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Abstract

Activated carbon is a porous material with several industrial applications. In this research, brazil nut shell was used to produce activated carbon, a promising raw material based on its low cost and high quality. Also, nut processing generates an extensive amount of waste, requiring proper disposal. The carbon was produced at temperatures of 600, 700, and 800 °C and activated by physical method using steam or CO₂. Yield was calculated, and the activated carbon was analyzed according to the following physical-chemical parameters: water content, volatile material, fixed carbon, ash, apparent density, and pH. Infrared spectroscopy and BET adsorption isotherms were also performed. The fixed carbon contents of the carbons produced at 700 and 800 °C remained above 84%. The pH values characterized them as basic. The surface areas of the carbon obtained at 800 °C were $397 \pm 8 \text{ m}^2 \text{ g}^{-1}$ and $325 \pm 7 \text{ m}^2 \text{ g}^{-1}$ in those which were activated by steam and CO₂, respectively.

Keywords: Activated carbon. Activation method. Adsorption.

1. Introduction

Bertholletia excelsa is popularly known as brazil nut tree and it is found in Amazon biome, in countries like Bolivia, Brazil, Ecuador, France and English Guianas, Peru and Venezuela (Ferreira and Tonini 2009). Its almond stands out for its market potential, sustaining innumerable communities in its regional economies (Salomão 2009).

As a seasonal product, the income generated by its extraction is concentrated during the harvest season, so an alternative income is necessary during the off-season period to guarantee the sustenance of the families that survive from this activity (Nogueira et al. 2014). Adding value to residues is an alternative for this period, like transforming shells into a valuable product.

The production of activated carbon is an alternative use of organic residues with high content of lignin, that will provide a high fixed carbon content, an essential characteristic to a good activate carbon. Low inorganic material content (ash), low volatile concentration and low cost are also important characteristics (Ilomuanya et al. 2017). Materials with high carbon content as agricultural residues (Yang

and Lua 2003; Shoaib and Al-Swaidan 2015), biomass (Vale et al. 2011) and industrial residues (Bouchelta et al. 2008) have been used for the activated carbon production. Brazil nut shell presents good characteristics to be used as a precursor for the production of activated carbon, besides being a residue widely available in the producing regions and not currently exploited in high value-added activities (Silva et al. 2022).

The activation also provides an increase on the porosity of the conventional carbon, which enables it to several uses, such as in health care (Vaziri et al. 2013; Ali et al. 2014; Melchior et al. 2017), skin care (Kulkarni et al. 2018), filtering (Nowicki et al. 2015) and others.

Different activation methods can be applied to the production of activated carbon, with emphasis on physical and chemical methods. Chemical activation is currently the most explored by scientists, but its high cost compared to physical methods, besides the need for an additional washing step, means that its application on commercial scale is unfavorable (Rimoli et al. 2019).

In this research, the aim was to evaluate Brazil nut shell as a precursor for carbon activated by physical methods, indicating the best carbonization/activation condition to its production.

2. Material and Methods

The Brazil nut shells were obtained from a processing industry located in Municipality of Sinop, central-northern region of Mato Grosso State, Brazil. Initially, the samples were washed in running water and then dried in the oven (Solab, SL-102) under forced circulation of air at 105 °C ± 3 °C until complete drying.

The experiments were developed in quintuplicate. Carbon were produced at 600, 700 and 800 °C and activated by the sample's exposition to atmosphere saturated by steam or CO₂, inert gases.

For carbon production, samples composed by 300 ± 5g of clean and dried shells were placed into a metal crucible measuring 340 mm of length and 68 mm of internal diameter which was heated in a furnace (Fortelab model T- 1200 /H-1z) at a rate of 10 °C.min⁻¹ until reaching the temperatures of 600 °C, 700 °C e 800 °C. After reaching the set temperature, the material was maintained at this condition for 30 minutes to ensure carbonization.

