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Production and characterization of activated carbon from wood wastes

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Production and characterization of activated carbon from wood wastes

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Resumen. Cedarwood *(Cedrela Angustifolia)* and teak *(Tectona Grandis)* woods are typically used for furniture manufacture because they have high durability, are light and easy to work. During these manufacturing process, large amount of these wastes is generated causing disposal environmental problems. In this paper, the residual wastes (sawdust) of Cedar (C) and Teak (T) are transformed into an activated material. The chemical composition of both biomass (C and T) was determinate by TGA (Thermogravimetric Analysis). Activated materials were characterized in surface area following the BET (Brunauer, Emmett and Teller) method, morphology using SEM (Scanning Electron Microscopy) and to know their functional groups a FTIR (Fourier Transform Infrared Spectroscopy) analysis was done. Their adsorption capacity was evaluated by removal of Methylene Blue (MB) and Congo Red (CR) from aqueous solutions.

1. Introduction

Activated commercial carbon is a very typical adsorbent to remove gaseous and aqueous contaminants. However, due to the high cost during its production and the environmental problems (it comes from fossil fuel) it is necessary to look for new sources to produce these materials. In a study published by British Petroleum, 2007 [1], coal reserves will only last 147 years (since 2006). Therefore, agroindustry wastes from oil palm [2], potato [3], wood [4, 5] become in good sources to produce activated carbon.

On the other hand, according to Food and Agriculture Organization of the United Nations (FAO) only about 28% of a tree is turned into sawn timber, the remaining 72% is waste that can be used as a fuel, in particles boards and others. These processes mentioned above generate about of 55% in wastes without any use [6], which makes the sawdust as a potential precursor for activated carbon.

Activated carbons produced from wood wastes are recognized for their good pore development, large surface area and high removal capacities. They are typical used as separators and purifiers in both gases and aqueous solutions, catalyst supports and, adsorbents to dyes removal or solvents recovery [7-10].

In this study, the production of activated carbon from two types of wood wastes, Cedar (C) and teak (T), was evaluated. Its chemical composition, morphology and textural properties were determined using different analysis techniques. And their application for dye removal from aqueous solutions was studied.

2. Methodology

2.1. Preparation and characterization of materials

C and T sawdust wastes were obtained from Sena-Calatrava, Itagüí, Antioquia. To get a homogeneous particle size, samples were passed through a sieve with size of <850µm. They were dried at 100°C for 24 hours. A chemical activation was performed with a ratio, biomass: $ZnCl_2$ 3M of 1:5 for C and 1:4 for

T (g:mL) and allowed to stir for 24 hours. After this time, both biomass was calcined at 550°C in a muffle for 60min with a heating rate of 15°C/min; then they were washed with a solution of HCl, 2M and kept under stirring at room temperature for three hours. After that, samples were passed through a filter paper (filter paper qualitative Advantec 2) and washed with distilled water, repeating the process until obtaining a pH between 6 and 7. Finally they were dried in an oven at 105°C for 12 h. Two activated carbons were obtained: Activated Cedar (AC) and Sctivated Teak (AT). To compare, both types of wood wastes were only subjected to calcination at 550°C for 30 minutes. They were called Calcined Cedar (CC) and Calcined Teak (CT).

Moisture, Volatile Material (VM), Fixed Carbon (FC) and ash of each biomass were determined by thermogravimetric analysis (TGA Q500, TA Instruments). The surface area of activated materials was determinate following the BET method (Brunauer, Emmett and Teller) [11], pore volume and size was found by nitrogen adsorption (ASAP 2020, Micrometrics) which is a standard procedure for the determination of adsorbents porosity [2]. Their morphology was determined by SEM analysis (Oxford Instruments). And FTIR (Shimadzu IRtracer 100) was used to know their functional groups.

2.2. Yield of dyes adsorption

Batch experiments were carried out in synthetic solutions of $50 mg \cdot L^{-1}$, at 150rpm and 2 hours to evaluated the yields of C, T, CC, CT, AC and AT for the adsorption of MB and CR. To determine the final concentration of MB and CR, calibration curves were performed and the equation for each one was obtained with a $R^2=0.9999$ (equation (1)) for MB and $R^2=0.9995$ (equation (2)) for CR. The absorbance was measured by using a spectrophotometer (VIS-DR 3900) at a wavelength of 665nm and 490nm for MB and CR, respectively.

$$x = \frac{y + 0.5242}{0.5205} \tag{1}$$

$$x = \frac{y - 0.0354}{0.0859} \tag{2}$$

Where "*y*" is the absorbance and "*x*" is the dye concentration in $mg \cdot L^{-1}$.

