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Activated Carbon Production from Biomass and its Use in Industrial Effluent Treatment

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Abstract. This study evaluates the feasibility of converting black wattle (*acacia mearsii*) bark residue - a byproduct of tannin extraction - into activated carbon (AC) for wastewater treatment. In southern Brazil, the industrial sector generates approximately 250 t/day of this residue, which is typically landfilled or composted. To add value to this waste, AC was produced via pyrolysis and chemical activation with H_3PO_4. The resulting material was characterized using elemental analysis, thermogravimetric analyses (TGA), scanning electron microscopy (SEM)/energy dispersive X-ray spectrometry (EDS) analyses, and Brunauer-Emmett-Teller (BET) and Barret-Joyner-Halenda (BJH) methods. Elemental analysis of the raw bark revealed a 51.4% carbon content. The produced AC achieved a specific surface area of 906 m²/g, significantly outperforming commercial activated carbon (CAC), which reached 597 m²/g. SEM images confirmed a highly developed porous structure. Performance tests for methylene blue dye removal demonstrated that the bark-derived AC is more effective than commercial alternatives, highlighting its potential as a high-performance, sustainable adsorbent for industrial effluent treatment.

Key words. Activeted carbon; biomass; effluent treatment; methylene blue dye

1. Introduction

Black wattle (*Acacia mearsii*) is a leguminous species not native to Brazil and its cultivation in the country is related to the extraction of wood and tannin. Tannin is a powerful coagulant used in the treatment and purification of water. The extraction of this compound, which is found in the bark of the tree trunk, ends up generating high rates of solid waste. Black wattle bark residues (BWBR) ends up being destined for organic composting, combustion or disposed of in landfills, creating costs for the generating companies. In the state of Rio Grande do Sul, in 2022, approximately 153 thousand tons of black wattle bark were produced, totaling 100% of the national production [1].

Activated carbon is a carbonaceous-based material that contains a well-developed internal pore structure with a high surface area and high porosity consisting of micro, meso and macropores. Furthermore, the functional groups present on the surface of this material make it a versatile material for materials science with applications in several areas, mainly in industrial processes, water treatment and for medicinal uses [2],[3].

The process used for carbon activation can be divided into two categories: chemical activation process and physical activation process. In chemical carbon activation processes, the starting materials are impregnated with a solution of a dehydrating agent (e.g. zinc chloride, sulfuric acid or potassium hydroxide) to retard the formation of tars during the carbonization process. These materials are then dried and carbonized (pyrolysis) at moderate temperatures (400 - 600 °C) in an inert atmosphere to produce the final adsorbent. In the physical activation process, nitrogen gas or carbon dioxide are used for the oxidation of carbonaceous matter at temperatures that can reach up to 1000 °C [4],[5].

Activated carbon can be produced from any organic substance that has a high carbon content in its composition. Lignocellulosic materials are formed by complex organic macromolecules often consisting of pectins, lignins, hemicellulose and celluloses, which may or may not be linked together. When chemically activated, these materials can be converted into highly porous activated carbons by undergoing hydrolysis, dehydration and condensation, and also by cross-linking reactions between the activating agent and the biopolymers that make up these materials [6],[7].

Research papers report the production of activated carbon from the use of various residual biomasses such as rice husks, orange peels, coffee grounds, melon seeds and wood sawdust, among others [8],[9]. In this context, it is observed that recent studies have pointed out the possibility of producing activated carbon from the use of lignocellulosic materials; however, there are no studies on the use of black wattle bark for this purpose. Thus, the objective of this work is to evaluate the feasibility of using this residue in the production of porous activated carbon with a high surface area, encouraging the use of alternative sources in the production of AC and also contributing to the reduction of economic costs and increasing environmental preservation. The activated carbon obtained from *Acacia mearsii* bark was tested in the adsorption of methylene blue, a cationic dye widely used in the textile industry for dyeing cotton and wool fabrics.

One of the main problems encountered by the textile sector is the removal of synthetic dyes from its effluents; it is estimated that of approximately 20 t/year of dyes consumed by the textile industry, approximately 20% are discarded as effluents; the main reason for this loss is related to the incomplete fixation of the dye to the fabric fiber during the dyeing process [10],[11]. The uncontrolled release of methylene blue dye into rivers and lakes, when not treated properly, affects not only the transparency of the water but also limits the passage of solar radiation, reducing natural photosynthetic activity, causing changes in the aquatic biota and resulting in acute and chronic toxicity in these ecosystems [10],[12].

