

Coffee pulp compost: Chemical properties and distribution of humic substances^{1,2}

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ABSTRACT

The distribution and chemical properties of humic substances (humic acids and fulvic acids) from a twelve-month-old static aerated compost pile of coffee pulp were studied. The compost was separated into five physical granule fractions (500, 1000, 2000, 4000 and 8000 μm) in order to determine the effect of particle size on humic substances, on nutrient composition, and on distribution. The objective of this study was to understand the chemical properties and distribution of humic substances in mature coffee pulp compost. There were no significant differences across fractions for total N and available P_2O_5 ; these fractions averaged 4.9 and 0.66%, respectively. Potassium (K) increased as fraction size increased, from 3.01 to 3.94%. Readily available nitrogen, NO_3 and NH_4 , was 7.19% of the total N. Eighty-nine percent of the total K was exchangeable K ions, and these varied within compost fractions from 2.82 to 3.33%. Approximately 50% of the total Ca and Mg were exchangeable ions; Ca ions varied from 2.13 to 3.01%, and Mg ions varied from 0.22 to 0.26% among fractions. Sequential extraction of organic carbon showed higher extractable carbon by the extraction with 0.1 N NaOH for 24 h, followed by 0.1 N NaOH for 2 h, and water for 2 h. The optical densities E4/E6 ratio ranged between 6.6 and 8.5 for humic acids (HA) extracted with NaOH for 2 h. The diversity in humic substances (HS) and exchangeable ions among compost fractions indicates that nutrient release is regulated by the size of the granules. In conclusion, these data demonstrate that smaller size particles have greater humification rates, and that N and available P are similar across particle sizes, thus providing an excellent soil compost amendment.

Key words: humic acids, fulvic acids, organic carbon, sequential extraction

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RESUMEN

Composta de pulpa de café: Propiedades químicas y distribución de sustancias húmicas

Se estudiaron las propiedades químicas y sustancias húmicas (ácidos húmicos y ácidos fúlvicos) de composta de pulpa de café de 12 meses; esta fue compostada en una pila estática aireada. La composta se dividió en cinco fracciones físicas de gránulos (500, 1000, 2000, 4000 y 8000 μm) con el fin de determinar el efecto del tamaño de las partículas en la cantidad de sustancias húmicas, composición de nutrientes, y distribución. El objetivo de este estudio fue entender las propiedades químicas y distribución de sustancias húmicas en composta madura de pulpa de café. No hubo diferencias significativas entre las fracciones para N total y P_2O_5 disponible; el promedio fue de 4.9 y 0.66%, respectivamente. El potasio (K) aumentó de 3.01% a un 3.94% a medida que aumentó el tamaño de la fracción. Nitrógeno disponible, NO_3 y NH_4 , representó 7.19% del total de N. Un 89% del K total estaba en forma intercambiable y solo varió de 2.82 a 3.33%. Aproximadamente el 50% del total de Ca y Mg estaba en forma intercambiable. La concentración del ion de Ca varió de 2.13 a 3.01%, y la del el ion de Mg varió de 0.22 a 0.26% entre las fracciones. La extracción secuencial de carbono orgánico fue mayor para el extractante 0.1 N NaOH por 24 h, seguido por 0.1 N NaOH por 2 h y finalmente agua por 2 h. La relación de densidades ópticas E4/E6 osciló entre 6.6 y 8.5 para ácidos húmicos que se extrajeron con NaOH durante 2 h. La diversidad en sustancias húmicas e iones intercambiables entre fracciones indica que la liberación de nutrientes está regulada por el tamaño de los gránulos. En conclusión, estos datos demuestran que las partículas más pequeñas tienen mayores tasas de humificación, y que el N y P disponible son similares en todos los tamaños de las partículas. Estos resultados demuestran que la utilización de compostas es un método excelente para proveer nutrientes al suelo, y que la pulpa de café bien compostada puede convertirse en un recurso para las fincas agrícolas.

Palabras clave: ácidos húmicos, ácidos fúlvicos, carbón orgánico, extracción secuencial

INTRODUCTION

Coffee is the major agricultural industry in Puerto Rico, covering 57,000 acres in 14 municipalities in the humid central mountain region (Alamo et al., 2006). According to statistics from the Department of Agriculture of Puerto Rico, during the year 2007-2008 coffee production amounted to 7,500 tons of coffee beans at a farm value of over \$36.5 million US dollars. Assuming 41% of coffee pulp (CP) and 20% coffee bean by weight, the estimated CP for that year was 15,375 tons.

Coffee pulp is a by-product obtained from coffee processing; it is a wet feedstock with 85 to 95% water content. The CP has a high concentration of nutrients with a strong potential to impair water resources. Farmers perceive this organic byproduct as waste that needs to be processed at extra cost and effort; hence there is a willingness to remove it from their farms. Coffee pulp composition is basically carbohydrates

(210 to 320 g/kg), proteins (75 to 150 g/kg) and fats (20 to 70 g/kg) (Ulloa-Rojas et al., 2003; Fischer et al., 2001; Murkovic et al., 2006). Additionally it contains 2 to 9% total tannins, 0.8 to 2.8% of which are soluble tannins; 7% pectins; 1% caffeine; 3% chlorogenic acid (CGA); and 2% caffeic acids (Farah et al., 2006).

The amount and chemical forms (aliphatic and aromatic structures) of humic substances can be used to evaluate the long-term effects of the compost soil amendment. The proportion of aromatic carbon structures, measured by optical densities at 465 nm (E4) and 665 nm (E6), provides an indication of recalcitrant, stable non water-soluble, versus the aliphatic structure chain, which is water soluble and subject to labile pressures. This finding is important because it provides a degree of maturity of the humic substances (HS) (Tan, 2003). The HS chemical properties could be different among compost particles in a static pile. This difference could affect the short- and long-term agronomic benefits from humic compost on the soils.

One main objective of this study was to evaluate the chemical and physical properties of the particle sizes of coffee pulp (CP) compost obtained from an aerobic static process, including nutrient content. Another objective was to evaluate the labile and non-labile organic carbon distribution in the compost particle sizes to quantify the benefits of composted coffee pulp. This evaluation can provide an additional incentive for farmers to implement on-farm compost practices.

MATERIALS AND METHODS

Twelve-month-old coffee pulp from a static aerated compost pile was prepared at a compost pilot plant at the Adjuntas Agricultural Experiment Station using 6.6 m³ of coffee pulp per m³ of coffee parchment as an aeration compost base (Chong, 2009). Compost samples were collected in December 2008 after screening compost through a 0.95-cm screen (Orbit model 62, Delhi, IA). The samples were then passed through 8000-, 4000-, 2000-, 1000-, and 500- μ m sieves. Three replicate sieved samples were then placed and tied up in plastic bags and delivered the next day for laboratory testing. The compost fractions were analyzed for pH and electrical conductivity (1:5 compost: water ratio). Total organic carbon (TOC) content was determined by the Walkley and Black method (Nelson and Sommers, 1996). All data were expressed relative to dried mass.

Total Kjeldahl N and P and inorganic nitrogen (N-NH₄ and N-NO₃) were measured with an Autoanalyzer Technicon II (ATII). Inorganic nitrogen was extracted by shaking 0.5 g of fresh compost with 30 ml of

2N KCl for 30 min. Available P was obtained by using AOAC method 960.02. With this method the available P was calculated by subtracting citrate-insoluble P from total P. Total K, Ca and Mg of composts were extracted by the dry ash method and determined with a TJA atomic absorption spectrophotometer (AAS). Exchangeable K, Ca, and Mg were extracted with 1N NH_4OAc (pH 7) and were determined with AAS. Blank samples were used during the extraction procedure to correct for background contamination.

The humic acids (HA) and fulvic acids (FA) were sequentially extracted by the method of Wu and Ma (2002). The extraction was performed sequentially by using 10 to 100 w/v of compost to extracting solution. Samples were first extracted with water for 2 h, followed by a 0.1N NaOH solution for 2 h and 24 h, respectively. Compost suspensions were shaken at room temperature and then centrifuged at 3000 rpm for 10 min. The solution was then filtered through a 0.45 nylon membrane. An aliquot of this filtered solution (50 ml) was adjusted to pH < 2 with 12 N HCl, and then centrifuged at 10,000 rpm and filtered through a 0.45- μm nylon membrane. The HA was the solid, whereas the FA was in solution. The HA and FA were added for total HS. Organic carbon (OC) concentrations were determined with a Dohrmann TOC D190 carbon analyzer from the FA solution, and for HA after the HA pellet was re-dissolved in 50 ml of 0.1N NaOH.

The UV-Vis spectrum of HA solutions, 200 mg/L in 0.05 M NaHCO_3 , were measured by using a UV-Vis Beckman spectrometer at a range of 700 to 190 nm. The optical densities at 465 nm (E4) and at 665 nm (E6) were recorded. These values were used for calculation of the E4/E6 ratio (Chen et al., 1977; Amir et al., 2003).