3. Results and discussion

3.1. Materials characterization

A typical chemical composition of different types of wood is: VM (78 and 84%), FC (6 and 16%) and ash (0.4 and 2.4%) [4,5]. As can be seen in Table 1, both biomass (T and C) are found within the previous ranges. With these analyses it is possible to say that during the calcination process, about 83% in mass was lost due to the elimination of VM, being available about 15% of FC to be activated. These results indicate that C and T can be used for the manufacture of activated carbon.

Table 1. Biomass properties.

Biomass	Moisture (%)	Proximate analysis (Wt %)[a]		
		VM	FC	Ash
C	6.02	83.76	15.41	0.83
T	5.02	83.92	15.67	0.41

C: cedar; T: teak; VM: volatile material; FC: fix carbon. [a]dry basis

Figure 1 shows SEM analysis for the two types of materials obtained here, calcined and activated (CC, CT, AC and AT). The micrographs show significant changes in the morphology of the studied biomass. Images for calcined materials, CC and CT, show a development of average porosity, which is in agreement with the determined values of surface areas $378.92 m^2 g^{-1}$ for CC and $311.51 m^2 g^{-1}$ for CT (Table 2); this porosity is generated by the release of volatile compounds during the calcination process

[3]. On the other hand, the micrographs for the activated materials (chemical activation), AC and AT, show a discontinuous surface caused by the dehydration action of the activating agent, in this case $ZnCl_2$ [12-14]. This dehydration leads to the good development of porosity and therefore to the increase of the surface area, with values of $1375.67 m^2 g^{-1}$ and $996.65 m^2 g^{-1}$ for AC and AT, respectively.
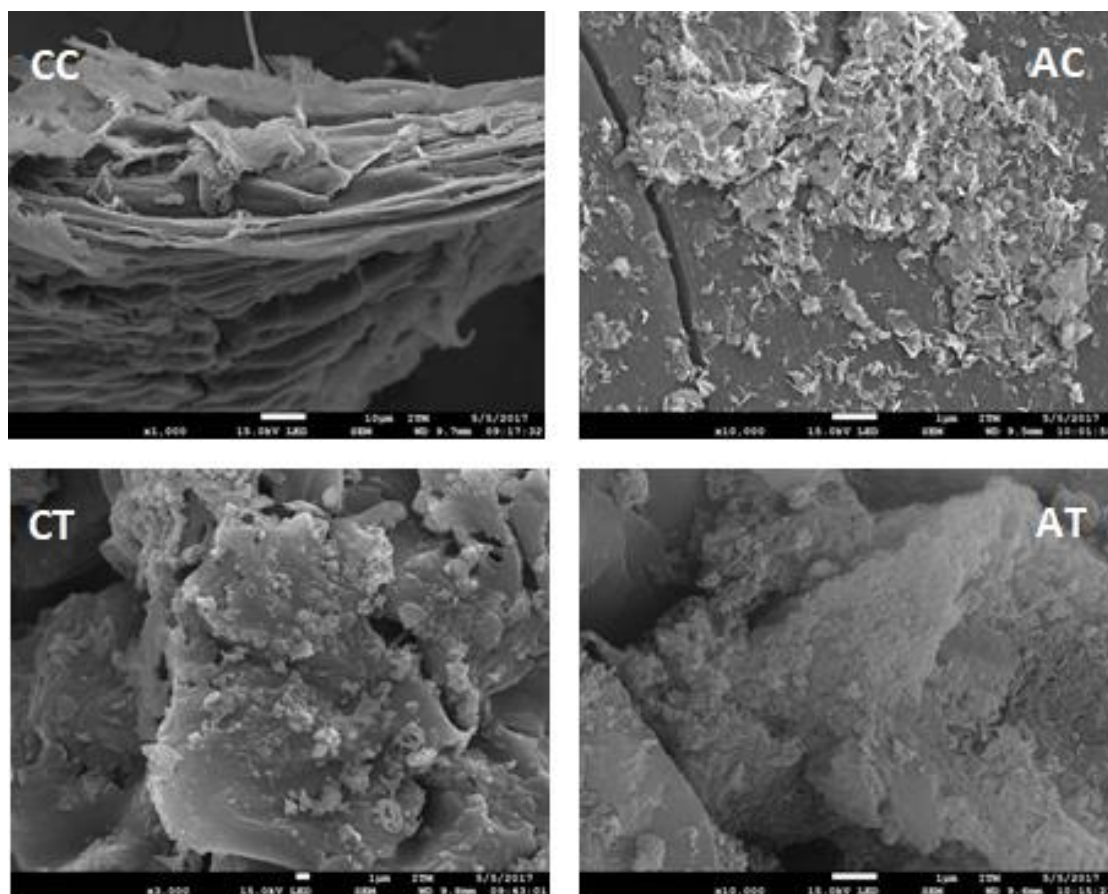

Figure 1. SEM micrographs for calcined and activated materials. CC: calcined cedar; AC: activated cedar; CT: calcined teak; AT: activated teak.

