USE OF SAWDUST *Eucalyptus* sp. IN
THE PREPARATION OF ACTIVATED CARBONS

Utilização de serragem de *Eucalyptus* sp. na preparação de carvões ativados

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ABSTRACT

Wood sawdust is a solid residue, generated in the timber industry, which is of no profitable use and can cause serious environmental problems if disposed inadequately. The aim of this study was to use the eucalyptus sawdust in the preparation of activated carbons AC) and test them as adsorbents of methylene blue (MB) and phenol, representative pollutants from aqueous effluents of various industries. The eucalyptus sawdust was characterized by instrumental analysis such as elementary analysis (CHNS-O), thermogravimetric analysis (TGA), infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The activated carbons were prepared by physical activation with carbon dioxide AC_CO_2, (10° C min^{-1}, 850° C, 1h) and by chemical activation with potassium carbonate $AC\_K_2CO_3$ (10° C min^{-1}, 850° C, 3h). The AC_CO_2 and $AC\_K_2CO_3$ were characterized by CHN-O, TGA, FTIR, N_2 adsorption/desorption (BET) to evaluate the specific surface area and SEM. The resulting activated carbons were tested for their ability to adsorb MB and phenol in water. The activated carbons produced in this work were predominantly microporous and showed specific surface area of about 535 m^2 g^{-1}. The $AC\_K_2CO_3$ was more effective in the adsorption of MB (81 mg g^{-1}) and phenol (330 mg g^{-1}) than AC_CO_2 (32 mg g^{-1} and 172 mg g^{-1}, respectively, for MB and phenol).

Index terms: Agro-industrial residues, adsorbents, pollutants.

RESUMO

A serragem é um resíduo sólido, gerado na indústria madeireira, que não tem uso rentável e pode causar sérios problemas ambientais quando disposta inadequadamente. Neste estudo, objetivou-se utilizar a serragem de eucalipto na preparação de carvões ativados (AC) e testá-los como adsorventes do corante azul de metileno (MB) e fenol; moléculas que representam poluentes de efluentes industriais. A serragem de eucalipto foi caracterizada por análises instrumentais, tais como: análise elementar (CHNS-O), análise termogravimétrica (TGA), espectroscopia na região do infravermelho (FTIR) e microscopia eletrônica de varredura (SEM). Os carvões ativados foram preparados por ativação física com dióxido de carbono AC_CO_2, (10° Cmin^{-1}, 850^oC, 1h) e pela ativação química com carbonato de potássio- $AC\_K_2CO_3$ (10° Cmin^{-1}, 850° C, 3h). O AC_CO_2 e $AC\_K_2CO_3$ foram caracterizados por CHN-O, TGA, FTIR, adsorção/dessorção de N_2 (área BET) para avaliar a área superficial específica e SEM. Os carvões ativados resultantes foram testados quanto à capacidade de adsorver MB e fenol na água. Os carvões produzidos apresentaram características de material microporoso, apresentando área superficial média de cerca de 535 m^2g^{-1}. O $AC\_K_2CO_3$ foi mais eficiente na adsorção do MB (81mgg^{-1}) e fenol (330mgg^{-1}) que o AC_CO_2 (32mg g^{-1} e 172 mg g^{-1}, respectivamente, para MB e fenol).

Termos para indexação: Resíduos agroindustriais, adsorventes, poluentes.

INTRODUCTION

In Brazil, a land with a large reforested area, many eucalyptus species are widely planted because they are important suppliers of raw material for various industrial purposes (SILVEIRA et al., 2004; TRUGILHO, 2009; CUSTÓDIO; BARBIN, 2009; DOMINGUES et al., 2010).

The reuse of waste biomass is becoming an issue of ever growing importance, not only for economic reasons but also for environmental ones (MARTINS et al., 2007).

Biomass, mainly derived from agricultural solid waste, is a preferable option for activated carbon (AC) precursors (GONÇALVES et al., 2007). Those materials are cheap, renewable and abundantly available (DIN et al., 2009). Thus, one of the possibilities of using such waste is in the manufacture of ACs, which are a well-known materials used in an increasing number of environmental applications like water and wastewater treatment, gas filters, and so forth. (POLLARD et al., 1992; DIAS et al., 2007).

