

## REMOVAL OF PHENOL BY ADSORPTION ON ACTIVATED CARBON FROM AQUEOUS SOLUTION

E.F.Tchikuala<sup>1</sup>, P.A.M. Mourão<sup>2</sup>, J.M.V. Nabais<sup>3</sup>

1 Departamento de Química, Escola de Ciências e Tecnologia, Centro de Química de Évora, Instituto de Investigação e Formação Avançada, Universidade de Évora, Rua Romão Ramalho nº59, 7000-671 Évora; Departamento de Ciências Exactas, Universidade Katyavala Bwila, Rua José Falcão nº67, 1725 Benguela, Angola (tchikuala@hotmail.com)

2 Departamento de Química, Escola de Ciências e Tecnologia, Centro de Química de Évora, Instituto de Investigação e Formação Avançada, Universidade de Évora, Rua Romão Ramalho nº59, 7000-671 Évora (pamm@uevora.pt)

3 Departamento de Química, Escola de Ciências e Tecnologia, Centro de Química de Évora, Instituto de Investigação e Formação Avançada, Universidade de Évora, Rua Romão Ramalho nº59, 7000-671 Évora (jvn@uevora.pt)

### ABSTRACT

The work now reported aims the preparation, characterization and application of activated carbons on the phenol adsorption from liquid phase. The carbon samples were produced from Angolan woods wastes, namely Baobab and Nuati. All precursors were previously carbonized and then activated, by physical activation with CO<sub>2</sub>, or by chemical activation with H<sub>3</sub>PO<sub>4</sub> and KOH. The activated carbons samples produced by CO<sub>2</sub> activation have apparent surface higher than 800m<sup>2</sup>/g, pore volume from 0.36 to 0.40cm<sup>3</sup>/g, mean pore width around 0.74nm and basic nature. The chemical activated samples show apparent surface area between 395 and 1682m<sup>2</sup>/g, pore volume from 0.17 to 0.65cm<sup>3</sup>/g and mean pore width around 1.08nm. The adsorption of phenol, a hazardous and problematic pollutant, from aqueous solutions reach a maximum adsorption capacity around 240mg/g at an equilibrium concentration of 1mg/L.

**Keywords:** Activated Carbon, Adsorption, Phenol, Wood Wastes

### INTRODUCTION

The search of new and less expensive precursors, in particular natural or industrial wastes, for the activated carbon (AC) production for wastewater treatment is an important area in environmental sciences. Phenols are one of the organic pollutants typically discharged into the environment causing unpleasant taste and odor, being very toxic, even at low concentrations. The sources of this hazardous compounds are very distinct, but also very common, namely in chemical, paint, paper, resin, dyes, pesticides industrial sectors. All this facts, turns imperative their identification, quantification and removal from different mediums, in particular from liquid phase [1-3].

The adsorption process using activated carbons (ACs) is often considered, amongst the different methods used to remove pollutants, from aqueous or gaseous phases, as the most efficient [1-5]. These features put the pressure on the production of cheaper adsorbents, in particular ACs, in a large scale from a selection of relatively low cost wastes, containing high carbon content, like lignocellulosic resources, making this process and products economical viable [3,4].

The existence of enormous amounts of wood wastes in Angola, usually used only as source of heat energy, creates the possibility of use those wastes for the production of ACs. Furthermore, if implemented, the setup of an industrial unit for the production of ACs could be a very powerful chance for the population of this developing country, where the environmental problems associated to this development are rising. At last, the test of the ACs produced on the adsorption of an important probe molecule, like phenol, from liquid phase give us much information about the properties of the ACs produced from those wood precursors [4,5].

## EXPERIMENTAL

The wood wastes, from Baobab and Nuati trees, collected in Angola, region of Benguela, were crushed in fragments of size up to 3 mm before its pre-washing with an acid aqueous solution of  $H_2SO_4$ . The suspension was filtrated and the solid remnant washed with distilled water, until the pH doesn't change, and then oven dried at  $110^\circ C$ .

The production of ACs were done in a horizontal tubular furnace. In the case of physical activation, the first step was the carbonization of the precursor under nitrogen, followed by the activation with  $CO_2$ , by maintaining the carbonized material under  $CO_2$  flux for a specific time at  $800^\circ C$ , in order to obtain the desired level of burn-off. In the case of chemical activation, the precursors were impregnated with the chemical agent,  $H_3PO_4$  and KOH, and the pre-dried mixture was heated under a flux of nitrogen at  $400^\circ C$ . In the two processes, after activation, the samples were cooled to room temperature under an inert atmosphere and then removed from the furnace. In the case of chemical activated samples, the carbon material was washed to remove the excess of chemical agent and other residual substances, like ash. The samples designations uses the following nomenclature (Wood Name)-(CO<sub>2</sub>) for the physical activated samples, and (Wood Name)-(chemical activating agent), for chemical ACs.

The precursors were characterized by thermogravimetric analysis and helium pycnometry. The content in cellulose and lignin was made by Agroleico (Porto Salvo, Portugal) using Portuguese Standards NP2029 and ME-414, respectively. All ACs samples were characterized by nitrogen adsorption at 77K, FTIR, CHNS-O elemental analysis and determination of pH of point of zero charge.

