



Carbon mineralization and distribution of nutrients within different particle-size fractions of commercially produced olive mill pomace

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ABSTRACT

Composting is a realistic option for disposal of olive mill pomace (OMP) by making it suitable as a soil amendment for organic farming. The chemical and physical characteristics and contribution of particle-size fractions to total nutrients and carbon mineralization of seven commercial composts of OMP (COMP) were investigated. Higher proportions of manure, co-composted with OMP, reduced the organic matter (OM), total carbon and C:N ratio of the product, but increased the content of nutrients and fine particles. The fine particles had higher nutrient contents, but less OM and carbon and, unlike larger particles, did not exhibit any phytotoxicity. Less than 1.5% of added carbon was mineralized in whole compost, but a lower rate was found with larger particles. Separation of COMP by particle size fractionation and application as a soil conditioner is recommended for better optimization of COMP with the <1 mm fraction providing the higher quality compost.

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1. Introduction

Olive mill pomace (OMP) is the main by-product of the two-stage olive oil extraction process. Currently, between 2.5 and 4.0 million tonnes of OMP are produced annually in the Andalusia region of southern Spain. OMP is a potentially harmful by-product for the environment arising from the phytotoxic and antimicrobial properties, mainly due to the phenolic and lipid constituents (Paredes et al., 1987; Perez et al., 1992), and therefore, direct application to rivers or soil is not permitted under regional environmental regulations. Thus in recent years, efforts have been intensified to provide better solutions for the safer disposal of OMP in the environment.

One of the main options involves recycling OMP organic matter and nutrients through composting and subsequent land application back onto organic olive tree farms. Composting OMP (COMP) enables it to be sanitized; the mass and volume of the product are reduced and stabilized prior to land spreading. This approach helps to improve soil fertility, reduces the use of inorganic fertilizers in olive oil farming and for a commercial enterprise, can provide an additional source of revenue for the olive mill economy. Despite this, the current amount of COMP produced annually in Andalusia is relatively small (at around 70,000 t), but has increased exponentially during the last 5 years (Álvarez de la Puente et al., 2010).

There are few studies on the agrochemical characterization of the final COMP product (e.g. Alburquerque et al., 2006), however,

these studies have been undertaken using only a limited number of types of COMP, which were produced in small quantities and on a small experimental scale. Typically before composting, OMP is mixed with olive tree leaves, which are gathered along with the olive fruit, twigs and small branches, as well as straw and manure, or any other locally available animal or plant material which may be added to increase the nutrient content. The relatively high diversity of the bulking agents and their mixing proportions will therefore, be responsible for some inherent heterogeneity in the COMP, not only in carbon (C), nitrogen (N), phosphorus (P) and potassium (K) contents, but also in the particle size distribution of the final product.

The relationship between particle-size distribution of an exogenous source of organic matter (OM), and the C and N dynamics in soils after OM application has been demonstrated for sludge compost by Doublet et al. (2010) among others. Similarly, C mineralization and turnover was seen to differ according to the particle-size fraction in a cattle slurry compost (Grilo et al., 2011; Fanguiero et al., 2007), also from a sludge-straw mixture (Robin et al., 2008) and in an aerobically digested sewage sludge composted along with screened green waste compost, stored yard trimmings and crushed wood pallets (Doublet et al., 2010).

Nitrogen availability in compost has also been shown to be related to particle size: increasing as particle size decreased in sludge compost (Tester et al., 1979). Generally, N mineralization is greater in the fine and water-soluble fractions than in coarser fractions, at least for manure and city refuse composts (Aoyama, 1985) and in composted sewage sludge (Doublet et al., 2010). These results, which can most likely be explained by differing OM characteristics,

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suggest that the size of fractions in compost contributes substantially to the total C and N dynamics of mineralization after the compost is incorporated into soil. This is of particular interest, because depending on the range in particle sizes, composts could therefore provide a means for storage of C in soil (i.e. from the larger sized compost fractions), or a source of available N (i.e. from the finer compost fractions).

As far as we know, there have been no studies investigating the particle size distribution of COMP which is produced commercially. The characterization of the particle size distribution of composts is of importance, because the relatively high volume of COMP produced in olive mills could make it economically feasible to undertake an industrially-based fractionation of composts and thereby generate different composts with the desired qualities through particular combinations of different particle-sizes.

In the present study, the characterization of particle size fractions including C and N distribution within those fractions and the potential contribution to C mineralization were investigated for seven out of the eleven commonly occurring COMP products found in southern Spain.

2. Methods

2.1. Collection and particle size fractionation

Seven ready-to-apply olive mill pomace composts were collected from several olive mills located in different provinces of Andalusia (Almería, Cádiz, Córdoba, Jaén and Málaga). All the composting methods used aerated heaps and the duration of the composting period was similar at between 7 and 9 months. However, composts from these olive mills differed in their composition and in the proportions of primary materials such as olive mill pomace (OMP), olive leafy material (OLM), manure and straw. Table 1 shows the proportions of the raw materials co-composted with the OMP.

At each site, five compost samples were collected from five individual and separate heaps. Each compost sample consisted of five bulked subsamples taken from five different positions within the heap. After collection, samples were transported on the same day to the laboratory and stored in a cold environment (4 °C) prior to analysis. Each sample was air-dried.

Sub-samples of the air-dried compost (500 g) were passed successively through eight large sieves to obtain ten different size fractions: whole/unseparated, >12, 9.5–12, 6.0–9.5, 2.0–6.0, 1.0–2.0, 0.425–1.0, 0.212–0.425, 0.045–0.212 and <0.045 mm. Each fraction was oven dried at 85 °C (Smith, 1973), weighed and stored in a dry environment before further analyses. In the current study, compost fractions greater than 6.0 mm were not considered for further analyses as they were comprised mainly of stones. In addition, because the contribution by mass of some of the particle fractions was very low, the majority of measurements were concentrated on three bulked fractions in the range of particle sizes from <0.212, 0.212–1.0 and 1.0–6.0 mm.

Table 1
Proportions of the raw materials co-composted with olive mill pomace.

Composted olive mill pomace	OMP (%)	OLM (%)	Manure (%)	Straw (%)
COMP1	80	7	13 (sheep)	–
COMP2	75	12	13 (poultry)	–
COMP3	60	–	40 (sheep)	–
COMP4	70	8	12 (sheep)	10
COMP5	85	5	–	10
COMP6	80	20	–	–
COMP7	80	13	7 (poultry)	–

2.2. Analytical methods

All of the whole/unseparated compost samples and the 0.212–1.0 mm fraction were analyzed for pH in water extracts (1:10, w:v).

Before any further analyses, compost samples were ground to a powder (<1.0 mm) using a ball mill.

