

# Evaluation of “alperujo” composting based on organic matter degradation, humification and compost quality

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Received: 11 March 2008 / Accepted: 3 September 2008 / Published online: 24 September 2008  
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**Abstract** The main by-product generated by the Spanish olive oil industry, a wet solid lignocellulosic material called “alperujo” (AL), was evaluated as a composting substrate by using different aeration strategies and bulking agents. The experiments showed that composting performance was mainly influenced by the type of bulking agent added, and by the number of mechanical turnings. The bulking agents tested in this study were cotton waste, grape stalk, a fresh cow bedding and olive leaf; the latter showed the worse performance. Forced ventilation alone was revealed to work inadequately in most of the experiments. The composting process involved a substantial degradation of the organic substrate with average losses of 48.4, 28.6, 53.7 and 57.0% for total organic matter, lignin, cellulose and hemicellulose, respectively. Both organic matter biodegradation and humification were greatly influenced by the lignocellulosic nature of the starting material, which led to low organic matter and nitrogen loss rates and a progressive increase in more humified substances, as revealed by the end-values of the humification indices. The resulting composts were of good quality

in terms of nutrient content, stabilised and non-phytotoxic organic matter and low heavy metal content. This demonstrates that composting technology can be used as an alternative treatment method to turn AL into compost that can be used as organic amendments or fertilisers for agricultural systems.

**Keywords** Composting · Humification · Organic fertilisers and amendments · Organic matter degradation · Two-phase olive-mill by-product

## Introduction

The agro-industrial sector generates large amounts of biodegradable wastes and by-products, which need to be suitably disposed of in order to reduce their environmental impact. A clear example of this is the edible olive oil sector, a very important industry in the Mediterranean area that produces great amounts of organic residual materials, both liquid and solid, depending on the system used to extract the olive oil (traditional press, and continuous three or two-phase centrifugation). In addition, these materials are considered to be a potential pollution source for the environment and constitute a major management problem for the olive oil producing countries.

The main olive-mill by-product in Spain is “alperujo” (AL), which results from the widespread

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implementation of the two-phase centrifugation system; although the same product may be called wet husk, two-phase solid olive-mill by-product, two-phase olive pomace or olive husk. AL is a very wet and pasty lignocellulosic material, difficult to handle and transport, which includes potentially pollutant compounds such as phenols and fats. Apart from the treatments to recover its residual oil (second centrifugation, and drying followed by chemical extraction), several methods have been considered for AL disposal (Aragón and Palancar 2001; Borja et al. 2006; Miranda et al. 2007; Morillo et al. 2008; Rincón et al. 2008), including physical, chemical and biological processes. Amongst the last of these, composting is increasingly considered a good way of improving profits and the viability of the olive oil production process, and much research work has been carried out in the recent decades to recycle the organic matter and nutrient contents of olive-mill wastes by composting, as noted in Azbar et al. (2004) and Roig et al. (2006).

Composting is a biological aerobic process managed under controlled conditions. It involves a complex transformation of the raw organic substrates, which are degraded and transformed into stable organic matter containing “humic-like” compounds which distinguish them from those found in more evolved materials (native soils, coals and peats). These substances, which are the most active fraction in compost, positively affect soil ecology, structure, fertility and productivity and hence are considered a critical factor for agricultural production (Senesi 1989; Chen et al. 1994; Haider 1994). Substrate composition is a key controlling factor for both the degradation and humification processes; the high lignocellulosic fraction of AL may be expected to affect both the composting performance and the compost quality, as lignin limits degradation and is recognised to be an important precursor of humic substances (Charest and Beauchamp 2002; Komilis and Ham 2003; López et al. 2006; Smidt et al. 2008).

The present work summarizes the most relevant results gained in successive co-composting experiments of AL with several organic wastes as bulking agents. In particular, it is reported how forced ventilation, turning, and the type of bulking agent added to AL affected the composting performance, organic matter degradation and humification of the composting substrate. Finally, with the aim of

demonstrating the feasibility of AL composting to produce quality end-products, which are non-phyto-toxic and rich in partially humified organic matter, compost characteristics are presented.

## Materials and methods

### Collection and characterisation of raw materials

#### *Olive-mill by-product*

Three different AL samples (AL1, AL2 and AL3) were used for the composting experiments. All three samples exhibited unfavourable composting properties, such as: high moisture content, high lignin content, acidic pH and unbalanced C/N ratio (Table 1). In addition, the high lipid content may affect physical substrate properties, leading to an incorrect distribution of air, water and dissolved substances during the process.

#### *Bulking agents*

The unfavourable AL properties led to a wide search for organic wastes such as cotton waste (CW), maize and barley straw, rice husk, spent mushroom compost, grape marc, grape stalk (GS), olive leaf (OL), almond husk and a fresh cow bedding (FCB), which were analysed to check their suitability as bulking agents. CW, GS, OL and FCB were selected for the composting experiments taking into account their physico-chemical characteristics, availability and commercial cost. All of them generally showed lower water and lignin contents than AL, accompanied by higher pH and electrical conductivity (EC) values (Table 1). CW and FCB showed the lowest C/N ratio, the latter also having the greatest nutrient content.