2. Materials and methods

A. Carbon pyrolysis and activation

BWBR were obtained from a tannin producing company based in Vale do Rio dos Sinos, in the state of Rio Grande do Sul, Brazil. First, the residues underwent a selection process, where the smallest particles (< 5 mm) were selected. The particles were then dried in an oven at 105 °C for 24 hours in order to obtain a standard precursor material. After drying, the samples were weighed and stored at room temperature.

For impregnation of the samples, phosphoric acid (H_3PO_4) was used as an activating agent from a commercial 85 wt% P.A. solution, which was diluted to a concentration of 28 wt%. Thus, quantities of 20 g of the precursor material were added to 200 ml of H_3PO_4 solution and the activation time of the black wattle bark was 12 hours under constant stirring. After impregnation, the samples were dried in an oven at a temperature of 70 ºC before proceeding to the pyrolysis stage [13],[14]. In the pyrolysis (carbonization) stage, the impregnated samples were placed inside a cylindrical stainless steel reactor measuring 30 cm in height and 10 cm in diameter, located in a muffle furnace. The samples were then heated to 500 °C at a heating rate of 5 °C/min and maintained at this temperature for 2 hours. The entire process took place in a nitrogen (N_2) atmosphere with a constant flow of 200 ml/min inside the reactor. At the end of the pyrolysis, the samples were cooled in a current of N_2 and maintained at room temperature. After the pyrolysis stage, the chemically activated carbon was washed with distilled water to remove excess activation agent. The samples were then dried in an oven for 12 hours at a temperature of 105 °C to completely remove absorbed water. The identification was in the form AC/H_3PO_4, where “AC” refers to Activated Carbon and H_3PO_4 to the activation agent.

For comparison purposes, charcoal was produced from black wattle bark residues without the presence of activating agents. For this purpose, three samples of BWBR were pyrolyzed without any type of impregnation, where the same carbonization patterns of the chemically activated charcoal were maintained, varying only the heating rates of the reactor at values of 5, 20 and 60 ºC/min. These charcoals were named as follows: C/05, C/20 and C/60, where the letter “C” refers to charcoal and the numeral represents the heating rate to which each sample was subjected. The yield of the charcoal produced was determined based on the initial mass of the precursor and the final mass of the charcoal on a dry basis calculated by Equation 1, where Mi represents the mass of precursor material in grams before pyrolysis in the reactor and Mf represents the mass in grams of the activated charcoal obtained at the end of the process.

$$\text{Yield (\% by mass)} = (Mf/Mi) \times 100 \quad (1)$$

B. Sample characterization

Immediate and elemental analyses of the precursor material, BWBR, were performed on a LECO TruSpec analyzer to determine the mass percentage of carbon as well as the sulfur, nitrogen, hydrogen and oxygen contents, according to ASTM D5373 (1993). The surface area and total pore volume of the produced coals were evaluated by the Brunauer-Emmett-Teller (BET) and Barret-Joyner-Halenda (BJH) methods, respectively, through N_2 adsorption using a Quantachrome analyzer.

Thermogravimetric analyses (TGA) were performed to analyze the thermal degradation characteristics of the precursor material and the samples of the produced coals. Weight loss was calculated as a function of temperature, using a LECO TGA 701 thermogravimetric analyzer. The heating rates used were 5 and 10 °C/min with a nitrogen flow of 20 mL/min, and the mass was constantly weighed. To evaluate the morphology of the coal surfaces, scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDS) analyses were obtained using a LEO EVO 50HV digital microscope (Carl Zeiss) equipped with an energy dispersive analyzer that provides the SEM image and the EDS spectrum using the same sample.

For comparison purposes, commercial activated carbon (CAC) from the Synth brand was selected and characterized using the BET, SEM and EDS methods in the same manner described for the carbons produced in this study.

C. Methylene blue adsorption tests

The methylene blue adsorption tests were performed in 250 mL Erlenmeyer flasks containing 50 mL of methylene blue dye solution and 0.05 g of the adsorbent material. The systems were shaken at 175 rpm at 25 ˚C for 10, 20, 30, 40, 50, 60, 70 and 80 minutes. After each contact time, the samples were removed and vacuum filtered, and the remaining dye concentrations were determined by reading on a UV-Vis spectrophotometer (T80-PG Instruments, GB) at a wavelength of 665 nm.

