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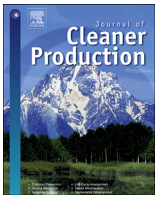

Comparative analysis of phytotoxicity and compost quality in industrial composting facilities processing different organic wastes

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ABSTRACT

A comparative analysis of industrial composting facilities processing Vegetal Residues, Sewage Sludge, Municipal Solid Waste, Agri-food Waste or Olive Mill Waste was performed. The evolution of phytotoxicity during composting was analyzed by measuring the germination index. Physico-chemical parameters (pH, electrical conductivity, organic matter, reducing sugars, phenolic compounds, humic substances, soluble organic carbon, N–NH$_4^+$ and N–NO$_3^-$) and heavy metals content were evaluated in final products. The material became non-phytotoxic at the cooling phase or at the end of composting in facilities processing Olive Mill Waste, Sewage Sludge, and Agri-food Waste. In facilities processing Municipal Solid Waste and Vegetal Residues, the material never lost phytotoxicity. All composts, except Municipal Solid Waste, fulfill requirements of the Spanish legislation for heavy metals content. Phytotoxicity was attributed to the high electrical conductivity and pH of the Vegetal Residues compost and heavy metal content of the Municipal Solid Waste compost. Germination index is recommended to track the performance of industrial composting. This work provides new insights for the better management of composting at the industrial scale.

1. Introduction

Composting is a dynamic, biological and aerobic process in which organic matter is stabilized after passing through a thermophilic phase, fostered by the development of biological degradation, and whose performance is directly dependent on the activity of microorganisms. The parameters that affect its development must be properly adjusted and monitored during composting. The most common control parameters are temperature, humidity, pH, C/N ratio and particle size (Petric et al., 2012). The correct control of the process contributes to obtain a stable, storable and transportable final product that is suitable for soil application. This final product, compost, improves soil characteristics by increasing infiltration, water retention, and thermal inertia; it also favors natural control of pests and provides nutrients for plant growth (Wei and Liu, 2005).

The raw materials processed by composting are very diverse in composition and characteristics. However, it is possible to obtain good quality composts irrespectively of materials employed provided that adequate management of the process control parameters is applied (Goyal et al., 2005). Conversely, assuming that every process, whatever its starting material is, will work the same, may lead to the production of immature or low agronomic quality compost (Silva et al., 2014). Consequently, it is essential to consider the peculiarities of each raw material and composting conditions should be adjusted accordingly (Goyal et al., 2005).

A composting process that is not well-performed results in an insufficiently stabilized organic matter or an immature compost that can affect soil environment and plant growth, be a source of disease and cause damage to crops by phytotoxicity (Cui et al., 2017). Compost quality is closely related to its stability and maturity. Maturity describes the ability of a product to be used effectively in agriculture for the growth of plants and relates to phytotoxicity aspects. Stability is the resistance of the organic matter in compost against further microbial decomposition, it is not equivalent to maturity, although both characteristics are often correlated since stability indicates the absence of biodegradable material, which includes several phytotoxic substances (Oviedo-

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Ocaña et al., 2015). One of the most employed methods for the evaluation of phytotoxicity is the determination of the germination index (GI) (Barral and Paradelo, 2011). The simplicity, short term, low cost and adaptability of the method for all types of substrates makes it preferable to other options. Despite some studies questioned the interpretation of GI results because different procedures are employed (Luo et al., 2018), it has been widely used in composting to detect operational problems in facilities and validate final compost quality (Barral and Paradelo, 2011). When properly performed, composting promotes the loss of phytotoxicity and may generate a product with phytostimulant effect (Selim et al., 2012).

The success of composting as an eco-friendly technology for waste management has caused the volume of waste treated in this way to explode in recent years (Ros et al., 2006). Composting is becoming a fundamental tool in the circular economy model promoted in Europe and the number of industrial composting facilities is growing throughout European geography (Razza et al., 2018). This new reality needs to be managed with appropriate rigor. There are big differences between industrial composting and small-scale composting. At small-scale the control of the process is high and the results are usually excellent if properly managed. Although high-quality composts can be obtained in industrial composting, the products generated are usually very variable in composition and characteristics (Barrena et al., 2014). Also, the situation of saturation faced by many industrial facilities is one of the main obstacles that limit the management of the process and the quality of composts obtained. In the particular case of facilities processing municipal solid waste, saturation joins an ineffective organic waste separation that causes the compost produced cannot be used as soil organic amendment. The use of simple tools such as GI for monitoring process performance and compost quality in industrial composting facilities would help to address operational weakness and improve waste processing for the production of quality compost. GI is listed in the quality assessment regulation of compost for commercialization in most European countries (Cesaro et al., 2015). Besides, other conventional physicochemical characteristics and biological activities are also required to fully evaluate the quality of compost obtained (Azim et al., 2018) that in turn may be correlated to GI (Luo et al., 2018).

This study aimed to compare the evolution of phytotoxicity during composting and final compost quality of industrial composting facilities processing different raw materials. For this, a comparative analysis of 15 industrial composting facilities that use five different starting raw materials, i.e. vegetal residue (VR), sewage sludge (SS), municipal solid waste (MSW), agri-food waste (AW) and olive mill waste (OMW) was performed. The phytotoxicity was determined throughout the composting process by measuring germination index (GI) and relationships were established for final products with physico-chemical parameters and heavy metals content to identify those that most influence the GI values and the quality of compost produced.

2. Material and methods

This section is aimed to describe the methodology used in this work. First, the detail of the composting facilities that were part of the study and the sampling method is described. Subsequently, all the analytical methods are detailed. Finally, the data analysis used is presented. To facilitate the understanding of the methodology, Table 1 and a Supplementary Fig. 1 are provided.

2.1. Composting facilities and sampling

The samples used for the experimental activity were collected at 15 full-scale composting facilities operating in the Southeast of Spain (Almeria, Granada, Murcia, and Jaen). The facilities were classified according to the main input waste: vegetal residue (VR), sewage sludge (SS), municipal solid waste (MSW), agri-food waste (AW) and olive mill waste (OMW), which is the pomace obtained from two-phase centrifugation olive mill decanter. Although this later residue is an agri-food waste coming from olive processing for olive oil extraction, it was considered separately from other agri-food wastes because of their special characteristics (Albuquerque et al., 2006). Three facilities were sampled for each input waste in order to have enough representativeness. Table 1 shows the facilities and their main treatment characteristics.

Samples were collected in each facility at significant composting phases as follows: mixture used for composting (raw materials, RM); material before reaching thermophile temperatures (mesophilic phase, MES); material at thermophile temperatures (50 °C) (thermophilic phase, TER); material after thermophilic phase was accomplished and temperature decreased up to ambient (cooling phase, COO); material at the middle of maturation phase (MAT); and final product (FP). A total amount of 90 samples (5 input wastes, three facilities per waste and six composting phases for each) were collected from November 2016 to November 2017. The entire sampling process took place in one day in facilities working in continuous, i.e. vegetal residue (VR), sewage sludge (SS), and municipal solid waste (MSW), while those processing agri-food waste (AW) and OMW, as they worked discontinuously because of the seasonality of the raw material, sampling was performed when each specific composting phase was reached.

Samples were taken from composting/raw material piles by collecting a bulk-integrated sample obtained from equal amounts (about 300 g) collected from nine different locations in each pile, considering three different levels without repetition of depth, wide and length (Supplementary Fig. 1), giving a final mass of approximately 3 kg. Then, the integrated sample was manually mixed and reduced to three sub-samples of about 1 kg, which were later used to carry out all the analytical procedures. In the case of MSW samples, only the biodegradable fraction was analyzed (organic matter), which means that all improper materials (plastic, glass, metal, etc.) were manually removed. After this, the sample was shredded in Moulinex Cousine Companion HF800A13 (Moulinex, Barcelona, Spain) prior to analytical. Samples were immediately stored in vacuum bags and frozen at −20 °C. Before analysis, samples were thawed at room temperature for 24 h. Phytotoxicity analyses were performed in all samples, while final products were used for the analysis of pH, electrical conductivity (EC), organic matter (OM), reducing sugars (RS), phenolic compounds (PC), humic substances (HS), soluble organic carbon (SOC), $N{-}NH_4^+$, $N{-}NO_3^-$ and heavy metals, according to procedures explained in analytical. Samples for most chemical analyses were air-dried at 40 °C overnight and ground to <1 mm, while those for phytotoxicity analyses, conductivity, pH, RS, PC, $N{-}NH_4^+$, $N{-}NO_3^-$ and SOC were freshly processed.

2.2. Analytical methods

The dry matter of the samples was determined after 24 h at 105 °C in order to express all data on a dry weight basis. The total organic matter content (OM) was determined by weight loss on ignition at 550 °C for 3.5 h (Kakezawa et al., 1992). The electrical conductivity (EC), pH, $N{-}NH_4^+$ and $N{-}NO_3^-$ were determined in a 1:10 (w/v) water extract. $N{-}NH_4^+$ and $N{-}NO_3^-$ were analyzed in filtered water extract by using Hach 9663 probe (Hach, Loveland, USA) and Nitrachek 404 probe (KPG Products Ldt., Hove, United Kingdom).

Phytotoxicity tests were analyzed by measuring the germination index (GI) in seeds of *Lepidium sativum* L. according to the method

Table 1
Main characteristics of the industrial composting facilities.

Main input waste	Code	Mixture for composting	Method of composting[a]	BP[b] (months)	Total[b] (months)
Vegetal Residue	VR1	Mostly cucumber and zucchini crop residues: stalks, leaves	Open air- Turned windrows	3	4
	VR2	Mostly cucumber, zucchini crop residues: stalks, leaves	Open air- Turned windrows	3	4
	VR3	Mostly pepper crop residues: stalks, leaves	Open air- Turned windrows	1	3
Sewage Sludge	SS1	Sewage sludge + straw (1:1 v/v)	Open air- Turned windrows	2.5	3.5
	SS2	Sewage sludge + pruning wastes (1:1 v/v)	Open air Turned windrows	2	3
	SS3	Dried sewage sludge (1:2 v/v)	in-vessel- Tunnel composting (turning by augers)	1	3
Municipal Solid Waste	MSW1	Municipal solid waste[c]	in-vessel Turned windrows in bays	1.5	3.5
	MSW2	Municipal solid waste[c]	in-vessel Turned windrows in bays	2.5	4.5
	MSW3	Municipal solid waste[c]	in-vessel Tunnel composting (turning by augers)	1	3
Agri-food Waste	AW1	Citric sludge[c] + palmtree prunings (1:3 v/v)	Open air- Turned windrows	5	8
	AW2	Cull tomatoes + tomato plant (stalks and leaves)	Open air- Turned windrows	4	6
	AW3	Citric sludge[d] + pig slurry + pruning wastes (mainly palmtree) (3:1:1.5 v/v)	Open air- Turned windrows	2	4
Olive Mill Waste	OMW1	OMW + chicken manure + straw (20.2:3.6:1 w/w)	Open air -Turned windrows	6	8
	OMW2	OMW + olive leaves + manure (12.5:3.5:1 w/w)	Open air - Turned windrows	5	7
	OMW3	OMW + manure + olive leaves (1:0.45:unknown w/w)	Open air - Turned windrows	3	5

[c] MSW: All facilities processed mixed municipal solid waste.

[c] Citric sludge: semi-solid residue in the form of juice centrifugation pulp obtained as waste products.

[a] Composting piles had variable dimensions depending on composting method. Most piles were about 2–3m high, 7–15 m length and 3–5 m width.

[b] Duration of biooxidative phase-BP (phase with thermal fluctuations); Total time of composting process.

of Zucconi et al. (1981). Briefly, water was mixed with compost samples to reach a moisture content equivalent to 65%. After 30 min contact time, a 10% aqueous extract was obtained and filtered through a 0.45 μm pore size membrane, 4 mL of the filtrate was added to 12 cm square dishes containing 25 seeds of *L. sativum* on top of one sheet of filter paper as support. Four replicate dishes were used for each sample providing a total of 100 testing seeds. Controls with 4 mL distilled water were used as reference. The seeds were placed in a growth chamber at 25 °C for 48 h in darkness. After this period, the number of seeds germinated was counted and the radical length was measured. The Germination Index (GI) was calculated as shown in equation (1) by multiplying the germinated seed number (G) and length of roots (L) and expressed as percentage (GI %) with respect to the control as follows:

$$GI\,(\%) = (Ls \times \%Gs) / (Lc \times \%Gc) * 100 \qquad (1)$$

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.

Soluble organic carbon (SOC), reducing sugars (RS) and phenolic compounds (PC) were analyzed in a K_2SO_4 extract according to López-González et al. (2013) as follows: 10 g of sample were added of 40 mL of 0.5 M K_2SO_4, after shaking at 200 rpm and 30 °C for 30 min, the extract was recovered by filtration. Soluble organic carbon (SOC) was measured in the extract by using TOC – VCSN analyzer (Shimadzu Co., Kyoto, Japan). Reducing sugars (RS) were analyzed by the DNS method (Miller, 1959), in which 1 mL of the extract was added of 3 mL DNS, after heating at 100 °C for 15 min, 200 μL of this solution were placed in 96 well microplates and absorbance was measured at 550 nm in microplate reader spectrophotometer Eon (Biotek Winooski, VT, USA), obtaining reducing sugar concentration by using glucose standard curve. Total phenolic compounds (PC) were analyzed according to Marambe and Ando (1992) using Folin-Ciocalteau reagent. Briefly, 0.5 mL of extract was mixed with 7 mL distilled water and 0.5 mL Folin-Ciocalteau reagent (Sigma-Aldrich, Missouri, USA), after 3 min contact, 1 mL of 20% (w/v) Na_2CO_3 and 3 mL distilled water were added and the mixture was left in darkness for 30 min. Finally, 200 μL were transferred to 96 well microplates and absorbance was measured at 725 nm in a microplate spectrophotometer reader Eon (Biotek Winooski, VT, USA). Total phenol concentration was obtained by using a standard curve made with p-hydroxybenzoic acid (Sigma Aldrich Corporation, St. Louis, USA).

Humic substances (HS) were extracted using a sodium pyrophosphate alkaline solution and fractionated depending on their solubility at different pH values, according to Ciavatta et al. (1991), as follows: 2 g of dried sample was added of 100 mL sodium pyrophosphate (pH 13, 0.1N) and shaken at 120 rpm and 60–65 °C for 48 h. The total humic extract (THE) was separated from solid material by filtration through cellulose acetate membrane 0.8 μm (Sartorius Group, Göttingen, Germany). An aliquot of 25 mL of the was acidified up to pH 1.5 with 9M H_2SO_4 and the precipitated humic acid (HA) fraction was separated from supernatant containing non-acid precipitable fraction by centrifugation at 5000 rpm for 20 min. The HA fraction was solubilized in 25 mL 0.1M sodium pyrophosphate for total organic carbon analysis. The supernatant containing non-acid precipitable fraction was passed through a 5 cm^3 polyvinylpyrrolidone (PVP) packed column pre-equilibrated with 0.005 M H_2SO_4, after adding 25 mL 0.5N NaOH, the flow-through was collected as the fulvic acid (FA) fraction. Total organic carbon was measured in HA and FA fractions by using a TOC – VCSN analyzer (Shimadzu Co., Kyoto, Japan). The maturation index was calculated as the ratio between humic and fulvic acid carbon (HA/FA).

Heavy metals (Cr, Cu, Cd, As, Ni, Zn, and Pb) were analyzed in 0.15 g of samples digested with 6 mL HNO_3 and 2 mL H_2SO_4 at 200 °C for 30 min in high-pressure microwave digester (Milestone Ethos Touch Control; Milestone SRL, Sorisole, Italy) by inductively coupled plasma mass spectrometry (ICPMS) (Thermo Scientific XSeries 2ICP-MS; Thermo Fisher Scientific, Waltham, USA).

2.3. Data analysis

Analytical was performed at least in triplicate. Simple descriptive analysis was applied to determine the average value of the quantitative variables. The mean values of each parameter were tested for statistically significant differences using one-way analysis of variance (ANOVA), considering the facility and the raw material, and the comparison of parameter means was performed by the Fisher's Least Significant Difference (LSD) at $P < 0.05$. Normality and homogeneity of the variances were checked using the Shapiro–Wilk and Levene tests, before ANOVA. The presence of categories within final compost samples collected from facilities composting different raw materials was investigated using stepwise linear discriminant analysis (DA), in order to find simple equations for estimation of the composition of these wastes from easily analyzable parameters. In addition, the Spearman correlations between the different parameters studied were calculated and multiple regression analysis with stepwise selection of variables was carried out. All of these analyses were carried out using Statgraphics Centurion XVII version 17.1.1 (Stat-Point, Inc., Virginia, USA).

3. Results and discussion

The results are described and discussed in this section. The evolution of phytotoxicity in the materials throughout the main composting phases is presented, followed by a detailed description of the final composts produced. Finally, correlation and discriminant analyses of results obtained for final products are discussed.

