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NEW ALTERNATIVE FOR ADSORPTION OF 2,4-DICHLOROPHENOXYACETIC ACID HERBICIDE (2,4-D)

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Abstract

The adsorptive capacity of charcoal from the husk of the brazil nut fruit, called "ouriço" (the hard ball with nuts inside) for the herbicide dichlorophenoxyacetic acid (2,4-D) was evaluated. Activated carbons were produced from the brazil nut in a tubular oven at 800 °C and activated with CO_2 or water steam. The specific surface area was determined by the Brunauer, Emmett and Teller (BET) method, demonstrating the mean density of micropores. Analysis of N_2 adsorption/desorption isotherms was undertaken and the morphology of activated carbons was visualized by Scanning Microscopy (SEM). The activated carbons were successfully obtained and had a specific surface area of 395 $m^2.g^{-1}$ and 401 $m^2.g^{-1}$ after activation with either CO_2 or water steam, respectively. The highest pore mean density occurred with a diameter of 1.17 nm for carbons activated in both atmospheres. The graph of the adsorption/desorption isotherms of N_2 showed Type I isotherms, regardless of the activation atmosphere. The SEM analysis showed that, for both activation atmospheres, pore formation occurred in the shape of uniform honeycomb craters. Adsorption kinetics followed the pseudo-second order model, indicating chemisorption. Regardless of the activation atmosphere, the activated carbon from the brazil nut "ouriço", was highly efficient for 2,4-D adsorption.

Keywords: Activated charcoal. Brazil nuts. Herbicide. Micropores.

1. Introduction

Environmental contamination by chemical compounds is a significant problem due to serious effects on human health. These compounds can be absorbed by the population orally, through water consumption (Faria et al. 2007; Salman and Hameed 2010) or food contaminated by the dermal or respiratory route (Santos et al. 2007).

Intensive pesticide use in crops is one of the main causes of environmental pollution in rural areas (Vieira et al. 1999), especially the pollution of rivers and groundwater (Aksu and Kabasakal 2004; Salman and Hameed 2010).

Among the most used herbicides worldwide: dichlorophenoxyacetic acid (2,4-D) is the third most common in the US and Canada to control broadleaf weeds (Júnior et al. 2002). In Brazil, its use stands out

in soybean crops, mainly for weed control and in pastures, due to its low cost and selectivity (Aksu et al. 2004; WHO 2017).

These characteristics corroborate epidemiological studies that suggest the association between human exposure to 2,4-D and two types of cancer: soft tissue sarcoma and non-Hodgkin's lymphoma. The World Health Organization recommends a maximum concentration of 30 µg. L^{-1} of 2,4-D in drinking water (WHO 2017), requiring the removal of pesticides from water and effluents (Hameed et al. 2008). One of the most used water treatment techniques is filtration. However, the existing filter with physical characteristics has a very low capacity to remove certain molecules, such as pesticides. Adsorption on activated carbon is a technology successfully used in the removal of organic compounds (pesticides, cyanotoxins, drugs etc.) (Letterman et al. 1999; Mello et al. 2015).

Activated carbons produced from wood pyrolysis have interconnected pores smaller than 2 nm and large surface area, generally ranging from 300 to 1500 $m^2.g^{-1}$, which makes these adsorbent materials very effective (Herzog et al. 2006). For water treatment, activated carbon is desirable due to its high pollutant adsorption capacity and fast adsorption kinetics (Kearns et al. 2014).

Finding a low-cost precursor material that combines sustainability and social development with ideal technical characteristics is the subject of much research. There are countless precursors from agricultural and/or forestry activities that have the potential to produce activated charcoal, especially those considered residues. Using waste as a raw material for the production of activated carbon transforms them into a co-product, increasing the competitiveness of the productive activity (Ionnidou and Zabaniotou 2007).

Plant residues from the Amazon contain important precursor materials for the production of activated carbon. Generally they are renewable, available in large quantities and at low cost when compared to other precursor materials (Melo et al. 2015).

The brazil nut “ouriço” is the fruit of the chestnut tree (*Bertholettia excelsa*, Lecythidaceae) (Scussel et al. 2014), a tree typical of the Amazon region (Yang 2009) that protects the almonds (Scussel et al. 2014, Santos et al. 2006). As it is a residue from the processing of brazil nuts, “ouriços” are a low-cost precursor material, found easily and in large quantities. Considering the manually collection of nut, the “ouriço” can be easily removed from the forests by the collectors and sent for processing. In addition to being lignocellulosic, a characteristic that allows obtaining quality activated carbon (Nogueira et al. 2014).

