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ORIGINAL PAPER

Two-phase olive mill waste composting: enhancement of the composting rate and compost quality by grape stalks addition

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Abstract Two-phase olive mill waste (TPOMW) is a semisolid sludge generated by the olive oil industry. Its recycling as a soil amendment, either unprocessed or composted, is being promoted as a beneficial agricultural practice in the Mediterranean area. One of the major difficulties when composting TPOMW is the compaction of the material due to its dough-like texture, which leads to an inadequate aeration. For this reason, the addition of bulking agents is particularly important to attain a proper composting process. In this study we followed the evolution of two composting mixtures (A and B) prepared by mixing equal amounts of TPOMW and sheep litter (SL) (in a dry weight basis). In pile B grape stalks (GS) were added (10% dry weight) as bulking agent to study their effect on the development of the composting process and the final compost quality. The incorporation of grape stalks to the composting mixture changed the organic matter (OM) degradation dynamics and notably reduced the total amount of lixiviates. The evolution of several maturation indices (C/N, germination index, water soluble carbon, humification indices, C/N in the leachates) showed a faster and improved composting process when GS were added. Moreover, chemical (NH_4^+, NO_3^-, cation exchange capacity, macro and micronutrients, heavy metals) and physical properties (bulk and real densities, air content, total water holding capacity, porosity) of the final composts were analysed and confirmed the superior quality of the compost where GS were added.

Keywords Two-phase olive mill waste · Grape stalks · Sheep litter · Leachates · Maturity

Introduction

European olive oil industry manufactures more than 75% of the olive oil produced worldwide (Roig et al. 2006). In the last decade the two-phase centrifugation system has been implemented in many productive areas, notably in Spain, which is the leading world producer. This system has considerably reduced the amount of water needed and the wastes generated during the olive oil extraction. However, in spite of its environmental advantages, olive mill waste disposal problems still remain unsolved. In fact, the semisolid sludge produced, known as two-phase olive mill waste (TPOMW), causes more disposal problems than the olive husk of the previous three-phase system and it demands for new environmental and economically viable management alternatives. At

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present, the most consolidated disposal option is a second extraction of the remaining oil and later combustion (Roig et al. 2006). Even so, the large proportion of organic matter and valuable nutrients present in olive mill wastes turns them into a valuable resource for beneficial utilization in agricultural soils.

Millions of tons of TPOMW are produced every year, most of them in Southern Mediterranean regions, where the concentration of organic matter in soil is extremely low and climate conditions, intensive agricultural practices and erosion have led to severe soil degradation in many areas. The utilisation of raw TPOMW as soil amendment has been proposed by several researchers who found positive results on crop yield (López-Piñeiro et al. 2006), with C sequestration aims (Sanchez-Monedero et al. 2008) or as biocide against fungi, weeds and nematodes (Cayuela et al. 2008a). However, olive mill wastes not always can be directly applied to soil since they exhibit toxic properties to several plants and soil micro organisms and can modify the equilibrium of soil nutrients. These inconveniences have led to the study of a broad number of possible remediation and treatment strategies (Sampedro et al. 2007; Aloui et al. 2008). Among them, composting TPOMW with other agricultural by-products has been proposed to obtain a stabilised, safe, balanced soil amendment (Cayuela et al. 2005; Alburquerque et al. 2006).

One of the main difficulties underlined by several researchers while composting TPOMW is the physical consistency of the residue, with a high moisture, low porosity and a doughy texture that makes aeration particularly difficult (Alburquerque et al. 2006; Cayuela et al. 2006). As a consequence, the addition of bulking agents is required to guarantee the correct development of the process.

TPOMW has been co-composted with different residues such as sheep litter (Cayuela et al. 2005), olive leaves (Alburquerque et al. 2006) or grape stalks (Baeta-Hall et al. 2005). However, the improvement of process development and physico-chemical quality produced by addition of grape stalks to a composting mixture has not been evaluated so far.