ACs have a very complex pore structure, large surface area, good catalytic activity and chemical stability, and a variety of oxygen-containing functional groups on

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the surface (JUAN; KE-QIANG, 2009). The characteristics of ACs depend on the activation methods as well as on the properties of the starting materials. There are two activation methods in the production of activated carbons: physical (or thermal) and chemical (MARSH; RODRÍGUEZ-REINOSO, 2006). The physical activation method involves carbonization of the raw material and the subsequent activation at high temperature in a carbon dioxide atmosphere or with steam. The chemical activation method involves the carbonization of the raw material previously impregnated with a chemical agent such as zinc chloride, phosphoric acid, potassium hydroxide or others (DUMAN et al., 2009).

ACs have demonstrated themselves to be highly efficient for the removal of many compounds such as dyes, metal ions, and low molar mass organic compounds (GONÇALVES et al., 2007; OLIVEIRA el at., 2009; DURAL et al., 2011; MAHMOODI et al., 2011; NIETO-DELGADO et al., 2011).

Methylene blue (MB) which is the most commonly used substance for dying cotton, wood and silk can cause some harmful effects. Therefore, the removal of such dye from waste streams before discharge to public water sources is of primary concern (DENG et al., 2009).

Phenol is frequently derived from petrochemicals, plastics, paints, pharmaceutical and steel industries (CANIZARES et al., 2006). Excessive presence of phenol and its derivatives in natural water is considered as a serious threat to human health and overall water quality (BHATNAGAR, 2007). The discharge of industrial wastes (generated in chemical industry activities) and the leaching from agricultural and forest land (when the application of chemical fertilizers and pesticides is made) are the main sources of organic pollution in several water streams (DIAS et al., 2007). Phenolic compounds are very harmful to organisms even at low concentrations due to their toxicity and carcinogenic properties. Thus, it is recommended to fully remove the phenol from industrial effluents before entering the water stream (OZKAYA, 2006).

In this article, the preparation and characterization of activated carbons from eucalyptus sawdust were reported and the adsorption equilibrium of methylene blue and phenol on those ACs were evaluated.

MATERIAL AND METHODS

Preparation of the activated carbons

Eucalyptus sp. sawdust samples, used as a carbonaceous precursor for the production of activated carbons, were donated by a sawmill in the city of Lavras, Brazil. The sawdust was dried at 80°C for 6 hours, sieved into a uniform size (25 mm) and stored at room temperature.

Two methods of activation were used to prepare the ACs: physical activation with CO_2 (AC_CO_2) and chemical activation with K_2CO_3 (AC_K_2CO_3).

To produce the AC_K_2CO_3, the eucalyptus sawdust was impregnated (1 g g^{-1} K_2CO_3) with K_2CO_3 using 100 mL of distilled water, 8 g of the eucalyptus sawdust and 8 g of K_2CO_3. The mixture was heated at 80° C under stirring until complete drying and put in an oven at 110° C for 24 h. Then, the material was placed in a tubular furnace and heated at a rate of 10° C min^{-1} until 500° C and held at this temperature for 3 h. The resulting material was washed to remove the remaining chemical agent and dried for 24 h at 80° C.

In the physical activation (to produce AC_CO_2), 10 g of the eucalyptus sawdust was previously pirolysed in a tubular furnace at 500° C for 3 h (heating rate of 10° C min^{-1}) under a N_2 atmosphere (200 mL min^{-1}). The pirolysed material was activated using a 150 mL min^{-1} CO_2 flow at 850° C (heating rate of 10° C min^{-1}) for 1 h.

Characterization of the activated carbons

The surface area and pore size distribution of the materials was obtained by means of N_2 adsorption/desorption isotherms at 77K in an Autosorb-1, Quantachrome. The specific surface area was calculated by the BET method and the pore size distribution was calculated by DFT.

Surface morphology of the produced activated carbons was investigated using scanning electron microscopy (SEM). The samples were prepared in stub holders with double face carbon tape, placed on an aluminum sheet and covered with gold, using a Balzers SCD 050 evaporator.

Analyses of CHN-O of AC_K_2CO_3, AC_CO_2 and of eucalyptus sawdust were obtained using a Thermo equipment, Flash EA 1112 series.

