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VALORIZATION OF A CAMEROONIAN SPECIES: MOABI (*BAILLONELLA TOXISPERMA Pierre*) INTO ACTIVATED CARBONS

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ABSTRACT

Activated carbons have been prepared from the chips of Moabi by chemical activation. We first carried out the chemical characterization (chemical composition, functional groups) of the chips of Moabi.The chemical characterization gave us extends of cellulose (40.5%) and lignin (29.6%) while an average pore volume is equal to 52.1% and its degree of crystallinity founded is 52%.We also characterize the sample with the scanning electron microscopy.

Secondly, the optimizations of conditions for preparation have been done. The parameters investigated concerned temperature (600 and 700°C), heating rate(1 and 3°C), the residence time (2 and 5 hours) for the carbonization process while, the concentration of NaOH (20%, 40% and 60%), the time of the impregnation (2 and 6hours) and the impregnation ratio of biomass/reagent(NaOH)(1/20 and 1/15) for the activation process. The calculation of the iodine numbers conducted to choose the best conditions of preparation of our activated carbons. The optimum conditions retained were: 700°C, 1°C/min and 2 hours for carbonization process and 20%, 2 hours and 1/15 for activation process, which gave us the iodine number of 629 ± 13mg/g. This Iodine number obtained is compared to those of commercial activated carbons

Keywords: Optimization, activated carbon, moabi, chemical activation, iodine number.

INTRODUCTION

Since the 19th century, charcoal has been replaced by news energies sources (petroleum, nuclear energy)[1]. Nuclear disasters and depletion of petroleum resources have giving a lot of interest to the use of charcoal. During the last two decades, industrials and researchers turned back to agricultural and forestry wastes to prepare charcoal which can constitute an alternative solution to those problems cited above. This waste has the advantage to be environmental friendly, renewable, abundant and available in a lot of form and quantity. Among the charcoals, special attention is paid to the activated carbon because they are highly porous, carbon adsorbents, hydrophobic and non polar. Activated carbon is highly demand in the industry for its reductive, adsorbent and dehydrated properties. The activated carbon can be obtained by chemical or physical activation of biomass material. The chemical activation of plant material in order to obtain activated carbon has been the subject of many studies[1-5]. The advantage of chemical activation is to operate at low carbonization temperatures and obtain large specific surface areas. Nowadays, activated carbons are widely used in the extraction of chemical species in the aqueous or in the gas phase due to their excellent adsorption capacity due to their large specific surface area and porosity development. Thus, activated carbons have a privileged place in the purification of water, fading sugars, recovery of volatile solvents, dyes fixing and gas treatment[6-10].

In the present work, we report the preparation of activated carbons from the chips of Moabi using NaOH as activating agent. The objective of the study is twofold. Firstly, it is to optimize the factors influencing the carbonization and activation with NaOH solution. On the other hand, we want to valorize the residues of Moabi which is abundant but less used locally. The purpose of this study is to obtain activated carbons from heavy wood, as performing as those already marketed.

MATERIALS AND METHODS

Sampling and characterisation

The chips of moabi used in this study were collected from a Small and Medium Enterprise (ETS BOIS EQUATORIAL) in Yaoundé (Cameroon). The chips of Moabi still wet were oven dried at 110°C and store in the polyethylene bag. One part of them were converted into sawdust with diameter less than or equal to 0.40 mm using a mill blade RETSCH SM100. We characterized the sawdust chemically by CTFT (Centre Technique Forestier Tropical) protocols and standard French T12011 and the physical properties including the density following IAWA (International Association of Wood Anatomists) Code. The density is given by formula:

$$\mathbf{d} = \frac{m_2}{(m_1 + m_2) - m_3} \quad \text{(I)}$$

where m_1, m_2, $m_{3(g)}$ represent respectively the mass of the tank filled with water, the mass of a sample of dry material and the mass of flask containing water and the sample. Humidity ratio was also determined according to standard French T12011. Average pore volume (C) has been determined by the formula:

$$\mathbf{C} = \left[\frac{1 - \rho_0}{\gamma_s}\right] \times 100 \quad \text{(II)}$$

Where

ρ_0: specific density of dry wood (g/cm^3) and γ_s: specific weight of the timber without pores (1.5 g/cm^3).

