



ISSN: 2230-9926

Available online at <http://www.journalijdr.com>

IJDR

International Journal of Development Research

Vol. 11, Issue, 10, pp. 50969-50993, October, 2021

<https://doi.org/10.37118/ijdr.22955.10.2021>



RESEARCH ARTICLE

OPEN ACCESS

CHARACTERIZATION OF BIOCHAR PRODUCED FROM SHELL OF *Theobroma grandiflorum*

Fabrício M. Lisbôa*¹, Selma de O. Freitas¹, Michelle S. Ramos¹, Melissa A. Zamai²
and Michely A. Zamai²

¹Professor. Federal Institute of Education, Science and Technology of Rondônia, Address: RO 257, Km 13, Rural Area, Ariquemes – Rondônia; ²Student. Federal Institute of Education, Science and Technology of Rondônia, Address: RO 257, Km 13, Rural Area, Ariquemes – Rondônia

ARTICLE INFO

Article History:

Received 08th August, 2021

Received in revised form

26th September, 2021

Accepted 11th October, 2021

Published online 30th October, 2021

Key Words:

Carbon Sequestration,
Environment Pollution,
Soil Amendment, Waste.

*Corresponding author: *Fabrício M. Lisbôa*,

ABSTRACT

Biochar can be defined as vegetable biomass, which has undergone pyrolysis in a low oxygen environment. It can contribute to chemical, physical and biological improvements in the soil. Different biomasses can be used in the production of biochar. However, characterization studies are needed before its recommendation. The aim of this work was to test the temperatures: 300, 400 and 500 °C in *Theobroma grandiflorum* for biochar production and to characterize the samples, submitted to different temperatures, to verify which temperature presents the best chemical and physical properties. The increase in temperature to 500 °C promoted an increase in carbon, nitrogen, nutrients, as well as pH and electric conductivity. There was a reduction in the original mass and in the values of polyphenols, cellulose, density and the aromaticity index.

Copyright © 2021, *Fabrício M. Lisbôa et al.* This is an open access article distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

Citation: *Fabrício M. Lisbôa, Selma de O. Freitas, Michelle S. Ramos, Melissa A. Zamai and Michely A. Zamai.* "Characterization of biochar produced from shell of *Theobroma Grandiflorum*", *International Journal of Development Research*, 11, (10), 50969-50993.

INTRODUCTION

Biochar can be defined as vegetable biomass, which has undergone pyrolysis in a zero or low oxygen environment, being better described as soil amendment. It is suggested that the carbon components in biochar are highly recalcitrant, with reported residence times of 100 to 1,000 years, approximately 10 to 1,000 times longer than the residence time of most soil organic matter (Verheijen *et al.*, 2010). In addition to its use in the preservation of carbon, biochar can contribute to chemical, physical and even biological characteristics of the soil. To be considered ideal, Rezende *et al.* (2011) pointed out that biochar must have an inert internal structure, which preserves carbon in the soil for many years, and a reactive external structure to interact with the environment. Among these interactions, the interaction of biochar with organic soil structures stands out; water retention; retention of H⁺ and OH⁻ ions, controlling soil pH; retention of nutrients that can be made available to plants and retention of toxic elements (Al³⁺). Initially, combustion leads to loss of mass of the original material, due to the degradation of volatile compounds, in

addition to the degradation of compounds such as hemicellulose and cellulose, while proportional increase in lignin occurs. However, numerous positive properties can be observed after pyrolysis, such as: elevation of: pH; Electric conductivity; cation exchange capacity; increase in macro and micronutrients and increase in the C:N ratio (Verheijen *et al.*, 2010). In addition, the release of volatile materials contributes to increasing the formation of pores that serve as a habitat for the soil microbiota and water retention in it. The properties of biochar depend on the composition of the material to be used and the temperature used. When the pyrolysis temperature is high, less biochar is generated and the microstructure develops more effectively. Carbon is also partially released as a volatile material, but another part of it remains as fixed carbon. However, if the temperature is too high, the loss of carbon and other elements of the functional group on the surface are excessive (Tan *et al.*, 2014). According to Bahng *et al.* (2009), one of the technologies used to produce biocarbons is pyrolysis, in which the biomass is elevated at temperatures ranging from 400 to 600 °C in the absence of oxygen.