The thermogravimetric analysis of the materials was performed in a Shimadzu DTG-60AH. For the analyses, approximately 10 mg of each sample were heated at 10° C min^{-1} from 25 to 900° C under N_2 flow.

The functional groups on the AC samples were analyzed by Fourier Transform Infrared (FTIR) spectroscopy. All spectra were collected on a Digilab Excalibur, FTS 3000 series, operating in the infrared region of 400-4000 cm^{-1} with a 8 cm^{-1} resolution. The samples were prepared as KBr tablets.

Adsorption tests

The adsorption tests were conducted using solutions of methylene blue dye and phenol, whose molecular structures are shown in Figure 1.

Adsorption isotherms were obtained using 10 mg of adsorbent materials in contact with 10 mL of solutions with different concentrations (25, 50, 100, 200, 500 and 1000 mg L^{-1}) of adsorbate (methylene blue or phenol). The solutions were kept under stirring for 24 hours at room temperature (25±2° C).

The determination of the equilibrium concentration was made by UV-visible spectroscopy (Shimadzu-UV-1601 PC) for methylene blue (l=665 nm) and phenol (l=270 nm).

The isotherms were evaluated using the Langmuir and Freundlich models.

RESULTS AND DISCUSSION

Characterization of the activated carbons

Figure 2 presents the N_2 adsorption/dessorption isotherms at 77K of AC_CO$_2$ and AC_K$_2$CO$_3$.

The area obtained for the AC was 528 and 539 $m^2 g^{-1}$ for AC_CO$_2$ and AC_K$_2$CO$_3$, respectively. According to Suhas and Ribeiro Carrot (2007) activated carbon may have surface areas ranging from 500 to 2000 $m^2 g^{-1}$, showing that the surface area found for AC_CO$_2$ and AC_K$_2$CO$_3$ is characteristic of a porous material. In terms of surface areas, those differences are not significant; however, the carbons were activated in different ways, so they have different surface chemical groups. As the carbons have different surface chemistry, even with values of surface areas so close, they have different behaviors depending on the adsorbate used (MB or phenol).

The isotherm exhibits high N_2 adsorption at low pressures, indicating predominantly microporous AC. We can also observe a very small variation in the amount of adsorbed N_2 with increasing pressure (p/p$_0$), which is typical for microporous materials. Figure 3 confirms the microporosity of AC_CO$_2$ and AC_K$_2$CO$_3$, since most of the pores are smaller than 20 Å.

The micrographs of the precursor material (eucalyptus sawdust) and of activated carbons AC_CO$_2$, AC_K$_2$CO$_3$ are shown in Figure 4.
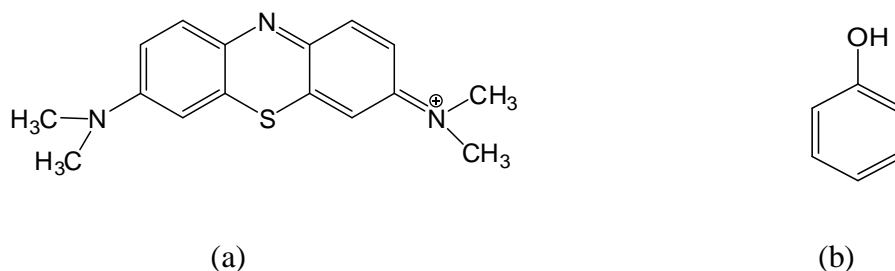

(a)   (b)

Figure 1 – Chemical structures of methylene blue (a) and phenol (b).