### Composting performance

Data were obtained from three successive composting experiments (pile 1, piles 2–3 and piles 4–5, Table 2). Four mixtures were made by adding the selected bulking agents (CW, GS, OL and FCB) to the three AL samples and placed in trapezoidal piles, with approximate dimensions of 1.5 m height and a 2 × 3 m base. Urea was added to piles 2 and 3 in

**Table 1** Main characteristics of the raw materials used in the composting experiments (dry weight)

Parameters	AL			Bulking agents			
	AL1	AL2	AL3	CW	GS	OL	FCB
Moisture (% f.w.)	71.3	63.9	55.6	11.5	5.9	7.3	46.1
pH <sup>a</sup>	4.97	5.20	5.08	6.80	4.40	5.60	7.47
EC <sup>a</sup> (dS m <sup>-1</sup> )	3.01	4.12	4.76	4.12	4.24	1.74	7.53
OM (g kg <sup>-1</sup> )	952.6	948.0	942.3	933.0	934.1	906.0	664.0
Lignin (g kg <sup>-1</sup> )	449.0	323.0	335.0	232.0	362.3	319.1	185.0
Cellulose (g kg <sup>-1</sup> )	207.0	206.6	205.0	392.3	213.0	193.3	122.1
Hemicellulose (g kg <sup>-1</sup> )	379.3	356.4	406.0	207.8	167.2	217.0	325.3
Fat content (g kg <sup>-1</sup> )	116.3	77.5	112.0	21.0	12.6	32.5	2.9
C/N ratio	44.2	57.9	39.6	22.4	60.1	39.4	19.0
N (g kg <sup>-1</sup> )	12.2	8.9	13.2	21.3	8.0	13.3	19.4
P (g kg <sup>-1</sup> )	0.9	1.1	0.8	1.8	0.6	0.8	2.5
K (g kg <sup>-1</sup> )	15.9	25.3	27.3	17.4	20.0	5.7	35.8

<sup>a</sup> Water extract 1:10

EC: electrical conductivity, OM: total organic matter

**Table 2** Composition and management of composting substrates

Pile	Composition (%)		Weight (kg)	Turnings	Forced ventilation <sup>a</sup>
	Fresh basis	Dry basis			
1	92.6 AL1 + 7.4 CW	(80/20)	2,700	1	Yes (60°C)
2	94.6 AL2 + 5.0 GS + 0.4 urea	(87/12/1)	2,600	3	Yes (55°C)
3	94.6 AL2 + 5.0 OL + 0.4 urea	(87/12/1)	2,600	3	Yes (55°C)
4	90 AL3 + 9 FCB + 1 AL compost	(87/11/2)	4,000	14	No
5	90 AL3 + 9 FCB + 1 AL compost	(87/11/2)	4,000	14	Yes (55°C)

<sup>a</sup> Ceiling temperature for continuous air blowing

order to reduce the high initial C/N value, while mature compost from pile 3 was added to piles 4 and 5 as inoculum. Complete details of the composting performance can be consulted in Cegarra et al. (2000, 2006) and Alburquerque et al. (2006a).

In the composting experiments special attention was paid to the effects of the bulking agent and forced ventilation. A combination of the Rutgers system, which maintains a temperature ceiling in the pile through on-demand removal of heat by forced ventilation (Finstein et al. 1985), and mechanical turning was employed, except in pile 4, which was only turned. Turnings were performed once (week 8) in pile 1, three times (weeks 4, 10 and 21) in piles 2 and 3, and 14 times in piles 4 and 5. The active phase of composting (bio-oxidative period) was considered as finished when the temperature of the piles was close to ambient and re-heating did not occur, at which time air blowing and/or mechanical turning

were stopped and the materials left to mature (3–4 months). The total composting time was 42 weeks for pile 1, 49 weeks for piles 2–3 and 36 weeks for piles 4–5. Moisture was maintained within the range 40–55% by periodically sprinkling the piles with water.

Representative samples were collected from the composting materials, homogenised, and subdivided into three sub-samples in the laboratory. One of them was dried in an oven at 105°C for 24 h to determine its moisture content, the second was frozen at –20°C and kept for the determination of NH<sub>4</sub>-N and NO<sub>3</sub>-N, and the third sub-sample was freeze-dried and ground to less than 0.5 mm before analysis.

#### Analytical methods

The following parameters were determined in the composting materials: pH, EC and contents of total

nitrogen (N),  $\text{NH}_4^-$  and  $\text{NO}_3^-$ -N, P, K, Ca, Mg, Na, Fe, Cu, Mn, Zn, Pb, Cr, Ni, Cd, total organic matter (OM), total organic carbon (C), fats, lignin, cellulose, hemicellulose (as the difference between holocellulose and cellulose), total water-soluble organic carbon (WSC), carbohydrates and phenols as well as the germination index (GI), according to the methods previously described by Albuquerque et al. (2004) and Cegarra et al. (2006). After isolation of the lignin fraction, its organic carbon content (LC) was determined by automatic microanalysis (ANA 1500 Carlo Erba microanalyser). Losses of N, OM, lignin, cellulose, hemicellulose, fats and WSC were calculated taking into account the apparent increase in the ash content resulting from the loss of dry matter weight in order to better reflect overall changes (Stentiford 1993; Paredes et al. 1996).

#### Extraction, purification and characterisation of humic-like substances

Composting samples were treated with 0.1 M NaOH (1:20 w/v) and shaken in sealed bottles for 24 h. After centrifugation (20 min at 23,500g), the supernatant (alkali-extractable fraction) was carefully removed and adjusted to pH 2.0 by adding concentrated  $\text{H}_2\text{SO}_4$ . The precipitate obtained (humic-like acids, HAs) was allowed to coagulate for 24 h at 4°C, then separated by centrifugation from the supernatant fulvic fraction (FF) as above. The separation of non-humified organic matter (NH) from FF and the subsequent isolation of the fulvic-like acids (FA) was made according to Sequi et al. (1986) by using a column packed with polyvinylpyrrolidone. The organic carbon in the total alkali-extractable fraction (TEC) and that existing as FFC and NHC were determined using an automatic carbon analyser for liquid samples (SKALAR formacs<sup>HT</sup> TOC analyser), whereas humic-like acid carbon (HAC) and fulvic-like acid carbon (FAC) were calculated by subtracting FFC from TEC and NHC from FFC, respectively. The percentage of humic-like acids (PHA) defined as “ $(\text{HAC}/\text{TEC}) \times 100$ ”, the humification index (HI) as “ $\text{NHC}/(\text{HAC} + \text{FAC})$ ” (Sequi et al. 1986) and the degree of humification (DH) as “ $[(\text{HAC} + \text{FAC})/\text{TEC} \times 100]$ ” (Ciavatta et al. 1988), were calculated from the above carbon values.

