PHYSICOCHEMICAL MATURITY PARAMETERS IN THE COFFEE PULP-COW MANURE VERMICOMPOSTED MIXTURE

PARÁMETROS FISICOQUÍMICOS DE MADUREZ EN LA MEZCLA VERMICOMPOSTEADA PULPA DE CAFÉ-ESTIÉRCOL VACUNO

Samia Berenice Flores-Solórzano¹, David Espinosa-Victoria²* y José Antonio Serrano-Casillas³*

¹Universidad Veracruzana, Facultad de Ciencias Químicas, Xalapa, Veracruz, México. ²Colegio de Postgraduados, Programa de Edafología, Montecillo, Texcoco, Estado de México, México. ³Universidad Veracruzana, Facultad de Ciencias Agrícolas, Xalapa, Veracruz, México.

*Corresponding author (nequodah@gmail.com)

SUMMARY

Determining the degree of maturity in organic matter is an aspect of interest in the composting process. The objective of this research was to measure the mineralization rate of different mixtures of coffee pulp with bovine manure and to evaluate maturity parameters related to humification, to be used in the characterization of the quality of vermicomposted mixture of coffee pulp:bovine manure. Treatments established for the pre-composting and vermicomposting processes were: 100 % coffee pulp (control), 50 % coffee pulp with 50 % bovine manure (50 CP:50 CM) and 25 % coffee pulp with 75 % bovine manure (25 CP:75 CM). CO₂ emission of the treatments, measured through the microbial respiration in the composts, was in accordance with the humification process, with a greater rate in the more humified treatments. Values of the polymerization index (PI), humic acids percentage (HAP), humification rate (HR) and humification index (HI) of the 50 CP:50 CM and 25 PC:75 CM treatments were associated with the humification parameters for maturity of compost of different origin as reported in literature. The maturity parameters sensitive to the coffee pulp with bovine manure vermicompost were PI, HAP, pH and cation exchange capacity, whose content defined the quality of the final product. Results showed that the 50 CP:50 CM mixture improved the humification process.

Index words: bovine manure, coffee residues, humification, vermicompost quality.

INTRODUCTION

Coffee is one of the main export crops worldwide; according to ICO (2020), it is produced particularly in developing countries and is consumed predominantly in industrialized ones. During the production process of coffee, a large amount of waste is generated. Generally, the waste is dumped into water bodies or stored near the wet processing area, which produces bad odors, pollution of the water table and eutrophication of rivers and lagoons (Cervantes et al., 2015).

Environmental pollution due to coffee by-products can be reduced by composting or vermicompost, as coffee pulp is a good source for growing earthworms (Aranda and Barois, 2000). This process consists of the transformation of raw organic matter into useful products through aerobic fermentation (Iglesias, 2008). The degradation time of the waste decreases with the activity of earthworms; in addition, vermicomposting is capable of reducing pathogenic bacteria and promoting production of the humic substances (Atiyeh et al., 2000). Vermicompost is already used by some coffee producers in Mexico (Aranda et al., 1999) but it is important to extend its use to improve soil structure and environmental impact.

Another important organic waste generated in Mexico is cow manure (CM), around 34,037,141 head of cattle...
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are raised in Mexico (INEGI, 2019), and only 1.85 % of its manure is managed (INEGI, 2017). untreated animal
waste causes air pollution and contributes to the excess
of bacteria and nitrates in soil and groundwater (Coulibaly
et al., 2010). Vermicomposting has been proposed by
numerous authors (Lazcano et al., 2008; Olivares-Campos
et al., 2012; Sierra et al., 2013) to stabilize manure sources
to reduce composting time and improve their agronomic
potential.

The determination of the maturity degree of organic
matter in composts ensures the stability and safety of the
product. There are physicochemical and microbial parameters to determine the maturity of a compost (SEC,
2008), which are associated with the degradation process.
The waste, which contains a high percentage of soluble
organic carbon, leads to a high CO₂ production derived
from microbial activity, this activity can promote the
degradation of organic matter and its humification (Defriri
et al., 2005). The CO₂ production rate is used to measure
mineralization in composts (Rivero and Hernández, 2001).