The phenol adsorption from liquid-phase was carried out at  $25^\circ C$  and acid medium, pH  $\sim 3$ , on Erlenmeyer flasks hermetically closed, under agitation on a thermostated shaker bath. After the contact time of 24h, the AC suspensions were filtered, and the residual pollutant concentration determined by UV/Visible spectrophotometry, using a PerkinElmer Lambda 850 spectrophotometer, at a wavelength of 269nm. The phenol was purchased from Aldrich with a purity greater than 99.99%.

## RESULTS AND DISCUSSION

The precursors show different characteristics, namely density,  $1.5076$  and  $1.4550g/cm^3$ , and contents in cellulose, 36.9 and 46.4%wt, hemicellulose, 9.2 and 13.9%wt and lignin, 7.4 and 24.6%wt, respectively for Baobab and Nuati woods.

The characterization of the ACs samples, by nitrogen adsorption/desorption isotherms, indicates the presence of porosity with a good structural characteristics, as can be seen in Table 1. All isotherms are of type I according to the IUPAC classification [6], which indicates that ACs are of microporosity nature. Nevertheless, some differences can be seen among the produced ACs, in particular it is clearly visible the higher apparent surface area, external area and pore volume for the samples activated with  $H_3PO_4$ . The lower values are obtained with the Nuati wood wastes when the activation is made with KOH.

Representative FTIR spectra are shown on Fig. 1. We can see, in this case for samples produced from Baobab, that different activating agents produces ACs with dissimilar surface chemistry as well. A complete discussion will be presented at the congress.

Table 1. Textural characterisation of the ACs.

Sample	$A_{BET}$ ( $m^2/g$ )	$A_s$ ( $m^2/g$ )	$V_s$ ( $cm^3/g$ )	$L_0$ (nm)
Baobab – CO <sub>2</sub>	875	44	0.40	0.74
Nuati – CO <sub>2</sub>	801	22	0.36	0.74
Baobab – KOH	915	376	0.32	-*
Nuati – KOH	395	23	0.17	1.08
Baobab – H <sub>3</sub> PO <sub>4</sub>	1682	813	0.57	-*
Nuati – H <sub>3</sub> PO <sub>4</sub>	1660	803	0.65	-*

( $A_{BET}$  – apparent surface area,  $A_s$  - external area determined by the alpha-s method,  $V_s$  - pore volume from alpha-s method,  $L_0$  - mean pore width, \* values of  $L_0$  not estimated because the isotherms present hysteresis at low pressure).

Figure 1. FTIR spectra of ACs prepared from Baobab.

All samples present adsorbed amounts of phenol around 125mg/g at equilibrium concentration of 0.5mg/L. When the equilibrium concentration double the maximum amount adsorbed is superior to 240mg/g. More data will be presented at the congress.

## CONCLUSIONS

The goal of this work, the production of ACs from Angola wood wastes, and posterior application for the removal of phenol from liquid phase, was achieved. This lignocellulosic precursors wastes presented a good potential for the production of ACs by physical activation with CO<sub>2</sub> and by chemical activation with KOH and H<sub>3</sub>PO<sub>4</sub>. The ACs surface chemistry reflects the nature of the activating chemical agent. Regarding the porous structure, the ACs produced shows high pore volume and surface areas. This mixture of properties makes the ACs samples on suitable adsorbents to be used in liquid phase adsorption, in particular for small molecules of hazardous substances, like phenolic compounds as phenol. In conclusion, this study have proven the suitability of the Angolan Nuati and Baobab wood to produce interesting ACs, which contributes to the valorization of this wastes.

## References

- [1] R.C. Bansal, Meenakshi Goyal, Activated Carbon Adsorption. Boca Raton: CRC Press, 2005.
- [2] Moreno-Castilla C. Adsorption of organic molecules from aqueous solutions on carbon materials. Carbon 42 (2004) 83–94.
- [3] J.M. Misihairabgwi, A. Kasiyamhuru, P. Anderson, C.J. Cunningham, T.A. Peshkur, I. Ncube. Adsorption of heavy metals by agroforestry waste derived activated carbons applied to aqueous solutions, African Journal of Biotechnology 13 (2014) 1579-1587.
- [4] P.A.M. Mourão, P.J.M. Carrott, M.M.L. Ribeiro Carrott, Application of different equations to adsorption isotherms of phenolic compounds on activated carbons prepared from cork, Carbon 44 (2006) 2422–2429.
- [5] I.P.P. Cansado, P.A.M. Mourão, A.I. Falcão, M.M.L. Ribeiro Carrott, P.J.M. Carrott, The influence of the activated carbon post-treatment on the phenolic compounds removal, Fuel Processing Technology 103 (2012) 64-70.
- [6] M. Thommes, K. Kaneko, A. Neimark, J.P. Olivier, F. Rodriguez-Reinoso, J. Rouquerol, K.S.W. Sing, Physisorption of gases, with special reference to the evaluation of surface area and pore size distribution (IUPAC Technical Report). Pure Appl. Chem. 87(9-10) (2015) 1051–1069.

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