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Industrial composting of low carbon/nitrogen ratio mixtures of agri-food waste and impact on compost quality

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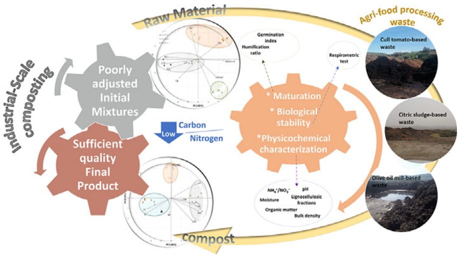
GRAPHICAL ABSTRACT



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ABSTRACT

The agri-food waste (AW) require amendments for composting to adjust nutritional and physicochemical deficiencies. The theoretical mixtures formulation is difficult to reach on an industrial scale. The main objective of this work was to evaluate to what extent the composition of AW-based mixtures determines the quality of the final compost produced at the industrial scale. Raw materials having the same AW share characteristics, irrespectively of the amendments added, but their compost were different. All the materials were biological stable at the cooling phase, and mature enough at the end, although the degree of humification did not match with the absence of phytotoxicity. The final compost had sufficient quality even though the AW-based raw materials have a low C/N ratio (< 20) and other characteristics such as high electrical conductivity ($13\ \mathrm{mS{\cdot}cm^{-1}}$) and pH ($< 8.5$) that are unfavorable for composting. The management operations during industrial composting correct the deficiencies of raw materials.

1. Introduction

The generation of residues in the different stages of the agri-food chain production, from farm to food processing industry and consumer, contributes to food losses, resource wastage, and causes negative environmental impact at several levels (Torres-León et al., 2018). Around 90 million tons of food waste is produced annually in the European Union (EU), most (72%) coming from household and food processing sectors (Stenmark et al., 2016). The later, hereinafter referred agri-food waste (AW), are residues of a high organic load produced during raw materials processing to foodstuff. They include materials in liquid or solid/semisolid form, such as bagasse, pomace, peels, trimmings, stems, shells, bran, seeds, and discards of production. Among them, the waste derived from olive oil extraction (Olive mill waste) and the production of fresh and processed citrus and vegetables are the most common in the Mediterranean countries and have high relevance in the EU (Fritsch

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et al., 2017; Muscolo et al., 2018). The seasonality of these agri-food processing sectors leads to the production of large amounts of organic waste concentrated locally in a short period, which complicates the management. Inadequate disposal causes environmental problems, including greenhouse gas (GHG) emissions, because of their high biodegradability, moisture, and microbial load (Fritsch et al., 2017).EU policies are currently promoting bioeconomy-based initiatives to prevent the loss of potential resources and mitigate the environmental damage caused by organic waste (Fritsch et al., 2017). Composting is one of the main treatments recommended for AW, because of its economic and environmental benefits, besides its pivotal role in the circular economy philosophy. This process allows the biological stabilization of the material under aerobic conditions and leads to a product, named compost, which has properties as fertilizer and soil improver, so as the organic matter returns to the soil closing the loop (Meena et al., 2016). For composting, AW usually requires amendment with other materials because of their imbalance of nutrients and high water content (Muscolo et al., 2018). Pomace or sludges from fruit processing are mixed with C-rich materials such as wood chips, pruning, or cereal straw to increase the C/N ratio to 25–30 and reduce the moisture, while olive mill waste are added of N-rich materials such as farm slurry or manure (Du et al., 2018; Tortosa et al., 2020). There are many reports in the literature on successful composting of AW amended with different materials, being the majority performed on a lab or pilot scale (Voběrková et al., 2020), whereas studies on an industrial scale are scarce (Cesaro et al., 2015; Du et al., 2018; Siles-Castellano et al., 2020). The number of facilities dedicated to industrial composting has increased significantly throughout the European geography (Razza et al., 2018). It is estimated that around 1/3 of the compost produced in the EU is made from food waste (Cesaro et al., 2015). Most AW industrial composting facilities face saturation because of the seasonality on the waste generation. In addition, they have to deal with heterogeneous raw materials, lack of suitable amendments, and operational constrains that make it difficult to reach theoretical optimal conditions for composting both in the mixtures used and the management of the process (Cesaro et al., 2015). Consequently, the compost produced in large-scale processes is not always of the best quality (Silva et al., 2014). The quality of compost is mainly defined by its stability and maturity. The stability relates to the resistance of the organic matter against further microbial decomposition. Respirometric analysis (oxygen uptake by a compost sample) is one of the best techniques to measure the stability of materials during composting (Almeira et al., 2015). The maturity relates to stability because a mature compost should be stable enough. Furthermore, maturity describes the degree of completeness of composting so that the product could be used in agriculture (Bernal et al., 2017). The degree of organic matter humification besides seed germination bioassays are generally accepted as criteria of maturity (Cesaro et al., 2019) and both are important to define the endpoint of a composting process.

This study evaluates the performance of six full scale composting facilities processing different AW and the effect of initial mixtures in the quality of the final compost obtained. Representative samples collected from AW industrial composting plants were characterized to determine: i) the suitability of the raw materials to start and guarantee the development of the composting process, ii) the stability and maturity degree of the material at critical phases of the composting, iii) the quality of the final compost.

2. Material and methods

2.1. Composting plants: Raw materials and sampling

Samples were collected from six industrial composting plants located in Southern Spain (Almeria, Jaen, and Alicante) that process AW as the main input waste, namely cull tomato (CT), olive mill waste (OMW), and citric sludge (CS) from the tomato processing, olive oil extraction, and citric juice production industries, respectively. Table 1 shows the raw mixtures used to build up the composting piles and the operating conditions followed by each facility . The plants coded M1-CS and M3-CS processed citric sludge (CS), the plant M2-CT, cull or discarded tomatoes (CT), and the plants M4-OMW, M5-OMW, and M6-OMW, olive mill waste (OMW). Different amendments/bulking agents were used in each facility to reach the optimum structure and C/N around 25–30 on the basis of theoretical calculations and fixed recipe. All plants operated with open-air turned windrows. All windrows had similar dimensions 2–3 m high × 3–4 m wide and variable length. Turnings were applied with truck loaders every 7 to 15 days, according to the temperature inside the pile, up to reaching the cooling phase (end of bio-oxidative phase), when the temperature inside the pile was below 40 °C. The duration of that phase was different in each plant, from 2 to 6 months. The maturation phase lasted for 2–3 months. The composition of the mixtures for composting, as well as the operations and time of each phase were set by each facility according to their protocols. Therefore, the conditions and characteristics described are based on the information provided by technical managers of the plants.