According to the author, the pyrolysis process can be divided into three subclasses: slow pyrolysis (heating of ≈ 500 °C and heating rate of $0.1-1$ °C.S⁻¹), rapid pyrolysis (heating of 600-1000 °C and heating rate $10-200$ °C.S⁻¹) and flash pyrolysis (heating 800-1000 °C and heating rate > 1000 °C.S⁻¹). Angin and Şensöz (2014) reported an increase in the content of carbon, ash, pH and specific surface area, to the detriment of the release of volatile materials, with an increase in the temperature of production of the biochar from 400 to 700 °C. The higher pyrolysis temperatures were responsible for the release of gases, which can contribute to the greenhouse effect. Using pyrolysis temperatures of 200, 300, 400 and 500 °C, Chen *et al.* (2014), reported an increase in pH, ash content, and organic carbon, while the levels of N, O, H decreased. Through Nuclear Magnetic Resonance, they verified that there was a loss of structures such as cellulose and hemicellulose, and the concentration of aromatic groups with increasing temperature, these aromatic groups are responsible for the greater permanence of carbon in the soil.

These data indicate that the best temperature for biocavão production is between 500 to 700 °C. Regarding the different materials that can be used in the production of biochar, these have been reported the most diverse, requiring the study of characterization before the most accurate recommendation of different biomasses. Originating in the Amazon region, *Theobroma grandiflorum* is one of the main and most used fruits in the region. Their shells, which can represent 37 to 41% of the fruit's composition. However, they do not have a range of reuse other than making crafts and producing energy (Gondim *et al.*, 2001). The use of *T. grandiflorum* shell for biochar production has both agronomic and environmental potential. From an environmental point of view, we have the opportunity to use 100% of the fruit and avoid waste. From an agronomic point of view, the shells may have potential for biochar production and, depending on its chemical and physical attributes, can be recommended as a soil amendment in food production. Thus, the aim of this work was to test the temperatures: 300, 400 and 500 °C in *T. grandiflorum* shells for biochar production and to characterize the samples, submitted to different temperatures, to verify which temperature presents the best chemical and physical properties.

MATERIALS AND METHODS

Temperature Test: Prior to the test temperature, the *T. grandiflorum* shells were dried at 105 °C to constant weight. After, the shells were milled in a Willey Knife Mill (SL-32 Solab). The material was placed in reactor (SP-1200DRP7), and based on the literature, the temperatures to be used as treatments for degradation of hemicellulose, cellulose and lignin were respectively: 300 °C; 400 °C and 500 °C (Chen *et al.*, 2014). These temperatures were selected because they displayed the best results in other studies, and facilitate comparisons with other studies. The type of pyrolysis used was slow pyrolysis, with a heating rate of $0.1-1$ °C.S⁻¹ (Bahng *et al.*, 2009). The heating rate was 1 °C.S⁻¹ until reaching the desired temperature. When reaching the desired temperature value, it was kept constant for 15 minutes, when the reaction is finished. After cooling in a desiccator, the material obtained was stored in an airtight environment until further analysis.

Remaining mass (Rm): In order to verify the mass of the material after the temperature rise, the remaining mass (Rm) was calculated, according to the formula:

$$Rm = \text{mass of final sample} / \text{mass of initial sample} \times 100$$

Analytical Procedures: The analyzes were performed in triplicates, the result being expressed as the average of the same.

Density: The verification of the density of the material is an important factor, since it reflects changes in its structure. As the density increases, there is a tendency to decrease its porosity. To determine the density, according Embrapa (2011), 100 ml plastic beaker was filled up to approximately the 50 ml mark with the dry

substrate. Then, this beaker was dropped, under the action of its own mass, from a height of 10 cm, for 10 consecutive times. The volume obtained (ml) was read. Then, the material was weighed (g), discounting the mass of the beaker. The procedure was repeated three times with different subsamples. The mean value of the measurements was expressed as a whole number. The determination of the density value was obtained by the following formula: Density (kg.m^{-3}) = $\text{mass (g)} / \text{volume (ml)} \times 1000$

pH: To determine the pH, 10 g of the sample was used with 25 ml of water, which was shaken, and left to rest for 1 hour, when reading with a digital pH meter GEHAKA (PG1800), previously calibrated with standard solutions of 4.0 and 7.0 (Embrapa, 2011).

Electrical Conductivity (EC): The determination of Electrical Conductivity was performed to evaluate the content of water-soluble electrolytes in the materials. The samples were weighed (50 g) and transferred to erlenmeyer with 50 ml of water, and left to rest. The conductivity was determined of filtrate extraction using the MS TECNOPON conductivity meter (mCa 150P) and values expressed in mS.cm^{-1} at 25 °C (Embrapa, 2011).

Chemical analysis: The macro and micronutrient determinations in the initial samples and in the samples treated with different temperatures were performed according to Embrapa (2000). The samples with 1 mm diameter sieves, were dried in an oven at 65 °C, until reaching constant weight. The elements were determined by solubilization in nitric acid (65%) and perchloric acid (70%), in atomic absorption spectrometry. The carbon analysis was performed by oxidation by dichromate in an acid medium, determined colorimetrically at 600 nm (Anderson and Ingram, 1992). The nitrogen determination was carried out through sulfuric solubilization followed by the Kjeldahl method (Embrapa, 2000). The determination of the contents of recalcitrant molecules such as Polyphenols, Cellulose and Lignin, were carried out based on the solubility in detergent solution, according to the method of Robertson and Van Soest (1981).

Fourier Transform Infrared Spectroscopy by Diffuse Reflectance: Fourier Transform Infrared Spectroscopy analyzes were conducted by Diffuse Reflectance using Shimadzu spectrometer (DRS-8000A). This technique avoids errors in the preparation of KBr pellets, reduces water band interference, and the IR ray interacts with the particles to produce better defined and better spectra (Haberhauer and Gerzabek, 1999; Azuaje *et al.*, 2012). Each sample was mixed and ground with potassium bromide (KBr), after being oven dried at 60 °C, and the reading was performed with the diffuse reflectance accessory, employing 40 scans and 4 cm^{-1} resolution, in the spectral range of $4,000$ to 400 cm^{-1} . The spectra and band intensity data were obtained by the Shimadzu IR Solution 1.6 program after adjusting the overall spectrum baseline at three points: $4,000$, $2,000$ and 400 cm^{-1} . The aromaticity level of the molecules was verified through the aromaticity index (AI), calculated according to Dick *et al.* (2008) by the expression I_{C-C} / I_{C-H} , where I_{C-C} is the absorption intensity around $1,640 \text{ cm}^{-1}$ and I_{C-H} is the absorption intensity around $2,920 \text{ cm}^{-1}$.

Results Analysis

The experiment was conducted in a completely randomized design, with four treatments and three replications being applied. The Treatments were:

1. T000: original material dried at 105 °C (control);
2. T300: material dried at 105 °C in an oven and heated to 300 °C;
3. T400: material dried at 105 °C in an oven and heated to 400 °C;
4. T500: material dried at 105 °C in an oven and heated to 500 °C.

All results of characterization of the material were subjected to analysis of variance (ANOVA).

In the case of significant effects indicated in ANOVA, comparisons of means were made by Tukey's test ($\alpha = 5\%$).

RESULTS

The remaining mass decreased with increasing temperature, varying from 32.36%, at 300 °C, to 17.73% at 500 °C (Table 1). The increase in temperature provided an increase in the ashes (5.63 to 19.1 g.kg⁻¹) The carbon (C) contents were lower in the green material, 424.56 g.kg⁻¹, increasing significantly by 500 °C to 465.50 g.kg⁻¹. However, the highest values were observed at intermediate temperatures of 300 and 400 °C (488.62 and 488.36 g.kg⁻¹, respectively). The same behavior was observed with nitrogen (N), which increased from 4.76 g.kg⁻¹ to 6.63 g.kg⁻¹, as the temperature increased to 500 °C. Regarding Polyphenols and Cellulose, there was a decrease as the temperature increased to 500 °C (Table 1). Polyphenols decreased from 8.41 g.kg⁻¹ to 1.05 g.kg⁻¹ and Cellulose decreased from 307.23 g.kg⁻¹ to 13.23 g.kg⁻¹. The opposite behavior was observed for Lignin and Ashes, with an increase in values as the temperature increased. Lignin increased from 438.20 g.kg⁻¹ to 896.40 g.kg⁻¹ and Ashes increased from 5.63 g.kg⁻¹ to 19.10 g.kg⁻¹.

Table 1. Values of remaining mass (Rm), ash, carbon (C), nitrogen (N), polyphenols, cellulose and lignin of biochar produced from *T. grandiflorum*'s shell at different temperatures

Variable	T000	T300	T400	T500	*CV (%)
Rm (%)	-	32.36a	22.86b	17.73c	25.96
Ashes (g.kg ⁻¹)	-	5.63c	15.63b	19.10a	1.16
C (g.kg ⁻¹)	424.56c	488.62a	488.36a	465.50b	0.11
N (g.kg ⁻¹)	4.76c	7.83a	7.89a	6.63b	6.22
Polyphenols (g.kg ⁻¹)	8.41a	1.29b	0.96c	1.05c	2.24
Cellulose (g.kg ⁻¹)	307.23a	44.33b	16.76c	13.23d	0.30
Lignin (g.kg ⁻¹)	438.20d	922.76a	894.43c	896.40b	0.04

a-n Different letters indicate significant differences within the same column at p-value < 0.05. *Coefficient of variation.

The increase in temperature promoted an increase in pH and Electric conductivity (E.C.). The pH values increased from 4.33 to 9.34. While the E.C. values increased from 1.258 mS.cm⁻¹ at 25 °C to 5.268 mS.cm at 25 °C (Table 2).

Table 2. Values of pH and Electric conductivity (E.C.) of biochar produced from *T. grandiflorum*'s shell at different temperatures

Variable	T000	T300	T400	T500	*CV (%)
pH	4.33d	6.83c	9.01b	9.34a	0.14
E.C. (mS.cm ⁻¹ at 25 °C)	1,258d	1,594c	3,567b	5,268a	0.03

a-n Different letters indicate significant differences within the same line at p-value < 0.05. *Coefficient of variation.

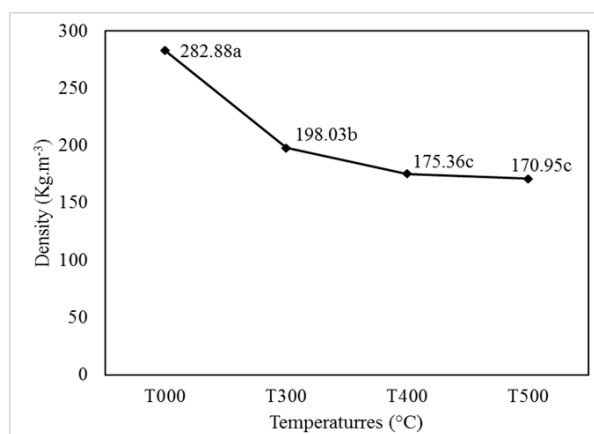
Regarding the nutritional composition of biochar, the increase in temperature promoted an increase in the contents of all the elements evaluated, increasing significantly with the increase in temperature (Table 3).

Table 3. Values of macronutrients, micronutrients and some evaluated elements of biochar produced from *T. grandiflorum*'s shell at different temperatures

Variable	T000	T300	T400	T500	*CV (%)
Ca (g.kg ⁻¹)	0.26d	0.68c	0.95b	1.30a	0.89
Mg (g.kg ⁻¹)	0.26d	0.76c	1.15b	1.59a	0.75
K (g.kg ⁻¹)	6.37d	17.46c	28.09b	36.74a	0.61
Na (mg.kg ⁻¹)	33.52d	119.03c	127.59b	150.85a	0.97
P (mg.kg ⁻¹)	29.56d	84.31c	100.51b	132.34a	0.34
Fe (mg.kg ⁻¹)	15.23d	33.61c	36.31b	60.29a	1.50
Cd (mg.kg ⁻¹)	1.79d	1.96c	2.06b	2.17a	0.50
Co (mg.kg ⁻¹)	2.30d	2.44c	2.53b	2.60a	0.40
Cr (mg.kg ⁻¹)	2.85c	3.12b	3.29a	3.29a	0.36
Cu (mg.kg ⁻¹)	5.76c	7.15b	7.75b	8.82a	4.20
Li (mg.kg ⁻¹)	20.08d	21.35c	25.71b	36.21a	0.76
Mn (mg.kg ⁻¹)	1.19d	2.43c	3.44b	6.43a	0.23
Mo (mg.kg ⁻¹)	3.63d	4.14c	4.50b	4.89a	0.37
Zn (mg.kg ⁻¹)	10.57d	20.14c	25.83b	38.64a	0.62

a-n Different letters indicate significant differences within the same line at p-value < 0.05. *Coefficient of variation

Among macronutrients, potassium (K) had the highest values in relation to the others: 6.37 g.kg⁻¹, in the original material and 36.74 g.kg⁻¹ at 500 °C, followed by phosphorus (P): 29.56 g.kg⁻¹, in the original material, 132.34 g.kg⁻¹ at 500 °C. Sodium (Na) showed a strong increase with temperature rise: 32.52 mg.kg⁻¹, in the original material and 150.85 mg.kg⁻¹ at 500 °C. Among micronutrients, the highlight was iron (Fe): 15.23 mg.kg⁻¹ in the original material and 60.29 mg.kg⁻¹ at 500 °C. The values of density of the biochar decreased significantly with the increase of the temperature to 500 °C, varying from 282.88 Kg.m⁻³ to 170.95 Kg.m⁻³. However, there was no significant increase between temperatures of 400 and 500 °C (Figure 1).



a-n Different letters indicate significant differences at p-value < 0.05. Coefficient of variation (CV): 1.96%.

Figure 1. Values of Density of biochar produced from *T. grandiflorum*'s shell at different temperatures

The Fourier Transform Infrared Spectroscopy spectra, by Diffuse Reflectance, showed similarities between the spectra at 300, 400 and 500 °C (Figure 2). These spectra were different from the original material regarding the presence of band 3,300 cm⁻¹, H bond of the OH group; band 2,922 cm⁻¹, of C-H stretching vibration in the aliphatic group; and band 1,050 cm⁻¹ of C-O binding in polysaccharides (Wang *et al.*, 2013). All spectra showed the band 1,720 cm⁻¹, related to the C=O bond; band 1,650 cm⁻¹ of aromatic ring C=C stretching vibration and conjugated ketone and quinone C=O stretching vibration; and band 1,250 cm⁻¹ of OH plane deformation (Zang *et al.*, 2020).

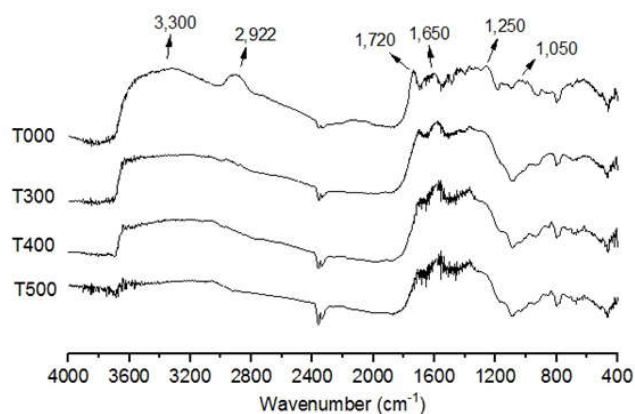
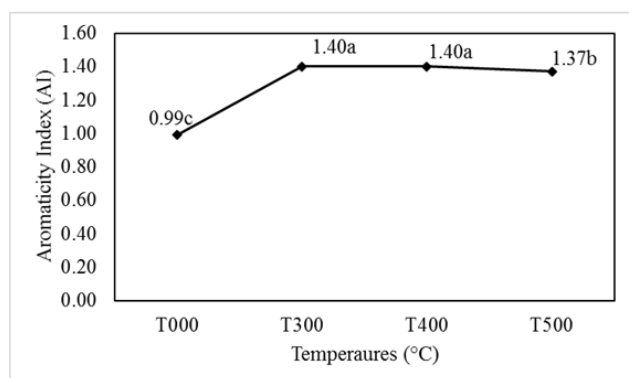


Figure 2. Fourier Transform Infrared Spectroscopy spectra, by Diffuse Reflectance, of biochar produced from *T. grandiflorum*'s shell at different temperatures

The aromaticity index (AI) showed an increase from the original material to temperatures of 300 and 400 °C (0.99 to 1.40), with a progressive decrease in 500 °C (1.37) (Figure 3).



