher than the 317 mg kg⁻¹ of the AL + SM piles), which decreased sharply during the process (Fig. 2). In our experiment, the T_N losses should be attributable largely to NH₃ volatilisation (especially in the piles with PM). This phenomenon is observed commonly in PM-composting experiments (Mahimairaja et al., 1994; Koenig et al., 2005) and can be related to the pH evolution (fast substrate alkalisation during AL composting) that transforms NH₄⁺ into volatile NH₃, which can provoke important environmental problems during

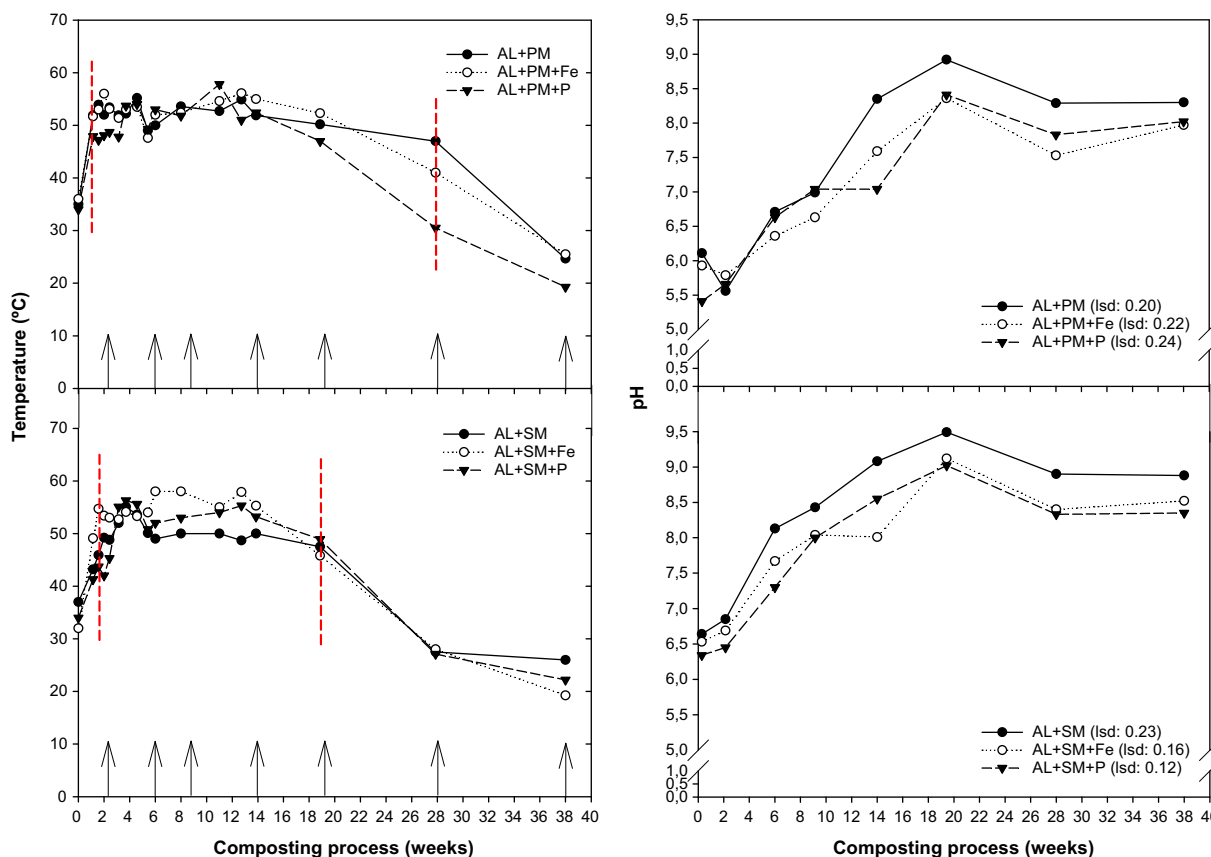


Fig. 1. Evolution of temperature and pH during composting (AL: “alperujo”, PM: poultry manure, SM: sheep manure, Fe: iron-enriched mineral amendment and P: phosphorous-enriched mineral amendment). Arrows showed the mechanical turnings applied. For each pile, the least significant difference test (Lsd) at $p < 0.05$ was calculated.

