

## Chemical and spectroscopy characterization of a compost from food waste applying the hot composting Berkeley method

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Received: 13 April 2020 / Accepted: 14 July 2021 / Published online: 05 March 2022

### Abstract

**Purpose** This study aims to characterize the compost produced under low input conditions in thirty days of composting from food wastes applying the hot composting Berkeley method (HCBM) by <sup>13</sup>C NMR CP/MAS techniques combined with evaluation of chemometric, physical and chemical parameters.

**Method** The composting process from food waste was carried out following the recommendations of the Hot Composting Berkeley method. At 7, 9, 11, 13, 15, 17, 19, 21, 23 and 25 days turning was carried out, totaling eleven turns and the temperature, pH, electrical conductivity, dry density, potential CO<sub>2</sub> emission and potential NH<sub>3</sub> emission were quantified. The metal content in the compost was determined after 25 days. The structural modifications of compost process were monitored by <sup>13</sup>C-NMR CP MAS spectroscopic and chemometrics analysis.

**Results** Composting takes place immediately and continues for 15 days through transformation to more labile lipid, carbohydrate and proteinaceous molecules (C<sub>Alkyl</sub>-H,R; C<sub>Alkyl</sub>-O; C<sub>Alkyl</sub>-O,N) with increasing temperature and pH. After 15 days, there is a CO<sub>2</sub> and NH<sub>3</sub> emission reduction as well as preservation of recalcitrant molecules with more aromatic and nonpolar characteristics (C<sub>Aromatic</sub>-H,R; C<sub>Aromatic</sub>-O,N).

**Conclusion** The compost obtained at 25 days of composting by applying HCBM has greater hydrophobicity and aromaticity and is rich in mineral elements important for plant nutrition. The application of HCBM to food waste allowed to obtain a stabilized compost in ecological production conditions.

**Keywords** Humification, Composting, Spectroscopy, Gas emission

### Introduction

The compost obtained from natural sources, food, and organic waste, as well as the soluble humic compounds (humic substances - SH) constitute an alternative to reduce the contaminant charge of these environmental residues while having high agronomic value (Mbarek et al. 2019; Dadi et al. 2019). The compost incorporated in soil, mainly in degraded areas, contributes to the accumulation of moisture, improved infiltration and

hydraulic conductivity (Kranz et al. 2020). There is a consensus in the literature that compost stimulates plant growth, improves flowering, fruiting and increases the presence of beneficial microorganisms in soil (Stewart-Wade 2020).

Composting is a biological process of organic waste degradation and transformation into a stabilized material and releases CO<sub>2</sub> and water vapor. This process consists of aerobic and thermophilic bio-oxidation, which takes place through diversified and intense microbiological activity that generates heat, raising temperatures up to over 80 °C (Azim et al. 2018; Clark 2019). Composting takes place in two distinct stages: the mesophilic stage, with temperatures varying between 25 and 45 °C, and the thermophilic stage, reaching 45 to 85 °C. These stages are reached through the action of different microorganisms, such as bacteria, fungi and actinomycetes, which act on the organic material according

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to the physical and biochemical characteristics of the environment (Inácio et al. 2009; Maccari et al. 2020).

During the composting process, different complex molecules undergo different transformations due to the degradation that takes place by microorganismal action. In composting, there is an initial degradation of the most labile compounds, followed by the preservation of the most recalcitrant molecules (El Hajjouji et al. 2008). The composting of chicken manure and swine waste is dominated by C-alkyl-O structure mineralization, and therefore, a quantity of C-aromatic structures is preserved (Huang et al. 2017). Cao et al. (2019) showed that the C-aromatic, C<sub>Alkyl</sub>-O and C-phenolic structures can function as indicators of the degree of humification of rice husk composting.

The regular duration of a composting process varies between 90 and 120 days and depends on the raw material used, the method and the degree of stabilization and maturity (Balmori et al. 2019). The “hot Berkeley method” foresees obtaining compost in approximately 3 weeks (Raabe 2015; Salmon 2012). In this method, some premises are necessary, such as the raw material size which must be between 0.5 and 1.5 inches (approximately 1.3 to 4 cm) and requires a C:N ratio of approximately 30:1 for the compost pile. The relative moisture content of the pile should be kept at approximately 50%, and the pile dimensions should be close to 36”×36”×36” (approximately 1.0 m × 1.0 m × 1.0 m). Nevertheless, constant turning is important to guarantee the necessary aeration for biological activity and to prevent the temperature from rising to the point of inhibiting the growth of microorganisms and interrupting the process. Thus, the more rotations there are, the faster the composting process will take place (Veeresh et al. 2010; Maqbool and Rehman 2015).

Heterogeneity in degradation and the various transformations that take place is one of the reasons why decreasing composting duration can affect the compost quality. In many cases, the composting process is not properly studied or monitored, generating a lower quality compost when obtained in shorter times, since there is a relationship between the compost quality and the production times (de Souza et al. 2019; García et al. 2013).