Samplings were performed at three different times matching with the following phases of the composting process: at the beginning of composting, raw material mixture (I); after bio-oxidative phase was ended, cooling phase (C); and at the end of the process, final product (F). The timing for C and F samplings varied among plants as indicated in Table 1. Samples (~500 g) taken from nine points of each windrow at three levels of depth, width, and length, were thoroughly mixed and, then, divided into three replicates for analytical. Samples were stored in vacuum bags frozen at −20 °C and thawed at room temperature for 24 h before carrying out the analyses. Fresh samples were used for the analysis of electrical conductivity (EC), pH, bulk density (BD), soluble organic carbon (SOC), germination index (GI), and Dynamic Respiration Index (DRI). For the analysis of organic matter (OM), total carbon (C) and nitrogen (N), $N-NH_4^+$ and $N-NO_3^-$, lignocellulose fractions (Lignin, Cellulose, and Hemicellulose) and humic-like fractions (Total Humic Extract, Humic, and Fulvic acids) the samples were air-dried at 40 °C overnight and ground to $<$ 1 mm (see Sections 2.2.1 to 2.2.3).

2.2. Analytical methods

2.2.1. Chemical and physicochemical parameters

Chemical and physicochemical parameters were analyzed in raw material (I) and final product (F). The moisture content was determined by drying at 105 °C for 24 h. All data were expressed on a dry weight basis. Electrical Conductivity (EC) and pH were measured in a 1/5 diluted sample. Bulk Density (BD) was determined using PVC cylinders of known dimensions, where the sample was placed without tightening. The sample weight was noted before, and after drying in an oven for 24 h at 60 °C. The BD value was calculated according to the expression: BD ($g\ cm^{-3}$) = (dry sample weight (g))/(cylinder volume)(cm^3)). Organic matter (OM) was determined as the weight loss of 1 g dry and ground sample, after ignition at 550 °C for 3.5 h. Total carbon (C) and nitrogen (N) were determined in solid samples by dry combustion using LecoTruSpec C-N Elemental Analyzer (LecoCo, ST Joseph MI, USA). Soluble organic carbon (SOC) was analyzed according to López-González et al. (2013) using TOC – VCSN analyzer (Shimadzu Co., Kyoto, Japan). $N-NH_4^+$ and $N-NO_3^-$ were determined in filtrated solution from 1/10 (w/v) dilution of wet sample incubated for 30 min at 200 rpm using Hach 9663 probe (Hach, Loveland, USA) for $N-NH_4^+$, and Nitrachek 404 probe (KPG Products Ldt., Hove, United Kingdom) for $N-NO_3^-$. Cellulose (CEL), Hemicellulose (HC), and Lignin (LIG) fractions were determined using the fiber analyzer Ankom (Ankom Tech., Macedon, NY, USA), according to the method established by the manufacturer.

2.2.2. Respirometric test

The biological stability of samples was evaluated using the Dynamic respiration index (DRI) following the methodology proposed by Adani

Table 1

Composition of the input waste mixtures and composting conditions in the industrial plants.

Plant Code	Input waste mixture[a]			Operating conditions[b]	
	AW	Amendments (A)	Mixture ratio AW/A (units)	Time to reach cooling phase (Months)	Maturation phase duration (Months)
M1-CS	Citric Sludge	Palm tree pruning	1 / 3 (v/v)	5	3
M2-CT	Cull Tomatoes	Tomato stalks and leaves	1 / 4 (v/v)	4	2
M3-CS	Citric Sludge	Pig slurry + Palm tree pruning	3 / 1 + 1.5 (v/v)	2	2
M4-OMW	Olive Mill Waste	Chicken manure + straw	20 / 4 + 1 (w/w)	6	2
M5-OMW	Olive Mill Waste	Cow Manure + olive leaves	12 / 1 + 3.5 (w/w)	5	2
M6-OMW	Olive Mill Waste	Cow Manure + olive leaves	1 / 0.45 + unknown (w/w)	3	2

[a] Agri-food waste (AW): Citric sludge (CS) is a semi-solid residue generated from the centrifugation of citric pulp in the citric juice production industry; Cull tomatoes (CT) are fruits that are discarded in the tomato processing industry because are defective, damaged, or immature; Olive Mill Waste (OMW) is called "alpeorujo" in Spain, it is a semisolid waste from the olive oil extraction industry that comprises the solid and aqueous fraction of the olive together with some remaining oil. It is generated in the two-phase horizontal decanter that separates olive oil from olive pomace. The amendment (A) palm tree pruning was shredded before mixing.

[b] All plants operated with open air turned windrows. Turnings applied every 7 to 15 days driven by temperature inside the pile. Cooling phase was reached when the temperature inside the pile $< 40\,^{\circ}\mathrm{C}$. All windrows dimensions around 2–3 m high × 3–4 m wide and variable length.

et al. (2006) and using the respirometer described on Ponsá et al. (2010). Briefly, a 500 mL reactor with 100 g sample at 60% moisture was placed in a water bath at 37 °C. Airflow in the reactor was adjusted through an airflow controller. Inlet air passed through a humidifier at the same temperature of the reactor to avoid water losses and moisture changes. Exhaust air from the reactor was measured by an oxygen sensor (Alphasense Ltd., Essex, UK). Airflow meters and oxygen sensors were connected to a data acquisition system to continuously record values. DRI was calculated from the average value of oxygen consumed during the most active 24 h of biological activity and expressed as g of oxygen consumed per kg of organic matter (OM) and per hour (g O_2 kg^{-1} OM h^{-1}) (Barrena et al., 2014).

2.2.3. Humification parameters

The humic-like fractions were analyzed according to Ciavatta et al. (1991) with slight modifications. For this purpose, 2.0 g of sample were added of 100 mL of pyrophosphate alkaline solution (0.1 M NaOH and 0.1 M $Na_4P_2O_7 \times 10\ H_2O$, pH 13), and shaken in a thermostatic water bath at 120 rpm for 48 h and 65 °C. After extraction, samples were filtered through a 0.8 µm cellulose acetate membrane (Sartorius Group, Göttingen, Germany), getting the Total Humic-like Extract (THE). This solution (THE) was further fractionated into Humic-like Acids (HA) and Fulvic-like Acids (FA). HA fraction was obtained by acidifying 25 mL of THE up to pH 1.5 using 9 M H_2SO_4. The precipitated HA was obtained by centrifugation at 5,000 rpm for 20 min, and then it was dissolved in 25 mL 0.1 M $Na_4P_2O_7 \times 10\ H_2O$. The supernatant obtained in the previous step was retained through a 5 cm^3 polyvinylpyrrolidone (PVP) packed column pre-equilibrated with 0.005 M H_2SO_4, and eluted after adding 25 mL 0.5 N NaOH. The flow-through was collected as the FA fraction. The carbon content of the fractions containing the Humic-like Acids (HA) and the Fulvic-like Acids (FA) (C_{HA}, C_{FA}) was measured using TOC – VCSN analyzer (Shimadzu Co., Kyoto, Japan) and expressed as a C percentage of sample dry weight. The humification ratio C_{HA}/C_{FA} was calculated as a maturity index.

2.2.4. Phytotoxicity test

The potential phytotoxicity of each sample was evaluated using Germination Index (GI), according to Zucconi et al. (1981) slightly modified. An aqueous extract, from a 65% moisture sample diluted with distilled water (1/10), was obtained after centrifugation at 10,000 rpm for 10 min and filtration (0.45 µm). The extract (4 mL) was added to filter paper deposited on a 12 × 12 cm sterile square plate and 25 seeds of *Lepidium sativum* were placed on the filter paper. Four plates were used for each tested sample, giving a total set of 100 seeds per sample. The same seeds set with distilled water added to the filter were used as control. The plates were incubated at 25 °C for 48 h in darkness. The number of germinated seeds and the root length of each seed were determined. The Germination Index (GI) was calculated using the following expression: $GI\ (\%) = (Ls \times \%Gs) / (Lc \times \%Gc) \times 100$

Where: Ls: Length of roots (mm) in seeds treated with compost extract sample; Gs: Number of seeds treated with compost extract sample that germinated; Lc: Length of roots (mm) in seeds treated with water (control); Gc: Number of seeds treated with water (control) that germinated.

2.3. Data analysis

All measurements were conducted at least in triplicate, and data are presented as the mean. Normality testing of the datasets was performed using the Shapiro-Wilk test and, when required, transformations were applied to ensure the assumption of parametricity, which was validated using the Levene test. For representation, the data were back-transformed into the original values. A significance level of $p < 0.05$ was used for all statistical analyses. The comparison of mean values for the six composting plants or sampling time was performed through Analysis of variance (ANOVA) and post hoc Least Significant Difference Fisher (LSD) multiple comparison test. Multivariate analysis was conducted to identify similarities–dissimilarities among samples with a Cluster Analysis (CA), and to quantify the significance of variables that explain the groupings from different composting plants through a Principal Component Analysis (PCA). The evolution of stability (DRI) and maturity (GI and C_{HA}/C_{FA}) of the material during composting were represented using box and whiskers plots , which represents median, lower (Q1) and upper (Q3) quartiles of the data, as well as the minimum, maximum, and outliers values of the parameter. All data analyses were performed using Statgraphics Centurion 18 (StatPoint Technologies Inc., Virginia, USA).

3. Results and discussion

3.1. Characterization of mixtures for composting.

The composition of raw materials determines its potential compostability and allows predicting whether sufficient quality standards in the final product will be achieved if an adequate operation is applied. Table 2 shows the main chemical and physicochemical characteristics that define the quality for composting of the initial mixtures used in the studied facilities. The moisture was very variable among samples, only two of them (M3-CS and M5-OMW) fell in the optimum range for composting (50–65%) while in the others it was too high, > 75% (M1-CS and M2-CT) or too low, < 50% (M4-OMW) or < 30% (M6-OMW). Moisture is a critical parameter for the success of composting, low values (< 50%) may completely preclude the start of the process while high ones (> 65%) cause anaerobic conditions which also compromise it (Xu et al., 2020). The optimal moisture content in the composting process contributes to a better degradation of organic matter and to the

Table 2

Chemical and physicochemical parameters of the initial mixtures in the AW composting plants.

Composting Plant	Moisture (%)	pH	EC(mS cm^{-1})	BD(g cm^{-3})	OM(%)	C(%)	N(%)	C/N	SOC (%)	Holocel(%)	Lig(%)
M1-CS	79.05^{d}	7.52^{d}	3.76^{a}	0.07^{a}	88.6^{d}	41.2^{c}	4.50^{e}	9.18^{a}	0.09^{a}	36.3^{d}	9.58^{b}
M2-CT	83.15^{d}	4.79^{a}	13.00^{c}	0.09^{a}	60.1^{b}	32.8^{a}	2.62^{d}	12.60^{b}	0.21^{a}	35.6^{cd}	10.90^{c}
M3-CS	65.59^{c}	6.67^{c}	2.82^{a}	0.21^{b}	62.5^{b}	35.5^{b}	2.13^{c}	16.70^{c}	0.14^{a}	26.0^{a}	8.02^{a}
M4-OMW	47.86^{b}	7.04^{c}	3.47^{a}	0.37^{c}	48.8^{a}	32.6^{a}	1.52^{a}	21.40^{e}	1.73^{c}	30.5^{b}	13.80^{d}
M5-OMW	62.48^{c}	5.52^{b}	3.05^{a}	0.18^{b}	74.8^{c}	36.1^{b}	1.90^{b}	19.00^{d}	1.55^{b}	33.5^{c}	10.80^{dc}
M6-OMW	26.53^{a}	6.63^{c}	7.20^{b}	0.20^{b}	74.8^{c}	42.8^{d}	2.32^{c}	18.40^{d}	2.02^{d}	34.3^{cd}	10.50^{bc}
	***	**	**	**	**	**	**	***	***	*	*
LSD	7.61	0.44	1.06	0.10	6.0	1.3	0.20	1.62	0.152	2.35	1.24

Abbreviations: EC: electrical conductivity; BD: bulk density; OM: organic matter; C: total carbon; N: total nitrogen; C/N: carbon–nitrogen ratio; SOC: soluble organic carbon; Holocel: cellulose + hemicellulose; Lig: lignin. Data are mean values (n = 3). Values within columns with different superscript letters denote a statistically significant difference based on Fisheŕs LSD paired post hoc comparisons at 95% confidence level. LSD (Least Significant Difference) interval and significance of ANOVA test ***$p < 0.001$, **$p < 0.01$, *$p < 0.05$ are shown. Composting plant code as in Table 1: Citric sludge (M1-CS, M3-CS); Cull tomatoes (M2-CT); Olive mill waste (M4-OMW, M5-OMW, M6-OMW).

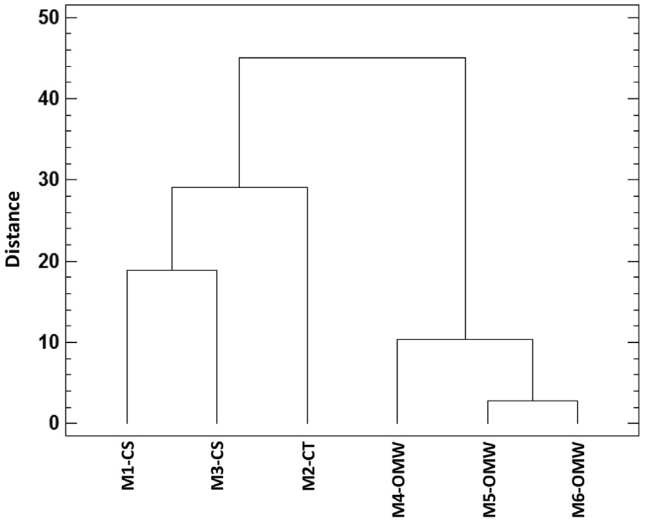

Fig. 1. Cluster analysis dendrogram of the initial mixtures used in the six composting plants evaluated according to their chemical and physicochemical composition (variables used for the analysis included in Table 2). Code of samples as in Table 1, mixtures identified by the main component CS citric sludge; CT cull tomato, OMW olive mill waste. Ward's method and the Euclidean distance were used as measures of homogeneity between groups of mixtures.

maintenance of temperature for a longer period, favoring microbial activity (Silva et al., 2014). Nevertheless, the moisture can be easily adjusted by watering of simply letting the material to dry during turnings so the composting can proceed. Most raw materials had pH close to neutral values, which are considered optimal for composting (López-González et al., 2013), only two mixtures were acidic, i.e. M2-CT (4.8) and M5-OMW (5.5). pH values below 6, especially in combination with the increasing temperatures at the start of the composting (first mesophilic phase), are a possible adverse factor in large-scale AW-based composting, negatively influencing the transition to the thermophilic phase and, therefore, the development of the entire process (Sundberg et al., 2004). The electrical conductivity (EC) was, in general, close to the value considered adequate for raw materials (around 4 mS·cm^{-1}) (Jara-Samaniego et al., 2017). However, the mixture made with cull tomatoes (M2-CT) had a high EC (13 mS·cm^{-1}) which may be explained by EC values of the tomato plant residues used as amendment, even though such high salt content materials have been successfully composted earlier (López-González et al., 2013). Bulk Density (BD) is considered an important parameter for composting in the initial mixtures since it influences the gases interchange and heat transfer through materials, which are required for the activity of the microorganisms. BD values around or below 0.2–0.35 g·cm^{-3} are adequate for composting (Jara-Samaniego et al., 2017; Chang et al., 2019). Most raw materials used in the AW plants had BD close or lower to that range because the amendments or bulking agents lowered the typical high BD of slurry materials such as CS and OMW. The organic matter (OM) content ranged from 87% to 49%, which were adequate for composting (Bernal et al., 2017) although slightly low in the mixture M4-OMW. The C/N ratio for raw materials in the range of 25–30 is assumed as an optimum environment for microorganisms in composting and it is one of the key parameters considered in the formulation of the initial material (Bernal et al., 2017; Xu et al., 2020). The composting facilities of this study tested the C and N content of the AW, and chose the appropriate amendments to adjust the C/N ratio, based on its composition, costs, proximity, and availability. This is important since C serves as a source of energy and N is used for building cell structures (Iqbal et al., 2015). However, while the mixtures were theoretically formulated to have a C/N ratio around 20–30, the analysis revealed that all the samples registered values below that range. The lowest (C/N $<$ 17) corresponded to CS and CT-based mixtures (M1-CS, M3-CS, and M2-CT) while the highest (C/N = 18–21) to mixtures made with olive mill waste (M4-OMW, M5-OMW y M6-OMW). Soluble organic carbon (SOC) represents the easily assimilable fraction and, therefore, the main carbon source directly available for the microorganisms (Bernal et al., 2017). The mixtures made with OMW had higher SOC values (1.5–2%) than those of CS or CT (SOC $<$ 0.2%). Accordingly, it is expected that the process would start earlier in the OMW mixtures (Jurado et al., 2014). However, high SOC content by itself does not guarantee a prompt increase of temperature because of the microbial activity, the specific composition of that fraction also determines that fact. Thus, OMW is rich in hydrosoluble phenols that can be a part of SOC but because of its antimicrobial activity, they can delay the start of the composting instead. The lignocellulose content of the initial mixtures is mainly provided by the vegetal amendments added to the AW. In this fraction, the lignin is critical because of its high resistance to biodegradation influences the rate of degradation of organic matter during composting (Toumela et al., 2000). In general, the higher the lignin content, the less the degradability of the material, and the longer the composting process (López-González et al., 2013). According to this, the mixtures made with CS (M1-CS and M3-CS), both with palm tree pruning as amendment, had the lowest lignin content ($<$ 9.5%) and are expected to biotransform faster than the CT and OMW mixtures that had lignin contents higher than 10%. The prolonged composting time for these two materials has been demonstrated earlier (López-González et al., 2013; Alburquerque et al., 2009). The cluster analysis allowed grouping the initial mixtures according to the previously discussed parameters. The dendrogram classified the materials mainly by the AW of the mixture (Fig. 1). It had two main clusters, one included OMW-based mixtures and the other the two CS mixtures, that grouped together, and CT. The most similar OMW mixtures were M5-OMW and M6-OMW, which on the other hand had similar components in the

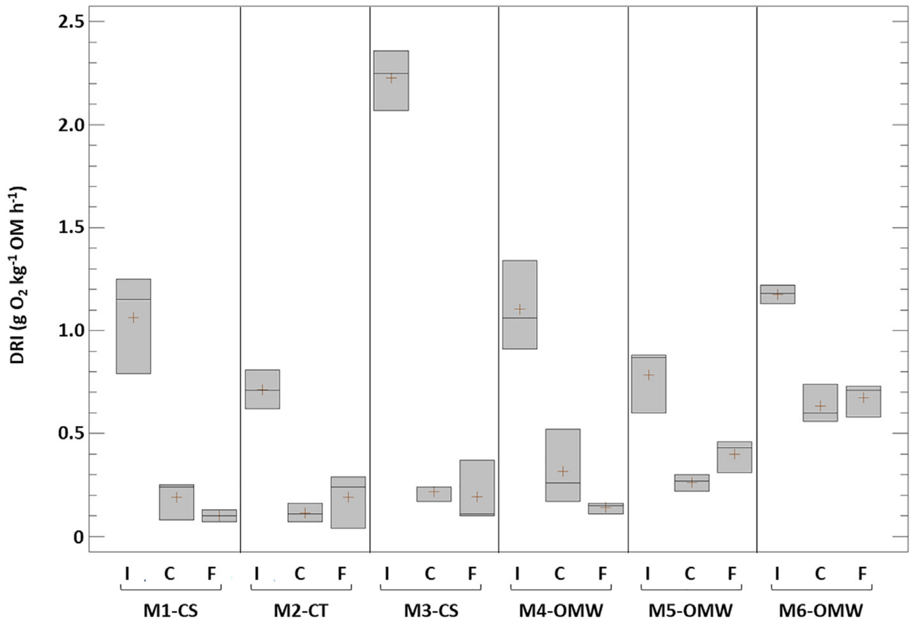

Fig. 2. Evolution of stability through Dynamic Respiration Index (DRI) in the material of the six composting plants: box and whisker plot showing DRI values at three composting phases Initial (I), Cooling (C) and Final product (F). Code of samples as in Table 1, composting plants identified by the main component of the raw mixture: CS - citric sludge; CT - cull tomato, OMW - olive mill waste.

mixture (see Table 1).