The aim of this work was to quantify and evaluate the physico-chemical changes originated by the addition of a bulking agent on a composting mixture made of TPOMW and sheep litter (SL). Our hypothesis was that the incorporation of grape stalks would improve not only the rate and development of the process, but also the quality of the final product.

Materials and methods

Composting pile elaboration

Two composting piles (A and B) were prepared by mixing two-phase olive mill waste (TPOMW) (total organic C: 55%; total N: 1.0%; pH: 5.5) and sheep litter (SL) (total organic C: 27%; total N: 2.3%; pH: 8.9) (50:50, on a dry weight basis). Grape stalks (total organic C: 37%; total N: 1.8%; pH: 8.0) were added to pile B in a proportion of 10% (dry weight). Grape stalks are characterised by a high ramification structure that leads to excellent bulking properties.

Both mixtures were composted in trapezoidal piles (1 m high and 2 × 3 m base) by the Rutgers system with occasional turnings. Pile A underwent seven windrow turnings whereas pile B only five. Water was regularly added (around 30 l each time) to maintain appropriate moisture (Fig. 1). In total, 1.3 m^3 of water were added to each pile along the whole process. During the composting period, the leachates produced from each pile were measured, sampled and stored at 4°C until chemical analysis.

Compost sampling was made by mixing five subsamples from different locations of the pile at five different stages of the composting process:

- I: Initial mixture (day 1)
- M: Mesophilic phase (day 35)
- T1: Thermophilic phase (day 126)
- T2: Thermophilic phase [day 201 (pile A) and 163 (pile B)]
- F: Final mature compost obtained (day 279 (pile A) and 241 (pile B))

Chemical analysis

Total nitrogen (TN) and organic carbon (TOC) were determined by automatic elemental microanalysis. Carbon losses were calculated according to the equation:

$$C - \text{loss}\ (\%) = 100 - 100[(X_1 C_2)/(X_2 C_1)]$$

where X_1 and X_2 are the initial and the final ash concentrations, and C_1 and C_2 are the initial and final

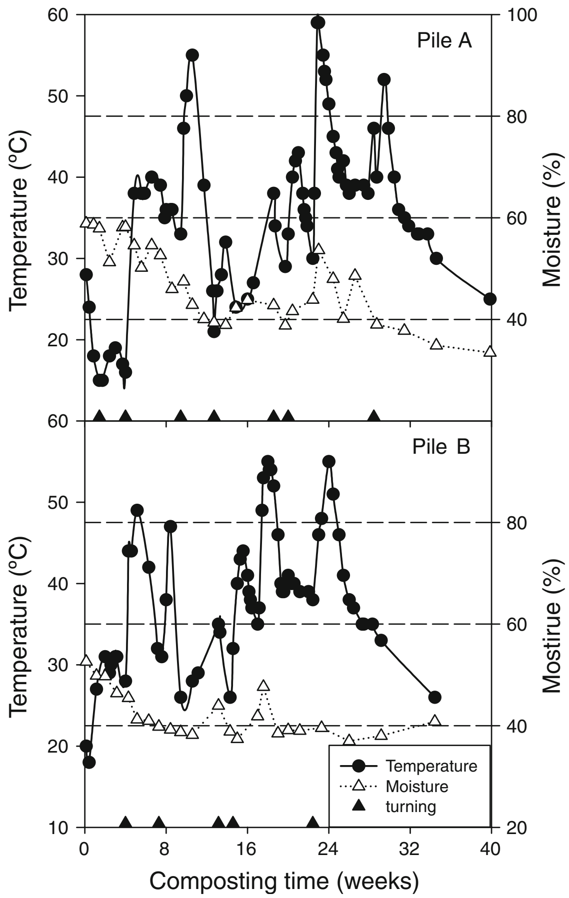

Fig. 1 Temperature and moisture evolution of pile A (two-phase olive mill waste + sheep litter) and B (two-phase olive mill waste + sheep litter + grape stalks) during composting. *Triangles* in the *x*-axis indicate the turning time of the piles

TOC concentrations. Phytotoxicity, measured by germination index (GI), was assayed by the *Lepidium sativum* test (Zucconi et al. 1981). NH_4^+-N was extracted with 2 M KCl from the frozen subsamples and determined by a colorimetric method based on Berthelot's reaction (Sommer et al. 1992), adding sodium citrate to complex divalent cations. NO_3^-, SO_4^{2-}, Cl^- and $H_2PO_4^-$ were determined by HPLC ion chromatography in a (1:20 w/v) water extract. Water soluble carbon (WSC) and water-soluble nitrogen (WSN) in the leachates were measured with a TOC analyzer. Extractable carbon (Cext) was measured in a 0.1 M NaOH extraction (20:1 w/v) and the fulvic acid carbon (FAC) after precipitation of the humic acid at pH 2 in the supernatant solution. The humic acid carbon content (HAC) was calculated by subtracting FAC to the Cext. Cation exchange capacity (CEC) was determined with $BaCl_2$-triethanolamine (Roig et al. 1988). Lignin and cellulose were determined by the American National Standards Methods (ANSI/ASTM 1977). Macro and micronutrients and heavy metals were analysed after HNO_3/$HClO_4$ digestion by atomic absorption spectroscopy. P was measured spectrophotometrically as ammonium molybdovanadate phosphoric acid. Boron was determined in dry digestion by spectrophotometry with azometine-H. Faecal coliforms counts were determined in a 1:4 (w:v) quarter-strength Ringer's solution suspension, by the most probable number (MPN) method (APHA 1985).

Physical properties

Selected physical properties were determined in the final compost. The coarseness index, expressed as weight percentage of particles with diameter >1 mm (Richards et al. 1986), and shrinkage, estimated as volume lost by the medium after drying at 105°C (Martinez 1992). The granulometry of compost was determined using the sieving tray analysis method (Martinez 1992). The air–water relationships and the rest of physical properties were measured according to de Boodt et al. (1974).

Results and discussion

General evolution of the composting process

The composting process lasted for 40 weeks in pile A and 35 weeks in pile B. Such long composting periods have been previously reported for this kind of material and are due, on the one hand, to the presence of fats and phenols with recognized antimicrobial properties in TPOMW, which delays the beginning of the process and, on the other hand, to the high concentration of lignin, which needs specific enzymes and high temperatures to be degraded (Baeta-Hall et al. 2005; Alburquerque et al. 2006; Cayuela et al. 2006). The pattern of temperature was not modified by the addition of grape stalks (Fig. 1). High temperatures (over 70°C) are common during TPOMW composting (Cayuela et al. 2005). However, the aeration method used in this study (Rutgers system) prevented temperature to increase above 60°C since continuous air blowing was activated over

55°C (Fig. 1). In both piles successive turnings were necessary to reactivate the process and increase temperatures, but the addition of GS in pile B decreased the number of turnings needed from seven (in pile A) to five (in pile B). The temperatures reached in both piles and the various turnings performed during composting guaranteed the sanitisation of the mixtures, as can be inferred from the faecal coliforms counts (Table 3).

Table 1 shows the evolution of some selected chemical properties during composting. The addition of grape stalks did not modify the TOC/TN dynamics, being this one of the most extensively used indices for establishing compost maturity. pH significantly increased in both piles during the thermophilic phase as previously reported for this kind of material. Such high pH increase is related to the decarboxilation of organic anions during the aerobic decomposition of TPOMW and the stabilisation of pH may be used as a stability index during TPOMW composting (Cayuela et al. 2008b).

