

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

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25
26 **Abstract.**

27
28 Nowadays, the main concern for the Spanish olive oil industry is the disposal of
29 the huge amounts (about 5 Mt in the last olive oil campaign, 2010-2011) of the two-
30 phase olive-mill waste or “alperujo” (AL) that it produces. Here, we have studied the
31 technical feasibility of using composting to transform AL into commercial organic

32 amendments and fertilisers. For this, six piles of 20 t each, prepared by mixing AL and
33 either poultry (PM) or sheep manure (SM), were composted. In addition, Fe and P-
34 enriched acidic mineral amendments were added at the beginning of the process in order
35 to reduce alkalinisation and nitrogen losses. As composting proceeded, substantial
36 organic matter degradation, progressive humification, increases of pH and germination
37 indices and decreases in fats and water-soluble organic fractions were detected. Also,
38 the addition of the mineral amendments was effective with regard to reducing the pH,
39 water-soluble organic carbon and polyphenols, hemicellulose fraction and nitrogen
40 losses. According to the Spanish legislation for fertiliser production (PRE/630/2011),
41 the composts obtained were suitable for preparing commercial organic amendments and
42 fertilisers within categories such as *AL Compost*, *Humic Organic Amendment*, *Compost*
43 *Organic Amendment* and *NPK Organic Fertiliser*. Also, by mixing with organic and/or
44 mineral fertilisers, the composts were optimal substrates for the production of 19 types
45 of *Organo-mineral Fertilisers*, either as solid or liquid formulations.

46

47 **Keywords:**

48 Commercial organic fertilisers, composting, olive mill wastes, “alperujo”, poultry and
49 sheep manures.

50

51 **1. Introduction.**

52

53 The agrofood industry produces a great variety of organic wastes that potentially
54 can be used as soil fertilisers and amendments due to their high contents of organic
55 matter (OM) and plant nutrients (Martínez-Blanco et al., 2011). A clear example of this
56 issue is the olive oil industry, which has a relevant economic and social importance in
57 Mediterranean countries and produces a huge amount of wastes, depending on the
58 extraction system applied. As the two-phase centrifugation system is the main one used
59 by the Spanish industry, the major concern for this sector is the high production of
60 “alperujo” (AL) and its disposal (Alburquerque et al., 2004). AL is an acidic and very-
61 wet solid olive-mill waste, containing phenolic and lipidic fractions which are related to
62 phytotoxic and antimicrobial effects when AL is used as a soil amendment. At present,
63 AL is usually treated with a second centrifugation, or dried and then subjected to
64 chemical extraction, to extract the residual oil. Finally, the resulting wastes are used to
65 obtain thermal or electrical energy for sale or for use in the olive mill, mainly through
66 combustion of the dried wastes although they are also considered valuable materials for
67 co-firing with coal in power utilities (Gogebakan and Selçuk, 2009).

68

69 Alternative methods such as composting, considered an appropriate low-cost
70 technology for organic waste recycling and organic fertiliser production
71 (Arvanitoyannis and Varzakas, 2008; Ruggieri et al., 2009), may well represent an
72 acceptable solution for AL disposal, adding value to this waste and enhancing the
73 sustainability of the olive oil production system (Vlyssides et al., 2004; Salomone and
74 Ioppolo, 2011). The AL composting process has been evaluated by studying several
75 factors at both the pilot plant (Madejón et al., 1998; Alburquerque et al., 2006a; Cegarra
76 et al., 2006; Alfano et al., 2008) and industrial scale (Filippi et al., 2002; Cayuela et al.,

77 2004; Sánchez-Arias et al., 2008). These studies focused mainly on the composting
78 aeration system employed (mechanical turnings, forced ventilation or a combination of
79 both) and the bulking agents added to improve the deficient physical properties of AL.
80 Animal manures, such as those of poultry (PM) and sheep (SM), seem to be appropriate
81 and effective bulking agents and also show an inoculant effect and add nutrients,
82 especially available nitrogen compounds (~~Solano et al., 2001;~~ Kelleher et al., 2002;
83 Cayuela et al., 2004; Alfano et al., 2008; Canet et al., 2008). However, some
84 disadvantages related to AL composting and the agricultural use of AL composts should
85 be taken into account. Some examples are: nitrogen losses during the process, alkaline
86 compost pH and an unbalanced nutrient content. In several composting experiments,
87 inorganic amendments rich in different elements—~~such as Fe, P, Mg, Ca, Al, K and Na~~
88 - were employed, which reduced the pH and ammonia volatilisation significantly
89 (~~Witter and Kirchmann, 1989; Moore Jr. et al., 1995 and 1996;~~ Kithome et al., 1999;
90 ~~Mc Crory and Hobbs, 2001;~~ Koenig et al., 2005). Also, S^o and Fe-enriched acidic
91 mineral amendments were employed satisfactorily in AL composting (Roig et al., 2004;
92 Canet et al. 2008; Sánchez-Arias et al. 2008). However, the addition of such materials
93 can increase substrate salinity and reduce compost quality.

