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

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

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SUMMARY

Determining the degree of maturity in organic matter is an aspect of interest in the composting process. The objective of this research was 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). CO_2 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.

RESUMEN

La determinación del grado de madurez de la materia orgánica constituye un aspecto de interés en el proceso de compostaje. El objetivo de esta investigación fue medir la tasa de mineralización de distintas mezclas de pulpa de café con estiércol vacuno y evaluar parámetros de madurez relacionados con la humificación, para ser utilizados en la caracterización de la calidad de la mezcla vermicomposteada pulpa de café:estiércol vacuno. Los tratamientos establecidos para los procesos de precomposteo y vermicomposteo fueron: 100 % pulpa de café (testigo), 50 % pulpa de café con 50 % estiércol vacuno (50 PC:50 EV) y 25 % pulpa de café con 75 % estiércol vacuno (25 PC:75 EV). La emisión de CO_2 de los tratamientos, medida por la respiración microbiana de las compostas estuvo en concordancia con los procesos de humificación, con una mayor tasa en los tratamientos más humificados. Los valores del índice de polimerización (IP), porcentaje de ácidos húmicos (PAH), tasa de humificación (TH) e índice de humificación (IH) de los tratamientos 50 PC:50 EB y 25 PC:75 EV estuvieron asociados con los parámetros de humificación para madurez de compostas de distinto origen reportados en la literatura. Los parámetros de madurez sensibles al proceso de vermicomposta de pulpa de café con estiércol vacuno fueron IP, PAH, pH y capacidad de intercambio catiónico, cuyo contenido definió la calidad del producto final. Los resultados mostraron que la mezcla 50 PC:50 EV mejoró el proceso de humificación.

Palabras clave: Calidad de vermicomposta, estiércol vacuno, humificación, residuos de café.

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 CO_2 production derived from microbial activity, this activity could promote the degradation of organic matter and its humification (Defrieri *et al*., 2005). The CO_2 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 CO_2 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 CP.50 CM) and 25 % coffee pulp with 75 % cow manure (25 CP.75 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 CP.50 CM treatment, and 0.75 kg of coffee pulp with 2.25 kg of cow manure for the 25 CP.75 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

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

Waste	pH	Moisture (%)	Total N (%)	C/N	OM (%)
CP (control)	6.86	78.7	3.85	11.18	74.22
CM	6.42	79.35	1.67	20.59	59.27

CP. coffee pulp, CM: cow manure, C/N: carbon nitrogen ratio, OM: organic matter.

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_{TO}) 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 (CO_2 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 $BaCl_2$ and titration was carried out with H_2SO_4. The procedure was carried out for 31 days, and the CO_2 emission was expressed in mg of C-CO_2 per gram of compost. To obtain the emission rate, the accumulated CO_2 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 $Na_2P_2O_7 \cdot 10H_2O$, and 5 g of compost or vermicompost were used. To separate the humic substances, the extract was acidified to pH 2.0 with H_2SO_4 for 24 h. Subsequently, the supernatant in the form of fulvic acids was decanted and the precipitate (humic acids) was measured by chemical titration with NaOH.

The humification indices used to evaluate the humification process in the vermicompost were: humification ratio (HR): $(C_{EX}/C_{TO}) \times 100$, humification index (HI): $(C_{HA}/C_{TO}) \times 100$, humic acids percentage (HAP): $(C_{HA}/C_{EX}) \times 100$ and polymerization index (PI): C_{HA}/C_{FA}, where C_{EX} corresponds to the extractable humic carbon (HA + FA), C_{TO} is the total organic carbon, C_{HA} is the carbon of humic acids and C_{FA} the carbon of fulvic acids (Roletto *et al*., 1985; Senesi, 1989).

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 CO_2 (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-CO_2 g^{-1} of compost, respectively (Figure 1). According to Defrieri *et al*. (2005), the high production of CO_2 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.

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.75CM: 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

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

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

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

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, EC: 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; iIn 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 ($P \leq 0.05$), 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 ($P \leq 0.01$) 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.

