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# **Co-composting of olive husks with high moisture contents: organic matter dynamics and compost quality.**

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## **Abstract**

As the adoption of new generation, two-phase olive oil extraction systems becomes more widespread, there is an increasing need to evaluate the feasibility of co-composting processes to treat the resulting olive husk by-products that often present particularly elevated moisture contents with respect to traditional two-phase systems. The present work compares process parameters, organic matter dynamics and compost quality during the co-composting of two olive husks having different initial moisture contents, with the adoption of agricultural organic residues as bulking/absorbent agents and forced aeration. Despite the elevated moisture content, the co-composting conditions adopted were effective in reducing the elevated contents of water soluble organic matter, phenolic and lipidic compounds as well as reducing the extreme phytotoxicity of the experimental husk. Organic matter evolution during composting showed that the stabilization process in the two husks was comparable, and final compost quality was adequate in both cases.

## **Keywords**

Olive-mill wastes, olive husks, organic matter degradation, water extractable organic matter, phytotoxicity.

## 1. Introduction

The disposal of olive mill wastes represents a problem of environmental relevance particularly in Mediterranean countries responsible for about 99% of the annual worldwide production of olive oil (Roig et al. 2006). In this area Spain, Italy, Greece, Portugal, Tunisia, Turkey and Syria are the main producers, with the first four alone representing about 75% of the world production (Baeta-Hall et al., 2005). Two-phase olive oil extraction technologies that deliver a single byproduct in the form of moist olive husks are becoming more wide spread with respect to three-phase systems that also produce relatively large volumes of waste waters and therefore considered to have a higher environmental impact (Alfano et al. 2008). However, the high moisture contents of two-phase husks reduces their economical value, and their disposal has become a new problem for the oil extraction industry. Moreover, two-phase husks are often rich in potentially phytotoxic and bacteriostatic substances (e.g. polyphenols and organic acids), and elevated salt concentrations that may cause negative impacts on crops and environment when the husks are directly used as fertilizers.

In recent years several methods have been proposed for the utilization of the husks produced by two-phase systems (Roig et al. 2006) through their direct application as soil amendment, combustion for energy production or anaerobic digestion for the production of biogas. Even if it was found that raw husks may have a beneficial effect on crop yield due to the high content of some plant nutrients (Lopez-Pinero et al. 2006), they often show high content of toxic compounds for plants and soil micro-organisms. For this reason a number of studies were carried out to find adequate treatment strategies that favour the degradation of these toxic compounds. Among them, composting is one of the possible and most promising technologies to obtain a stable and mature soil amender.

The main problem encountered during the composting of husks is that their semisolid consistency often leads to a reduced porosity and compact texture. As a consequence, the addition of bulking materials is required to favour oxygen diffusion during the process (Albuquerque et al. 2006; Cayuela et al. 2006; Cayuela et al. 2010). Moreover, the composting of these materials is becoming even more challenging, as new generation oil extraction systems that enable the recovery of the crushed olive stones present within the husks for energy production, are further increasing their moisture contents. As, in the near future a worldwide diffusion of this typology of olive mill technology will probably occur, identifying the best technology for the treatment of these water-saturated husks, taking in consideration their consistency, the absence of the olive stones and their high phytotoxicity, is highly necessitated.

Based on these considerations the aim of the present work was to evaluate (i) the feasibility of co-composting of olive-mill husks obtained from the new generation olive mills, (ii) compost quality and organic matter evolution during the composting of these husks in experimental feedstocks and comparison to a reference mixture involving the traditional olive-mill husks. In order to achieve these objectives, composting of both experimental and traditional husks was carried out by means of a laboratory scale composter and process parameters (moisture, temperature, volatile solids and C/N ratio) monitored. To confirm the effectiveness of composting in the degradation of phytotoxic compounds, water extractable organic carbon and soluble phenols were measured throughout the process while a seed bioassay was used to evaluate changes in matrix phytotoxicity with time. Moreover the

evolution of compounds particularly abundant in olive husks, such as lipids and polyphenols, were monitored.

## 2. Materials and methods

### 2.1 Composting procedure and sampling

The fresh olive oil husk was collected from a new generation two-phase centrifugation olive mill (<sup>®</sup>DMF – Peralisi) in which the raw byproduct (ExH) was composed of olive pulp and endogenous olive water and had a moisture content of 78.1%. The reference husk (TrH) was collected from a traditional two-phase plant with a moisture content of 47.1%.

Various preliminary trials using ExH and TrH were carried out (data not shown) in order to identify the ideal feedstock mixture in terms of the maximum proportion between husk and absorbent/bulking material to avoid the leaching of free water and provide adequate porosity. Based on these preliminary trials, two feedstocks were prepared according to the following proportions: Mixture A, composed of TrH 80% + olive tree pruning 20% (w/w); Mixture B, composed of ExH 70% + olive tree pruning 22% + cereal straw 8% (w/w/w). Table 1 reports the main characteristics of all the constituents of the two feedstocks.

About 200 L of each mixture were composted in an experimental laboratory composter that consists of a 240 L steel cylinder. Air was pumped from the bottom of the cylinder by connecting an air compressor to a perforated PVC tube. A constant air flow ( $35 \text{ L h}^{-1} \text{ kg}^{-1}$  dry weight) during thermophilic phase was maintained for both the mixtures. When the temperature was  $> 55^\circ\text{C}$  the air flow was automatically increased until temperature was  $< 55^\circ\text{C}$ . Temperature was monitored every minute by a probe fixed in the middle of the composting mass. Twice a week the moisture of the mixture was also measured and water addition was made when the water content was below 50% in order to maintain the moisture between 50% and 60%. The thermophilic phase was considered concluded when the temperature was stable at  $45^\circ\text{C}$ . At the end of the thermophilic phase the material was placed in an open container without forced air until the curing phase was completed (126 days). During both thermophilic and curing phases, samples were collected at 0, 7, 19, 47 and 126 days. Once in the laboratory, collected samples were divided into two portions. One portion was frozen while the other was freeze-dried, crushed to pass through a 0.5 mm sieve and thoroughly mixed.