94

95 The recycling of organic wastes for agricultural purposes is crucial in order to
96 sustain soil productivity in Mediterranean areas, where the soil OM content is very low
97 (~~Martinez-Mena, 2002;~~ Lasaridi et al., 2006; Ruggieri et al., 2009; Martínez-Blanco et
98 al., 2011). Therefore, composting may act as an appropriate disposal option for
99 biodegradable wastes. However, compost of satisfactory quality is required to establish
100 a fertiliser market, which could promote an efficient recycling strategy for AL. Several
101 countries have developed their own quality criteria for composted materials (ECN-QAS

102 in Europe, STA and CCQC in the USA, RAL in Germany, BSI PAS 100 in the UK,
103 CIC in Italy, etc.). Thus, AL composts with an acceptable nutrient content could be
104 obtained and both the Spanish legislation for fertilisers (PRE/630/2011) and the 2nd
105 draft of the Biological Treatment of Biowaste (European Commission, 2001) should be
106 considered as the best references for assessment of their quality and potential as
107 commercial products. Their high OM content and the possibility of obtaining the
108 European eco-label (European Commission, 2010) as *Soil Improvers*, due to their low
109 content of heavy metals, make their commercialisation attractive, as mentioned by
110 Canet et al. (2008).

111

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

115

116 **2. Materials and methods.**

117

118 **2.1. Composting methodology.**

119

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

124

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

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

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

128

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

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

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

132

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

140

141 A minimum of 30 sub-samples per pile were taken, mixed and homogenised.
142 This sample was considered representative of the composting substrates and later it was
143 divided into three fractions in the laboratory: the first was frozen (-20 °C) and kept for
144 the determination of NH_4^+ and NO_3^- , the second was dried in an oven at 105 °C for 24
145 hours, to determine its moisture content, and the third was freeze-dried and ground to
146 less than 0.5 mm prior to analysis. The water-soluble organic carbon (WSC) was
147 determined, using an automatic analyser for liquid samples (Shimadzu TOC Analyser),
148 for 1:20 (w/v) water extracts produced by shaking for 2 hours. The NH_4^+ was
149 determined using a colorimetric method based on Berthelot's reaction (Kempers and
150 Zweers, 1986; Sommers et al., 1992), NO_3^- was measured by HPLC after 1:20 (w/v)
151 water extraction and the phytotoxicity was determined using the germination index (GI)

152 test with cress (*Lepidium sativum*) and radish (*Raphanus sativus*) seeds (Zucconi et al.,
153 1981; Satisha and Devarajan, 2007). The macro and micronutrients (P, K, Ca, Mg, Na,
154 S, Fe, Cu, Mn, Zn) and heavy metals were determined by Inductively Coupled Plasma
155 Optical Emission Spectrometry (ICP-OES) after microwave digestion. The humification
156 indices - humification ratio (HR), humification degree (HD) and percentage of humic
157 acids (P_{HA}) - were calculated according to Albuquerque et al. (2009). Other parameters,
158 including electrical conductivity (EC) and pH, OM, total nitrogen (T_N), total organic
159 carbon (T_{OC}), total fat content, water-soluble phenolic substances (WSPH), water-
160 soluble carbohydrates (WSCH), lignin, cellulose and hemicelluloses, were determined
161 according to methods described previously (Albuquerque et al., 2004; Cegarra et al.
162 (2006). Losses of total OM, lignin, cellulose, hemicellulose and T_N were calculated by
163 taking into account the apparent increase in the ash content resulting from the loss of
164 dry weight, in order to reflect better the overall changes (Viel et al., 1987; Paredes et al.,
165 1996).

166

167 **2.2. Raw material characterisation.**

168

169 For the composting experiments, AL was collected from “Aceites Guadalentín
170 S.L.”, an olive mill in Jaén (Spain), and the bulking agents (poultry: PM, and sheep
171 manure: SM) from farms near the composting plant. The AL showed high moisture
172 (55.8%), fat and OM contents, the latter composed mainly of lignin (344.7 g kg^{-1}),
173 acidic pH and moderate nitrogen (poor in inorganic forms), P and micronutrient
174 contents (Table 1). With respect to the bulking agents, the PM had a higher OM content
175 than the SM, as well as higher nitrogen content (about 20% as ammonium), while the
176 SM had higher electrical conductivity (EC) and Ca and Fe contents (Table 1). Rice

177 husk, used as animal bedding, constituted an important proportion of the PM. The
178 addition of two acidic mineral additives to the composting mixtures was evaluated also,
179 with regard to their ability to enrich the end-products of composting in essential plant
180 nutrients (Fe and P), reduce the alkalisation resulting from the composting process and
181 improve the agronomic potential of the composts by reducing the nitrogen losses during
182 the process. The Fe-enriched mineral amendment used was a by-product of titanium
183 oxide synthesis (Sanchez-Arias et al., 2008), whereas the P-enriched mineral was a
184 commercial fertiliser (GSSP, Granular Simple Super Phosphate, ICL Fertilisers
185 company).

186

187 **2.3. Statistical analyses.**

188

189 A descriptive statistical analysis (the mean and the absolute and relative errors)
190 was calculated for each pile. Also, we performed some inferential statistical analyses
191 such as the analysis of variance (ANOVA) within treatments (piles and time), assuming
192 a normal distribution of the data and homoscedasticity. For *post-hoc* analysis, we used
193 the Tukey test ($p < 0.05$) and the Least Significant Difference (LSD) ($p < 0.05$), to
194 determine changes in the analysed parameters for each pile with time. All these analyses
195 were calculated using the SSPS 17.0 program for Windows XP. Another inferential
196 statistical analysis was a regression study of some of the experimental data, especially
197 OM losses. We fitted them to two models: zero-order and first-order kinetic models,
198 with important correlation coefficients. This analysis was performed with SigmaPlot
199 2000 software for Windows, Version 10.0.