Data collected from each fraction size were analyzed statistically by using SAS. Analysis of variance (ANOVA) was performed to evaluate the effects of compost granule sizes. Significant means were separated by least significant difference (LSD) tests at $P \leq 0.05$.

RESULTS AND DISCUSSION

Nutrient analysis

No fractions showed any significant differences for total N and P values; however, total K varied from 3.01 to 3.94% (Table 1). Averaged across fractions, total N was 4.89%, and it ranged from 4.29 to 5.31% having a standard deviation of 0.38%. Out of the total N, extractable NO_3^- ranged from 0.33 to 0.37%, and NH_4^+ nitrogen was less than a fraction of one percent. The organic N content averaged 92.9% across all fractions. Total P values averaged 0.46% and ranged from 0.43 to

TABLE 1.—*Nutrient composition of coffee pulp fractions.*

Compost fraction (μm)	Total N	N-NO ₃	N-NH ₄	Total P	Available P ₂ O ₅	Total K	Ca	Mg
			%				%	
500	4.81 a ¹	0.33 b	8.11×10^{-4} b	0.43 a	0.60 a	3.01 c	4.71 b	0.46 c
1000	4.95 a	0.36 a	1.01×10^{-3} ab	0.50 a	0.75 a	3.51 b	5.27 a	0.56 ab
2000	5.09 a	0.35 ab	9.41×10^{-4} ab	0.44 a	0.65 a	3.48 b	5.54 a	0.53 b
4000	4.29 a	0.37 a	1.11×10^{-3} a	0.45 a	0.68 a	3.62 b	5.19 ab	0.51 bc
8000	5.31 a	0.33 b	1.03×10^{-3} ab	0.48 a	0.63 a	3.94 a	5.09 ab	0.62 a

¹Within columns, means followed by the same letter are not significantly different at $P < 0.05$.

0.50%; available phosphorous oxide (P_2O_5), averaged 0.66% (0.30% of P), which is 65% of the total, and had a standard deviation of 0.06%.

Total K content increased with increasing fraction size. Potassium in mature compost is usually not incorporated into organic matter because it is highly soluble. Thus, potassium can easily move from fractions when the organic matter is mineralized, which explains the lower K content in the smallest fraction. Although not examined during this experiment, the aggregate distribution and amounts of different particle sizes could influence availability of nutrients.

The CEC was different among fractions, with the lowest average in the smallest fraction; it ranged from 203 to 262 cmol/kg (Table 2). In general, the exchangeable ions K, Ca and Mg had some of the lowest content in the smallest fraction. The Na ion concentration had no significantly different values across all fractions (Table 2). On average, 89% of the total K was in the exchangeable form, which ranged from 2.82 to 3.33% among fractions. Total Ca content ranged from 4.71 to 5.54% and the exchangeable portion ranged from 2.13 to 3.01%, being approximately 50% of the total Ca. Similarly, total Mg content was between 0.46 and 0.62%, and exchangeable Mg accounted for approximately 44.6% of the total Mg (Table 1). The CP fractions evaluated had no significant differences in electrical conductivity (EC), averaging 3.33 mS/cm (data not shown). The pH varied with no discerning trend, ranging from 6.72 to 6.84 (Table 3).

Carbon analysis

The TOC is comprised of biological carbon, which might include algae, mites, bacteria and fungi, plus humins, lignins, pectins, and humic substances (HS). The relationship between C and N quantities in compost is both an indication of maturity and an index of humification degree, which increases as the C:N ratio decreases (Marinari et al., 2007). The CP feedstock used in this study had a C:N ratio of 13:1. The C:N ratio of the compost fractions ranged from 4.72:1 to 8.88:1 (Table 3).

TABLE 2.—*Exchangeable nutrient content and cation exchange capacity of coffee pulp compost fractions.*

Compost fraction (μ m)	CEC cmol/kg	K	Ca	Mg	Na
			%		
500	203 c ¹	2.82 b	2.13 b	0.22 c	0.14 a
1000	246 ab	2.99 ab	2.88 a	0.23 b	0.16 a
2000	262 a	3.33 a	3.01 a	0.24 b	0.14 a
4000	242 ab	3.33 a	2.60 ab	0.24 ab	0.15 a
8000	225 bc	3.09 ab	2.34 b	0.26 a	0.17 a

¹Within columns, means followed by the same letter are not significantly different at $P < 0.05$.