### 2.2 Analytical methods

Moisture content was determined by weight loss upon drying at  $105^\circ\text{C}$  in an oven for 24 h. Electrical conductivity and pH were determined in a 1:10 compost:water extract ratio (ANPA, 2001). Total volatile solids (VS) were determined by weight loss upon ashing at  $550^\circ\text{C}$  for 24 h in a muffle furnace. Total organic carbon (TOC) was determined by an elemental analyser (EA 1110 Carlo Erba, Milan, Italy; ANPA, 2001). Organic matter (OM) loss during composting was calculated both in terms of OM loss and C loss. OM loss was calculated according to the equation described by Viel et al. (1987) and recently used also by Altieri et al. (2011) to describe OM loss during composting of olive mill waste:

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

where  $X_1$  and  $X_2$  represent the initial ash content and the ash content at a time  $t$ , respectively. TOC concentration was corrected using the following equation:

$$\text{TOC}_{(\text{corrected})} (\text{g kg}^{-1}) = \text{TOC} (X_1/X_2)$$

where  $X_1$  and  $X_2$  represent the initial ash content and the ash content at a time  $t$ , respectively. Fresh samples were used for determination of total Kjeldahl-N (TKN) and  $\text{NH}_4^+$ -N by means of macro and micro-Kjeldahl distillation methods, respectively (ANPA, 2001). Total organic N was calculated by the difference between TKN and  $\text{NH}_4^+$ -N.

Water-extractable organic matter (WEOM) and its hydrophilic (Hi) and hydrophobic (Ho) fractions were obtained, and C content measured, as described in Said-Pullicino et al. (2007). Water-extractable organic carbon (WEOC) data were corrected by using the same equation previously reported for TOC correction. WEOM extracts were also analysed for water-soluble phenols with a modified version of the Folin-Ciocalteu method (Box 1983) as described by Said-Pullicino and Gigliotti (2007). The concentration of polyphenols was determined by extraction with methanol at room temperature three times for 30 minutes each. After solvent evaporation and re-solubilisation in water, phenolic compounds were determined by a modified Folin-Ciocalteu method as described by Montedoro et al. (1992). Concentrations were calculated against a calibration curve prepared with six different concentrations of gallic acid (from 5 to 90 mg L<sup>-1</sup>) and expressed in mg g<sup>-1</sup> gallic acid equivalents. The total lipid content was determined after extraction with hexane at 60°C three times for 15 minutes each followed by another extraction overnight. After solvent evaporation, total lipid content was determined gravimetrically (Greenberg et al. 1998). Compost maturity was estimated by means of a *Lepidium sativum* L. seed germination bioassay as proposed by Zucconi et al. (1985) and modified by Said-Pullicino et al. (2007). In detail, deionised water was added to the fresh compost samples to attain a moisture content equivalent to 85% (wet weight) and after a contact period of 2 h, the aqueous extracts were obtained by centrifugation and filtration through a 0.45 µm membrane filter. The concentrated extracts together with three dilutions (25%, 50% and 75% in deionised water) were used as germination media. A Whatman filter paper (Number 42) placed inside a 9 cm diameter, sterilized, disposable Petri dish was wetted with 1 mL of each germination solution and 10 *Lepidium sativum* L. seeds were placed on the paper. Deionised water was used as a control and five replicates were set out for each treatment. The Petri dishes were placed in sealed plastic bags to minimise water loss while allowing air penetration, and were then kept in the dark for 2 d at 20 °C. After the incubation period the number of germinated seeds and the primary root length were measured and expressed as a percentage of the control (germination index). Data were analyzed statistically against compost age and concentration of media.

In order to characterise the final compost obtained, total P, total K and total Cd, Cu, Hg, Ni, Pb and Zn contents were determined on the mature composts and compared with the limits defined by the Italian legislation for municipal or green waste compost. Total P was measured spectrophotometrically after digestion of the samples with concentrated H<sub>2</sub>SO<sub>4</sub>/HClO<sub>4</sub> (ANPA, 2001). Total K was analysed by flame atomic emission spectroscopy, whereas total Cd, Cu, Ni, Pb and Zn were analysed by flame atomic absorption spectroscopy (AA 6800, Shimadzu Corp., Tokyo, Japan) both after sample digestion with concentrated HNO<sub>3</sub>/HClO<sub>4</sub> (ANPA, 2001). In the same extract, Hg concentration was determined by atomic absorption spectroscopy with hydride generation (ANPA, 2001).

All chemical analyses were carried out in triplicate and the standard error (SE) was calculated.

### 3. Results and discussion

#### 3.1 Temperature, moisture, pH and EC

The temperature profiles during the active phase for the composting of both mixtures (A and B) are reported in Figure 1. Peak temperatures were obtained after 8 days for the mixture A and 7 days for the mixture B. The rapid establishment of thermophilic conditions due to the self-heating of organic matter as a result of microbial respiration are indicative of an adequate feedstock composition capable of supporting aerobic microbial degradation. In both mixtures, matrix temperatures were higher than 50°C for at least 10 days and higher than 45°C for the whole duration of the active phase. This long duration of the active phase was probably due to the hydrolysis of semi-recalcitrant molecules that, transforming in simpler moieties, are available for the thermophilic microorganisms that sustain microbial activity throughout the active phase. During the curing phase the temperature in both mixtures decreased gradually reaching room temperature after about 95 days of process (data not shown).

Moisture content decreased from 42.3 to 28.3% for mixture A and from 62.6 to 38.4% for mixture B (Figure 1). Most of the reduction occurred during the curing phase since water addition to mixture A during the active phase maintained water content between 50% and 60%. Moisture content for mixture B was found to be higher than 60% for most of the active phase as a consequence of the higher water content of the feedstock. For both mixture A and B, the moisture content at the end of the curing phase allowed efficient material handling thereafter.

