






PREPARATION AND CHARACTERIZATION OF CARBON FROM THE FRUIT OF BRAZIL NUT TREE ACTIVATED BY PHYSICAL PROCESS

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ABSTRACT – Activated carbon is the name of a big group of materials that presents high degree of porosity and, consequently, an extended internal surface area, with physical and chemical adsorption properties. Innumerable raw materials can be used as precursors, especially biomass. The objective of this study was to obtain activated carbon from physical activation of the fruit of Brazil nut tree (“ouríço”) and to evaluate its physical and chemical properties in function of the differences between the temperatures and atmospheres of activation. The samples were carbonized at 3 different temperatures and the carbons were activated under atmosphere saturated by CO₂ or steam. The results showed the influence of activation temperature and atmosphere on physicochemical characteristics of carbon. The carbon from woody Brazil nut seed capsule activated by CO₂ and steam at different temperatures had a microporous profile, indicating its use to adsorb organic molecules of small dimensions. Basic characteristic was observed on samples and the carbon produced was thermally stable. Best quality was attributed to carbon activated by steam at 800 °C.

Keywords: Activated carbon; Adsorption; Activation method.

PREPARAÇÃO E CARATERIZAÇÃO DO CARVÃO DO FRUTO DA CASTANHEIRA-DO-BRASIL ATIVADO POR PROCESSOS FÍSICOS

RESUMO – Carvão ativado é o nome de um grande grupo de materiais que apresentam alto grau de porosidade e, conseqüentemente, extensa área superficial interna, com propriedades adsorptivas de caráter físico e químico. Inúmeros materiais podem ser utilizados como precursores, especialmente biomassa. O objetivo deste trabalho foi obter carvão ativado a partir da ativação física do fruto da castanheira-do-brasil (“ouríço”) e avaliar suas propriedades físico químicas em função das diferenças entre as temperaturas e atmosferas de ativação. As amostras foram carbonizadas em 3 diferentes temperaturas e o carvão produzido foi ativado sob atmosfera saturada por CO₂ e vapor d’água. Os resultados mostraram a influência da temperatura e atmosfera de ativação nas propriedades físico-químicas do carvão. O carvão do ouríço ativado por CO₂ e por vapor apresentou perfil microporoso, indicando seu uso como adsorvente de moléculas orgânicas de pequenas dimensões. Foi observado um caráter básico nas amostras e o carvão produzido foi termicamente estável. Melhor qualidade foi atribuída ao carvão ativado por vapor d’água a 800 °C.

Palavras-chave: Carvão ativado; Adsorção; Ativação.



1. INTRODUCTION

Activated carbon is the name for a big group of porous materials based on a carbonic matrix. Carbonaceous materials are carbonized after their impregnation with chemicals, it is called chemical activation; or a char is treated with oxidizing gases, a process named as physical activation. These conditions accord a high degree of porosity and, consequently, an extended internal surface area, withal physical and chemical adsorption properties (Rodríguez-Reinoso and Silvestre-Albero, 2016).

The main differences between activation process are: carbonization and activation are unseparated steps during chemical activation, whereas they are independent during physical activation (Prauchner and Rodríguez-Reinoso 2012). Physical activation presents several advantages and disadvantages compared to chemical activation. The main advantages are the low cost, considering there is no need of acquiring activating agents, such as acid or basic chemicals, and there is no need of an additional washing stage. Moreover, these chemicals can be corrosive (Maciá-Agulló et al., 2004).

The characteristics of activated carbons are due to the precursor material and the activation method (Bhatnagar and Sillanpää, 2010). A plenty of lignocellulosic materials were employed as precursors (Ahmad et al., 2007; Daud et al., 2000; Jaguaribe et al., 2005; Melo et al., 2015; Tsai et al., 2001). However, other materials such as animal bones and petroleum coke can also be used (Djilani et al., 2016; Kawano et al., 2008).

The woody Brazil nut (*Bertholletia excelsa*) seed capsule has high lignocellulose content (Rambo et al., 2015; Scussel et al., 2014) which is an important feature for obtaining a high quality carbon (Yang et al., 2007). As a byproduct, it has low cost, which, added to the lower cost of physical activation, may produce a cheaper activated carbon, to be used: in the treatment of water supply (Borges et al., 2016), in medical and environmental applications (Alkhatib and Zailaey, 2015), in natural gas storage (Gottipati et al., 2012), in the filtering of compounds used in agricultural activity (Melo et al., 2015), and others.