Then, steam at 1 kgf.cm⁻² of pressure, produced by a vertical boiler (EIT) was injected into the metal crucible through the furnace for 40 minutes, physically activating the carbon,

The remaining samples were carbonized at each set temperature followed by CO₂ activation at a pressure of 1 kgf.cm⁻², also for 40 minutes.

After activation, the carbon was cooled inside de crucible by natural convection for one hour. The samples were taken from the crucible, weighed, and packed in closed containers.

Activated carbons were analyzed by the following physicochemical parameters: yield, water content, volatile material, ash and fixed carbon content (ASTM D1762-84 2007). Apparent density was determined according to the procedures set by ASTM D2854-09 (ASTM D2854-09 2009), pH and bulk density were determined according to ASTM D3838-80 (ASTM D3838-80 1999).

All physicochemical data were submitted to Kolmogorov-Smirnov normality test, followed by the Kruskal-Wallis no parametric test and compared to the Simes-Hochberg post-test (p > 0.05), using Action Stat[®] software (version 3.7, Estatcamp Team (2014).

Fourier transform infrared spectroscopy (FTIR) from 4000 to 400 cm⁻¹ (Perking Elmer Spectrum BX FT-IR System) was performed to identify the functional groups present in the sample (Rimoli et al. 2019).

The specific surface area of activated carbons were estimated by the Brunauer, Emmett and Teller method (BET). The samples were preheated at 120 °C under vacuum for 12 hours and the nitrogen adsorption isotherms obtained at the boiling temperature of the nitrogen (Micromeritics Tristar II Kr 3020) (Sing et al. 1985; Rimolli et al. 2019).

Activated carbon yield was determined by weighing the precursor material that filled the container and the material removed from the oven, after activation. The result was expressed as a percentage in wet basis (Rimolli et al. 2019).

3. Results

Different yields were found among the temperatures ($p < 0.05$), for steam and CO₂, they were inversely proportional to the carbonization temperatures. Comparing yield between the atmospheres at the same temperature, the results for 600 °C were similar ($X^2 = 3.15$, $p = 0.76$). At 700 e 800 °C, carbons activated under atmospheres saturated by CO₂ got higher yield than those activated by steam (Table 1). There was no difference for the water content of activated carbons at any temperature. However, comparing the results for the activation atmosphere at the same temperature, steam resulted in higher water content values, excepted for the temperature of 800 °C ($X^2 = 0.01$; $p = 0.91$) (Table 1).

The CO₂ activation caused the highest value of volatile material at of 600 °C, but at 800 °C the results were similar for both atmospheres ($X^2 = 2.45$; $p = 0.11$). There was an inverse proportionality for volatile material content and temperature for both atmospheres (Table 1).

Ash content increased as the temperature rose, either for CO₂ or steam. Comparing the results between atmospheres at the same temperature, there were no significant differences ($X^2 = 0.27$; $p = 0.60$) (Table 1).

Temperature positively influenced results for fixed carbon. The highest record for fixed carbon was observed at 800 °C, regardless of the atmosphere ($X^2 = 0.53$; $p = 0.46$) (Table 1).

Samples of carbon activated by steam presented an increase in their pH values as the temperature rose. Regardless of activation atmosphere, at the same temperature, pH values were similar ($X^2 = 1.84$; $p = 0.17$) (Table 1). All the samples presented alkaline characteristic.

There were no differences for apparent density, neither for atmosphere ($X^2 = 0.50$; $p = 0.77$) or temperature ($X^2 = 1.32$; $p = 0.25$) (Table 1).

Table 1. Mean values \pm standard error for physical-chemical analysis of the carbons activated by steam or CO₂.