The HAs were successively washed by shaking for 15 min twice with 0.1 M HCl, then once with

distilled water. After centrifugation as above, the supernatant fractions were discarded and the resultant purified HAs freeze-dried and characterised for C, H, S and N by using an ANA 1500 Carlo Erba microanalyser, whereas O was calculated as the difference from 100% (ash-free dry weight).

#### Statistical analyses

Statistical analyses of data and correlation studies were made using the SPSS 15.0 Programme for Windows, and curves fitting to OM-losses experimental data were obtained using the Programme SigmaPlot 2000 for Windows, Version 10.0. Data were subjected to ANOVA and differences between means were determined using the Tukey's test. To obtain additional information about the change in the parameters during the composting phase a *t*-test for related samples was performed.

## Results and discussion

### Composting process control

Given the physical properties of AL, which would lead to compaction and restrict gas transfer and distribution through the composting substrate (Filippi et al. 2002; Baeta-Hall et al. 2005), several aeration strategies in combination with bulking agent addition were tested, and their effect on pile temperature was monitored. In our experiments, aeration was first supplied by forced ventilation, but was only revealed to be effective when performed in conjunction with mechanical turning. Pile 1 reached the thermophilic phase with temperatures clearly higher than 45°C in the eighth week, coinciding with the only turning performed. Piles 2 and 3 showed a long activation phase and only after the first (week 4) and second (week 10) turning, respectively, high thermophilic temperatures were measured regardless of the intense forced ventilation supplied (Albuquerque et al. 2006a). In contrast, high thermophilic temperatures were quickly recorded in piles 4 and 5 (Cegarra et al. 2006), which was related to the better oxygen distribution favoured by either the frequent turnings (pile 4) or the conjunction of both forced ventilation and turning (pile 5), as well as to the considerable improvement in the substrate properties provoked by

FCB addition. The nutrient availability of FCB and its inoculum effect, together with the rather low water content of AL3 compared with AL1 and AL2 (Table 1), might have enhanced substrate aeration and accelerated the composting start-up phase. Therefore, turning encouraged composting by promoting physical mixing and homogenisation of the raw components, and re-distribution of microorganisms, moisture and nutrients. Furthermore, by improving the porosity of the AL substrates the efficacy of the forced ventilation system improved. Baeta-Hall et al. (2005) and Cayuela et al. (2006) also noted how turning led to better aerobic conditions, faster evolution and higher humification efficiency in the AL composting process. The pH was considered a very suitable indicator of the AL composting progress in our experimental conditions since it was closely related to the substrate aeration conditions, low increases in pH coinciding with low temperature periods and degradation rates.

AL substrate moisture decreased during composting, water addition being conditioned by pile weight, substrate temperature and water retaining capacity, composting time, forced ventilation, number of turnings and environmental conditions. Water consumption was greater in piles 2 and 3 (2.7 and 2.4 l kg<sup>-1</sup> of initial dry weight, respectively) than in piles 1 and 5 (2.0 l kg<sup>-1</sup>) because of the longer composting time in the first two, whereas pile 4 showed the lowest consumption (1.7 l kg<sup>-1</sup>) due to the shorter composting time and the lack of forced ventilation. Therefore, moisture conditions should be carefully taken into account before putting into full scale operation AL composting since they clearly influence the physical substrate properties and hence oxygen availability. Optimisation of water consumption may well result in a reduction of production costs.

#### Nitrogen turnover and losses

There was a gradual increase of the total N concentration in all cases (from 14–16 to 22–26 g kg<sup>-1</sup>), which generally coincided with the most intense organic matter degradation periods as a result of the weight loss and the concentration effect. In none of the experiments was the NH<sub>4</sub>-N decrease accompanied by a clear increase in NO<sub>3</sub>-N, which was only

detected at very low concentrations at the end of composting (<108 mg kg<sup>-1</sup>).

N-losses during AL composting were much lower than those recorded for other composting substrates, such as mixtures containing sewage sludge (Witter and López-Real 1988; Sánchez-Monedero et al. 2001) or manures (Martins and Dewes 1992). The highest N-losses were detected in pile 5 (Table 3) due to the intense periods of forced ventilation and the rapid increase in pH. According to Witter and López-Real (1988), Liang et al. (2000) and Jeong and Kim (2001), the above mentioned conditions favour ammonia formation and volatilisation. On the contrary, pile 3 registered a final gain of 9.9%. This different behaviour can be attributed to the lower OM decomposition rate of pile 3, which would have contributed to nitrogen immobilisation and subsequently to less ammonia formation and volatilisation. Moreover, the negative N-losses calculated during the initial composting phase in piles 1–3 (Table 3) can be explained by biological nitrogen fixation phenomena, as revealed by Paredes et al. (1996) during the composting of olive-mill wastewater sludge.

#### Organic matter degradation

During the whole process, the OM content fell from initial values of 940 g kg<sup>-1</sup> in piles 1–3 and 906 g kg<sup>-1</sup> in piles 4 and 5 to approximately 900 and 800 g kg<sup>-1</sup>, respectively (Table 3). The resultant OM-losses (Fig. 1), calculated as a percentage of the initial content, reflected clear differences between piles. Pile 5 showed the highest losses (60.5%) and pile 3 the lowest (34.4%, Table 3). In addition, piles 2 and 3 exhibited an initial lag phase in OM loss evolution of 4 and 8 weeks, respectively (Fig. 1), even when regular forced ventilation was supplied from the start of the experiment. As mentioned before, the lag period coincided with periods of low temperatures and persistent acidity. The degradative process in both piles was only activated by turning, although pile 3 always showed a lower progress of the degradation process. In contrast, pile 1 and, especially, 4 and 5 showed a rapid initiation of the degradation process without lag phases (Fig. 1).