The objective was to analyze the adsorption capacity for 2,4-D of brazil nut “ouriço” charcoal, physically activated in two distinct atmospheres, CO_2 and water steam.

2. Material and Methods

The activated carbons used were produced from the carbonization for five hours at 800 °C of the brazil nut “ouriço”, a temperature that produced the best quality coal among the temperatures tested. Followed by physical activation, in the reactor, at 800 °C for 40 min with an atmosphere modified by the injection of CO_2 or water steam (Rimolli et al. 2019).

BET analysis (Brunauer, Emmett and Teller) (Micromeritics Tristar II Kr 3020) was developed at the Laboratory of Solids and Surfaces (LSS) of the Chemistry Institute of the Federal University of Rio Grande do Sul (UFRGS) and scanning electron microscopy (SEM) at the Electronic Microscopy Center of the same university. Micropore size distribution was evaluated by the DFT (Density Functional Theory) method. The morphology of the samples was obtained by SEM (Jeol JSM 6060 scanning electron microscope), voltage of 10 Kv.

All analyzes referring to 2,4-D adsorption were performed at the Laboratory of Pharmaceutical Nanotechnology, Federal University of Mato Grosso (UFMT). Quantification by HPLC was performed by constructing analytical curves with a 2.4-D standard (Sigma Aldrich) with 98% purity, constructed in the range between 0.6 to 30 µg. mL^{-1} and prepared from stock solutions at a concentration of 500 µg. mL^{-1}. The system used was a Varian Pro Star 325 liquid chromatography with an ultraviolet (UV) detector. Separation was performed on a Phenomenex Luna C18 column (250 x 4.6 mm, 5 µm). The mobile phase consisted of 80% H_2O (pH 3.0) and acetonitrile (80:20). The acquisition time was 7 minutes, with a flow of 1 $mL.min^{-1}$, injection volume of 20 µL and detection at 230 nm.

To study the adsorption and equilibrium kinetics, 20.0 mL of 2.4D solution (5 µg. mL^{-1}) was prepared and added to a 50.0 mL Falcon tube with 0.010 g of brazil nut "ouriço" carbon activated with CO_2 or water steam at 800 °C. The samples were shaken in an SP Labor shaker incubator at 150 rpm and at 25 °C for specific time periods (analyzed every 2 min for the first 30 min, between 30 and 60 min, analyzed every 5 min, and between 60 and 120 minutes, analyzed every 30 min), filtered and analyzed in HPLC.

The isotherm tests followed the international standard ASTM 3860-98 (2008). Kinetic models were applied to interpret the experimental data in order to understand the mechanisms that control the adsorption process, as well as the mass transfer in solution and chemical interactions.

The first kinetic model used to adjust the experimental data was the pseudo-first order, defined by Lagergren, which is based on the adsorption capacity of solid-liquid systems (Njoku and Hameed 2014). Adsorption kinetics was described by the pseudo-second order equation, according to the model used to adjust the data (Ho Mckay 1999).

3. Results

The specific surface area determined by the BET method for activated carbon with CO_2 at 800 °C was 395 $m^2.g^{-1}$ and for activated carbon with water steam at 800 °C it was 401 $m^2.g^{-1}$. The N_2 adsorption/desorption isotherms for carbon activated with either CO_2 or water steam indicated high N_2 adsorption at low pressures (Figure 1a) and greater pore density at 1.17 nm (Figure 1b).

The SEM analysis showed that carbon activated with either CO_2 (Figure 2) or water steam (Figure 3) presented structural modifications of the precursor, with pore formation in the shape of cavities similar to a well-organized pore network, as uniform honeycomb craters.

The analytical parameters of linearity, precision, accuracy, specificity and robustness were developed and confirmed. The amount of 2,4-D adsorbed after 2 minutes of contact with carbon activated with CO_2 was 3.48 $mg.g^{-1}$, whereas for carbon activated with water steam it was 6.17 $mg.g^{-1}$. That is, the removal percentage of 2,4-D after 2 minutes of contact with CO_2 activated carbon was 54.62 %, while the 2,4-D removal percentage with water steam activated carbon was 67.39 % for the same period (Figure 4).