The levels’ decrease of food waste in the cities constitute an environmental emergency with objectives outlined by the scientific community until 2030 (Ghinea et al. 2019). There are several challenges for

the composting of food waste, including those related to toxicity to plants (Voběrková et al. 2020). In this sense, one of the improvement alternatives has been the incorporation of additives to compost and co-composting, however, the times of these processes still exceed 60 days and the mechanisms associated with transformations are not yet established (Awasthi et al. 2020). As obtaining quality compost in relatively short times is an environmental requirement and knowing the mechanisms associated with this process is highly important, it is that in the present study we propose an alternative to solve these problems. Some studies have characterized the compost obtained by the Berkeley method (Nawawi et al. 2016), however, to the best of our knowledge, here we present one of the few studies that perform a spectroscopic monitoring of the composting process.

In this scenario, we hypothesize that the hot composting Berkeley method allows to obtain a viable compost for future use in agriculture in terms of stability, structural quality and nutrients and can be obtained from municipal waste residues in up to thirty days of composting. To test this hypothesis, the present study aimed to characterize the compost obtained from food waste by applying the Berkeley method and using chemical-physical (NH<sub>3</sub>, CO<sub>2</sub>, pH, temperature, electrical conductivity, density), spectroscopy (<sup>13</sup>C NMR CP/MAS) and chemometric techniques (Principal component analysis).

## Materials and methods

### Location and construction of the composting patio

Implementation of the composting process was carried out by building a patio at a low cost and with minimal environmental impact. The composting yard was located at the Federal Rural University of Rio de Janeiro (UFRRJ), Seropédica-RJ, located in Baixada Fluminense, at an altitude of 26 m and coordinates of 22°45’S and 43°40’W. The site for patio construction was chosen due to its proximity to the sources of waste production, physical characteristics of the soil and easy accessibility. The constructive technique used was “pau-a-pique”, using wooden and bamboo structures covered with a clay mass, sand and dry straw to fill in the walls and stalls. Due to the materials used, the construction guaranteed the appropriate aesthetic and thermal comfort characteristics (Fig. 1).



**Fig. 1** Construction of the composting patio for the application of Hot Composting Berkeley Method in food waste. Original photos by Bruna Marraccini Precioso de Oliveira and “Composta Rural Group”

### Composting process

The composting process was carried out following the recommendations of the hot composting Berkeley method (Raabe 2015). The raw materials were collected from organic residues produced in university residences and restaurants in the locality. The residues were collected daily, and, as a source of dry matter, grass clippings from the fields at the university campus were used. Irrigation was carried out with each addition of new material at all times to ensure a good distribution of humidity without causing waterlogging.

Organic waste was separated and allocated in cataloged and specific buckets for this purpose. The waste was collected daily and transported to the composting patio, and, in this way, the pile was incremented with the residues and covered with dry straw in a ratio of 1:1 (food waste): (dry straw) (mass:mass) until the pile reached an approximate height of 1.20 m. On the fifth day, the pile started to be turned on alternate days until the thirtieth day, as counted from the beginning of composting. Subsequently, the patio was divided into stalls, and then the compost was manually transported to the front stall, which was denoted the first stall. Then, turnover was carried out following the “mirror stalls” scheme. At 7, 9, 11, 13, 15, 17, 19, 21, 23 and 25 days, turning was carried out, totaling eleven turns.

### Evaluation of chemical and physical parameters

#### Determination of chemical and physical parameters

The parameters of temperature, pH, electrical conductivity, dry density, potential CO<sub>2</sub> emission and potential NH<sub>3</sub> emission were performed at 7, 9, 11, 13, 15, 17, 19,

21, 23 and 25 days according to the number of turns. After each turn, three composite samples were collected, which were homogenized in a plastic bucket and then subjected to the quartering process to reduce the size until a volume of 250 ml was reached. The reserved fractions were identified and then frozen for further analysis of pH, electrical conductivity (EC), density and potential CO<sub>2</sub> and NH<sub>3</sub> emissions.

#### Temperature determination

The average environment and compost temperatures were measured immediately before the start of all compost turning. After the last turning, the compost remained in the stall, without any intervention for another 15 days, when the temperature was measured again. The average temperatures were calculated from five replicates of recorded temperatures with a skewer thermometer at five different points in the pile (Oliveira et al. 2014).

#### pH value and electrical conductivity determination

The pH analyses were performed in a distilled water solution 5:1 (v/v), and the electrical conductivity was determined in the same aqueous extract obtained for the pH measurement, according to the methods of Oliveira et al. (2014).

#### Dry density determination of compost

The density on a dry basis was determined by quantifying the wet mass and dry matter content of the material contained in the 250 ml pots. The dry material was obtained after drying in an oven at 60 °C for 3 days (Oliveira et al. 2014).

### Determination of potential emissions of CO<sub>2</sub> and NH<sub>3</sub>

Potential emissions of CO<sub>2</sub> and NH<sub>3</sub> were quantified according to methods described by Oliveira et al. (2014) with an incubation temperature to 30 °C. Briefly, 20 g of sample was placed in a 500 ml plastic pot with a container with NaOH to capture CO<sub>2</sub> and another container containing boric acid to capture NH<sub>3</sub>. After 5 days of incubation at 30 °C, 2.0 ml of 1.0% barium chloride (m:v) was added to the NaOH (0.1 mol/L<sup>-1</sup>) to precipitate carbonate, which was then titrated with HCl (0.5 mol/L<sup>-1</sup>) using phenolphthalein as an indicator. The turning point from pink to colorless was observed. The amount of CO<sub>2</sub> emitted was determined using the equation described by Oliveira et al. (2014). NH<sub>3</sub> determination was performed by titration with H<sub>2</sub>SO<sub>4</sub> (0.025 mol/L<sup>-1</sup>) and calculated by the equation described by Oliveira et al. (2014). Only samples that showed a change in the color of the solution from lilac to green showed evidence of NH<sub>3</sub> emission, and these were taken to titration.

