Physicochemical and Microbiological Properties and Humic Substances of Composts Produced with Food Residues

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Received: September 28, 2017      Accepted: November 7, 2017      Online Published: December 15, 2017
doi:10.5539/jas.v10n1p180          URL: https://doi.org/10.5539/jas.v10n1p180

Abstract

The consolidation of a wide and effective management system of solid residues, especially biodegradable ones, is one of the great challenges of current society. Composting was evaluated as an option of organic fertilization for soil enrichment, using raw food residues in substitution to bovine manure. The compost piles were built with 30% of biodegradable residues mixed with 70% of ground tree pruning material. The effects of different proportions of food residues (FR) and bovine manure (BM) as source of carbon were tested in 5 treatments (T₁ = 15%BM + 15%FR, T₂ = 20%BM + 10%FR, T₃ = 10%BM + 20%FR, T₄ = 30%BM and T₅ = control, 30%FR), in randomized blocks, under open field conditions for 90 days. The pH, temperature and moisture content of the compost were measured weekly. The aged compost was evaluated for physicochemical and microbiological properties and carbon contents in the humic substances. The analyses of the results indicated that all studied composts reached the maturation stage with satisfactory contents of humic substances, macronutrients, and micronutrients, indicating that food residues can be used as a source of carbon in compost piles to produce organic fertilizers. The contents of the evaluated chemical contaminants were much lower than those established in the main legislation and current normative instructions and, in terms of contamination by pathogens, there was the absence of total coliforms, thermo tolerant coliforms, and Salmonellas.

Keywords: nutrients recycling, organic fertilization, C/N ratio

1. Introduction

A number of solid residues disposed of by the population is extremely large and, when inadequately disposed, they become a problem for society, because they can cause environmental impacts, modifying the quality of soil, air and water bodies, which represents a risk to the public health (Bulcão et al., 2010).

Thus, to face the situation in the social context of the country, there arise individual (garbage collectors) and collective (associated groups, cooperatives, and companies) initiatives whose operations are based on the process of collection, separation, reuse and/or recycling of these materials.

Currently, there are two recycling associations in the city of Mossoró/RN: Associação Comunitária Reciclando para a Vida (ACREVI) and Associação dos Catadores de Material Reciclável de Mossoró (ASCAMAREM) (Cavalcanti et al., 2011). These associations work with the selective collection and collaborate to minimize the negative impacts that the residues can cause to the environment (Ferreira et al., 2012).

However, as in most recycling associations in Brazil, there are no programs for the recycling of biodegradable materials and their disposal in landfills is common, causing serious environmental problems. From the sustainability point of view, biodegradable residue must efficiently return to the economic and productive cycle, to guarantee their final disposal.
In this context, composting becomes an option for the reuse of biodegradable residues as one form of production of organic fertilization, which can be used to enrich soil, since it provides nutrients that are essential to its fertility, besides meeting sanitary, social, ecological and economic aspects (Inácio & Miller, 2009).

Nevertheless, to avoid negative environmental impacts of the use of the composting of biodegradable residues in agriculture, it is necessary to define parameters relative to physicochemical and microbiological quality, besides the possible contamination by pathogens or heavy metals. Thus, composting was evaluated as an option for an organic source of fertilization in agriculture using raw material food residue in substitution to bovine manure.

2. Material and Methods

The experiment was carried out in the courtyard of the Associação Comunitária Reciclando para a Vida (ACREVI), located in the Nova Vida neighborhood, municipality of Mossoró - RN (5º14′9″ S; 37º18′59″ W; 18 m asl.).

The biodegradable residues (bovine manure and food residues) used for the composts were collected in popular restaurants and households of the local urban area. Filling materials consisted of tree pruning residue collected by the urban cleaning service of the municipality. In total, approximately 7 tons of vegetal material, 1,500 kg of bovine manure and 1,500 kg of food residue were collected. All piles were built with 70% of the vegetal material (V) and 30% of bovine manure (BM) and food residues (FR), as recommended by Pereira Neto (2007).

The effects of different proportions of food residues (FR) and bovine manure (BM) as source of carbon were tested in 5 treatments (T1 = 15%BM + 15%FR, T2 = 20%BM + 10%FR, T3 = 10%BM + 20%FR, T4 = 30%BM and T5 = 30%FR), in randomized blocks, under open field conditions during 90 days.