Organic matter, total carbon and total N (TN), potential N mineralization (PNM), available organic carbon (AOC), total water soluble inorganic N and total water soluble inorganic P, and total P and K contents were determined for the whole compost and <0.212 mm, 0.212–1.0 and 1.0–6.0 mm fractions.

Organic matter was determined by loss on ignition (LOI) at 550 °C for 3 h (Nelson and Sommer, 1982). Total C (TC) and N (TN) contents were determined using a CHN auto-analyzer (Carlo Erba NA200, Milan Italy). Potential N mineralization (PNM) was analyzed in fresh samples following Lober and Reeder (1993). Briefly, compost samples were incubated over 7 days at 40 °C in syringes with the air expelled to produce anaerobic conditions and after incubation, ammonium concentrations were analyzed by automated segmented-flow colorimetry. Water soluble carbon (WSC) and hot water carbon (HWC) were determined after Ghani et al. (2003). Briefly, 2 g of COMP was mixed with 35 ml of distilled water for 30 min shaking, and the supernatant was filtered and stored (WSC). The sediment of the WSC extracts were incubated with 35 ml of distilled water for 16 h at 80 °C, after which the supernatant was filtered and stored (HWC). Total organic C of the WSC and HWC extracts (filtered supernatants) were analyzed by a Skalar Formacs analyzer. Available organic C (AOC) was calculated as the sum of both WSC and HWC. Total water soluble inorganic N (TWSIN) and phosphorus (TWSP) were analyzed in the WSC extracts. TWSIN was determined after nitrate and ammonium analyses on an automated segmented-flow colorimetry and TWSP by the analyses of phosphate after a persulphate oxidation of the WSC extracts. Total P and total K contents were analyzed by an ICP-MS Agilent Series 7500 after a HNO₃/HClO₄ digestion (Sommer and Nelson, 1972).

Polyphenol, acid detergent fiber and lignin, and cellulose contents and germination index were measured on the whole/unseparated compost samples and <1.0 mm and 1.0–6.0 mm fractions. Total extractable polyphenol contents were determined using Folin-Ciocalteu reagent following an adaptation of the method of Anderson and Ingram (1993).

Acid detergent fiber (ADF) and acid detergent lignin (ADL) were measured following Van Soest (1963). Briefly, ADF was determined using acid detergent solution with cetyltrimethyl ammonium bromide (CTAB) and ADL after digesting the samples with H₂SO₄ (ADL data were corrected for ash content). The percentage of cellulose was estimated by the difference between ADF and ADL (Goering and Van Soest, 1970).

Compost phytotoxicity was measured using the Zucchini test (Zucchini et al., 1981). Briefly, 10 seeds of *Lepidium sativum* were incubated with 5 ml of compost extract (1:10) after 7 days in a Petri dish. The germination index (GI) was calculated according to: $GI = (G/G_0) \times (L/L_0) \times 100$, where G and G₀ are the germination percentages of each COMP extract and the control (distilled water), respectively, and L and L₀ the mean of the root lengths of the samples and control, respectively.

The potential mineralization of organic C was measured in the whole compost and <0.212, 0.212–1.0 and 1.0–6.0 mm fractions during a 28 day incubation period in soil at 25 °C in the dark and under aerobic conditions. All the organic samples were measured after air-drying but were not ground. The soil we used was sampled from the surface layers (top 10 cm) of an experimental plot at Rothamsted Research, North Wyke, Devon, SW England (50° 46' N, 3° 54' W, elevation; 180 m asl), from the Hallsworth soil series: a typical poorly drained, silty clay loam; USDA typic haplaquept. Some important

physical and chemical properties of the soil were reported elsewhere (Hatch et al., 1998). The main analytical soil characteristics were as follows: organic C 5%; total N 0.6%, C:N ratio 9.2 and pH 5.1. After sampling, the soil was sieved to 2 mm and kept moist at 4 °C until use. The incubated mixtures were prepared in triplicates with 44.8 g of wet soil (or 2 g soil organic C) in 1 L Kilner jars. All the organic amendments (between 2 and 11 g depending on the size fraction C content) were based on the same amount of organic C (equivalent to 1 g C). The water content of the mixtures was adjusted to 60% of the water holding capacity and was maintained during the entire incubation period by weighing and, if necessary, adding de-ionized water. The control was prepared with soil and the same amount of water, but without receiving any COMP or particulate fractions. The CO₂-C production rate was determined after three hours and again after 1, 2, 7, 14, 21 and 28 days of incubation. At each sampling, two gas samples were taken from the headspace of each Kilner jars: one just before the jars were hermetically sealed (time 0) and another, after three hours of incubation. Linearity for the increase of CO₂ concentrations in the headspace during the three hours was checked in a preliminary assay. The gas samples were analyzed using an infra-red gas analyzer (ADC type 225 MK3, Hoddesdon, Herts, UK) for CO₂ concentration.

2.3. Statistical analysis

Differences in the measured variables among the COMP samples were tested using a one-way ANOVA and Fisher post hoc test. Correlation among measured variables was tested with the Pearson-moment correlation coefficient. Differences between measured variables among particle-size fractions of the COMP were tested by two-way ANOVA. Assumptions of analysis of variance (homocedasticity and normality) were tested and assured by using transformed data sets [$\log(\text{dependent variable value} + 1)$] where necessary. Significance was accepted at $P < 0.05$ in all cases. Principal Component Analysis (PCA) was applied using values of the whole COMP. Only the first principal component (PC1) was selected for the management of cases, since this explained most of the variability in the data. Product-moment correlations were performed between COMP physico-chemical properties and scores on the PC1 for the interpretation of the new axes. Statistical analyses were performed using STATISTICA 6.0 scientific software.

3. Results and discussion

3.1. Characterization of whole (non-fractionated) COMP

The main characteristics of the seven COMPs, which are currently produced in Andalusia, differ markedly according to the raw material co-composted with OMP.

Compost organic matter content (measured as LOI) ranged from 27.2% to 87.9% for COMP3 and COMP6, respectively, and averaged 60.6% (Table 2). These values were similar or slightly higher than the experimentally-produced COMP of Albuquerque et al. (2006), which ranged between 34.4% and 60.5%, but were higher than the LOI content typically found for cow, sheep and poultry manures and similar to those found for horse, pig and rabbit manures (Moral et al., 2005). Highest values were found in those composts which included a relatively high proportion of OLM (COMP 6 and 7) or OLM and straw (COMP4 and COMP5), and might be due to incomplete LOI degradation of the larger particle size and the high lignin content of LOI. As expected, compost total C was closely related to LOI ($r = 0.80$; $P < 0.01$) (Table 2) and averaged 30.8%. The soils in olive groves are characterized by relatively low organic matter contents (typically < 1%), and thus the high LOI and TC of COMP make them suitable for application to the soil as a practical