The time required to obtain the maximum 2,4-D adsorption with carbon activated with CO_2 at 800 °C was 35 min (4.89 $mg.g^{-1}$) and for carbon activated with water steam, also at 800 °C, it was 120 min (8.85 $mg.g^{-1}$). For carbon activated with CO_2, it took 35 min for the adsorption process to reach equilibrium, while for carbon activated with water steam, it was 30 min (8.32 $mg.g^{-1}$) (Figure 5).

The pseudo-first-order and pseudo-second-order models were used to describe the 2,4-D adsorption mechanism, performing linear adjustments under non-equilibrium conditions. For carbon activated with CO_2, the pseudo-first order model presented an R^2_{adj} value of 0.4024, k1 of 0.0219 min^{-1} and qe of 0.9370 $mg.g^{-1}$. The pseudo-second order model had a value of R^2_{adj} of 0.9972, k2 of 0.0943 $g.mg^{-1}.min^{-1}$ and qe of 4.8197 $mg.g^{-1}$ (Table 1).

The pseudo-first order model fitted to the experimental data for carbon activated with water steam, which presented an R^2_{adj} value of 0.5795, k1 of 0.0359 min^{-1} and qe of 2.1560 $mg.g^{-1}$. The R^2_{adj} value of the pseudo-second order model was 0.9942, k2, 0.0283 $g.mg^{-1}$.min-1 and qe was 8.8896 $mg.g^{-1}$ (Table 1).

Based on the value of R^2_{adj}, the pseudo-second order model was the one that best fit the experimental data (Figures 6 and 7).

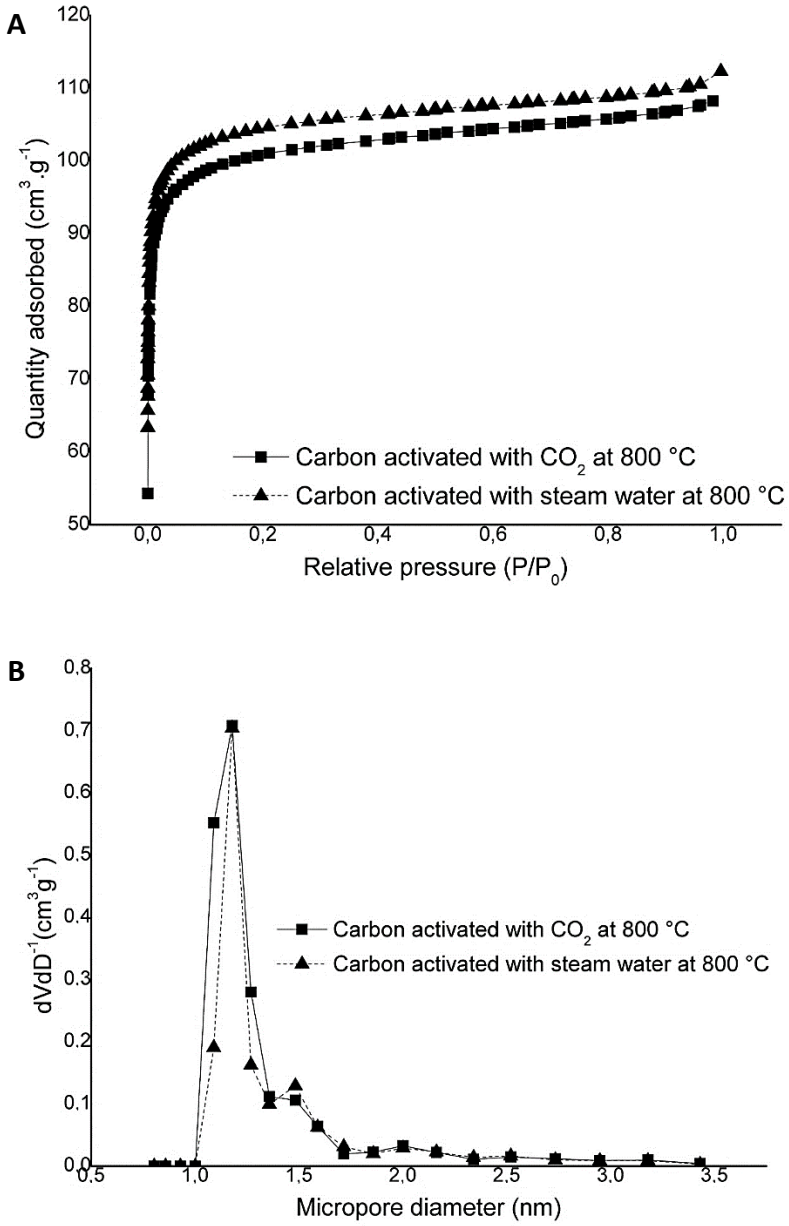

Figure 1. A - N2 adsorption/desorption isotherm at 120 °C for activated carbons and B - pore volume distribution for activated carbons.