3.2. Evolution of stability and maturity during composting

The stability of the material during composting was measured with the Dynamic Respirometric Index (DRI) that reflects the degree of decomposition during the biotransformation and provides an indirect measure of the biological activity (Wichuk and McCartney, 2010). It helps to detect incorrect management practices (e.g. feeding of the pile with fresh materials), to monitor the performance of the process, and make corrections in operations such as the number of turnings or watering. Fig. 2 shows the evolution of DRI in the six composting plants studied, from the initial values in the raw mixtures (I) to the cooling phase (C) and the final product (F). In general, DRI steep dropped from the initial values to the lowest ones after passing the bio-oxidative phase, at the cooling phase, with no significant further changes during maturation. This trend matches the more intense biological activity at the bio-oxidative phase, which leads to the stabilization of the material. During that phase, the DRI dropped > 80% in CS and CT-based materials, while that decrease was lower (46% to 70%) in OMW-based materials. This is likely due to the more recalcitrant nature of OMW (Alburquerque et al., 2009; Martínez-Gallardo et al., 2020). Moreover, the presence and amount of manure in the mixture determined a higher decrease in the DRI during the bio-oxidative phase. Thus, in the two CS-based mixtures, the one that lacked manure (M1-CS) got an 82% DRI decrease, while in the M2-CS that contained pig slurry, DRI dropped 90%. For OMW-based mixtures, the one containing a high proportion of chicken manure (M4-OMW) stabilized more than the other two mixtures with DRI decrease of 72% at cooling phase and 87% at the end, in comparison to decreases of 66% for M5-OMW and 46% for M6-OMW, this later having the lowest manure ratio. In addition, it is worth mentioning that the dispersion of DRI data in all plants decreased as the composting proceeded, as noticed by the decreasing length of boxplot from the initial values to those at cooling and final product (Fig. 2). This can be related to the homogenization of the materials during composting because of turnings and OM degradation. At the cooling phase, the DRI reached values between 0.6 and 0.1 g O_2/kg OM h^{-1} which are in the range of the classification made by Adani et al. (2004) for stable materials. According to that, it can be stated that all industrial composting plants operated well during the critical bio-oxidative phase in which more intense operational activities (e.g. turning, watering) are required. In addition, no feeding of the pile with fresh materials that would have led to DRI increase seems to have been done, except for the M5-OMW plant in which DRI doubled its value during maturation. This practice at the industrial scale leads to the production of final materials insufficiently stable.

Maturity degree is, besides stability, the main criteria to decide what composting operations apply and when a process is finished because the compost obtained is good enough The humification ratio C_{HA}/C_{FA} and the Germination Index (GI) were analyzed to determine the maturity degree in AW materials during composting. The evolution of the humification ratio was quite different depending on the AW (Fig. 3). C_{HA}/C_{FA} increased in OMW and CS-based materials while in the CT-based mixture that ratio decreased. Only OMW-based materials and M1-CS reached final humification ratio higher or close to 1 that is considered typical for mature compost (Azim et al., 2018). The GI provides parallel information to that of C_{HA}/C_{FA} about the state of organic matter decomposition and, thus, the maturity. Nevertheless, the GI results obtained in the different materials did not exactly match with those of degree of polymerization (Fig. 4). The GI results are better interpreted considering the classification criteria for the phytotoxicity of a material based on its germination index (Zucconi et al., 1981; Emino and Warman, 2004). Samples with $GI < 50\%$ are highly phytotoxic; $50\% < GI < 80\%$, moderately phytotoxic; $GI = 80–100\%$, non-phytotoxic, and, $GI > 100\%$, phytostimulants. Initially, all the raw materials were phytotoxic ($GI < 50\%$), especially those of CS and CT ($GI = 0$) (Fig. 4), which is logical at the beginning of any composting process, since the transformation of the waste has not started yet (Selim et al., 2012). In the early stage of a composting process short-chain organic acids are produced that are toxic to different organisms. However, these acids are decomposed further, eliminating the phytotoxicity (Wichuk and McCartney, 2010). Under normal conditions, as the composting process progresses, the GI values should gradually increase, reaching values close to 100%, at the end, if the degree of maturity of the final product is sufficient (Huang et al., 2017). Despite the low degree of humification (C_{HA}/C_{FA}) of CS and CT-based materials at the cooling and final phase (Fig. 3), they were those that gave higher GI values yet at the cooling phase ($GI > 80\%$). Even more, final M1-CS and M2-CT were considered plant growth-promoting substrates, reaching GI values higher than 100%. Conversely, M5-OMW, the most humidified final material (Fig. 3), was the most phytotoxic final product (GI 55%) that surprisingly was no phytotoxic at the cooling phase (GI 78%) (Fig. 4). This can be related to the potential incorporation of fresh material during the maturation phase, as it was also evidenced by the

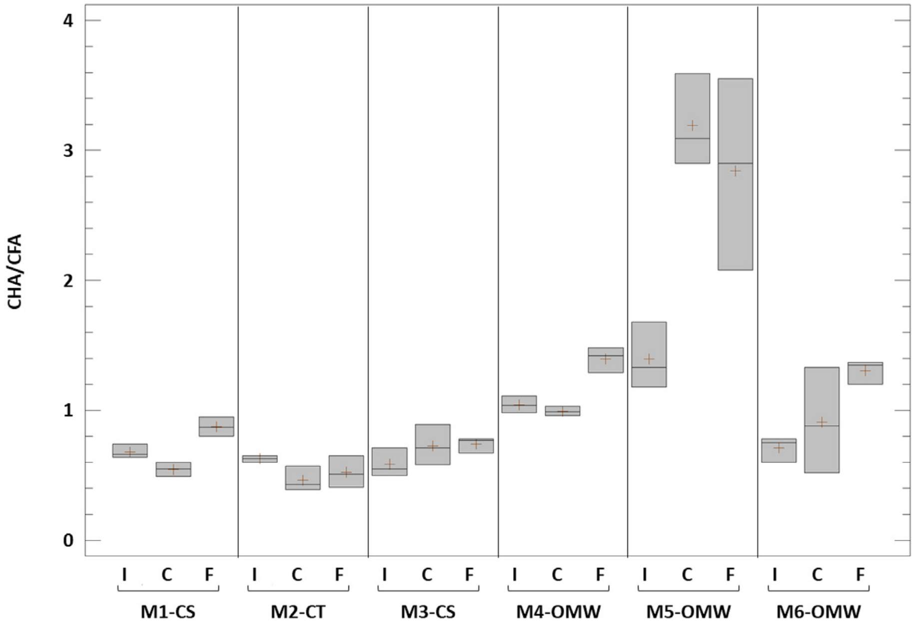

Fig. 3. Evolution of maturity through humification ratio (CHA/CFA) in the material of the six composting plants: box and whisker plot showing CHA/CFA values at three composting phases Initital (I), Cooling (C) and Final product (F). Code of samples as in Table 1, composting plants identified by the main component of the raw mixture: CS - citric sludge; CT - cull tomato, OMW - olive mill waste.

increase in DRI. The other OMW facilities, although having great variability in results and dispersion in the data, especially M6-OMW, managed to obtain final products with a certain maturity considering GI (between 80 and 100%). The disagreement between the GI and C_{HA}/C_{FA} results in the OMW management plants could be due to the particular characteristics of OMW. The complex interaction between different phenolic substances present in the OMW gives this material a toxic effect (phytotoxic and antimicrobial). This condition, together with the release of phenolic compounds after the degradation of the lignin present in many organic residues, would lead to this deceleration in eliminating the phytotoxic potential of the samples and can hinder the actual polymerization rate during humification (Martínez-Gallardo et al., 2020).