### Quantification of metallic and nonmetallic elements in the compost

The metal content in the compost (Cu<sup>2+</sup>, Mn<sup>2+</sup>, Fe<sup>3+</sup>, Zn<sup>2+</sup>, Cd<sup>2+</sup>, Pb<sup>2+</sup>, Ni<sup>2+</sup>, Mg<sup>2+</sup>, Al<sup>3+</sup> and Ca<sup>2+</sup>) was determined after 25 days of composting using an atomic absorption spectrophotometer (Varian 55B). As a reference, a NIST 2782-industrial sludge certified sample was used. The compost samples were dried at 60 °C for three days and then milled. Digestion of samples was performed according to the EPA 3050 method, and 1.0 g of the milled compost was placed in a tube. Then, 5 ml of HNO<sub>3</sub> was added for 15 minutes at 95 °C. Subsequently, 10 ml of HNO<sub>3</sub> was added for 2 hours, removed from the block and combined with 8 ml of H<sub>2</sub>O<sub>2</sub>. After the end of effervescence, the solution was increased to 50 ml and filtered to quantify the elements. The value obtained was an average of the triplicate analyses (EPA 1996).

### Characterization of compost by <sup>13</sup>C NMR CP/MAS

A <sup>13</sup>C NMR CP/MAS analysis was performed at the Analytical Center of PPGQ/UFRRJ. The <sup>13</sup>C-NMR CP MAS spectroscopic analysis was performed in a Bruker AVANCE II NMR instrument at 400 MHz, equipped with a 4 mm Narrow MAS probe and operating in resonance sequence from <sup>13</sup>C to 100.163 MHz. To obtain the spectra, the previously lyophilized compost samples

were placed in a rotor (sample holder) of zirconium dioxide (ZrO<sub>2</sub>) with Kel-F caps with a rotation frequency of 8±1 kHz. The spectra were obtained by collecting 3000 data points for the same number of scans at an acquisition time of 34 ms and with a 5s recycle delay. The contact time for the 1H ramp sequence was 2 ms. Spectral collection and elaboration were performed using Bruker Topspin 2.1 software. Free induction decays were transformed by applying a zero-filling equal to 4k, and then an adjustment by 70 Hz line broadening was carried out. The regions were then integrated to obtain the relative number of structures as a function of total area. The spectra were analyzed using ACD/Labs software v.12.01 (Freeware Academic Edition). The spectra were then divided into different regions according to the carbon type using the PeakPicking tool. The regions were integrated over the total area using the “integration manual” tool.

The integrated regions were as follows: nonfunctionalized alkyl carbons (C<sub>Alkyl</sub>-H,R), 0–45 ppm; C-methoxy and N-alkyl-C (C<sub>Alkyl</sub>-O,N), 45–60 ppm; O-alkyl (C<sub>Alkyl</sub>-O), 60–90 ppm; di-O-alkyl C (anomeric carbons) (C<sub>Alkyl</sub>-di-O), 90–110 ppm; aromatic carbons (C<sub>Aromatic</sub>-H,R), 110–140 ppm; O, N – aromatic C (C<sub>Aromatic</sub>-O, N), 140–160 ppm; carboxyl C (C<sub>COO</sub>-H,R), 160–185 ppm; and carbonyl C (C<sub>C=O</sub>), 185–230 ppm. The aromaticity index was calculated according to (C<sub>Aromatic</sub>-H,R) + (C<sub>Aromatic</sub>-O,N) \* 100/total area, and the aliphaticity index was calculated as 100- aromaticity (%). The hydrophobicity index (HB/HI) was calculated as follows: (0 ppm-46 ppm) + (110 ppm-156 ppm)/(46 ppm-110 ppm) + (156 ppm-186 ppm) (Keeler et al. 2006; Song et al. 2008).

### Chemometric analysis applied to <sup>13</sup>C NMR CP/MAS spectral data

Chemometric analysis was performed by Unscrambler Software (version 10.4) (Camo Software AS, Nedre Vollgate 8, Oslo, Norway). Loading of <sup>13</sup>C-NMR CP MAS spectra generated an initial matrix of samples: variable (12 x 4090). The <sup>13</sup>C-NMR CP MAS matrix was subjected to line plotting for visual inspection and later the matrix was transformed through normalization, smoothing and baseline correction. To obtain the average spectra, the tool used was the descriptive statistics and then the matrix accessed for plotting in the line form of average obtained in the analysis. The matrix corresponding to the average spectra for each type of