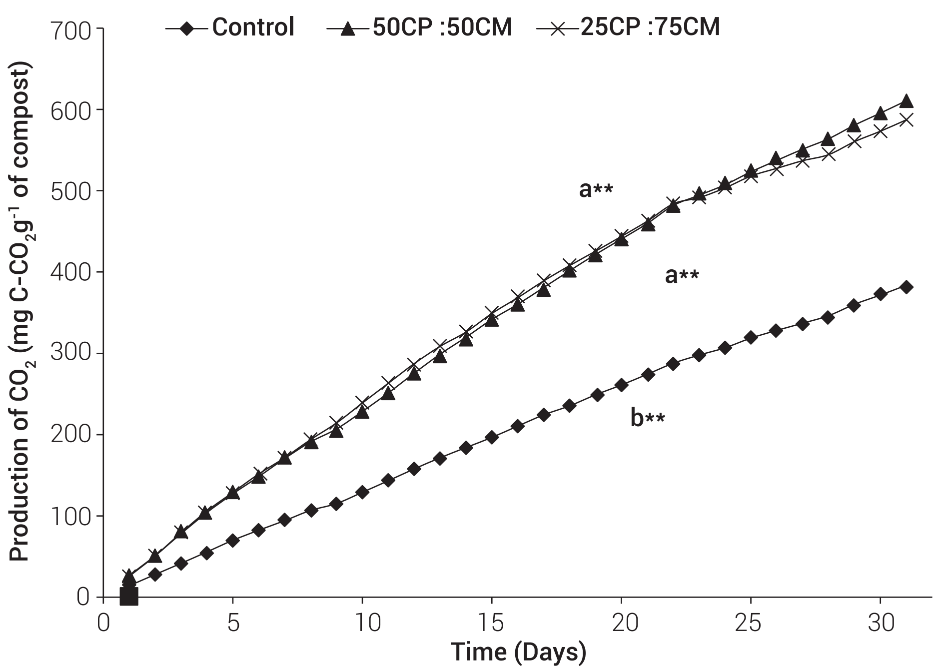

Figure 1. Accumulative production of CO_2 in the composts. Treatments with the same letter are not statistically different (Tukey, *$P \leq 0.05$, **$P \leq 0.01$). Control (100 % coffee pulp), 50 CP.50 CM (50 % coffee pulp with 50 % cow manure) and 25 CP.75 CM (25 % coffee pulp with 75 % cow manure).

Table 4. Humification indices of coffee pulp-cow manure treatments at the end of the precomposting and 60 days after vermicomposting.

Treatment	HR (%)	HI (%)	HAP (%)	PI (%)
		Precomposting		
Control	9.57 ± 0.15 b*	4.67 ± 0.09 b**	48.78 ± 0.7 b**	0.95 ± 0.03 b**
50 CP.50 CM	10.29 ± 0.5 ab*	5.78 ± 0.18 a**	56.25 ± 0.8 a**	1.29 ± 0.04 a**
25 CP.75 CM	11.55 ± 1.2 a*	5.34 ± 0.49 a**	46.25 ± 1.7 c**	0.86 ± 0.06 c**
		Vermicomposting		
Control	8.85 ± 0.3 b*	4.37 ± 0.09 b**	49.41± 1.38 c**	0.98 ± 0.05 c**
50 CP.50 CM	10.27 ± 0.68 a*	6.13 ± 0.18 a**	59.74 ± 1.05 a**	1.48 ± 0.07 a**
25 CP.75 CM	11.18 ± 0.94 a*	6.10 ± 0.49 a**	54.55 ± 2.99 b**	1.21 ± 0.14 b**

Values with the same letter within columns and composting phases are not statistically different (Tukey, $*P \leq 0.05$, $**P \leq 0.01$). Precomposting and vermicomposting were analyzed separately. CP. coffee pulp, CM: cow manure, HR: humification ratio, HI: humification index, HAP. humic acids percentage, PI: polymerization index.

Table 5. Maturity parameters sensitive to the vermicomposting process of coffee pulp-cow manure mixtures

	pH	CEC (cmol kg^{-1})	Organic matter (%)	HAP (%)	PI (%)
Parameter established	5.5 - 8.5	> 40	20 – 50	> 50	> 1.0
Control	6.93 c	31.55 a	68.72 a	49.41 c	0.98 c
50 CP.50 CM	7.63 b	34.64 b	61.77 b	59.74 a	1.48 a
25 CP.75 CM	8.24 a	36.48 b	61.49 b	54.55 b	1.21 b

Values with the same letter within columns are not statistically different (Tukey, $*P \leq 0.05$, $**P \leq 0.01$). CP. coffee pulp, CM: cow manure, CEC: cation exchange capacity, HAP. humic acids percentage, PI: polymerization index.

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 CO_2 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 CO_2 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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