The biochemical transformation of organic matter by microorganisms during composting occurs mainly in the water-soluble phase and the study of changes in this fraction can be relevant for assessing stability and maturity (Said-Pullicino et al. 2007). Chanyasak and Kubota (1981) proposed the ratio C/N in aqueous extract (WSC/WSN) as a proper method for determining maturity. However, the concentration of WSN is so low in some composting materials, that there are often analytical problems for its determination. Another option, that we propose in this study, is the measurement of the C/N ratio in the leachates of the compost. In the leachates a natural concentrated compost extract is obtained and, although the total concentration may change depending on the water drenched, the nutrients ratio follow a specific pattern. Figure 2 shows the evolution of WSC/WSN in the leachates of both piles. The first leachates were produced after 6 weeks in pile A and 2 weeks in pile B. Both showed a similar initial WSC/WSN ratio around 27. However, the WSC/WSN in the leachates was a parameter able to discriminate between the composting piles, showing a faster decrease and therefore a faster composting process in pile B. The measurement of the total volume of leachates along the whole composting period showed that addition of grape stalks in pile B decreased the total amount of lixiviates, since even though both piles were irrigated with the same volume of water, the total volume of lixiviates was 58 l in pile A and 33 l in pile B.

A faster decrease in phytotoxicity was also observed in pile B, with a GI of 60% after 1 month. Low ammonium concentrations were monitored throughout the composting time, which is in agreement with the findings of previous works for this kind of material (Alburquerque et al. 2006). Nitrification was facilitated in pile B probably due to the better

Table 1 Evolution of selected chemical characteristics during composting of two-phase olive mill wastes with sheep litter (pile A) and two-phase olive mill waste with sheep litter and grape stalks (pile B)

Sample	TOC/TN	pH	GI (%)	WSC (%)	NH_4^+ (mg kg^{-1})	NO_3^- (mg kg^{-1})	CEC (mEq 100 g^{-1})
A-I	25.7^a	7.13^a	9^a	7.51^a	30^a	n.d	47.2^a
A-M	24.5^a	7.09^a	0^a	6.87^b	34^a	n.d.	50.7^a
A-T1	18.9^b	9.02^b	–	6.17^c	78^b	n.d.	69.1^b
A-T2	17.6^c	9.31^c	61^b	5.78^c	133^d	n.d.	69.2^b
A-F	15.5^d	9.50^d	74^c	3.35^d	105^c	<10	72.6^b
B-I	22.9^B	6.93^A	5.1^A	5.58^A	193^c	n.d.	60.7^A
B-M	28.6^A	7.70^B	60.5^B	5.17^B	68^A	n.d.	61.5^A
B-T1	16.5^C	9.46^C	–	2.71^D	73^A	n.d.	70.1^B
B-T2	15.9^C	9.51^C	97.9^C	3.12^C	67^A	n.d.	82.2^C
B-F	12.7^D	9.44^C	87.8^C	2.88^D	129^B	387	85.3^D

Results are the mean of three replicates. For each pile, values in the same column followed by the same letter are not significantly different according to Waller–Duncan test ($P < 0.001$)

TOC total organic carbon, *TN* total nitrogen, *GI* germination index, *WSC* water soluble carbon, *CEC* cation exchange capacity

Composting piles A and B were independently statistically analysed. Small letters show differences in pile A and capital letters in pile B

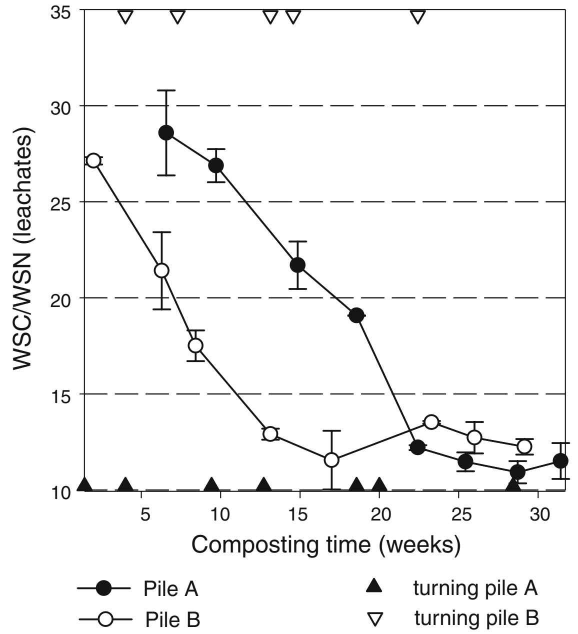

Fig. 2 Evolution of C/N ratio in the leachates during the composting of two-phase olive mill waste and sheep litter without grape stalks (*pile A*) and with grape stalks (*pile B*). *Vertical bars* represent standard deviations. *Triangles* in *x*-axis show when turnings were made

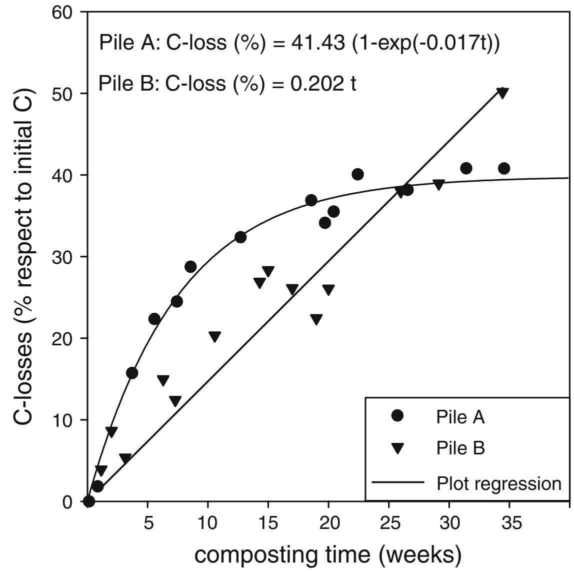

Fig. 3 Carbon mineralization dynamics during composting of two-phase olive mill wastes with sheep litter (*pile A*) and two-phase olive mill waste with sheep litter and grape stalks (*pile B*)

structure that allowed oxygenation during the maturation period (Sánchez-Monedero et al. 2001). Grape stalks significantly increased the cation exchange capacity of the compost.

Carbon mineralization dynamics

Carbon losses during composting are directly related to microbial respiration and organic matter degradation. Figure 3 shows the percentage of carbon losses respect to initial C throughout the composting process for both piles. The addition of GS notably modified the C mineralization dynamics. Thus, the organic matter degradation profile followed a first-order kinetic equation in pile A, and a zero-order kinetic equation in pile B. The incorporation of grape stalks slowed down the mineralization of organic matter during the first stage and this is probably because the bulking agent had a higher particle size, reducing the surface available for micro organisms. Also Paredes et al. (2002) found a zero-order kinetic mineralization pattern when maize straw was added as bulking agent during olive mill wastewater sludge composting (Fig. 4).

Evolution of the humification indices

We used four indices widely used in literature to follow the humification process during composting: the humification ratio (C_{ext}/TOC × 100), the humification index (HAC/TOC × 100), the percentage of humic acids (HAC/C_{ext} × 100) and the polymerisation degree (HAC/FAC) (Iglesias-Jimenez and Perez-Garcia, 1992) (Table 2). Compared to compost from other origins, TPOMW composts show very high concentration of humic-like substances. In addition, the analysis of humic fractions showed that humification reactions were remarkably favoured by the addition of GS. Thus, in pile B the humification indices show a high maturation degree already at T1 stage (after 126 days), while in pile A this level of humification was reached only at the end of the process (after 279 days). This fact can be related also with the mineralization dynamics showed in Fig. 2. The grape stalks favoured humification reactions against mineralization of organic matter at the beginning of the process.