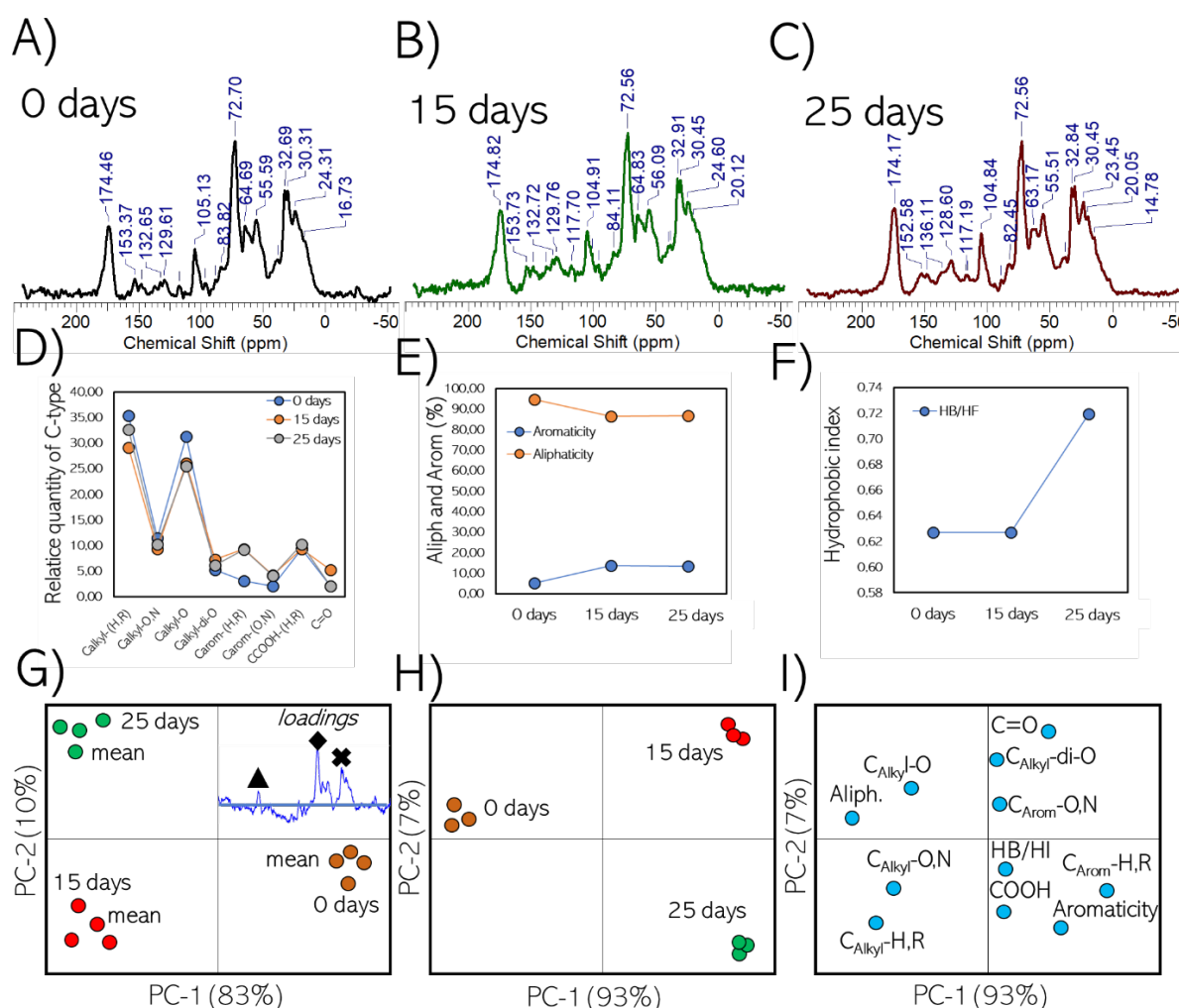
compost was then added to the original matrix for multivariate analysis of the data. Principal component analyses (PCA) were performed from the standardized matrix. The model adopted for PCA used a total of seven components, automatic identification of outliers. A NIPALS algorithm and a cross validation were used. The scores and loadings were presented in different graphs and the latter were plotted as a line for the visualization of weights in the form of a spectral pattern.

## Results and discussion

### Characterization by $^{13}\text{C}$ NMR CP/MAS at different composting times

Structural characterization of the compost allowed for the identification of the modifications that occurred

during composting for all present structures (Fig. 2). Modification that occurred in terms of the quantity of components that conformed to the compost was identified both at 15 and 25 days of composting and compared to the beginning of the process (0 days). Characterization by  $^{13}\text{C}$  NMR CP/MAS showed, in the three moments of composting, an abundant presence of aliphatic carbons corresponding to waxes, cutins, cutan, etc. ( $\text{C}_{\text{Alkyl-H,R}}$ : 0-45 ppm). It was also possible to identify methoxy carbons ( $\text{RO-CH}_3$ ) and  $\text{C}_\alpha$  ( $-\text{CO-CHR-NH}$ ) of polypeptides ( $\text{C}_{\text{Alkyl-O,N}}$ : 45-60 ppm). The peaks present between 60 and 90 ppm highlighted the presence of  $-\text{C-OH}$  belonging to cellulose and hemicellulose, as well as ligninic fragments ( $\text{C}_{\text{Alkyl-O}}$ ), and peaks corresponding to the ( $\text{C}_{\text{Alkyl-di-O}}$ ) of anomeric carbon carbohydrates and  $\text{C}_2$  of the guaiacil and syringil structures (90-110 ppm). Peaks appeared