^{a-b}Different letters indicate significant differences at p -value < 0.05. Coefficient of variation (CV): 0.71%.

Figure 3. Aromaticity Index (AI) of biochar produced from *T. grandiflorum*'s shell at different temperatures.

DISCUSSION

The results showed that there was a decrease in the remaining mass as a function of the temperature increase, with their corresponding increase in the amount of ashes (Table 1). This reduction was over 80% to 500 °C. These may be related to water loss, volatile compounds as well as complex components such as cellulose (Zhang *et al.*, 2020). As shown in Table 1, cellulose and polyphenols decreased with temperature increase. Zhao *et al.* (2018) characterizing biochars from different plant sources, obtained a remaining mass of, approximately, 18 to 41%, at a temperature of 500 °C. Carbon and nitrogen values increased up to 400 °C (Table 1). After 500 °C there was a decrease in their values. This behavior may be associated with an increase in lignin values, in relation to original material. According to Zhang *et al.* (2020), this indicated that the aromatic structure forms at increasing temperatures due to the decomposition of cellulose and other macromolecules in biochar. There was an increase in pH values with increased temperature applied in biochar production (Table 2). Zhao *et al.* (2018) explained that the effect of pyrolysis temperature on biochars pH is related to two main aspects. The first one: the increasing of pyrolysis temperature increase the formation of biochar ash which contains alkaline minerals. Other aspect is the biochar produced at lower temperatures may contain greater densities of acidic functional groups, as phenolic and carboxylic groups, thus decreasing biochar pH. In fact, we observed an increase in ash production, as can be seen in Table 1.

The first one: the increasing of pyrolysis temperature increase the formation of biochar ash which contains alkaline minerals. Other aspect is the biochar produced at lower temperatures may contain greater densities of acidic functional groups, as phenolic and carboxylic groups, thus decreasing biochar pH. In fact, we observed an increase in ash production, as can be seen in Table 1. As pH, electrical conductivity increased significantly with increasing temperature, to 500 °C (Table 2). As already shown, the increase in temperature provides more ash and the mineral content interferes with both pH and electrical conductivity. While pH is more dependent on alkaline metals such as sodium and potassium, electrical conductivity is most affected by alkaline earth metals such as calcium and magnesium (Intani *et al.*, 2018). Despite the fact that we have not found specific data in the literature on the nutritional composition of biochars produced from *T. grandiflorum*'s shell, several authors highlight the increase in chemical elements with the increase in temperature. In this study (Table 3), among the macronutrients, the elements that enhance in greater quantity were K (increase of 5.7) and P (increase of 4.5). With the exception of macronutrients, the elements that enhance in greater quantity were Na (increase of 4.5) and Fe (increase of 4.0). Conz (2015) evaluated several materials for the production of biochars and obtained significant increases (at 650 °C) of 4.1 to K; 1.7 to P and 1.2 to Fe. Oliveira *et al.* (2015), obtained an increase (at 500 °C) of 8.0 to K; 3.3 to P and 0.8 to Fe, in relation

to the original plant material studied. In these studies, Na was not evaluated. As shown in figura 1, the values of biochar density decrease with increase of temperature. Density and porosity are physical inversely proportional properties. During temperature increase, pores may be filled with material decomposition, and partially blocking porosity (Ghani *et al.*, 2013). This may cause an increase in density values. There was no significant difference between the density values at temperatures of 400 and 500 °C, which were lower than the density values of the original material and the temperature of 300 °C. According to the findings of Ghani *et al.* (2013), this may be an indication that the rate of pore formation exceeded that of destruction, due to pore enlargement and collapse at the beginning of the process. The Fourier Transform Infrared Spectroscopy spectra, by Diffuse Reflectance (Figure 2), showed water loss from the original material, when subjected to different temperatures. What was evident with the decrease of the band 3,300 cm^{-1} . In addition, there was a decrease in the bands related to aliphatic groups (2,922 cm^{-1}). Zhang *et al.* (2020) also observed the presence of bands around 2,920 cm^{-1} at lower temperatures of biochar (300-400 °C) which were attributed to the existence of compounds such as cellulose. After an increase in temperature (300, 400 and 500 °C), there was also a loss of compounds such as polysaccharides, evidenced by the absence of the 1,050 cm^{-1} band (Wang *et al.*, 2013). The decrease in water and carbon compounds less resistant to temperature corroborates the data, shown here, on loss of mass of the material when subjected to high temperatures and may be indicative of the loss of compounds such as polyphenols and cellulose, which also decreased after temperature increase (Table 1).