3. Results and discussion

The results of the immediate analysis, elemental analysis and calorific value of the black wattle bark residue (BWBR) are presented in Table I.

Table I. - Immediate analysis, elemental analysis and calorific value of the black wattle bark residue (BWBR)

Analysis	Parameter	BWBR
Immediate analysis (%)	C_{fixed}[a]	20.1
	Ash[a]	3.8
	Volatile matter[a]	76.2
	Moisture[b]	57.6
Elemental analysis (%)	C[a]	51.4
	H[a]	5.2
	S[a]	0.1
	N[a]	1.4
Calorific value (kcal/kg)	PCI[a]	4405

[a]dry basis
[b]as received

The immediate BWBR analysis showed a high percentage of volatile matter (76.2%, d.b.) and moisture (57.6%), and a low ash content (3.8%, d.b.). The elemental analysis of the material indicated a carbon content of 51.4%, characteristic of a good precursor material for the production of activated carbon. Similar results were observed in studies of the pyrolysis of soybean residue, olive pomace and olive pit, respectively [4],[15],[16].

The average yields in the production of activated carbons are presented in Table II. The values did not present great variations between the different samples and demonstrate that the chemical activation through H_3PO_4, as well as the variations applied in the heating rate of the reactor did not significantly influence the yield of the products. The yield values obtained experimentally corroborate those found in the literature.

Table II. - Yield, surface area (S_{BET}) and pore volume of the analyzed coal

Sample	Yield (%)*	S_{BET} (m^2/g)	Pore Volume (cm^3/g)
AC/H_3PO_4	37.2	906	0.27
C/05	41.7	12	0.05
C/20	39.3	41	0.04
C/60	38.3	54	0.04
CAC	-	597	0.22

* in bulk

The results obtained for the surface area (S_{BET}) and pore volume analyses of the produced carbons together with the results of the commercial activated carbon are presented in Table II. Samples C/05, C/20 and C/60, which correspond to the carbons not chemically activated, presented the lowest surface area values (12, 41 and 54 m^2/g, respectively). The AC/H_3PO_4 sample activated with phosphoric acid presented the highest specific surface area value (906 m^2/g), a value approximately 35% higher than the surface area of the commercial activated carbon (CAC) sample used as a reference, which was 597 m^2/g. Likewise, the total pore volume of the activated AC/H_3PO_4 sample (0.27 cm^3/g) was higher than the value obtained for the commercial activated carbon (0.22 cm^3/g). Similar studies using residues from the olive oil industry to produce activated carbons with H_3PO_4 showed surface area of up to 393 m^2/g [17]. Other studies shown in the literature present results similar to the present work, with surface area and total pore volume of the activated carbon produced in the order of 1040 m^2/g and 0.55 cm^3/g, respectively [18].

Thermogravimetric analysis (TGA) shows the mass loss of the analyzed materials as a function of temperature variation. Figure 1 shows the TGA curves for the BWBR and for the charcoals with and without chemical activation.

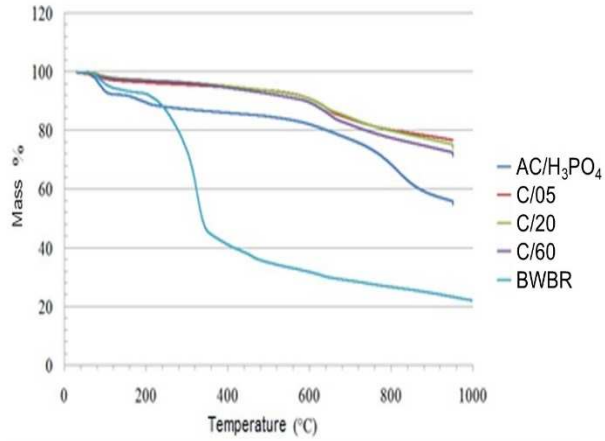

Fig.1. Thermogravimetric analysis of the black wattle bark residues samples and the charcoals with and without chemical activation AC/H_3PO_4, C/05, C/20 and C/60