In the present study, activated carbon was obtained from physical activation of woody Brazil nut

seed capsule. Its physical and chemical properties were characterized in function of the differences between the temperatures and atmospheres of activation.

2. MATERIAL AND METHODS

Samples of woody Brazil nut seed capsule were fragmented into 2 to 3 cm pieces and then washed in running water and kept in an oven with forced air circulation at 105 °C for 24 hours for drying.

Cleaned and dried material was fitted inside a metal cylindrical container (length: 720 mm; internal diameter: 68 mm) and the filled container was placed in a tubular oven (FT-1200/H1Z, Fortelab, São Carlos, SP, Brazil) with heat rate of 10 °C/min. The oven was adjusted to the pyrolysis temperature and the samples were maintained at these temperatures for five hours. The tested temperatures were 600 °C, 700 °C and 800 °C. Shortly after the pyrolysis of the material, the atmosphere was saturated with an oxidizing agent for 40 minutes, for the activation of the carbon. The tested agents were CO₂ (pressure of 0.58 kgf.cm⁻²) and steam at 1 kgf.cm⁻² from a vertical boiler (EIT-VL, EIT, Paçandu, PR, Brazil).

Water, volatile, ash and fixed carbon contents were determined according to the method standardized by ASTM D 1762-84 (ASTM D 1762-84, 2007). The results were expressed as a percentage in dry basis, except for water content which was expressed as a percentage in wet basis. The pH values were measured using the standard test method ASTM D 3838-80 (ASTM D 3838-80, 1999) and bulk density by the standard test method ASTM D 2854-09 (ASTM D 2854-09, 2009). 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.

The absorption bands in the infrared region of the activated carbon samples were determined in a Fourier transform (FTIR) spectrometer equipped with an attenuated total reflectance (ATR), (Spectrum BXII, Perkin Elmer, Billerica, MA, USA) in the region between 4000 and 400 cm⁻¹, with resolution of 16 cm⁻¹.

Samples of activated carbon were submitted to BET (Brunauer, Emmett e Teller) analysis (Tristar II Kr 3020, Micromeritics, Norcross, GA, USA) to

determine the specific surface area. The distribution for microporous size was analyzed by DFT (Density Functional Theory) method.

The thermogravimetric analysis (TGA) curves were determined in a thermogravimetric analyzer (SDT Q600, TA Instruments, New Castle, DE, USA), only for the samples that presented the smallest contents of water and volatile and the highest yield, pH and content of fixed carbon, for both atmospheres. The explored temperature ranged from 20 °C to 1000 °C at a heat rate of 10 °C.min⁻¹, under atmosphere of ultrapure air (100 mL.min⁻¹).

A full 2³ factorial coupled with statistical analysis of the results, by using analysis of variance (ANOVA)

was carried using Action Stat Pro (ESTATCAMP and DIGUP, 2017). Five replications were used for all the physicochemical properties analyzed.

3. RESULTS

The temperature influenced the yield (F= 51.69; p< 0.05), pH (F= 35.95; p< 0.05), volatiles content (F= 123.04; p< 0.05), ash content (F= 8.56; p< 0.05) and fixed carbon (F= 95.51; p< 0.05). There was no significant interaction between temperature and atmospheres of activation for any parameters. Physicochemical composition data of the activated carbon followed normal distribution, by Anderson-Darling testing (Table 1).

Table 1 – Yield, Water content, Volatile, Ash, Fixed carbon, Bulk density and pH for samples activated by CO₂ and steam at temperatures of 600, 700 and 800 °C

Tabela 1 – Rendimento, teor de água, voláteis, cinzas, carbono fixo, massa específica e pH para amostras ativadas por CO₂ e vapor a temperatura de 600, 700 e 800 °C

Temperature (°C)	CO ₂	Steam	p1
Yield (%)			
600	26.38±0.51 a	25.82±0.36 a	0.4004
700	23.78±0.46 b	24.48±0.46 a	0.3135
800	21.86±0.42 c	21.34±0.43 b	0.4126
Water content (%)			
600	1.53±0.09 a	0.48±0.05 a	< 0.001
700	1.55±0.18 a	0.58±0.20 a	0.0072
800	1.24±0.28 a	0.45±0.08 a	0.0284
Volatile (%)			
600	11.10±0.41 a	10.26±0.43 a	0.1935
700	7.47±0.32 b	6.96±0.14 b	0.1954
800	6.42±0.18 b	5.83±0.19 c	0.0588
Ash (%)			
600	2.08±0.12 a	1.72±0.06 a	0.0308
700	1.79±0.08 a	1.69±0.09 a	0.4859
800	2.12±0.07 a	2.09±0.08 b	0.7813
Fixed carbon (%)			
600	85.27±0.42 a	87.53±0.40 a	0.0044
700	89.18±0.44 b	90.74±0.30 b	0.0198
800	90.20±0.17 b	91.61±0.24 b	0.0014
Bulk density (g.cm ₋₃)			
600	0.24±0.01a	0.24±0.01 a	0.4323
700	0.25±0.01a	0.25±0.01 a	0.7355
800	0.25±0.01 a	0.23±0.02 a	0.0946
pH			
600	9.00±0.08 a	9.17±0.04 a	0.1302
700	9.10±0.06 a	9.14±0.08 a	0.1302
800	9.58±0.04 b	9.64±0.08 b	0.4938

*Means followed by the same letter in the column, for the same parameter, don't differ by Tukey test at p<0.05.

¹H₀: μ_{t, vapor} = e H₁: μ_{t, vapor} ≠ μ_{t, CO₂}

Higher results for yield were obtained from carbon activated by CO₂ at 600 °C, and from those activated by steam at 600 °C and 700 °C. Smaller contents of volatile were acquired at temperatures of 600 °C and 700 °C for the activation with CO₂, and at 800 °C by steam activation. Steam at 800 °C allows higher ash contents. For fixed carbon, either CO₂ and steam at 700 °C and 800 °C allowed higher results. The temperature of 800 °C, for both activation atmospheres, allowed greater basicity to samples (Table 1).

The activation by steam, regardless of the temperature, allowed smaller water contents, compared to those activated by CO₂. The results for ash content of samples activated by steam and CO₂, were different only for the temperature of 600 °C. Steam allowed higher fixed carbon under any temperature (Table 1).

The spectrum on infrared region (IR) for precursor showed vibrational bands at 3348, 2924, 1736, 1654, 1594, 1510, 1466, 1416, 1370, 1330, 1228, 1160, 1107, 1026 and 668 cm⁻¹. For carbon activated by CO₂, there were vibrational bands at 2884 cm⁻¹ for all temperatures; at 1584 cm⁻¹ only for temperatures of 600 and 700 °C; 1134 cm⁻¹ for 700 °C and at 984 and 1563 cm⁻¹ for temperature of 800 °C (Figure 1a). For carbon activated by steam, there were vibrational bands at 2928 cm⁻¹ for 600 e 800 °C; 953 cm⁻¹ for 700 and 800 °C; 1127 cm⁻¹ for 800 °C; 2891 cm⁻¹ for 700 °C; 920, 847 and 771 cm⁻¹ for temperature of 600 °C (Figure 1b).

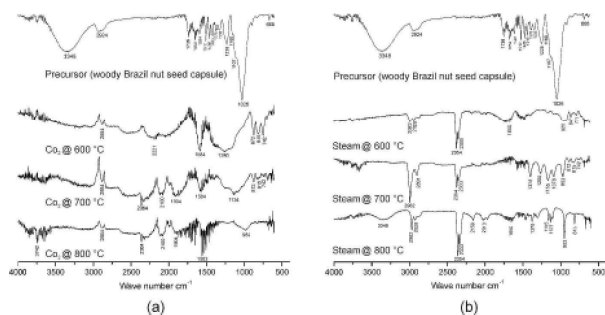


Figure 1 – Spectrum on infrared region for precursor material, carbon activated by CO₂ at any temperature (a) and carbon activated by steam at any temperature (b).

Figura 1 – Espectro na região do infravermelho para o material precursor; carvão ativado por CO₂ em todas as temperaturas (a) e carvão ativado por vapor em todas as temperaturas (b).

The specific surface area for samples activated by CO₂ at 600, 700 and 800 °C were 15.8, 55.0 and 395.0 m²g⁻¹, respectively. From steam activation the results were 125.0, 320.0 and 401.0 m²g⁻¹, for samples activated at 600, 700 and 800 °C, respectively.

The N₂ sorption/desorption isotherm for carbon activated by both atmospheres showed high adsorption of N₂ under low pressures (Figure 2 a and b) and higher intensity of porous of 1.16, 1.17 and 1.17 nm, for temperatures of 600, 700 and 800 °C, respectively (Figure 3a). For the samples activated by steam at 600 °C, the porous of 1.26 nm was the most founded, followed by 1.0 and 1.7 nm for samples activated at 700 and 800 °C for the same atmosphere, respectively (Figure 3b).