According to Wang *et al.* (2013) the increase in temperature provides the loss of OH and aliphatic groups, which improve the formation of pores due to the development of aromatic ring structures. Considering that there is a decrease in density with an increase in porosity, this hypothesis is consistent with the behavior of the studied samples, since there was a decrease in the density of samples with an increase in temperature (Figure 1). This was also clear when we noticed an increase in AI between the original material and the biochars. The aromaticity index (IA) is an index used to check the level of recalcitrance. It is calculated from the ratio between the absorption intensity of aromatic (1,640 cm^{-1}) and aliphatic (2,920 cm^{-1}) groups, the greater the absorption at 1,640 cm^{-1} , the greater the AI value (Dick *et al.*, 2008). In the present study (Figure 3), we verified an increase in the aromaticity of the original sample when submitted to 300 and 400 °C, however, there was a decrease when the temperature was 500 °C. According to Tan *et al.* (2014), higher pyrolysis temperatures lead to the loss of hydrogen, this promotes the direct connection between oxygen and carbon, thus integrating oxygen into the carbon chain forming simple carbon-oxygen bonds. Thus, it is believed that, up to a temperature of 400 °C, formation of condensed structures occurs, formation of aromatic rings, from 500 °C the formation of simple bonds between carbon and oxygen leads to an increase in aliphatic structures and a decrease in aromaticity index values from that temperature.

CONCLUSION

The increase in temperature to 500 °C promoted an increase in carbon, nitrogen, nutrients, as well as pH and electric conductivity, in relation to the original material. On the other hand, there was a reduction in the original mass and in the values of polyphenols, cellulose and density. The increase in temperature also promoted the loss of aliphatic compounds and the proportional increase of more recalcitrant compounds, such as lignin, at 400 °C, from which, the reduction of part of these compounds occurs, with an increase at 500 °C, also evidenced by the aromaticity index.

Acknowledgment: To Federal Institute of Education, Science and Technology of Rondônia. And Federal University of Rondônia and State University of North Fluminense for their support during the conduct of the analyzes.