3.3. Final compost quality

The characteristics related to the quality of the final compost produced in the six facilities are shown in Table 3. The pH in almost all of the compost was within the range adequate for compost (6–8) (Bernal et al., 2017; Azim et al., 2018), except for two OMW-based compost (M5-OWM and M6-OMW) and one CS-compost (M3-CS) that had pH $>$ 8. The high pH value is known to affect the germination of seeds (Barral and Paradelo, 2011) and that could explain the moderate phytotoxicity (GI $<$ 80%) of these compost (Zucconi et al., 1981; Emino and Warman, 2004) in comparison to the other final materials that were phytostimulant (GI $>$ 100%). The electrical conductivity indicates the total salt content in the compost and provides a measure of its agronomic potential as an organic amendment (Huang et al., 2017; Yang et al., 2015). According to Awasthi et al. (2014), EC up to 4 mS cm^{-1} is considered suitable for compost to be applied in the soil. In this sense, only M1-CS (7 mS cm^{-1}) and M2-CT (5 mS cm^{-1}) outrange that value. Noteworthy, the unfavorable high initial EC value of CT-based raw materials was lowered during composting (from 13 to 5 mS cm^{-1}) likely because of salts washing effect of watering. The BD influences the

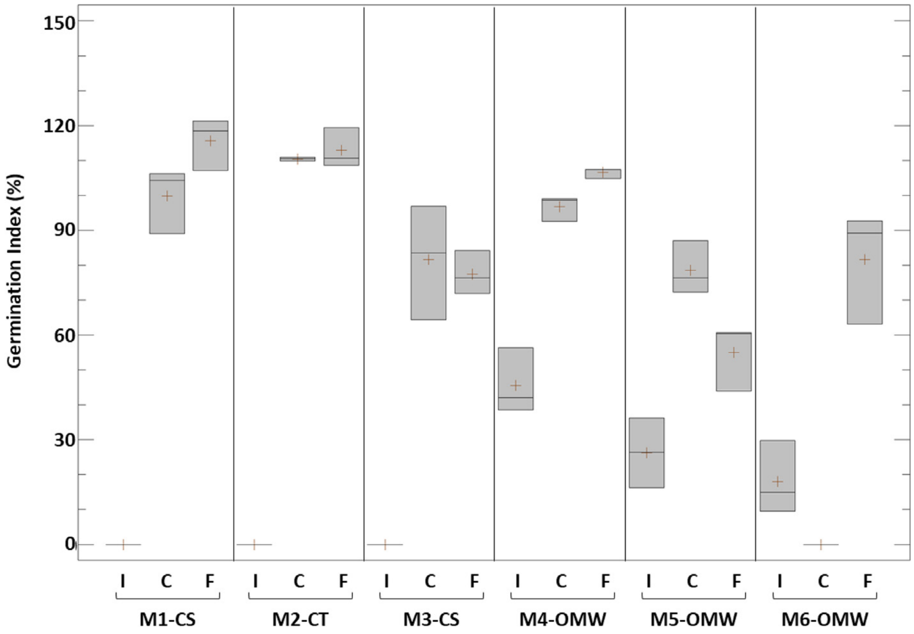

Fig. 4. Evolution of maturity through germination index (GI) in the material of the six composting plants: box and whisker plot showing GI values at three composting phases Initital (I), Cooling (C) and Final product (F). Code of samples as in Table 1, composting plants identified by the main component of the raw mixture: CS - citric sludge; CT - cull tomato, OMW - olive mill waste.

Table 3
Characterization of the final compost from the AW composting plants.

Composting Plant	pH	EC(mS cm^{-1})	BD(g cm^{-3})	OM(%)	C(%)	N(%)	C/N	SOC (%)	NH_4^+/NO_3^-	C_{HA}/C_{FA}	GI(%)	DRI(g O_2 kg^{-1}OM h^{-1})
M1-CS	6.64^{a}	7.24^{d}	0.15^{a}	74.1^{d}	37.0^{c}	3.28^{e}	11.3^{a}	0.01^{a}	0.04^{a}	0.87^{ab}	116.0^{c}	0.10^{a}
M2-CT	7.83^{c}	5.10^{c}	0.56^{d}	49.1^{b}	25.3^{a}	2.05^{b}	12.4^{b}	0.10^{a}	0.14^{a}	0.52^{a}	113.2^{c}	0.19^{a}
M3-CS	8.67^{b}	2.72^{ab}	0.29^{b}	52.1^{b}	30.5^{b}	2.58^{d}	11.8^{ab}	0.14^{a}	0.73^{b}	0.74^{a}	77.5^{b}	0.19^{a}
M4-OMW	7.23^{d}	4.57^{c}	0.53^{d}	32.3^{a}	25.4^{a}	1.45^{a}	17.5^{d}	0.32^{b}	0.08^{a}	1.40^{b}	107.3^{c}	0.14^{a}
M5-OMW	9.46^{e}	3.55^{b}	0.26^{b}	60.2^{c}	31.8^{b}	2.03^{b}	15.7^{c}	0.73^{c}	0.09^{a}	2.84^{c}	55.2^{a}	0.41^{b}
M6-OMW	8.62^{d}	2.44^{a}	0.39^{c}	82.1^{e}	40.8^{d}	2.21^{c}	18.5^{e}	1.28^{d}	0.13^{a}	1.31^{b}	81.7^{b}	0.67^{c}
	***	**	***	***	**	**	*	***	*	**	***	*
LSD	0.09	0.87	0.03	3.0	1.3	0.08	0.8	0.15	0.04	0.56	16.2	0.17

Abbreviations: EC: electrical conductivity; BD: bulk density; OM: organic matter; C: total carbon; N: total nitrogen; C/N: carbon–nitrogen ratio; SOC: soluble organic carbon; C_{HA}/C_{FA}: humification ratio; GI: Germination index; DRI: Dynamic respiration index. Data are mean values (n = 3). Values within columns with different superscript letters denote a statistically significant difference based on Fisheŕs LSD paired post hoc comparisons at 95% confidence level. LSD (Least Significant Difference) interval and significance of ANOVA test ***$p < 0.001$, **$p < 0.01$, *$p < 0.05$ are shown. Composting plant code as in Table 1: Citric sludge (M1-CS, M3-CS); Cull tomatoes (M2-CT); Olive mill waste (M4-OMW, M5-OMW, M6-OMW).