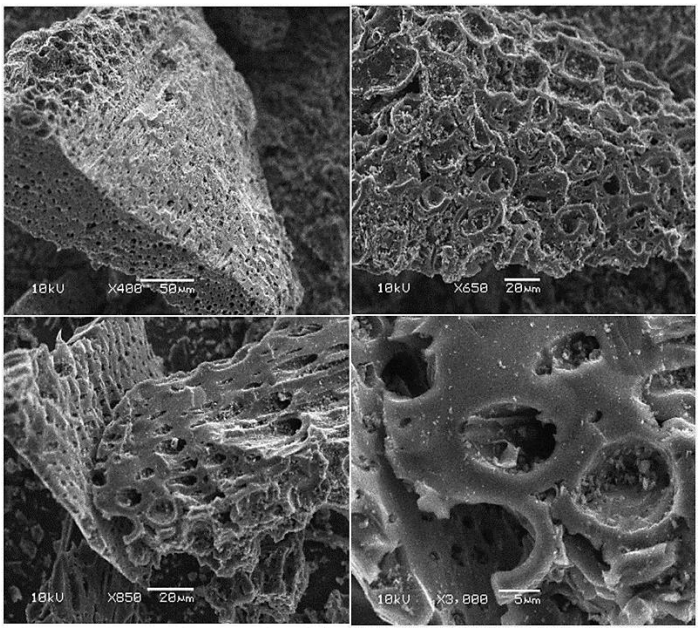

Figure 2. SEM micrograph of Brazil nut "ouriço" charcoal with CO_2 at 800 °C.

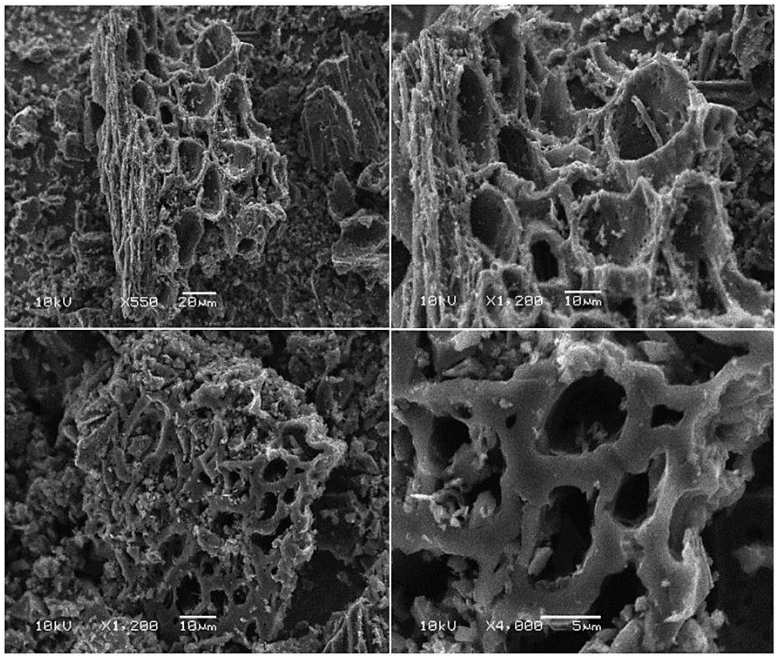

Figure 3. SEM micrograph of Brazil nut "ouriço" charcoal with water steam at 800 °C.