Temperature (°C)	Steam		CO ₂		p^1
	Yield (%)				
600	36.75 \pm 0.88 a		38.63 \pm 0.47 a		0.076
700	32.04 \pm 0.23 b		34.70 \pm 0.03 b		0.009
800	26.82 \pm 0.11 c		31.53 \pm 0.08 c		0.009
Water Content (%)					
600	1.99 \pm 0.45 ab		0.50 \pm 0.20 a		0.016
700	1.28 \pm 0.12 b		0.54 \pm 0.14 a		0.009
800	2.14 \pm 0.24 a		1.81 \pm 0.31 b		0.917
Volatile Material (%)					
600	13.97 \pm 0.27 a		16.63 \pm 0.42 a		0.009
700	08.90 \pm 0.14 b		10.78 \pm 0.20 b		0.009
800	08.80 \pm 0.32 b		09.18 \pm 0.09 c		0.117
Ash Content (%)					
600	4.07 \pm 0.34 a		3.82 \pm 0.03 a		0.601
700	4.06 \pm 0.07 a		4.09 \pm 0.04 b		0.601
800	4.96 \pm 0.21 b		4.59 \pm 0.08 c		0.347
Fixed Carbon (%)					
600	79.95 \pm 0.33 a		79.02 \pm 0.41 a		0.117
700	85.74 \pm 0.19 b		84.57 \pm 0.31 b		0.009
800	84.09 \pm 0.36 c		84.40 \pm 0.35 b		0.464
pH					
600	7.64 \pm 0.09 a		7.84 \pm 0.43 a		0.174
700	8.23 \pm 0.06 b		8.09 \pm 0.43 a		0.114
800	8.54 \pm 0.06 c		8.40 \pm 0.36 a		0.174
Apparent Density (g cm ⁻³)					
600	0.25 \pm 0.033 a		0.21 \pm 0.010 a		0.075
700	0.23 \pm 0.010 a		0.23 \pm 0.015 a		0.601
800	0.23 \pm 0.013 a		0.23 \pm 0.006 a		0.250

*Means followed by the same letter in the column do not differ by the Simes-Hochberg test with $p > 0.05$. ¹H₀: $p_{\text{steam}} = p_{\text{CO}_2}$ e H₁: $p_{\text{steam}} \neq p_{\text{CO}_2}$.

The spectrum on infrared region (IR) for brazil nut shell showed vibrational bands at 2360, 2342, 1606, 1030 and 665 cm⁻¹ (Figure 1). For carbon activated by steam, there were noises between 4000 and 3600 cm⁻¹, as well as important peaks at wave numbers near do 2900 cm⁻¹ for the higher temperatures. For all temperatures, there were vibrational bands near to 2360, 2340, 1560 cm⁻¹. Peaks of 1160 and 1148 were verified at 700 and 800°C.