Infra-Red spectrum were recorded with an Infra-Red Fourier Transform Spectroscopy in Diffuse Reflection mode on KBr pellets made of 15 % clay in oven-dried KBr using a Bruker, IFS 55 spectrometer. The spectrum, recorded from 4000 cm^{-1} to 600 cm^{-1} with a resolution of 4 cm^{-1}, was obtained by accumulating 200 scans.

The XRD analysis was done on a Bruker AXS model D8 Advance diffractometer, with Cu-Kα radiation, under 40 kV and 30 mA operating conditions, to identify the constituent phases of the whole bulk samples. The data were recorded between 2° and 45° for whole-bulk samples using a step scan 0.02° and a step time of 2.0 s .The powder mounts were prepared through gentle hand mortar grinding (~1g of bulk wood) until the particle size was decreased below Ø< 250µm, and placed afterwards on sample holder, with gentle pressure so as to limit any orientation. The XRD analysis can be used to determine the degree of crystallinity with the formula:

$$\mathrm{CrI} = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad \text{(III)}$$

Where I_{002} is the maximal intensity of diffraction pick of (002) plan and I_{am} the intensity of the sample amorphous phase.

The morphological characterisation of the sample was also done by the scanning electron microscope (SEM) using Philips apparatus.

Optimization of parameters of preparation

Carbonization process

The factors studied were: the carbonization temperature, the heating rate and the residence time. As part of our work where the production of

charcoal is to promote, the slow pyrolysis technique is the most interesting. We define two levels for each factor studied (Tableau IV)
The study of the thermal parameters of screening is performed on four samples of our raw material. We establish the following test matrix (Table V)

Preparation procedure of charcoals
Charcoals were prepared according to the following experimental protocol:

The chips were dried for 24 hours in an oven at 110°C. The oven dried chips were then carbonized in a closed reactor (in the absence of oxygen) placed in a furnace with the require heating rate, to the known carbonization temperature, until the precise residence time. After cooling, the charcoals obtained were first ground and sieved to obtain an 80µm fraction (diameter ≤ 80 µm). This fraction was oven dried for 24 hours at 110 ° C and stored in sealed containers placed in a desiccator until they were ready to be studied. Charcoals obtained were characterized by measuring their iodine number.

The experimental setup used is an ISUNI mark electric furnace with automatic regulation, having the temperature programmer relied to the furnace by a type K thermocouple and having a quartz reactor.

This adsorption can be used to control the efficiency of the charcoal in the water treatment[1].The adsorption of iodine from the aqueous phase is a useful tool for product control in the manufacture of activated carbon. The sample studied is characterized in terms of iodine number (mg/g) using iodine solution of 0,200±0,001 N. A volume of 20 ml of iodine solution was treated by 0,2000±0,0001g of activated carbon. After equilibrium, the waste iodine of the filtration was titrated by 0,1N of sodium thiosulfate. The formula[11] as follows is used:

$$\text{Iodine number (mg/g)} = \frac{25{,}4 \times (20 - V_n)}{m_{ca}}$$

(IV)

Where m_{ca}(g): mass of activated carbon and V_n (ml): volume of sodium thiosulfate at equilibrium. Iodine and sodium thiosulfate used in this study are manufactured by Riedel-de-Haën.

Optimization of the impregnation parameters
The effect of the activating agent on the efficiency of the activated carbon obtained was studied through these factors: the concentration of the activating agent, the time of the impregnation and the ratio biomass/ impregnation solution. The activating agent used is NaOH. We define these levels for each factor studied (Table VI)
The methodology of the experimental design provides the maximum information with the minimum experiments. This methodology carries out the complete study of the influence of all factors on the given process and its optimization[12].

We establish the following test matrix using the methodology cited above (Table VII).

Preparation procedure of activated carbons
Activated carbons were prepared according to the following experimental protocol:
The chips were oven dried for 24 hours at 110°C. These chips were then treated for a limited time of the impregnation, in a NaOH aqueous solution of known concentration, in the precise Impregnation ratio of biomass/reagent. After the impregnation, the chips are drained in the open air for at least 12 hours before being carbonized in a closed reactor (in the absence of oxygen) placed in an electric furnace described above. Carbonization was carried out at the optimum thermal parameters determined above. After

cooling, the charcoals obtained were washed with 5 % hydrochloric acid and distilled water, and finally dried. These charcoals were first ground and sieved to obtain a 80µm fraction (diameter ≤ 80 µm). This fraction was oven dried for 24 hours at 110°C and stored in sealed containers placed in a desiccator until they were ready to be studied. Activated carbons obtained were characterized by measuring their iodine number (mg/g) according to the protocol described above.

