Evolution of organic matter from sewage sludge and garden trimming during composting

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Abstract

To use compost appropriately in agriculture it is extremely important to estimate the stabilization level of the organic matter. In this work, two different piles of compost were studied by means of (i) humification parameters (degree of humification—DH, humification rate—HR, humification index—HI) prior to and after enzymatic hydrolysis of the extracted organic carbon, (ii) water-soluble organic carbon (WSOC) and (iii) water-soluble nitrogen. A significant relationship between composting time, WSOC and humification parameters after enzymatic hydrolysis (DH\textsubscript{enz}, HR\textsubscript{enz}, HI\textsubscript{enz}) was found.

Keywords: Compost; Enzymatic hydrolysis; Humification parameters; Water-soluble organic carbon; Water-soluble nitrogen

1. Introduction

The considerable amount of organic matter (OM) and the appreciable concentration of mineral nutrients in sewage sludge and garden trimming provide an excellent source of plant nutrition when applied to soil after an appropriate composting period (Terman and Mays, 1973; De Haan, 1981; Chen et al., 1996; Zach et al., 1999). The agronomical quality of compost produced from these biosolids is limited mainly by their chemical composition as well as by the stability and maturity of the OM (Iglesias-Jimenez and Perez-Garcia, 1992; Govi et al., 1993; Chefetz et al., 1996; Bernal et al., 1998; Marchiol et al., 1999). Organic components of compost undergo several important transformations during the composting process, (Tiquia and Tam, 2000; Sánchez-Monedero et al., 2001) producing metabolites which exhibit inhibiting or stimulating effects on plant growth (Wong et al., 2001). The OM from sewage sludge and garden plant trimming must be well stabilized before its addition to soil, otherwise it could have a toxic effect on plants (Atiyeh et al., 2001). Many different methods have been proposed to substantiate OM quality during the composting process. Nicolardot et al. (1982) proposed monitoring the respiration intensity during composting, while Nappi (1989) attempted to correlate the degree of stabilization with nitrogen mineralization. A number of bioassay methods on cress (Lepidium sativum L.) seedling growth (Zucconi and de Bertoldi, 1987), cress seed germination and physiological parameters of sunflower (Helianthus annuus L.) plants (Baca et al., 1990) have been proposed to evaluate compost maturity; however none of these methods give an absolute parameter. Compost is a very heterogeneous biomass and the different chemical methods exploited to determine maturity level are only suitable for certain families of materials. Genevini et al. (1992) reported a poor correlation between the degree of humification (DH), compost protein and hemicellulose rich materials due to the formation of humic-like molecules (Ciavatta et al., 1990a). The relationship between composting time and maturation level can be increased by treating an organic extract with a sequence of unspecific hydrolytic enzymes (Ciavatta et al., 1990a). This enzymatic treatment, together with the amount of water-soluble organic carbon (WSOC) and water-soluble nitrogen (WSN), provide a more thorough understanding of the composting process and compost stability.

The aim of this work was to follow the evolution of the OM from sewage sludge and yard trimming during the composting of two piles of compost, using humification parameters, enzymatic hydrolysis, WSOC and WSN.
2. Methods

2.1. Sampling and preparation of samples

Two different compost samples ("A" and "B") were taken from a composting plant over a period of 160 days of composting in a static pile. The blend of raw materials in trial "A" at the beginning of the composting process was made up of 70% (v/v) plant trimming (mowed and pruned) and 30% (v/v) sewage sludge of either urban or agro-industrial origin. The raw materials in trial "B" were made up of 60% (v/v) plant trimming and 40% (v/v) urban and agro-industrial sewage sludge mixture. Samples were obtained following the procedure suggested by I.P.L.A. (1998), dried in an air-forced oven at 40°C until they reached a constant weight and then sieved to 8 mm. The fraction <8 mm (ϕ) was crushed using a Tecator Cyclotec, 1093 (PBI), until all the material passed through a 0.25 mm sieve.

2.2. Determination of ash content, pH and electrical conductivity

The ash content was determined as follows: 5 g of dry sample was placed in an oven at 650°C until a constant weight was achieved (about 6 h) (I.P.L.A., 1998).

A suspension of 5 g of sample and 50 ml of distilled water was stirred for 30 min at 25°C and, after filtration, the pH was measured using a Crison pH-meter (Micro TT 2022, Spain).

The electrical conductivity of the same solution was then measured using specific equipment (Halosis SAT type, Italy).

2.3. Determination of nutrients and heavy metals

The nutrient and heavy metal content was determined in accordance with Italian and E.C. official chemical methods for fertilisers (M.A.F., 1986).

2.4. Extraction and fractionation of the organic carbon

Two grams of compost sample was prepared as reported in Section 2.1 and extracted with 100 ml of NaOH 0.1 M + Na4P2O7 0.1 M solution, at 65°C using a Dubnoff shaker bath at 110 rpm for 24 h under N2 atmosphere. The suspension was centrifuged at 5000×g for 30 min and then filtered through a 0.8 μm filter and fractionated into humic and humic-like materials and non-humified (NH) organic carbon (M.A.F., 1986; Ciavatta et al., 1990b). The total organic carbon (TOC) in the solid and liquid samples was measured using the methods suggested by Ciavatta et al. (1989, 1991), respectively.

2.5. Enzymatic hydrolysis

Enzymatic hydrolysis of the liquid samples was carried out as follows: 50 mg of extracted organic C (TEC) was placed into a 100 ml Erlenmeyer flask. The volume was brought to around 20 ml with de-ionized H2O and then the solution was acidified drop by drop to pH 8 with 85% H3PO4. Next, selected enzymes (Merck, Germany) were sequentially added to the suspension. The enzymes used were (i) 5 ml of a solution containing 150 μg ml⁻¹ of lipase (porcine, 130 FIP-units mg⁻¹), (ii) 10 ml of a solution of 15 μg ml⁻¹ of lysozyme (from hen’s egg white, 15,000 units mg⁻¹), and (iii) 10 ml of a solution of 40 μg ml⁻¹ of pronase (from Streptomyces griseus, 95 PUK mg⁻¹). The enzyme solutions were added to the organic extract sequentially (lipase, followed by lysozyme, and finally pronase). Each enzymatic treatment with a single enzyme was carried out in a thermostatic shaker bath at 37°C and 80 oscillations per min for 90 min. Following the three incubations, the suspension was filtered through a 0.45 μm Millipore filter, and then fractionated following the procedure suggested by Ciavatta et al. (1990a).

2.6. Water-soluble organic carbon and nitrogen

WSOC and WSN were extracted according to Chen et al. (1998). The determination of TOC in solution was carried out by dichromate oxidation (Ciavatta et al., 1991), while the total nitrogen in solution was determined by acid digestion followed by automatic titration (Kjeldhal) (M.A.F., 1986).

2.7. Statistical analysis

Statistical analysis was performed using a Sigma Plot version 2000 software programme.

3. Results and discussion

3.1. Compost characteristics

The main chemical characteristics of the composting trial samples at each specific stabilization step are reported in Table 1. The different moisture contents were due to temperature changes throughout the year period. Trial “A” was conducted throughout the spring-summer season and trial “B” in wintertime. As can be seen in Fig. 1, it is possible to appreciate that the lower TOC content of sample “B” compared to sample “A” is dependent upon the different composition of the initial blends of raw materials. The TOC and total Kjeldhal nitrogen (TKN) content decreased during the composting process due to the mineralization of the OM by microorganisms.
The data for TKN, phosphorus, potassium, magnesium, manganese and sodium (Table 2), was within the normal range for these nutrients (Korboulevsky et al., 2002). Total DTPA, extractable heavy metals and other trace elements present in the different trial samples at the end of the process are shown in Table 3.

3.2. Extractable and humified organic carbon

The extractable (TEC), humified (HA + FA) and NH C content is detailed in Fig. 1. As reported by Ciavatta et al. (1990b, 1993) the different trials confirmed that TOC and TEC decreased predominantly during the first phase of composting due to the intense mineralization process. The NH fraction is lower than the humified fraction in every sample analyzed. The humified C content was overestimated particularly at the beginning of the composting process, because a part of the NH material interferes with the fractionation of humic substances (Ciavatta et al., 1990a).

The DH, the humification ratio (HR) and the index of humification (HI) of the samples illustrated the evolution of the organic fraction (Figs. 2–4). The high DH and HR values (Figs. 2 and 3) at the beginning of the process are due to the interfering substances (mainly proteins and lipids), while more authentic humification parameter values can be obtained after an enzymatic hydrolysis of the extracts (Ciavatta et al., 1990a).