The compost piles were built with a conical shape, 1.60 m high and 2.00 m wide, spaced and parallel to one another to facilitate the turning, passage of materials and access. The manual turning of the compost piles occurred every three days in the first week of composting and, subsequently, every ten days. The piles were daily irrigated using tap water.

During the maturation process, the piles were weekly monitored through measurements of the parameters temperature, moisture, and pH. In addition, the contents of total organic carbon (TOC), total nitrogen (TN) and C/N ratio were determined in four periods, 2 during the degradation stage of the composts (beginning and at 10 days from the building of the piles) and the others at 30 and 60 days after the beginning of the composting and during maturation.

TOC was determined through the wet oxidation method with external heating, while TN was determined through the wet digestion method in the open system, using a block digester (Tedesco et al., 1995; Santos et al., 2009). The C/N ratio was obtained by the ratio between the TOC and nitrogen, expressed in percentage.

After maturation, the composts were analyzed for humic substances (Swift, 2001), macro and micronutrients, heavy metals (Santos et al., 2009), total coliforms, thermo tolerant coliforms and Salmonella (Siqueira, 1995). For the determination of humic substances, the samples of each treatment were ground, passed through a 60-mesh sieve (0.210 mm) and subjected to the fractioning of humic substances (Swift, 2001). From this fractioning, the fractions corresponding to fulvic acids, humic acids, and humins were obtained through the differential solubility in acidic and alkaline solutions.

The contents of macro- and micronutrients and chemical contaminants were determined through wet digestion in a closed system using a microwave oven as a source of heat and concentrated nitric acid to digest the compost. Then, the liquid extract obtained from this process was analyzed for the contents of Cu, Zn, Fe, Mn, Cd, Ni, Pb, Ca and Mg, through atomic absorption spectrophotometry, and P, Na and K, through calorimetry and flame spectrophotometry, respectively (Santos et al., 2009).

Total coliforms and thermo tolerant coliforms were evaluated using the most probable number (MPN) technique, also known as a multiple-tube method (Siqueira, 1995).

All analyses were performed in triplicate. The results were subjected to analysis of variance (p < 0.05), using the statistical program ASSISTAT 7.7 (Silva & Azevedo, 2016). Tukey test was used to compare the mean values.

3. Results and Discussion

3.1 Composition and Characteristics of the Compost piles

Table 1 shows the composition and characteristics of the piles during the composting process. At the end of the composting, the results showed that there is no variation of temperature (35 °C) between the studied treatments and the observed values allow the reduction and/or elimination of pathogens. However, there were small
variations of pH (7.3 to 7.8), TOC (7 to 11%) and TN (0.5 to 0.8%). Regarding the C/N ratio, the piles of the treatments with 30% of food residues (OC_4) showed higher C/N ratio than the other treatments, while the OC_4 compost pile showed the lowest moisture content (48%).

Table 1. Composition and parameters presented in the piles during the composting process