200

201 **3. Results and discussion.**

3.1. Composting process evolution.

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

Figure 1 shows acidic pH values at the beginning of the process, which increased during the thermophilic phase and reached their highest values at the end of the 20th week (8.9 and 9.5 for the AL+PM and AL+SM piles, respectively). Later, all piles showed a slight decrease until the end of composting, that led to final average values of 8.1 and 8.6 for the AL+PM and AL+SM piles, respectively. This behaviour is observed commonly in AL composting (Cayuela et al., 2006; Cegarra et al., 2006), with end-material pH values higher than 8.5.

226 The addition of the acidic amendments led to decreases in pH along the
227 composting process (around 0.5 pH units, Figure 1). Some amendments have been
228 employed successfully to control the evolution of pH in AL composting. Roig et al.
229 (2004) found that the pH decreased by 1.1 units when S^o was added during the curing
230 period of the composting process, while Mari et al. (2005) obtained similar results by
231 adding the same amendment at the end of the thermophilic phase. Finally, Sánchez-
232 Arias et al. (2008) strongly decreased the pH values of AL composts by adding 20%
233 (dry weight basis) of the same Fe mineral amendment used in our experiment; this
234 reduced the biological influence on the organic matter oxidation and increased the
235 chemical oxidation.

236

237 The T_N content of all piles increased during composting (Table 2), from initial
238 values of 18.4 and 15.5 g kg⁻¹ to final ones of 20.6 and 18.9 g kg⁻¹ for the AL+PM and
239 AL+SM piles, respectively. In the former, the T_N content was higher, due to the higher
240 nitrogen content of the PM (32.3 g kg⁻¹, compared to 17.7 g kg⁻¹ for SM). During the
241 thermophilic phase, the AL+PM piles showed an initial decrease in T_N content. This
242 could be explained by the initial NH₄⁺ content in the AL+PM piles: 1,742 mg kg⁻¹
243 (much higher than the 317 mg kg⁻¹ of the AL+SM piles), which decreased sharply
244 during the process (Figure 2). In our experiment, the T_N losses should be attributable
245 largely to NH₃ volatilisation (especially in the piles with PM). This phenomenon is
246 observed commonly in PM-composting experiments (Mahimairaja et al., 1994; Koenig
247 et al., 2005) and can be related to the pH evolution (fast substrate alkalisation during
248 AL composting) that transforms NH₄⁺ into volatile NH₃, which can provoke important
249 environmental problems during PM disposal (Mahimairaja et al., 1994; Mondini et al.,
250 1996). Also, the NH₃ volatilisation effect was noted in the evolution of the T_{OC}/T_N ratio,

251 which showed an initial increase in the AL+PM piles followed by a decrease after the
252 20th week, until the thermophilic phase (Table 2).

253

254 The T_N content of AL composts is within the range 16.6-26.2 g kg⁻¹, as shown
255 previously (Cayuela et al., 2004; Albuquerque et al., 2006a and Cegarra et al., 2006).
256 The addition of the acidic mineral amendments rich in Fe and P reduced the T_N losses.
257 At the end of the process, AL+PM+Fe and AL+PM+P exhibited T_N losses of 31.9 and
258 31.6%, respectively, lower than for AL+PM (43.1%); also, AL+SM+Fe and AL+SM+P
259 had T_N losses of 29.4 and 20.9%, respectively, compared to 32.4% for AL+SM. Acidic
260 mineral amendments have been used widely to control the pH during composting and to
261 reduce nitrogen losses. Hence, Koenig et al. (2005) tested different amendments
262 intended to control ammonia losses during composting, obtaining good results with
263 respect to the progression of pH. Also, Ekinci et al. (2000), composting short paper
264 fibre with broiler litter, demonstrated that ammonia volatilisation was lower when the
265 pH of the composting substrate was around 7; it increased considerably with
266 alkalinisation pH (≥ 8).

267

268 The initial WSC values depended on the bulking agent and the proportion added
269 to the AL. Hence, piles made with PM showed values close to 55 g kg⁻¹ while SM piles
270 had values around 41 g kg⁻¹ (Table 2). The WSC decreased during the process in all
271 piles, when the thermophilic phase was more intense. At the end of the process, the
272 AL+PM and AL+SM piles exhibited values, in the order of 17 and 12 g kg⁻¹,
273 respectively, similar to that recommended by Bernal et al. (1998) as a maturity index for
274 composts of different sources. Canet et al. (2008) obtained final WSC values of 11 and
275 10 g kg⁻¹ after composting AL with SM or rabbit manure, respectively. Albuquerque et

276 al. (2006a) reported final values around 30 g kg^{-1} after composting AL with several
277 bulking agents. Their higher values may be related to the lesser amount of bulking agent
278 employed: AL comprised 86 % (dry weight basis) compared to about 50% (Canet et al.,
279 2008) or 37 and 57% in our experiments.