approach to increase soil organic matter contents. Compost TN ranged between 1.06 (COMP4) and 2.0% (COMP2) and the highest was achieved for the two composts (COMP2 and COMP7) made with poultry manure (Table 2), and are typically at the lower end of the N contents of manures (Moral et al., 2005), but within the range of compost made from plant residues without additives (Goyal et al., 2005). The C:N ratio of the whole compost ranged from 27.2 to 35.8 for COMP4, COMP5 and COMP6, but was much lower (10.5–18.7) for COMP1, COMP2, COMP3 and COMP7, all of which included sheep or poultry manures. Total K was highest for COMP2 (2.39%), which included a high proportion of poultry manure, but lowest for COMP4 (1.06%) and COMP5 (1.47%), both of which included straw (Table 2). These relatively high levels of K in the compost agree with the general characteristics of olive mill waste and by-products due to the high total K content of olive fruits. The composts with the highest contents of total P were found typically for those which included manures (COMP1, COMP2, COMP3 and COMP7) as raw material, whereas it was lowest for OMP composted with only OLM or straw. Available water soluble inorganic N varied by more than one order of magnitude for the whole compost and was highest for the compost which included 13% poultry manure (COMP2; $1504 \mu\text{g g}^{-1}$), or a high proportion of sheep manure (COMP3; $900 \mu\text{g g}^{-1}$) (Table 2). For available water soluble P, there seemed to be no pattern for the whole compost, being higher for COMP1, 2 and 7 and the lowest for COMP5 made with straw, OMP and OLM.

Lignin contents (ADL) ranged from 7.6% of the COMP3 (Table 3), which included a high proportion of sheep manure, to 35.3% in the COMP4 (with straw and manure as the primary bulking materials). Polyphenol contents were relatively low and similar for all the composts, except for COMP3 with 9.38 mg g^{-1} (lowest value) and COMP2 with 21.1 mg g^{-1} (highest value).

3.2. Characterization of particle size fractions of COMP

The relative proportion of the COMP fractions varied depending on the COMP type. Overall, more than 51.6% of the COMP mass was found in particles between 1.0 and 6.0 mm (Table 2). For COMPs 1, 2, 3 and 4, all including sheep or poultry manure, between 51.6% and 63.6% of the total COMP was composed of particles 1.0–6.0 mm, although COMP7, which also included poultry manure, was the exception. These COMPs showed similar percentages of particle size fractions to those reported by Fangueiro et al. (2010) for duck manure, suggesting that during composing, manures provide a relatively good enrichment of fine fractions. On the other hand, COMP5 COMP6 and COMP7, resulting from composting OMP with a high percentage of OLM and without any, or only a small proportion of manure showed the highest content of particles 1.0–6.0 mm. Typically, the content of fine fractions (<0.212 mm) was lower than 15.0%.

3.3. Concentration, mass and distribution of C, N, P and K within the separated fractions

Generally, the LOI and TC contents increased with particle size, both being significantly higher in fractions 1.0–6.0 mm than in the finer fractions; although with some exceptions (see Table 2). For COMP3, neither LOI, nor TC were significantly different among separated fractions, and for COMP2, TC was only significantly lower for the <0.212 mm fraction. Doublet et al. (2010), for sludge compost made with sewage sludge, screened refuse, yard trimmings and pallets, and Fangueiro et al. (2007), for two types of dairy slurries found, as in this study, an increase in the TC content with larger particle sized fractions, although these differences were more modest. It is likely that the lack of OLM in the initial mixture made COMP3 more homogeneous and with a much lower LOI content

Table 2

Some properties of unseparated (whole) and size-fractions of COMP. Data are the mean \pm standard deviation ($n = 3$). Different capital letters in the same column denote significant differences ($P < 0.05$) between whole COMPs, whereas different lower case letters denote significant differences between fractions and whole for each individual COMP type.