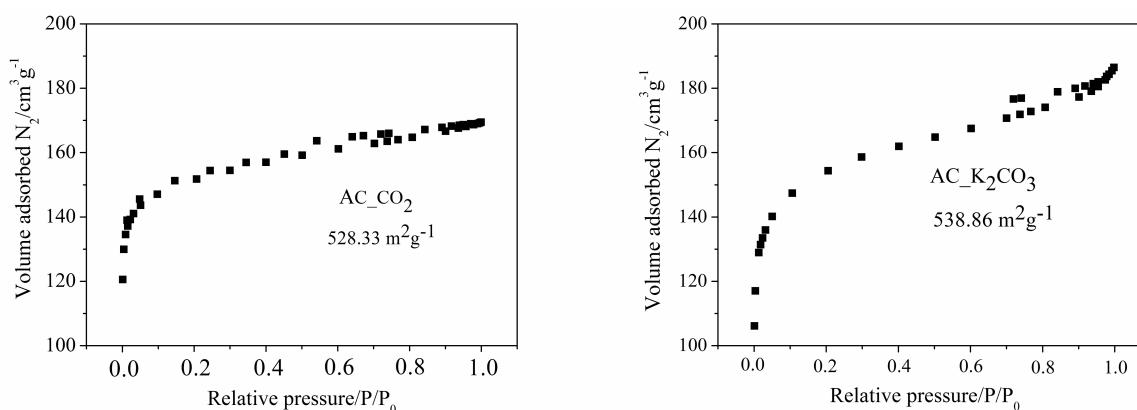Figure 2 – N_2 adsorption/desorption isotherms at 77 K, for AC_CO$_2$ and AC_K$_2$CO$_3$.

It is observed that the morphology of the precursor material changed after the pyrolysis and activation process, forming a structure with larger pores which can be attributed to the decomposition of labil compounds during the process.

The carbon, hydrogen, nitrogen and oxygen contents found in the sawdust and in the activated carbons by means of elemental analysis are presented in Table 1.

Comparing the ACs with the sawdust, it is observed that the carbon content increases and the levels of hydrogen and oxygen decrease. This is because the process of pyrolysis and activation causes a release of volatile compounds, such as water and smaller hydrocarbons.

The physically activated carbon (AC_CO_2) has the higher carbon content compared to the chemically activated ($AC\_K_2CO_3$).

There is an increase in the C/H values when comparing the sawdust and activated carbons, which indicates an increase of the degree of aromaticity after the
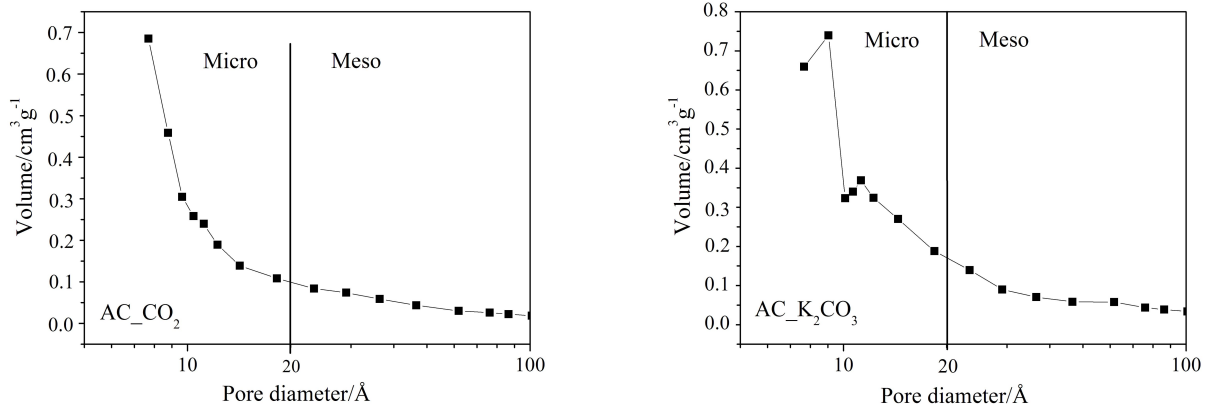