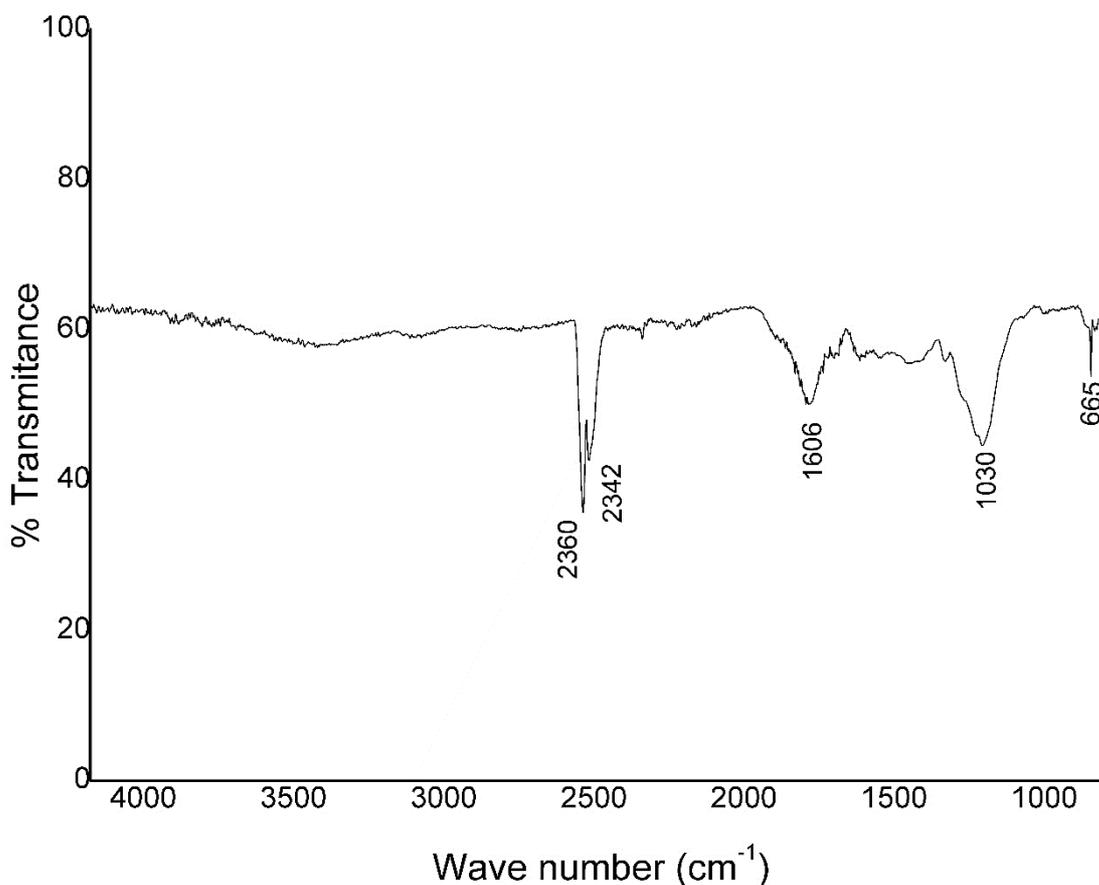


Figure 1. Infrared spectrum of brazil nut shell.

Other important functional groups could be verified by peaks between 950 and 700 for all temperatures (Figure 2A). Samples of carbon activated by CO₂ showed noises between 4000 and 3500 cm⁻¹, for all temperatures. Peaks near to 2900 cm⁻¹ disappeared as the temperature increased. Vibrational bands between 2200 and 1900 cm⁻¹ were checked for samples activated at temperatures of 700 and 800°C. A region marked by many noises at wave numbers near to 1500 cm⁻¹ were observed for all temperatures as well as vibrational bands between 900 and 700 cm⁻¹ (Figure 2 B).

The specific surface area of the carbons activated by steam were higher compared to those obtained from samples of carbon activated by CO₂, for all considered temperature (Table 2).

Table 2. Surface area of carbons produced at 600, 700 e 800 °C, activated by steam or CO₂

Activation	Temperature (°C)	Surface Area (m ² g ⁻¹)
Water Vapor	600	141 ± 4
	700	338 ± 7
	800	397 ± 8
CO ₂	600	90 ± 3
	700	199 ± 5
	800	325 ± 7