The study of the trends in OM loss led to a clear insight into the progress of substrate degradation during composting. As shown in Table 4 and Fig. 1,

**Table 3** Losses of total nitrogen, organic matter, lignin, cellulose and hemicellulose (% of the initial content) at different composting stages

Pile	Composting phase	N	OM	LIG	CEL	HEMIC
1	I	0.0cd	0.0g [933.9]	0.0f	0.0g	0.0i
	T	-0.4cd	18.7f [919.9]	12.9cd	22.7f	19.5h
	LB	11.2abc	42.1cd [891.0]	29.7b	53.8bc	48.4de
	M	15.9ab	43.0cd [889.5]	30.4b	55.1bc	50.5cde
2	I	0.0cd	0.0g [945.2]	0.0f	0.0g	0.0i
	T	-10.2f	14.0f [936.8]	4.6ef	24.4f	21.4h
	LB	13.2abc	46.0bc [903.0]	24.5bc	50.7bc	45.1e
	M	11.9abc	50.0abc [896.0]	26.8b	54.1bc	51.5cde
3	I	0.0cd	0.0g [940.6]	0.0f	0.0g	0.0i
	T	-2.8de	3.9g [938.4]	1.6f	5.4g	6.7i
	LB	-9.2ef	31.1e [916.1]	10.1e	24.4f	39.6fg
	M	-9.9f	34.4de [912.2]	14.3cd	33.2e	44.9e
4	I	0.0cd	0.0g [906.0]	0.0f	0.0g	0.0i
	T	3.9cd	30.5e [870.1]	9.4e	21.0f	33.0g
	LB	10.7abc	45.2bc [840.9]	23.1bc	43.3d	54.9cd
	M	6.5bc	54.1ab [815.5]	30.2b	60.0ab	68.1ab
5	I	0.0cd	0.0g [906.6]	0.0f	0.0g	0.0i
	T	16.3ab	44.9bc [842.5]	26.6b	35.7e	47.6de
	LB	22.7a	51.2abc [825.6]	32.0ab	51.4bc	60.2bc
	M	18.3ab	60.5a [792.9]	41.4a	66.1a	70.0a
Pile		*	*	*	*	*
Composting phase		*	*	*	*	*
Pile × composting phase		*	*	*	*	*

\* Significant at  $P < 0.001$

For each parameter, values followed by the same letters are not statistically different according to the Tukey's test at 5% probability level

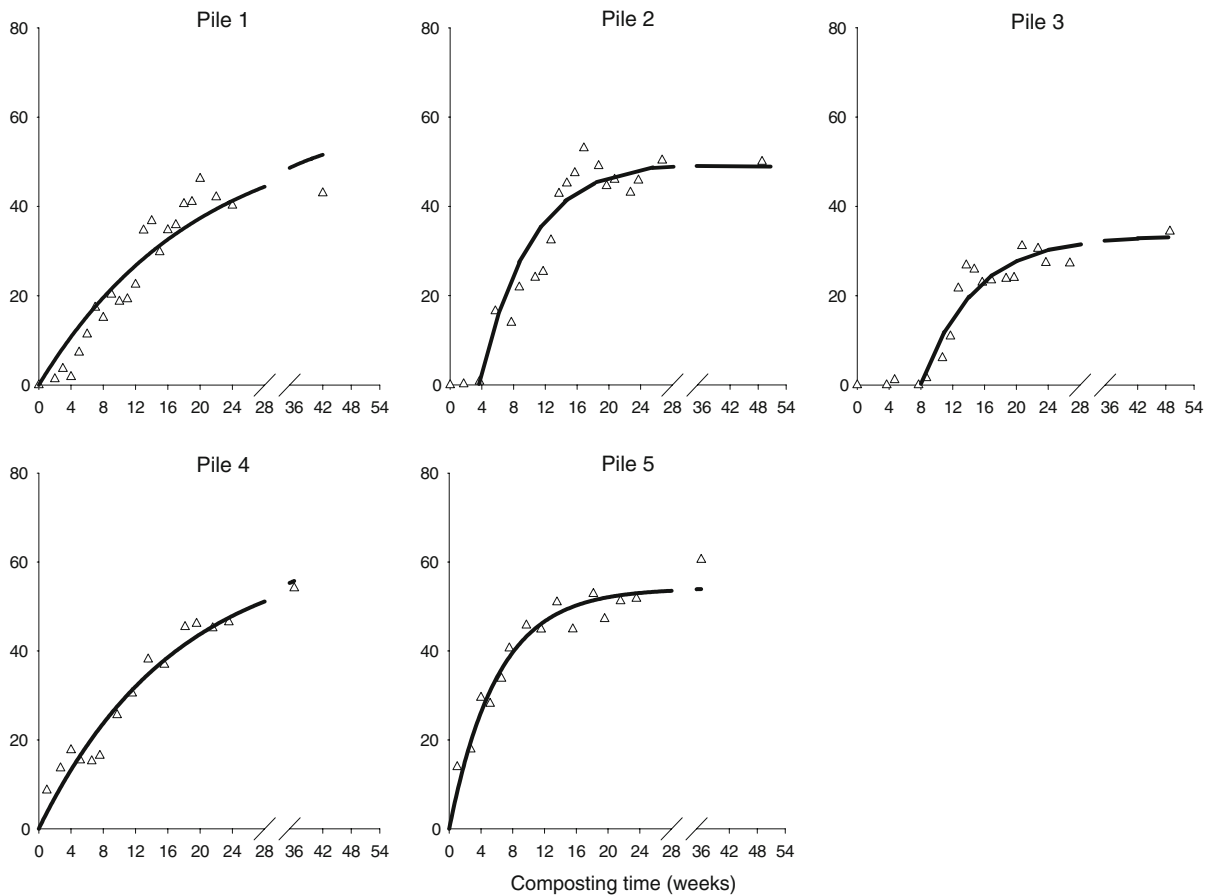
N: total nitrogen, OM: total organic matter, LIG: lignin, CEL: cellulose; HEMIC: hemicellulose. Figures between square brackets represent the OM content ( $\text{g kg}^{-1}$  dry weight)

I, T, LB and M: initial, thermophilic (coinciding with maximum temperatures at about the tenth week), late bio-oxidative and mature phases, respectively

OM-losses followed a first-order kinetic model " $A(1 - e^{-kt})$ " in all the piles as generally found in composting experiments, where  $A$  is the maximum degradation of OM (%),  $k$  the rate constant ( $\text{days}^{-1}$ ) and  $t$  the composting time (days). The curve-fitting for piles 2 and 3 were made considering  $t = 0$  after the mentioned lag phase. The  $A$  and  $k$  values obtained were within the range found by different authors in other composting experiments with lignocellulosic materials (Benito et al. 2003; García-Gómez et al. 2003; Bustamante et al. 2008): 46.0–78.7% and 0.0048–0.0294  $\text{days}^{-1}$ , respectively.