The TGA analysis of black wattle bark residues (BWBR) shows three well-defined mass losses. The first mass loss, below 200 °C, is due to the loss of water from the material. The second mass loss is related to the degradation of oxygen-containing groups on the surface of the material, such as cellulose and hemicellulose. Then, a third mass loss is observed, which is related to more stable carboxylic groups that decompose above 400 °C, such as lignin molecules, which require a wider temperature range for degradation [19]. Also in Figure 1, it can be observed that the coals produced with and without chemical activation presented greater thermal stability in relation to the precursor material and more discrete mass losses, around 20 to 40% by mass. Figures 2a and 2b show the EDS analysis spectrum for the elemental composition of the samples of the coal without chemical activation C/60 and the activated carbon AC/H_3PO_4, respectively. In C/60 carbon, the presence of carbon, oxygen, calcium and gold can be observed in the energy dispersive spectrum (Figure 2a), while for AC/H_3PO_4 activated carbon, the calcium element was not detected, but rather the presence of phosphorus resulting from the H_3PO_4 activation agent used (Figure 2b).

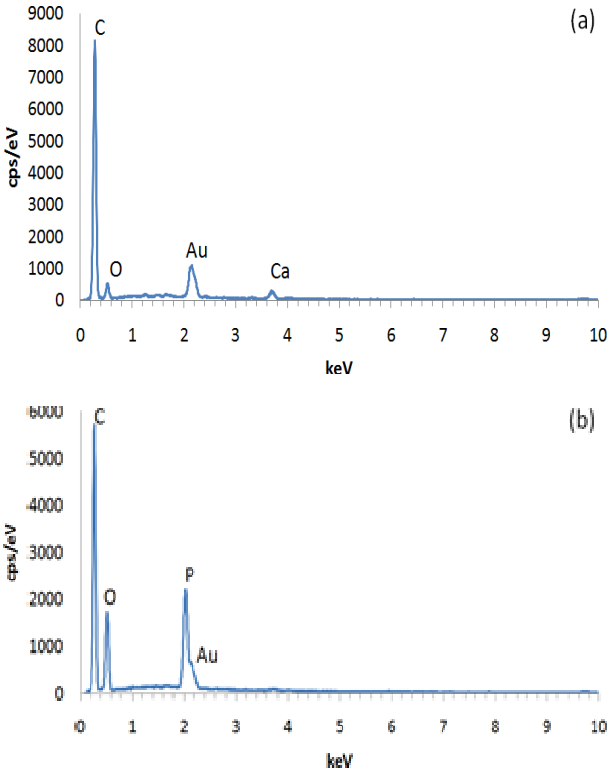

Fig.2. Distribution of elements on the surface of coal samples (a) C/60 and (b) AC/H_3PO_4 by energy dispersive X-ray mapping (EDS)

Table 3 presents the values of the elemental components present in the areas analyzed by EDS and it can be observed that the coals presented a high carbon content in their matrix, close to 60% for AC/H_3PO_4 and 70% for C/60 coal. Commercial activated carbon (CAC) still presented a higher percentage of carbon on the surface (75.7%) in relation to the other samples analyzed. The appearance of small amounts of gold in the EDS analyses is related to the procedure of metallization of the samples with this element for carrying out the analysis.

Table III. - Percentage values of elements on the surface of coals analyzed by EDS

Elemental Composition (% by mass)						
Sample	C	O	Ca	P	Au	Si
AC/H_3PO_4	59	24.3	-	10.2	7.2	-
C/60	68.9	8.4	4.4	-	19	-
C/20	60.1	11.4	11.2	-	15.8	0.9
C/5	58.7	12.7	7.8	-	15.8	2.5
CAC	75.7	7.81	2	-	11.9	1.9

The Scanning Electron Microscopy (SEM) technique was used to investigate the physical morphology of the surfaces of the obtained carbons.

Figure 3a illustrates the image of the AC, where a homogeneous surface structure of the carbon can be observed, with elongated pore cavities and oval-shaped openings. The images of the carbons C/60 and C/05 produced without chemical activation (Figures 3b and 3c) show a surface without relevant presence of fissures and macropores, but rather the formation of a lamellar structure with ridges and closed pores, probably due to the presence of calcium carbonate, since the elements calcium and oxygen appear in the EDS analysis for these samples. This would explain the low value of their surface area as presented by the BET method analysis. In the image of activated carbon with H_3PO_4 (Figure 3d), an irregular and heterogeneous surface morphology can be observed with a well-developed porous structure, which corroborates the high value of the surface area presented through the BET analysis of the respective sample.