Figure 2 shows pH and electrical conductivity profiles of the aqueous extract of the composting mixtures with time. The initial pH values for mixture A and B were clearly influenced by the acidic nature of the husks used in the experiment (Table 1). During the early stages of composting low pH values are typically due to anaerobic conditions that are established in the waste materials prior to the commencement of the composting process, resulting in the formation of low molecular weight fatty acids (Said-Pullicino et al. 2007). Moreover, these particular feedstock materials are known to have an acidic pH as a result of the elevated presence of phenolic compounds (Paredes et al. 1999). As aerobic conditions are provided through forced aeration of the composting mass, an increase in pH is observed as organic acids are degraded or phenolic compounds are oxidised. During the process, the mineralisation of proteins, amino acids and peptides generally leads to the release of ammonium or volatile ammonia that also contribute to an increase in pH. Our results confirm these findings; in fact the pH evolution reflects the behavior of N: increasing N concentration in the samples during composting coincided with an increase in pH values. At the end of the curing phase pH values were 7.4 and 8.1 for mixtures A and B respectively, also reflecting the N content of the mixture (17.6 and 28.5 g kg<sup>-1</sup> for mixture A and B, respectively). These pH values were compatible with the agricultural use of compost

Soluble salts as estimated by electrical conductivity (Figure 2), were higher for mixture B as a consequence of the higher soluble salts content in the experimental husk with respect to the traditional one. Throughout the composting process values of electrical conductivity were in the range of 0.3–1.6 dS m<sup>-1</sup> for mixture A and 1.5–3.3 dS m<sup>-1</sup> for mixture B. In both mixtures the highest values were reached at the end of the process, when the decrease in the total mass, attributable to the mineralisation of organic matter, causes a relative concentration

in soluble salts.

### *3.2 Organic matter degradation*

During composting, significant OM loss occurred in both mixtures resulting in a decrease in VS and TOC throughout the process, but particularly during the curing phase (Table 2). In the two mixtures OM loss was very similar and, at the end of the process, was 47.1% and 48.0% for mixtures A and B, respectively. These values are close to the range of 55-68% loss normally reported for the composting of sewage sludge, animal manure and olive mill waste with different lignocellulosic materials used as bulking agents (Garcia-Gomez et al. 2003). The slightly lower OM loss observed may be due to the presence of relatively stable organic compounds in our husks probably represented by lipids, polyphenols and pectin.

When olive oil industry wastes are processed the degradation of fats is necessitated due to their potentially high content in the husk. At the beginning of the process fat content in mixture B was higher with respect to mixture A (Table 3). In both mixtures their degradation was initially very slow probably due to the selective degradation of more labile substances present in the feedstock. At the end of the active phase about 80% of lipids were degraded in both mixtures, but in mixture B their concentration was still 3 fold with respect to mixture A. At the end of the curing phase the concentration of lipids was the same in both mixtures with a percentage degradation of 94 and 97% for mixtures A and B, respectively. These results confirm the efficiency of the stabilization process since these compounds are adequately degraded. Similar results were reported by Cayuela et al. (2006) who found a 90% reduction in lipids during the composting of two-phase olive mill waste under aerated conditions.

Another class of compounds generally present in the husks are polyphenols. These compounds are considered to be of particular concern due to their phytotoxic effect. In the fresh feedstock total phenol concentration in mixture B was double that in mixture A (Table 3). In both mixtures the degradation of total phenolic compounds started immediately and after the first 7 days the percentage of degradation was of 55% and 44% for mixture A and B, respectively. This decrease in total phenol concentrations continues during the process, and at the end of the active phase the percentage of degradation was 69% and 76% for mixtures A and B, respectively. This behaviour confirms data from Altieri et al. (2011) who reported a reduction in polyphenols concentration during the static composting of olive mill waste by about 80% after 120 days of composting. In contrast, during the curing phase the concentration of total phenolic compounds tended to increase probably due to a concentration effect of these compounds.

### *3.3 Nitrogen evolution and C/N ratio*

The changes in TKN of the composting mixtures with time are reported in Table 4. In both mixtures data were strongly related to the variations in organic N which was the major nitrogenous constituent. This was particularly true for the mixture B where  $\text{NH}_4^+-\text{N}$  concentration was very low when compared to organic N forms (2.2 and 5.3% of mineral N to TKN at the beginning and at the end of the process, respectively). In mixture A the proportion of TKN as  $\text{NH}_4^+-\text{N}$  was 27.2% and 4.5% at the beginning and end of the process, respectively. The loss of ammonia in the mixture A may be attributed both to the volatilisation at relatively high pH and temperature, nitrification process and immobilization of N by



microbial assimilation. The lack of increase in  $\text{NH}_4^+\text{-N}$  towards the beginning of the process was expected for substrates rich in cellulose, hemicelluloses and lignin due to lower substrate degradation. In fact, a significant part of the protein contained in the fresh mixture is not ammonified at all during composting (Körner and Stegmann, 2002) and therefore the general increasing trend in organic N can be attributed to a concentration effect as a consequence of degradation of organic compounds which reduced the dry mass. Mixture A showed the highest N increase (about 2.2 fold the initial N content) probably as a consequence of its lower content of ligno-cellulosic materials.

The TOC (data not corrected for ash content) to organic N (C/N) ratio (Table 4) gives an indication of N availability for the process of biological degradation and the decrease in this ratio with composting time has been widely reported as an indicator of stability for composting processes, even if the large variation in raw materials sometimes does not allow to use this parameter as a valid indicator. In our experiment, both mixtures show a decrease in the C/N ratio. The final C/N ratio for mixtures A and B is comparable with the values obtained in the co-composting of olive mill waste as reported by Hachicha et al. (2009) and are compatible (for mixture B) or very close (for mixture A) with limit values reported in Italian law for an adequate agronomical use of compost (Table 5).

### *3.4 Water soluble organic carbon*

The variation in WEOC with composting time (Fig. 3) in both mixture A and B shows a general decrease during the active and curing phases. Mixture B had initial WEOC concentrations that were relatively high ( $83 \text{ g kg}^{-1}$  on a dry weight basis) indicating a potential phytotoxic effect of the feedstock. On the other hand, WEOC concentration in the mixture A was initially  $26 \text{ g kg}^{-1}$ , a value normally found at the start of composting of different feedstocks (Said-Pullicino et al. 2007). In both mixtures WEOC values tended to decrease with composting probably as a result of labile OM degradation in this fraction. At the end of the curing phase WEOC concentrations were of  $11 \text{ g kg}^{-1}$  and  $24 \text{ g kg}^{-1}$  for mixtures A and B, respectively. The concentration of WEOC during composting is related to the equilibrium between various reactions which increase or decrease the amount of dissolved organic matter (Said-Pullicino et al. 2007). An increase in WEOC concentrations could be due an increasing contribution of soluble microbial products (e.g. enzymes, microbially-derived sugars and cell wall constituents) or a release of soluble low-molecular weight compounds during organic matter degradation (e.g. phenols and sugars from the depolymerisation of ligno-cellulosic constituents). On the other hand, the reduction in WEOC depends on the continuous mineralisation of soluble organic compounds, and re-polymerization and condensation pathways that lead to the formation of complex organic substances that have a lower solubility in water and tend to flocculate out of solution. The contribution of each of these processes changes with composting time and, generally, at the end of the composting the balance of these processes lead to a decrease in WEOC concentration. Some authors consider that compost is sufficiently mature when WEOC concentration is below  $10 \text{ g kg}^{-1}$ , even if according to Paredes et al. (2002) this value has to be below  $7 \text{ g kg}^{-1}$ . In our experiment at the end of the process mixture A reached values of  $10 \text{ g kg}^{-1}$ , whereas mixture B had a WEOC concentration higher than  $10 \text{ g kg}^{-1}$  and this may demonstrate that mixture B did not reach

stability. But, as demonstrated by Adani et al. (2003) the composition of the initial mixture in composting highly influences the final value of WEOC in a mature compost.