| Piles | C %  | T °C  | U%  | pH   | TOC% | TN%  | C/N% |
|-------|------|-------|-----|------|------|------|------|
| OC_1  | 70 VM| St. ≈ 35 | St. 55 | St. ≈ 6.0 | St. ≈ 23 | St. 0.9 | St. ≈ 26 |
|       | 15 BM| Int. ≈ 55 | Int. 45 | Int. 7.1-7.2 | Int. ≈ 12 | Int. 0.8 | Int. ≈ 15 |
|       | 15 FR| En. ≈ 35 | En. 51 | St. 7.8 | En. ≈ 11 | En. 0.8 | En. ≈ 13 |
| OC_2  | 70 VM| St. ≈ 35 | St. 55 | St. 6.0 | St. ≈ 19 | St. 0.8 | St. ≈ 23 |
|       | 20 BM| Int. ≈ 55 | Int. 45 | Int. 6.9-7.2 | Int. ≈ 10 | Int. 0.7 | Int. ≈ 14 |
|       | 10 FR| En. ≈ 35 | En. 51 | En. 7.3 | En. ≈ 9 | En. 0.7 | En. ≈ 13 |
| OC_3  | 70 VM| St. ≈ 35 | St. ≈ 40 | St. 6.4 | St. ≈ 19 | St. 0.7 | St. ≈ 25 |
|       | 10 BM| Int. ≈ 55 | Int. 28-40 | Int. 5.8-6.9 | Int. ≈ 9 | Int. 0.5 | Int. ≈ 19 |
|       | 20 FR| En. ≈ 35 | En. 51 | En. 7.6 | En. ≈ 7 | En. 0.5 | En. ≈ 14 |
| OC_4  | 70 VM| St. ≈ 35 | St. ≈ 20 | St. 6.8 | St. ≈ 17 | St. 0.9 | St. ≈ 19 |
|       | 30 BM| Int. ≈ 55 | Int. 32-35 | Int. 6.8-7.2 | Int. ≈ 11 | Int. 0.7 | Int. ≈ 15 |
|       | 0 FR | En. ≈ 35 | En. 48 | En. 7.8 | En. ≈ 10 | En. 0.7 | En. ≈ 14 |
| OC_5  | 70 VM| St. ≈ 35 | St. ≈ 61 | St. 5.5 | St. ≈ 17 | St. 0.7 | St. ≈ 24 |
|       | 30 BM| Int. ≈ 55 | Int. 55-62 | Int. 5.8-6.0 | Int. ≈ 10 | Int. 0.6 | Int. ≈ 17 |

Note. C = composition; T = temperature; U = moisture; VM = vegetal material; TOC = total organic carbon; TN = total nitrogen; C/N = nitrogen carbon ratio; BM = bovine manure; FR = food residues; St. = start; Int. = intermediary; En. = end.

3.2 Carbon Contents in Humic Substances

According to the ANOVA, the variable carbon fractions of the humic acids (C-HA), fulvic acids (C-FA) and humin (C-HUM) were significantly influenced (p < 0.01) by the proportions of food leftovers of the compost piles (Table 2).

Table 2. Analysis of variance of carbon fraction contents of the humic substances in organic composts produced with different proportions of bovine manure and food residues

| Variation sources | Freedom degree | Carbon fractions |
|-------------------|----------------|-----------------|
|                   |                | C-AH            | C-AF            | C-HUM           |
| Treatments        | 4              | 0.22**          | 3.61**          | 1341.18**       |
| Error             | 10             | 0              | 0              | 0.08            |
| Total             | 14             | 0.91           | 14.45          | 5365.49         |
| CV (%)            |                | 1.86           | 1.82           | 0.19            |

Note. ns; *, ** not significant, significant at the 0.05 and 0.01 level of significance according to the F test; CV = coefficient of variation.

In general, among the fractions of humic substances, there was higher C content in humins (C-HUM), followed by humic acid (C-HA) and fulvic acid (C-FA) (Figure 1). This high C content in C-HUM is possibly due to the lower solubility of this fraction associated with the high polymerization and, consequently, higher degree of stabilization.
Figure 1. Mean values of the fractions C-HA (A), C-AF (B) and C-HUM (C) in organic composts produced with different proportions of bovine manure and food residues.

*Note.* Averages followed by the same letter do not differ statistically by Tukey test at the 5% probability level.

The mean values of the fractions C-HUM, C-HA and C-FA stood out in the control pile (30% BM) (Figure 1), which allows to infer that the decrease of diversity can cause increment in the mineralization rates, increasing the interaction of these fractions with the mineral portion present in the organic compost. It was observed that the means of the C-HA fractions did not differ statistically (p < 0.05) in the piles OC1 (15% BM and 15% FR) and OC3 (10% BM and 20% FR), in relation to the control, which were more satisfactory in the evaluation of this fraction. However, these piles differed from OC2 (20% BM and 10% FR) and OC5 (30% FR), which were considered as the worst in the estimate of C-HA.

Although OC1 and OC3 are similar to the control and considered as the most significant piles in the C-HA fraction evaluation, the control (OC4) is even more expressive in the analysis of this fraction. Thus, when C-HA was evaluated in the other piles, it suffered a more relevant decrease in OC2 (38.3%), compared with the control. In the other piles, this reduction varied from 18 to 20% approximately, compared with OC4.