Humic substances are complex and heterogeneous
mixtures of functional groups such as alcohols, carboxylic
acids, ketones, quinones, phenolics, among others; they are
divided into fulvic acids, humic acids and humins, formed
from chemical reactions in the biodegradation of plants
and microbial debris in a process called humification (IHSS,
2007). First, fulvic acids (FA) are made, in this phase the
aliphatic chains predominate, then the aromatic nuclei
increase to constitute humic acids. (Moreno and Moral,
2008). Substances continue condensing into humins over
the years (Stevenson, 1994). Humic acids have a higher
content of C, H, N, molecular weight and polymerization
degree, compared to fulvic acids (Steelink, 1985).

A similar process occurs in composting, a gradual
increase in the fraction of humic acids and a parallel
decrease in fulvic acids (Chefetz et al., 1996). The
substances produced in the composting process have
a nature and behavior like those produced in the soil
environment; however, they should be defined as humic-
like substances (Senesi et al., 1996). Several publications
suggest the inclusion of humic substances within the
quality parameters of the compost (Acosta et al., 2006;
Azim et al., 2018; Pierre et al., 2009). Roletto et al. (1985)
proposed the following indices to evaluate the compost
humification: humification ratio (HR) ≥ 7.0, humification
index (HI) ≥ 3.5, humic acids percentage (HAP) ≥ 50 and
polymerization index (PI) ≥ 1.0.

In the present study, the CO₂ emission rate of the
coffee pulp with cow manure in different proportions
was evaluated, along with maturity parameters related
to humification to be used in the characterization of the
coffee pulp-cow manure vermicompost.

MATERIALS AND METHODS

Origin of coffee pulp and cow manure

Coffee pulp (CP) from the coffee zone of Huatusco de
Chicuellar, Veracruz, Mexico, located at 19° 13' N latitude
and 96° 58' West longitude was used. It was a proportional
mixture of different varieties of coffee plants: Colombia,
Costa Rica, Oro Azteca, Catimor, Caturra, Garnica, Borbón
and San Román. This coffee pulp was the result of the wet
process system to extract the coffee bean. Cow manure
(CM) came from cattle of Colegio de Postgraduados,
Campus Montecillo, Texcoco, State of Mexico. The cattle
were fed on a mixture of oats, alfalfa and 17 % Clayton
Malta® commercial feed. The parameters measured in
the raw waste were pH, total N, % moisture, C/N ratio and
organic matter content (Table 1).

Treatments

The established treatments were: 100 % coffee pulp
(control), 50 % coffee pulp with 50 % cow manure (50
CP50 CM) and 25 % coffee pulp with 75 % cow manure
(25 CP75 CM) for pre-composting and vermicomposting
processes. To obtain these proportions, 3 kg of coffee pulp
were weighed for the control, 1.5 kg of coffee pulp with 1.5
kg of cow manure for the 50 CP50 CM treatment, and 0.75
kg of coffee pulp with 2.25 kg of cow manure for the 25
CP75 CM treatment. In all cases the weighing was carried
out on a wet weight basis.

Pre-composting was carried out for 26 days in a
greenhouse using four replicates per treatment, placing
3 kg of mixture in 30 × 50 × 20 cm plastic containers
used as composters. Vermicomposting was carried out
in a controlled environment chamber (Sherer®, Marshall,
Michigan, USA) at 30 °C. Forty grams of Californian red
worm (Eisenia fetida) were used for each treatment after
the pre-composting phase.

A randomized complete block experimental design with
four replicates per treatment was used. To determine
significant differences between the treatments, analysis
of variance (ANOVA) and Tukey test were applied to the
data using the SAS statistical package Version 9.1 (SAS
Institute, 2004).

Sampling

Compost samples were taken for analysis at the end of
the pre-composting period. Vermicompost samples were
analyzed 60 days after inoculation of the earthworms. The
samples were dried at room temperature, then ground and sieved on 2 mm mesh as indicated in the NMX-FF-109-SCFI-2008 standard for earthworm humus test methods (SEC, 2008).

**Evaluated variables**

The pH was determined by the potentiometric method, using a digital potentiometer (Conductronic PC45®, Mexico) at the 1:5 ratio. Organic matter (OM) and total organic carbon (C_{tot}) were determined using the loss on ignition method at 400 °C (Dabadie et al., 2018). The cation exchange capacity (CEC) was determined through the ammonium distillation method (Rodríguez and Rodríguez, 2002). Total nitrogen was determined by the micro-Kjeldhal method according to NOM-021-RECNAT-2000 (SEMARNAT, 2002).