280

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

295 The WSCH developed in a rather similar way to WSC (Table 2), reaching end-
296 values close to 3 and 1 g kg^{-1} for the AL+PM and AL+SM piles, respectively. Previous
297 authors have suggested WSCH as the main carbon source for the microflora responsible
298 for the initial degradation phase (thermophilic biodegradation). Sánchez-Monedero et
299 al. (1999) evaluated different composting mixtures prepared with municipal solid waste
300 and noted that the WSCH fell sharply during the first three weeks of composting (by >

301 60% of the initial values). Also, Albuquerque et al. (2006a), who composted AL with
302 grape stalks and olive leaves, showed that the WSCH decreased during the process, to
303 reach final values of 10 g kg^{-1} .

304

305 Other important water-soluble organic compounds used to evaluate the maturity
306 of AL compost are the WSPH, which are related to the phytotoxic properties of the
307 olive-mill wastes (Ait-Baddi et al., 2003; Albuquerque et al., 2006b; Sampedro et al.,
308 2008). In our case, the WSPH decreased in all piles during composting, reaching similar
309 end-values close to 2 g kg^{-1} (Table 2). It should be added that WSCH and WSPH are
310 liberated also by the degradation of lignocellulosic materials, leading to their increased
311 concentrations in the water phase which then decrease as a consequence of microbial
312 activity. As mentioned before, the degradation pathway prevailed in our case, since a
313 continuous decrease occurred during the composting process. Also, Sánchez-Monedero
314 et al. (1999) reported that decreases in the WSPH were inversely and strongly correlated
315 with the main humification indices, suggesting that these compounds could act as
316 precursors for the humification process during composting.

317

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

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

329

330 The main components of the AL organic substrate (lignin, cellulose,
331 hemicelluloses and fats) were also studied during composting (Table 2). The relative
332 contents of lignin and cellulose increased during the process (especially in the
333 thermophilic phase) while hemicellulose showed the opposite behaviour, a decrease.
334 The two acidic mineral amendments decreased the lignin losses by 8 and 16% compared
335 to the controls: the non-amended mixtures AL+PM and AL+SM, respectively. The
336 cellulose losses showed a similar behaviour. Only the hemicellulose losses increased
337 when the acidic amendments were used, by about 7 and 4% relative to the non-amended
338 mixtures. The decreases in the biodegradation rates of the lignin and cellulose fractions
339 could have been due to the chemical affinity of the Fe and P amendments for certain
340 functional groups in the lignocellulosic matrix of the organic matter, as remarked upon
341 by other authors. Merdy et al. (2002a) found that the Fe³⁺-lignin complex showed
342 certain stability, lignin being a good adsorbent of this nutrient in calcareous soils where
343 it is not available to plants. Merdy et al (2002b) studied the interaction of both Fe and
344 Mn with the lignocellulosic fractions from barley straw, showing redox process
345 involving Fe and quinone-like structures on the lignin surface. Also, Karthikeyan et al.
346 (2004) used solid wood residues to reduce the orthophosphate content in waste water,
347 due to ionic change process and other Lewis interactions between orthophosphate and
348 the wood lignocellulosic fraction.

349

350 Another important organic fraction of AL is its high residual fat content

351 (Albuquerque et al., 2004), which should be monitored during composting. Here, the
352 initial high fat contents of the two groups of mixtures (75 and 55 g kg⁻¹ in the AL+PM
353 and AL+SM piles, respectively) decreased sharply during the first three months of
354 composting to reach end-values close to 2 g kg⁻¹ (Figure 2), a phenomenon common in
355 AL composting studies (Madejón et al., 1998; Albuquerque et al., 2006b).

356

357 Humic parameters, such as the humification indices, have been employed as
358 quality criteria for AL composting (Tomati et al., 2001; Albuquerque et al., 2009).
359 Table 3 shows that HD and P_{AH} increased during AL composting in all piles due to the
360 incorporation of OM into the humic fraction (HA and FA-like). During the early
361 composting phase, the HR decreased considerably since non-humic materials were
362 initially co-extracted in the alkali-soluble fraction. At the end of the process, the
363 AL+SM piles generally showed higher HR, HD and P_{HA} values than the AL+PM piles
364 (29.8, 75.7 and 68.1% versus 20.0, 70.8 and 63.2%, respectively), related to the more-
365 easily-degradable organic matrix of the AL+SM piles which could have enhanced the
366 humification of the substrate, in accordance with the OM losses discussed previously.

367

368 Phytotoxicity is one of the most-important criteria for evaluating the suitability
369 of organic materials for agricultural purposes (Zucconi et al., 1981; Bernal et al.,
370 1998). All piles showed a clear increase in the GI during the first three months of
371 composting (Table 2) and reached end-values typical of non-phytotoxic materials (81
372 and 78% for the AL+PM composts and 76 and 96% for the AL+SM composts, for the
373 cress and radish GI, respectively). These increases in the GI could have been related to
374 the decreases observed for substances with phytotoxic properties: ammonium, WSPH
375 and fats Albuquerque et al. (2006b).