According to the means of C-FA, there was statistical difference between almost all piles in the final process. This demonstrated that the FA fraction is more sensitive to changes than HA. However, the pile OC4 was superior to the others regarding the quantification of C-HA, while OC2 and OC3 did not exhibit statistical differences, being considered as the worst in the estimate of this fraction.

In the quantification of the humin fraction, all piles were different (p > 0.05), with the control (30% BM) as the most significant and OC3 (10% BM and 20% FR) as the least significant. This statistical difference in the piles can be a consequence of the microbial decomposition, which is particular, in its time, for each composting process (Matiz et al., 2015).

Silva et al. (2009) point out that there are studies in the literature relating the maturation degree of a compost to the characteristics of the humic compost present, associating this parameter to the polymerization degree. Thus, it can be inferred that the produced composts were mature, since the final values of this C-HA/C-FA ratio were 1.63, 2.33, 3.00, 1.25, and 1.54, for the samples collected in the final stage in the compost piles OC1, OC2, OC3, OC4, and OC5, respectively.

### 3.3 Contents of Macro and Micronutrients of the Composts

According to the ANOVA, there was a significant effect of the proportions of food residues in the compost on the variables nitrogen, phosphorus, potassium, calcium, copper, iron and zinc (p < 0.01), besides magnesium and manganese (p < 0.05) (Table 3).
Table 3. Analysis of variance of the nutrients in organic composts produced with different proportions of bovine manure and food residues

| FV   | GL  | N   | P   | K   | Ca  | Mg  | Cu  | Mn  | Fe   | Zn  |
|------|-----|-----|-----|-----|-----|-----|-----|-----|------|-----|
| Treatments | 4   | 4.51** | 0.10** | 0.13** | 11.02** | 0.09* | 36.60** | 1499.65* | 1,031,226.33** | 214.00** |
| Error | 10  | 0.02 | 0.00 | 0.00 | 0.77 | 0.02 | 3.22 | 267.03 | 139,875.65 | 10.46 |
| Total | 14  | 18.18 | 0.39 | 0.61 | 51.78 | 0.52 | 178.63 | 8668.94 | 5,523,661.83 | 960.58 |

CV (%) | 1.9 | 6.03 | 12.41 | 15.44 | 11.07 | 36.01 | 27.29 | 21.6 | 10.24 |

Note. *; ** significant at the 0.05 and 0.01 level of significance according to the F test; CV = coefficient of variation

There was greater concentration of N and Ca in all organic composts, compared with the other analyzed macronutrients (Figure 2). In general, N stood out as the primary macronutrient in largest amount, since it showed the highest means in OC₁, OC₂ and OC₄. The addition of the inoculant bovine manure, on the piles (Table 1) that formed these organic composts, may have been a factor that favored the increase in the concentration of this nutrient.

Figure 2. Mean values of macronutrients Nitrogen (A), Phosphorus (B), Potassium (C), Calcium (D) and Magnesium (E) in organic composts produced with different proportions of bovine manure and food residues

Note. Averages followed by the same letter do not differ statistically by Tukey test at the 5% probability level.
The other composts, OC3 and OC5, showed higher percentage of Ca, possibly due to the increment of food leftovers in the composition of the piles (Table 1), which favor the formation of these composts.

Assessing wastewater biosolids composting, Matiz et al. (2015) verified that in regarding the physicochemical and nutritional characteristics, none of the treatments showed significant differences and their characteristics were similar and their values within norm.

As to the evaluated cationic micronutrients, Fe stood out over the others, exhibiting a higher concentration in all organic composts (OC), equivalent to 94.0, 96.6, 92.4, 94.5, and 94.5% in OC1, OC2, OC3, OC4, and OC5, respectively. These values may have been due to the site where the experiment was installed, because the piles were built in an association of recyclable material collectors, where the piles are accommodated on the floor in contact with residues in the soil. Similar results were found by Primo et al. (2010), who analyzed the nutritional quality of organic compost produced with tobacco residues and observed high Fe concentrations.

In general, OC3 stood out as the most significant in the evaluation of Cu, Mn and Fe (Figure 3). However, OC3 was not as relevant in the quantification of Fe as it was for Cu and Mn. The OC3 was considered as the worst in the estimate of the analyzed micronutrients.

