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Strategies to produce commercial liquid organic fertilisers from "*alperujo*" composts

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ABSTRACT

Composting is considered an economically-friendly procedure for producing commercial solid organic amendments and fertilisers from the two-phase olive mill waste (called "*alperujo*": AL), main by-product of the Spanish olive oil industry. AL composts are characterized by a noticeable organic matter content, mainly of lignocellulosic nature, which determines their humic properties. In this study, we have assayed several extraction conditions in order to release commercial liquid organic fertilisers from AL composts. The following conditions were tested: extraction time, extraction temperature, heat (70 °C) time applied, extraction ratio, extractant agent and alkali concentration. Their effects on organic fraction (total organic, polyphenol- and carbohydrate-like carbons), nutrient concentration and extraction efficiency were evaluated. In general terms, an increase in the extraction time and the combined use of alkali and heat increased significantly the amount of organic carbon solubilised from the compost, affecting the nature of the alkali-soluble organic matter and even showing a chemical degradation of the humic fraction in some cases. The extraction ratio modified the concentration of the organic and inorganic fraction in the extracts, and also their polyphenol and carbohydrate content. The use of a 24 h extraction with 1 M KOH (1:4 or 1:5, extraction ratios) and heat (4 h at 70 °C) allowed us to extract the required amount of C and K from AL compost, being necessary an external source of N and P to complete the fertiliser formulations according to the current Spanish legislation for *Organo-mineral Fertiliser* production (RD 506/2013, 2013).

1. Introduction

Composting is considered a low cost technology for transforming organic wastes and by-products into quality materials that can be used as soil amendments and/or fertilisers. A clear example is found in the Spanish olive oil industry, which generates a large amount of a solid by-product called "*alperujo*" (AL) (Alburquerque *et al.*, 2004). Composting has been shown as an effective treatment for adding value to AL (Muktadirul Bari Chowdhury *et al.*, 2013) through the production of commercial solid organic amendments and fertilisers (Tortosa *et al.*, 2012). Also, AL composts have shown a good performance in agricultural experiments (Alburquerque *et al.*, 2006, 2007; Fernández-Hernández *et al.*, 2013; Altieri *et al.*, 2014; Nair *et al.*, 2014). Therefore, the quality of the AL composts has been recognised by the Spanish authorities, including a category for AL compost as an organic amendment in the Spanish legislation for fertiliser production (RD 506/2013, 2013). Such composts are characterized by a high content of organic matter due to the lignocellulosic nature of AL, which forms the basis of their humic properties (García-Gómez *et al.*, 2005; Alburquerque *et al.*, 2009a; Serraniá *et al.*, 2013). Moreover, the composting process is associated with a high degradation of the main components of the organic matter (lignin, cellulose, hemicellulose and proteins); delivering simple carbohydrates and polyphenols which have been proposed in the literature as relevant precursors for the humification process (Sánchez-Monedero *et al.*, 1999; García-Gómez *et al.*, 2005).

The humic substances are considered the most important fraction of organic matter in agricultural soils (Stevenson, 1994). This fact is related with the improvement of physico-chemical and

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biological soil properties and also, with positive effects on plant physiology, nutrient uptake and root development (Trevisan *et al.*, 2010). In addition to these facts, composts can be also used for the extraction of soluble humic substances with potential to be used in agriculture, nowadays becoming more commonly found in commercially available liquid fertilisers (Valdrighi *et al.*, 1996; Bidegain *et al.*, 2000; Eyheraguibel *et al.*, 2008). Ait Baddi *et al.* (2013) and Masmoudi *et al.* (2013) characterized the humic fraction from AL composts showing that the alkali-extracted humic-like substances presented characteristics close to those of standard humic materials. Moreover, Caravaca *et al.* (2006) and Kohler *et al.* (2008) have shown positive effects of the alkali-soluble organic matter application from AL composts on some important legumes crops (*Retama sphaerocarpa* L. and *Medicago sativa* L.).

In Tortosa *et al.* (2012), we underlined the importance of producing high quality composts in establishing a fertiliser market, which could promote an efficient recycling strategy for AL. Also, we demonstrated the technical feasibility for producing commercial solid organic amendments and fertilisers from AL composts according to the Spanish legislation for fertiliser production (RD 506/2013, 2013). This article constituted a continuation to the former and focuses on the technical feasibility of obtaining liquid organic fertilisers from AL composts, in order to diversify the range of commercial products obtained from AL in either solid (compost) and liquid (*Organo-mineral Fertiliser*) forms. For this, we have tested different extraction conditions in order to optimize the solubilisation of both organic carbon and nutrients with particular emphasis on the humic-like component and its lignocellulosic fraction, especially important due to its positive effects on plant growth.

2. Material and methods

2.1. Composting performance and compost characterisation

A total of seven AL composts were used in the present study and they were obtained in previous experiments where AL was co-composted with animal manures (cow, poultry and sheep manure) using the mechanical turning strategy. Complete details of the composting performance (bulking agent selection, mixing ratio, etc.), process evolution and compost characterisation can be consulted in Cegarra *et al.* (2006) and Tortosa *et al.* (2012). The agrochemical characterisation of the AL composts is presented in Table 1.