The ratio between the hydrophobic to hydrophilic (Ho:Hi) fraction of WEOC has been shown to be a good index of compost stability with values  $>1$  being obtained towards the end of the composting process (Said-Pullicino et al. 2007). The fractionation of WEOC into hydrophobic and hydrophilic fractions (Fig. 3) showed that during the active phase both fractions were responsible for the decrease in WEOC. This is in contrast with the findings of Said-Pullicino et al. (2007) that attribute the decrease in WEOC during the active phase to a degradation of the more labile hydrophilic fraction. However, the presence of soluble phenolic compounds in the husks may explain the relatively high values of hydrophobic C obtained at the beginning of the process particularly for mixture B, resulting in an elevated Ho:Hi ratio. During the composting process the concentration of the hydrophilic fraction decreased 2.6 and 6.5-fold for mixtures A and B respectively, while the hydrophobic fraction decreased 2.2-fold for both mixtures. This confirms that although the major process involved in the loss of WEOC during the composting process can be attributed to the biodegradation of labile organic compounds that form part of the hydrophilic fraction, the hydrophobic fraction is also subject to significant degradation possibly as a result of the biotic or abiotic oxidation of the phenolic constituents present in this fraction. This suggests that, for these materials, the elevated concentration of hydrophobic constituents at the beginning of the composting process and their rapid degradation with composting time may conceal the changes in this fraction generally attributed to the stabilisation process. Although the faster degradation of the hydrophilic fraction with respect to the hydrophobic fraction during the active phase results in an increase in the Ho:Hi ratio, the decreasing values of WEOC as well as Ho:Hi ratio towards the end of the composting process may suggest that even after 126 d of composting, significant degradation of the hydrophobic fraction is still underway.

The relatively high values of hydrophobic C for mixture B were confirmed by the elevated concentrations of soluble phenolic compounds obtained for this mixture (Fig. 4) with respect to mixture A. Their concentration for both mixtures rapidly decreases during the first days of composting possibly as a result of their biotic degradation or abiotic oxidation. Thereafter their concentration remains constant during most of the composting process. This seems to suggest that following an initial, rapid degradation of soluble phenolic compounds, a balance between the oxidative degradation of these compounds and their release in solution as a result of the depolymerisation of insoluble phenolic constituents is established. Similar results have been reported by Said-Pullicino et al. (2007) for the composting of urban refuses.

### *3.5 Compost phytotoxicity*

Results for the seed bioassays used to evaluate changes in compost phytotoxicity at the beginning of the composting process, at the end of the active phase and at the end of the curing phase, are reported in Figure 4. The germination index obtained for mixture B at the beginning confirms a very high phytotoxicity of the experimental husk used. During the stabilisation process a clear decrease in the phytotoxicity with composting time occurred demonstrating the efficiency of the composting process even with problematic matrices such as those used in this work. In a recent bioassay experiment, Del Buono et al. (2011) also showed that the elevated phytotoxicity of these husks was significantly reduced after aerobic

treatment. During the active phase the GI increased from 0.6 to 61% and then, during the curing phase, this value reached 66%, exceeding the value of 60% that indicates an appropriate reduction in compost phytotoxicity (Zucconi et al. 1985). The very high phytotoxicity of mixture B coincides with a high concentration of water soluble organic substances and toxic constituents such as organic fatty acids (evident from the initial very low pH value) and phenolic compounds. These factors are considered the most important in determining phytotoxicity with the lack of oxygen due to high microbial activity, the accumulation of other toxic compounds such as alcohols, ammonia and toxic nitrogen compounds, the immobilisation of nitrogen with high C:N ratios, and the presence of heavy metals and mineral salts (Said-Pullicino et al. 2007).

In mixture A changes in the GI are not evident, as a consequence of the relatively high GI of the initial feedstock (about 80%). The higher initial GI value in the mixture A with respect to mixture B is justified by the lower EC, Hi-WEOC and soluble phenols content.

### *3.6 Final compost quality*

Table 5 shows the main chemical characteristics of the composts obtained by mixtures A and B. All the chemical parameters established by the Italian law for compost commercialisation were satisfied by the final composts produced. Since the composition of the two husks ExH and TrH was very different, the final chemical composition of the resulting composts also differed significantly. Even though composting greatly improved all chemical parameters of mixture B, some characteristics of this compost are worse with respect to compost A. In particular the moisture and EC were higher in compost B, whereas the humic-like substances content was lower in this compost with respect to compost A. Both composts showed a very low concentration in heavy metals; this result was expected as the feedstocks used in this experiment are generally poor in contaminant elements such as heavy metals.

## **4. Conclusions**

The composting of the experimental husk used in this work was achieved in about 4 months, despite the particular chemical and physico-chemical characteristics of this olive oil waste. The phytotoxicity of the initial mixture decreased during composting and, at the end of the process, showed GI values compatible with the values generally indicated for mature compost and not significantly different respect to the compost obtained with the traditional two-phase husk. The changes in phytotoxicity are attributable to the degradation of toxic compounds, principally contained in the water soluble organic matter fraction. The two composts obtained showed all parameters within the limits defined by Italian law for these products. Moreover, despite the compact texture, high moisture content and elevated concentrations of potentially toxic compounds in the experimental husk, the compost obtained is a fertiliser with a high quality both from an environmental and an agronomical point of view. These results obtained in an experimental composter may be replicated in real scale plants adjusting the forced aeration according to the available equipment.