3.1. Evolution of phytotoxicity during composting

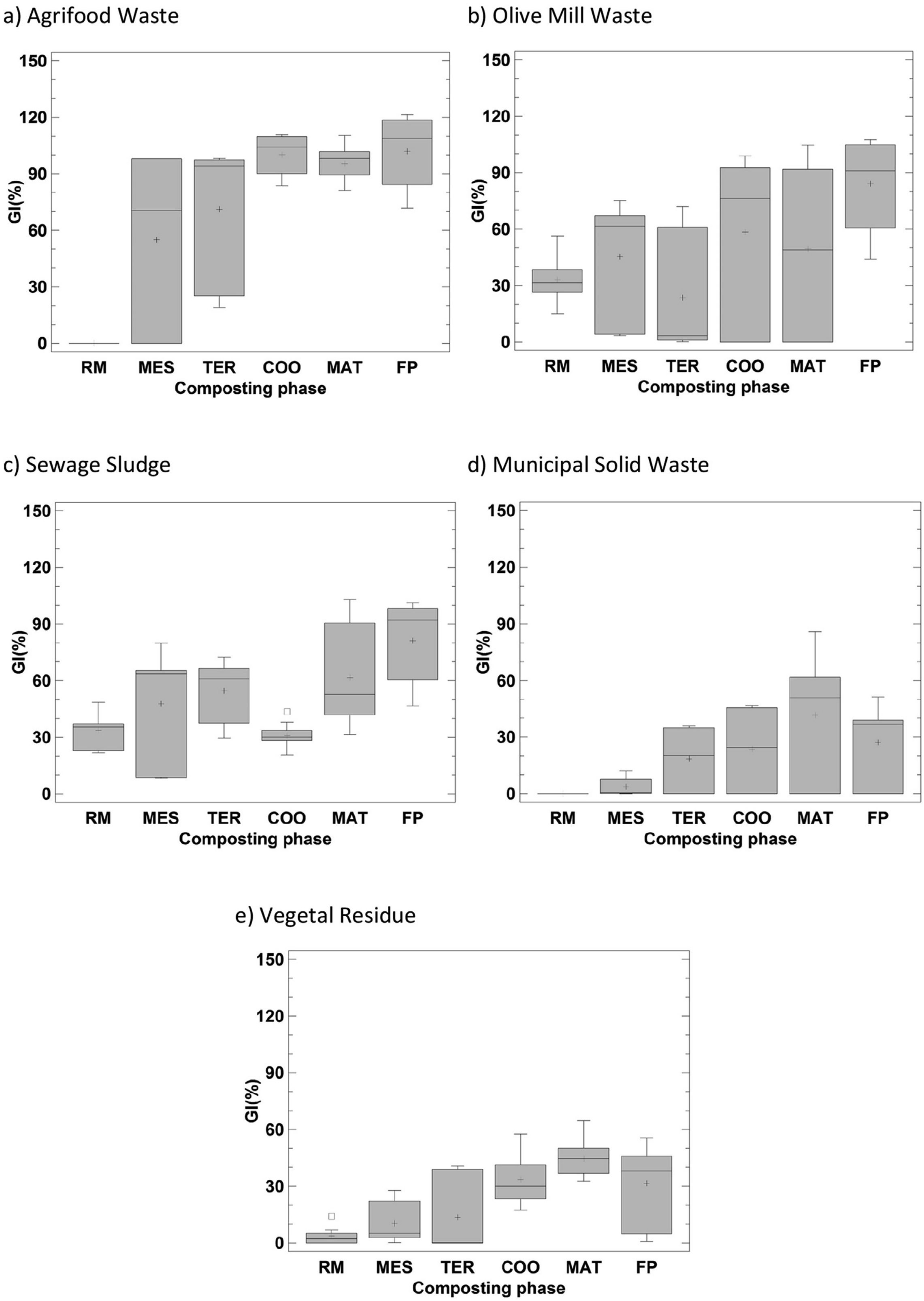
The evolution of the germination index (GI) was studied throughout all phases of composting in 15 industrial facilities processing five different raw materials, three for each material, whose results per raw material are shown in Fig. 1. Detailed GI values for each facility can be found (Supplementary Table S1). According to Emino and Warman (2004), materials with GI values lower than 50% are highly phytotoxic, between 50% and 80% are moderately phytotoxic, higher than 80% are no-phytotoxic, and materials with a GI higher than 100% have phytonutrient or phytostimulant effect. All raw materials were phytotoxic, having GI values far below 50% that is relatively common for unprocessed materials (Selim et al., 2012). In general, GI values showed an increasing trend from the beginning of the composting process, as it has been also reported in composting of poultry manure (Young et al., 2016) and other animal manures (Huang et al., 2017), and co-composting of sewage sludge and organic fraction of municipal solid waste (Zhang et al., 2018). However, the behavior was quite different depending on the raw materials. In the case of Olive Mill Waste (OMW), Sewage Sludge (SS) and Agrifood Waste (AW) GI reached values above 80% at the end of the process or earlier, thus losing phytotoxicity. On the contrary, in the facilities processing Municipal solid waste (MSW) and Vegetal residue (VR), the material never had GI values higher than 50%, so even final compost was phytotoxic. Besides, it is necessary to emphasize that there was an important variability of GI values between facilities processing similar raw materials, which was evidenced by the wide range of GI values obtained, especially at intermediate phases of composting (mesophilic, thermophilic, cooling and maturation) that was much lower for final products. This is typical for industrial composting (Barrena et al., 2014) and may relate to differences in processing practices between facilities, including, initial mixture composition, method of composting, time to reach final product and intermediate operations such as turnings, moisture adjustment, fresh material addition during intermediate phases, etc. Unfortunately, it was not possible to find a pattern to explain differences in GI values based on processing parameters other than those shown in Table 1 because of the scarce information provided by some industrial facilities where the process was subjected to little control and monitoring.

Agri-food waste was the material that better performed and sooner reached GI higher than 80%. An average GI of 100% was obtained at the cooling phase and remained around this value up to the end of the process with slight differences between the three facilities (Fig. 1a). The main dispersion of results among facilities was obtained at mesophilic and thermophilic phases. In two of the three facilities that processed citric sludge (AW1) and cull tomatoes (AW3) as the main input residue, non-phytotoxic levels were reached even at the mesophilic and thermophilic phases; while for the third facility that processed citric sludge (AW2), this occurred at the cooling phase, having GI values close to zero at the mesophilic phase. The difference between this last facility and the other that processed citric sludge (AW1) was the use of pig slurry in the initial mixture that could be the reason of the shorter duration of the bio-oxidative phase (2 months) and the different pattern of GI evolution during initial phases. However, this did not impact much the evolution of phytotoxicity in further phases following a similar pattern than in the other facilities.