Table 1
Moisture, pH, electrical conductivity and ash content in compost samples during the composting process

<table>
<thead>
<tr>
<th>Sample</th>
<th>Days of composting</th>
<th>Moisture (%)</th>
<th>pH (dil. 1:5)</th>
<th>ECw (dS m⁻¹)</th>
<th>Ash (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>A', B'</td>
<td>A'</td>
<td>B'</td>
<td>A'</td>
<td>B'</td>
</tr>
<tr>
<td>1</td>
<td>0</td>
<td>67</td>
<td>68</td>
<td>7.41</td>
<td>7.17</td>
</tr>
<tr>
<td>2</td>
<td>15</td>
<td>38</td>
<td>62</td>
<td>8.67</td>
<td>7.45</td>
</tr>
<tr>
<td>3</td>
<td>30</td>
<td>17</td>
<td>45</td>
<td>7.84</td>
<td>8.23</td>
</tr>
<tr>
<td>4</td>
<td>40</td>
<td>16</td>
<td>12</td>
<td>7.43</td>
<td>8.42</td>
</tr>
<tr>
<td>5</td>
<td>70</td>
<td>12</td>
<td>49</td>
<td>7.51</td>
<td>8.47</td>
</tr>
<tr>
<td>6</td>
<td>100</td>
<td>12</td>
<td>53</td>
<td>7.70</td>
<td>8.38</td>
</tr>
<tr>
<td>7</td>
<td>130</td>
<td>11</td>
<td>50</td>
<td>7.70</td>
<td>8.12</td>
</tr>
<tr>
<td>8</td>
<td>160</td>
<td>11</td>
<td>48</td>
<td>7.70</td>
<td>7.79</td>
</tr>
</tbody>
</table>

A' = Compost A.
B' = Compost B.
All analyses were carried out in triplicate and relative standard deviations were less than 5%.

Fig. 1. TOC, extracted (TEC), humified (HA + FA) and NH carbon in samples of compost in trials A and B.

Table 2
Total content of various nutrients in compost samples at different stages of maturation

<table>
<thead>
<tr>
<th>Sample</th>
<th>TKN (%)</th>
<th>P₂O₅ (%)</th>
<th>K₂O (%)</th>
<th>MgO (%)</th>
<th>Na (%)</th>
<th>Mn (mg kg⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>A'</td>
<td>B'</td>
<td>A'</td>
<td>B'</td>
<td>A'</td>
<td>B'</td>
</tr>
<tr>
<td>1</td>
<td>2.22</td>
<td>1.87</td>
<td>1.17</td>
<td>1.32</td>
<td>1.22</td>
<td>0.97</td>
</tr>
<tr>
<td>4</td>
<td>1.56</td>
<td>1.10</td>
<td>1.08</td>
<td>1.22</td>
<td>0.77</td>
<td>0.94</td>
</tr>
<tr>
<td>8</td>
<td>1.55</td>
<td>1.01</td>
<td>1.39</td>
<td>1.30</td>
<td>0.83</td>
<td>0.90</td>
</tr>
</tbody>
</table>

A' = Compost A.
B' = Compost B.
All analyses were carried out in triplicate and relative standard deviations were less than 5%.
3.3. Humification parameters after enzymatic hydrolysis

Continuously lower DH values were obtained after the enzymatic hydrolysis of the TEC of compost samples when compared to the untreated samples (Fig. 2). In trial “A”, prior to enzymatic hydrolysis, the DH showed an irregular trend with a negligible relationship between composting time and DH \( (R^2 = 0.35; P = \text{ns}) \). After enzymatic hydrolysis, the DH\textsubscript{enz} values decreased and showed a more regular increasing trend during composting. The relationship between time and DH\textsubscript{enz} was statistically significant \( (R^2 = 0.90; P < 0.005) \) (Fig. 2). In trial “B”, the DH trend, both before and after enzymatic hydrolysis, was very similar to that observed in trial “A” (Fig. 2). The relationship between time and DH was statistically significant after the enzymatic treatment \( (R^2 = 0.96; P < 0.0005) \).

The enzymatic treatment was highly effective on the HR\textsubscript{enz} (Fig. 3). The relationship between HR and time, before and after the enzymatic treatment, increases from \( R^2 = 0.01; P = \text{ns} \) to \( R^2 = 0.83; P < 0.05 \) in trial “A” and from \( R^2 = 0.16; P = \text{ns} \) to \( R^2 = 0.86; P < 0.01 \) in trial “B”. It must be emphasized that the HR\textsubscript{enz} showed an asymptotic trend after about 3 months of composting.