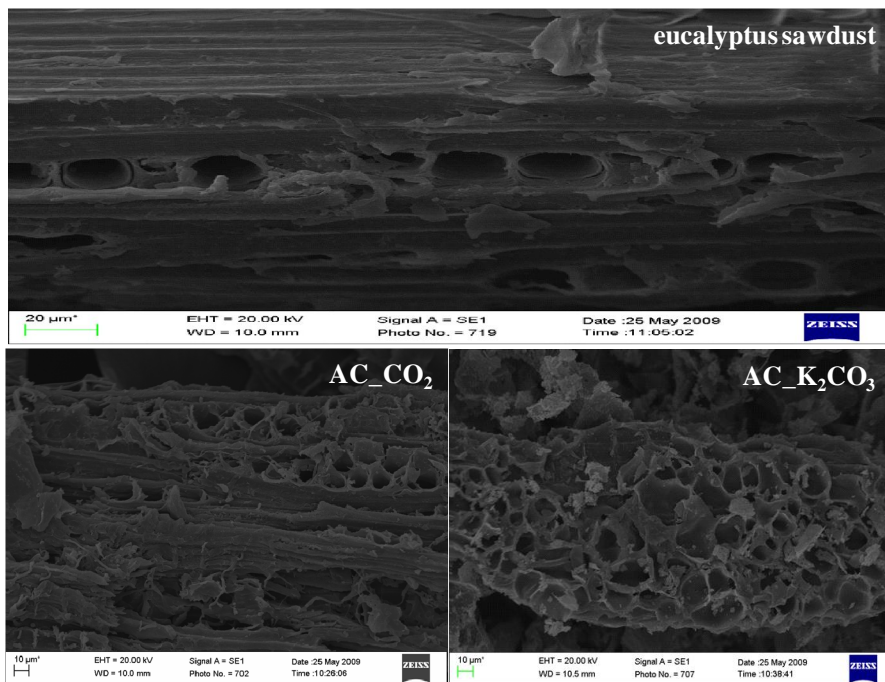Figure 3 – Pores size distribution of AC_CO_2 and $AC\_K_2CO_3$.

Figure 4 – SEM photographs for eucalyptus sawdust, AC_CO_2 and $AC\_K_2CO_3$.

process of pyrolysis and activation. The C/H has been accepted as an indication of condensation reactions or flavoring reactions, common in the carbonization and activation process (CHATTOPADHYAYA et al., 2006).

Elementary analysis shows that drastic changes occurred in the material and indicates the efficiency of the process of pyrolysis and activation, since the material produced contains higher amount of fixed carbon and aromatic units.

The FTIR spectrum of sawdust (Figure 5) shows a broad band around 3400 cm^{-1}, corresponding to the axial OH deformation. It also presents signals at 2930 cm^{-1}, which characterize symmetric and asymmetric vibrations of -CH$_2$- groups. A band near 1600 cm^{-1}, related to the stretching of lead C=C of aromatic groups; a band near the region of 1230 cm^{-1}, related to the vibration of the aromatic lignin ring, and the bands between 1000 and 1050 cm^{-1}, assigned to the stretching of C-O groups of lignin, cellulose or hemicelluloses or C-O-C group of cellulose and hemicelluloses, are also observed.

The infrared spectrum shows some functional groups present in the macromolecular constituents of wood (lignin, cellulose and hemicellulose). The molecules those containing these functional groups are modified during the pyrolysis and activation and, some of these molecules, when in the surface of the material, may make the activated carbons more acidic or alkaline. The spectra of AC_CO$_2$ and AC_K$_2$CO$_3$ are typical for charcoal and the absence of some bands related to functional groups that are present in sawdust, indicate the pyrolysis of the material. In these spectra we can observe a broad band around 3400 cm^{-1} (OH); a band near 1600 cm^{-1} (C=C of aromatic groups) and a band in the region of 1230 cm^{-1} (vibration of the aromatic rings).

According to Martins et al. (2007), charcoal from eucalyptus sawdust (pyrolysed at 500° C) presents bands between 3700 and 3600 cm^{-1}, corresponding to free -OH group, a broad band between 3400 and 3000 cm^{-1} that can be attributed to the presence of functional groups associated to phenolic OH, alcohols and carboxylic acids. The 1700 cm^{-1} band features double bonds of C=C or C=O, the 1620 cm^{-1} band is attributed to aromatic rings and the 1350 and 1450 cm^{-1} bands correspond to CH groups.

Table 1 – Elementary analysis of sawdust and activated carbons.