Microbial respiration (CO₂ emission) of the coffee pulp and its different mixtures with cow manure was evaluated according to Anderson (1982). Fifty grams of mixture were weighed per treatment and placed into a closed glass flask. A vial with NaOH was placed inside it for all treatments, including the control. Every 24 h the contents of the vial were precipitated with BaCl₂ and titration was carried out with H₂SO₄. The procedure was carried out for 31 days, and the CO₂ emission was expressed in mg of C-CO₂ per gram of compost. To obtain the emission rate, the accumulated CO₂ was divided by the total time (31 days).

The extractable carbon (AH + AF) was obtained through the Kononova-Belchikova method (Cabrera et al., 2002) and measured with Walkley and Black (1934) procedure. The extractant solution was Na₄P₂O₇ ⋅ 10H₂O, and 5 g of compost or vermicompost were used. To separate the humic substances, the extract was acidified to pH 2.0 with H₂SO₄, and its different mixtures with cow manure was evaluated (Figure 1). According to Defrieri et al. (2005), the high production of CO₂ is related to an active development of the microbial metabolism, this suggests that the addition of cow manure promoted the microbial activity in the compost and improved the mineralization of the coffee pulp.

The CEC values obtained at the end of the pre-composting and vermicomposting processes (Table 2) were higher than the CEC value of the compost made only with coffee pulp. The high CEC values are explained by the increase of humic substances in the process (Mayhew, 2004) and the concentration of minerals as there is less organic matter.

### RESULTS AND DISCUSSION

#### Physicochemical characteristics and maturity parameters

The NMX-FF-109-SCFI-2008 standard establishes an optimum range of 20 to 50 % for the content of organic matter; however, values outside this range were obtained, which was also observed by Bernal et al. (1998) and Raviv et al. (2004) in a stable and mature compost. Bernal et al. (1998) confirmed a decrease in all parameters (C/N ratio, total N and pH) using a mixture of poultry manure, cotton waste and olive-mill wastewater, from 21.1 to 9.4 C/N ratio and from 81.5 to 64.8 % OM. Raviv et al. (2004) used cow manure with 81 % OM and 23 initial C/N, and obtained 70 % OM and a final C/N ratio of 15.6; both confirmed pH lower than 8. The OM percentage depends on the characteristics and operational conditions (Richard and Zimmerman, 1995), the manure sources showed greater organic matter content.

A decrease in the percentage of organic matter was observed when adding cow manure to the coffee pulp (Table 2), as reported by Pierre et al. (2009) when applying goat manure in a mixture with coffee waste. The decrease in OM is associated to the evolution of CO₂ (Benito et al., 2009). At the end of 31 days, the control and the 50 CP:50 CM and 25 PC:75 CM treatments accumulated 383.82, 610.79 and 587.47 mg C-CO₂ g⁻¹ of compost, respectively (Figure 1). According to Defrieri et al. (2005), the high production of CO₂ is related to an active development of the microbial metabolism, this suggests that the addition of cow manure promoted the microbial activity in the compost and improved the mineralization of the coffee pulp.

### Table 1. Initial physicochemical characteristics of coffee pulp and cow manure.

<table>
<thead>
<tr>
<th>Waste</th>
<th>pH</th>
<th>Moisture (%)</th>
<th>Total N (%)</th>
<th>C/N</th>
<th>OM (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP (control)</td>
<td>6.86</td>
<td>78.7</td>
<td>3.85</td>
<td>11.18</td>
<td>74.22</td>
</tr>
<tr>
<td>CM</td>
<td>6.42</td>
<td>79.35</td>
<td>1.67</td>
<td>20.59</td>
<td>59.27</td>
</tr>
</tbody>
</table>

Humic substances

Contrary to what was reported in literature, the extractable humic C (HA + FA) decreased after vermicomposting; however, extractable C increased as cow manure was added (Table 3), which agrees with the microbial respiration data (Figure 1), as the highest CO$_2$ values were present in the treatments with cow manure. The treatments in which cow manure was incorporated had a faster mineralization that led to the transformation of organic matter in a more compact time. The CO$_2$ emission rates of the treatments with cow manure (50 CP:50 CM: 18.95 ± 2.13, 25 CP:75 CM: 19.70 ± 2.23) were significantly higher than that of the control (12.38 ± 1.71) with only coffee pulp.

In the composting process, the amount of extractable C could depend on the type of substrate or manure used. The results of the present experiment differ from those obtained by Pierre et al. (2009), who did not find significant differences in terms of extractable C when using different proportions of goat manure with coffee pulp; also, Paredes et al. (2000) reported that the extractable C remained constant throughout the process when analyzing mixtures of residues of different origin, including manures; thus, it is not appropriate to consider it as a maturity parameter of organic matter.