Significant differences in the relationship between HI calculated before and after the enzymatic treatment were observed (Fig. 4). The statistical parameters showed a good relationship between time and HI\textsubscript{enz} after the en-

### Table 3
Total and DTPA extractable heavy metals and some trace elements in samples of compost at the end of the composting period

<table>
<thead>
<tr>
<th>Sample</th>
<th>B</th>
<th>Total</th>
<th>DTPA</th>
<th>Co</th>
<th>Total</th>
<th>DTPA</th>
<th>Fe</th>
<th>Total</th>
<th>DTPA</th>
<th>Cu</th>
<th>Total</th>
<th>DTPA</th>
<th>Zn</th>
<th>Total</th>
<th>DTPA</th>
</tr>
</thead>
<tbody>
<tr>
<td>A\textsuperscript{'}</td>
<td>9</td>
<td>n.d.</td>
<td>28</td>
<td>7.0</td>
<td>30,640</td>
<td>1432</td>
<td>146</td>
<td>13</td>
<td>212</td>
<td>93</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>B\textsuperscript{'}</td>
<td>66</td>
<td>n.d.</td>
<td>33</td>
<td>7.2</td>
<td>19,872</td>
<td>800</td>
<td>164</td>
<td>28</td>
<td>312</td>
<td>98</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cd</td>
<td>3.2</td>
<td>&lt;0.5</td>
<td>35</td>
<td>&lt;1</td>
<td>&lt;0.5</td>
<td>n.d.</td>
<td>46</td>
<td>8</td>
<td>55</td>
<td>12</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cr</td>
<td>1.3</td>
<td>&lt;0.5</td>
<td>52</td>
<td>&lt;1</td>
<td>&lt;0.5</td>
<td>n.d.</td>
<td>57</td>
<td>5</td>
<td>41</td>
<td>11</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

A\textsuperscript{'} = Compost A.
B\textsuperscript{'} = Compost B.

All analyses were carried out in triplicate and relative standard deviations were less than 5%.

**Fig. 2.** Relationship between DH, degree of humification after enzymatic hydrolysis (DH\textsubscript{enz}) and days of composting in samples of compost of trials A and B, ns = not significant.

**Fig. 3.** Relationship between HR, humification ratio after enzymatic hydrolysis (HR\textsubscript{enz}) and days of composting in samples of compost of trials A and B, ns = not significant.
zymatic treatment: \( R^2 = 0.82, \ P < 0.05 \) (Fig. 4, trial “A”) and \( R^2 = 0.95, \ P < 0.01 \) (Fig. 4, trial “B”).

The presence of the humic-like substances was significantly higher at the beginning of the composting process. These substances were used by the microorganisms both as an energy source and for synthesis of humic substances (Ciavatta et al., 1990a; Adani et al., 1995, 1997).

### 3.4. Total water-soluble organic carbon and nitrogen

The concentration of WSOC in the water extract progressively decreased with maturation time in both cases (Fig. 5). These results are in agreement with those previously reported by Chen et al. (1998). At the beginning of composting, the WSOC content was 1630 mg L\(^{-1}\) in trial “A” and 1400 mg L\(^{-1}\) in trial “B” and after 160 days it decreased respectively to 38% and 16% of the initial values. A good correlation was found between composting time and WSOC:

\[
y = 9E-05x^2 - 0.0183x + 1.2699 \\
R^2 = 0.82; P<0.05
\]

\[
y = 2E-05x^2 - 0.0041x + 0.553 \\
R^2 = 0.39; P = ns
\]

At the same time the WSN decreased from 280 to 180 mg L\(^{-1}\) and from 200 to 98 mg L\(^{-1}\) in trial “A” and “B” respectively. The WSN contents showed a good correlation

\[
y = 3E-05x^2 - 0.0104x + 1.4221 \\
R^2 = 0.95; P<0.001
\]

\[
y = 9E-06x^2 - 0.0014x + 0.4982 \\
R^2 = 0.23; P = ns
\]
with maturation time, but no significant correlation with the humification parameters. These results confirm that the microbial biomass subtracts nutrients, including soluble N, for their metabolism during composting.

4. Conclusion

Composting is a biological, aerobic process in which microorganisms utilize OM for their metabolism. The parameters usually used to determine the maturity of compost (DH, HR, HI) are often overestimated due to the presence of humic-like substances such as proteins, lipids and intermediate microorganism metabolites. A significant relationship between composting time and WSOC was found. Enzymatic treatment of the whole extract produced very interesting results. The DH, HR and HI calculated after enzymatic hydrolysis (DH_{enz}; HR_{enz}; HI_{enz}) showed a more realistic trend because this treatment removes the humic-like substances. Furthermore, the statistically significant relationship found between humification parameters after enzymatic hydrolysis (DH_{enz}; HR_{enz}; HI_{enz}) and WSOC demonstrates that it is possible to monitor the composting maturation process more easily and rapidly avoiding longer and more expensive analytical procedures.

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