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## References

- Albuquerque, J.A., González, J., García, D., Cegarra, J., 2006. Effects of bulking agent on the composting of “alperujo”, the solid by-product of two-phase centrifugation method for olive oil extraction. *Process Biochemistry* 41, 127-132.
- Alfano, G., Belli, C., Lustrato, G., Ranalli, G., 2008. Pile composting of two-phase centrifuged olive husk residues: Technical solutions and quality of cured compost. *Bioresource Technology* 99, 4694-4701.
- Altieri, R., Esposito, A., Nair, T., 2011. Novel static composting method for bioremediation of olive mill waste. *International Biodeterioration and Biodegradation* 65, 786-789.
- ANPA (Agenzia nazionale per la protezione dell’Ambiente) 2001. *Metodi di analisi del compost. Manuali e linee guida* 3, 2001.
- Baeta-Hall, L., Sàagua, C M., Bartolomeu, M.L., Anselmo, A.M., Rosa, M.F. 2005. Biodegradation of olive oil husks in composting aerated piles. *Bioresource Technology* 96, 69-78.
- Box, J.D., 1983. Investigation of the Folin-Ciocalteu phenol reagent for the determination of polyphenolic substances in natural waters. *Water Resources* 17, 511-522.
- Cayuela, M.L., Sánchez-Monedero, M.A., Roig, A., 2006. Evaluation of two different aeration systems for composting two-phase olive mill wastes. *Process Biochemistry* 41, 616-623.
- Cayuela, M.L., Sánchez-Monedero, M.A., Roig, A., 2010. Two-phase olive mill waste composting: enhancement of the composting rate and compost quality by grape stalks addition. *Biodegradation* 21, 465-473.
- Del Buono, D., Said-Pullicino, D., Proietti, P., Nasini L., Gigliotti, G., 2011. Utilization of olive husks as plant growing substrates: phytotoxicity and plant biochemical responses. *Compost Science & Utilization* 19, 52-60.
- García-Gomez, A., Roig, A., Bernal, M.P., 2003. Composting of the solid fraction of olive mill wastewater with olive leaves: organic matter degradation and biological activity. *Bioresource Technology* 86, 59-64.
- Greenberg, A.E., Clesceri, L.S., Eaton A.D., 1998.. *Standard methods for the examination of water and wastewater*, 20th ed. American Public Health Association, Washington, USA.
- Hachicha, S., Cegarra, J., Sellami, F., Hachicha, R., Drira, N., Medhioub, K., Ammar, E., 2009. Elimination of polyphenols toxicity from olive mill wastewater sludge by its co-composting with sesame bark. *Journal of Hazardous Materials* 161, 1131-1139.
- Körner, I., Stegmann, R., 2002. N-dynamics during composting –overview and experimental results. In: Insam, H., Riddech, N., Klammer, S. (eds.): *Microbiology of Composting*, Springer-Verlag, Berlin, Germany, pp. 143–154.
- López-Piñero, A., Fernandez, J., Rato Nuñez, J.M., García-Navarro, A., 2006. Response of soil and wheat crop to the application of two-phase olive mill waste to Mediterranean agricultural soils. *Soil Science* 171, 728-736.
- Montedoro, G., Servili, M., Baldioli, M., Miniati, E., 1992. Simple and hydrolyzable phenolic

- compounds in virgin olive oil. 1. Their extraction, separation, and quantitative and semiquantitative evaluation by HPLC. *Journal Agricultural Food Chemistry* 40 (9), 1571–1576.
- Paredes, C., Cegarra, J., Roig, A., Sánchez-Monedero, M.A., Bernal, M.P., 1999. Characterization of olive mill wastewater (alpechin) and its sludge for agricultural purposes. *Bioresource Technology* 67, 111-115.
- Paredes, C., Bernal, M.P., Cegarra, J., Roig, A., 2002 Bio-degradation of olive mill wastewater sludge by its co-composting with agricultural wastes. *Bioresource Technology* 85, 1-8.
- Roig, A., Cayuela, M.L., Sánchez-Monedero, M.A., 2006. An overview on olive mill wastes and their valorisation methods. *Waste Management* 26, 960-969.
- Said-Pullicino, D., Erriquens, F.G., Gigliotti, G., 2007. Changes in the chemical characteristics of water extractable organic matter during composting and their influence on compost stability and maturity. *Bioresource Technology* 98, 1822-1831.
- Said-Pullicino, D., Gigliotti, G., 2007. Oxidative biodegradation of dissolved organic matter during composting. *Chemosphere* 68, 1030-1040.
- Trinchera, A., Leita, L., Sequi, P. 2006. *Metodi di Analisi per i Fertilizzanti*. Ministero delle Politiche Agricole e Forestali, Osservatorio Nazionale Pedologico e per la Qualità del Suolo Agricolo e Forestale. CRA- Consiglio per la Ricerca e la sperimentazione in Agricoltura, ISNP – Istituto Sperimentale per la Nutrizione delle Piante.
- Viel, M., Sayag, D., Peyre, A., André, L., 1987. Optimization of in-vessel co-composting through heat recovery. *Biological Wastes* 20, 167-185.
- Zucconi, F., Monaco, A., Forte, M., de Bertoldi, M., 1985. Phytotoxins during the stabilization of organic matter. In: Gasser, J.K.R. (Ed.), *Composting of Agricultural and other wastes*, Elsevier Applied Science Publishers, Barking, Essex, UK, pp. 73-85.

Table 1. Properties of the organic materials used in the two feedstocks<sup>a</sup>

	TrH	ExH	Olive tree pruning	Cereal straw
Moisture (%)	47.1 (2.1)	78.1 (3.8)	28.1 (1.4)	27.8 (1.3)
TOC (g kg <sup>-1</sup> )	493.0 (14.0)	524.3 (9.3)	545.8 (6.1)	542.7(11.7)
TKN (g kg <sup>-1</sup> )	8.3 (0.5)	16.2 (0.6)	14.0 (0.5)	7.0 (1.4)
C/N	59.4	32.4	39.0	77.5
EC (dS m <sup>-1</sup> )	0.67 (0.05)	2.58 (0.08)	nd <sup>b</sup>	nd <sup>b</sup>

TOC, Total organic carbon; TKN, Total Kjeldahl nitrogen; ES, Electrical conductivity.

<sup>a</sup> Except for moisture, all data are expressed on a dry weight basis; Values represent the mean with standard error in brackets;

<sup>b</sup> not determined.