PM disposal (Mahimairaja et al., 1994; Mondini et al., 1996). Also, the NH_3 volatilisation effect was noted in the evolution of the $T_{\text{OC}}/T_{\text{N}}$ ratio, which showed an initial increase in the AL + PM piles followed by a decrease after the 20th week, until the thermophilic phase (Table 2).

The T_{N} content of AL composts is within the range 16.6–26.2 g kg^{-1} , as shown previously (Cayuela et al., 2004; Albuquerque et al., 2006a and Cegarra et al., 2006). The addition of the acidic mineral amendments rich in Fe and P reduced the T_{N} losses. At the end of the process, AL + PM + Fe and AL + PM + P exhibited T_{N} losses of 31.9 and 31.6%, respectively, lower than for AL + PM (43.1%); also, AL + SM + Fe and AL + SM + P had T_{N} losses of 29.4 and 20.9%, respectively, compared to 32.4% for AL + SM. Acidic mineral amendments have been used widely to control the pH during composting and to reduce nitrogen losses. Hence, Koenig et al. (2005) tested different amendments intended to control ammonia losses during composting, obtaining good results with respect to the progression of pH. Also, Ekinci et al. (2000), composting short paper fibre with broiler litter, demonstrated that ammonia volatilisation was lower when the pH of the composting substrate was around 7; it increased considerably with alkalisation pH (≥ 8).

The initial WSC values depended on the bulking agent and the proportion added to the AL. Hence, piles made with PM showed values close to 55 g kg^{-1} while SM piles had values around 41 g kg^{-1} (Table 2). The WSC decreased during the process in all piles, when the thermophilic phase was more intense. At the end of the process, the AL + PM and AL + SM piles exhibited values, in the order of 17 and 12 g kg^{-1} , respectively, similar to that recommended by Bernal et al. (1998) as a maturity index for composts of different sources.

Canet et al. (2008) obtained final WSC values of 11 and 10 g kg^{-1} after composting AL with SM or rabbit manure, respectively. Albuquerque et al. (2006a) reported final values around 30 g kg^{-1} after composting AL with several bulking agents. Their higher values may be related to the lesser amount of bulking agent employed: AL comprised 86% (dry weight basis) compared to about 50% (Canet et al., 2008) or 37 and 57% in our experiments.

The mineral amendments affected the final WSC values in the AL + PM + Fe and AL + SM + Fe composting piles, which were decreased by nearly 34% compared to their respective controls: piles AL + PM and AL + SM. In the P-enriched piles, only for AL + SM + P was the decrease statistically significant, the value being almost 27% lower than in the AL + SM pile. The interactions of Fe and P with the water-soluble organic fraction in soil (and in composting substrates) are influenced by factors such as their chemical structure and conformation, element/carbon ratio and pH (Stevenson, 1994). Gu et al. (1994) showed that the interactions with soil iron oxides could happen due to their chemical binding with carboxylic and hydroxyl functional groups from the water-soluble fraction of the organic matter. Also, Nierop et al. (2002) showed that Fe^{2+} and Fe^{3+} interactions with dissolved organic matter, and their flocculation capacity, were greater in an acidic soil than in other types of soil. Guggenberger and Kaiser (2003) employed iron oxides incrustated in biofilms in order to reduce dissolved organic matter in a wastewater technology treatment.

The WSCH developed in a rather similar way to WSC (Table 2), reaching end-values close to 3 and 1 g kg^{-1} for the AL + PM and AL + SM piles, respectively. Previous authors have suggested WSCH as the main carbon source for the microflora responsible for the initial degradation phase (thermophilic biodegradation).

Table 2
Evolution of some parameters during composting of AL + PM and AL + SM piles (AL: "alperujo", PM: poultry manure, SM: sheep manure, Fe: iron-enriched mineral amendment and P: phosphorous-enriched mineral amendment).