The thermogravimetric analysis (TGA) curves, for both activation methods, showed two inflection points defining three ranges for mass loss. Considering

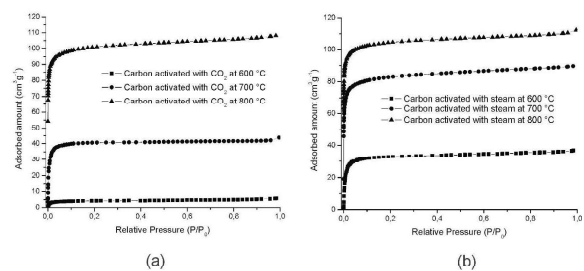


Figure 2 – Isotherm of sorption/desorption of N₂ at 120 °C for carbon activated by CO₂ (a) and steam (b) at any temperatures.

Figura 2 – Isotermas de sorção/dessorção de N₂ a 120 °C para o carvão ativado por CO₂ (a) e vapor (b) em todas as temperaturas.

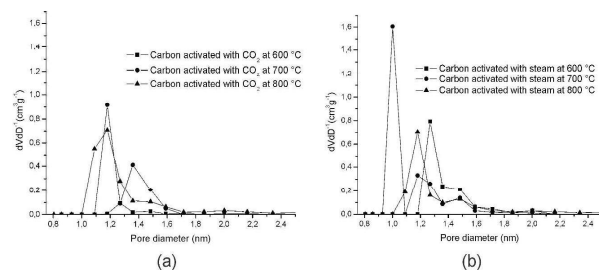


Figure 3 – Distribution of amount of porous diameter for carbon activated by CO₂ (a) and by steam (b) at any temperature.

Figura 3 – Distribuição dos diâmetros de poros para o carvão ativado por CO₂ (a) e por vapor (b) em todas as temperaturas.

the samples activated by CO_2 , there was a mass loss of 3.08 % up to 150 °C, 0.67 % at temperatures ranging from 600 to 700 and 8.62 % above 700 °C. For the activation by steam, mass losses were 9.79 %, 0.56 % and 16.78 % for temperatures up to 150 °C, between 600 and 700 °C and above 700 °C, respectively (Figure 4).

4. DISCUSSION

Yields were influenced by the temperature but not by the atmosphere. This fact allows to infer that yield is defined during carbonization step, when volatile is released, and high rates of mass loss are observed (Lua and Guo, 2001; Rodríguez-Reinoso et al., 1995; Yang and Lua, 2003). So, since higher temperatures are acquired, smaller yields are observed. Similar behavior was observed for activated carbon from pistachio husk (Yang and Lua, 2003) and from palm trunk (Ahmad et al., 2007), where higher yields were getting from lower temperatures of carbonization, regardless of the activation atmosphere. Whereas the composition of biomass in general, yields close to 30 % are considered fair (Nobre et al., 2015b).

Neither the temperature nor the atmosphere of activation influenced water content, since water is volatilized up to 200 °C during carbonization step (Pastor-Villegas et al., 1999). However, higher temperatures of carbonization can contribute to hygroscopicity due to increasing on porous formation (Boas et al., 2010) The hygroscopic feature of the activated carbon allows chemical-physical adsorption

of water inside the porous (Ahmad et al., 2007; Anisuzzaman et al., 2015). So, during the sample handling, water may be absorbed from air moisture. Lower water contents are desirable, since high water contents are related to decreasing on mechanic resistance of activated carbon, consequently, dust generation (Boas et al., 2010).

Releasing of volatile is due to the change to vapor phase of several molecules, by the action of the carbonization temperature (Silva and Brito, 1990). The atmosphere change during the activation step is also important for the residual volatile in the activated carbon. During this stage, the gas permeates into solid matter, contributing to desorption, distillation and removal of volatile that still existed in carbon. Also, activation gases contribute to stabilization of the radical acquired during thermal decomposition, boosting the volatile release (Ahmad et al., 2007). The formation of micro and macro pores is influenced by volatile release, which means that high volatile content in the activated carbon may imply a low surface area (Zhang et al., 2004). Volatile contents higher than 11 % were presented by other studies in similar conditions of carbonization and activation of the biomass (Ahmad et al., 2007; Róz et al., 2015; Yang and Lua, 2003), disclosing the high quality of the activated carbon from woody Brazil nut seed capsule.