In the case of Olive Mill Waste, GI evolution was very different in the three facilities, final compost had GI average values around 84%, one of them was phytotoxic (GI 55%) while the other two had GI>80% (Fig. 1b). A wide dispersion of results was obtained for the three facilities at all intermediate phases. The material from one facility (OMW3) had GI values close to zero up to maturation phase, in other facility (OMW2) GI peaked at cooling phase (78%) but then decreased to 55%, while in the third facility (OW1) an increasing trend was obtained throughout composting and material lost phytotoxicity at cooling phase. This shows that differences in operating conditions together with the composition of initial mixture (Table 1) may have a great impact on GI other than the type of main input waste. Further studies are required to determine the influence of operating conditions at industrial scale on the GI results.

For the Sewage Sludge, the differences in GI values between facilities were some of the lowest (Fig. 1c). This parameter increased continuously for all three facilities, except at the thermophilic phase in which GI decreased, likely due to the release of phytotoxic compounds such as phenolic acids, organic acids and ammonia during organic matter decomposition and the increased solubility of heavy metals (Guo et al., 2012). The final compost had an average GI of 81%, ranging from 99.8% to 52.7%.

The two materials that worst performed were Municipal Solid Waste (MSW) and Vegetal Residue (VR) whose phytotoxicity was not eliminated during composting and led to phytotoxic final products (GI<50%) (Fig. 1d and e). The justification for this result cannot be restricted only to the nature of the waste because there are several successful lab or small-scale composting experiences with VR (Gavilanes-Terán et al., 2016) and MSW (Obuotor and Odeyemi, 2017) that led to non-phytotoxic compost. The reasons for the poor performance likely lies in the management procedures in the facilities. In the case of Vegetal Residue (VR), the three facilities processed plant residues from intensive horticulture whose management is complicated by the presence of non-biodegradable polyethylene ropes used for staking plants that are tightly intertwined to the plant. These elements are manually removed in small scale composting (López-González et al., 2013) but at the industrial



a) Agrifood Waste

b) Olive Mill Waste

c) Sewage Sludge

d) Municipal Solid Waste

e) Vegetal Residue

Fig. 1. Evolution of phytotoxicity (germination index, GI) during the composting phases (raw materials, RM; mesophilic phase, MES; thermophilic phase, TER; cooling phase, COO; maturation phase, MAT; and final product, FP) in industrial facilities processing a) Agri-food Waste, b) Olive Mill Waste, c) Sewage Sludge, d) Municipal Solid Waste, and e) Vegetal Residue. Symbol + in box-plot indicates mean values (n = 9, 3 facilities x 3 replicates).

scale, they usually remain with plant stalks and leaves up to cooling phase and are removed further by passing the material through a trommel. The fine plant material recovered is not yet stabilized but it is not subjected to further processing other than accumulating it in piles for maturation where it tends to warm again. Consequently, the material cannot be considered finished and this is why final material retains the phytotoxicity. Besides, it is a common practice to use composting leachates to water the windrows that also affect phytotoxicity levels. This situation can also be applied to MSW processing facilities but in this case, the inert materials mixed with organic matter are the elements that are retained up to the maturation phase, in which they are separated from organic matter by sieving. In fact, the process performed at the MSW facilities studied is not considered composting as such but mechanical-biological treatment. Because of procedures required at an industrial scale for processing RV and MSW, the material was probably finished early without being subjected to a sufficient thermal profile to ensure proper biotransformation. The presence of unstable organic material in both processes are expected to cause phytotoxicity in the final materials (Cesaro et al., 2019). Also, in both cases, the addition of fresh material during intermediate phases could have been produced because of saturation of the volume of waste to be treated, and it is something that obviously could affect the composting process.

The correct management of the process together with the favorable nature of the waste are the two great challenges that lie ahead in the field of industrial composting. Germination index is a parameter easy to determine that could help to monitor the performance of composting in industrial facilities as well as to verify final product safety. However, it is important to use the integrated information of different parameters when evaluating the risk of the application of organic material to the soil. The relationship between this parameter and other characteristics in the final products obtained in each facility is discussed in the following section.

3.2. Final products characterization

Several physico-chemical parameters and heavy metals content were analyzed in final products from each facility and correlation and discriminant analysis were performed in order to find relationships between maturity, expressed as GI, and parameters determined in the samples.

3.2.1. Physico-chemical parameters

Table 2 shows the values of physico-chemical parameters in final products obtained from each industrial facility which were likely to affect the phytotoxicity and help to define the quality of final products. In general, there were significant differences for nearly all parameters among compost from different facilities, even for those coming from the same initial materials.

High pH and electrical conductivity values in compost are known to affect seed germination (Barral and Paradelo, 2011). Most final products had pH values in the range of those established as usual for compost (pH 6–9) (Cayuela et al., 2008), except the compost from VR1, VR3 and OMW2 facilities that had pH values of 9.18, 9.68 and 9.46. These alkaline pH may partially explain the low GI values of the compost, being 2.7, 46.4 and 55.0%. The electrical conductivity (EC) varied in a wide range (2.44–17.36 mS cm^{-1}) in compost analyzed. Compost from Vegetal Residue (VR) and one from MSW (MSW3) had the highest levels of electrical conductivity (higher than 8 mS cm^{-1}), and also showed high phytotoxicity (low GI values), especially for VR2 and MSW3, whose EC value (>10 mS cm^{-1}) was probability responsible for the low GI value of the compost (2.66 and 0%). In the case of compost from VR, the justification to the high EC values may be related to the origin of the plant residues processed at the facilities. As stated above, they are plant residues from the intensive horticulture that uses inorganic fertilization and lead to residues having high conductivity because the salts are not lost through irrigation of the vegetal, but instead concentrate on the residue as a result of drip irrigation practices carried out in the study area, where water losses are minimal (Aznar-Sánchez et al., 2011). However, these residues have been successfully composted at pilot plant scale (López-González et al., 2013). Other causes that are intrinsic to industrial scales, such as the use of leachates from composting to water the piles, may be the main reason for the high EC levels and the resulting phytotoxicity. This explanation may be also applied to MSW3 compost, but in this case, the complex mixture of raw materials from mixed municipal solid waste could add a factor to consider.