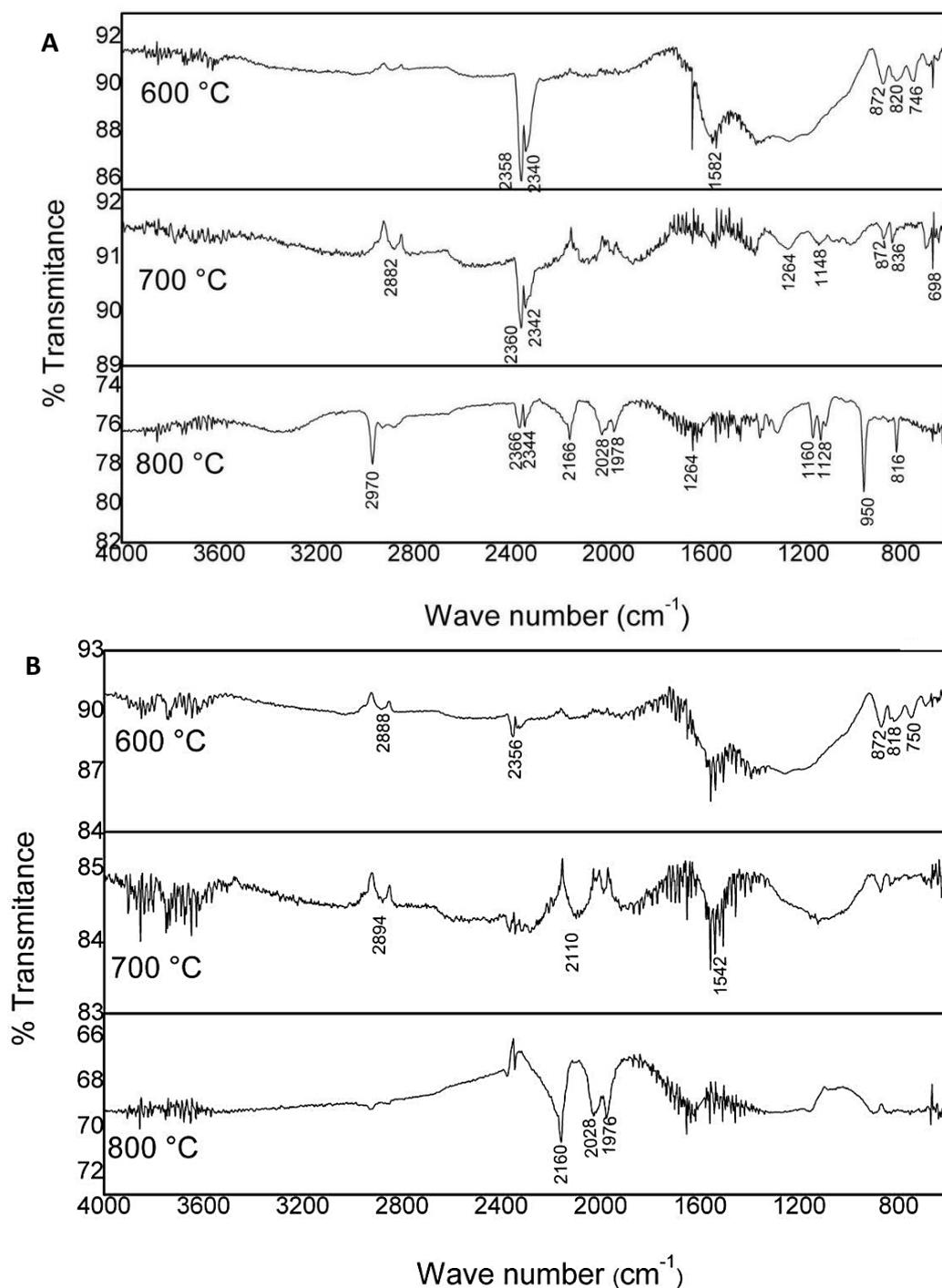


Figure 2. A - Infrared spectrum of carbons activated with water vapor and B - Infrared spectrum of carbons activated with CO_2 .

N_2 sorption/adsorption isotherms showed an increase in the adsorption capacity of the carbons as the temperature of activation rose. For carbon activated by steam (Figure 3A), the adsorption rates were higher than those obtained for carbons activated by CO_2 at the same pressure, regardless the temperature (Figure 3B).

4. Discussion

The raise on the temperature increased the evaporation of volatile materials and the burning of carbon, explaining the decrease in yield values as the temperature rose (Shoib and Al-Swaidan 2015). The yields calculated for activated carbon from brazil nut shell were similar than those obtained for different precursors, like coffee bean's activated carbon which was activated by physical methods and presented yields from 35 to 50% (Aznar 2011) or activated carbon from pecan nut shell, which got a yield of 26% (Ahmedna et al. 2000). Carbon from brazil nut shell, thermally activated at 800 °C, presented a yield of

28% (Melo et al. 2015), whereas activated carbon from the fruit of brazil nut, produced at the same conditions of these research, obtained yield values from 21 to 27% (Rimoli et al. 2019).