Ash content of activated carbon is related to the composition of precursor and the characteristics of the process of carbonization and activation, since the latter may allow the contamination of the carbon with inert matter (Collet, 1956; Silva and Brito, 1990). Lower ash content may also contribute to indicate the quality of activated carbon (Jaguaribe et al., 2005). As a non-carbonic mineral additive, it may impair adsorption due to changes in interaction between carbon surface and the species to be adsorbed (Bautista-Toledo et al., 2005). Besides blocking pores in carbon and its hydrophilic characteristic, ash increases water adsorption rather than compounds of interest (Ahmedna et al., 2000; Brum et al., 2008). In general, commercial activated carbon presents ash contents ranging from 10% to 15% (Jaguaribe et al., 2005; Lopes et al., 2013), far above those found in this work.

High content of fixed carbon confers a matrix able to generate functional groups or surficial complex, that serves as bond sites to adsorption of compounds of

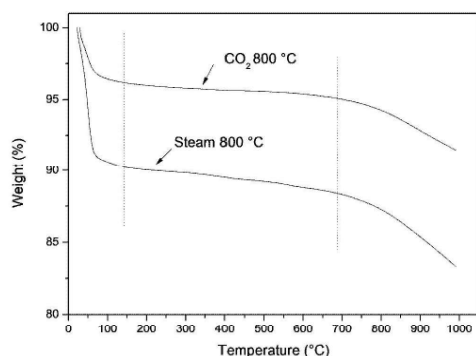


Figure 4 – TGA curves from samples of carbon activated by CO_2 and steam at 800 °C.

Figura 4 – Curvas TGA das amostras de carvão ativado por CO_2 e por vapor a 800 °C.

interest (Aznar, 2011; Mohan and Pittman Jr., 2006). Negative correlation with yield (Brito et al., 1987) and positive correlation with temperature (Róz et al., 2015) are observed during carbonization step. Increasing on temperature leads to losses of condensable and non-condensable compounds, as CO and CO₂ (Pinheiro et al., 2005; Róz et al., 2015; Yang et al., 2007), reducing yield and fixing residual carbon.

pH may indicate the presence of groups on carbon surface. High values show a low index of acid groups on activated carbon (Strelko and Malik, 2002). Complexes formed between carbonic matrix and oxygen atoms can determine the acidic or basic characteristics of the activated carbon (Aznar, 2011; Mohan and Pittman Jr., 2006; Wibowo et al., 2007). The basic characteristic of the activated carbon is due to the high temperature of activation and to the activation atmosphere. Both parameters collaborate to the breakdown of bonds in the carbonic matrix and to a new arrangement with gases that compose the atmosphere (Leon y Leon et al., 1992; Mohan and Pittman Jr., 2006; Pereira et al., 2003; Wibowo et al., 2007).

The chemical surface of the activated carbon determines the capacity to retain water, catalytic characteristics, acidic or basic feature and the capacity of adsorption (Salame and Badosz, 2001). It is related to the presence of heteroatoms (oxygen, hydrogen and nitrogen), also the atom of carbon inside the matrix (El-Sayed and Badosz, 2004; Salame and Badosz, 2001).

There was a broad band at 3348 cm⁻¹ in the precursor's spectrum (woody nut seed capsule), attributed to a vibrational stretch of hydroxyl group (O-H) linked to hydrogen for alcohols and phenols, possibly due to moisture of the sample (Ramos et al., 2009; Yang and Lua, 2003). The same band was founded for other precursors used for activated carbon production (Ramos et al., 2009). Vibrations corresponding to ν (C-H) of alkanes (CH₃ and CH₂) were observed at 2924 cm⁻¹ (Ramos et al., 2009; Yang and Lua, 2003). CH₂ and CH₃ groups are confirmed for bands at 1446 and 1370 cm⁻¹, typical for angular deformation of these groups (Gomez-Serrano et al., 1996; Jagtoyen et al., 1992; Yang and Lua, 2003). Potential olefinic ν (C=C) absorption in the sample allows signals at 1654 cm⁻¹, other two bands around 1510 and 1416 cm⁻¹ can be due to stretch of C=C in

aromatic rings (Yang and Lua, 2003). A band at 1330 cm⁻¹ can be due to ν (C=O) vibrations of carboxylated groups and C-O stretch vibration is observed between 1300 and 900 cm⁻¹. The band at 1228 cm⁻¹ and the shoulder at 1660 cm⁻¹ indicate the presence of esters (R-CO-O-R'), ethers (R-O-R') or other phenolic groups on precursor. The other shoulder observed at 1107 cm⁻¹ associated to the band at 1026 cm⁻¹ can be associated to alcohol groups (R-OH). Finally, the band at 668 cm⁻¹ represents γ (OH) (Yang and Lua, 2003).