376

377 **3.2. Agrochemical characteristics of the AL composts relevant to the**
378 **production of commercial organic amendments and fertilisers.**

379

380 The mature composts showed alkaline pH and low EC values (Table 1), in
381 agreement with the results reported by Roig et al. (2004), Sanchez-Arias et al. (2008)
382 and Canet et al. (2008), while the total and mineral nitrogen contents were slightly
383 higher in the AL+PM composts than in the AL+SM composts. The total OM content
384 was high in both types of compost (750.6 and 573.8 g kg⁻¹, respectively), as was the
385 lignin content; around 50% of the total organic content. The water-soluble organic
386 fraction was low, as indicated by the values of WSC, WSCH and WSPH, but slightly
387 higher in AL+PM composts than in AL+SM composts. All the composts had
388 humification indices which characterised them as humified materials. The heavy metal
389 content was, in general, low and did not produce any phytotoxic effects, according to
390 the GI values of cress and radish (> 70%).

391

392 According to the new Spanish legislation for fertilisers (PRE/630/2011), these
393 composts could be used in several commercial categories, such as *AL Compost* (OM >
394 45%, moisture > 40%, T_{OC}/T_N < 20 and total polyphenols < 0.8%), *Humic Organic*
395 *Amendment* (OM > 25%, 5% in the total humic extract and 3% in humic acids),
396 *Compost Organic Amendment* (OM > 35% and T_{OC}/T_N < 20) and *NPK Organic*
397 *Fertiliser* (T_{OC}/T_N < 15 and N+P₂O₅+K₂O > 4%, more than 1% of each nutrient).
398 According to the minimum requirements, the six composts were defined as *AL*
399 *Compost*. The AL+PM and AL+SM composts could be used to obtain the categories
400 *Humic Organic Amendment* and *Compost Organic Amendment*, while only *NPK*

401 *Organic Fertiliser* could be made with the AL+SM compost, due to the high T_{OC}/T_N
402 value of the AL+PM compost, mainly because of the different evolution of the OM
403 during composting of this pile and the rice husk content of PM, not for the content of
404 nutrients. Another category described in the Spanish legislation is *Organo-mineral*
405 *Fertilisers*, made by mixing organic fertilisers with other organic and/or inorganic
406 fertilisers. Using AL+SM composts as an organic fertiliser (*NPK Organic Fertiliser*), it
407 could be possible to prepare at least 19 types of commercial organic fertiliser, in solid
408 and liquid forms.

409

410 **3.3. Cost of the composting process**

411

412 For the industrial production of marketable products, it is necessary to check the
413 economical viability of the process. In our experimental conditions, the total cost to
414 produce 60 t of compost was around 2,150€ (36€ per t of compost obtained or 31€ per t
415 of AL treated). We have identified the following operational costs:

416

417 1. Raw material supplied. According to the composting performance we
418 prepared six composting piles of 20 t each (120 t in total), using 69.6 t of AL, 29.0 t of
419 PM, 20.6 t of SM, 0.4 t of Fe- and 0.4 t of P-enriched materials. The AL was supplied
420 by the olive mill without cost. The manures and the mineral acidic amendments cost
421 around 1,050€ in total (including transport).

422

423 2. Composting equipment. The composting experiments were carried out in
424 an outdoor facility close to the olive mill and dedicated to AL storage (without any
425 additional cost). A backhoe loader was used for preparing the composting mixtures and

426 turning the composting piles, spending 25 working hours in total (240€).

427

428 3. Labour cost. Two men were necessary to carry out the composting

429 experiment. This required close to 35 working-hours, with an approximate cost of 630€.

430

431 4. Water consumption. An aspersion system was necessary to keep the

432 moisture content in the piles around 40%, with a cost of 200€. The total water

433 consumption was estimated to be 40 m³, with an approximate cost of 30€.

434

435 The estimated cost for AL composting in our experiment was lower than the 37

436 and 55€ per t of AL treated for industrial composting processes in Italy and Spain,

437 respectively (Cubero et al., 2011). This difference must arise from the amortization

438 costs included in the latter processes (construction and maintenance of installations,

439 composting machine, etc.), as well as to the different bulking agents (type and

440 proportion) and composting technology used.

441

442 **4. Concluding remarks.**

443

444 This study revealed that composting is an effective technology for adding value

445 to AL through its transformation into commercial organic amendments and fertilisers.

446 The manures assayed in our experiment acted as appropriate co-substrates for AL

447 composting and the mechanical turnings applied were enough to aerate the piles,

448 homogenising the substrate simultaneously. Several parameters were important for the

449 control and monitoring of the composting process, such as temperature, moisture, pH,

450 water-soluble fractions, OM losses and germination index. The addition of the Fe and P-

451 enriched acidic mineral amendments reduced the pH values (by around 0.5 units during
452 the process) and also the WSC, WSPH and hemicellulose contents.

453

454 The composts obtained lacked phytotoxicity and had valuable contents of
455 nutrients and high contents of OM with humic properties and predominance of the
456 lignocellulosic matrix. According to the new Spanish legislation for fertilisers
457 (PRE/630/2011), these composts satisfied the minimal requirements for commercial
458 categories such as *AL Compost*, *Humic Organic Amendment*, *Compost Organic*
459 *Amendment* and *NPK Organic Fertiliser*. These AL composts can be mixed with other
460 fertilisers to obtain new, valuable solid and liquid formulations. In addition, *in-situ*
461 composting of AL can be considered viable from an economic point of view, according
462 to our results.