Pile	Composting time (weeks)	T _N (g kg ⁻¹)	T _{OC} /T _N	WSC (g kg ⁻¹)	WSCH (g kg ⁻¹)	WSPH (g kg ⁻¹)	Lignin (g kg ⁻¹)	Cellulose (g kg ⁻¹)	Hemicellulose (g kg ⁻¹)	GI of cress (%)	GI of radish (%)
AL + PM	0	19.2	23.3	57.3	15.7	9.0	296.0	161.8	376.2	0	0
	6	13.7	31.4	19.5	9.6	7.1	447.9	137.3	327.4	14	48
	19	14.4	29.7	12.7	4.6	4.9	469.1	140.6	295.0	69	62
	38	20.2	19.5	19.4	3.2	2.0	428.2	176.6	208.7	71	82
AL + PM + Fe	0	17.0	27.3	50.0	14.0	7.0	308.0	168.1	376.8	0	0
	6	14.0	28.9	23.2	10.4	5.2	441.1	150.0	295.7	9	58
	19	17.1	24.9	12.9	3.6	4.4	431.4	131.8	281.1	82	78
	38	20.6	19.5	13.5	2.0	2.1	467.4	182.8	152.2	94	76
AL + PM + P	0	19.0	22.2	57.0	16.0	8.0	290.3	175.4	360.4	0	0
	6	16.1	27.2	24.3	10.6	5.7	444.0	171.5	208.6	18	52
	19	14.7	29.8	16.2	9.3	4.6	452.6	104.4	322.1	60	61
	38	21.0	18.7	19.3	4.2	3.4	457.2	178.6	129.1	77	77
AL + SM	0	15.7	24.7	45.2	12.1	6.0	375.5	161.7	294.4	0	0
	6	16.5	24.1	24.0	8.1	7.8	423.5	179.8	176.8	24	41
	19	18.6	16.6	19.8	4.3	6.9	348.7	192.0	135.9	74	76
	38	18.6	13.5	16.4	1.5	2.1	350.6	146.1	131.0	81	85
AL + SM + Fe	0	16.2	24.3	38.0	10.0	5.5	319.4	169.4	281.5	0	0
	6	14.7	26.7	22.5	8.5	6.3	396.8	171.7	179.9	60	37
	19	18.4	18.2	17.7	5.0	4.0	359.1	252.9	50.3	94	94
	38	19.2	14.2	10.6	2.2	1.1	375.0	179.2	92.0	70	102
AL + SM + P	0	14.6	27.0	41.0	9.0	6.0	324.3	176.0	271.6	0	0
	6	14.6	27.3	24.1	9.0	5.7	420.5	199.8	147.3	21	59
	19	18.0	18.9	19.1	4.6	3.3	377.7	160.3	83.8	57	96
	38	19.1	15.0	11.7	1.2	1.3	374.1	188.1	104.0	78	101
ANOVA	Time	**	**	**	**	**	***	**	*	***	***
	Pile	*	**	**	**	**	***	**	**	NS	NS
	Time × Pile	*	**	**	**	**	***	**	*	NS	NS

T_N: total nitrogen, T_{OC}: total organic carbon, WSC: water-soluble organic carbon, WSCH: water-soluble carbohydrates, WSPH: water-soluble phenols and GI: germination index.

NS: not significant and ***, ** and *: significant at $p < 0.001$, 0.01 and 0.05, respectively.

Sánchez-Monedero et al. (1999) evaluated different composting mixtures prepared with municipal solid waste and noted that the WSCH fell sharply during the first three weeks of composting (by > 60% of the initial values). Also, Albuquerque et al. (2006a), who composted AL with grape stalks and olive leaves, showed that the WSCH decreased during the process, to reach final values of 10 g kg⁻¹.

Other important water-soluble organic compounds used to evaluate the maturity of AL compost are the WSPH, which are related to the phytotoxic properties of the olive mill wastes (Ait Baddi et al., 2003; Albuquerque et al., 2006b; Sampredo et al., 2008). In our case, the WSPH decreased in all piles during composting, reaching similar end-values close to 2 g kg⁻¹ (Table 2). It should be added that WSCH and WSPH are liberated also by the degradation of lignocellulosic materials, leading to their increased concentrations in the water phase which then decrease as a consequence of microbial activity. As mentioned before, the degradation pathway prevailed in our case, since a continuous decrease occurred during the composting process. Also, Sánchez-Monedero et al. (1999) reported that decreases in the WSPH were inversely and strongly correlated with the main humification indices, suggesting that these compounds could act as precursors for the humification process during composting.