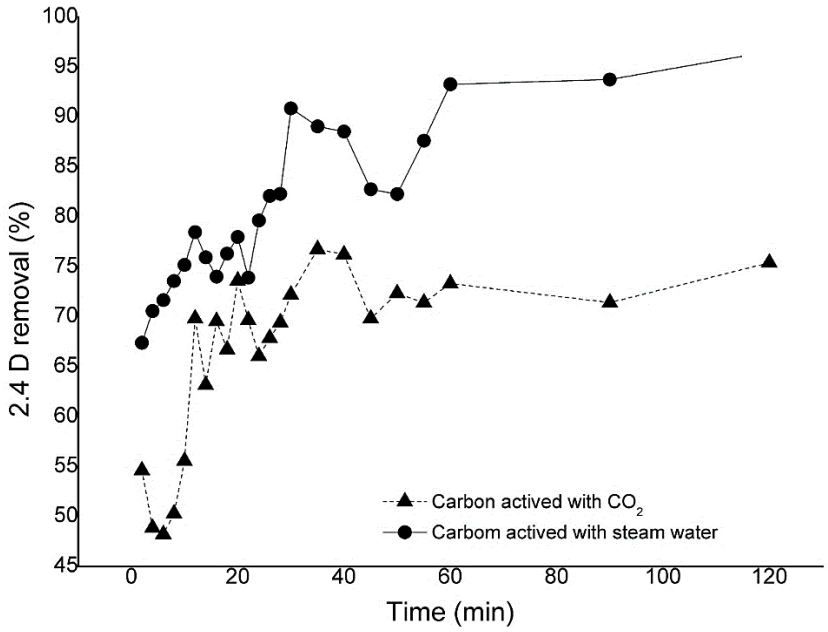

Figure 4. Removal percentage of 2,4-D on the activated carbon of the Brazil nut "ouriço" with CO2 or water steam as a function of time.

Table 1. Kinetic model parameters for 2,4-D adsorption with carbon activated with CO_2 or water steam at 800 ºC.

Parameters	Carbon activated with CO_2	Carbon activated with water steam
C_0 (mg.L^{-1})	3.1855	4.58
$q_{e,exp}$ (mg.g^{-1})	4.8900	8.32
Pseudo-first order		
k_1 (min^{-1})	0.0219	0.0359
q_e (mg.g^{-1})	0.9370	2.1560
R^2_{adj}	0.4024	0.5795
RMSE		
Pseudo-second order		
k_2 (g.mg^{-1}.min^{-1})	0.0943	0.0283
q_e (mg.g^{-1})	4.8197	8.8896
R^2_{adj}	0.9972	0.9942
RMSE		

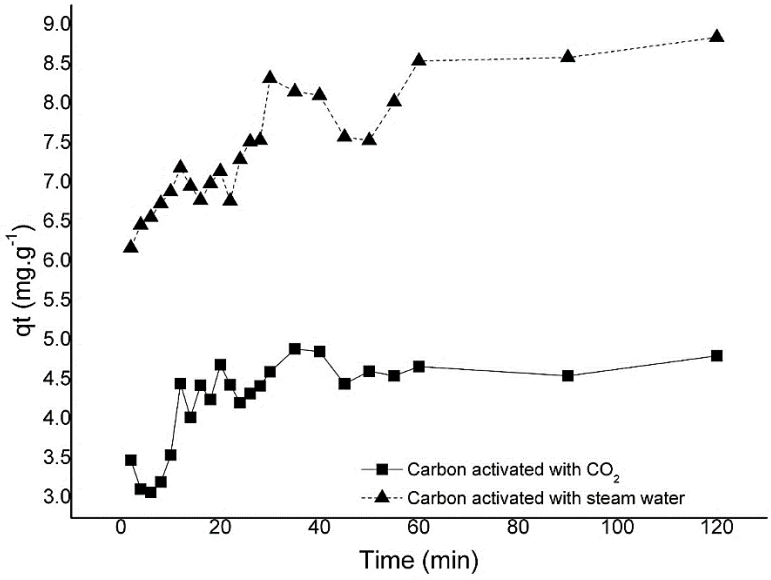

Figure 5. Adsorption of 2,4-D per gram of activated carbon with CO2, pH 6.44, or with water steam, pH 6.63, at 800 °C.