Table 2. Organic matter loss during composting<sup>a</sup>

Time (days)	Volatile Solids (g kg <sup>-1</sup> )		Total Organic C (g kg <sup>-1</sup> ) <sup>b</sup>		Organic Matter loss (%)	
	Mixture A	Mixture B	Mixture A	Mixture B	Mixture A	Mixture B
0	956 (11.4)	922 (6.6)	462 (5.4)	479 (2.6)	0.0	0.0
7	953 (13.6)	910 (12.9)	441 (6.8)	413 (1.9)	6.7	14.5
19	957 (9.8)	880 (21.5)	496 (3.6)	272 (11.5)	0.0	38.0
47	945 (22.2)	900 (12.1)	376 (2.5)	345 (1.2)	20.9	23.9
126	920 (19.8)	860 (9.7)	253 (1.8)	264 (0.7)	47.1	48.0

<sup>a</sup> Values represent the mean expressed on a dry weight basis with standard error in brackets;

<sup>b</sup> Data corrected for ash content.

Table 3. Changes in the concentration of fats and polyphenols during composting<sup>a</sup>

Time (days)	Fats (g kg <sup>-1</sup> )		Polyphenols (g kg <sup>-1</sup> ) <sup>b</sup>	
	Mixture A	Mixture B	Mixture A	Mixture B
0	44.0 (1.4)	107.0 (6.6)	3.78 (0.22)	7.08 (0.54)
7	42.0 (3.6)	74.0 (12.9)	1.69 (0.04)	4.00 (0.23)
19	40.0 (9.8)	20.0 (1.5)	1.94 (0.02)	1.27 (0.11)
47	7.5 (0.2)	22.5 (2.1)	1.17 (0.02)	1.65 (0.05)
126	2.5 (0.8)	2.5 (0.7)	1.50 (0.11)	1.95 (0.11)

<sup>a</sup> Values represent the mean expressed on a dry weight basis with standard error in brackets;

<sup>b</sup> Expressed as gallic acid equivalents.

Table 4. N and C/N behaviour during composting<sup>a</sup>

Time (days)	Total Kjeldahl N (g kg <sup>-1</sup> )		C/N		N-NH <sub>4</sub> <sup>+</sup> (g kg <sup>-1</sup> )		Organic N (% of total N)	
	Mixture A	Mixture B	Mixture A	Mixture B	Mixture A	Mixture B	Mixture A	Mixture B
0	8.1 (0.2)	18.5 (0.3)	57.0	25.9	2.2 (0.02)	0.4 (0.02)	72.8	97.8
7	9.8 (0.1)	17.8 (0.3)	48.1	26.8	1.5 (0.01)	0.5 (0.03)	84.7	97.2
19	9.3 (0.1)	18.0 (0.5)	52.2	23.3	1.0 (0.01)	0.8 (0.01)	89.2	95.6
47	8.9 (0.2)	24.0 (0.5)	53.0	18.4	1.5 (0.02)	0.6 (0.01)	83.1	97.5
126	17.6 (0.3)	28.5 (0.3)	26.2	16.6	0.8 (0.02)	1.5 (0.03)	95.5	94.7

<sup>a</sup> Values represent the mean expressed on a dry weight basis with standard error in brackets

Table 5. Chemical characteristics of the final composts obtained<sup>a</sup>

	Compost A	Compost B	Italian law limits
Moisture (%)	28.3 (1.6)	38.4 (0.8)	< 50
pH	7.4 (0.02)	8.1 (0.07)	6.0 – 8.5
EC (dS m <sup>-1</sup> )	1.55 (0.01)	3.29 (0.24)	
TOC (g kg <sup>-1</sup> )	461 (3.4)	473 (1.3)	> 200
TKN (g kg <sup>-1</sup> )	18 (0.4)	29 (0.5)	
Total organic N (g kg <sup>-1</sup> )	17	27	> 80% of TKN
C/N	26.2	16.6	< 25
Humic acid + Fulvic acid C (g kg <sup>-1</sup> )	292 (11.8)	142 (3.9)	> 70
Total P (g kg <sup>-1</sup> )	2.7 (0.2)	5.1 (0.5)	
Total K (g kg <sup>-1</sup> )	7.6 (0.1)	33.3 (0.2)	
Total Cd (mg kg <sup>-1</sup> )	0.8 (0.03)	0.3 (0.06)	< 1.5
Total Cu (mg kg <sup>-1</sup> )	28.3 (0.5)	57.1 (0.5)	< 150
Total Hg (mg kg <sup>-1</sup> )	0.22 (0.01)	< 0.05 <sup>b</sup>	< 1.5
Total Ni (mg kg <sup>-1</sup> )	< 0.05 <sup>b</sup>	2.6 (1.1)	< 50
Total Pb (mg kg <sup>-1</sup> )	14.4 (0.2)	< 0.5 <sup>c</sup>	< 140
Total Zn (mg kg <sup>-1</sup> )	27.8 (0.4)	41.2 (1.0)	< 500

EC, Electrical conductivity; TOC, Total organic carbon; TKN, Total Kjeldahl nitrogen

<sup>a</sup> Except for moisture, all data are expressed on a dry weight basis; Values represent the mean with standard error in brackets;

<sup>b</sup> limit of sensitivity of the method used.