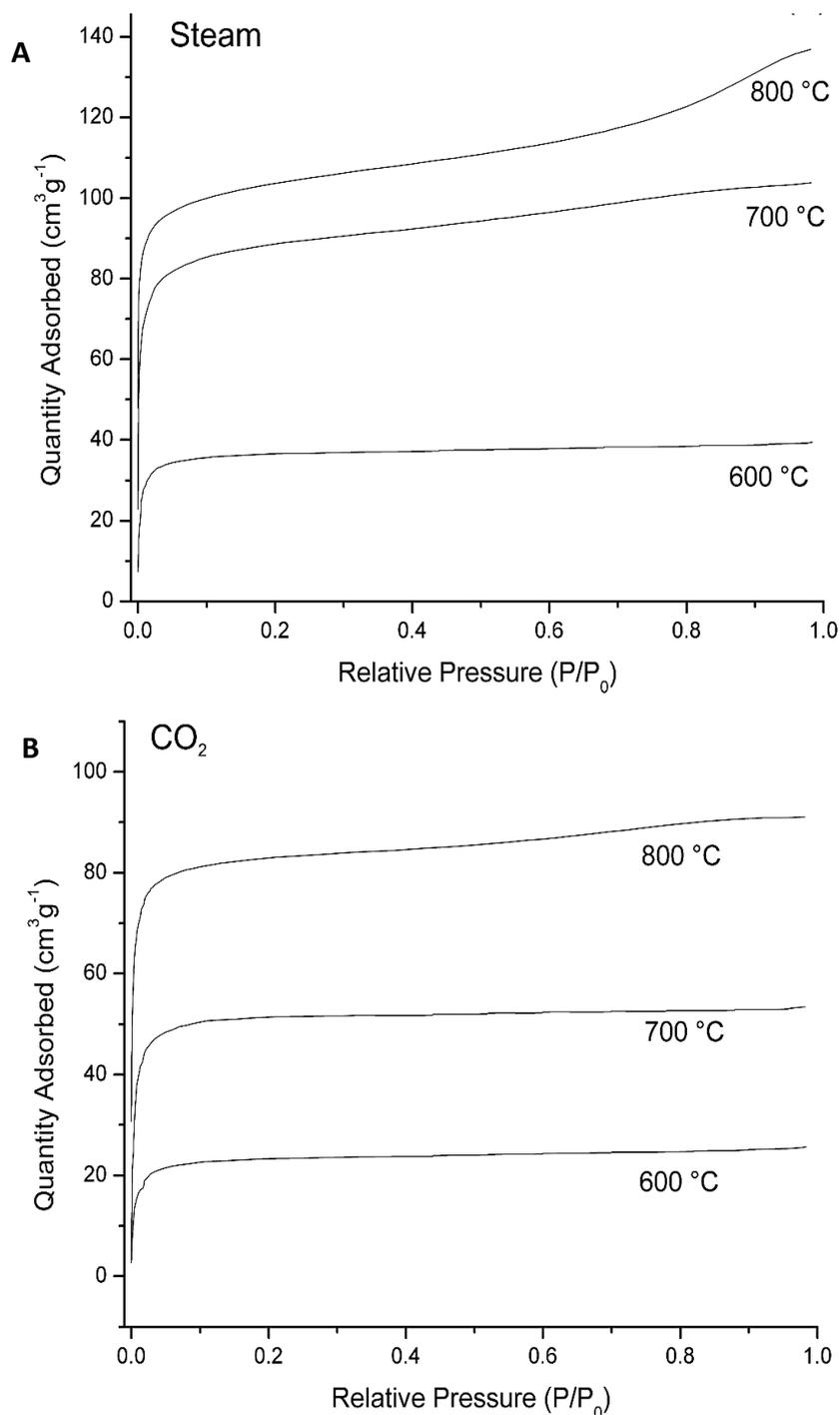


Figure 3. (A) Amount of gas adsorbed on activated carbon by water vapor using Brunauer, Emmett and Teller (BET) method. (B) Amount of gas adsorbed on activated carbon by CO₂ using Brunauer, Emmett and Teller (BET) method.