quality of the final product in terms of mechanical properties such as strength, porosity, and ease of compaction (Estrella-González et al., 2019). In general, the compost had BD values suitable for its agronomic application and, in the specific case of M3-CS, M5-OMW and M6-OMW, reached values close to those generally considered optimal (0.32 g·cm^{-3}) (Jara-Samaniego et al., 2017). The OM was within the range established in the literature 30–60% for compost (Fialho et al., 2010) and comply with the threshold established in the Spanish regulation 35–40% (BOE, 2017), except for M1-CS (74%) and M6-OMW (82%), which had high OM proportion at the end of a composting process. However, those values do not represent an issue for the final compost since both materials were stabilized enough, as evidenced by the DRI values. All the final materials reached biological stability (< 0.5 g O_2 kg^{-1} MO-h^{-1}) (Lasaridi and Stentiford, 1998; Adani et al., 2006). The only exception was observed in M6-OMW (0.67 g O_2 kg^{-1}OM h^{-1}), which is considered moderately stabilized (Adani et al., 2004). In any case, as other authors also reported (Scaglia et al., 2000; Adani et al., 2006), DRI < 0.5 (g O_2 kg^{-1}OM h^{-1}) are related to maturation, and, thus biologically stable. Related to OM, the fraction of organic carbon readily available to microorganisms (SOC) usually decreases during composting to values around 0.9% in the compost (Jurado et al., 2014), which is in the range of values found in all compost obtained. The C/N ratio of final materials ranged between 18.5 and 11, thus pass the standard (C/N ≤ 25) for mature compost (Bernal et al., 2017) and fulfilled the Spanish legislation threshold C/N < 20 (BOE, 2017). This means that even though initial C/N in the raw materials was lower than optimal for composting, no nitrogen losses were produced during composting, which is the main issue of low C/N mixtures. The ratio NH_4^+/NO_3^- is considered another maturation index. It is also known as the oxidation index of mineral forms of nitrogen or nitrification index, so as the lowest its value the highest the maturity (Bernal et al., 2017). Values below or in the range of 0.5 to 3 are reported by Onwosi et al. (2017) for mature compost with a more restrictive value below 0.16 stated by Bernal et al. (2017) for a fully mature compost. Accordingly, all AW compost fulfilled this last value, except M3-CS material (0.73) that also can be considered mature. The degree of polymerization and hence the humification was higher in OMW-based compost, in which the ratio $C_{HA}/C_{FA} > 1$, than CS or CT-based materials which according to that ratio are not mature enough. As stated above, this result mismatch those obtained with other maturity-related parameters such as GI and NH_4^+/NO_3^- that revealed all materials were mature at the end of composting. This supports the need for a certain range of parameters to measure to determine the maturity and quality of the final compost (Estrella-González et al., 2020). The cluster analysis classified final compost according to the previously discussed parameters in two main groups, one including two OMW-based compost (M5-OMW and M6-OMW) and the other a miscellaneous of the remaining materials (Fig. 5).

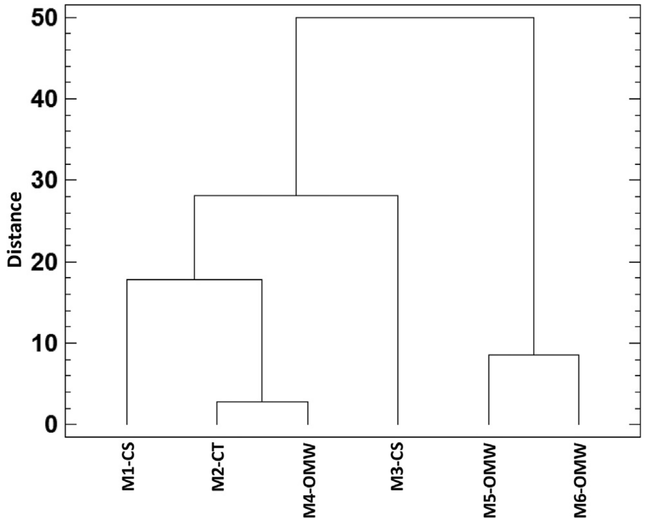

Fig. 5. Cluster analysis dendrogram of the final products of the six composting plants evaluated according to their chemical and physicochemical composition (variables used for the analysis included in Table 3). Code of samples as in Table 1, mixtures identified by the main component CS citric sludge; CT cull tomato, OMW olive mill waste. Ward's method and the Euclidean distance were used as measures of homogeneity between groups of mixtures.

3.4. Comparison between initial mixtures and final compost

Fig. 6 depicts the parameters that most influenced the differentiation of the samples for the initial mixtures and the final composts, which also confirm the findings from the cluster analysis. The grouping of the samples changed after composting from the initial, that separated mixtures made with the same AW, to the final compost in which only two OMW compost (M5 and M6) had similar characteristics. In the initial mixtures the two PC explained 66% of the variability. The variables that most differentiated samples were pH and OM for CS-based mixtures, EC for CT-mixture, whereas OMW-based mixtures clustered with many parameters related to SOC, C/N, BD, and lignin content. For the final products (PC 64%), EC and GI had a high load for the miscellaneous group M1-CS, M2-CT, and M4-OMW, while N mineralization ratio was the main driver for M3-CS separation due to its high value in this material. In the case of the two OMW-compost that clustered together (M5-OMW and M6-OMW) the carbon-related parameters (DRI, SOC, C_{HA}/C_{FA}, OM, and C/N) besides pH had a strong influence in their grouping.

To establish a ranking of the raw materials according to their suitability for composting and relate them with the quality of the compost produced, the values obtained for key parameters were compared with those reported in the literature as optimum for initial and final