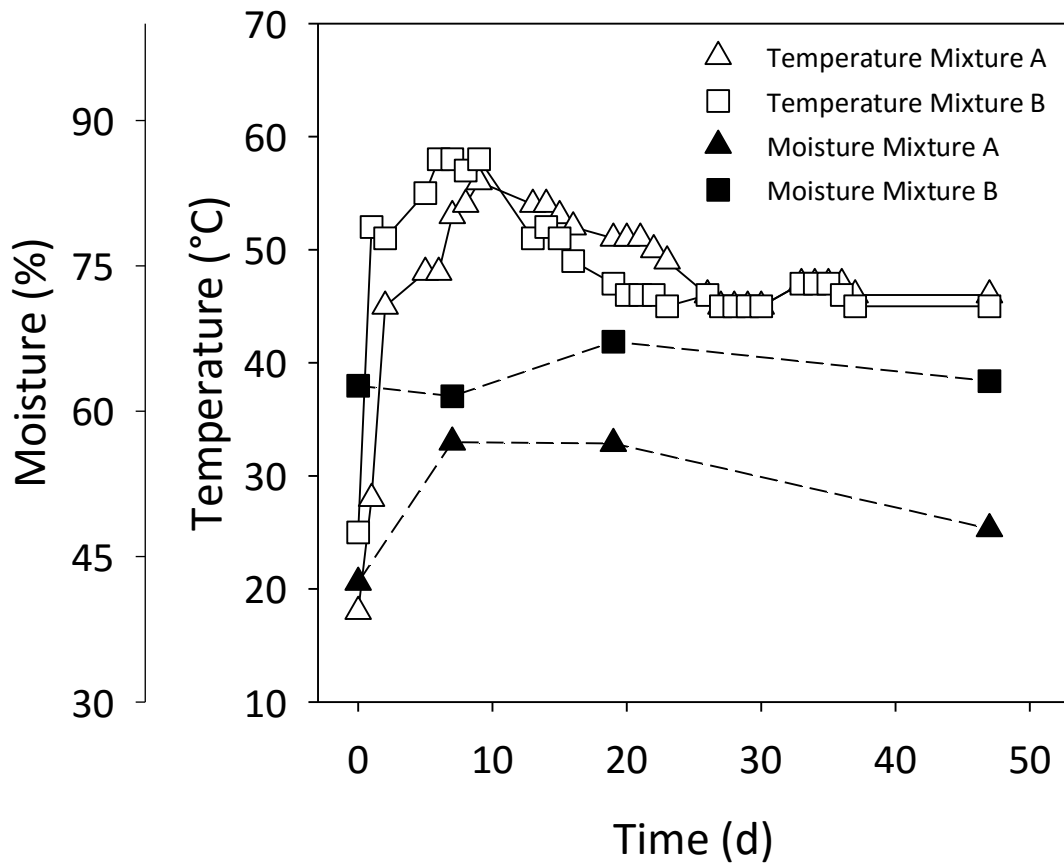


Fig. 1. Temperature and moisture profiles during the active composting phase of mixtures A and B.

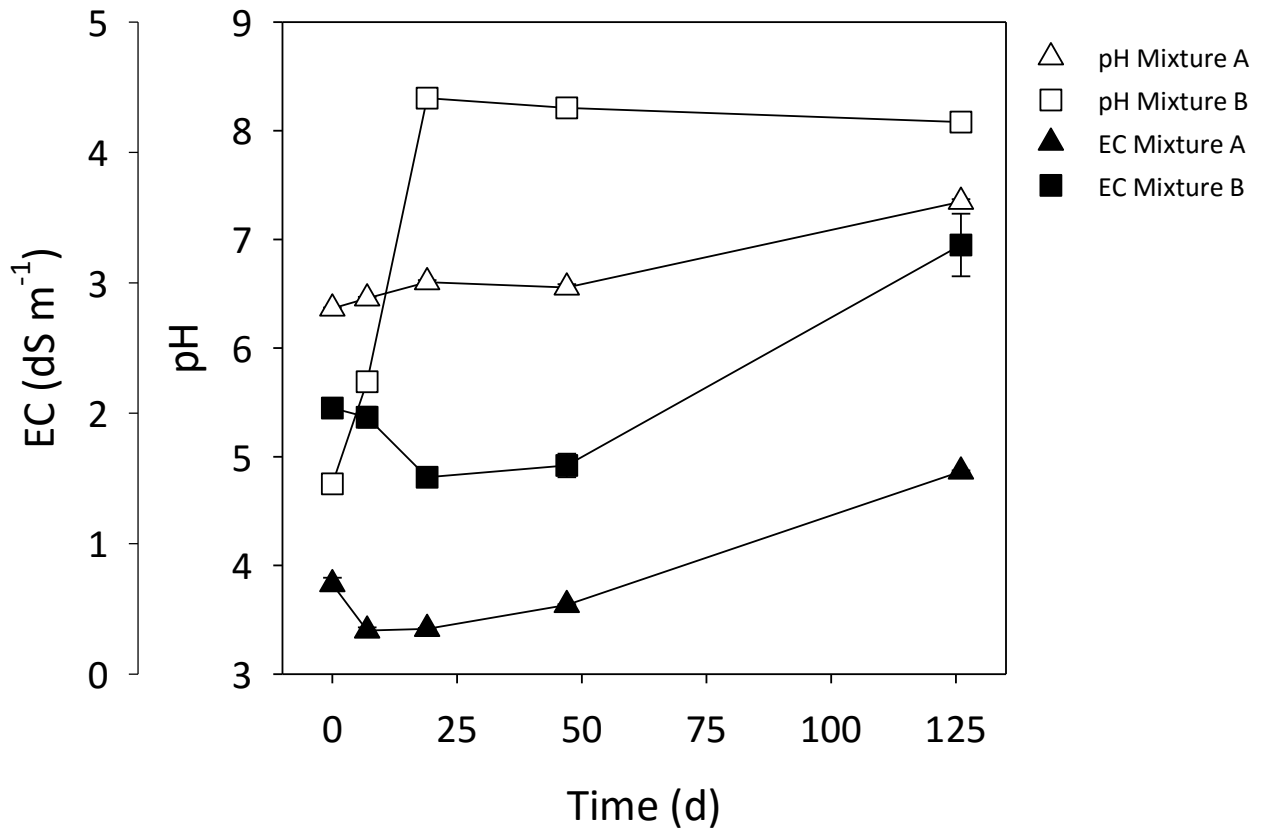


Fig. 2. pH and electrical conductivity (EC) profiles during the composting of mixtures A and B. Error bars indicate SE.