463

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465

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469

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652

653

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

<i>Parameters¹</i>	<i>AL</i>	<i>PM</i>	<i>SM</i>	<i>Mature composts</i>					
				<i>AL+PM</i>	<i>AL+PM+Fe</i>	<i>AL+PM+P</i>	<i>AL+SM</i>	<i>AL+SM+Fe</i>	<i>AL+SM+P</i>
pH²	5.38	7.50	8.51	8.30	7.97	8.02	8.88	8.52	8.35
EC² (dS m⁻¹)	4.64	8.47	11.33	1.70	1.84	2.44	1.69	1.70	2.03
OM (g kg⁻¹)	907.9	805.5	456.5	745.6	760.3	746.0	561.2	572.1	588.2
Lignin (g kg⁻¹)	344.7	129.7	211.4	428.2	467.4	457.2	350.6	375.0	374.1
Cellulose (g kg⁻¹)	178.7	149.9	113.6	176.6	182.8	178.6	146.1	179.2	188.1
Hemicellulose (g kg⁻¹)	350.3	307.0	109.7	208.7	152.2	129.1	131.0	92.0	104.0
T_{OC} (g kg⁻¹)	486.0	398.4	251.6	396.6	403.1	393.2	251.9	272.3	288.4
T_N (g kg⁻¹)	13.4	32.3	17.7	20.2	20.6	21.1	18.6	19.2	19.1
NH₄⁺ (mg kg⁻¹)	63	5,915	889	141	163	203	46	60	71
NO₃⁻ (mg kg⁻¹)	16	19	520	23	37	44	6	23	19
T_{OC}/T_N	36.3	12.3	14.3	19.5	19.5	18.7	13.5	14.2	15.0
Fat content (g kg⁻¹)	99.2	14.9	5.1	2.1	2.1	4.1	2.1	2.1	2.1
WSCH (g kg⁻¹)	19.7	20.9	4.5	3.2	2.0	4.2	1.5	2.2	1.2
WSPH (g kg⁻¹)	9.0	9.3	3.3	2.0	2.1	3.4	2.1	1.1	1.3
WSC (g kg⁻¹)	86.5	67.8	35.4	19.4	13.5	19.3	16.4	10.6	11.7
P (g kg⁻¹)	0.8	2.2	2.2	2.0	1.9	2.2	1.8	1.8	2.0
K (g kg⁻¹)	10.4	13.5	16.5	11.0	7.9	6.5	12.8	12.7	10.2
Ca (g kg⁻¹)	8.0	47.5	100.9	29.4	20.1	18.7	65.0	72.4	65.2
Mg (g kg⁻¹)	3.1	5.5	18.7	5.7	4.9	4.9	12.7	10.6	10.3
Na (g kg⁻¹)	0.3	4.1	3.9	8.3	6.0	8.2	7.6	6.2	6.2
S (g kg⁻¹)	1.1	4.0	13.2	2.5	3.6	4.6	5.2	5.4	5.4
Fe (g kg⁻¹)	2.4	1.9	4.1	3.9	9.0	4.8	6.5	11.2	6.0
Cu (mg kg⁻¹)	22	29	51	32	22	27	16	14	19
Mn (mg kg⁻¹)	56	322	226	183	193	145	167	199	131
Zn (mg kg⁻¹)	17	79	185	92	54	39	41	55	49
Pb (mg kg⁻¹)	4	4	12	8	30	37	11	17	24
Cr (mg kg⁻¹)	19	23	19	14	12	13	24	22	28
Ni (mg kg⁻¹)	55	49	25	21	29	35	50	42	80
Cd (mg kg⁻¹)	nd	nd	nd	nd	2	7	nd	1	2

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

¹Data based on dry weight. ²Water extract 1:10. EC: electrical conductivity, OM: total organic matter, T_{OC}: total organic carbon, T_N: total nitrogen, WSCH: water-soluble carbohydrates, WSPH: water-soluble phenols, WSC: water-soluble organic carbon, and nd: not detected.

Table 3. Evolution of the humification ratio (RH), humification degree (HD) and the percentage of humic acids (P_{HA}) during composting (AL: “alperujo”, PM: poultry manure, SM: sheep manure, Fe: iron-enriched mineral amendment and P: phosphorous-enriched mineral amendment).