From the above, it can be said that the activation process is efficient since it manages to increase the surface area 3.6 times for AC and 3.2 times for AT respect to the calcination process. This parameter is one of the most important to take into account for activated carbons. It is widely known that in a greater surface area, there are more available active sites, which allows the material to be promising for using in adsorption processes [15].

The IUPAC (International Union of Pure and Applied Chemistry) classifies the micro-, meso- and macroporosity of the materials depending on the pore size: micropores ($d_p < 2nm$), mesopores ($2nm < d_p < 50nm$) and macropores ($>50nm$). Table 2 shows the pore size for calcined and activated materials. These values indicate that CC, CT, AC and AT have a mesoporous structure with tendency to microporous. In the same way, an increase in pore volume of AC and AT in comparison to CC and CT is observed. According to some authors [4,5,16] activation parameters such as, time, temperature and activation method strongly influence textural properties of the activated carbons. For example, they reported that as the temperature increases from 700 to 900°C, the yield of the material is reduced and the effect of the activation time is more pronounced (4 hours). The decrease in yield with increasing temperature is due to the removal of carbon atoms during the activation stage, which leads to the creation of a large number of new pores, enlargement of existing pores and opening of blocked pores [5,16].

Table 2. Properties of calcined and activated materials.

Materials	Surface area, BET ($m^2 g^{-1}$)	d_p (nm)	Pore volumen ($cm^3 g^{-1}$)
CC	378.92	2.19	0.21
CT	311.51	2.19	0.17
AC	1375.67	2.73	0.94
AT	996.65	2.55	0.63

CC: calcined cedar; CT: calcined teak; AC: activated cedar; AT: activated teak; d_p: pore size

Other important parameters to consider for activated carbons are the functional groups on their surface which may influence the adsorption process [4,5,16]. The functional groups commonly found in activated carbon are: alcohols, aromatic rings, aromatic compounds and aliphatic chains [17].
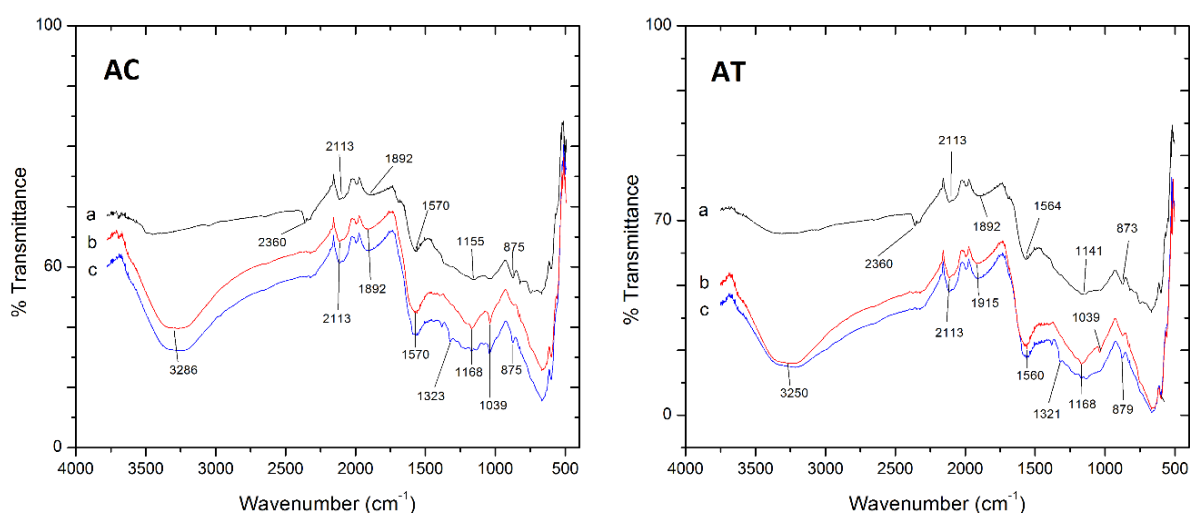

Figure 2. FTIR spectra for activated carbons. AC: activated cedar; AT: activated teak. a: before the adsorption process and b, c: after the adsorption process using CR and MB, respectively.