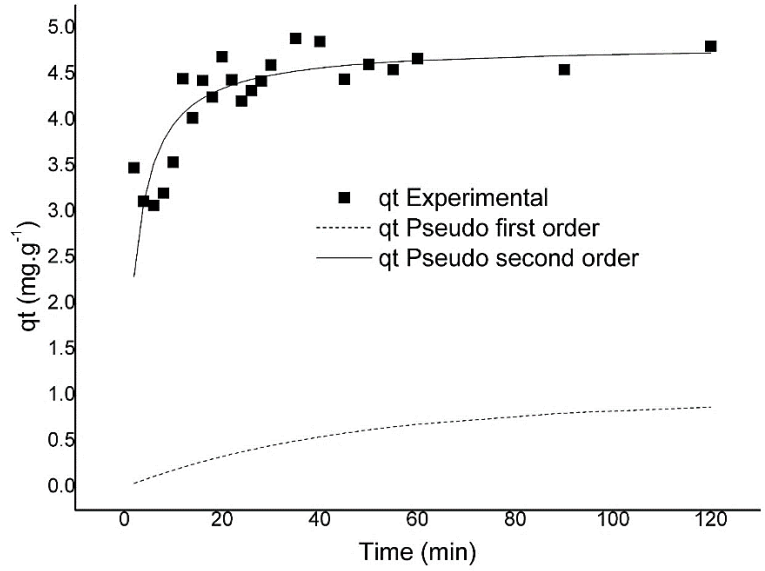

Figure 6. Kinetic modeling for 2,4-D adsorption on activated carbon with CO_2 at 800 °C.

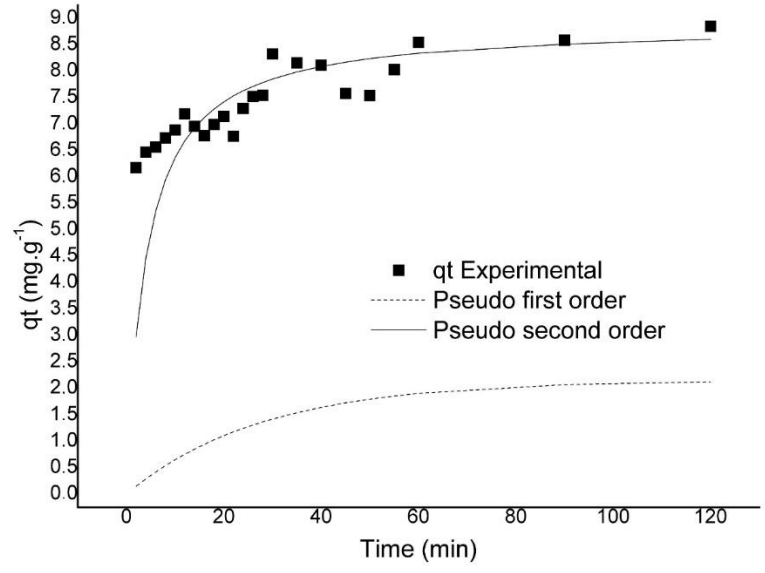

Figure 7. Kinetic modeling for 2,4-D adsorption on activated carbon with water steam at 800 °C.

4. Discussion

The high values of the specific surface area obtained for both carbon activated with CO_2 or with water steam (Herzog et al. 2006) indicate the quality of the brazil nut "ouriço" as a precursor material for the production of activated charcoal, in addition to being a low-cost, renewable and abundantly available material.

The microporous profile of carbon activated with CO_2 or with water steam presented a higher volume of N_2 adsorption at low pressures (Sun and Webley 2010) and peaks with lower pore diameter at 2 nm (20 Å) (Ahmad et al. 2007) This data is important for the adsorptive result. Activated carbons have a microporous profile that provides high adsorption capacity for small organic molecules, with potential for retention of common gases and solvents (Nobre et al. 2015).

Through the BET analysis it was possible to obtain a graph of the adsorption and desorption isotherms of the process, whose geometry was defined by the surface and porosity properties of the activated carbons (Sing et al. 1985). The isotherms of carbons activated with CO_2 or water steam were Type I (Figure 1a), which are typical of microporous materials with relatively small external surfaces. In this case, adsorption limits are determined by the volume of accessible micropores rather than the inner surface area (Sing et al. 1985). Type I isotherms are revealed in cases when chemisorption occurs (Shaji and Zachariah 2017). The same type of isotherm was found for activated carbon from babassu biomass (Oliveira et al. 2016).

The pore distribution analysis showed that between the two atmospheres there is little variation in the BET surface area, which can also be observed in the photographs of the surface of the samples (Figures 2 and 3). This indicates that the activation atmosphere did not influence the pore distribution and that the brazil nut "ouriço" is an excellent alternative as a precursor material for the production of activated carbon, regardless of activation with CO_2 or water steam.

Scanning electron microscopy analysis showed that activated carbons presented pore dilation and a defined arrangement with the formation of organized craters (hive type). The porous surface is the result of the elimination of volatile compounds formed by the decomposition reactions during carbonization. This pore formation plays an important role in the adsorption operation, mainly due to the size of the adsorbed molecules (Rimolli et al. 2019).