Organic matter (OM) of final products was in the range of values described in the literature for composts, 30–60% (Fialho et al., 2010) except for compost AW1 and OMW3 whose OM content was higher than 70%. Surprisingly none of them were phytotoxic, having GI above 80%. Other studies found a negative correlation between the amount of organic matter and the GI in materials such

Table 2
Properties of final compost from industrial composting facilities[a].

Compost[b]	pH		EC (mS cm^{-1})		OM (%)		RS (%)		PC (%)		SOC (%)		NH_4^+ (%)		NO_3^- (%)		HA/FA		GI (%)	
VR1	9.18	i	8.48	e	48.43	de	0.12	de	0.021	d	0.68	gh	0.05	cd	0.16	cd	0.54	a	45.79	bc
VR2	8.08	f	17.36	g	63.30	i	0.21	g	0.008	bc	0.48	f	0.14	g	0.34	g	1.40	def	2.66	a
VR3	9.68	k	9.97	f	39.64	c	0.09	bcd	0.028	e	0.38	ef	0.08	ef	0.30	fg	0.98	abcd	46.43	bc
MSW1	8.66	h	4.97	c	53.91	g	0.11	d	0.017	d	0.61	g	0.04	c	0.20	de	1.78	f	32.73	b
MSW2	7.50	d	5.58	c	38.05	c	0.22	gh	0.018	d	0.38	e	0.06	de	0.28	f	1.17	bcd	45.31	bc
MSW3	6.00	a	10.29	f	63.65	i	0.26	h	0.007	abc	1.78	j	0.04	c	0.05	a	0.96	abcd	0.00	a
SS1	7.72	e	4.67	bc	47.19	d	0.07	abc	0.033	f	0.27	d	0.08	f	0.08	ab	1.32	cde	91.08	de
SS2	8.26	g	2.72	a	26.09	a	0.06	ab	0.005	ab	0.16	bc	0.05	cd	0.10	ab	1.76	ef	99.80	ef
SS3	8.52	h	5.52	c	50.18	ef	0.22	g	0.010	c	0.23	cd	0.07	ef	0.08	ab	1.13	bcd	52.68	c
AW1	6.64	b	7.24	d	74.07	j	0.10	cd	0.005	ab	0.01	a	0.01	a	0.25	ef	0.87	abc	115.64	g
AW2	7.83	e	5.10	c	49.10	de	0.04	a	0.008	bc	0.10	ab	0.04	bc	0.29	fg	0.52	a	112.89	fg
AW3	8.67	h	2.72	a	52.10	fg	0.10	cd	0.004	ab	0.14	bc	0.08	f	0.12	bc	0.74	ab	77.46	d
OMW1	7.23	c	4.57	bc	32.30	b	0.16	ef	0.003	a	0.33	de	0.01	a	0.12	bc	1.40	def	106.54	fg
OMW2	9.46	j	3.55	ab	60.23	h	0.19	fg	0.005	ab	0.73	h	0.03	ab	0.26	f	2.84	g	55.01	c
OMW3	8.62	h	2.44	a	82.07	k	0.61	i	0.003	a	1.28	i	0.01	a	0.08	ab	1.31	cde	81.66	d

[a] Data are mean values (n = 3), those with the same letter in the same column are not significantly different from each other (LSD, $p < 0.05$). Abbreviations: EC: electrical conductivity; OM: organic matter; RS: reducing sugars; PC: Phenolic compounds; SOC: soluble organic carbon; HA/FA: humic acids/Fulvic acids ratio; GI: germination index. All data are on a dry weight basis.

[b] Compost code as in Table 1: Vegetal Residues (VR), Municipal Solid Waste (MSW), Sewage Sludge (SS), Agri-food Waste (AW), Olive Mill Waste (OMW).

as sewage sludge (Kebibeche et al., 2019). However, it seems that it is the biodegradability of organic matter nor its amount the factor that could influence the phytotoxicity. The soluble fractions of organic matter, i.e. soluble organic carbon (SOC) and reducing sugars (RS), constitute the main source of energy for the growth and activity of the microorganisms, therefore, similar to total organic matter, they decrease during composting (Zmora-Nahum et al., 2005). Both parameters were in typical values for composting, except for OMW3 compost that had the highest SOC and RS values, as it was also for OM content, with none relation with GI values.

The content of any of the inorganic forms of nitrogen in the compost is of great importance to determine its quality (Cesaro et al., 2019). In the compost analyzed, the amount of ammonia nitrogen (NH_4^+) was relatively low in comparison to nitric nitrogen (NO_3^-) which is related to the stabilization of organic matter. According to Bernal et al. (1998) high $N{-}NH_4^+$ or $N{-}NH_4^+/N{-}NO_3^-$ ratio higher than 0.16 lead to phytotoxic effects. However, in this study, this effect was not noticed since compost with these characteristics were not phytotoxic.

Phytotoxicity is related to the high concentration of phenolic compounds (Pinho et al., 2017). This fraction reached the highest values (>0.03%) in compost from Vegetal Residue VR3 and Sewage Sludge SS1. In the first case, phenolic compounds could contribute to the phytotoxicity of material (GI 46.4%) but that was not the case for SS1 compost that was not phytotoxic (GI 91%). It should be noted that the compost produced from OMW, a residue containing a high phenolic load (Dermeche et al., 2013), had the lowest phenolic compounds concentration because of their depletion during the composting process.