**Fig. 2**  $^{13}\text{C}$  NMR CP/MAS average spectra obtained from descriptive statistics for the spectral replicates ( $n = 3$ ) of the compost in three composting moments (A: 0 days, B: 15 days, C: 25 days)

Variation in structure relative quantity (D), aromaticity and aliphaticity (E) and hydrophobicity index (F). G) PCA, scores and loadings (x: non functionalized aliphatic carbon, ♦: functionalized aliphatic carbon and ▲: carboxyl carbon) obtained from pure spectra. H) and I) Scores and loadings respectively obtained in PCA carryout from the relative number of structures.

between 110 and 140 ppm corresponding to aromatic structures ( $C_{\text{Aromatic}}\text{-H,R}$ ) of a ligninic nature as well as between 140 and 160 ppm corresponding to functionalized carbon peaks ( $C_{\text{Aromatic}}\text{-O,N}$ ). Between 160 and 180 ppm, there was an intense peak corresponding to ester and carboxylic carbons, and those between 180 and 230 ppm corresponded to the carbonyl carbons ( $C=O$ ) of aldehydes and ketones (Fig. 2a, 2b and 2c).

As a result of fast transformation during the composting process, the contents of the components present in the compost have been intensely modified, generating a final compost with different chemical properties (Fig. 2d, 2e and 2f). In composting, the aliphatic structures underwent the most intense transformation and therefore decreased, especially the structures corresponding to fatty acids, waxes and carbohydrates; therefore, the aromatic structures were more predominant and preserved at the end of composting (Fig. 2d). This change in structure quantity caused the compost obtained at the end of the composting process to be more aromatic, less aliphatic (Fig. 2e) and therefore more hydrophobic (Fig. 2f).

The chemometric analyses confirmed the information about the transformations of different molecules at different composting times (Fig. 2g, 2h and 2i). PCA of the pure spectra (93% of variance explained) showed a clear separation and grouping of spectra at the three different moments during composting; all spectra corresponding to the samples collected on day 0 (beginning of composting) grouped at positive values of PC-1 (83% of the explained variance), and, oppositely, all spectra corresponding to samples collected on days 15 and 25 grouped along the negative axis of PC-1 (Fig. 2g). Loadings expressed as a spectral pattern clearly showed that aliphatic carbons corresponding to lipids and carbohydrates ( $C_{\text{Alkyl}}\text{-H,R}$ ;  $C_{\text{Alkyl}}\text{-O}$  and  $\text{COOH}$ ) were present in the starting material and decreased intensely after composting times of 15 and 25 days. The loading plots also showed that aromatic structures were characteristic of the compounds formed at 15 and 25 days, and this type of structure was predominant at the end of the composting process (Fig. 2g).

The composting process is dramatically heterogeneous, mainly due to the high variability in quantity and quality of raw materials. In this sense and as shown in this study, spectroscopic monitoring allows to understand the mechanism of molecular transformation. The studies by Huang et al. (2017) show that the composting decomposition phase is preceded by a

mineralization of O-Alkyl-C type structures and subsequent preservation of C-aromatic structures. The FTIR and  $^{13}\text{C}$  NMR CP/MAS techniques applied to the composting of pig slurry also showed that more labile structures decompose at the beginning of the process (Martín-Mata et al. 2016).

Our results agree with those published in the literature, where an initial transformation of aliphatic and carbohydrate structures is reported in the composting process, therefore preserving more aromatic, nonpolar and hydrophobic structures with the advancement of the composting process (Baddi et al. 2004; de Souza et al. 2019). The studies carried out by Tomati et al. (2001) also showed that during the composting process, there is a preservation of aromatic structures and high modification of polysaccharide fragments.

## Chemical and physical characterization of the compost

### Temperature

The temperature variation in the composting process is shown in Fig. 3a. The average room temperature was 27.1 °C, while in the compost pile, the temperatures were thermophilic throughout the process, with a peak value at 76 °C by the sixth turning (day 15), followed by a constant decline. During the last turning, the average temperature was 59.9 °C, indicating permanence in the thermophilic phase. After 15 days, the compost showed an average temperature of 36 °C, characterizing a biostabilized compost (Kiehl 2002).

The decrease in temperature matches the structural information obtained for the compost by  $^{13}\text{C}$  NMR CP/MAS characterization, where lipids and carbohydrates may have been initially metabolized in the composting process (Fig. 1d). High temperatures during composting are due to the intense microbial activity inside the pile, which may have benefited from the carrying out of turning and irrigation, providing aeration and humidity for microorganism reproduction and activity. Authors such as Tognetti et al. (2007) also found thermophilic temperatures during most of the food waste composting process. Barrena et al. (2006) found thermophilic temperatures even after compost stabilization in the maturation phase and associated them with the low thermal conductivity of large windrows and with the similarity of the surface layers of the windrows with the ambient temperatures.

## pH values

The pH variation in the composting process is shown in Fig. 3b. The pH of the initial composting process was acidic due to the organic acids produced by the decomposition of easily degradable carbon sources (Leal et al. 2007). The pH increased until it reached its peak on the fifteenth day and then decreased slightly to a pH value of 8.37 at 25 days, coinciding with the moment of greatest decrease in the relative quantity of aliphatic nitrogen structures corresponding to peptides ( $C_{\text{Alkyl-O,N}}$ ) (Fig. 1d). It has been reported that an increase in pH value during the composting process may result from protein degradation, generating alkaline reaction compounds that promote the consumption of protons and the release of ammonia (Jahnel et al. 2000; Leal et al. 2007; Kiehl 2002).