The validated analytical method proved to be linear, precise, exact, specific and robust. The kinetic experiments showed that the 2,4-D adsorption was directly proportional to the contact time between the adsorbent and the adsorbate. Faster adsorption was also observed during the early stages and slow more when it approached equilibrium. Both carbons activated with CO_2 or with water steam proved to be efficient in removing 2,4-D. However, the carbon activated with water steam proved to be superior at removing 2,4-D in the first minutes. This is because this activated carbon had lower water and volatile material contents, and a higher percentage of fixed carbon (Rimolli et al. 2019).

The amount of 2,4-D adsorbed at equilibrium reflects the maximum adsorption capacity of activated carbon under operating conditions (Njoku and Hameed 2011). In addition to the higher percentage of adsorption for activated carbon with water steam, it reached equilibrium more quickly. These results contribute to demonstrating the superiority of carbon activated with water steam rather than CO_2.

The 2,4-D adsorption kinetics studies found equilibrium times of 8 h for corncob charcoal chemically activated with H_3PO_4 (Njoku and Hameed 2011), between 4 and 7 h and 30 min for date pit activated charcoal (Hameed et al. 2008), and between 4 and 6 days for granular commercial charcoal (6 – 16 mesh) (Aksu and Kabasakal 2004), demonstrating the high quality of brazil nut "ouriço" charcoal activated with CO_2 or water steam.

The study of adsorption kinetics enabled the optimization of processes and determined the solute adsorption rate that controls the residence time of the adsorbent at the solid-solution interface (Zeferino et al. 2014). The pseudo-second order model has been successfully applied to the adsorption of herbicides (Ho 2006) and was the one that best fits the experimental data. Experimental data from the adsorption kinetics study of 2,4-D and carbofuran pesticides with commercial granular activated carbon were also better described by the pseudo-second order model (Salman and Hameed 2010).

It is believed that the removal of 2,4-D from the aqueous solution using activated carbon occurs through the transport of 2,4-D from the boundary layer to the outer surface of the adsorbent. That is, by film diffusion in the transfer of 2,4-D from the surface to the intraarticular active sites and by the adsorption of 2,4-D by the sorbent's active sites (Aksu and Kabasakal 2004). The pseudo-second order model suggests that the adsorption rate is more dependent on the availability of adsorption sites on the surface of the activated carbon than on the concentration of 2,4-D in the solution (Liu 2008). This expression describes chemisorption involving valence forces through the sharing or exchange of electrons between 2,4-D and activated carbon as covalent forces and ion exchange (Ho 2006).

5. Conclusions

This study presents valuable insights into the adsorption capacity of brazil nut “ouriço” charcoal, demonstrating its effectiveness in removing 2,4-D. The results emphasize the significance of activation atmosphere, with steam activation at 800°C yielding the best results. The findings contribute to the development of sustainable and efficient adsorbents for the removal of organic contaminants from aqueous systems. Further investigations could delve into optimizing the activation parameters, such as temperature and activation time, to achieve even higher adsorption capacities. Additionally, the research could explore the influence of other variables, such as pH and initial concentration of the pollutant, to better understand the charcoal's performance under different conditions.

Authors' Contributions: RIMOLI, M.F.S.: conception and design, acquisition of data, analysis and interpretation of data, drafting the article, and critical review of important intellectual content; NOGUEIRA, R.M.: conception and design, acquisition of data, analysis and interpretation of data, drafting the article, and critical review of important intellectual content; DE CASTRO, P.M.: conception and design, acquisition of data, analysis and interpretation of data, drafting the article, and critical review of important intellectual content; MERLO, A.A.: conception and design, acquisition of data, analysis and interpretation of data, drafting the article, and critical review of important intellectual content; SINHORIN, A.: conception and design, acquisition of data, analysis and interpretation of data, drafting the article, and critical review of important intellectual content; KERKHOFF, J.: conception and design, acquisition of data, analysis and interpretation of data, drafting the article, and critical review of important intellectual content; FERRARINI, S.R.: conception and design, acquisition of data, analysis and interpretation of data, drafting the article, and critical review of important intellectual content; PIRES, E.M.: conception and design, acquisition of data, analysis and interpretation of data, drafting the article, and critical review of important intellectual content. All authors have read and approved the final version of the manuscript.

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