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

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

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

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

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

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

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

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

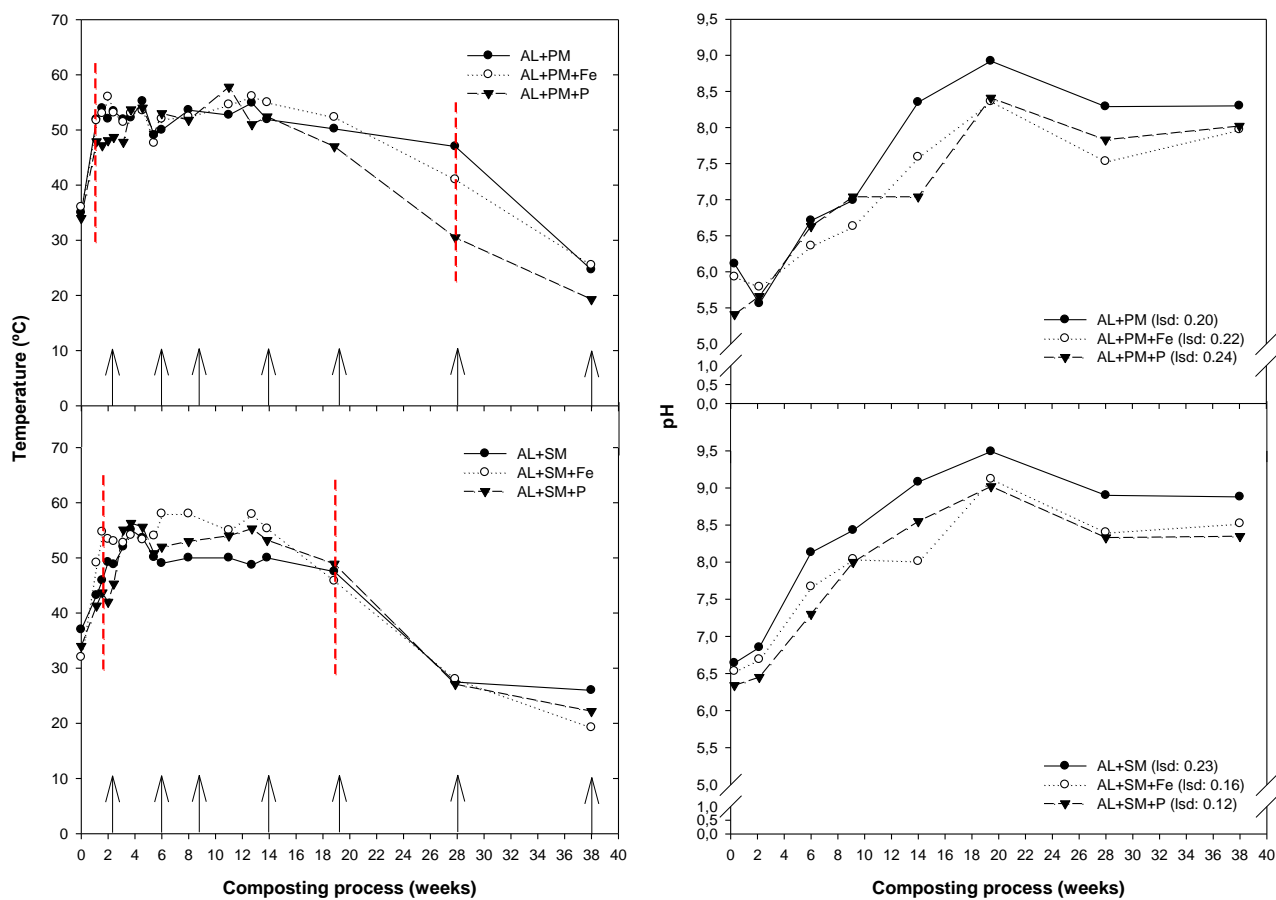
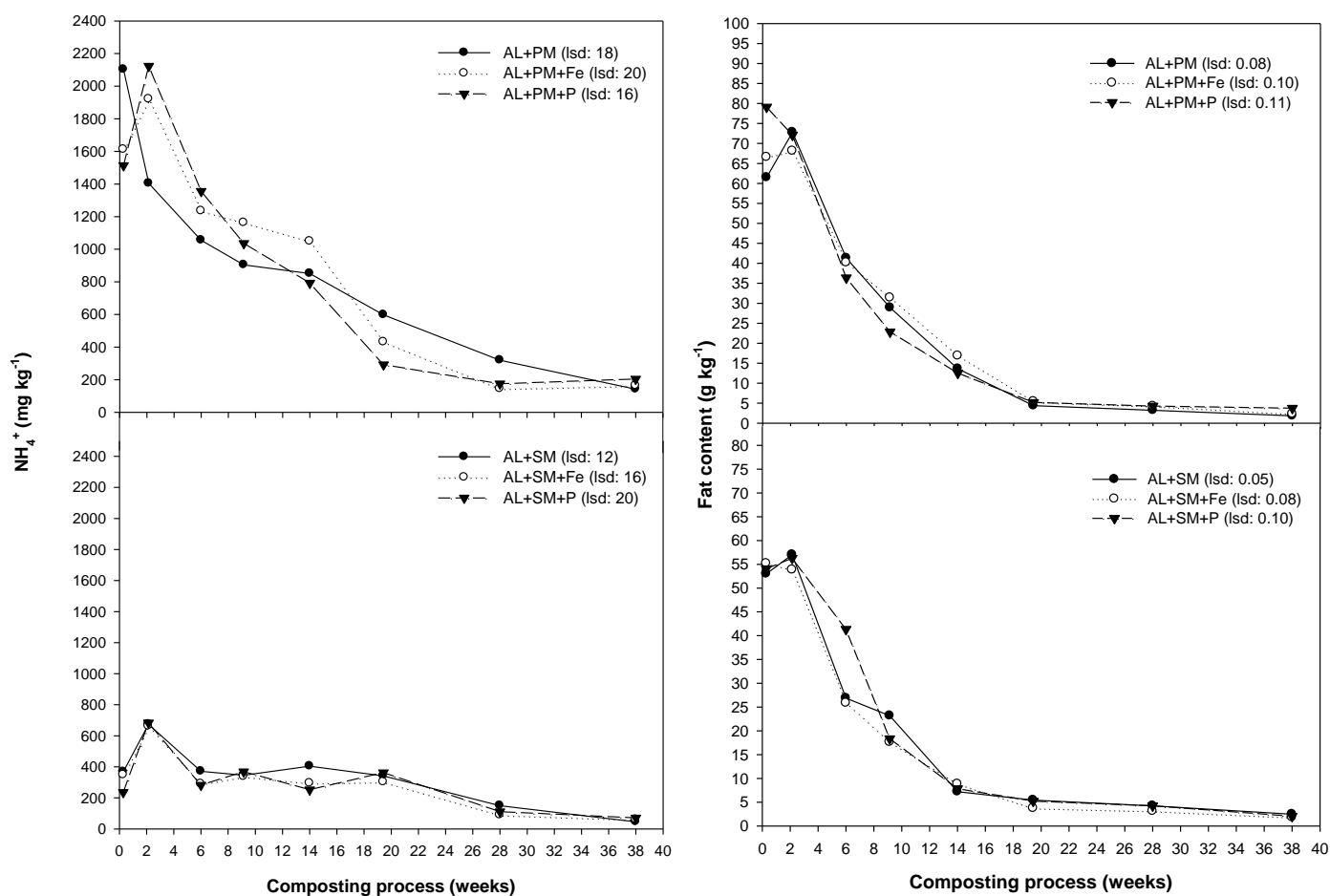