When the parameters of the first-order kinetic model were compared (Table 4), clear differences

among piles were observed in accordance with the aeration strategy and the bulking agent added. Pile 5 showed the highest  $k$  and  $A \times k$  values, reflecting that the combination of the high number of turnings (14) and prolonged forced ventilation periods (on/off cycles of 5/15 min during the most active phase of the process) led to the greatest OM decomposition rate. Similar results to pile 5 were obtained in pile 2 after the lag period, pile 2 combined 3 turnings and also prolonged forced ventilation periods (on/off cycles of 3/15 min). On the contrary, pile 3 (3 turnings and on/off cycles for forced ventilation of 3/15 min as in pile 2) showed lower kinetic parameter values, which must be attributed to the scant



**Fig. 1** Evolution of OM-losses (% of initial value) during composting. Lines represent curve-fitting to experimental data

**Table 4** Parameters of the first-order kinetic model “ $A(1 - e^{-kt})$ ” fitted to experimental data of OM-losses; “A”, the maximum loss (%), “k”, the rate constant ( $\text{days}^{-1}$ ), “t”, the composting time in days and “ $A \times k$ ”, the mineralisation rate ( $\% \text{ OM days}^{-1}$ )

Pile	A	k	$A \times k$	RMS	F
1	55.5 (8.0)	0.0073 (0.0017)	0.41	22.3	183.3*
2	50.3 (2.5)	0.0226 (0.0036)	1.14	27.3	137.9*
3	33.2 (2.1)	0.0212 (0.0038)	0.70	13.7	127.2*
4	63.5 (6.2)	0.0083 (0.0014)	0.53	12.4	313.9*
5	54.1 (1.9)	0.0236 (0.0026)	1.28	12.4	317.6*

\* Significant at  $P < 0.001$ , Standard error in brackets. Statistical parameters: residual means square (RMS) and F factor

improvement in the AL physical structure achieved by the bulking agent added to this pile (OL) which was unable to keep optimum aerobic substrate conditions for a further progress of the degradation

process. Finally, pile 1 showed A, k and  $A \times k$  values close to pile 4 which are consistent with high OM-losses at the end of the process (A values) but, at a lower rate compared to piles 2, 3 and 5 (k and  $A \times k$  values, Table 4). These results must be related to the less sustained and intense oxygen supply of pile 1 (one turning combined with on/off cycles of 0.5/14.5 min for forced ventilation) and 4 (only turned, 14) compared to piles 2, 3 and 5.

The total time needed for AL composting, particularly the thermophilic phase, which led to bio-oxidative periods of 26 weeks (piles 1, 4 and 5) and 34 weeks (piles 2–3), were much longer than that needed for other substrates such as manures, sewage sludge and municipal solid wastes. This was mainly due to the high AL lignin content that is not easily degraded by microorganisms (Faure and Deschamps 1991; Whitney and Lynch 1996; Charest and Beauchamp 2002). In this respect, other lignocellulosic

materials have also shown long composting times (Eiland et al. 2001; Madejón et al. 2001; Charest and Beauchamp 2002) and, in the particular case of AL, this has been explained by the large amounts of oily substances which release energy and favour the further degradation of highly recalcitrant compounds such as lignin, leading to an extended degradation process and hence longer composting times (Manios et al. 2006).

Substrate aeration greatly conditioned lignin degradation in our experiments (Table 3), which was higher in piles 1–2 than in 3 and in pile 5 than in 4. As mentioned before, OL (pile 3) was a worse bulking agent than CW and GS (piles 1 and 2, respectively), leading to more deficient aerobic conditions in pile 3, while pile 5 was better aerated than pile 4 which was only turned. In accordance with these results, Haider (1994) and Pichler and Kogel-Knabner (2000) noted that lignin degradation is limited by deficient aeration conditions. Losses of cellulose and hemicellulose were generally greater than 50% by the end of composting (Table 3), pile 3 showing the lowest (33.2% and 44.9%, respectively) and pile 5 the highest loss (66.1% and 70.0%, respectively), as was similarly found for lignin. The faster and greater degradation of the three biopolymers in pile 5 was the consequence of the better substrate aeration conditions mentioned above, which favoured its degradation as a whole.

All piles followed a similar biodegradation pattern, in that cellulose and hemicellulose were more extensively degraded than lignin. Figure 2 depicts a comparison between the lignin (LC) and non-lignin (NLC) carbon content before and after composting. As can be seen, all the substrates initially showed a higher NLC content because of the pre-eminence of readily biodegradable organic compounds (simple carbohydrates, lipids and peptides, starch, pectins, aminoacids, etc.). On the contrary, LC was predominant and a significant increase in the LC/NLC ratio ( $P < 0.001$ ) was recorded in all the piles after composting (Fig. 2) due to the greater resistance of lignin to biodegradation. This provoked a progressive stabilisation of substrates, the degradation rate gradually decreasing as composting progressed due to a strong diminution in the available carbon sources.