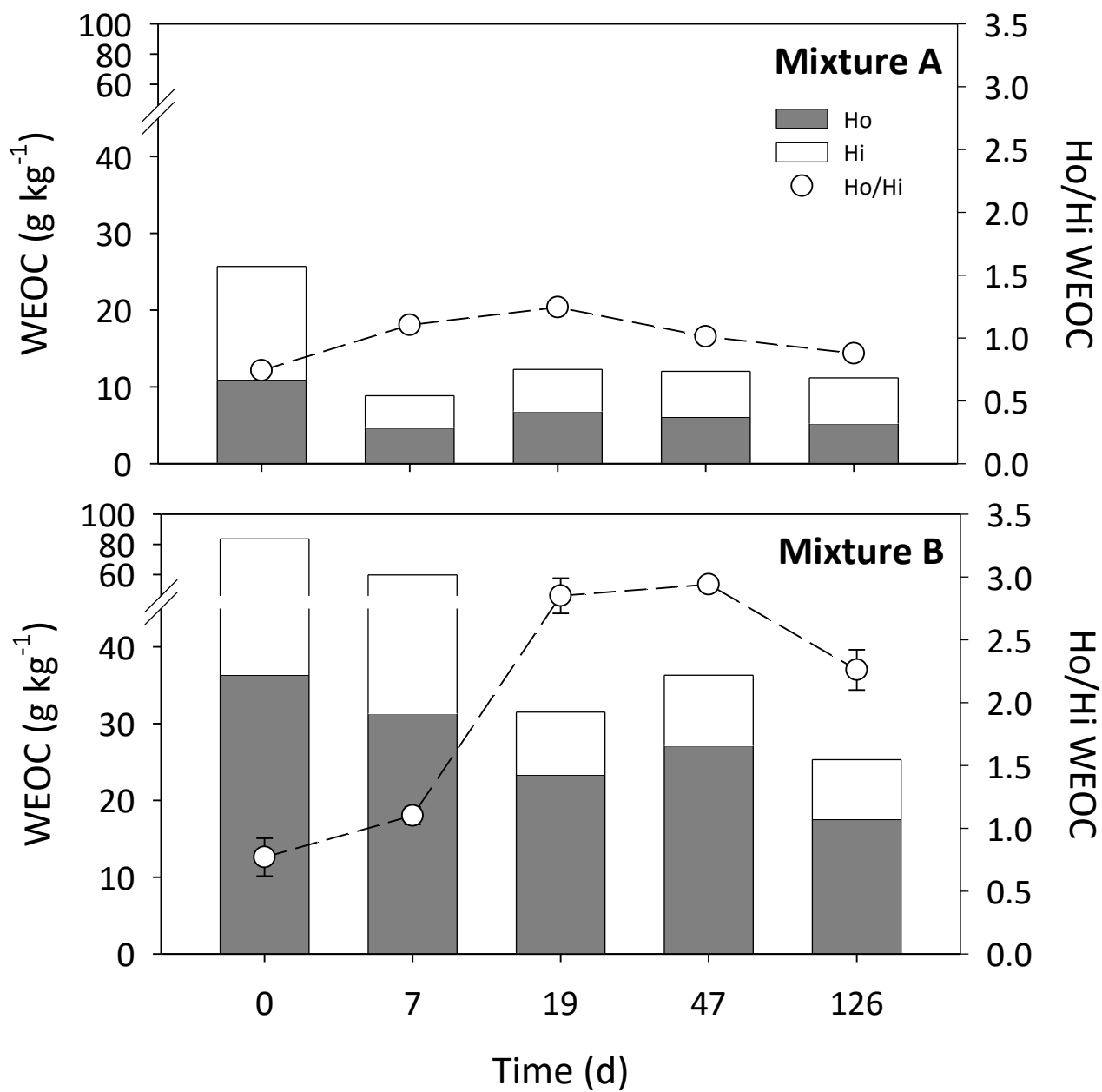


Fig. 3. Variations in water extractable organic carbon (WEOC) content, and distribution between hydrophobic (Ho) and hydrophilic (Hi) fractions during the composting of mixtures A and B. Error bars indicate SE.

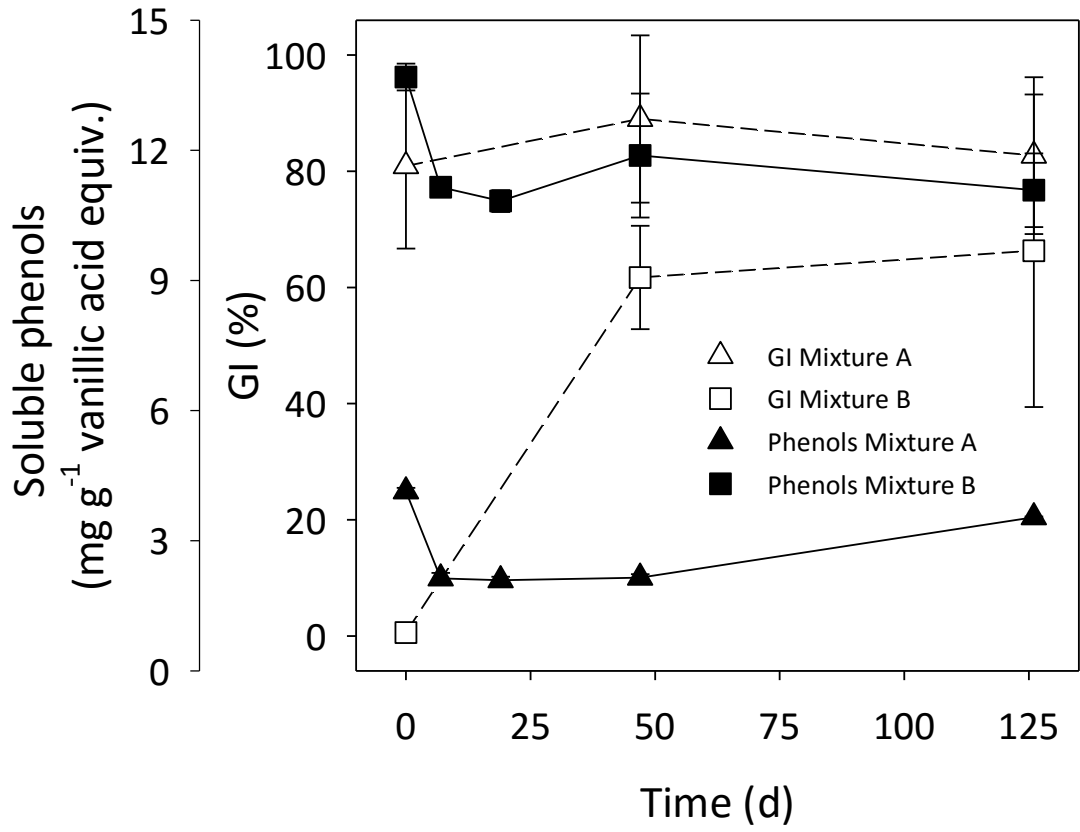


Fig. 4. Variations in the germination index (GI) and soluble phenols during the composting of mixtures A and B. Error bars indicate SE.