Regarding Cu, the organic composts that stood out in the evaluation of this micronutrient were OC1 and OC3, while OC2 was statistically equal to OC3 and OC4. Despite that, OC3 was the most expressive in the evaluation of Cu, because when it was analyzed in the other composts, there were reductions of 38.9% in OC1, 65.6% in OC2, 70.4% in OC4 and 85.4% in OC5 (Figure 3).

![Figure 3. Mean values of micronutrients Iron (A), Copper (B), Manganese (C) and Zinc (D) in organic composts produced with different proportions of bovine manure and food residues](image)

*Note.* Averages followed by the same letter do not differ statistically by Tukey test at the 5% probability level.

For the micronutrient Mn, more relevant values were observed in the composts OC3 and OC4, while OC1 and OC2 were statistically equal and OC3 was the least prominent in the Mn analysis. It should be pointed out that, when Mn was evaluated in the OCs with statistically lower contents, OC4 showed reductions of approximately 42, 38 and 64% in comparison to OC1, OC2, and OC3, respectively.

In the quantification of the nutrient Zn, higher contents were observed in OC1, but it did not differ statistically from OC2. OC3 was the organic compost with lowest contents of Zn, but it was statistically similar to OC4, which did not differ from OC4. It is ratified that Zn suffered a decrease in OC3, compared with the other analyzed
composts. These reductions were approximately of 85, 48, 74, and 63% referring to OC2, OC3, OC4, and OC5, respectively.

In general, the organic composts OC1 and OC4 stood out in the quantification of the macronutrients and OC3 in the evaluation of micronutrients. However, despite these differences in the quantification of nutrients in the different organic composts, it should be pointed out that there are no specifications of standard limits for the contents of nutrients analyzed in organic composts, because both macro and micronutrients are related to the type of matter used in the production of the compost or in the composition of the compost piles. Since these contents do not exist, studies conducted in this research line are adopted as reference, as well as concentrations considered as ideal for agriculture.

The values obtained in the present study indicate that the analyzed organic composts had good quality, since, according to Malavolta (1980), they have macro and micronutrients in adequate concentrations for agriculture.

3.4 Heavy Metals

According to the summary of the ANOVA, only nickel (Ni) showed significant effect (p < 0.01) of the proportions of food residues in the composting (Table 4).

Table 4. Analysis of variance of chemical contaminants in organic composts produced with different proportions of bovine manure and food residues

| Treatments | GL | Ni    | Cd    | Pb    |
|------------|----|-------|-------|-------|
| Treatments | 4  | 8.64**| 0.01**| 24.05*|
| Error      | 10 | 0.04  | 0.02  | 7.31  |
| Total      | 14 | 34.96 | 0.26  | 169.31|
| CV (%)     |    | 5.51  | 52.79 | 76.9  |

Note. ns; ** not significant and 0.01 level of significance according to the F test; CV = coefficient of variation.

As in the quantification of nutrients, there is not a specific legislation containing the acceptable concentrations of the metals that are considered as chemical contaminants to soil and/or plant; instead, there are proposals of resolution referring to the quality parameters for the organic compost (CONAMA resolution proposal 02/2016). This proposal presents the quality contents that the organic compost must have for any allowed application as fertilizers and soil conditioners, including agricultural, gardening and landscaping applications.

Thus, the values referenced in the present research were based on the proposals of resolution, studies and acceptable contents established by some countries of Europe and the United States (Table 5).

Table 5. Acceptable contents of chemical contaminants in mg kg⁻¹ for organic compost and the values found in the research

| Countries/Resolutions | Ni | Cd | Pb |
|-----------------------|----|----|----|
| Germany               | 50 | 15 | 150|
| Austria               | 200| 6  | 900|
| Switzerland           | -  | 3  | 150|
| Italy                 | 200| 10 | 500|
| Netherlands           | 50 | 2  | 20 |
| EUA                   | 100| 10 | 500|
| CONAMA Prop. Res 02/2016 | 70 | 1.5| 150|
| OC1                   | 0.8| 0.2| 1  |
| OC2                   | 4.4| 0.3| 5.3|
| OC3                   | 4.2| 0.3| 4.3|
| OC4                   | 4.1| 0.3| 0.5|
| OC5                   | 5.1| 0.3| 3.6|

Note. OC1 (15%FR + 15%BM); OC2 (10%FR + 20%BM), OC3 (20%FR + 10%BM), OC4 (30%BM) and OC5 (30%FR).
Based on the adequate values of chemical contaminants presented in Table 5, all analyzed OCs contained adequate concentrations of the metals Ni, Cd and Pb, because the observed values were much lower than those established by the mentioned references.