	Fraction (mm)	Relative proportion	pH	LOI (%)	TC (%)	AOC (mg TOC g ⁻¹)	TN (%)	TSWIN ($\mu\text{g g}^{-1}$)	C:N	K (%)	P (%)	TSWP ($\mu\text{g g}^{-1}$)
COMP1	Whole	100	7.99 \pm 0.30 ^{Aa}	65.0 \pm 2.1 ^{Aa}	26.7 \pm 1.4 ^{Aa}	16.2 \pm 2.01 ^{Aa}	1.59 \pm 0.1 ^{Aa}	131.9 \pm 16 ^{Aab}	16.8 \pm 1.4 ^{Aab}	1.64 \pm 0.09 ^{Aa}	0.33 \pm 0.02 ^{Aa}	178.9 \pm 47 ^{Aa}
	1.0–6.0	63.6		72.2 \pm 1.0 ^b	32.2 \pm 2.5 ^b	14.8 \pm 1.49 ^a	1.50 \pm 0.4 ^a	123.0 \pm 36 ^a	22.5 \pm 7.8 ^a	1.11 \pm 0.17 ^b	0.18 \pm 0.05 ^b	169.3 \pm 31 ^b
	0.212–1.0	20.5	7.00 \pm 0.57 ^b	54.2 \pm 1.3 ^c	28.3 \pm 0.9 ^{ab}	15.0 \pm 1.42 ^a	2.48 \pm 0.1 ^b	131.8 \pm 14 ^{ab}	11.4 \pm 0.8 ^b	1.45 \pm 0.04 ^c	0.33 \pm 0.02 ^a	186.5 \pm 45 ^b
	<0.212	15.7		42.4 \pm 0.1 ^d	22.6 \pm 1.5 ^c	13.3 \pm 1.48 ^a	2.25 \pm 0.1 ^b	165.0 \pm 7.6 ^b	10.0 \pm 0.2 ^b	1.63 \pm 0.06 ^a	0.40 \pm 0.02 ^c	162.8 \pm 50 ^b
COMP2	Whole	100	8.30 \pm 0.02 ^{Ba}	51.6 \pm 4.0 ^{Ba}	21.1 \pm 0.6 ^{Ba}	23.4 \pm 0.52 ^{Ba}	2.00 \pm 0.1 ^{Ba}	1504.1 \pm 36 ^{Ba}	10.5 \pm 0.3 ^{Ba}	2.39 \pm 0.01 ^{Ba}	1.19 \pm 0.04 ^{Ba}	820.2 \pm 39 ^{Ba}
	1.0–6.0	51.6		57.4 \pm 1.9 ^b	29.1 \pm 6.9 ^b	20.9 \pm 2.09 ^a	2.06 \pm 0.6 ^{ab}	1083.0 \pm 308 ^{ab}	14.3 \pm 1.1 ^b	2.01 \pm 0.30 ^a	0.55 \pm 0.30 ^b	868.1 \pm 179 ^a
	0.212–1.0	34.4	8.28 \pm 0.03 ^a	51.9 \pm 3.1 ^a	30.7 \pm 0.5 ^b	20.9 \pm 5.64 ^a	2.71 \pm 0.0 ^{bc}	869.3 \pm 74 ^b	11.3 \pm 0.5 ^a	2.21 \pm 0.13 ^a	0.88 \pm 0.02 ^c	958.3 \pm 108 ^{ab}
	<0.212	13.9		47.6 \pm 0.4 ^a	25.0 \pm 1.7 ^{ab}	22.4 \pm 1.66 ^a	3.17 \pm 0.0 ^c	1272.7 \pm 28 ^a	7.9 \pm 0.7 ^c	2.05 \pm 0.18 ^a	1.31 \pm 0.05 ^a	1534.8 \pm 568 ^b
COMP3	Whole	100	8.20 \pm 0.02 ^{Ba}	27.2 \pm 2.4 ^{Ca}	18.4 \pm 2.9 ^{Ca}	11.9 \pm 0.47 ^{Ca}	1.60 \pm 0.3 ^{Aa}	899.6 \pm 49 ^{Ca}	11.1 \pm 0.9 ^{Ba}	2.11 \pm 0.05 ^{Ca}	0.44 \pm 0.01 ^{Ca}	138.4 \pm 26 ^{Ca}
	1.0–6.0	56.1		31.0 \pm 1.8 ^b	18.0 \pm 1.8 ^a	11.5 \pm 1.11 ^a	1.34 \pm 0.4 ^a	909.6 \pm 123 ^a	14.1 \pm 3.6 ^a	2.36 \pm 0.37 ^a	0.40 \pm 0.12 ^a	179.5 \pm 37 ^{abc}
	0.212–1.0	28.7	6.90 \pm 0.38 ^b	31.1 \pm 0.6 ^b	18.8 \pm 0.2 ^a	12.4 \pm 0.37 ^a	1.85 \pm 0.1 ^a	884.6 \pm 70 ^a	10.2 \pm 0.4 ^a	2.32 \pm 0.12 ^a	0.48 \pm 0.00 ^a	211.5 \pm 70 ^b
	<0.212	15.0		28.8 \pm 1.0 ^{ab}	15.8 \pm 2.6 ^a	9.68 \pm 0.93 ^b	1.65 \pm 0.2 ^a	830.3 \pm 36 ^a	9.6 \pm 0.02 ^a	2.25 \pm 0.03 ^a	0.43 \pm 0.05 ^a	118.5 \pm 33 ^c
COMP4	Whole	100	8.19 \pm 0.11 ^{ABa}	54.0 \pm 1.5 ^{Ba}	35.0 \pm 0.8 ^{Da}	4.61 \pm 0.34 ^{Da}	1.06 \pm 0.0 ^{Cab}	35.2 \pm 1.1 ^{Da}	33.1 \pm 0.4 ^{Ca}	1.06 \pm 0.04 ^{Da}	0.22 \pm 0.00 ^{Da}	134.9 \pm 26 ^{Ca}
	1.0–6.0	62.6		65.1 \pm 0.1 ^b	41.8 \pm 1.3 ^b	5.36 \pm 0.20 ^b	0.96 \pm 0.1 ^a	38.0 \pm 6.4 ^a	44.6 \pm 9.8 ^b	0.93 \pm 0.01 ^b	0.14 \pm 0.01 ^b	141.2 \pm 24 ^a
	0.212–1.0	24.2	8.11 \pm 0.16 ^a	45.0 \pm 1.4 ^c	30.6 \pm 1.5 ^c	3.74 \pm 0.19 ^c	1.25 \pm 0.1 ^{ab}	34.4 \pm 6.1 ^a	24.5 \pm 2.0 ^{ac}	1.00 \pm 0.06 ^{ab}	0.19 \pm 0.01 ^{ac}	115.4 \pm 61 ^a
	<0.212	13.0		50.5 \pm 0.3 ^d	27.3 \pm 2.2 ^c	4.88 \pm 0.37 ^a	1.35 \pm 0.1 ^b	67.1 \pm 15 ^b	20.2 \pm 0.8 ^c	1.02 \pm 0.01 ^{ab}	0.17 \pm 0.01 ^c	192.4 \pm 19 ^a
COMP5	Whole	100	7.45 \pm 0.15 ^{Ca}	70.2 \pm 1.5 ^{Da}	38.2 \pm 0.3 ^{Ea}	18.3 \pm 0.53 ^{Aa}	1.07 \pm 0.0 ^{Ca}	17.3 \pm 1.9 ^{Da}	35.8 \pm 0.9 ^{Da}	1.47 \pm 0.13 ^{Ea}	0.19 \pm 0.01 ^{Ea}	57.7 \pm 3.4 ^{Da}
	1.0–6.0	80.0		71.3 \pm 0.5 ^a	37.8 \pm 0.7 ^a	18.7 \pm 1.72 ^a	1.01 \pm 0.1 ^a	15.4 \pm 3.1 ^a	37.6 \pm 4.7 ^a	1.24 \pm 0.14 ^b	0.13 \pm 0.00 ^b	60.8 \pm 9.8 ^{ab}
	0.212–1.0	13.3	6.62 \pm 0.53 ^b	66.7 \pm 0.4 ^b	36.1 \pm 0.05 ^a	19.9 \pm 0.51 ^a	1.28 \pm 0.1 ^b	16.3 \pm 3.7 ^a	28.3 \pm 2.7 ^b	1.41 \pm 0.03 ^a	0.17 \pm 0.03 ^c	77.4 \pm 28 ^{ab}
	<0.212	6.6		59.3 \pm 0.5 ^c	31.3 \pm 3.0 ^b	18.9 \pm 0.80 ^a	1.31 \pm 0.1 ^b	24.1 \pm 1.7 ^b	23.9 \pm 0.5 ^b	1.33 \pm 0.04 ^{ab}	0.17 \pm 0.00 ^c	100.4 \pm 28 ^b
COMP6	Whole	100	7.73 \pm 0.07 ^{Da}	87.9 \pm 2.4 ^{Ea}	39.0 \pm 0.4 ^{Ea}	15.8 \pm 0.10 ^{Aa}	1.43 \pm 0.1 ^{Aa}	42.6 \pm 2.1 ^{Da}	27.2 \pm 1.3 ^{Eab}	1.48 \pm 0.07 ^{Eab}	0.16 \pm 0.01 ^{Ea}	96.9 \pm 5.1 ^{CDa}
	1.0–6.0	84.8		86.3 \pm 1.1 ^a	42.5 \pm 0.08 ^b	16.6 \pm 2.55 ^a	1.47 \pm 0.4 ^a	38.3 \pm 9.3 ^a	30.1 \pm 8.5 ^a	1.30 \pm 0.22 ^a	0.11 \pm 0.04 ^b	102.8 \pm 28 ^a
	0.212–1.0	11.4	7.11 \pm 0.12 ^b	76.0 \pm 1.5 ^b	40.9 \pm 1.6 ^{ab}	20.1 \pm 0.35 ^b	2.26 \pm 0.2 ^b	70.2 \pm 10 ^a	18.2 \pm 3.0 ^{bc}	1.76 \pm 0.18 ^{cb}	0.19 \pm 0.02 ^a	128.3 \pm 22 ^a
	<0.212	3.6		62.4 \pm 2.0 ^c	33.2 \pm 1.7 ^c	16.4 \pm 2.30 ^a	2.28 \pm 0.01 ^b	158.2 \pm 63 ^b	14.5 \pm 0.8 ^c	1.63 \pm 0.06 ^{ac}	0.18 \pm 0.01 ^a	194.0 \pm 55 ^b
COMP7	Whole	100	8.34 \pm 0.06 ^{Ba}	67.6 \pm 0.9 ^{ADa}	36.7 \pm 0.8 ^{DEa}	25.0 \pm 2.47 ^{Ba}	1.95 \pm 0.0 ^{Ea}	275.7 \pm 2.6 ^{Ea}	18.7 \pm 0.3 ^{Fa}	2.02 \pm 0.15 ^{Ca}	0.38 \pm 0.02 ^{Fa}	308.1 \pm 21 ^{Ea}
	1.0–6.0	85.2		72.3 \pm 0.6 ^b	35.2 \pm 1.5 ^{ab}	28.2 \pm 1.34 ^a	2.06 \pm 0.2 ^a	280.4 \pm 126 ^a	17.1 \pm 1.0 ^a	2.07 \pm 0.05 ^a	0.28 \pm 0.04 ^b	313.5 \pm 23 ^a
	0.212–1.0	10.8	8.66 \pm 0.03 ^b	66.0 \pm 0.3 ^c	33.4 \pm 0.73 ^{bc}	28.5 \pm 3.50 ^a	2.71 \pm 0.2 ^b	275.1 \pm 54 ^a	12.4 \pm 1.4 ^b	1.63 \pm 0.04 ^a	0.46 \pm 0.04 ^{ac}	299.3 \pm 25 ^a
	<0.212	3.9		63.3 \pm 0.5 ^d	31.3 \pm 1.7 ^c	29.0 \pm 2.24 ^a	2.95 \pm 0.1 ^b	375.2 \pm 34 ^a	10.6 \pm 0.04 ^b	2.15 \pm 0.53 ^a	0.47 \pm 0.08 ^c	439.8 \pm 51 ^b
MEAN	Whole	100.0	8.03 \pm 0.33	60.6 \pm 18.9	30.8 \pm 8.6	16.5 \pm 6.9	1.54 \pm 0.4	415 \pm 571	21.9 \pm 10	1.74 \pm 0.45	0.42 \pm 0.36	247 \pm 264
	1.0–6.0	69.2 \pm 13.9		65.1 \pm 17.4	33.8 \pm 8.3	16.6 \pm 7.2	1.49 \pm 0.5	355 \pm 449	25.8 \pm 12	1.58 \pm 0.56	0.25 \pm 0.16	262 \pm 278
	0.212–1.0	20.5 \pm 9.2	7.52 \pm 0.80	55.9 \pm 15.1	31.3 \pm 6.9	17.2 \pm 7.8	2.08 \pm 0.6	325 \pm 385	16.7 \pm 7.3	1.68 \pm 0.46	0.38 \pm 0.25	282 \pm 306
	<0.212	10.3 \pm 5.3		50.7 \pm 12.4	26.7 \pm 6.1	16.4 \pm 8.1	2.14 \pm 0.7	413 \pm 467	13.9 \pm 6.1	1.72 \pm 0.46	0.45 \pm 0.40	366 \pm 452