REFERÊNCIAS

- Anderson, J.D., Ingram, J.S.I. 1992. Tropical soil biology and fertility: a handbook of methods. 2nd Ed. Oxford University Press, Wallingford. 240p.
- Angin, D., Şensöz, S. 2014. Effect of pyrolysis temperature on chemical and surface properties of biochar of rapeseed *Brassica napus* L. . International Journal of Phytoremediation. 7, pp. 684–693. DOI: <https://doi.org/10.1080/15226514.2013.856842>
- Azuaje, E.I., Comerford, N.B., Harris, W.G., Reeves III, J.B. 2012. Grunwald, S. Loblolly and slash pine control organic carbon in soil aggregates and carbon mineralization. Forest Ecology and Management. 263, pp. 1-8. DOI: <https://doi.org/10.1016/j.foreco.2011.09.030>
- Bahng, M.K., Mukarakate, C., Robichaud, D.J., Nimlos, M.R. 2009. Current technologies for analysis of biomass thermochemical processing: A review. Analytica Chimica Acta. 651, pp. 117–138. DOI: <https://doi.org/10.1016/j.aca.2009.08.016>
- Chen, C.P., Cheng, C.H., Huang, Y.H., Chen, C.T., Lai, C.M., Menyailo, O.V., Fan, L.J., Yang, Y.W. 2014. Converting leguminous green manure into biochar: changes in chemical composition and C and N mineralization. Geoderma. 4, pp. 581–588. DOI: <http://dx.doi.org/10.1016/j.geoderma.2014.06.021>
- Dick, D.P., Martinazzo, R., Dalmolin, R.S.D., Jacques, A.V.A., Mielniczuk, J., Rosa, A.S. 2008. Impacto da queima nos atributos químicos e na composição química da matéria orgânica do solo e na vegetação. Pesquisa Agropecuária Brasileira. 43, pp. 633-640. DOI: <https://doi.org/10.1590/S0100-204X2008000500011>
- EMBRAPA. 2000. Métodos de Análise de Tecidos Vegetais Utilizados na Embrapa Solos. Embrapa Solos. Rio de Janeiro. URL: <https://ainfo.cnptia.embrapa.br/digital/bitstream/item/62212/1/Metodo-de-analise-de-tecido.pdf>
- EMBRAPA. 2011. Manual de métodos de análise de solo. Embrapa Solos. Rio de Janeiro. URL: <https://ainfo.cnptia.embrapa.br/digital/bitstream/item/104933/1/Manual-de-Mtdos-de-Anilise-de-Solo.pdf>
- Ghani, W.A.W.A.K., Mohd, A.; Silva, G., Bachmann, R.T., Taufiq-Yap, Y.H., Rashid, U., Al-Muhtaseb, A.H. 2013. Biochar production from waste rubber-wood-sawdust and its potential use in C sequestration: Chemical and physical characterization. Industrial Crops and Products. 44, pp. 18-24. DOI: <https://doi.org/10.1016/j.indcrop.2012.10.017>
- Gondim, T.M.S., Thomazini, M.J., Cavalcante, M.J.B., Souza, J.M.L. 2001. Aspectos agronômicos do cupuaçu. Embrapa Acre. Rio Branco. URL: <https://www.infoteca.cnptia.embrapa.br/bitstream/doc/498481/1/doc67.pdf>
- Haberhauer, G., Gerzabek, M.H. 1999. Drift and transmission FT-IR spectroscopy of forest soils: an approach to determine decomposition processes of forest litter. Vibrational Spectroscopy. 19, pp. 413–417. DOI: <https://doi.org/10.1016/S0924-203198.00046-0>
- Intani, K., Latif, S., Cao, Z., Muller, J. 2018. Characterization of biochar from maize residues produced in a self-purging pyrolysis reactor. Bioresource Technology. 265, pp. 224-235. DOI: <https://doi.org/10.1016/j.biortech.2018.05.103>
- Rezende, E.I.P., Angelo, L.C., Santos, S.S., Mangrich, A.S. 2011. Biocarvão Biochar. e Sequestro de Carbono. Revista Virtual Química. 3, pp. 426-433. URL: <file:///C:/Users/Usuario/AppData/Local/Temp/204-1811-3-PB.pdf>
- Robertson, J.B., Van Soest, P.J. 1981. The detergent system of analysis and its application to human foods. In James, W.P.T.; Theander, O. eds.. The analysis of dietary fiber in food. Marcel Dekker. New York. pp. 123-158.
- Tan, C., Yaxin, Z., Hongtao, W., Wenjing, L., Zeyu, Z., Yuancheng, Z., Lulu R. 2014. Influence of pyrolysis temperature on characteristics and heavy metal adsorptive performance of biochar derived from municipal sewage sludge. Bioresource Technology. 164, pp. 47–54. DOI: <https://doi.org/10.1016/j.biortech.2014.04.048>
- Verheijen, F., Jeffery, S., Bastos, A.C., Van Der Velde, M., Diafas, I. 2009. Biochar Application to Soils: A Critical Scientific Review of Effects on Soil Properties, Processes and Functions. Joint Research Centre. European Communities. Ispra. URL: https://publications.jrc.ec.europa.eu/repository/bitstream/11111111/13558/1/jrc_biochar_soils.pdf
- Wang, Y., Hu, Y., Zhao, X., Wang, S., Xing, G. 2013. Comparisons of biochar properties from wood material and crop residues at different temperatures and residence times. Energie Fuel. 27, pp. 5890-5899. DOI: <https://doi.org/10.1021/ef400972z>
- Zhang, X., Zhang, P., Yuan, X., Li, Y., Han, L. 2020. Effect of pyrolysis temperature and correlation analysis on the yield and physicochemical properties of crop residue biochar. Bioresource Technology. 296, pp. 1-8. DOI: <https://doi.org/10.1016/j.biortech.2019.122318>
- Zhao, B., O'Connor, D., Zhang, J., Peng, T., Shen, Z., Tsang, D.C.W., Hou, D. 2018. Effect of pyrolysis temperature, heating rate, and residence time on rapeseed stem derived biochar. Journal of Cleaner Production. 174, pp. 977-987. DOI: <https://doi.org/10.1016/j.jclepro.2017.11.013>