The bands at: 2928 cm⁻¹ for carbon activated by steam at 600 and 800 °C; 2891 cm⁻¹ also for activation by steam at 700°C; and 2884 cm⁻¹ for activation by CO₂ at all temperatures, can be attributed to ether group (-O-CH₃) and/or to C-H bonds, symmetric or asymmetric, of methyl and methylene groups (Ahmad et al., 2007). Bands at 1584 cm⁻¹ for carbons activated by CO₂ at temperatures of 600 and 700 °C and at 1134 cm⁻¹ for temperature of 800 °C represent the stretch of carbonyl group in quinone, also the structure of γ -pyrone with strong vibrations of the C=O and C=C combination (Ahmad et al., 2007; Tsai et al., 2001). At 1127 cm⁻¹, the band found in the samples activated by steam at 800 °C, and at 1134 cm⁻¹ for carbon activated by CO₂ at 700 °C, represent ketones, alcohols, pyrones and aromatic deformations in C-H plan (Ahmad et al., 2007). The bands at 984 cm⁻¹ (carbon activated by CO₂ at 800 °C), 953 cm⁻¹ (carbon activated by steam at 700 and 800 °C) and 920 cm⁻¹ (carbon activated by steam at 600 °C) can be due to vibration of ethers (-C-O-C-) (Ahmad et al., 2007). The bands at 847 and 771 cm⁻¹ found in the samples activated by steam at 600 °C can be related to C-H out of the plan in aldehydes -CHO, compounds of pyranose and others benzene derivatives (Ahmad et al., 2007).

The basic characteristic of the carbon confers greater capacity of adsorption of acidic substances (Leon y Leon et al., 1992; Mohan and Pittman Jr., 2006; Pereira et al., 2003; Wibowo et al., 2007). The formation of structures of pyrone, ether and groups carbonyl contributes to the basic characteristic of the produced carbon.

The results for specific surface area, both for the carbon activated by CO₂ and by steam, are considered fair for activated carbons from wood pyrolysis (Herzog et al., 2006). That fact indicates the quality of woody

Brazil nut seed capsule as a precursor for activated carbon production, besides being a raw material of low cost, renewable and abundantly found.

Independent of the atmosphere of activation, the samples presented a microporous profile, confirmed by the higher adsorption volume of N₂ under low pressure (Sun and Webley, 2010) and by the identification of peaks at diameter smaller than 2 nm (20 Å) (Ahmad et al., 2007). Activated carbons with microporous profile provide high capacity of adsorption of organic molecules of small dimensions, with potential to retain gases and common solvents (Nobre et al., 2015a).

From BET analysis, the microporous profile was confirmed for both activation process, since the isotherms were type I, typical of microporous materials with external surfaces relatively small (Sing et al., 1985). Type I isotherms confer characteristic of chemisorption for activated carbon (Shaji and Zachariah, 2017), on that case, the adsorption is limited by the volume of accessible micropores instead of internal surface area (Sing et al., 1985). This type of isotherm is typical for carbon produced from biomass as a precursor (Oliveira et al., 2016; Yang and Lua, 2003). Pore distribution was not affected by activation atmosphere, indicating that woody Brazil nut seed capsule can be used as a precursor independent of the atmosphere used.

From thermogravimetric curves, both carbons activated by CO₂ and activated by steam, at 800 °C, presented similar thermal stability. The initial mass loss observed at temperatures up to 200 °C can be explained by water evaporation (Pastor-Villegas et al., 1999), which could have been adsorbed during the storage or handling of the samples (Oliveira et al., 2016). The second mass loss, occurred between 600 and 700 °C, could be due to the decomposition of groups formed on carbon surface during activation process (carbonyl groups, ethers, structures of pyrone) and/or also by the decomposition of the carbon skeleton (Oliveira et al., 2016). The small loss of mass over a wide temperature range provides the desired stability to activated carbon.

5. CONCLUSIONS

Temperature and atmosphere of activation had significant effects on physico-chemical characteristics

but did not present interaction between them. Samples presented a microporous profile for both atmospheres, indicating their high capacity to adsorb organic molecules of small dimensions. Pore distribution was not affected by activation atmosphere and the results for specific surface area confirmed the quality of woody Brazil nut seed capsule as precursor for activated carbon. Considering the results, activation by steam at 800 °C led an activated carbon with better quality.

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