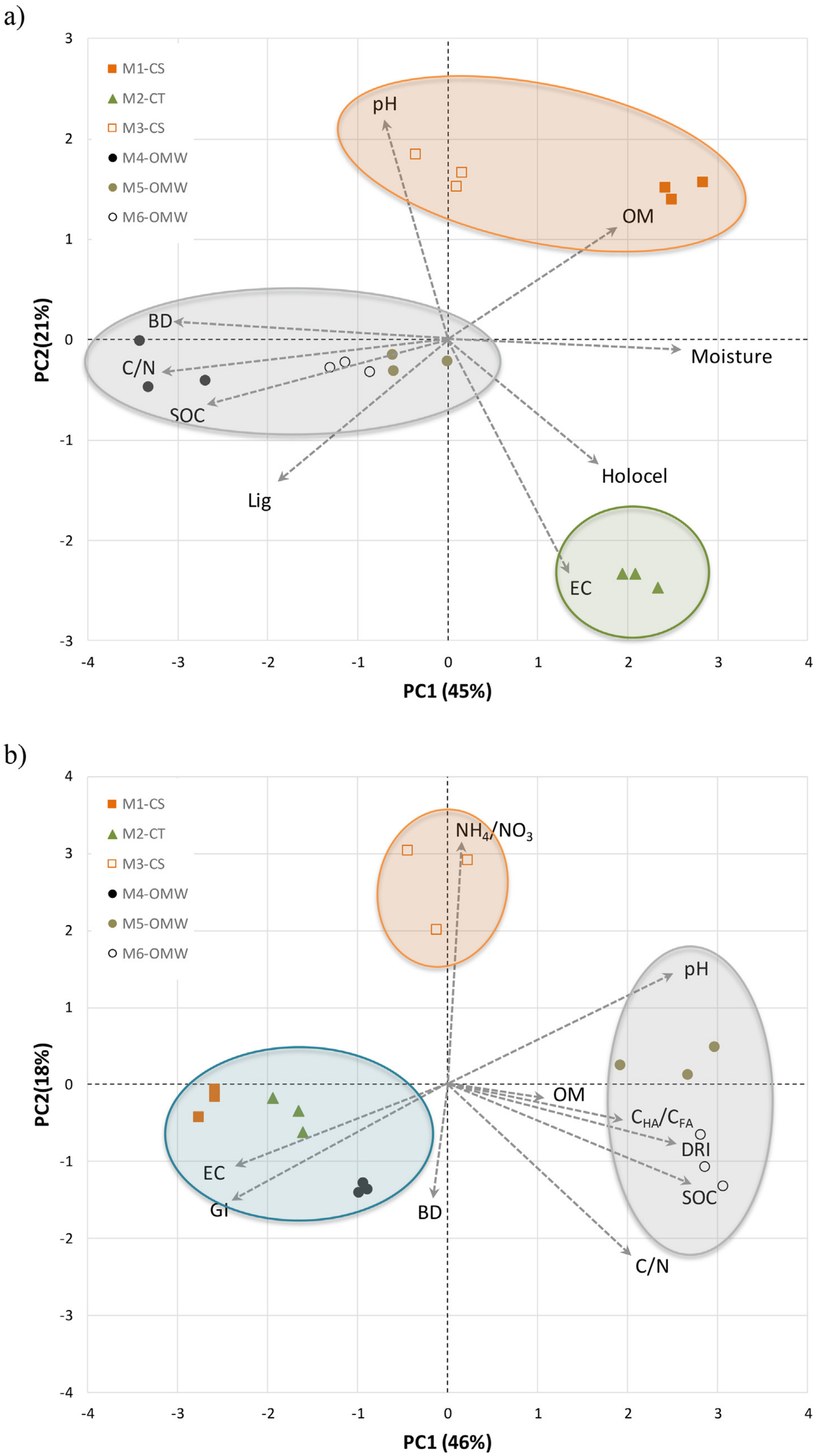

Fig. 6. Principal components analysis biplot for the a) raw materials and b) final products of the six AW composting plants (variables used for the analysis included in Tables 2 and 3). Code of samples as in Table 1, mixtures identified by the main AW component CS citric sludge; CT cull tomato, OMW olive mill waste.

Table 4
Scoring of raw materials and final compost from AW composting plants*.

Material	Parameter (Units)**	Optimal Values***	M1-CS	M2-CT	M3-CS	M4-OMW	M5-OMW	M6-OMW		
Raw materials	Moisture (%)		50 – 60[a]		0	0	0	1	0	0
	pH	5.5 – 8[a]	1	0	1	1	1	1		
	EC (mS cm^{-1})	< 4[b]	1	0	1	1	1	0		
	BD (g cm^{-3})	0.1 – 04[c]	1	1	1	1	1	1		
	C/N	25 – 35[a]	0	0	0	0	0	0		
	Total Score raw materials	0.60	0.20	0.60	0.80	0.60	0.40			
Compost	pH	6.5 – 8.5[a]	1	1	0	1	0	1		
	EC (mS cm^{-1})	< 4[b]	0	1	1	0	1	1		
	OM (%)	> 20 [a,d]	1	1	1	1	1	1		
	C/N	< 15 – 20[a,d]	1	1	1	1	1	1		
	C_{HA}/C_{FA}	> 1[a]	1	0	0	1	1	1		
	NH_4^+/NO_3^-	< 0.16[e]	1	1	0	1	1	1		
	GI (%)	> 80[f]	1	1	1	1	0	1		
	DRI (g O_2 kg^{-1} OM h^{-1})	< 0.4 [a,g]	1	1	1	1	1	0		
	Total Score Compost	0.88	0.88	0.63	0.88	0.75	0.88			

* The scores are based on the comparison of values ± standard deviation of each material with the optimal values reported in literature (mean values in Table 2 for raw materials and Table 3 for compost): values in the range of optimal values = 1; values out of the range of optimal values = 0. The total scores are calculated as the sum of scores/number of parameters. Composting plant code as in Table 1: Citric sludge (M1-CS, M3-CS); Cull tomatoes (M2-CT); Olive mill waste (M4-OMW, M5-OMW, M6-OMW).

**Abbreviations: EC: electrical conductivity; BD: bulk density; OM: organic matter; C/N: carbon–nitrogen ratio; C_{HA}/C_{FA}: humification ratio; GI: Germination index; DRI: Dynamic respiration index.

***References for optimal values: (a) Bernal et al., 2017; (b) Jara-Samaniego et al., 2017; (c) Azim et al., 2018; (d) BOE, 2017; (e) Bernal et al., 1998; (f) Emino and Warman, 2004; (g) Adani et al., 2004.

composting materials. The results of the scoring are shown in Table 4, in which the closest total score is to 1 the better the quality of the material, either as a substrate for composting or as compost of quality. In general, all the raw materials were of unacceptable quality for composting because of the low C/N ratio, none of the mixtures reached the minimum required (C/N = 25). Considering the other parameters, the best raw material was M4-OMW (0.8), followed by M5-OMW, M3-CS, and M1-CS (0.6), while M2-CT (0.2) as the one with the lower quality for composting. Despite the low suitability of some of the initial AW mixtures, all the final compost had sufficient quality, reaching scores higher than 0.6, which means that a minimum of five parameters out of eight, which are related to stability and maturity, were in the optimal range for compost of quality. Noteworthy, M2-CT compost was among the ones with the highest score (0.88) even if it came from the initial mixture having the lowest quality. All compost had scores of 0.88, except M5-OMW (0.75) and M3-CS (0.63), both having pH > 8.5. Previous studies have reported on the feasibility of carrying out adequate food-based waste composting processes starting with materials having low C/N ratio (Zhu, 2007; Voběrková et al., 2020). The results obtained could be indicative of the possibility to obtain compost of quality without the need for a precise adjustment of the initial C/N ratio for composting of AW.

4. Conclusions

The raw materials for composting having the same AW share similar chemical and physicochemical characteristics, irrespectively of the amendments added. The AW materials were stabilized at the cooling phase. The compost produced from the same AW does not necessarily share similar characteristics. Despite the low suitability of some initial AW mixtures, all the final compost had sufficient quality. It is possible to produce quality compost from AW-based materials that have a low C/N ratio and other characteristics that are unfavorable for composting. The management operations during composting correct the deficiencies.

CRediT authorship contribution statement

Ana B. Siles-Castellano: Investigation, Methodology, Formal analysis, Writing - original draft. **María J. López:** Conceptualization, Formal analysis, Data curation, Supervision, Writing - review & editing. **Macarena M. Jurado:** Conceptualization, Investigation, Data curation, Writing - original draft. **Francisca Suárez-Estrella:** Conceptualization, Investigation, Methodology, Resources. **Juan A. López-González:** Investigation, Methodology. **María J. Estrella-González:** Investigation, Methodology. **Joaquín Moreno:** Conceptualization, Supervision, Project administration, Funding acquisition, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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