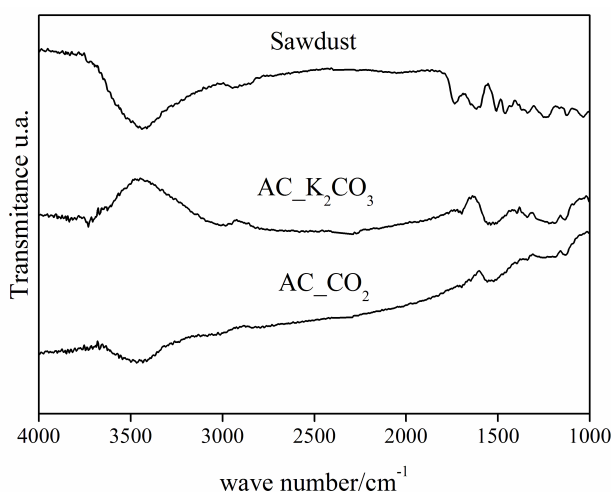| Materials	C (%)	H (%)	N (%)	O* (%)	C/H
Sawdust	45.5	6.2	0.13	48.1	7.34
AC_K$_2$CO$_3$	80.4	2.79	0.13	16.7	28.82
AC_CO$_2$	89.3	1.72	0.19	8.61	51.92

*Calculated from difference.

Figure 5 – FTIR spectrum of eucalyptus sawdust, AC_CO$_2$ and AC_K$_2$CO$_3$.

The characteristics of thermal degradation of lignocellulosic materials are strongly influenced by the chemical composition of these materials (cellulose, hemicelluloses and lignin) (ANTAL; VARHEGY, 1995) and the TG curves provide a semi-quantitative understanding of thermal degradation processes that occur during thermochemical conversion (GONZÁLEZ et al., 2009). Balci et al. (1993) showed that pyrolysis of wood proceeds by successive degradation of its principal lignocellulosics constituents (hemicelluloses, cellulose and lignin) which vary in thermal behavior. According Wielage et al. (1999) and Gröndahl et al. (2003), hemicelluloses decompose before the cellulose and lignin. García-Ibáñez et al. (2006) identified two areas of pyrolysis: the first, called the active zone, can be attributed to the volatile components generated during the decomposition of cellulose and hemicelluloses and the second, passive area, is due to the conversion of lignin (and part of the cellulose).

The thermogravimetric analysis data of the sawdust and of activated carbons (AC_CO_2 and $AC\_K_2CO_3$), under nitrogen atmosphere, are shown in Table 2. All materials showed a small weight loss at a temperature close to 100°C, which can be attributed to water loss.

The thermal degradation of sawdust occurs in two steps. The first weight loss occurs around 240° C due to degradation of hemicelluloses and part of cellulose. The second sawdust weight loss occurs at about 350° C due to the degradation of remaining cellulose and lignin. The mass stabilization of sawdust occurs at 540° C with a weight loss about 40%.

AC_CO_2 thermal degradation begins at temperatures around 450° C, stabilizing at 640° C. The residual mass CA_CO_2 was around 80%.

$AC\_K_2CO_3$ exhibit strong weight loss at temperatures ranging from 300 to 740° C. The residual mass presented by $AC\_K_2CO_3$ was about 85%.

The activated carbons show higher thermal degradation temperatures than the sawdust, as well as higher thermal stabilization temperatures. AC_CO_2 showed the highest initial thermal degradation temperature (450° C) while $AC\_K_2CO_3$ showed the highest thermal stabilization temperatures (740° C) and highest percentage of residual mass (85%). The activated carbons, compared to sawdust, have a higher thermal stability.

Figure 6 shows the adsorption isotherms of methylene blue dye (MB) and phenol using different activated carbons.

Table 2 – Summary of TG degradation temperatures of eucalyptus sawdust, AC_CO_2 and $AC\_K_2CO_3$.

Materials	DT in N_2 (°C)						RM (%)
	Initial	Final	Initial	Final	Initial	Final	
	Loss of water		Degradation		Degradation		
Sawdust	80	100	240	350	350	540	60
AC_CO_2	80	100	450	640	---	---	80
$AC\_K_2CO_3$	80	100	350	740	---	---	85