Regarding the OM degradation during composting, evaluated by the OM losses (Fig. 3), the data from the AL + PM piles fitted a zero-order kinetic model "OM loss = ht " (where " h " is the slope, % OM week⁻¹, and " t " the composting time in weeks), while the AL + SM piles followed a first-order kinetic model "OM loss = $A(1 - e^{-kt})$ " where " A " is the maximum degradation of OM (% OM), " k " the rate constant (week⁻¹) and " t " the composting time in weeks. This difference in behaviour is probably related to the more-recalcitrant nature of PM, as mentioned already, and the differing proportions of bulking agent added to the AL. The AL + SM piles showed more-intensive degradation rates than the AL + PM piles during the

thermophilic period; these decreased slightly at the end of the process, giving final OM losses of 53.4 and 57.3% in the AL + PM and AL + SM piles, respectively.

The main components of the AL organic substrate (lignin, cellulose, hemicelluloses and fats) were also studied during composting (Table 2). The relative contents of lignin and cellulose increased during the process (especially in the thermophilic phase) while hemicellulose showed the opposite behaviour, a decrease. The two acidic mineral amendments decreased the lignin losses by 8 and 16% compared to the controls: the non-amended mixtures AL + PM and AL + SM, respectively. The cellulose losses showed a similar behaviour. Only the hemicellulose losses increased when the acidic amendments were used, by about 7 and 4% relative to the non-amended mixtures. The decreases in the biodegradation rates of the lignin and cellulose fractions could have been due to the chemical affinity of the Fe and P amendments for certain functional groups in the lignocellulosic matrix of the organic matter, as remarked upon by other authors. Merdy et al. (2002a) found that the Fe³⁺-lignin complex showed certain stability, lignin being a good adsorbent of this nutrient in calcareous soils where it is not available to plants. Merdy et al. (2002b) studied the interaction of both Fe and Mn with the lignocellulosic fractions from barley straw, showing redox process involving Fe and quinone-like structures on the lignin surface. Also, Karthikeyan et al. (2004) used solid wood residues to reduce the orthophosphate content in wastewater, due to ionic change process and other Lewis interactions between orthophosphate and the wood lignocellulosic fraction.

Another important organic fraction of AL is its high residual fat content (Albuquerque et al., 2004), which should be monitored during composting. Here, the initial high fat contents of the two groups of mixtures (75 and 55 g kg⁻¹ in the AL + PM and AL + SM piles, respectively) decreased sharply during the first three months of composting to reach end-values close to 2 g kg⁻¹ (Fig. 2),