Table 3

Content of acid detergent lignin (ADL), acid detergent fibre (ADF), cellulose and polyphenols of unseparated (whole) COMP and size-fractions of COMP. Data are the mean \pm standard deviation ($n = 3$). Different capital letters in the same column denote significant differences ($P < 0.05$) between whole COMP. Different lower case letters denote significant differences between fractions and whole for each individual COMP type.

	Fraction (mm)	Relative proportion	ADL (%)	ADF (%)	Cellulose (%)	Polyphenols (mg g ⁻¹)
COMP1	Whole	100	18.1 \pm 1.5 ^{Aa}	55.6 \pm 3.9 ^{Aa}	37.5 \pm 2.7 ^{Aab}	11.8 \pm 1.6 ^{Aa}
	1.0–6.0	63.7	25.3 \pm 3.4 ^a	53.1 \pm 5.3 ^a	27.7 \pm 7.0 ^a	10.9 \pm 1.4 ^a
	<1.0	36.3	5.6 \pm 8.6 ^b	60.0 \pm 3.6 ^a	54.6 \pm 11 ^b	13.5 \pm 5.4 ^a
COMP2	Whole	100	12.3 \pm 4.1 ^{Bab}	40.9 \pm 5.1 ^{Bab}	28.6 \pm 2.8 ^{Ba}	21.1 \pm 0.4 ^{Ba}
	1.0–6.0	51.7	16.7 \pm 1.1 ^a	52.1 \pm 1.6 ^a	35.4 \pm 2.6 ^a	18.7 \pm 1.5 ^a
	<1.0	48.3	7.5 \pm 9.6 ^b	28.9 \pm 9.2 ^b	21.4 \pm 4.0 ^a	23.6 \pm 1.8 ^a
COMP3	Whole	100	7.6 \pm 0.3 ^{Ca}	66.3 \pm 11 ^{Ca}	58.8 \pm 11 ^{Ca}	9.38 \pm 0.9 ^{Ca}
	1.0–6.0	56.2	9.7 \pm 3.0 ^a	67.3 \pm 2.9 ^a	57.5 \pm 5.8 ^a	10.6 \pm 0.6 ^a
	<1.0	43.8	4.8 \pm 3.2 ^a	65.2 \pm 26 ^a	60.4 \pm 28 ^a	7.7 \pm 2.3 ^a
COMP4	Whole	100	35.3 \pm 3.6 ^{Da}	69.2 \pm 3.9 ^{Ca}	33.9 \pm 2.9 ^{ABab}	14.6 \pm 1.0 ^{Da}
	1.0–6.0	62.7	45.5 \pm 3.5 ^b	71.5 \pm 1.3 ^a	26.0 \pm 4.8 ^a	10.3 \pm 0.4 ^a
	<1.0	37.3	18.1 \pm 12 ^c	65.4 \pm 8.5 ^a	47.3 \pm 4.6 ^b	21.9 \pm 2.7 ^b
COMP5	Whole	100	19.6 \pm 1.8 ^{Aa}	52.7 \pm 3.4 ^{Aa}	33.0 \pm 2.2 ^{ABa}	10.7 \pm 1.4 ^{ACa}
	1.0–6.0	80.1	38.3 \pm 12 ^b	43.5 \pm 0.3 ^a	28.5 \pm 1.4 ^a	10.8 \pm 0.8 ^a
	<1.0	19.9	15.0 \pm 1.1 ^a	89.5 \pm 16 ^b	51.2 \pm 12 ^b	10.3 \pm 5.7 ^a
COMP6	Whole	100	29.0 \pm 1.1 ^{Ea}	66.7 \pm 4.2 ^{Ca}	37.6 \pm 3.4 ^{Aa}	13.0 \pm 0.8 ^{ADa}
	1.0–6.0	84.8	35.6 \pm 3.1 ^a	67.1 \pm 4.2 ^a	31.5 \pm 3.0 ^a	11.3 \pm 1.4 ^a
	<1.0	15.2	2.0 \pm 3.4 ^b	64.2 \pm 20 ^a	72.2 \pm 30 ^b	22.9 \pm 6.1 ^b
COMP7	Whole	100	31.3 \pm 0.7 ^{DEa}	70.8 \pm 2.0 ^{Ca}	39.4 \pm 1.3 ^{Aa}	12.8 \pm 1.5 ^{ADa}
	1.0–6.0	85.2	39.7 \pm 1.8 ^a	78.4 \pm 1.0 ^a	38.6 \pm 2.4 ^a	13.1 \pm 0.2 ^a
	<1.0	14.8	0.1 \pm 0.2 ^b	27.0 \pm 9.2 ^b	43.9 \pm 5.7 ^a	11.7 \pm 5.6 ^a
MEAN	Whole	100.0	21.9 \pm 10.3	60.3 \pm 11.0	38.4 \pm 9.7	13.5 \pm 3.8
	1.0–6.0	69.2 \pm 14.0	26.8 \pm 13.7	61.8 \pm 12.5	35.0 \pm 10.9	12.3 \pm 3.0
	<1.0	30.8 \pm 14.0	10.9 \pm 13.4	57.2 \pm 22.1	50.1 \pm 15.7	16.7 \pm 6.4