TABLE 3.—*The pH, total organic carbon and C/N ratio at given coffee pulp compost fractions.*

Granule size μm	pH	TOC %	C/N
500	6.78 b ¹	22.7 c	4.72
1000	6.73 cd	32.4 b	6.54
2000	6.72 d	36.5 a	7.15
4000	6.77 bc	38.1 a	8.88
8000	6.84 a	38.5 a	7.25

¹Within columns, means followed by the same letter are not significantly different at $P \leq 0.05$.

The smallest fraction (500 μm) had the lowest C:N ratio and TOC content, thus suggesting a greater humification in smallest fractions (Table 3). The TOC, which had the lowest quantities in the smallest fraction, 22.7%, increased to 36.5% in the 2000- μm fraction, and averaged 38.3%; across the two larger fractions, between which there was no significant difference (Table 3).

The contact time had a strong effect on the extraction of HS with water and 0.1 M NaOH. The HS in water ranged from 3.80 to 4.4 g/kg dry compost mass; HS in 0.1 M NaOH for 2 h ranged from 17.0 to 23.8 g/kg; HS in 0.1 N NaOH for 24 h ranged from 38.0 to 47.6 g/kg dry compost mass. The above data showed that distribution of extractable OC across the three sequential extraction steps increased in the following order: water < 0.1 N NaOH for 2 h < 0.1 N NaOH for 24 h. This order corresponds mainly to an increase in the amount of humic acids (HA) rather than fulvic acids (FA) as indicated by the HA/FA distribution (Table 4). In general, HA of the compost granules extracted over a 24-h period had lower E4/E6 values than HA extracted for 2 h. This result indicates that a greater amount of less soluble and more recalcitrant HA was extracted by 0.1 M NaOH over a period of 24 h. These results were supported by the value of HA/FA ratio, which was higher for organic matter from 24-h extracts than from 2-h extracts (Table 4).

Greater amounts of total HS were obtained in larger particles; however, smaller particles had greater recalcitrance HS as shown by E4/E6 ratio at 2 h (Table 5). Small particles arise from larger particles. As larger particles break down, the superficial area increases; thus, microorganisms have greater opportunity to decompose and increase the humified fractions of organic matter. Smaller particles have greater superficial area. This area is one of the reasons for their lower content of HS and higher HS/TOC ratio but greater aromatic HS. As larger particles became small, greater amounts of liable organic carbon were consumed and/or humified into aromatic humic substances.

TABLE 4.—Organic carbon contents of sequentially extracted CP compost. Mass of compost is expressed on a dry weight basis.

Granule size μm	Water 2 h				0.1 M NaOH 2 h				0.1 M NaOH 24 h			
	OC _{FA}	OC _{HA}	OC _{FA+HA}	HA/FA	OC _{FA}	OC _{HA}	OC _{FA+HA}	HA/FA	OC _{FA}	OC _{HA}	OC _{FA+HA}	HA/FA
	g/kg				g/kg				g/kg			
500	2.3 b ¹	1.5 c	3.8	0.65	5.9 a	15.3 b	21.2	2.58	9.3 c	28.7 e	38.0 e	3.07
1000	2.4 b	1.6 c	4.0	0.67	6.3 a	17.5 a	23.8	2.76	9.5 b	30.2 c	39.7 c	3.16
2000	2.6 a	1.5 c	4.0	0.59	6.3 a	17.1 ab	23.4	2.71	9.6 b	28.8 d	38.4 d	3.01
4000	2.4 b	1.8 b	4.2	0.73	6.2 a	14.2 c	20.4	2.29	9.8 b	33.1 b	42.9 b	3.37
8000	2.4 b	2.0 a	4.4	0.86	5.8 b	11.3 d	17.0	1.95	11.5 a	36.1 a	47.6 a	3.13

¹Within columns, means followed by the same letter are not significantly different at $P \leq 0.05$.

TABLE 5.—The total humic substances, extractable fulvic and humic acids, ratio of humic substances to total organic carbon, and absorbance of HA measured at 465 nm (E4) and 665 nm (E6) at given coffee pulp compost fractions.

Granule size μm	HS %	FA %	HA %	HS/TOC %	HA NaOH 2h E4/E6
500	6.07 c ¹	1.74 c	4.33 c	26.8	7.50
1000	6.76 ab	1.83 b	4.93 ab	20.9	7.60
2000	6.59 b	1.85 b	4.74 b	18.0	8.10
4000	6.75 ab	1.84 b	4.90 ab	17.7	8.10
8000	6.90 a	1.97 a	4.94 a	17.9	8.50

¹Within columns, means followed by the same letter are not significantly different at $P \leq 0.05$.