Moreno and Moral (2008) and Chefetz et al. (1996) indicated that in the humification process there is a gradual increase in the fraction of humic acids and a parallel decrease in fulvic acids. In the present study, although an increase in humic acids was observed, a decrease in fulvic acids was only observed in the 50 CP:50 CM treatment (Table 3); probably the extraction method influenced the results.

Table 2. Physicochemical characteristics of the coffee pulp-cow manure treatments at the end of precomposting and 60 days after vermicomposting

<table>
<thead>
<tr>
<th>Treatment</th>
<th>pH</th>
<th>Organic matter (%)</th>
<th>C$_{TO}$ (%)</th>
<th>CEC (cmol kg$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Precomposting</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>7.36 b**</td>
<td>67.95 ± 0.79 a**</td>
<td>39.06 ± 0.45 a*</td>
<td>19.59 ± 1.86 b**</td>
</tr>
<tr>
<td>50 CP:50 CM</td>
<td>7.87 a**</td>
<td>65.17 ± 1.35 b**</td>
<td>37.45 ± 0.78 b*</td>
<td>21.11 ± 0.32 a**</td>
</tr>
<tr>
<td>25 CP:75 CM</td>
<td>8.05 a**</td>
<td>66.18 ± 1.32 ab**</td>
<td>38.03 ± 0.76 ab*</td>
<td>20.43 ± 1.44 a**</td>
</tr>
<tr>
<td>Vermicomposting</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>6.93 c**</td>
<td>68.72 ± 0.23 a**</td>
<td>39.49 ± 0.13 a*</td>
<td>31.55 ± 1.34 b**</td>
</tr>
<tr>
<td>50 CP:50 CM</td>
<td>7.63 b**</td>
<td>61.77 ± 1.04 b**</td>
<td>35.34 ± 0.59 b*</td>
<td>34.64 ± 1.03 a**</td>
</tr>
<tr>
<td>25 CP:75 CM</td>
<td>8.24 a**</td>
<td>61.49 ± 0.95 b**</td>
<td>35.49 ± 0.55 b*</td>
<td>36.48 ± 0.74 a**</td>
</tr>
</tbody>
</table>

Values with the same letter within columns and composting phases are not statistically different (Tukey, *P ≤ 0.05, **P ≤ 0.01). Precomposting and vermicomposting were analyzed separately. CP: coffee pulp, CM: cow manure, C$_{TO}$: total organic carbon, CEC: cation exchange capacity.

Table 3. Extractable humic carbon values and humic substances in the coffee pulp-cow manure treatments at the end of precomposting and 60 days after vermicomposting

<table>
<thead>
<tr>
<th>Treatment</th>
<th>EHC (%)</th>
<th>HA (%)</th>
<th>FA (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Precomposting</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>3.74 ± 0.09 b*</td>
<td>1.82 ± 0.05 b**</td>
<td>1.91 ± 0.05 b**</td>
</tr>
<tr>
<td>50 CP:50 CM</td>
<td>3.85 ± 0.13 b*</td>
<td>2.17 ± 0.05 a**</td>
<td>1.69 ± 0.08 b**</td>
</tr>
<tr>
<td>25 CP:75 CM</td>
<td>4.39 ± 0.42 a*</td>
<td>2.03 ± 0.18 ab**</td>
<td>2.36 ± 0.26 a**</td>
</tr>
<tr>
<td>Vermicomposting</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>3.49 ± 0.14 b*</td>
<td>1.72 ± 0.03 b**</td>
<td>1.77 ± 0.12 a**</td>
</tr>
<tr>
<td>50 CP:50 CM</td>
<td>3.62 ± 0.18 ab*</td>
<td>2.16 ± 0.07 a**</td>
<td>1.46 ± 0.11 b**</td>
</tr>
<tr>
<td>25 CP:75 CM</td>
<td>3.97 ± 0.33 a*</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Values with the same letter within columns and composting phases are not statistically different (Tukey, *P ≤ 0.05, **P ≤ 0.01). Precomposting and vermicomposting were analyzed separately. CP: coffee pulp, CM: cow manure, EHC: extractable humic carbon, HA: humic acids, FA: fulvic acids.
Humification Indices

The humification indices obtained after pre-composting and vermicomposting (Table 4) were compared with the parameters established by Roletto et al. (1985) to determine the composts maturity. The humification ratio (HR) indicates the percentage of extractable humic carbon contained in the total organic carbon. All the treatments surpassed the HR parameter. Roig et al. (1988), among others, indicated that the percentage of extractable humic carbon (C_{EX}/C_{TO}) cannot be considered as an adequate index of humification of organic matter, since it remains practically constant throughout the process in mixtures of waste of different origin (Paredes et al., 2000). The humification index is the total of humic acids contained in the organic carbon, which must be ≥ 3.5 for a stable compost. Besides, humic acids are used because they have a higher molecular weight and degree of polymerization (Steelink, 1985).