#### Humification process

Humification progress was studied by monitoring changes during composting in the carbon content of

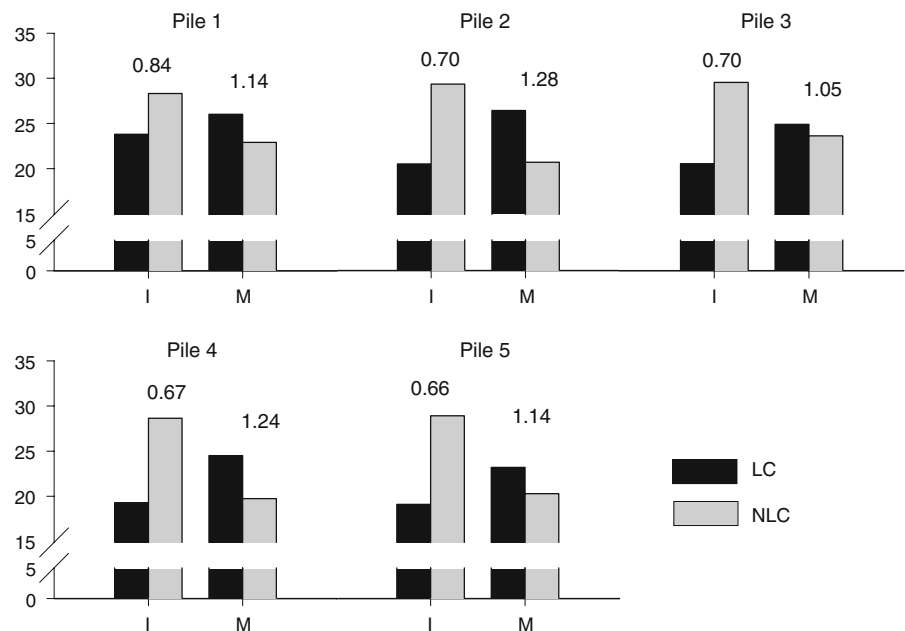
alkali extracted humic-like substances and characterising their main fraction (HAs) through elemental analysis (Table 5). Although alkaline extraction methods raise problems due to the co-extraction of non-humic components, determination of the DH and HI humification indices (based on the separation of non-humified and humified materials in the FF) has been considered more accurate than determining the classical indices to evaluate the stability and humification degree of composting organic substrates (Sequi et al. 1986; Ciavatta et al. 1988; Tomati et al. 1995, 2000; Mondini et al. 2006).

According to the *t*-test results for related samples, significant changes were observed in DH, HI, and PHA coinciding with the most active phase of composting (bio-oxidative), while they were not significant during the maturation period (Table 6); Mondini et al. (2006) reported similar results and noted how the humification process mainly occurred during the thermophilic phase, when lignocellulosic materials were being composted. In our case, DH and PHA increased and HI decreased in all the piles (Tables 5, 6), the mentioned increase meaning a growing predominance of humified substances (HAC + FAC) and the presence of more polymerised compounds (HAC), while the decrease indicates diminution of non-humified compounds (NHC). The end-values obtained for these indices (DH > 74%, HI < 0.35 and PHA > 67%) suggest OM stability according to the results found by Tomati et al. (1995, 2000) and Mondini et al. (2006) in composts made of lignocellulosic materials (olive-mill and green wastes, respectively). Piles 4 and 5 exhibited higher DH and lower HI values than piles 1, 2 and 3 at the end of the process ( $P < 0.001$ , Table 5), which suggest a greater predominance of the humified substances in these piles in accordance with the better overall progress of composting in piles 4 and 5 as mentioned before.

In addition, there were significant correlations between organic fraction losses and the humification indices when the piles were considered together (Table 7). The above findings must be connected to the fact that the degradation process during composting reduced the amount of non-humified compounds extracted in alkali conditions, as shown by the above mentioned decrease in NHC, and also resulted in the release of compounds which were incorporated into the humification pathways through reorganisation and



**Fig. 2** Content of LC and NLC carbon as % dry weight, before (I) and after (M) composting. NLC was calculated as the difference between C and LC, and numbers on the bars represent the LC/NLC ratio



molecular condensation reactions (Varadachari and Ghosh 1984; Ertel et al. 1988; Sánchez-Monedero et al. 1999), leading gradually to more humified, polymerised and oxidised compounds. In this respect, lignin is known to play a major role in the humification of lignocellulosic wastes, since lignin partially degraded by oxidation and de-polymerisation reactions may form the basic structure of humic substances, and also liberate aromatic and phenolic by-products to form humic structures (Ertel et al. 1988; Hatcher and Spiker 1988; Serra-Wittling et al. 1996; Veeken et al. 2000). All these phenomena contributed to increasing the content of humic-like substances in the AL compost and its ligno-humic core.

As composting progressed, the HAs gradually became richer in N and O and poorer in H and C leading to decreases in the H/C ratio and increases in N/C and O/C ratios (Table 5). As shown in Table 6, these changes were only statistically significant between I and T phases for H/C and N/C ratios, and between T and LB for O/C (Table 6). The H/C decrease is likely related to increasing aromaticity, while the N/C increase suggests more nitrogen involved in the humic-like structures through condensation reactions between lignin or components derived from lignin and nitrogenous compounds, and also as a result of sugar-amine condensation

(Stevenson 1994). The O/C increase might have been connected with the appearance of more oxidised molecules in the HA structure. Similar results were found by Sánchez-Monedero et al. (2002) and Huang et al. (2006).

Compared with those from soils (Schnitzer and Preston 1986; Stevenson 1994) and coals (García et al. 1996), the HAs extracted from AL compost exhibited lower O/C and higher H/C and N/C ratios (Table 8). This reflects the much younger form of the AL compost HAs, which would still have similar structures to the native plant material, the time required for composting being much shorter than the much longer periods needed for humification in soils or for plant material fossilisation. All this leads to very oxidised and aromatic humic structures, poor in nitrogen (Chen et al. 1996).

The elemental composition of the HAs was similar at the end of composting in all the piles (Table 8), as also similar to the HAs from composts made from other agro-industrial wastes (Senesi et al. 1996; Sánchez-Monedero et al. 2002) which reflected the same origin of these raw organic wastes in which lignocellulose is a major component. Therefore, the long bio-oxidative phase of the AL composting process and the high lignin content of AL substrate led to humification parameters and atomic ratios similar among all piles at the end of the process.