that the others composts. Overall, there were no significant differences in available organic C among the separated fractions, suggesting that the increase in TC observed in the larger fractions is associated with the presence of less soluble and more recalcitrant compounds. In fact, for all the COMPs, the lignin content (ADL) in the <1 mm particle fractions was significantly lower than that of 1.0–6.0 mm fractions, however, for COMP3, differences were not significant (Table 3). There was no clear trend for the ADF and cellulose contents. For some COMPs, there were no significant differences between the <1 mm and 1.0–6.0 mm fractions (COMP1, 3, 4 and 6 and for COMP2, 3 and 7, with ADF and cellulose, respectively). This result was the opposite of that reported by Fanguiero et al. (2007, 2008) who showed that a higher lignin content was found in the finer particle fractions of dairy slurry, probably because the slurries had already been subjected to microbial decomposition in the digestive tract of the animal, considerably changing the apparent structure and composition of the material. The kinetics and efficiency of biological and physico-chemical degradation of composts depend on the organic contents and on the particle size distribution. Typically, hydrolysis rates and particle size are negatively correlated because smaller fractions have a higher surface to volume ratio available for bacterial colonization and reaction (Masse et al., 2005). Thus, during composting the lignin content of small particle sized materials was reduced, whereas larger fractions were not, showing the highest lignin and fiber content after composting due to their low degradability. The relatively high LOI and TC contents of the COMP particles between 1.0 and 6.0 mm, made this fraction an excellent source of organic matter and C, and a useful soil conditioner, which when applied to olive oil farms could improve soil organic matter in those soils characterized by low organic matter contents.

TN concentration tended to increase with decreasing particle size and this was true for all COMPs, except COMP3 (Table 2). Total water soluble inorganic N and inorganic P were significantly higher in the fine fractions (Table 2) for all the COMP samples, especially for fractions smaller than 0.212 mm, although this depended on the particular COMP. For COMP3 and 7 there were no significant differences among separated fractions for TSWIN, and for the other COMP types, only the <0.212 mm fraction showed higher levels

than the other larger sized fractions. Similar to the trend in total N, total P and TSWP, concentrations tended to be higher in the finer fractions. For total P this was true in COMP1, 2, 4, 5 and 7 and for K in COMP1, 4, 5 and 6 (Table 2). Other authors have reported that 93% of the P in swine manure was contained in particles <0.3 mm (Vanotti et al., 2002), or 95% of P in manure is associated with fractions sizes <0.055 mm (Powers et al., 1995). There were no significant differences in K contents between particle fractions and the whole COMP, suggesting that this element was distributed uniformly between the different sized fractions of COMP.

No differences were observed in the polyphenol contents (Table 3), except for COMP4 and COMP6, where there was an increase in the 1.0–6.0 mm fraction.

More than 40% of the COMP mass of total N, P and K was in the <1 mm fractions for COMP1, 2 and 3 which included sheep or poultry manure during composting, whereas for COMP5, 6 and 7, the contribution of <1 mm fractions was lower than 30% (Fig. 1). This highlighted the influence of the raw materials in the final composition of COMP, when a high proportion of manure increased the finer particles with high nutrients contents, whereas a high amount of OLM produced an increase in the larger particles with a high content of different C compounds.

3.4. Carbon and N mineralization

To combine the COMP results, a principal component analysis (PCA) for whole and particle fractions was performed using the analyzed chemical variables. The score values of the PC axis contain information on how the whole COMP and their particle fractions relate to each other, whereas the loading values contain information on how the variables relate to each other. The first PC describes the largest variation in the data, whereas the second PC is constrained to be orthogonal to the first and describes the second largest variation. Fig. 2 shows the scores of the PCA carried out on the seven whole COMP samples and three size fractions (1.0–6.0, 0.212–1 and <0.212 mm). The different proportion of raw materials used during composting of OMP, resulted in diverse types of COMP, which differed in their chemical properties, as well as in the relative proportions of particle size fractions and their