Figure 1. Evolution of temperature and pH during composting (AL: “alperujo”, PM: poultry manure, SM: sheep manure, Fe: iron-enriched mineral amendment and P: phosphorous-enriched mineral amendment). Arrows showed the mechanical turnings applied. For each pile, the least significant difference test (lsd)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



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

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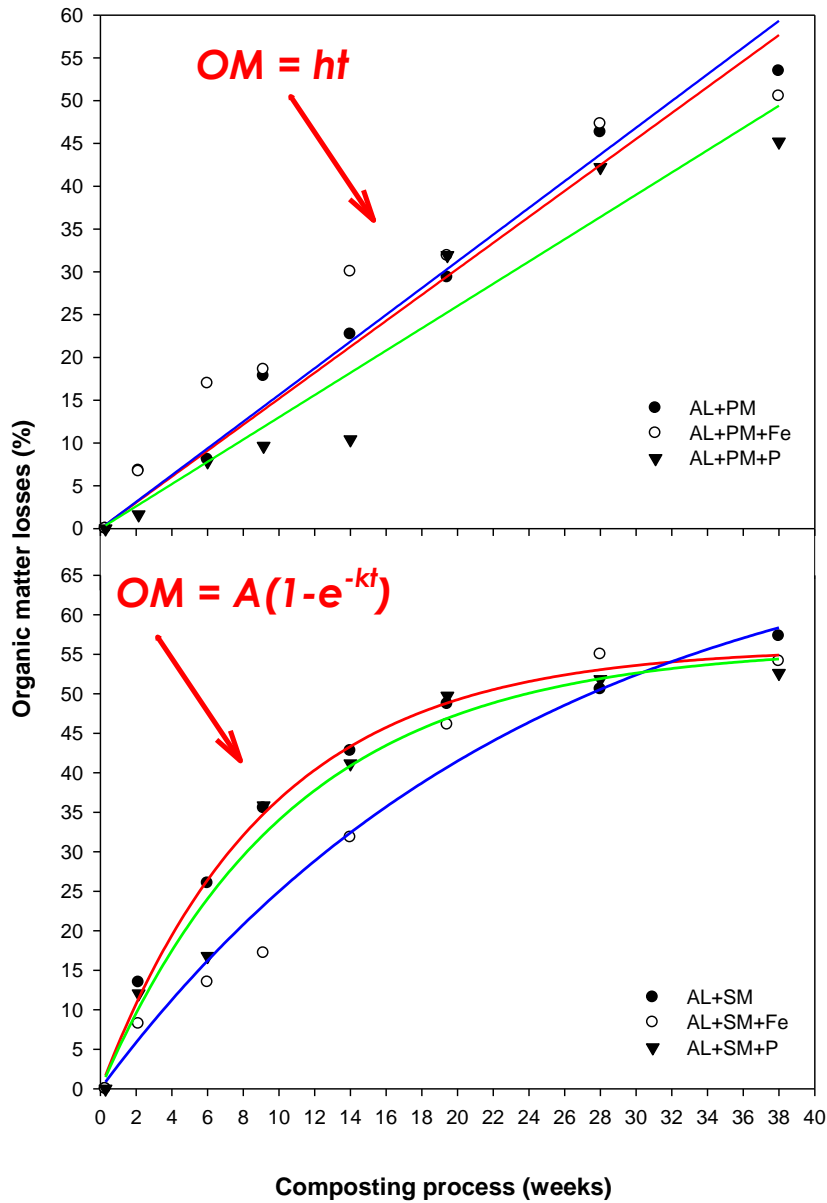
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44 **Figure 3.** Evolution of the organic matter losses during composting process (AL:
45 “alperujo”, PM: poultry manure, SM: sheep manure, Fe: iron-enriched mineral
46 amendment and P: phosphorous-enriched mineral amendment). AL+PM piles fitted to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