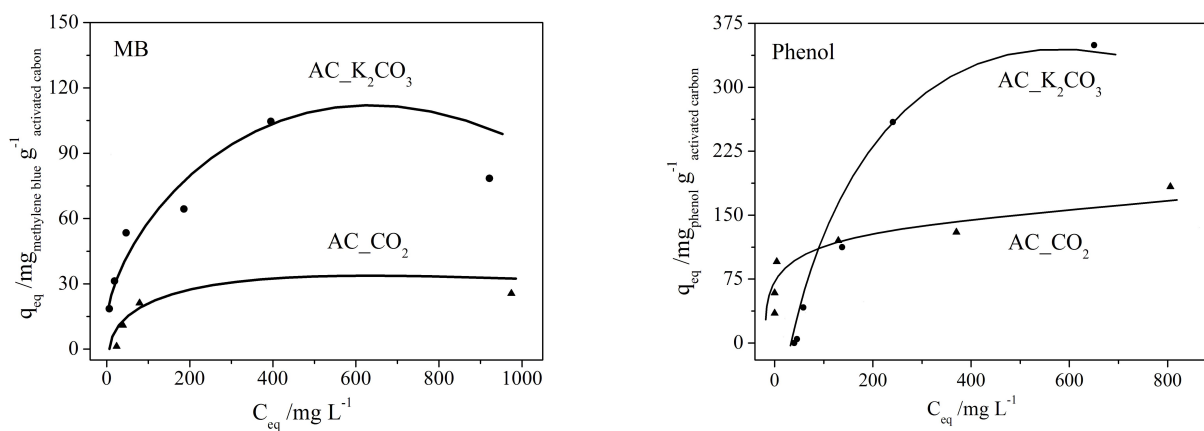DT = Degradation temperature; RM = Residual mass at 900° C

Figure 6 – Methylene blue dye and phenol adsorption isotherms.

The experimental results were adjusted according to the models of Langmuir and Freundlich. The best fit for MB was observed for the Langmuir model. The adsorption results are summarized in Table 3. $AC\_K_2CO_3$ (81 mg g^{-1}) was considerably more efficient in the adsorption of MB than AC_CO_2 (32 mg g^{-1}).

Adsorption of phenol on the different ACs was adjusted according to the Langmuir and Freundlich models. $AC\_K_2CO_3$ fits better to the Freundlich model and AC_CO_2, to the Langmuir model. The adsorption results of phenol are summarized in Table 4.

$AC\_K_2CO_3$ has a higher adsorption capacity for phenol (330 mg g^{-1}) compared to AC_CO_2 (172 mg g^{-1}) (Figure 6).

Table 3 – Langmuir parameters for the adsorption of methylene blue on activated carbons.

Activated carbons	Langmuir parameters		
	q_m (mg g^{-1})	K_L (L mg^{-1})	R^2
AC_CO_2	32	0.004	0.79
$AC\_K_2CO_3$	81	0.034	0.99

q_m = maximum amount of adsorption (mg g^{-1}); K_L = Langmuir constant (L mg^{-1}); R^2= coefficient of determination

Table 4 – Freundlich and Langmuir parameters for the adsorption of phenol on activated carbons.

Activated carbons				
	Freundlich parameters	K_F (mg g^{-1}) (L g^{-1})$^{1/n}$	n	R^2
$AC\_K_2CO_3$		1.26	1.11	0.92
	Langmuir parameters	q_m (mg g^{-1})	K_L(L mg^{-1})	R^2
AC_CO_2		172	0.071	0.96

K_F = Freundlich constant (mg g^{-1}) (L g^{-1})$^{1/n}$; 1/n= parâmetro de Langmuir; q_m = maximum amount of adsorption (mg g^{-1}); K_L = Langmuir constant (L mg^{-1}); R^2= coefficient of determination

CONCLUSION

The activated carbon obtained from the eucalyptus sawdust has certain characteristics such as surface area over 500 m² g⁻¹, high microporosity and potential for adsorption of pollutants commonly found in industrial effluents such as phenol and dye. This means that waste of eucalyptus sawdust can be used as precursor in the preparation of activated carbons (AC), using physical (CO_2) or chemical (K_2CO_3) activation. The two ACs prepared are microporous with a specific surface area around 535 m² g⁻¹ under the preparation conditions. Although AC_CO_2 and $AC\_K_2CO_3$ have several similarities, they have different adsorption capacities with respect to the tested contaminants (methylene blue and phenol). The $AC\_K_2CO_3$ was most effective in the adsorption of methylene blue (81 mg g⁻¹) and phenol (330 mg g⁻¹).

ACKNOWLEDGEMENTS

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