The first humic substances that are formed during earlier stages of composting are the FA. Therefore, high concentration of FA could be associated with an incomplete composting process. The FA, an easily extractable organic carbon, decreases with composting age, and its rate of disappearance depends on the composition of the compost source material (Wu and Ma, 2002). Wu et al. (2002) reported that when concentration of FA drops below 9.4 g/kg, the evolution of CO₂ is very slow, a sign of the compost's stability. The low range of water (2 h) and NaOH (2 h) extractable FA in the samples of this study, 2.2 to 2.6 g/kg and 5.8 to 6.3 g/kg, respectively, and the high concentration of humic acids and cation exchange capacity indicate that the coffee compost pile was mature at the sampling time (Tables 2 and 4).

The structural quality, aliphatic-aromatic, of the HS provides a qualitative index of compost stability. The absorbance of HA extracted with 0.1M NaOH for 2 h and measured at 465 nm (E4) and 665 nm (E6), has been used as a qualitative measurement of compost stability (Wu and Ma, 2002). Specifically the E4/E6 ratio provides a tool for estimating relative size of humic substances and humification degree, both of which factors have been directly correlated with aliphatic-aromatic structures (Campitelli and Ceppi, 2008; Tan, 2003). Low E4/E6 ratios indicate greater aromatic carbon networks, whereas high ratios suggest more aliphatic structures and macromolecules of low molecular weight (Tan, 2003; Stevenson, 1994). Over a period of two hours the NaOH-extracted humic acids had low E4/E6 ratios, regardless of particle sizes. This finding indicates the presence of HA with high molecular weight (Table 5). Humic acids from the largest granular sizes (8000 µm) showed the highest E4/E6 ratio (Table 5), thus indicating HA of lower molecular weight (Cheng et al., 1977; Stevenson, 1994), and also indicating the presence of the relatively largest proportions of aliphatic structures (Senesi et al., 2001) and the least maturity in the largest particles. Thus, low E4/E6 ratios indicate greater aromatic carbon networks, whereas high ratios suggest more aliphatic structures and macromolecules of low molecular weight (Tan, 2003; Stevenson, 1994). The HA extracted with 0.1M NaOH for 2 h from the largest granular sizes (8000 µm) had the highest E4/E6 ratio (Table 5), which indicates the presence of relatively large proportions of aliphatic structures and thus the lowest condensation degree and maturity (Senesi et al., 2001). The smallest ratios were found in the smallest fraction, thereby suggesting greater stable aromatic structures. The above data suggests that differences in the chemical composition of humic substances among particle sizes are associated with intrinsic characteristics of the feedstock rather than with an incomplete composting process.

This study shows that HS composition and its properties, HS to TOC ratio and labile C fractions, are related to the size of the particles. The finding of humified organic matter with different C network properties (aliphatic-aromatic content) helps to explain why the organic amendments usually have short- and long-term agronomic benefits. The short-term benefits of compost come from already mineralized nutrients that provide immediate plant nourishment. Long-term agronomic benefits in part come from better soil structure, porosity and water retention, and the further nutrient mineralization once the organic amendment is applied.

The smallest particle size had the greatest HS/TOC ratio, and lowest E4/E6 ratio and TOC. These findings suggest that during the development of the smaller particles, the increase in superficial area caused an increase in microorganism activity and environmental exposure. As a result, labile OM and C:N ratio decreased, increasing the lability of aliphatic structures and remnants of aromatic structures.

This study demonstrates that 12-month-old coffee pulp compost can be an excellent source of nutrients; these nutrients were generally stable across fractions, with the exception of K. The above data show that CP fractions had up to 65% organic matter ($\text{TOC} \times 1.68$), out of which a maximum of 6.9% were humic substances. Greater amounts of HA than FA were extracted across all fractions. The larger superficial area of the smallest fractions provides greater exposure to microorganisms and environmental effects thus causing greater decomposition of labile TOC as shown by higher recalcitrant forms, aliphatic-aromatic, HS relative to TOC, and FA-HA amounts. It is suggested that aliphatic-aromatic structure tests should be performed when compost is evaluated, given the fact that FA and HA concentration alone can provide misleading conclusions regarding the long-term effects of OM.

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