**Table 5** Humification indices and atomic ratios ( $\times 100$ ) of humic-like acids extracted at different composting stages

Pile	Composting phase	DH (%)	HI	PHA (%)	H/C	O/C	N/C
1	I	55.8	0.79	48.7	153	28	4
	T	72.5	0.38	65.8	127	36	7
	LB	75.2	0.33	68.8	116	40	8
	M	74.4	0.34	67.2	112	41	7
2	I	53.4	0.87	37.9	137	35	4
	T	63.4	0.58	52.7	125	38	5
	LB	74.6	0.34	66.6	110	45	6
3	I	46.4	1.16	27.4	150	31	3
	T	59.0	0.70	45.5	146	28	3
	LB	73.1	0.37	66.6	111	39	7
	M	75.9	0.32	68.2	116	38	7
4	I	53.1	0.88	39.4	157	31	3
	T	68.5	0.46	65.7	128	42	7
	LB	78.1	0.28	74.7	123	46	8
	M	79.0	0.27	75.3	119	46	8
5	I	54.8	0.82	41.0	156	32	3
	T	76.9	0.30	68.7	126	44	7
	LB	79.9	0.25	72.2	122	48	7
	M	82.1	0.22	72.3	121	46	7

DH:  $[(\text{HAC} + \text{FAC})/\text{TEC}] \times 100$ , HI:  $\text{NHC}/(\text{HAC} + \text{FAC})$ , PHA:  $(\text{HAC}/\text{TEC}) \times 100$ , TEC: total organic carbon extracted in 0.1 M NaOH, HAC: humic-like acid carbon, FAC: fulvic-like acid carbon and NHC: non-humified organic carbon from the fulvic fraction

I, T, LB and M: initial, thermophilic (coinciding with maximum temperatures at about the tenth week), late bio-oxidative and mature phases, respectively

#### AL compost characteristics

The addition of FCB (a material rich in mineral nutrients) to piles 4 and 5 and the resulting higher mineralisation led to end materials with a lower OM content and higher EC and nutrient values than were observed in piles 1, 2 and 3. Despite these differences, the characteristics of the AL compost were quite similar in all the piles, as shown in Table 9.

AL compost showed a pleasantly earthy smell, alkaline pH, moderate salinity and high C/N ratio. Nearly half of its high OM content was lignin, which is considered to be an important precursor of soil humic substances and responsible for the above mentioned high C/N values. Other important constituents were cellulose and hemicellulose, while fats

**Table 6** Differences of humification indices and atomic ratios ( $\times 100$ ) between composting phases (I: initial, T: thermophilic, LB: late bio-oxidative and M: mature), averaged for all piles, and values of the t-test for related samples

	I→T	T→LB	LB→M
$\Delta\text{DH}$	-15.4	-8.1	-0.9
<i>t</i>	-7.514*	-3.578***	-1.292ns
$\Delta\text{HI}$	0.42	0.17	0.01
<i>t</i>	11.107**	3.119***	1.200ns
$\Delta\text{PHA}$	-20.8	-10.1	-0.9
<i>t</i>	-8.011**	-2.974***	-1.014ns
$\Delta\text{H/C}$	20	14	1
<i>t</i>	3.899***	2.490ns	0.609ns
$\Delta\text{O/C}$	-6	-6	1
<i>t</i>	-2.229ns	-4.353***	1.395ns
$\Delta\text{N/C}$	-2	-1	0
<i>t</i>	-2.954***	-2.064ns	1.000ns

ns: not significant, \*, \*\*, \*\*\* Significant at a probability level  $P < 0.05, 0.01, 0.001$ , respectively

$\Delta = 1\text{st phase} - 2\text{nd phase}$

DH:  $[(\text{HAC} + \text{FAC})/\text{TEC}] \times 100$ , HI:  $\text{NHC}/(\text{HAC} + \text{FAC})$  and PHA:  $(\text{HAC}/\text{TEC}) \times 100$

and water-soluble carbohydrates and phenols were minor components (<1%). It also exhibited GI values higher than 70%, which indicated a clear detoxification of the initial substrate and was related with the suitable stability and maturity of the AL compost (Albuquerque et al. 2006b).

Compared with urban waste compost and manures (Cegarra et al. 1993; Pascual et al. 1997; Canet et al. 2000; Sager 2007), the AL compost was rich in K and organic N but poor in P, Ca, Mg, and micronutrients. The content of Na and potentially toxic harmful heavy metals was much lower in the AL compost. Moreover, more than 20% of C was quantified as TEC, around 70% of it being HAC (Table 9), which means that AL compost could also be used to produce commercial liquid organic fertilisers.

The above characteristics make AL compost suitable for agricultural requirements and suggest that it can be used as an effective product for plant growth according to European Guidelines (European Commission 2001) as well as for soil amendment due to the strong resistance of its lignocellulosic matrix to edaphic degradation (Albuquerque et al. 2006c). However, the low nitrogen mineralisation rate could limit its agronomic use due to its low content in

**Table 7** Correlation matrix between losses and humification indices

Indices	Losses					
	OM	Lignin	Cellulose	Hemicellulose	Fats	WSC
DH	0.947*	0.895*	0.910*	0.952*	0.956*	0.924*
HI	-0.913*	-0.851*	-0.879*	-0.917*	-0.947*	-0.921*
PHA	0.920*	0.843*	0.875*	0.922*	0.953*	0.902*

\* Significant at  $P < 0.001$  (correlated points = 20)

OM: total organic matter, WSC: total water-soluble organic carbon, DH:  $[(\text{HAC} + \text{FAC})/\text{TEC}] \times 100$ , HI:  $\text{NHC}/(\text{HAC} + \text{FAC})$  and PHA:  $(\text{HAC}/\text{TEC}) \times 100$

**Table 8** Comparison of the elemental composition of humic-like acids from AL compost ( $n = 5$ ) and those extracted from other humic materials (g  $\text{kg}^{-1}$  ash-free dry weight)