The degree of maturity of compost is one of the most important factors affecting its successful application for agricultural purposes (Said-Pullicino et al., 2007). The composting process involves the formation of fulvic acids (FA) as an intermediate step in the formation of humic acids (HA). The presence of humic fractions favors high GI values that relate to decreased phytotoxicity (Xi et al., 2016). In addition, they positively affect both the microbiota and the physico-chemical parameters of the soil (Suárez-Estrella et al., 2008). Because maturation implies the formation of humic substances, the degree of OM humification (HA/FA) is generally accepted as a criterion of maturity, immature composts have values less than 1, and greater than 1 or 3 for mature composts (Azim et al., 2018). The analyses carried out on the 15 composts of this study revealed that the HA/FA ratio was in the range of 0.58−1.78. All compost from OMW had AH/AF ratio higher than 1 but one of them was phytotoxic. On the contrary, all AW compost had AH/AF lower than 1 but none were phytotoxic. In addition, this relationship was neither found for the most phytotoxic compost, i.e. MSW and VR. This result may have two main outcomes, i) industrial facilities should ensure time spent in the process is sufficient to generate mature products; ii) the relationship between maturity indexes such as AH/AF ratio and phytotoxicity should be revisited.

3.2.2. *Heavy metal levels*

The content of heavy metals of compost is a limiting factor necessary to analyze for its safe application as an organic amendment in soil (Alvarenga et al., 2015). Table 3 shows the content of heavy metals of the composts from the different composting facilities, the limit values from Spanish legislation Royal Decree 506/2013 (BOE, 2013) and the corresponding classification based on these limits (Class A for high quality, Class B for medium and Class C for low quality). All heavy metals considered in the current regulation except mercury were analyzed. In addition, arsenic was also included in the study due to its growing importance as an emerging pollutant (Attanayake et al., 2015). Compost from sewage sludge and municipal solid waste have usually high heavy metals content (Alvarenga et al., 2015) while those produced from vegetal residues are considered optimal in terms of their potential heavy metals levels (Farrell et al., 2010). This was also detected in this study. The composts with the lowest content of heavy metals were those from OMW, VR, and AW. Only the copper content, slightly higher than the limit, prevented some compost from being Class A. On the contrary, all the composts from sewage sludge fit class B because of its copper and zinc content. Similar results were obtained by Alvarenga et al. (2015) for sewage sludge compost. The highest levels of heavy metals were found in compost from MSW. In two of the three compost (MSW1 and MSW3), the levels of some heavy metals were higher than the limit established for lower quality compost (Class C). The high values of these elements could be

Table 3
Heavy metal content of final compost from industrial composting facilities and the limit values from legislation.[a].

Compost[b] /Limit[c]	Cr (mg kg^{-1})		Cu (mg kg^{-1})		Cd (mg kg^{-1})		As (mg kg^{-1})		Ni (mg kg^{-1})		Zn (mg kg^{-1})		Pb (mg kg^{-1})		Compost Class[c]
VR1	3.51	b	91.87	bcd	0.25	ab	5.10	de	2.27	b	125.22	e	9.66	ab	B
VR2	1.18	a	53.78	ab	0.13	ab	1.09	a	1.61	ab	47.61	bc	3.30	ab	A
VR3	5.75	c	109.76	def	0.43	bcd	2.88	c	1.24	a	117.58	e	5.25	ab	B
MSW1	18.31	e	104.35	def	3.99	f	5.80	e	11.29	f	526.31	k	92.51	e	–
MSW2	28.40	h	135.87	f	0.90	e	2.39	bc	12.67	g	287.46	g	69.19	de	B
MSW3	13.51	d	1052.11	g	4.33	f	2.73	c	10.08	e	207.51	f	3021.58	f	–
SS1	26.50	g	118.80	def	0.65	de	4.71	d	20.44	i	382.55	j	62.54	cde	B
SS2	43.03	i	114.93	def	0.44	bcd	18.31	f	15.77	h	363.48	i	33.89	bc	B
SS3	80.06	j	134.89	ef	0.76	de	3.28	c	23.76	j	338.81	h	50.64	cd	B
AW1	24.77	f	30.84	a	0.06	a	0.99	a	12.59	g	56.56	c	6.16	ab	A
AW2	7.13	c	140.48	f	0.28	abc	5.74	de	3.35	c	48.58	bc	31.02	abc	B
AW3	18.65	e	96.44	cde	0.61	cde	1.45	ab	9.76	e	101.13	d	14.35	ab	B
OMW1	44.20	i	36.20	a	0.13	ab	1.63	ab	15.10	h	23.01	a	4.32	ab	A
OMW2	18.62	e	63.89	abc	0.11	ab	0.74	a	15.04	h	41.93	b	3.89	ab	A
OMW3	6.28	c	31.76	a	0.04	a	0.66	a	4.32	d	15.11	a	0.92	a	A
Limit (Class A)	70		70		0.7		–		25		200		45		
Limit (Class B)	250		300		2		–		90		500		150		
Limit (Class C)	300		400		3		–		100		1000		200		

[a] Data are mean values (n = 3), those with the same letter in the same column are not significantly different from each other (LSD, $p < 0.05$). All data are on a dry weight basis.

[b] Compost code as in Table 1: Vegetal Residues (VR), Municipal Solid Waste (MSW), Sewage Sludge (SS), Agri-food Waste (AW), Olive Mill Waste (OMW).

[c] Heavy metal limit and compost classification according to Spanish Regulation for organic fertilisers (Real Decreto 506/2013).

among the causes of the high phytotoxic level of MSW compost, as it has been also found in composting of sewage sludge and swine manure (Miaomiao et al., 2009). The compost of MSW1 contained 526.31 mg kg^{-1} of zinc, the highest value obtained. Even more relevant was the case of MSW3 final product whose levels of copper (1052.11 mg kg^{-1}) and lead (3021.58 mg kg^{-1}) prevent it to be used as an organic amendment. In fact, MSW final products analyzed are currently disposed of by landfilling. In these facilities, the bio-stabilization of mixed municipal waste through mechanical-biological treatment is pursued before landfilling so the product cannot be considered compost. Finally, arsenic reached the highest levels in compost from sewage sludge SS2 (18 mg kg^{-1}) but it was quite low in other compost. This element has been found in MSW compost at levels of 7.6 mg kg^{-1} (Montejo et al., 2015), also in industrial sludge from a treatment plant which cleans groundwater contaminated by arsenic (Maňáková et al., 2014) and compost from arsenic-rich hyperaccumulator *Pteris vittata* (Cao et al., 2010). Owing to the absence of threshold values for arsenic in Royal Decree 506/2013 (BOE, 2013), the results obtained can be compared to other international standards for compost quality such as those from United States Environmental Protection Agency that limits arsenic to 41 mg kg^{-1} (U.S. EPA, 2000), higher than the values obtained in the samples..