Figure 2 shows the FTIR spectrum for AC and AT before (a) and after the adsorption process with CR (b) and MB (c). It has been reported that the main interactions between the functional groups in activated carbon and dyes are: (i) hydrophobic interactions (Van der Waals - low energies), (ii) electron donor-acceptor interactions between the aromatic rings of activated carbon and -OH groups in dyes and, (iii) hydrogen bonds [17]. The adsorption process studied in this work may be governed by the interactions mentioned above. According to Figure 2, the main functional groups present in our materials are aromatic, aliphatic and alcohols, which according to their relative intensities can be explained as follows: interactions between $3280 cm^{-1}$ and $3250 cm^{-1}$ correspond to hydrophobic ones, between $2360 cm^{-1}$ and $2113 cm^{-1}$ are donor-acceptor electron interactions through C-H symmetrical stretches; signals at $1570 cm^{-1}$ and $1560 cm^{-1}$ correspond to donor-acceptor electron interactions through aromatic rings of activated materials and -OH group of dyes. Signals between $1323 cm^{-1}$ and $1321 cm^{-1}$ are donor-acceptor electron interactions by C-H inflections. And the ones between $1168 cm^{-1}$ and $1039 cm^{-1}$ are attributed to interaction donor-acceptor by stretching CO. These results are in very good agreement with the reported by Thue, *et. al.* (2006) [17].

3.2. Yield of the materials for dyes adsorption

Figure 3 shows the percentages in MB and CR removal using the materials before and after the transformation processes (C, T, CC, CT, AC, AT). It can be observed that for all cases, activated materials present a greater removal compared to calcined materials and biomass without any treatment. The percentages of removal for CR are given as: AC ~40%, CC ~15% and C ~6%.
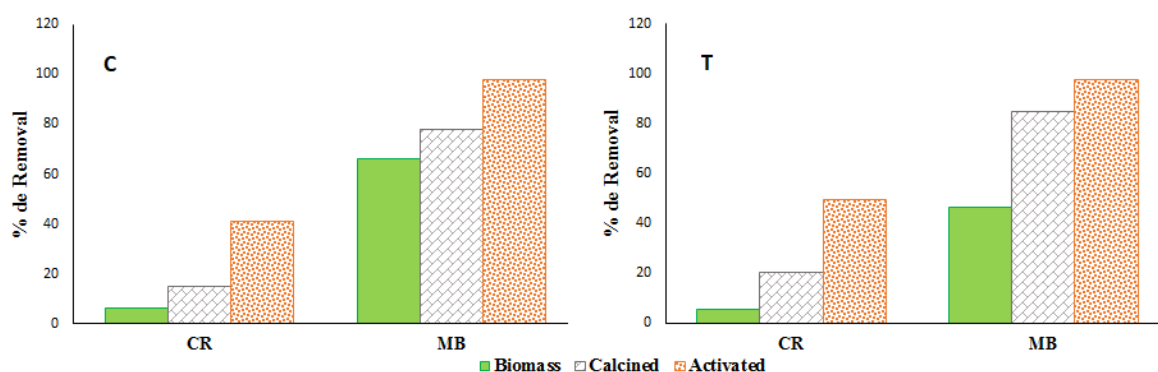
Figure 3. Dyes removal using materials before and after the transformation processes. C: cedar; T: teak; CR: Congo Red; MB: Methylene Blue. green: biomass; grey: calcined; orange: activated

The good performance of activated materials during dyes removal can be explained by adsorption inside the pores or by interaction of functional groups. In this case, it is suggested that the greater removal given by AC and AT for MB is related to dyes size. CR (Figure 4(b)) has a larger molecular size than MB. Therefore, for the adsorption of a CR molecule it is necessary more active sites (functional groups) than for a MB molecule. And, the pore size is a limiting factor for the adsorption of CR. Similar reports have been publish in the scientific literature [18,19].
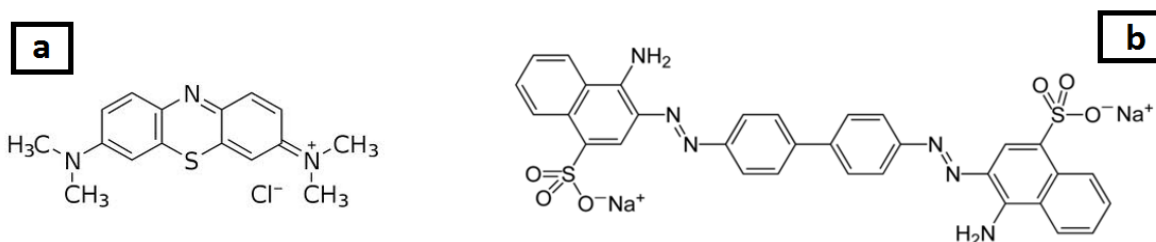

Figure 4. Chemical structure of (a) Methylene Blue (b) and Congo Red.

4. Conclusions

It was demonstrated that our activation method is suitable for the transformation of cedar and teak wastes into an activated carbon with a potential use for dyes removal from water. Activated materials presented surface areas of $1375.67 m^2 g^{-1}$ and $996.65 m^2 g^{-1}$ for AC and AT, respectively which are above the range of those that have been reported in the literature.

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