Lower water contents for higher temperatures may be explained by the fact of the temperature acts on increasing the porous formation during the carbonization or activation, contributing to the hygroscopicity of the carbon (Boas et al. 2010). Besides the temperature, the atmosphere of activation may contribute to porous formation, later to hygroscopicity. Steam is more effective than CO₂ in increasing the specific surface area of the carbon, which means more hygroscopicity (Dias Júnior et al. 2016), whether for the higher oxidizing power of the steam versus CO₂, or the atmosphere pressure during activation, which were higher for steam comparing to CO₂ in this research. Water content were higher for different precursors activated in similar conditions, as for buriti seed (*Mauritia flexuosa*) (3.72%) (Pinto et al. 2013),

cupuaçu (*Theobroma grandiflorum*) (8.2%), brazil nut woody capsule (*Bertholletia excelsa*) (7%). Water content lower than 8% is desirable to activated carbons (Ilomuanya et al. 2017).

Volatile materials represent organic substances in the precursor which can be evaporated by heating it. Thus, after carbonization and activation process, a low residual volatile material content in samples of carbon is expected, considering the high temperature to which the precursor is submitted (Kwiatkowski and Broniek 2017). Values from 12 to 19% were reported for activated carbons produced from pistachio shells, activated by CO₂ at temperatures between 725 and 825 °C (Yang and Lua 2003). Activated carbons produced from the fruit of brazil nut, at the same conditions than those used in this research, presented lower results, varying from 5.83 to 11.10%, consistent with those got in this research (Rimoli et al. 2019).

Results for the ash content comprise the inorganic material remained after carbonization, being mainly related to the composition of the precursor, but also to the process of carbonization and activation (Silva and Brito 1990; Yang and Lua 2003). The presence of a non-carbonic mineral may implicate on a reduction of adsorption capacity for the interaction between carbon surface and the mineral which compose ash (Rimoli et al. 2019). Similar results for ash content were reported for activated carbon from the same precursor, brazil nut shell (*Bertholletia excelsa*), presented an ash content of 4.84% (Melo et al. 2015). Values of 6.80, 3.10 and 5.90% were showed for carbons from other products, like cupuaçu (*Theobroma grandiflorum*), brazil nut fruit (*Bertholletia excelsa*) respectively (Junior 2010). The results for this research indicate outstanding quality activated carbons (Ilomuanya et al. 2017).

The temperature of carbonization and activation process is decisive to define the carbon fixation (Róz et al. 2015). Carbon fixation guarantees a formation of a complex matrix that allows surficial adsorption or the generation of functional groups, serving as a microscopic filter (Aznar 2011). As higher is the detachment of condensable and non-condensable gases during carbonization or activation process, higher levels of fixed carbon are reached, which is desirable for quality of activated carbon (Sekirifa et al. 2013; Ilomuanya et al. 2017).

The pH above 7.0 characterizes the carbon as basic and this parameter is directly related to the use of carbon as a filtering agent, capable of retaining compounds with the opposite charge. So, acid activated carbon favors the adsorption of basic compounds, whereas basic activated carbon is suitable for the adsorption of acidic compounds (Yang and Lua 2003). The high temperature of activation can explain the fact of the adsorbent develops basic oxides on its surface (Melo et al. 2015).

The apparent density is related to the yield, porous formation, and evaporation of volatile material, in other word, it is close related to the carbonization and activation process. The results obtained in this study are similar to those got for different precursors carbonized and activated by similar conditions. Similar results were observed for activated carbon from pistachio shell (Yang and Lua, 2003) and palm trunk (Ahmad et al. 2007), where higher yields were obtained with lower carbonization temperatures, independent of the activation atmosphere. Whereas the biomass composition in general, yielded about 30 % are considered regular (Nobre et al. 2015). Also studies showed volatile content higher than 11% under similar conditions of carbonization and biomass activation (Yang and Lua, 2003; Ahmad et al. 2007; Róz et al. 2015).