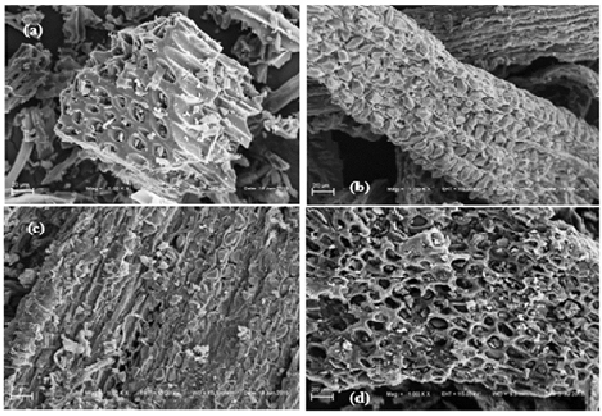

Fig.3. SEM images at 1000x magnification for (a) commercial activated carbon CAC, (b) carbon C/60, (c) carbon C/05, and (d) activated carbon AC/H_3PO_4

Regarding the removal of methylene blue from solutions containing this dye, which is widely used in the textile industry, the best result was obtained for the sample of activated carbon from black wattle bark with H_3PO_4 (AC/H_3PO_4), being more than 98% for the initial concentration of 200 mg/L in 80 min of experiment. This removal of the methylene blue dye was much higher than that obtained with CAC, as can be seen in Figures 4 and 5, probably due to the larger surface area of the AC from *Acacia mearsii* bark, in addition to the presence of functional groups on its surface that facilitate the adsorption of the dye.

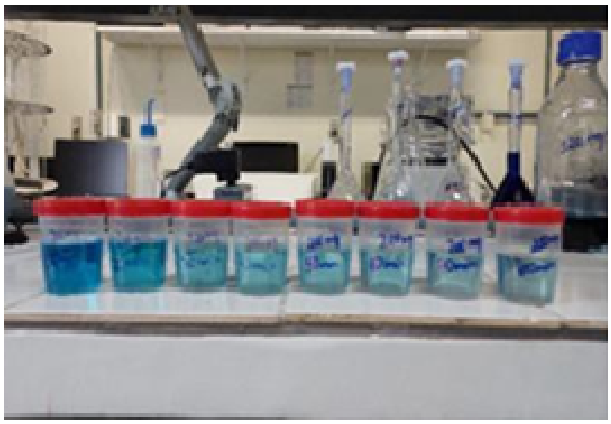

Fig.4. Methylene blue adsorption onto activated carbon produced from black wattle bark

Fig.5.-Methylene blue adsorption onto commercial activated carbon

From a techno-economic feasibility perspective, the conversion of black wattle bark residue into activated carbon shows great industrial potential. The high availability of this byproduct in southern Brazil - approximately 250 tons/day - ensures a stable supply of low-cost raw material. Technically, the produced AC outperformed commercial standards in both surface area and adsorption capacity. Economically, utilizing this waste reduces landfill disposal costs for tannin industries while generating a high-value product for the textile effluent treatment market.

4. Conclusion

Based on the results obtained, it can be seen that black wattle bark residue (BWBR) has the necessary characteristics to be used as a viable raw material for the production of activated carbon. The elemental analysis of BWBR indicated a carbon content of 51.4%, before the pyrolysis process and/or impregnation in solution of the activating agent, which is very important and desirable in the production of carbonaceous materials.

For the carbons obtained by pyrolysis and without chemical activation, the occurrence of a compact structure with partial obstruction of the pores and a low surface area was observed. On the other hand, the activated carbon with H_3PO_4 presented a reticulated and heterogeneous surface morphology with a very porous structure that allowed the emergence of a high surface area. Furthermore, BET analyses indicated that activated carbon with phosphoric acid can have up to 906 m^2/g, which is a surface area much higher than the surface area of commercial activated carbon (CAC) used as a reference for the experiments, which was 597 m^2/g.

The removal of methylene blue from solutions containing textile dye by activated carbon made from acacia bark was 98%, a value much higher than that obtained with commercial sample. This is probably due to the larger surface area of activated carbon made from acacia bark, in addition to the presence of functional groups on its surface that facilitate the adsorption of the dye.

Therefore, the results confirm that the production of activated carbon from black wattle bark is technically and economically viable for large-scale industrial applications.

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