The change in pH value during composting is impacted by the raw materials used, management type, degradation rate of organic molecules and the amount of ammoniacal nitrogen produced or volatilized. High temperatures throughout the process promote an increase in nitrogen mineralization, causing an increase in  $\text{NH}_4^+$  concentration and, consequently, an increase in pH value (Sánchez-Monedero et al. 2001; Tognetti et al. 2007).

## Electrical conductivity values and variation of density on a dry basis value

Electrical conductivity is a phytotoxicity indicator for the quality of compost. Degradation of organic materials increases the electrical conductivity due to mineralization of salts, indicating nutrient availability in their soluble form. Kiehl (2002) recommends that the electrical conductivity of the compost does not exceed  $4.0 \text{ mS/cm}^{-1}$ ; however, values higher than  $2.0 \text{ mS/cm}^{-1}$  already indicate salinity. It is important to note that the leaching of salts caused by rain or irrigation can reduce electrical conductivity over time (Abreu et al. 2000). In the present study, the obtained compost did not show high values of electrical conductivity during the composting process, with a value of  $2.11 \text{ mS/cm}^{-1}$  on the twenty-fifth day (Fig. 3c).

The variation in density on a dry basis in the composting process is shown in Fig. 3d. The density increased during the composting process, probably resulting from the increase in the degree of humification and polymerization of organic substances (Leal et al. 2007; Harrison 2008).

## Variation in potential carbon dioxide emission

The Fig. 3e shows the potential emission of carbon dioxide ( $\text{CO}_2$ ) during the composting process. The  $\text{CO}_2$  emission resulting from microbial activity assumes that the degradation of more labile substances causes a high release of energy in the form of heat and therefore  $\text{CO}_2$  emission, which explains the greater increase in emission observed on the eleventh (day 11) of the composting process (Jahnel et al. 2000). These results were clearly confirmed by  $^{13}\text{C}$  NMR CP/MAS characterization, which showed more nonpolar and hydrophobic structure preservation and predominant degradation of lipids and carbohydrates. The decrease in emissions throughout the composting process was due to the depletion of fast decomposing materials, preservation of the most resistant materials or those with a relatively high recalcitrance degree. In this phase, there is less organic matter degradation and less  $\text{CO}_2$  emission, which reveals the evolution of the compost to the final stabilization. Loadings on the PCA plot of the  $^{13}\text{C}$  NMR CP/MAS data reaffirm that the  $C_{\text{Aromatic-H,R}}$  and  $C_{\text{Aromatic-H,R}}$  structures predominated the composition of the compost at 15 and 30 days, especially when compared with that of the compost at 0 days (Fig. 1g).

Materials with  $\text{CO}_2$  emissions less than  $2 \text{ mg/g}^{-1}/\text{ms}/\text{day}^{-1}$  are considered very stable, those with emissions between 4 and 8 are stable, and those with emissions above  $8 \text{ mg/g}^{-1}/\text{ms}/\text{day}^{-1}$  are considered immature. Materials with  $\text{CO}_2$  emissions less than  $4.0 \text{ mg/g}^{-1}/\text{ms}/\text{day}^{-1}$  are considered stable. In this study, the  $\text{CO}_2$  emission reached a value of  $4.58 \text{ mg/g}^{-1}/\text{ms}/\text{day}^{-1}$  on day twenty-five (25) of composting, indicating a high level of stabilization in a short period of time (Bernal et al. 2009) (Fig. 3e).

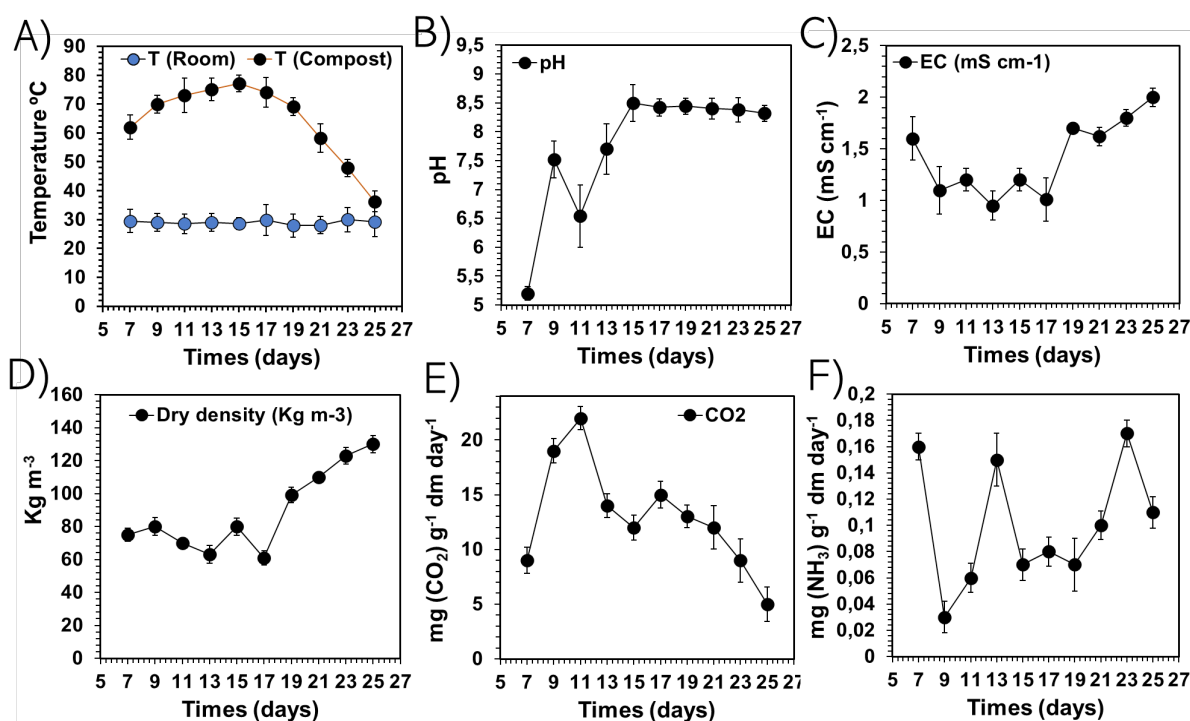
## Variation in the potential ammonia ( $\text{NH}_3$ ) emission