Final compost quality

Table 3 shows the main chemical properties of the final composts obtained. Both composts satisfied the

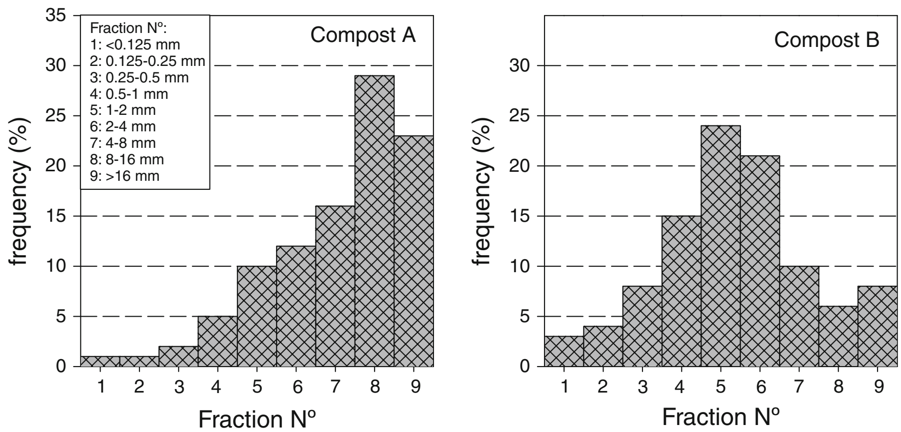

Fig. 4 Histograms of the different particle size fractions in composts A (two-phase olive mill wastes with sheep litter) and compost B (two-phase olive mill wastes with sheep litter and grape stalks)

Table 2 Analysis of the humic fractions and maturation indices based on humification during composting of two-phase olive mill wastes with sheep litter (pile A) and two-phase olive mill waste with sheep litter and grape stalks (pile B)

Composting time	Humic fractions (%)				Humification indices		
	C_{ext}	HAC	FAC	C_{ext}/TOC	HAC/TOC	HAC/C_{ext}	HAC/FAC
Pile A							
I	11.3^{a}	3.0	8.3^{a}	29.8	7.9	26.5	0.36
M	11.2^{a}	3.8	7.5^{b}	30.8	10.3	33.5	0.50
T1	8.2^{b}	2.3	5.9^{c}	25.3	7.0	27.7	0.38
T2	8.1^{b}	5.6	2.5^{d}	24.2	16.6	68.8	2.20
F	7.5^{c}	5.5	2.0^{d}	24.8	18.1	73.1	2.71
ANOVA	***		***				
Pile B							
I	9.5^{A}	3.2	6.3^{A}	31.7	10.7	33.8	0.5
M	7.8^{B}	2.8	5.0^{B}	22.8	8.3	36.3	0.57
T1	6.9^{C}	5.0	1.9^{C}	25.1	18.2	72.3	2.61
T2	6.9^{C}	5.1	1.8^{D}	22.7	16.7	73.5	2.77
F	6.7^{C}	5.2	1.6^{E}	28.2	21.7	76.8	3.32
ANOVA	***		***				

Results are the mean of three replicates. For each pile, values in the same column followed by the same letter are not significantly different according to Waller–Duncan test ($P < 0.001$)

ANOVA test: $***P < 0.001$

thresholds established in literature for common maturation indices such as TOC/TN ratio, lignin/cellulose ratio or GI. However, compost B always showed improved values such as lower phytotoxicity and higher humification degree (Table 3). Regarding chemical composition, the incorporation of grape stalks induced some important changes, among which, a lower electrical conductivity and a higher concentration of Fe, Mn and B. The concentration of K was, on the other hand, diminished. Heavy metals were below the limits established by the Spanish legislation in both piles, except for Ni, that slightly exceeded the maximum concentration in pile A.