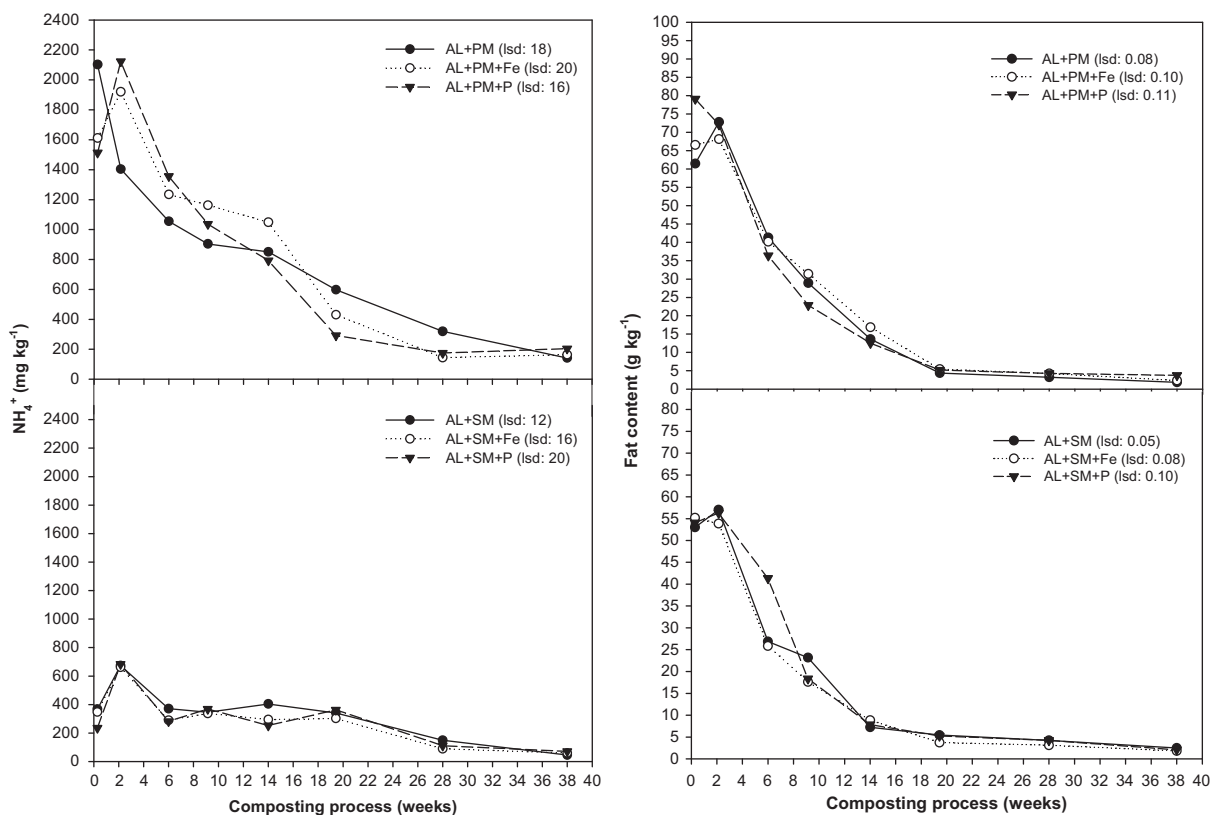


Fig. 2. Evolution of ammonium and fat content during composting (AL: “alperujo”, PM: poultry manure, SM: sheep manure, Fe: iron-enriched mineral amendment and P: phosphorous-enriched mineral amendment). For each pile, the least significant difference test (Isd) at $p < 0.05$ was calculated.

a phenomenon common in AL composting studies (Madejón et al., 1998; Alburquerque et al., 2006b).

Humic parameters, such as the humification indices, have been employed as quality criteria for AL composting (Tomati et al., 2001; Alburquerque et al., 2009). Table 3 shows that HD and P_{AH} increased during AL composting in all piles due to the incorporation of OM into the humic fraction (HA and FA-like). During the early composting phase, the HR decreased considerably since non-humic materials were initially co-extracted in the alkali-soluble fraction. At the end of the process, the AL + SM piles generally showed higher HR, HD and P_{HA} values than the AL + PM piles (29.8, 75.7 and 68.1% versus 20.0, 70.8 and 63.2%, respectively), related to the more-easily-degradable organic matrix of the AL + SM piles which could have enhanced the humification of the substrate, in accordance with the OM losses discussed previously.

Phytotoxicity is one of the most-important criteria for evaluating the suitability of organic materials for agricultural purposes (Zucconi et al., 1981; Bernal et al., 1998). All piles showed a clear increase in the GI during the first three months of composting (Table 2) and reached end-values typical of non-phytotoxic materials (81 and 78% for the AL + PM composts and 76 and 96% for the AL + SM composts, for the cress and radish GI, respectively). These increases in the GI could have been related to the decreases observed for substances with phytotoxic properties: ammonium, WSPH and fats (Alburquerque et al., 2006b).

3.2. Agrochemical characteristics of the AL composts relevant to the production of commercial organic amendments and fertilisers

The mature composts showed alkaline pH and low EC values (Table 1), in agreement with the results reported by Roig et al. (2004), Sánchez-Arias et al. (2008) and Canet et al. (2008), while

the total and mineral nitrogen contents were slightly higher in the AL + PM composts than in the AL + SM composts. The total OM content was high in both types of compost (750.6 and 573.8 g kg^{-1} , respectively), as was the lignin content; around 50% of the total organic content. The water-soluble organic fraction was low, as indicated by the values of WSC, WSCHE and WSPH, but slightly higher in AL + PM composts than in AL + SM composts. All the composts had humification indices which characterised them as humified materials. The heavy metal content was, in general, low and did not produce any phytotoxic effects, according to the GI values of cress and radish (>70%).