The humic acids percentage (HAP) represents the amount of humic acid contained in the extractable carbon. This parameter, like the others, will increase as the maturation time increases; in addition, this parameter allows to see the difference between the already humified material and the one that is still being transformed. As in compost there are no humins, the rest consists of fulvic acids. The 50 CP:50 CM treatment after precomposting reached more than 50 % of HAP (Table 4) as mentioned by Roletto et al. (1985). After vermicomposting, the treatments with manure exceeded this percentage. The best treatment was the one containing equal parts of coffee pulp and cow manure (P ≤ 0.05), the manure contributes positively to the humification of the coffee pulp vermicompost.

The polymerization index (PI) is the ratio between humic and fulvic acids. The best treatment was 50 CP:50 CM. The control treatment showed no humification after pre-composting. The treatment with the highest percentage of manure after pre-composting was the least humified; however, it exceeded the established value for the polymerization index (≥ 1) after vermicomposting. Table 5 shows the parameters with significant difference (P ≤ 0.01) between pre-composting and vermicomposting. These were compared with the parameters established in literature.

Manure treatments comply with the parameters proposed by Roletto et al. (1985); thus, these products attained maturity. The best treatment was 50 CP:50 CM; it was statistically different from the control and the 25 CP:75 CM was an appropriate treatment in terms of polymerization index and humic acids percentage. Likewise, the latter treatment presented a high degree of stability compared to the control and the 25 CP:75 CM treatment.

![Figure 1. Accumulative production of CO\textsubscript{2} in the composts. Treatments with the same letter are not statistically different (Tukey, *P ≤ 0.05, **P ≤ 0.01). Control (100 % coffee pulp), 50 CP:50 CM (50 % coffee pulp with 50 % cow manure) and 25 CP:75 CM (25 % coffee pulp with 75 % cow manure).](image-url)
The values established by the standard NMX-FF-109-SCFI-2008 are not sufficient as a quality guide for composts in terms of maturity; for instance, the CEC and OM % depend on the composition of the waste and the biodegradation process. It is necessary to have simultaneous mineralization and humification parameters to measure the stability of vermicompost and to compare the initial and final changes to ensure the transformation of residues. The humification parameters useful to evaluate the quality and the transformation process of the coffee pulp-cow manure vermicompost were the HAP and the PI, that showed significant changes before and after vermicomposting. The humification parameters showed the same trend as the CO₂ emission rate. The optimum mixture was 50 CP:50 CM, due to its higher degree of polymerization, neutral pH and greater cation exchange capacity and CO₂ emission rate. Compared to the control (coffee pulp 100%), the addition of manure contributes to the humification of the vermicompost, but the benefit is not proportional in the mixtures, since the 50 CP:50 CM treatment did not present significant differences compared to 25 CP:75 CM.

CONCLUSIONS

The values established by the standard NMX-FF-109-SCFI-2008 are not sufficient as a quality guide for composts in terms of maturity; for instance, the CEC and OM % depend on the composition of the waste and the biodegradation process. It is necessary to have simultaneous mineralization and humification parameters to measure the stability of vermicompost and to compare the initial and final changes to ensure the transformation of residues. The humification parameters useful to evaluate the quality and the transformation process of the coffee pulp-cow manure vermicompost were the HAP and the PI, that showed significant changes before and after vermicomposting. The humification parameters showed the same trend as the CO₂ emission rate. The optimum mixture was 50 CP:50 CM, due to its higher degree of polymerization, neutral pH and greater cation exchange capacity and CO₂ emission rate. Compared to the control (coffee pulp 100%), the addition of manure contributes to the humification of the vermicompost, but the benefit is not proportional in the mixtures, since the 50 CP:50 CM treatment did not present significant differences compared to 25 CP:75 CM.

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BIBLIOGRAPHY