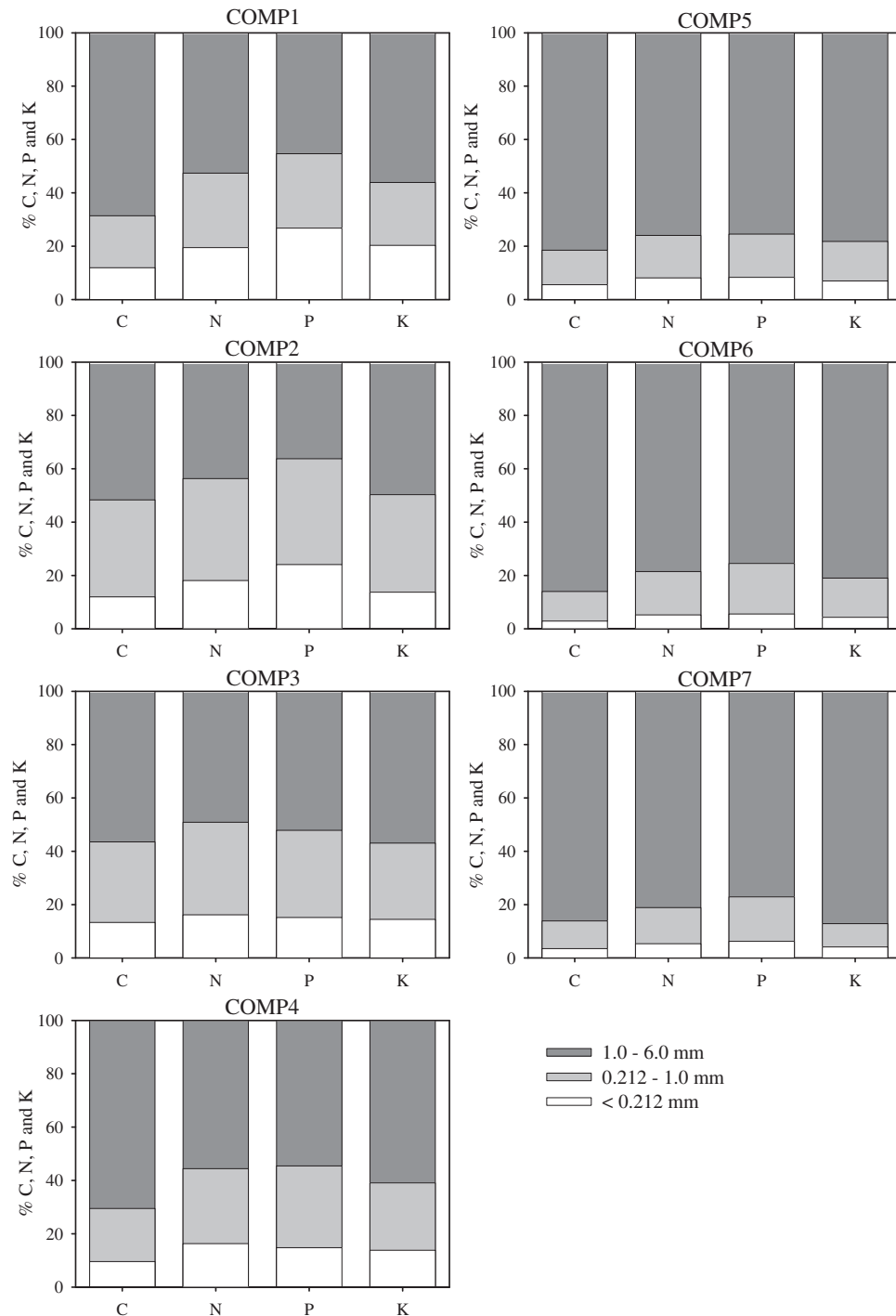


Fig. 1. Distribution of C, N, P and K mass (%) within particle-size fractions of the COMPs.

characteristics. The first PC (PC1), which explained 38.6% of the variability, was significantly negatively correlated with total N ($r = -0.75$; $P < 0.01$) and available N ($r = -0.80$; $P < 0.01$) and P ($r = -0.80$; $P < 0.01$), whereas it was positively correlated with the C:N ratio of the samples ($r = 0.76$; $P < 0.01$). On the other hand, PC2, which explained 26.1% of the data variability was positively correlated with LOI ($r = 0.84$; $P < 0.01$) and total C ($r = 0.77$; $P < 0.05$). Thus, those whole COMPs and size fractions with relatively high TN and available N and P, but with low C:N ratios, were located in the negative range of PC1, which suggested that this axis was related to COMP quality in terms of nutrients, and sorted the COMPs according to the pool of nutrients and organic matter and

C. This showed the high influence of raw materials in the quality of individual COMPs. COMP2 and COMP7, both made with poultry manure, were located to the far left of PC1 (e.g. COMPs with high TN, and available P and N contents and a relatively low C:N ratio), resulting in a high quality product. COMP3, made with a relatively high proportion of sheep manure, was also in the negative portion of the PC1, although close to zero, and finally, COMP1 and COMP4, made with only a small proportion of sheep manure (<13%), and COMP5 and 6, without manure, were positioned in the positive values of PC1 (e.g. low in TN and with a high C:N ratio), suggesting the low quality of these COMPs. In addition, there was a tendency for an increasing quality of COMP in the finer fractions. The following

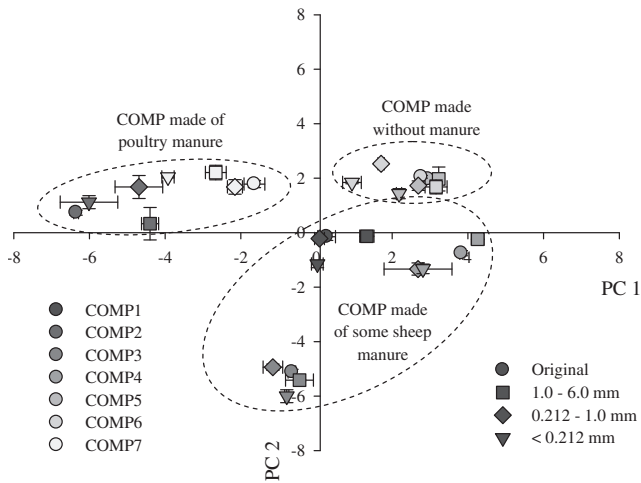


Fig. 2. Scores of the whole and the particle size fractions of the COMPs in the space defined by PC1 and PC2.

data analysis and presentation are based on this ranking of the COMPs.

C:N ratio increased significantly with increasing particle size, ranging 14.1–44.6 for 1.0–6.0 mm particles and 7.9–23.9 for fractions smaller than 0.212 mm (Fig. 3). Similar relationships between C:N ratio and particle size fractions were obtained by Fanguero et al. (2008) for cattle slurry and Doublet et al. (2010) for sludge compost. Typically, C:N for particles between 0.212 and 1 mm was lower than 18.2 in all COMPs, except for COMP4 and COMP5. Beauchamp and Paul (1989) suggested that during decomposition of manures, with C:N ratios below 15, are likely to result in positive (net) N mineralization after application to soil.

Potential N mineralization (PNM) is used by some authors as an index of short-term N availability (Griffin and Laine, 1983; Serna and Pomares, 1992). In our study PNM in fractions <0.212 mm were significantly higher than the 0.212–1 and 1.0–6.0 mm fractions in three of the four COMP combinations, being as much as three times higher in some cases. For COMP5 and COMP6 types, which were composed only of OMP and OLM, PNM was the lowest and there were no differences among particle size fractions (Fig. 3).