3.3. Correlations and discriminant analysis

A correlation analysis was carried out between physico-chemical parameters and GI values of compost (Table 4). The parameter with the highest significant correlation with GI was SOC ($R^2 = -0.68$). Cui et al. (2017) suggested a relationship between the amount of soluble fresh matter and the presence of phytotoxic compounds in compost. The other parameters evaluated did not significantly correlate with GI even though the correlation for some of them is well described in the bibliography (Barral and Paradelo, 2011). According to this, the chemical properties of compost were hardly correlated with maturity parameter used (GI). In all likelihood, the reason for this lies in the high heterogeneity of the compost samples analyzed in the study. However, the discriminant analysis separated the composts into well-differentiated groups on the basis of the raw materials. DA was applied to all parameters determined in this study except heavy metals and AH/AF ratio that were excluded in the stepwise DA because their tolerance level was below the established minimum ($p < 0.05$). The DA grouped samples according to the raw materials (Fig. 2). Two discriminant functions were obtained that explained 87.66% of the variation. The first discriminant function accounts for 64.44% of the variation and separated the samples into three groups (AW, VR, and the other compost), mainly based on GI, pH and EC values. In VR compost, the presence of compounds that caused high pH and conductivity values was the main cause of phytotoxicity. The opposite case is represented by AW compost whose higher GI values may be due to its lower pH and EC and reflect that the industrial composting process significantly reduced the presence of phytotoxic compounds such as ammoniacal compounds among other. The second function, which explains an additional 23.22% of the variation, separated MSW compost from AW, SS, and VR compost, with OMW at a middle point between both groups. In this case, samples are also separated mainly by pH, EC and GI values, and additionally by OM content. MSW differentiated from the other compost because of their low GI values and high OM content which may indicate that compost is not finished and whose values together with pH and EC are far from the SS, AW and RV compost. In the case of OMW compost, the disparity of GI results was notable, likely due to the presence of basic compounds that cause the mild phytotoxicity detected in OMW2, but not in the rest of OMW facilities. This may have been the reason for the intermediate positioning of these samples along function 2. These results confirm the differentiation between the final compost according to the starting materials used in industrial processes. In summary, despite the high dispersion of data obtained from the different facilities, it was possible to find a common pattern for compost produced from similar raw materials. VR and MSW compost had phytotoxic effect mainly because of high pH and EC and incomplete organic matter stabilization. On the other hand, it is worth noting the remarkable differentiation that was registered in the compost from agri-food waste (AW), which, as indicated above, had the highest GI values. These results suggest

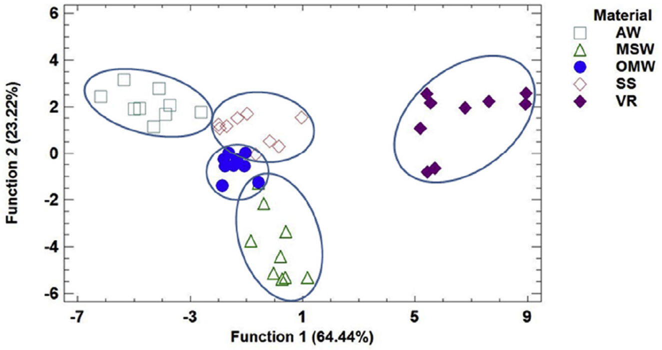


Fig. 2. Discriminant analysis loading plot of physico-chemical and GI data for final products from Vegetal Residue (VR), Municipal Solid Waste (MSW), Sewage Sludge (SS), Agri-food waste (AW) and Olive Mill Waste (OMW). Data are grouped in four classes: I, AW; II, OMW, SS; III, MSW; IV, RV.

Table 4
Spearman correlation matrix between germination index (GI) and physical-chemical parameters of finished compost (n = 15).

	pH	EC	OM	RS	PC	SOC	NH4	NO3	GI
AH/AF	0.09	-0.36	-0.04	0.21	-0.15	0.3	-0.14	-0.05	-0.1
pH		-0.18	-0.03	-0.05	0.18	0.24	0.23	0.19	-0.23
EC			0.12	0.12	0.49	0.16	0.32	0.33	-0.52
OM				0.45	-0.29	0.35	-0.25	-0.09	-0.22
RS					-0.24	0.67	-0.09	-0.22	-0.54
PC						0.03	0.57	0.2	-0.4
SOC							-0.13	-0.16	-0.68
NH4								0.14	-0.44
NO3									-0.04

Abbreviations: EC: electrical conductivity; OM: organic matter; RS: reducing sugars; PC: Phenolic compounds; SOC: soluble organic carbon; HA/FA: humic acids/Fulvic acids ratio; GI: germination index.
Blue colour represent positive correlation and red color negative correlation.

that it is essential to consider the intrinsic characteristics of each raw material to be composted since they are decisive to achieve a stable and non-phytotoxic compost. In addition, the operating conditions of the process would also have a high impact on the final material.

4. Conclusions

Industrial composting faces many obstacles that limit the quality of final products. Despite the high dispersion of characteristics of compost obtained from industrial facilities, this study confirms the effectiveness of the Germination index (GI) for monitoring composting performance at industrial scale and verify final product safety. The integration of other control parameters should be also considered to provide reliable information on compost quality for its safe use on soil. The phytotoxicity of OMW, SS, and AW was eliminated in industrial facilities at the cooling phase or the end of composting. In the facilities processing MSW and VR, the material never lost phytotoxicity. This was due to the high electrical conductivity and pH for RV compost and the heavy metal content for the MSW compost. All compost, except MSW, fulfill requirements of the legislation for heavy metal content.

Author contributions

The manuscript is written through contributions of all authors. All authors have given approval to the final version of the manuscript.

Declaration of competing interest

None.

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Appendix A. Supplementary data

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