According to the new Spanish legislation for fertilisers (PRE/630/2011, 2011), these composts could be used in several commercial categories, such as *AL Compost* (OM > 45%, moisture > 40%, $T_{OC}/T_N < 20$ and total polyphenols < 0.8%), *Humic Organic Amendment* (OM > 25%, 5% in the total humic extract and 3% in humic acids), *Compost Organic Amendment* (OM > 35% and $T_{OC}/T_N < 20$) and *NPK Organic Fertiliser* ($T_{OC}/T_N < 15$ and $N + P_2O_5 + K_2O > 4\%$, more than 1% of each nutrient). According to the minimum requirements, the six composts were defined as *AL Compost*. The AL + PM and AL + SM composts could be used to obtain the categories *Humic Organic Amendment* and *Compost Organic Amendment*, while only *NPK Organic Fertiliser* could be made with the AL + SM compost, due to the high T_{OC}/T_N value of the AL + PM compost, mainly because of the different evolution of the OM during composting of this pile and the rice husk content of PM, not for the content of nutrients. Another category described in the Spanish legislation is *Organo-mineral Fertilisers*, made by mixing organic fertilisers with other organic and/or inorganic fertilisers. Using AL + SM composts as an organic fertiliser (*NPK Organic Fertiliser*), it could be possible to prepare at least 19 types of commercial organic fertiliser, in solid and liquid forms.

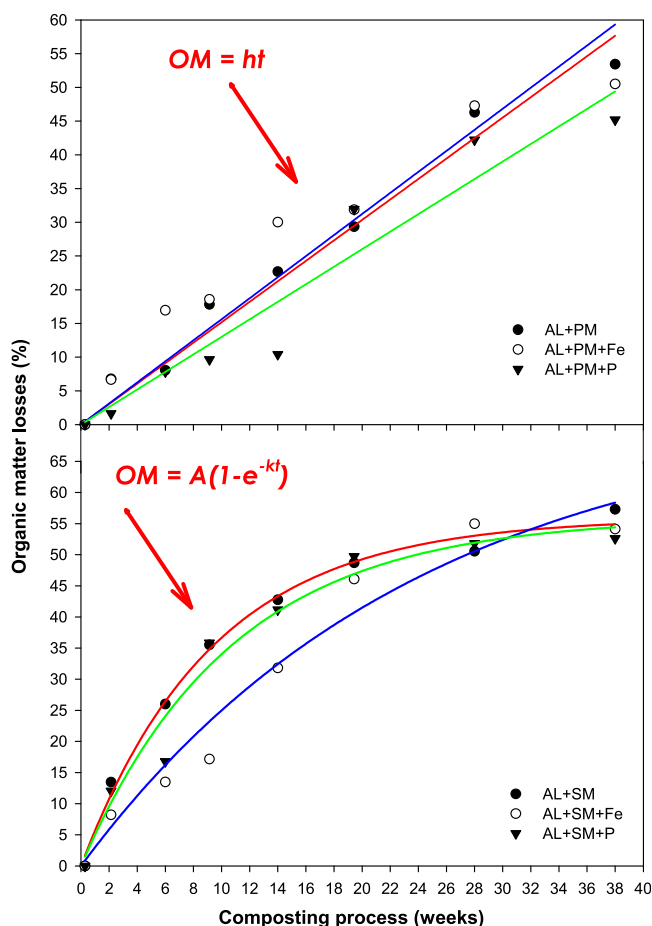


Fig. 3. Evolution of the organic matter during composting process (AL: “alperujo”, PM: poultry manure, SM: sheep manure, Fe: iron-enriched mineral amendment and P: phosphorous-enriched mineral amendment). AL + PM piles fitted to a zero-order kinetic model “OM loss = ht ” (where “ h ” is the slope, % OM weeks⁻¹, and “ t ” the composting time in weeks) and AL + SM piles followed a first-order kinetic model “OM loss = $A \cdot (1 - e^{-kt})$ ” where “ A ” is the maximum degradation of OM (% OM), “ k ” the rate constant (weeks⁻¹) and “ t ” the composting time in weeks.

3.3. Cost of the composting process

For the industrial production of marketable products, it is necessary to check the economical viability of the process. In our experimental conditions, the total cost to produce 60 t of compost was around 2150€ (36€ per t of compost obtained or 31€ per t of AL treated). We have identified the following operational costs:

1. Raw material supplied. According to the composting performance we prepared six composting piles of 20 t each (120 t in total), using 69.6 t of AL, 29.0 t of PM, 20.6 t of SM, 0.4 t of Fe and 0.4 t of P-enriched materials. The AL was supplied by the olive mill without cost. The manures and the mineral acidic amendments cost around 1050€ in total (including transport).
2. Composting equipment. The composting experiments were carried out in an outdoor facility close to the olive mill and dedicated to AL storage (without any additional cost). A backhoe loader was used for preparing the composting mixtures and turning the composting piles, spending 25 working-hours in total (240€).
3. Labour cost. Two men were necessary to carry out the composting experiment. This required close to 35 working-hours, with an approximate cost of 630€.