The potential ammonia emission values ( $\text{NH}_3$ ) in composting are shown in Fig. 3f. It is possible to observe high  $\text{NH}_3$  emissions on the seventh day of composting, which corresponded to the second turning of the compost pile. The increase in  $\text{NH}_3$  emissions is usually associated with high temperatures and protein degradation, which favors organic matter mineralization, resulting in ammonia volatilization and, consequently, a pH increase (Tuomela et al. 2000; Beck-Friis et al. 2001; Sánchez-Monedero et al. 2001; Fukumoto et al. 2003; Yang et al. 2008).

In this study, after nine days, there was an increase in  $\text{NH}_3$  emissions according to the increase in pH values and temperature of compost. In an experiment regarding the composting of swine manure was concluded that there is a direct relationship between the turning times of piles and  $\text{NH}_3$  emissions, which may cause N content reduction in compost. In this way, it was possible to understand that the behavior of  $\text{NH}_3$  emissions in this study had a relationship with high temperatures reached by the compost pile and the intense frequency of overturning during the entire process (Brito et al. 2008).

The gas emission is often a problematic characteristic, as composting becomes undesirable in plac-

es where it is carried out because of odor and insects that the decomposition of organic materials attracts. As obtained in this study, proper handling of compost piles is an important step in reducing gas emissions (Zhu-Barker et al. 2017). To solve these problems, alternatives such as the inclusion of earthworms and red mud to compost can reduce the  $\text{CO}_2$ ,  $\text{CH}_4$  and  $\text{N}_2\text{O}$  emission (Barthod et al. 2018). The biochar incorporation to the composting process also reduces gas emissions, especially when added to cow manure compost (Thomas et al. 2019).



**Fig. 3** Chemical and physical parameters quantified during composting process. A) Temperature, B) pH, C) Electrical conductivity, D) Density, E)  $\text{CO}_2$  emission and F)  $\text{NH}_3$  emission

### Variation of metallic and nonmetallic element contents during composting

Table 1 shows the contents of metals and nonmetals present in the compost after 15 and 25 days of composting. The contents of C, N and O decreased from 15 to 30 days of composting, while the H content increased. The OM content also decreased until the final stage of composting along with the C/N ratio. The  $\text{CO}_2$  losses, as well as the intense decrease in  $\text{NH}_3$ , justified the decrease in the amount of carbon and demonstrat-

ed the influence of mineralization on the final contents of these elements in compost. Evidently, the losses of these elements, as well as the high temperature peak after 15 days of composting, also justified the decrease in OM content until reaching 25 days of composting. The  $^{13}\text{C}$  NMR CP/MAS spectroscopy applied for compost characterization and chemometric analysis allowed us to observe that the losses of OM were due to the degradation of aliphatic fragments, mainly lipids, carbohydrates and peptides ( $\text{C}_{\text{Aliphatic}}\text{-H}$ , R;  $\text{C}_{\text{Aliphatic}}\text{-O}$ ;  $\text{C}_{\text{Aliphatic}}\text{-O,N}$ ;  $\text{C}_{\text{Aliphatic}}\text{-di-O}$ ) (Fig. 3).



**Table 1** Nonmetal and metal elements content present in the compost at 15 and 25 days of composting process

Elements	Units	15 days	Error	25 days	Error
C		40.55	±1.23	30.01	±1.11
N		5.21	±0.54	5.01	±0.61
H	%	13.22	±1.55	25.71	±1.31
S		0.31	±0.001	0.30	±0.001
O		40.71	±1.65	38.97	±1.44
OM		68.93	±5.62	51.01	±4.1
C/N	Not unit	7.78	±1.44	5.99	±1.33
Ca <sup>2+</sup>		422.30	±11.32	488.60	±10.98
Mg <sup>2+</sup>		80.99	±2.77	87.27	±2.88
Cu <sup>2+</sup>		0.21	±0.011	0.26	±0.014
Mn <sup>2+</sup>		3.54	±0.06	4.22	±0.002
Fe <sup>3+</sup>	mg/kg <sup>-1</sup>	70.65	±3.44	65.40	±2.68
Zn <sup>2+</sup>		1.02	±0.001	1.92	±0.001
Cd <sup>2+</sup>		0.01	±0.0001	0.01	±0.0001
Ni <sup>2+</sup>		0.02	±0.0001	0.05	±0.0001
Al <sup>3+</sup>		18.25	±3.8	25.80	±3.65
Pb <sup>2+</sup>		0.19	±0.002	0.18	±0.001