Due to loss of carbon during composting process, the concentration of heavy metals may increase thus, its monitoring is essential (Matiz et al., 2015). According to Manios and Stentiford (2006), and Cai et al. (2007) the results obtained at end of the process can be higher for compared with raw material possibly due to volatilization of the materials, transformation of organic matter into CO2 and NH3 material weight loss.

The significant effect of the contaminant Ni when subjected to the test of means demonstrated that OC3 showed higher contents of this heavy metal, while OC2, OC3, and OC4 were statistically similar, and OC1 was the least prominent in the evaluation of this metal (Figure 4).

![Figure 4. Mean values of contaminant Nickel in organic composts produced with different proportions of bovine manure and food residues](image)

*Note.* Averages followed by the same letter do not differ statistically by Tukey test at the 5% probability level.

Although OC5 is the most expressive in the analysis of Ni, the concentration found in this compost is still considered much lower than those observed in the literature. Nickel is a heavy metal that, at high concentrations, can cause serious physiological disorders or even the death of the plants. At low concentrations, however, it is considered as an essential nutrient to plants, participating in metabolic processes as activator of urease (Berton et al., 2006; Pires & Andrade, 2006; Wood et al., 2006).

### 3.5 Microbiological Quality of the Composts

The microbiological quality of the composts, the standards and criteria to confer their quality in relation to the analysis of Salmonella and coliforms, was evaluated following the Normative Instruction (NI) n° 27/2006 of the Secretariat of Agriculture and Livestock Defense - SDA, for fertilizers, correctives, inoculants and biofertilizers, as well as the resolution proposal 02/2016 (CONAMA process 02000.001228/2015-37). These references indicate maximum allowed concentrations of pathogenic agents for humans, animals and plants.

In this context, there was no contamination by Salmonella species in the produced organic composts. In addition, the numbers of total coliforms in all produced composts were lower than the maximum value allowed, compared with organic fertilizers. The decontamination may have been a consequence of the maintenance of temperature for adequate time during the composting process, because, according to Orrico Júnior et al. (2009) and Liang et al. (2003), for the composting process to have a significant reduction of pathogenic microorganisms, the material needs to achieve high temperatures and the temperature must remain for various days, because microbial biomass is influenced directly by it.

The highest value of total coliforms was observed in OC4, which probably occurred due to the higher percentage of bovine manure used in the composition of the pile (Table 1), which favored the formation of the organic compost OC4. Additionally, only OC4 showed values of thermotolerant coliforms, but they are within the limits for Class I organic composts (CONAMA resolution proposal 02/2016).

The removal and/or reduction of pathogens in organic composts is extremely relevant, especially when the organic compost is intended for the production of vegetables, which in general are consumed fresh and need to be pure and healthy, free from pathogens that can lead to diseases.
Therefore, the results ensure the stability of the produced composts, since there was a complete microbiological decomposition, free from pathogens and toxicity.

4. Conclusions

All the produced composts reached the maturation stage with satisfactory contents of humic substances, macronutrients and micronutrients, indicating that the food residues can be used as source of carbon in compost piles.

The concentrations of heavy metals in the organic composts were lower than those established in the main European and American legislations, as well as in the NI n° 27/2006 of the Secretariat of Agriculture and Livestock Defense (SDA) and in the resolution proposal 02/2016 (CONAMA process 02000.001228/2015-37).

The composts suffered a complete microbial decomposition, which led to the reduction and/or removal of contaminant microbiological agents, showing absence of total coliforms, thermotolerant coliforms and Salmonella.

The composting of biodegradable residues does not have factors of risk to the public health, emerging as an initiative and means of recycling of food leftovers, avoiding negative environmental impacts.

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