The final composts significantly differed in their physical properties. Figure 4 shows the histogram of frequencies of the composts different particle sizes. In compost A, the most abundant fraction was between 8 and 16 mm, whereas in compost B it was between 1 and 2 mm. In general, both composts exhibited physical properties in the optimum range for the ideal substrate (Table 4). Coarseness index was very high, but pile A clearly showed greater size of particles with

Table 3 Main chemical characteristics of composts obtained from two-phase olive mill wastes with sheep litter (compost A) and two-phase olive mill waste with sheep litter and grape stalks (compost B)

Chemical properties	Threshold values	Compost A	Compost B
TOC/TN	15[a]	15.5	12.7
Cellulose/lignin	<0.5[b]	0.47	0.39
CEC/TOC	>1.7[c]	2.4	3.6
GI (%)	>50[d]	74.2	87.8
Faecal coliforms (MPN g^{-1})	<1000[e]	21.68	<0.08
pH	6–9	9.50	9.44
EC (dS m^{-1})	–	7.31	4.98
K_2O (%)	–	4.7	3.8
P_2O_5 (%)	–	4.7	4.2
Cl^- (g kg^{-1})	–	13.1	6.1
SO_4^{2-}(g kg^{-1})	–	5.13	4.25
$H_2PO_4^-$(g kg^{-1})	–	1.00	1.06
Fe (g kg^{-1})	–	424	684
Cu (mg kg^{-1})	450[f]	31	36
Mn (mg kg^{-1})	–	104	131
Zn (mg kg^{-1})	1100[f]	72	86
B (mg kg^{-1})	–	198	230
Ni (mg kg^{-1})	120[f]	133	81
Cr (mg kg^{-1})	270[f]	58	75
Pb (mg kg^{-1})	150[f]	<5	<5
Cd (mg kg^{-1})	3[f]	<0.5	<0.5

[a, c] Roig et al. 1988; [b] Komilis and Ham 2003; [d] Zucconi et al. 1981; [e] BNQ Standards Canada 1995; [f] Spanish law of fertilisers 2005

Table 4 Main physical properties of composts obtained from two-phase olive mill wastes with sheep litter (compost A) and two-phase olive mill waste with sheep litter and grape stalks (compost B)

Physical properties	Optimum level[a]	Compost A	Compost B
Particle size (mm)	0.25–2.0	–	–
Most abundant fraction (mm)	–	8–16	1–2
Coarseness Index (CI) (%)	–	90	69
Bulk density (BD) (g cm^{-3})	<0.4	0.349	0.435
Real density (RD) (g cm^{-3})	1.45–2.65	1.796	1.822
Total pore space (TPS) (% vol.)	>85	80.6	76.2
Air capacity (AC) (% vol.)	20–30	25.8	18.0
Total water holding capacity (TWHC) (% vol.)	55–70	54.7	62.3
Shrinkage (% vol.)	<30	11.1	8.4

[a] Abad et al. 2001

more than 50% larger than 8 mm. This elevated particle size is due to the physical consistency of TPOMW that tends to form aggregates. The incorporation of GS improved the structure preventing the aggregation. Both composts exhibited similar real density; however, compost A showed better aeration properties (higher total pore space and aeration capacity) while pile B displayed better hydrological properties (higher water holding capacity and lower shrinkage), which is in accordance with the lower quantity of leachates obtained in pile B.

Conclusions

The composting of TPOMW is a long-lasting process compared to other organic residues. However, the addition of grape stalks to a 50:50 TPOMW:SL composting pile improved and accelerated the composting process by:

- Decreasing the composting time in at least 5 weeks, which was demonstrated by comparing several maturation and stability indices.

- Speeding the removal of phytotoxic compounds and the formation of humic substances.
- Improving structure and decreasing the number of necessary turnings.
- Decreasing the total amount of lixiviates in more than 40%.

On the other hand, grape stalks addition enhanced the quality of the end product by:

- Providing higher concentration of humic-like substances, cation exchange capacity and lower phytotoxicity.
- Improving the hydrological physical properties by yielding higher water holding capacity and lower shrinkage.

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