During the composting process, there was an increase in the concentration of the metals Ca<sup>2+</sup>, Mg<sup>2+</sup>, Mn<sup>2+</sup>, Cu<sup>2+</sup>, Ni<sup>2+</sup>, Zn<sup>2+</sup> and Al<sup>3+</sup>, which allowed an increase in the content of these elements in the compost. The Fe and Pb metals decreased their content until the end of composting. The elemental form of aluminum (Al<sup>3+</sup>) was found in high quantities compared to those of the other metals. However, it was possible that Al<sup>3+</sup> formed complexes of the inner sphere with functional groups of humic substances, and, consequently, the metal may have had low bioavailability. In addition, at pH values above 7.0, this element is found in the chemical formula Al(OH)<sub>4</sub><sup>-</sup> and is not toxic. Other potentially toxic elements, such as Cd<sup>2+</sup>, Pb<sup>2+</sup> and Ni<sup>2+</sup>, were found in low quantities and therefore do not pose risks to human health.

The presence of HM in the final composting product is a parameter to assess the quality (Silva et al. 2019). This is because the HM present in the compost can be leached to the soil (Fang et al. 2017), however, in low quantities such as those obtained in this study, the compost can be used as a soil improver without increasing the speciation of metals in the soil (Fang et al. 2016).

De Souza et al. (2019) showed that during the composting process, the transformation of organic compounds influences the metal dynamics of compost, where elements such as Cu<sup>2+</sup> and Zn<sup>2+</sup> can accumulate due to the interaction with the most preserved frag-

ments, while Fe and Pb can be leached and decrease in concentration by soluble complex formation. He et al. (2016) also showed that during the vermicomposting process, the tendency of an increase in heavy metal concentration occurred as a result of a decrease in their mobility.

### Environmental and agronomy importance of hot composting Berkeley methods

Several alternatives to optimize composting process have been the subject of research in recent years. The establishment of adequate composting times for obtaining of the final compost has been one of the most discussed. Monitoring and quantifying volatile compounds as well as determining the presence and diversity of fungi community has been some of the alternatives (Zhu et al. 2016; Gu et al. 2017). However, in the present study it was proven that after 25 days of composting, a compost produced from food waste, can be ready and stabilized applying the HCBM method. The studies carried out by de Souza et al. (2019) showed that after 30 days of composting, it is possible to obtain a stabilized final product, but without applying the HCBM method.

When the HCBM method is applied to food waste, there is a transformation mechanism initiated by the decomposition of more labile compounds such as fatty acids, peptides and carbohydrates fragments. This

means that an enrichment of raw material with these compounds can optimize the composting time, providing a reduction in up to 25 days. As is well known in the literature, stable compounds have greater aromaticity (Jindo et al. 2019). From an agronomic and environmental point of view, a compost obtained in these conditions and in reduced times has advantages, since a reduction of the waste and residues of this class in the cities can lead for a greater volume of compost for urban agriculture and even a reduction in the effects of odors and insects at production stations.

## Conclusion

The application of the hot composting Berkeley method (HCBM) to food waste allowed for us to obtain a stabilized compost in ecological production conditions. Composting occurred through an initial transformation in the first 15 days of polar and labile compounds belonging to lipids and carbohydrates ( $C_{\text{Alkyl}}\text{-H,R}$ ;  $C_{\text{Alkyl}}\text{-O}$ ;  $C_{\text{Alkyl}}\text{-O,N}$ ), with an increase in temperature and pH. After 15 days of composting, the  $\text{CO}_2$  and  $\text{NH}_3$  emissions were reduced, indicating a decrease in the transformation of the more labile compounds and the preservation of more recalcitrant molecules with aromatic and nonpolar characteristics ( $C_{\text{Aromatic}}\text{-H,R}$ ;  $C_{\text{Aromatic}}\text{-O,N}$ ). The final compost (25 days) produced by the HCBM method were hydrophobic and aromatic and therefore stabilized and rich in mineral elements important for plant nutrition ( $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Mn}^{2+}$ , and  $\text{Zn}^{2+}$ ).

**Acknowledgment** A.C. García (SisFAPERJ: 2012028010) thanks the Fundação de Amparo à Pesquisa do Estado do Rio de Janeiro (FAPERJ) for supporting the research through Edital No. 04/2018- JCNE. A.C. García also thanks the Brazilian National Research Council (CNPq) for the research grant fellowship (No. 306867/2018-4, CNPq-No. 09/2018, Bolsa de pesquisa PQ-2). This study was financed in part by the Coordenação de Aperfeiçoamento do Pessoal de Nível Superior-Brasil (CAPES), Finance Code 001.

## Compliance with ethical standards

**Conflict of interest** The authors declare that there are no conflicts of interest associated with this study.

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