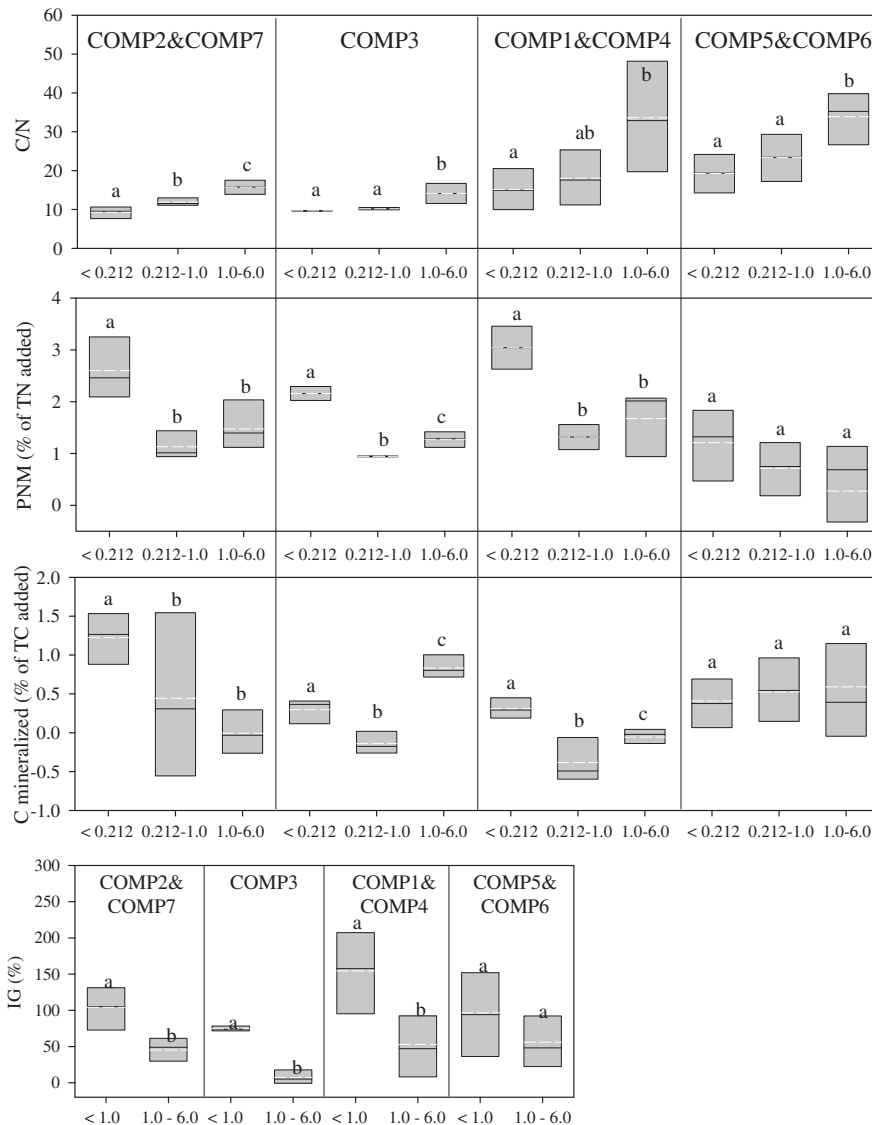


Fig. 3. Box-plot of C:N, potential mineralizable N (PNM), mineralizable C and germination index of particle size fractions of combinations of COMPs according to the Principal Component Analysis. Different letters stand for significant differences ($P < 0.05$) among particle size fractions.

The tendency of increasing the pool of N availability in the short-term in the finer fractions, agrees with the findings of Aoyama (1985) for manure and city refuse composts, Fanguiero et al. (2007, 2008 and 2010) for animal slurry and Doublet et al. (2010) for sludge compost, who observed that various short-term N availability forms were in the finer and water-soluble fractions. Thus, the finest fractions of the COMP largely contribute to the potential availability of N in the various COMPs.

The percentage of COMP derived C which was respired was very low for all the COMP types and was always lower than 1.5% and well below the ranges reported by Ajwa and Tabatabai (1994) for plant materials, animal manure and sewage sludge (21–62%), or by Bernal et al. (1998) for several composts made with manure, organic wastes and olive mill wastewater. The very low C degradability of the COMPs is indicative of the refractory nature of this type of compost. Indeed there are indications that, during the composting of OMP together with olive leaf remains, the structural and chemical properties of the fulvic acid fraction is modified favorably to provide complex stability (Plaza et al., 2007), increasing the degree of aromatic ring polycondensation, polymerization and humification. Moreover, Wu et al. (2000) suggested that the water soluble C is the most active fraction of C and is indicative of compost decomposability and stability. The whole COMP and the different fractions showed relatively low water soluble C contents (<1.5%) which might explain the very low C mineralization of the COMPs. The very low C mineralization of the COMP is a property that could have important consequences from a practical point of view for the storage and sequestration of organic C in soils, as a relatively large proportion of the COMP organic C remains in non-decomposable forms. This finding, obtained under laboratory conditions, would suggest that the evaluation of the actual soil C sequestration efficiency at the field scale of composted olive mill pomace would be of value.

There was no clear trend in C mineralization in the different size fractions. For COMP2 and COMP7 and COMP1 and COMP4, the highest percentage of mineralized C from that added was found with the finest particles (<0.212 mm), whereas that of the 1.0–6.0 mm fraction was close to zero (e.g. similar to the control). In contrast, C mineralization was highest for the fractions 1.0–6.0 mm for COMP3, and no differences among fractions were found for COMP5 and COMP6 (Fig. 3). This result disagrees with the findings of Doublet et al. (2010) who found that C mineralization of coarse fractions are typically lower than the finer fractions because the former are characterized by intrinsically low biodegradability with the lowest available surface area for colonization by the decomposer microorganisms. On the other hand, it has also been reported that the finer fractions might also show low C mineralization because these are partly transformed to more stabilized organic C during composting, resulting in relatively low decomposability. These two mechanisms, together with the variability in the raw materials and proportions of the initial mixing, might explain the lack of a clear trend in C mineralization related to particle size.

Overall, the germination index was higher than 80% (e.g. lack of phytotoxicity) in the fractions <1.0 mm, whereas it was lower than 50% (e.g. with some phytotoxicity) for fractions 1.0–6.0 mm, although this was not true for the COMP5 and six mixtures (Fig. 3). Thus, the fractionation of the compost produced in olive mills obtaining <1 mm phytotoxicity-free fractions is highly recommended for commercial purposes.

4. Conclusions

The separation and application of different COMP particle sizes could be advantageous for better optimization of COMP

management considering that the particles 1.0–6.0 mm have higher values for GI, total N, P and K and potentially mineralizable N, providing a higher quality compost. Alternatively, the larger particles had higher contents of organic matter and carbon and tended to have the lowest C mineralization and thus their application to olive groves as a soil conditioner could improve the structure and increase the organic matter in the these poorer soils and increase the storage of soil organic C.

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