Table 3

Evolution of the humification ratio (RH), humification degree (HD) and the percentage of humic acids (P_{HA}) during composting (AL: “alperujo”, PM: poultry manure, SM: sheep manure, Fe: iron-enriched mineral amendment and P: phosphorous-enriched mineral amendment).

Pile	Composting time (weeks)	HR (%)	HD (%)	P_{HA} (%)
AL + PM	I (0)	24.1	67.9	37.8
	LB (19)	17.6	73.1	65.5
	M (38)	18.6	71.3	63.2
AL + PM + Fe	I (0)	19.9	56.6	16.1
	LB (19)	17.2	71.2	65.5
	M (38)	18.5	66.0	63.5
AL + PM + P	I (0)	29.1	66.4	39.3
	LB (19)	14.4	64.3	52.2
	M (38)	22.9	75.2	72.0
AL + SM	I (0)	28.6	69.9	42.0
	LB (19)	31.0	80.6	78.9
	M (38)	34.3	79.8	78.7
AL + SM + Fe	I (0)	27.3	66.7	46.2
	LB (19)	21.7	73.3	71.1
	M (38)	30.5	75.4	63.8
AL + SM + P	I (0)	26.3	66.6	36.1
	LB (19)	20.2	75.3	72.8
	M (38)	24.6	72.0	61.9
ANOVA	Time	**	***	***
	Pile	**	*	*
	Time × Pile	*	*	*

I, LB and M: initial, late bio-oxidative and mature phases, respectively.

HR: $(C_{Ex} / T_{OC}) \times 100$, HD: $((C_{AH} + C_{AF}) / T_{OC}) \times 100$ and P_{HA} : $(C_{AH} / C_{Ex}) \times 100$. C_{Ex} : total organic carbon extracted in 0.1 M NaOH; T_{OC} : total organic carbon, C_{AH} : humic-like acid carbon and C_{AF} : fulvic-like acid carbon.

NS: not significant and ***, ** and *: significant at $p < 0.001$, 0.01 and 0.05, respectively.

4. Water consumption. An aspersion system was necessary to keep the moisture content in the piles around 40%, with a cost of 200€. The total water consumption was estimated to be 40 m³, with an approximate cost of 30€.

The estimated cost for AL composting in our experiment was lower than the 37 and 55€ per t of AL treated for industrial composting processes in Italy and Spain, respectively (Cubero et al., 2011). This difference must arise from the amortization costs included in the latter processes (construction and maintenance of installations, composting machine, etc.), as well as to the different bulking agents (type and proportion) and composting technology used.

4. Concluding remarks

This study revealed that composting is an effective technology for adding value to AL through its transformation into commercial organic amendments and fertilisers. The manures assayed in our experiment acted as appropriate co-substrates for AL composting and the mechanical turnings applied were enough to aerate the piles, homogenising the substrate simultaneously. Several parameters were important for the control and monitoring of the composting process, such as temperature, moisture, pH, water-soluble fractions, OM losses and germination index. The addition of the Fe and P-enriched acidic mineral amendments reduced the pH values (by around 0.5 units during the process) and also the WSC, WSPH and hemicellulose contents.

The composts obtained lacked phytotoxicity and had valuable contents of nutrients and high contents of OM with humic properties and predominance of the lignocellulosic matrix. According to the new Spanish legislation for fertilisers (PRE/630/2011, 2011), these composts satisfied the minimal requirements for commercial categories such as AL Compost, Humic Organic Amendment, Compost Organic Amendment and NPK Organic Fertiliser. These AL composts can be mixed with other fertilisers to obtain new, valuable solid and

liquid formulations. In addition, *in-situ* composting of AL can be considered viable from an economic point of view, according to our results.

Acknowledgements

The authors thank the “Aceites Guadalentín S.A” olive mill, for financial and technical support and for disposal of the raw material, and Dr. David J. Walker for improvement of the written English.

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