The infrared spectrum from raw brazil nut shell shows peaks at 2360 and 2342 cm⁻¹, indicating the presence of triple bond between carbons (C≡C) or between carbon and nitrogen (C≡N). The peak at 1606 cm⁻¹ may indicate the presence of aromatic ring and double bond between carbon and oxygen (C=O), while the peak at 1030 cm⁻¹ indicates the presence of an OH in the surface of carbon (Silverstein et al. 2005). The small number of functional groups is characteristic for raw materials. The exposure of temperature and oxidizing atmosphere during the carbonization and activation process allowing the formation of complex between nitrogen, carbon or oxygen atoms with the structure of the original material.

Carbon activated by steam presented noises between 4000 and 3800 cm⁻¹ characteristics of free OH from water. The appearance and increase in peak intensity for wave numbers between 3000 and 2880 cm⁻¹, which was observed for the increasing on the temperature, is characteristic of aliphatic CH bond (Saka 2012). The vibration stretching in alkyne group at bands between 2366 and 2340 cm⁻¹ decreases its intensity with the increasing of the temperature. Groups that IR peaks are between 2200 and 1975 cm⁻¹ present triple bond between carbons. The vibration stretching in 1160 and 1128 cm⁻¹ is a region associate

to alcohol groups. The presence of peaks between 880 and 740 cm⁻¹ for lower temperatures is related to C-H out of the plane in aldehydes -CHO, compounds of pyranose or other benzene derivatives. However, a band at 816 cm⁻¹ and the insertion of a band at 950 cm⁻¹ based on the increase of the temperature can be due to the presence of ethers.

The spectrum for carbons activated by CO₂ presented noises between 3928 and 3571 cm⁻¹ for all tested temperatures, indicating free OH from water. In the region between 2800 and 3000 cm⁻¹, characteristic of carbon double bond (C=C) there are peaks for samples activated at 600 and 700 °C, but it disappears as the temperature rises to 800 °C, indicating the breaking of this bond at this temperature. The region between 1970 and 2200 cm⁻¹ indicate the presence of alkyne (C≡C) or triple bond between carbon and nitrogen (C≡N). Bands in the region between 872 and 750 cm⁻¹ are characteristic for the presence of alkenes (C=C) or aromatic rings. The disappearance of any peaks indicates the breakage of the bonds (Gomez-Serrano et al. 1996).

The formation of functional groups in the surface of carbon begins at temperatures around 600 °C. When the temperature reaches 700 °C, most of acid functional groups disappear, while the basic functional groups are formed extensively (Liu et al. 2013; Shafeeyan et al. 2010). There was a reduction of carboxyl groups with the increasing of the temperature, indicating the removal of oxygen from the surface of the material. During pyrolysis, oxygenated compounds and hydrocarbons of low molecular weight are released (Borges et al. 2015).

The degradation of organic compounds due to the temperature of exposure may explain the increase of the surface areas, regardless the activation atmosphere. It favors the expulsion of volatile materials (Saka 2012), as well as the burning of the carbon helps to improve the surficial area (Yang and Lua 2003). Higher pressures are responsible for the compounds removal from carbon during activation, which can explain the better results for carbon activated by steam in comparison to those obtained from samples activated by CO₂.

From BET analysis, the microporous profile was confirmed for both activation process, since the isotherms were type I, typical for microporous materials with external surfaces relatively small, as well as the higher adsorptions volume of N₂ under low pressure (Rimoli et al. 2019). Micropores confer the capacity of the activated carbon to adsorb small organic molecules and common solvents. This type of isotherm is typical for carbon produced from biomass (Yang and Lua 2003).

5. Conclusions

Based on the analyzed parameters, the carbon produced from brazil nut shell had high quality. Regardless of the activation atmosphere, the temperature of 800 °C generates important characteristics to the activated carbon, such as high surface area and the presence of functional groups that confers basicity to it. Activated carbon from brazil nut shell appears as a promising adsorbent, either for physisorption or chemisorption of acid molecules. For industrial production of activated carbon from brazil nut shell, steam appears more suitable than CO₂, by economic reasons, since the technical analysis indicates similarity between them.

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