Parameters	AL compost	CV (%)	Soil humic acids <sup>b</sup>	Leonardite <sup>c</sup>	Lignite <sup>c</sup>	Agro-industrial waste compost <sup>d</sup>
C	547.9–593.6	3.5	534.0–0599.0	530.5–593.6	522.8–607.4	466.0–625.0
N	42.0–49.2	6.2	8.0–43.0	12.5–16.1	7.9–9.8	24.0–53.0
H	53.0–57.9	3.4	32.0–62.0	45.0–47.0	31.6–36.8	44.0–95.0
O	296.7–337.9	5.4	302.9–393.0	345.1–410.5	351.2–433.1	237.0–443.0
H/C <sup>a</sup>	109–121	4.3	73–102	91–103	62–83	100–167
O/C <sup>a</sup>	38–46	8.3	39–55	44–58	43–62	30–60
N/C <sup>a</sup>	6–8	10.1	3–6	2	1	3–9

CV: Coefficient of variation

<sup>a</sup> Atomic ratio  $\times 100$

<sup>b</sup> Humic acids from soil (Schnitzer and Preston 1986; Stevenson 1994)

<sup>c</sup> Leonardite and lignite (García et al. 1996)

<sup>d</sup> Agro-industrial waste composts (Senesi et al. 1996; Sánchez-Monedero et al. 2002)

readily available forms and provoke a scarce availability in the short term so that it may require the addition of supplementary nitrogen fertilisers (Alburquerque et al. 2007).

## Conclusions

Both the selection of an appropriate bulking agent (OL was the worst bulking agent assayed) and mechanical turning were the key factors for the proper AL composting performance in the experimental conditions tested. The best results were found in piles 4 and 5, which combined the use of FCB and the highest number of turnings. This treatment resulted in a clear reduction in the composting time, higher mineralisation of the substrate and better humification indices. In order to reduce operating costs, the use of mechanical turning without forced ventilation is recommended due to its effectiveness

and simplicity of implementation for future industrial scale development of AL composting.

As composting advanced, a gradual humification of the composting substrate, clear increases in pH and GI and decreases in C/N ratio, fats, and water-soluble compounds were detected. In addition, temperature, pH, OM loss and GI are considered the most suitable parameters to monitor the AL composting progress. The lignocellulosic nature of AL led to a long composting time, low nitrogen loss, and mineralisation rates and an end-product rich in humic-like compounds.

The resulting composts were rich in organic matter, had an acceptable nutrient content and showed values of humification (DH > 74%, HI < 0.35 and PHA > 67%) and maturity (GI > 70%) indices compatible with agronomic use. Thus, AL compost can be considered an effective product for plant growth and soil amendment and fertilisation, even if supplementary nitrogen is required. It also contained a large

**Table 9** Main characteristics of the AL compost ( $n = 5$ , dry weight)

Parameters	Mean	Range	CV (%)
pH <sup>a</sup>	8.78	8.49–8.99	2.3
EC <sup>a</sup> (dS m <sup>-1</sup> )	3.71	2.96–4.81	23.7
OM (g kg <sup>-1</sup> )	861.2	792.9–912.2	6.2
C (g kg <sup>-1</sup> )	464.3	434.8–489.0	5.3
TEC (g kg <sup>-1</sup> )	102.2	86.8–123.0	15.2
HAC (g kg <sup>-1</sup> )	71.8	64.1–82.6	10.9
N (g kg <sup>-1</sup> )	24.1	21.9–26.3	8.6
C/N ratio	19.5	16.6–22.3	13.4
NH <sub>4</sub> -N (mg kg <sup>-1</sup> )	137	86–185	31.6
NO <sub>3</sub> -N (mg kg <sup>-1</sup> )	67	31–108	55.1
P (g kg <sup>-1</sup> )	1.7	1.4–1.9	13.9
K (g kg <sup>-1</sup> )	37.2	27.5–42.7	16.7
Ca (g kg <sup>-1</sup> )	17.4	9.4–29.7	51.1
Mg (g kg <sup>-1</sup> )	3.6	1.9–5.7	48.4
Na (g kg <sup>-1</sup> )	2.6	1.0–4.1	55.2
Fe (mg kg <sup>-1</sup> )	987	525–1468	43.5
Cu (mg kg <sup>-1</sup> )	30	21–36	22.5
Mn (mg kg <sup>-1</sup> )	62	38–98	44.9
Zn (mg kg <sup>-1</sup> )	78	38–138	62.8
Pb (mg kg <sup>-1</sup> )	11	5–20	68.0
Cr (mg kg <sup>-1</sup> )	6	nd–12	97.0
Ni (mg kg <sup>-1</sup> )	7	4–10	35.1
Cd (mg kg <sup>-1</sup> )	nd	–	–
GI (%)	79	71–83	6.4
<i>Main organic components (g kg<sup>-1</sup> dry weight)</i>			
Lignin	409.4 [47.5]	387.2–430.4	4.4
Hemicellulose	282.9 [32.9]	245.9–314.2	10.2
Cellulose	181.8 [21.1]	156.7–208.0	11.2
Fats	8.1 [0.9]	4.4–13.9	47.9
Water-soluble carbohydrates	10.1 [1.2]	8.1–13.1	19.3
Water-soluble phenols	3.2 [0.4]	2.3–4.1	20.7

<sup>a</sup> Water extract 1:10

CV, coefficient of variation; nd, not detected; EC, electrical conductivity; OM, total organic matter; C, total organic carbon; TEC, total organic carbon extracted in 0.1 M NaOH; HAC, humic-like acid carbon; N, total nitrogen; GI, Germination index. Figures between square brackets represent percentages with respect to the total organic matter content

humic-like fraction, which if properly extracted, could be used to obtain alternative commercial liquid products for fertigation.

**Acknowledgements** The authors wish to thank Mr. P. Thomas for checking the English of the manuscript and Drs. M. P. Bernal and R. Clemente for their scientific comments and assistance in the statistical studies.

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