The AL composts showed common typical characteristics such as alkaline pH (round 8.5), moderate salinity (2.05 ± 0.48 dS m^{-1} of electrical conductivity) and a high organic matter content (close to 70%, especially of lignocellulosic nature: 407.4 ± 44.7, 179.9 ± 18.4 and 158.3 ± 69.8 g kg^{-1} of lignin, cellulose and hemicellulose contents, respectively). In addition, they presented a high total organic carbon (T_{OC}: 355.8 ± 85.9 g kg^{-1}), a total nitrogen content close to 20.3 ± 1.5 g kg^{-1} (predominantly organic) and a T_{OC}/T_N ratio ranged from 13.5 to 21.0, typical of AL composts (Muktadirul Bari Chowdhury *et al.*, 2013). The water-soluble organic fraction showed in general a low content according to their water-soluble organic carbon (WSC), polyphenols (WSPH) and carbohydrates (WSCH) values (16.8 ± 5.7, 2.1 ± 0.8 and 3.2 ± 2.4 g kg^{-1}, respectively). Macro and micronutrient contents were noticeable, especially K, Ca and Fe, those ranged between 13.9 ± 10.1, 40.0 ± 26.5 and 6.0 ± 3.5 g kg^{-1} of average, respectively. In general terms, the AL composts showed germination index (GI) values higher than 70% and a low heavy metal content that confirmed their non-phytotoxic characteristics. Finally, agrochemical characteristics of AL composts used were agreed with those reviewed by Muktadirul Bari Chowdhury *et al.* (2013).

Table 1
Agrochemical characteristics of the AL composts used (ALC1 from Cegarra *et al.*, 2006; ALC2-7 from Tortosa *et al.*, 2012).

Parameters[a]	AL composts						
	ALC1	ALC2	ALC3	ALC4	ALC5	ALC6	ALC7
pH[b]	8.99	8.30	7.97	8.02	8.88	8.52	8.35
EC[b] (dS m^{-1})	2.96	1.70	1.84	2.44	1.69	1.70	2.03
OM (g kg^{-1})	912.2	745.6	760.3	746.0	561.2	572.1	588.2
Lignin (g kg^{-1})	399.2	428.2	467.4	457.2	350.6	375.0	374.1
Cellulose (g kg^{-1})	208.0	176.6	182.8	178.6	146.1	179.2	188.1
Hemicellulose (g kg^{-1})	291.4	208.7	152.2	129.1	131.0	92.0	104.0
T_{OC} (g kg^{-1})	484.9	396.6	403.1	393.2	251.9	272.3	288.4
T_N (g kg^{-1})	23.1	20.2	20.6	21.1	18.6	19.2	19.1
T_{OC}/T_N	21.0	19.5	19.5	18.7	13.5	14.2	15.0
WSCH (g kg^{-1})	8.1	3.2	2.0	4.2	1.5	2.2	1.2
WSPH (g kg^{-1})	2.9	2.0	2.1	3.4	2.1	1.1	1.3
WSC (g kg^{-1})	27.0	19.4	13.5	19.3	16.4	10.6	11.7
P (g kg^{-1})	1.5	2.0	1.9	2.2	1.8	1.8	2.0
K (g kg^{-1})	36.2	11.0	7.9	6.5	12.8	12.7	10.2
Ca (g kg^{-1})	9.4	29.4	20.1	18.7	65.0	72.4	65.2
Mg (g kg^{-1})	1.9	5.7	4.9	4.9	12.7	10.6	10.3
Na (g kg^{-1})	4.1	8.3	6.0	8.2	7.6	6.2	6.2
S (g kg^{-1})	–	2.5	3.6	4.6	5.2	5.4	5.4
Fe (g kg^{-1})	0.5	3.9	9.0	4.8	6.5	11.2	6.0
Cu (mg kg^{-1})	33	32	22	27	16	14	19
Mn (mg kg^{-1})	44	183	193	145	167	199	131
Zn (mg kg^{-1})	50	92	54	39	41	55	49
Pb (mg kg^{-1})	20	8	30	37	11	17	24
Cr (mg kg^{-1})	12	14	12	13	24	22	28
Ni (mg kg^{-1})	10	21	29	35	50	42	80
Cd (mg kg^{-1})	nd	nd	2	7	nd	1	2
P_{HA} (%)	75.3	63.2	63.5	72.0	78.7	63.8	61.9
GI	79	71	94	77	81	70	78

Note: Data was shown as the mean value of four replicates with less than 5% of relative error.
[a] Data based on dry weight.
[b] Water 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. HR: $(ASC/T_{OC}) \times 100$, P_{HA}: $(ASC_{HA}/ASC) \times 100$, ASC: carbon extracted with NaOH 0.1 M, ASC_{HA}: carbon of acid humic-like subtances. GI: Germination index by Zucconi Test. nd: not detected and –: not determined.

2.2. Extraction conditions and humic-like fractionation

The current protocol for the preparation of organic fertilisers is based on the procedures proposed by the International Humic Substances Society (IHSS, http://www.ihss.gatech.edu/), which are essentially based on an extraction with a strong alkali (NaOH or KOH) under controlled conditions. These procedures were initially developed for comparative studies on the chemical properties of the solubilised organic matter, and its humic fraction (humic and fulvic acids). From a commercial point of view, the extraction efficiency (percentage of organic carbon solubilised with respect to the initial content in the compost), and especially the richness in organic carbon of the obtained extract (expressed by concentration, g L^{-1}), must be optimised to fulfil the legal requirements.

In the present study, both criteria were optimized by modifying several factors such as extractant concentration, extraction time, temperature and ratio (the relationship between the solid organic material and the liquid extractant). From an agricultural point of view, KOH was used instead of NaOH as alkali extractant since K is essential for plant nutrition and Na can affect negatively both soil properties and plant growth. In order to compare the extractant effectiveness, two concentrations (0.1 and 1 M) of KOH were tested. A representative AL compost was used (Cegarra *et al.*, 2006) and the following extraction conditions were tested: