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Stability and maturity of biowaste composts derived by small municipalities: Correlation among physical, chemical and biological indices.

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Abstract

Stability and maturity are important criteria to guarantee the quality of a compost that is applied to agriculture or used as amendment in degraded soils. Although different techniques exist to evaluate stability and maturity, the application of laboratory tests in municipalities in developing countries can be limited due to cost and application complexities. In the composting facilities of such places, some classical low cost on-site tests to monitor the composting process are usually implemented; however, such tests do not necessarily clearly identify conditions of stability and maturity. In this article, we have applied and compared results of stability and maturity tests that can be easily employed on site (i.e. temperature, pH, moisture, electrical conductivity [EC], odor and color), and of tests that require more complex laboratory techniques (volatile solids, C/N ratio, self-heating, respirometric index, germination index [GI]). The evaluation of the above was performed in the field scale using 2 piles of biowaste applied compost. The monitoring period was from day 70 to day 190 of the process. Results showed that the low-cost tests traditionally employed to monitor the composting process on-site, such as temperature, color and moisture, do not provide consistent determinations with the more complex laboratory tests used to assess stability (e.g. respiration index, self-heating, volatile solids). In the case of maturity tests (GI, pH, EC), both the on-site tests (pH, EC) and the laboratory test (GI) provided consistent results. Although, stability was indicated for most of the samples, the maturity tests indicated that products were consistently immature. Thus, a stable product is not necessarily mature. Conclusively, the decision on the quality of the compost in the installations located in developing countries requires the simultaneous use of a combination of tests that are performed both in the laboratory and on-site.

Keywords: composting; correlation; low-cost; maturity; stability.

1. Introduction

Composting is one of the most applied and effective technique for the treatment of the organic fraction of municipal solid wastes (biowastes) (Levis et al., 2010). In order to guarantee the safety of compost during its use in the agriculture, or as an amendment in degraded soils, certain quality criteria should be fulfilled. These are related to the content of pathogens, heavy metals, organic matter, nutrient content, stability and maturity (Soumare et al., 2003; Tognetti et al., 2007; Barral et al., 2007).

Stability is a term related to the resistance of the organic matter of a product against extensive degradation or towards major microbiological activity. Maturity describes the ability of a product to be used effectively in agriculture and is related to the growth of plants and to phytotoxicity aspects. In general, both criteria should be somehow correlated, since phytotoxic compounds are products of the microbial activity of unstable organic matter (Sullivan and Miller, 2001; Bernal et al. 2009; Komilis and Tziouvaras, 2009; Raj and Antil, 2011; Astrup et al., 2015). That is, an unstable or immature organic product hinders the growth of plants and negatively affects the quality of soil (Riffaldi et al., 1986; Wuet al., 2000; Tang et al., 2003).

A large number of physical, chemical and biological tests has been proposed to assess the stability and maturity of compost (Bernal et al., 1998; Barrena et al., 2006; Bernal et al., 2009); however, there is still no unique universally accepted test to assess both parameters (Komilis et al., 2011). Among the tests used, we can find some commonly used on-site and others that need a laboratory to be performed. The latter are usually characterized by complexity or demand higher costs and complicated logistics (Khalil et al., 2008).

In the developing countries, the composting of biowastes has not been an effective solution, which is one of the reasons of the generation of end-products that do not comply with quality standards (Hoornweg et al., 1999; OPS, 2005; Barreira et al., 2006; Kurian, 2007). The composting installations of small municipalities, which are herein defined arbitrarily as the ones with a population less than 12000 residents, are characterized by resource limitations as well as by the fact that their operation is usually realized by operators that are not well trained and cannot perform complex activities (Turan et al., 2009; Shekdar, 2009; Marmolejo, 2011).

In such installations, the stability and maturity of the product is usually determined on-site with relatively easy to perform techniques, such as odor, color, temperature, humidity, pH and electrical conductivity (Ruggieri et al., 2008). These techniques can be inconsistent with laboratory tests used to assess stability and maturity. For example, typical on-site quality criteria can be to obtain a dry material, of a dark color, of a relatively small particle size and of an odor similar that to soil. However, physical, chemical and biological quality analyses at the laboratory can indicate if the product meets the conditions of stability and maturity (Zucconi et al., 1981; CCQC, 2004; Icontec, 2003; Marmolejo, 2011; Cesaro et al., 2015).

The conditions of the operation of composting installations in small communities in developing countries are primarily governed by the lack of budget. On the other hand, there is a necessity to provide information on control schemes on the monitoring of the process and on the quality of the product. Based on the above, the primary objective of this work was to compare and correlate the results of the application of certain *laboratory* scale stability tests (volatile solids, C/N ratio, respirometric index, self-heating) and maturity tests (germination index) with some commonly employed *on-site* tests to assess stability (temperature, color, odor, moisture) and maturity (pH and electrical conductivity).

2. Materials and Methods

2.1 Initial conditions and experimental setup

The study was developed in an actual biowaste composting installation in Colombia that accepts source separated wastes derived from the selective collection of biowaste. The separate collection of the biowastes takes place 2 times per week, and the amount of biowastes collected per day is between 1.3 to 1.5 t. The wastes used here were organic and almost exclusively of residential origin, since commercial activities are very low in the area under study. Approximately 90% of the population performs a separation of biowastes at the source. The dominant constituents in those biowastes (93% of the total wet weight) were non-processed food, such as plantains and tubers, citric and other fruit as well as vegetables and legumes. The remaining 7% were processed food, some paper and cardboard, pruning and garden waste and non-biodegradable material. The average temperature in the location is 18°C and the average precipitation is 1500 mm/year. Monitoring of the quality of the biowastes that took place during the year showed that the raw material has a pH of 5.5±0.5, a moisture content of 76.7±3.2% (wet weight basis, wb), a TOC of 33.0±4.8% (dry weight basis, db), a total nitrogen content (N_T) of 1.6±0.5% (db), a C/N ratio of 21.7±5.3 and an ash content of 25.1±5.6% (db).

The study was based on the monitoring of 2 composting piles that were constructed by the generated biowastes during a period of 4 days (pile A1) and 3 days (A2). The piles were constructed approximately 24 hours after the entrance of the biowastes into the composting facility and after the removal of non-biodegradable materials that normally takes place in the plant. During the formation of the piles, the material was manually homogenized and sieved through a 10 cm square mesh.

The 2 piles had, each, a weight of around 1400 kg, a conical shape and an average height of 1.1 m and were placed on an impermeable base and covered by a roof. A minimum distance of 3 m was kept between the piles. The piles were turned manually with shovels.

In order to monitor the temperature profile of the piles, the tip of a 70 cm thermometer was placed in the center of the piles to take daily temperature readings. The moisture content was attempted to be maintained always above 40% (wb) (Agnew and Leonard, 2003), until the initiation of the maturation phase (considered when the temperature rapidly decreased to around 30±5°C after approximately 60 days), by spraying tap water with a hose. On site moisture measurements were performed using a moisture analyzer (Ohaus MB-35). The turning of the piles was done to avoid the compaction of the material and when temperatures higher or equal to 65°C were recorded. The monitoring of the process was done until the temperature of the piles approached ambient temperature (within a range of ± 5°C).

2.2 Stability and maturity tests

In this study, we used eleven (11) tests to determine the stability and maturity of the material. These tests were applied after the initiation of the cooling phase (maturation) until the end of the monitoring of the piles. Table 1 presents the stability and maturity tests that were applied in the laboratory as well as on-site. In addition, Table 1 includes the approximate capital cost of the necessary equipment used to perform these analyses. This is done in order to illustrate the cost differences between on-site (considered relatively cheap) and laboratory measurements (considered relatively expensive).When several alternatives are possible, the cost of each has been estimated.

For each measurement mentioned in Table 1, we followed the sampling protocols proposed by Sullivan and Miller (2001) which is based on the random collection of materials. Moisture was determined gravimetrically at 105ºC and the total organic carbon (TOC) was indirectly measured by the Walkley-Black method (Schulte, 1988; Sullivan and Miller, 2001; Icontec, 2003). pH and EC were measured on aquatic solutions prepared at a 5:1 (v/w) ratio utilizing, respectively, a pH meter (WTW™ Model 315i) and a conductivity meter (WTW™ model 325). Volatile solids were measured with the loss on ignition at 550ºC for 4 hours. All above parameters were determined following the protocols established in the Colombian

Technical Norm (NTC) 5167 (Icontec, 2003). Total N was measured according to the Kjeldahl method according to NTC 370 (Icontec, 1997). The germination tests were performed using radish seeds according to INN (2004), Varnero et al. (2007) and Komilis and Tziouvaras (2009). It is noted that radish seeds are considered an ideal choice for germination assays; actually, Komilis and Tziouvaras (2009) had observed that only the GI calculated by this type of seeds had a statistically significant negative correlation with certain stability indices. Extracts of 1:5 (g of wet material : mL distilled water) were prepared and after a 3 hour contact time, the liquid phase was filtered and 10 ml of the filtrate were removed and placed in each Petri dish using 10 radish seeds placed on filter paper. The control contained only the seeds with distilled water. The Petri dishes were incubated at the laboratory at 25°C. Duplicate measurements were performed. The germination index (GI) was calculated according to Varnero et al. (2007) by accounting for the number of seeds germinated and the total length of the seedlings in both the samples and the control.

The self-heating potential (SFH) was developed according to Brinton et al. (1995) by fixing the moisture of the material at around 35% (wb) using the fist test and by placing it in a thermally insulated vessel (Dewar flask). The recording of the temperature was done with 2 stainless steel stem (compost) thermometers (bimetallic 2” dial X 1.5” stem) during an 8 day period using measurements performed every 8 hours. During the SFH measurements, the sample was incubated in a covered vessel in laboratory temperature at 25ºC ± 2°C.

The respirometric index was calculated via measurements of CO_2 generation by the samples. We followed a static method suggested to measure the microbial activity of soils, as described by Álvarez-Solis and Anzueto-Martinez (2004) and Tognetti (2007). The sample (50 g wet weight) was incubated for 6 days at ambient temperature in an airtight vessel that included a CO_2 absorbent (10 mL of a 1N NaOH solution). At the end of the incubation, we added 2 mL of a 10% barium chloride solution, which precipitated the CO_2 as $BaCO_3$. Then, a phenolphthalein indicator was added in all samples, which were titrated with a 0.5 N HCl solution until the color of the index changed, and the volume of the acid consumed was recorded. A blank measurement was always performed at the same time with the actual samples. Equation 1 presents the calculation performed to estimate the RI, which was expressed in units of mg CO_2-C / (g OC day).

$$RI = \frac{(B - T) \cdot N_{HCl} \cdot 0.006 \cdot 1000}{DW \cdot OC \cdot d} \qquad (1)$$

where:

B: volume of acid consumed by the blank sample (mL)

T: volume of acid consumed by the sample (mL)

N_{HCl}: normality of HCl (0.5 N)

0.006: dilution factor

1000: factor to convert μg to mg

DW: dry weight of sample (mg)

OC: percentage of total organic carbon of the sample (expressed as a fraction on a dry weight basis, db)

d: days of incubation (6 d)

To determine odor, samples were placed in sealed vessels for 5 days. After that period, the vessels were opened and the odor was compared to that from vessels that contained soil. The odor test was performed throughout the study consistently by two of the authors and the determinations were always conducted at the same time by these two authors. According to Iglesia-Jimenez et al. (2008), organic materials with a high grade of maturity lack the characteristic odor of organic acids and should have an odor similar to wet soil. With regard to color, we made comparisons with the colors of stable and mature composts that had been obtained from previous composting experiments and had been carried out by a laboratory certified by the Colombian authority that deals with these issues (IDEAM, *Instituto de Hidrología, Meteorología y Estudios Ambientales de Colombia*, Colombia). The stable and mature composts that had been used for comparison pass the stability standards established by the Colombian law. The categorization of the color tones was: light brown, dark brown and almost black, which were respectively considered to indicate unstable, relatively stable and stable composts.

Measurements of VS, EC, pH, TOC, N_T and GI were performed in duplicates. If the coefficient of variation between duplicates was higher than 10%, the tests were repeated. The measurements of stability and maturity were compared to the suggested values present in the technical literature.

In order to identify if there were significant differences among the stability and maturity test results (i.e. temperature, humidity, VS, self-heating potential, pH, EC and GI) between the two piles, we applied regression models to the results of those tests that were performed 8 or 9 times during the test period for samples of each pile, respectively. The fitted regression model is presented in Equation (2) (Berthouex and Brown, 2002). Regression analysis was performed at a significance level of 5%.

$$Y_t = \beta_0 + \beta_1 \cdot t + \beta_2 \cdot Pile_t + \varepsilon_t \qquad (2)$$

Y_t: parameter that is analyzed.

t: sampling time (d) in which the parameter was measured.

$Pile_t$: binary variable that takes the value of 0, if the parameter was measured in pile A1, or 1 if the parameter was measured in pile A2, at time t.

β_0: model intercept; this parameter takes the value of Y_t in the cases that variables t and $Pile_t$ are zero.

β_1: slope associated with time; it changes Y_t for every unit of t

β_2: slope associated with the pile; it changes Y_t as long as the measurement took place in pile B

ε_t: random error.

Depending on the statistical significance of parameters β_1 and β_2, it can be concluded whether sampling time or the pile affects the parameter under analysis. For example, a non-statistically significant β_2 would indicate that the parameter under study is statistically similar in both piles (i.e. therefore, there is a good replication, since the parameter “pile” does not affect the result).

3. Results and Discussion

3.1 Temperature profiling

Figure 1 presents the temperature profile of the two piles during the whole composting process. Figure 1 also illustrates the times that turning of the piles was performed (blue and red dots) as well as the sampling times (dotted vertical lines).

According to Figure 1, the temperature profiles obtained in this study present the oxidative-thermophilic phase, the mesophilic phase and the cooling phase, as maturity of the material approached (Chiumenti et al., 2005). There was a rapid establishment of the thermophilic phase and the peak temperature (70°C) was reached at around 15-20 days from the initiation of the process. This is attributed to the presence of easily degradable matter and agrees with the temperature profile of composting experiments performed with local biowastes in the past (Oviedo et al., 2014). A rapid decline of the temperature took place after day 20, whilst small increases of the temperature were observed after each turning event. These temporary increases might be also related to the humidification of the piles and simply indicate that some material within the pile was still undecomposed even after the active thermophilic phase.

3.2 Stability and maturity indices

Table 2 presents the stability and maturity test results from both piles. The regressions models (see Eq. 2) calculated no significant differences between the 2 piles with regard to temperature ($p=0.70$), moisture ($=0.41$), self-heating ($p=0.27$), pH ($p=0.70$) and GI ($p=0.83$). On the other hand, there were statistical differences ($p<0.05$) between the 2 piles for the VS ($p=0.03$) and EC ($p<0.01$). In general, the behaviour of both piles can be considered as similar.

From Table 2, it can be observed that during the first 6 measurements (i.e. till day 141), the temperature was typical of the cooling-mesophilic phase (i.e. between 30ºC to 50ºC), whilst during the last 3 measurements the temperature was typical of a material in the phase of maturation. Actually, according to Dulac (2001) and Raj and Antil (2011), a criterion to consider a composting process finalized is that the temperature in the center of a pile should lie within a range of ±5ºC from the ambient temperature. In our case, some final pile temperatures were higher than the range suggested by the above authors. This indicates that, despite the temperature decline, some wastes can act as self-insulators and thus can preserve the heat emitted by the process (even at the phase of maturation).

The moisture steadily declined during the period of analysis due to the temperature increase and the turning of the material. Despite the generation of water by the biochemical process, there is a net water loss during open composting. In general, at the end of process, a moisture content less than 35% was maintained, which is a threshold value suggested by Icontec (2003) as well as of Saha et al. (2010) to designate stability. Thus, values below that limit would indicate a stable material. However, lower moisture contents during the active phase can severely limit the biological activity leading to a poor stabilization of the final product. The increase of the moisture around day 128 coincided with the wetting of the piles, resulting in a simultaneous small temperature peak and a temporary increase of the CO_2 production and of the self-heating potential. The last three factors (temperature, CO_2

production and self-heating potential) are indications of increased biological activity due to the turning of the piles and the wetting, as earlier discussed.

The substrates at the start of the process had a VS content around 75% (db) in both piles, whilst on the first measurement on day 70, the VS contents of piles A1 and A2, respectively, were 35% and 55% (db). Thus, a significant organic matter decomposition transformation occurred during the first stages of the process, despite the clear statistical differences between the two piles. According to Raj and Antil (2011), a material can be considered stable as long as a higher than 42% VS reduction takes place (VS reduction was calculated by Raj and Antil (2011) as the difference between the initial and final VS contents divided over the initial VS content). In our case, using the same approach, the VS reductions were above 49%, for pile A1, after day 100, and above 52%, for pile A2, after day 141, when the VS content was approximately the same for both piles: 37.1% in pile A1 and 36.0% in pile A2, respectively, Table 2). The differences in VS reduction observed between the two piles are normal in biological experiments and are attributed to differences in the advance of the organic matter transformation process. As can be seen, with regard to that parameter, and by accounting for the aforementioned suggested threshold value of Raj and Antil (2011), the materials in both piles can be considered stable throughout the whole monitoring period.

As the organic matter decomposes, the microbial respiration activity reduces as this is indicated by the CO_2 generation rates (Said-Pullicino et al., 2007; Defrieri et al., 2005). Some classification stability limits (Trautman and Krasny, 1997; CCQC, 2001) with regard to CO_2 evolution have been based on measurements performed at the mesophilic range (i.e. 34ºC and 37ºC). As there are no such limits for CO_2 evolution at ambient temperature, a caution is needed when comparing our microbial activity measurements (performed at ambient temperature) with those limits. For example, Trautman and Krasny (1997) proposed the following limits (in mg CO_2-C/g OC-d): <2 for very stable, 2 – 5 for stable, 5 – 10 for moderately stable, 10 – 20 for unstable and >20 for extremely unstable. In CCQC (2001), the suggested limits (in mg CO_2-C/g OM-d) are: <2 for very stable, 2 – 8 stable and > 8 less stable. Based on the classification of Trautman and Krasny (1997), and although the microbial respiration activity values are expected to be lower at ambient temperature compared to mesophilic temperatures, it appears that the material in the piles can be safely designated as either stable or very stable. The same conclusion is reached when all values are expressed on a per OM basis and compared to the limits of CCQC (2001), as indicated in Table 2. The lowest RI values, indications of very stable material, were obtained during the last measurements in both piles.

Since the start of the monitoring on day 70, the self-heating ΔT values were equal to or less than 5ºC. According to Brinton et al. (1995), ΔT values between 0ºC and 10ºC classify a material under stability grade V, which is a criterion for very stable final composts. The fluctuations of ΔT observed during the sampling period might be related to temporary reactivations of the biological activity due to the turning or wetting of the piles. As a result, with regard to that index, the material can be consistently considered as very stable.

According to Mohedo (2004), the color of a material during the composting process gets darker as the process evolves until it attains a dark brown or almost black color due to the formation of chromophor groups or due to the synthesis of melanoidines. The visual inspection of the material, in comparison with mature and stable compost from previous experiments, permitted us to identify the different color tones and to classify the material as consistently stable after day 128 in both piles. However, a black color was also recorded in earlier days in both piles (e.g. day 70 and 86 for pile A1 and day 70 for pile A2) but dark brown colors were still noted in some of the next sampling periods until the black colors became eventually permanent.

During the composting process, the odor is mostly affected by the presence of organic acids of low molecular weight the concentration of which reduces to negligible levels at the end of the process. Although gas chromatography can be used to measured organic acids and indirectly quantify odor levels, this procedure was not available in this study. Nevertheless, a characteristic odor of humid soil was continuously detected in our study beyond day 100 for pile A1 and consistently beyond day 128 for pile A2.

The C/N ratio has been used extensively in the literature as both a stability and maturity indicator during composting (Tognetti et al. 2007; Said-Pullicino et al., 2007); however, variable values of C/N ratio have been reported to determine the stability and maturity of composts. Some examples are: <17 (Moldes et al. 2007), <12 (Bernal et al., 1998), around 15 to 16 (Barral et al., 2007), between 15 to 20 (Rosen et al., 1993). Other authors support the idea that it is not possible to establish one unique C/N ratio value, since this depends on the characteristics of the material under treatment (Defrieri et al. 2005). The obtained C/N ratios (< 13) for both piles after day 141 can lead to the conclusion that at that stage the material in both piles fulfilled the criteria of stability and maturity with regard to that parameter; however, lower C/N ratios were clearly observed in pile A2 compared to those of A1. These differences agree with the statement of Brewer and Sullivan (2003) who reported that the C/N ratio itself is not an appropriate index of stability, but it is necessary to illustrate its development during the process rather than to use a unique threshold value.

Actually, the C/N ratio should be combined with other analytical tests and indices in order to characterize the quality of composts (Difrieri et al., 2005).

The pH remained at the alkaline range during the monitoring period with values between 9.2 and 9.7, for pile A1 and between 9.1 and 9.8, for pile A2. The pH increase to alkaline ranges is attributed to the consumption of protons during the decomposition of volatile fatty acids, the generation of CO_2 and the mineralization of organic nitrogen (Beck-Friis et al., 2001; Smars et al., 2002; Chiumenti et al., 2005). Tognetti et al. (2007) attribute the high pH at the end of a composting process of unshredded biowastes to the long and intensive thermophilic phase that can favor the ammonification of organic nitrogen. Alkaline pH is desirable since it limits the availability of heavy metals (Tiquia, 2005); on the other hand it can induce a micronutrient deficiency (Rosen et al., 1993). With regard to that parameter, the product can be categorized as immature (pH was arbitrarily considered as a maturity parameter in our work), since its pH was outside the desirable range of 7 and 8 that can render it suitable for agricultural applications (USDA, 1999). Actually, very alkaline pHs, as in our case, can limit the development of plants and cultivations.

At the beginning of the maturation process (Table 2), the GIs were quite low, apparently due to the continuing decomposition of organics that lead to the generation of phytotoxic compounds, such as volatile fatty acids. Those phytotoxic compounds, which are commonly microbial metabolic byproducts, are degraded during the process so that the final mature material eventually does not affect the plant growth (Zucconi et al., 1981, Bernal et al., 1998). Bernal et al. (2009) suggested that a compost can be considered immature at GI values < 80%, mature between GI 80% and 90% and highly mature at GI > 90%. However, it is noted that these threshold values highly depend on the type of seed used. Notably, Komilis and Tziouvaras (2009) showed that when radish seeds are used, a fair correlation between GI and CO_2 generation (expressed as RI) does exist. In this study, although there were many fluctuations in the GI values, an increasing trend seemed to exist with time as a general trend. By accounting for the GI limit (80%) proposed by Bernal et al. (2009), the material can be considered constantly immature from the start of the measurements to the end of the process.

The soluble salt content of the material can be estimated indirectly through measurements of electrical conductivity (EC) of a sample which is over-saturated with water. The elements that mostly contribute in the salinity are Na, K, Cl^-, NH_4^+, NO_3^- and SO_4^{2-}. EC values lower than 3.5 dS/m indicate a small quantity of available salts. According to the pertinent Chilean law (NCh 2880) a compost of high quality without restrictions in its use should have an EC lower than 3 dS/m (INN 2004). The values measured in both piles were higher than those proposed by Moldes et al. (2007) and INN (2004), since they were equal to 5.2 dS/m, for pile A1, and 3.6 dS/m, for pile A2, at the end of the monitoring period. Said-Pullicino et al. (2007) also measured high EC values (5.0 and 7.8 dS/m) during the composting of biowastes, pruning wastes and leaves; however, the compost product was not phytotoxic in germination tests. However, Manios et al. (2004) suggested that compost with high EC content could present a phytotoxic behavior affecting seed growth when used in large amounts. Although one would expect a decrease of the EC measurements during the process (Khalil et al., 2008), the EC values in this study were quite variable and a clear trend after day 70 was not evident.

3.3 Comparisons among tests of stability and maturity

Table 3 presents a grouping of the stability and maturity indices for each sampling time and each pile. The conditions of stability and maturity are indicated with a shaded color. As can be seen, after day 162 in pile A1 and after day 155 in pile A2, all stability indices conclude that the material is stabilized (except only in the case of the final moisture content of pile A2 which was marginally above the threshold value of 35% wb). The 3 maturity tests (pH, EC, GI), however, all concluded that the material was consistently immature throughout the test period (except in one case in which the EC was marginally below the threshold value). C/N ratio indicates that both composts were stable, but recent studies have demonstrated that this parameter should be evaluated according to biodegradable carbon instead of total organic carbon (Puyuelo et al., 2011). Therefore, it appears that there is no agreement between the maturity and stability test results. In addition, no agreement within the different stability test results was always present between day 70 and 155. Actually, there seem to exist consistent results within 2 groups of tests, the first group being the self-heating, RI, VS and odor and the 2nd being the group of three tests developed on-site (temperature, moisture, color). This verifies that there are some inconsistencies between the results of the tests performed in the laboratory with the ones performed on-site, as referred in other biowaste composting studies (Ruggieri et al., 2008).

Although at the end of the monitoring period, both the laboratory tests and the on-site tests indicated stability, this condition was indicated more rapidly (on day 86) in the laboratory tests, namely RI and the self-heating potential. This also indicates that the decisions based on only one quality test can be erroneous and it is necessary to simultaneously perform more tests to reach a decision (Komilis and Tziouvaras, 2009; Tontti et al., 2011).

Regarding the temperature, although this classic index shows the evolution of the process, it did not coincide with the findings of the other tests (RI, VS, self-heating and C/N) that indicated that the material was stable. Therefore, temperature has to be also combined with other tests to clearly designate (or not) the stability of a material. This is also true for the index of “color”, which although very easy to perform, it can be imprecise and subjective. For each type of tests, it is recommended to compare with other physicochemical and biological tests, as discussed in Raj and Antil (2011).

The results of the maturity tests, with an exception of the C/N ratio, are consistent among each other. Although both piles reached stability at the end of the monitoring period, the material was continuously still phytotoxic (low GI), with a high salt contents (high EC) as well as with alkaline pH values till the end of the monitoring period. This inconsistency between stability and maturity tests has been also shown in Komilis and Tziouvaras (2009). On the other hand, it is noted that the GI values could have been completely different if other types of seed had been used, as had been shown by Komilis and Tziouvaras (2009).

3.4 Correlation of quantitative indices

Figure 2 presents six correlation plots among some of the quantitative indices measured in this work. As can be seen, no correlation seems to exist among any of those indices. This is verified after calculating the coefficients of determination (R^2) of the resulting linear regression equations (values not shown), which were all non-statistically significant at $p<0.05$. This partly contradicts the findings of Komilis and Tziouvaras (2009), who had found a negative correlation between the GI of 3 types of seeds (cucumber, radish, spinach) and EC as well as a marginal statistically significant negative correlation between CO_2 generation and the GI (measured with radish seeds). On the other hand, the absence of a correlation between VS content and the radish seed based GI does agree with the findings of Komilis and Tziouvaras (2009).

4. Conclusions

The conclusions of this work are:

1. The tests traditionally performed on-site to monitor the evolution of the process, such as temperature, moisture and color, are not consistent with the laboratory tests in order to assess organic matter stability.
2. In the case of maturity, both the on-site (pH, EC) and the lab (GI) tests indicated that the material was consistently immature, which did not agree with the findings of the laboratory stability tests, in which a stability conditions was established in most of the sampling periods.
3. Stability does not necessarily indicate maturity.
4. No statistically significant correlations were found among any of the quantitative stability and maturity indices measured in this work, as indicated in Figure 2.

Based on the above conclusions, it is recommended to use a combination of lab and on-site tests in order to reach a decision regarding the quality of a product. To do that, it is ideally recommended to develop and adjust existing laboratory tests so that they can be used on-site.

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References

Agnew, J.M., Leonard, J.J., 2003. The physical properties of compost. Compost Science & Utilization, 11, 3, 238-264.

Álvarez-Solís, J.D., Anzueto-Martínez, D.J., 2004. Actividad microbiana del suelo bajo diferentes sistemas de producción de maíz en los altos de Chiapas, México. Agrociencia, 38,1, 13-22.

Astrup, T., Dimambro, M., Wallace, P., Howell, G., Frederickson, J., 2015. Static, dynamic and inoculum augmented respiration based test assessment for determining in-vessel compost stability. Waste Management, 42, 3-9.

Barral, M.T., Moldes, A., Cendón, Y., Diaz-Fierros, F., 2007. Assessment of municipal solid waste compost quality using standardized methods before preparation of plant growth media. Waste Management & Research, 25,2, 99-108.

Barral, M.T., Paradelo, R., 2011. Trace elements in compost regulation: The case of Spain. Waste Management, 31, 3, 407-410.

Barreira, L., Philippi, A., Rodrigues, M., 2006. Usinas de compostagem do estado de Sao Paulo qualidade dos compostos e processos de producao. Engenharia Sanitaria e Ambiental, 11, 4, 385-393.

Barrena, R., Vazquez, F., Sánchez, A., 2006. The use of respiration indices in the composting process: a review. Waste Management & Research, 24, 1, 37-47.

Beck-Friis, B., Smars, S., Jönsson, H., Kirchmann, H., 2001. Gaseous Emissions of Carbon Dioxide, Ammonia and Nitrous Oxide from Organic Household Waste in a Compost Reactor under Different Temperature Regimes. Journal of Agricultural Engineering Research, 78, 4, 423-430.

Bernal, M.P., Paredes, C., Sanchez-Monedero, M.A., Cegarra, J., 1998. Maturity and stability parameters of composts prepared with a wide range of organic wastes. Bioresource Technology, 63, 1, 91-99.

Bernal, M.P., Alburquerque, J.A., Moral, R., 2009. Composting of animal manures and chemical criteria for compost maturity assessment. A review. Bioresource Technology, 100, 22, 5444-5453.

Berthouex, P.M., Brown, L., 2002. Statistics for Environmental Engineers, second ed. Lewis Publishers, New York.

Brewer, L.J., Sullivan, D.M., 2003. Maturity and stability evaluation of composted yard trimmings. Compost Science & Utilization, 11, 2, 96-112.

Brinton, W.F., Evans, E., Droffner, M.,L., Brinton, R.B., 1995. A standardized Dewar test for evaluation of compost self-heating. Biocycle, 36, 1-16.

California Compost Quality Index (CCQC), 2001. Compost Maturity Index. http://www.calrecycle.ca.gov/organics/products/quality/compmaturity.pdf (accessed Oct. 2014).

Cesaro, A., Belgiorno, V., Guida, M., 2015. Compost from organic solid waste: Quality assessment and European regulations for its sustainable use. Resources, Conservation and Recycling, 94, 72-79.

Chiumenti, A., Chiumenti, R., Diaz, L., Savage, G., Eggerth, L.Y, Goldstein, N., 2005. Modern Composting Technologies. BioCycle. Journal of Composting and Organics Recycling. Singapore.

Defrieri, R.L., Jimenez, M.D.L.P., Effron, D., Palma, M., 2005. Utilización de parámetros químicos y microbiológicos como criterios de madurez durante el proceso de compostaje. Agriscientia, 22,1, 25-31.

Dulac, N., 2001. The Organic Waste flow in Integrated Sustainable Waste Management – The Concept. Waste Tools for Decision-makers: Experiences from the Urban Waste Expertise Programme (1995-2001). Netherlands, 49.

Hoornweg, D., Thomas, L., Otten, L., 1999. Composting and Its Application in Developing Countries. Published for Urban Development Division, The World Bank, Washington DC.

Icontec, 2003. Norma Técnica Colombiana. Productos para la industria agrícola. Productos orgánicos usados como abonos o fertilizantes y enmiendas de suelo. Instituto Colombiano de Normas Técnicas y Certificación. Bogotá.

Icontec, 1997. Norma Técnica Colombiana NTC 370. Abonos o Fertilizantes. Determinación de Nitrógeno Total. Instituto Colombiano de Normas Técnicas y Certificación. Bogotá.

Iglesias-Jiménez, E., Barral-Silva, M.T., Marhuenda-Egea, F.C., 2008. Indicadores de la estabilidad y madurez del compost. Moreno Casco, J., Moral Herrero, R.(Ed.), compostaje. Ediciones Mundi-Prensa, Madrid, p. 243-283.

INN – Instituto Nacional de Normalización (2004). Norma chilena de compost 2880- 2004 (NCh 2880-2004), Compost - Clasificación y requisitos, 23. Santiago de Chile, 27.

Khalil, A., Domeizel, M., Prudent, P., 2008. Monitoring of green waste composting process based on redox potential. Bioresource Technology, 99, 14, 6037-6045.

Komilis, D., Tziouvaras, I., 2009. A statistical analysis to assess the stability and maturity of six composts. Waste Management, 29, 1504-1513.

Komilis, D., Kontou, I., Ntougias, S., 2011. A modified static respiration assay and its relationship with an enzymatic test to assess compost stability and maturity. Bioresource Technology, 102, 10, 5863-5872.

Kurian, J., 2007. Experiencias de tratamiento de residuos sólidos urbanos en India. Residuos: La Revista Técnica del Medio Ambiente 97 (Mayo – Junio), 24 – 30.

Levis, J., Barlaz, M., Themelis, N., Ulloa, P., 2010. Assessment of the state of food waste treatment in the United States and Canada. Waste Management, 30, 1486–1494.

Manios, T., 2004. The composting potential of different organic solid wastes:

experience from the island of Crete. Environment International, 29, 1079–1108.

Marmolejo L., 2011. Marco conceptual para la sostenibilidad de los sistemas de aprovechamiento de residuos sólidos en cabeceras municipales menores a 20.000 habitantes del Valle del Cauca. [Tesis Doctoral]. Doctorado en Ingeniería, énfasis Ingeniería Sanitaria y Ambiental. Cali (Colombia): Universidad del Valle. Facultad de Ingeniería.

Mohedo, J. J., 2004. Estudio de la estabilidad durante el compostaje de residuos municipales. [Tesis Doctoral]. Doctorado en Ingeniería Química. Universidad de Córdoba, Cordoba (Argentina). Facultad de Ciencias.

Moldes, A., Cendon, Y., Barral, M.T., 2007. Evaluation of municipal solid waste compost as a plant growing media component, by applying mixture design. Bioresource Technology, 98, 16, 3069-3075.

OPS - Organización Panamericana de la Salud, 2005. Informe de la Evaluación Regional de los Servicios de Manejo de Residuos Sólidos en América Latina y el Caribe. Washington. D.C.

Oviedo, E.R., Marmolejo, L.F., Torres, P., 2014. Influencia de la frecuencia de volteo para el control de la humedad de los sustratos en el compostaje de biorresiduos de origen municipal. Revista Internacional de Contaminación Ambiental, 30, 1, 91-100.

Puyuelo, B., Ponsá, S., Gea, T., Sánchez, A., 2011. Determining C/N ratios for typical organic wastes using biodegradable fractions. Chemosphere. 85, 4, 653-659.

Raj, D., Antil, R.S., 2011. Evaluation of maturity and stability parameters of composts prepared from agro-industrial wastes. Bioresource Technology, 102, 3, 2868-2873.

Riffaldi, R., Levi-Minzi, R., Pera, A., De Bertoldi, M., 1986. Evaluation of compost maturity by means of chemical and microbial analyses. Waste Management & Research, 4, 4, 387-396.

Rosen, C.J., Halbach, T.R., Swanson, B.T., 1993. Horticultural uses of municipal solid waste composts. HortTechnology, 3, 2, 167-173.

Ruggieri, L., Gea, T., Mompeó, M., Sayara, T., Sánchez, A., 2008. Performance of different systems for the composting of the source-selected organic fraction of municipal solid waste. Biosystems Engineering, 101, 1, 78-86.

Said-Pullicino, D., Erriquens, F. G., Gigliotti, G., 2007. Changes in the chemical characteristics of water-extractable organic matter during composting and their influence on compost stability and maturity. Bioresource Technology, 98, 9, 1822-1831.

Saha, J.K., Panwar, N., Singh, M.V., 2010. An assessment of municipal solid waste compost quality produced in different cities of India in the perspective of developing quality control indices. Waste Management, 30, 2, 192–201.

Schulte, E.E., 1988. Recommended soil organic matter tests. In: Dahnke, W.C. (Ed.), Recommended chemical soil test procedures for the north central region. North Dakota State University, Fargo, pp. 29-31.

Shekdar, A.V., 2009. Sustainable solid waste management: An integrated approach for Asian countries. Waste Management 29, 4, 1438–1448.

Smårs, S., Gustafsson, L., Beck-Friis, B., Jönsson, H., 2002. Improvement of the composting time for household waste during an initial low pH phase by mesophilic temperature control. Bioresource Technology, 84, 3, 237-241.

Soumaré, M., Tack, F.M.G., Verloo, M.G., 2003. Characterisation of Malian and Belgian solid waste composts with respect to fertility and suitability for land application. Waste Management, 23, 6, 517-522.

Sullivan, D.M., Miller, R.O., 2001. Compost quality attributes, measurements, and variability. In: Stoffella, P.J., Kahn, B.A. (Eds.), Compost Utilization in Horticultural Cropping Systems. Lewis Publishers, Boca Raton, Florida, pp. 95–120.

Tang, J.C., Inoue, Y., Yasuta, T., Yoshida, S., Katayama, A., 2003. Chemical and microbial properties of various compost products. Soil Science and Plant Nutrition, 49, 2, 273-280.

Tiquia, S., 2005. Microbiological parameters as indicators of compost maturity. Journal of Applied Microbiology, 99, 4, 816 – 828.

Tognetti, C., 2007. Compostaje de residuos orgánicos urbanos: optimización del proceso para una mayor calidad del producto final. Tesis Doctoral. Universidad Nacional Comahue, San Carlos de Bariloche, Argentina, 175 pp.

Tognetti, C., Mazzarino, M. J., Laos, F., 2007. Improving the quality of municipal organic waste compost. Bioresource Technology, 98, 5, 1067-1076.

Tontti, T., Heinonen-Tanski, H., Karinen, P., Reinikainen, O., Halinen, A., 2011. Maturity and hygiene quality of composts and hygiene indicators in agricultural soil fertilised with municipal waste or manure compost. Waste Management & Research, 29, 2, 197-207.

Trautman, N., Krasny, M., 1997. Composting in the classroom: Scientific inquiry for high school students. http://cwmi.css.cornell.edu/compostingintheclassroom.pdf. (accessed Oct. 2014).

Turan, N.G., Çoruh, S., Akdemir, A., Ergun, O.N., 2009. Municipal solid waste management strategies in Turkey. Waste Management, 29, 1, 465-469.

USDA – United State Departament of Agriculture, 1999. Guía para evaluación y calidad y salud del suelo. USDA, Washington.

Varnero, M.M.T., Rojas, A,C., Orellana, R.R., 2007. Índices de fitotoxicidad en residuos orgánicos durante el compostaje. Revista de la Ciencia del Suelo y Nutrición Vegetal, 7 (1), 28-37.

Wu, L., Ma, L.Q., Martinez, G.A., 2000. Comparison of methods for evaluating stability and maturity of biosolids compost. Journal Enviromental Quality, 29, 2, 424-429.

Zucconi, F., Pera, A., Forte, M., de Bertoldi, M., 1981. Evaluating toxicity of immature compost. Biocycle, 22, 54–57.

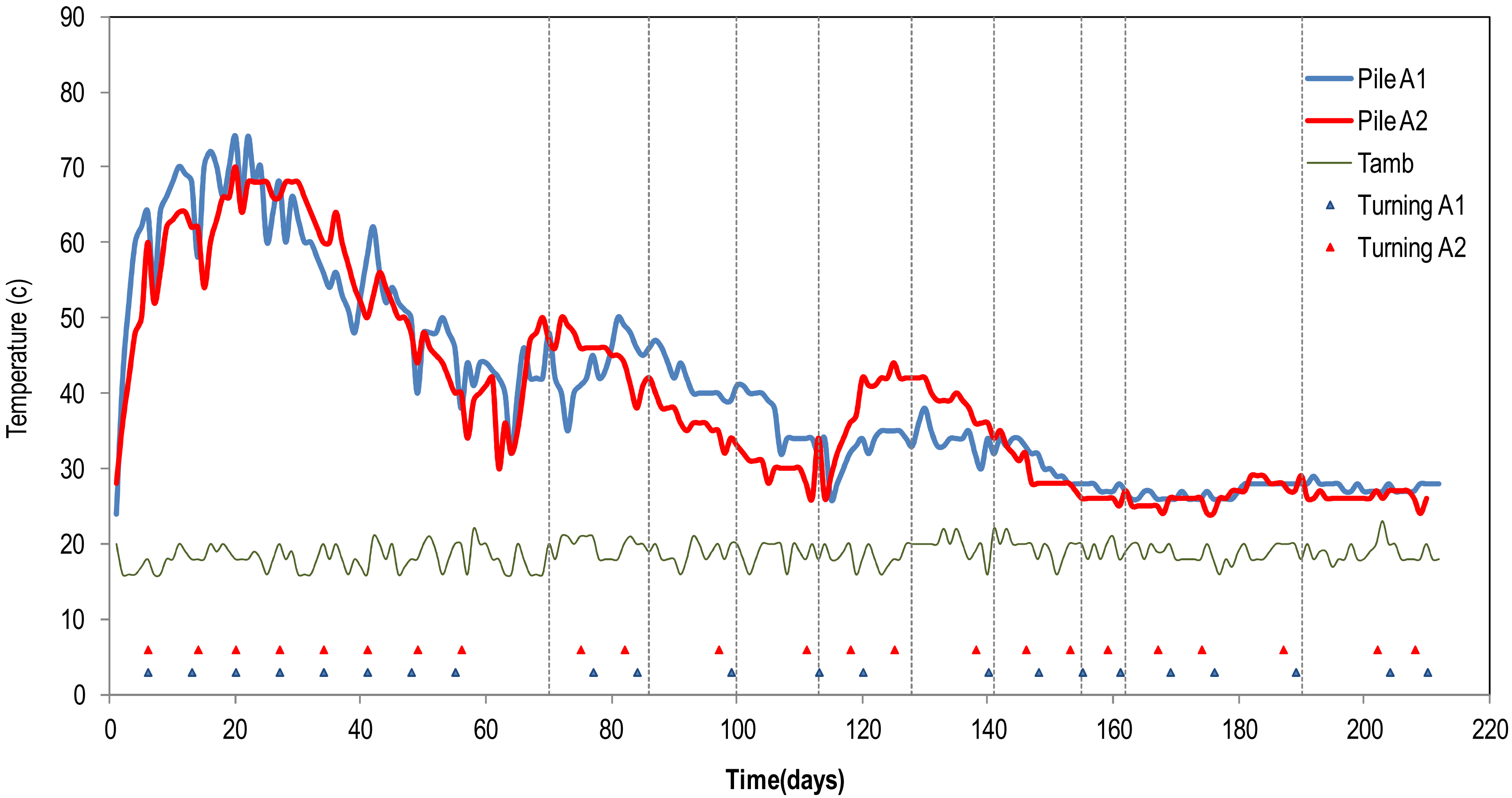

Figure 1. Temperature profile in the centre of both piles and ambient temperature (Tamb); dotted lines indicate sampling times and triangles indicate the turning events of the piles

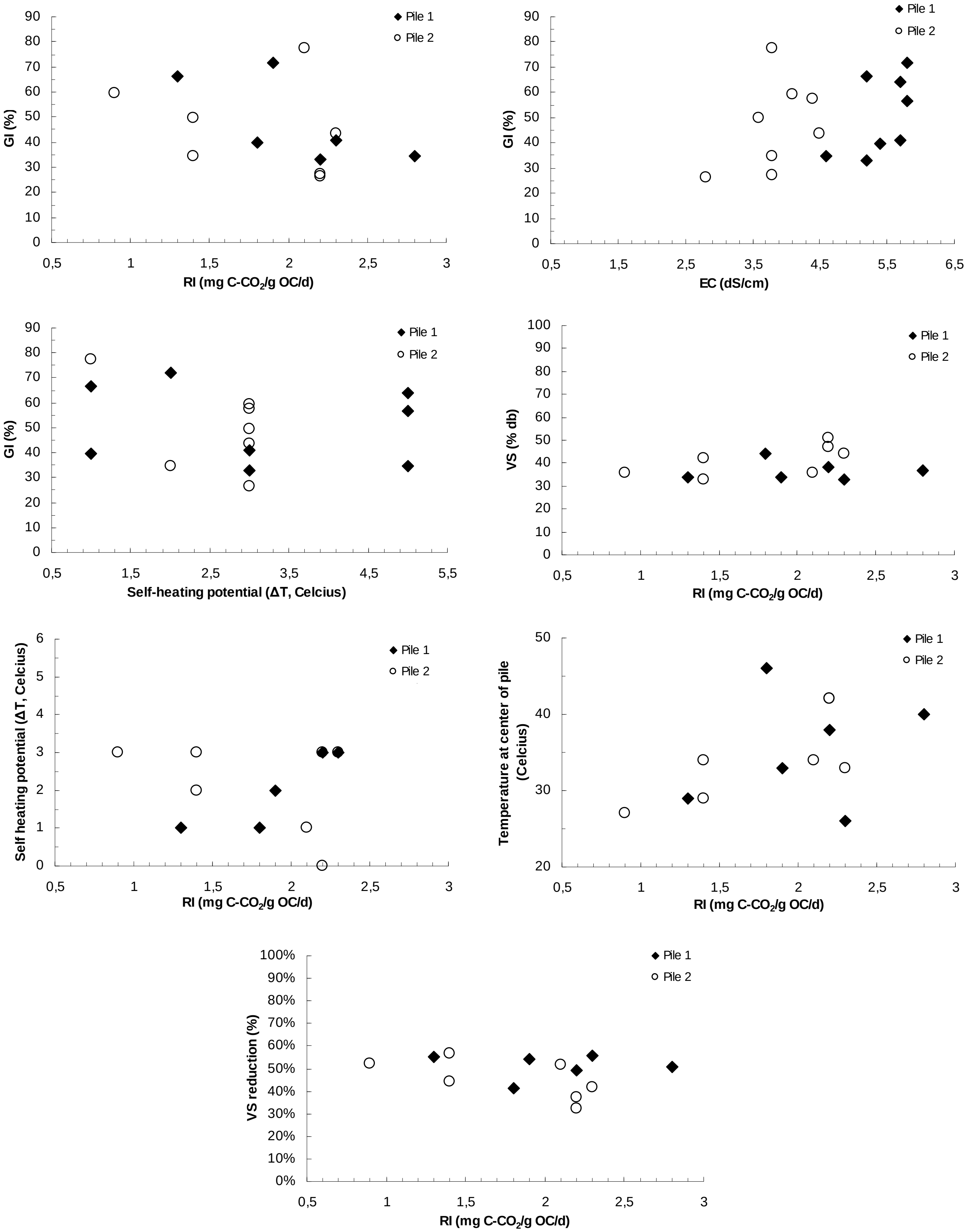

Figure 2. Correlations among several of the indices measured in this work

Table 1. Laboratory and on-site stability and maturity tests and estimates of purchase cost of necessary equipment

Site	Stability	Cost of stability test equipment (€)	Maturity	Cost of maturity test equipment (€)	Days of sampling from the start of the composting process
On-site	Temperature	< 500	pH	<500	
	Color	0	EC	<500	
	Odor	0[1]			
Laboratory	Moisture	< 1000	GI	<200	70, 86, 100, 113, 128, 141, 155, 162, 190
	Volatile solids (VS)	< 5,000			
	C/N ratio	< 80,000[2]			
	Respirometric index (RI) measured via the CO_2 generation	< 2,000[3]			
	Self-heating potential (SFH)	< 2,000			

Note: Self-heating and GI measurements were not performed on day 70; RI was not measured on days 70, 113 and 155; C/N ratio was not measured on days 70, 86, 100, 113 and 128.

[1]: Assuming that no olfactometry equipment is used but simply odor sensing by a panel of people.

[2]: Via the use of an elemental analyzer (10000 € for the purchase of a muffle furnace, to assess organic matter and then convert to total carbon based on a regression coefficient, and for a typical Kjeldalh apparatus).

[3]: If custom made Erlenmeyer flasks are used (up to 30,000 € if automated respirometers that record both O_2 consumption and CO_2 generation are used).

Table 2. Results of the laboratory and on-site tests to evaluate stability and maturity

Pile	Time (days)	Stability								Maturity		
		On-site				Laboratory				On-site		Laboratory
		Temperature (°C)	Moisture (% wb)	Color	Odor	VS (% db)	RI[1] (mg CO_2/g OC/d): (mg CO_2/g OM/d)	Self-heating (ΔT - °C)	C/N[2]	pH	EC (dS/m)	GI (%)
A1	70	40	51.0	Black	Putrid	34.8±0.6	-	-	-	9.2	4.1	-
	86	46	48.0	Black	Moist	44.0±3.1	1.8 : 0.66	1	-	9.5	5.4	39.7±1.5
	100	40	46.0	Dark brown	Earthy	37.1±2.1	2.7 : 0.64	5	-	9.4	4.6	34.7±4.9
	113	26	36.0	Dark brown	Earthy	38.5±3.2	-	5	-	9.6	5.7	64.0±7.4
	128	38	44.0	Black	Earthy	38.0±3.5	2.2 : 0.43	3	-	9.4	5.2	33.0±3.2
	141	33	35.0	Black	Earthy	34.4±0.5	1.9 : 0.36	2	10.0	9.4	5.8	71.8±18.5
	155	28	34.0	Black	Earthy	34.1±3.0	-	5	13.2	9.5	5.8	56.8±8.1
	162	26	32.0	Black	Earthy	33.2±2.7	2.3 : 0.32	3	10.6	9.5	5.7	40.9±2.7
	190	29	32.2	Black	Earthy	33.5±2.5	1.3 : 0.31	1	9.2	9.7	5.2	66.4±9.8
A2	70	47	48.0	Black	Putrid	54.9±1.2	-	-	-	9.3	3.7	-
	86	42	42.0	Dark brown	Earthy	50.7±3.0	2.2: 0.56	0	-	9.8	3.8	27.3±3.9
	100	33	36.0	Dark brown	Earthy	43.5±2.2	2.3: 0.46	3	-	9.3	4.5	43.6±5.1
	113	34	30.0	Dark brown	Moist	41.6±0.7	1.3: 0.25	2	-	9.5	3.8	34.7±4.1
	128	42	46.0	Black	Earthy	46.8±0.2	2.1: 0.42	3	-	9.1	2.8	26.5±1.5
	141	34	37.0	Black	Earthy	36.0±3.0	2.0: 0.42	1	8.2	9.6	3.8	77.5±22.4
	155	26	34.0	Black	Earthy	30.1±3.2	-	3	8.7	9.3	4.4	57.4±5.1
	162	27	31.0	Black	Earthy	35.9±0.1	1.2: 0.25	3	8.3	9.4	4.1	59.4±2.9
	190	29	36.5	Black	Earthy	32.4±3.1	1.4: 0.33	3	7.7	9.6	3.6	49.7±4.2

[1]: Measured at ambient temperature
[2]: The C/N ratio was considered here as both a stability and a maturity index.
Values are means (± standard deviation).

Table 3. Grouping of all samples with respect to each index *

Pile	Time (d)	Stability parameters								Maturity parameters		
		On site				Laboratory				On-site		Laboratory
		Temperature	Moisture	Color	Odor	VS	RI	Self-heating potenital	C/N ratio	pH	EC	GI
A1	70	NS	NS	S	NS	S	-	-	-	IM	IM	-
	86	NS	NS	S	NS	NS	S	S	-	IM	IM	IM
	100	NS	NS	NS	S	S	S	S	-	IM	IM	IM
	113	NS	NS	NS	S	S	-	S	-	IM	IM	IM
	128	NS	NS	S	S	S	S	S	-	IM	IM	IM
	141	NS	S	S	S	S	S	S	S/M	IM	IM	IM
	155	S	S	S	S	S	-	S	NS/IM	IM	IM	IM
	162	S	S	S	S	S	S	S	S/M	IM	IM	IM
	190	S	S	S	S	S	S	S	S/M	IM	IM	IM
A2	70	NS	NS	S	NS	NS	-	-	-	IM	IM	-
	86	NS	NS	NS	S	NS	S	S	-	IM	IM	IM
	100	NS	NS	NS	S	S	S	S	-	IM	IM	IM
	113	NS	S	NS	NS	S	S	S	-	IM	IM	IM
	128	NS	NS	S	S	NS	S	S	-	IM	M	IM
	141	NS	NS	S	S	S	S	S	S/M	IM	IM	IM
	155	S	S	S	S	S	-	S	S/M	IM	IM	IM
	162	S	S	S	S	S	S	S	S/M	IM	IM	IM
	190	S	NS	S	S	S	S	S	S/M	IM	IM	IM

Note: S: Stable, NS: Non-stable, M: Mature: IM: Immature. Conditions of stability (S) and maturity (M) are indicated in gray.

*It is considered that stability is indicated as long as: i) pile temperatures are within ±5ºC from ambient temperature, ii) moisture contents are below 35% (wb), iii) the color of the product is black, iv) there is an earthy smell, v) the VS reduction is below 42%, vi) the RI is below 5 mg C-CO_2 / g OC–d or below 8 mg C-CO_2 / g OM–d at mesophilic temperatures, vii) the self- heating potential is below 10ºC, viii) the C/N ratio is below 13. In addition, it was considered that maturity is indicated as long as: i) pH is between 7 and 8, ii) EC is below 3 dS/cm, and iii) GI is above 80%.