RESULTS

Characterisation of the raw material

The chemical composition and the physical properties of the raw material are shown in tables I and II. Each value is the mean of three replications ± Standard Dispersion (SD) due to the variability of the biomass properties. Table III presents the infrared bands characteristic of wood species (Moabi). Its degree of crystallinity is 52% where the maximal intensity of diffraction pick situated at reticular plan indexed I_{002} around $2\theta_{002}$ = 22.5°C and the intensity at I_{am} at $2\theta_{002}$ =18°C. FTIR spectrum and X-ray diffractogram of Moabi are presented in figures I and II. Scanning electron microscopy (SEM) of Moabi is also presented in figure III

Preparation of charcoals and activated carbons

The parameters used for the preparation of the charcoals and the activated carbons are summarized in tables V and VII. The iodine number of a commercial activated carbon indexed F100 prepared by CHEMV society from bitu Coal with 0.8 -1.0 mm of granulometry and 900m^2/g of BET surface area is found at 838 ± 13 mg/g.

DISCUSSION

Analysis of raw material (chips of Moabi)

Carbonization of the raw material depends on the nature and the properties of biomass and can influence the adsorbent properties of the charcoals obtained. It depends on the degradation temperature of the components of the raw material. Thermal degradation of plants is the result of separated degradation[20] of hemicelluloses within 200 and 260°C, cellulose within 240 and 350°C, lignin within 280 and 500°C. Then, carbonization temperature must be greater than this temperature range. This is why physicochemical characterization of the raw material is used to predict parameters in the pyrolysis process. Moreover, it is shown that the amount of charcoal is proportional to the fraction of lignin present in the biomass[17]. Moabi chemical composition displayed characteristics of tropical wood namely Lignin, cellulose, hemicelluloses, extractable and ash contents (Table I). Similar observations have been reported in the literature[18] where chemical composition of Moabi have been studied by the laboratory of CTFT with cellulose (40.8–46.1 %), lignin (30.3–27.7 %), pentosans (15.7–14.0 %).There are some differences with the values of extractable and ash content, respectly 7.35 % and 0.33 %. The balance of the chemical composition shows that the rate of extractable of our species is high due to the fact that chemical composition of wood changes with species, age, part of plant and growing environment.

The value of our density found in the present study shows that Moabi is a heavy wood which is in agreement with the literature[16] while its X-ray Diffractogram revealed the semi-crystallynity nature of Moabi. The scanning electron microscopy (SEM) is used to characterise morphologically our sample before and after the activation process in order to show the effect of the chemical activation on the porosity.

Effect of carbonization parameters

Effects of temperature, heating rate and residence time on the carbonization was evaluated through the iodine number test. Increasing temperature from 600 to 700°C showed an increase iodine number from 127 to 152 mg/g when the residence time at the final temperature is 2 hours and the heating rate is 3°C/min. This increasing temperature could been explained by the fact that at 600°C, the pores are not completely liberated by volatile components as observed by PORQUET[2] in the preparation of activated carbons magnetisable from Oak . Minimum activation temperature exists for chosen heating rate and residence time.

A further increase, in the residence time from 2 to 5 hours, when the carbonization temperature is 700°C and the heating rate 1°C/min illustrated a gradual decrease in iodine number from 159 to 108 mg/g. A longer residence time affected negatively the porosity of the charcoal. About 2 hours, the micropores are already completely liberated by volatile components. Similarly, increasing heating rate from 1 to 3°C/min showed a decrease in iodine number from 159 to 152 mg/g attributed by the fact that the rapid energy input does not homogeneously release micropores.

Effect of the activating agent

Effect of activating agent on the activation was investigated with its concentration, the time of impregnation and the ratio biomass/impregnation solution.

Increasing concentration of the activating agent from 20 to 60 % resulted in a decrease in iodine number from 629 to 362 mg/g or from 559 to 438 respectively with the ratio biomass/impregnation solution of 1g/15ml or 1g/20ml when the time of impregnation is 2 hours. This increasing caused blockage of the pores leading to a dramatic decrease in accessible area. Additionally, increasing concentration would intensify a vigorous activation reaction, which leads to transition of micropores–mesopores into macropores lowering the charcoal yield as confirm by FOO[19] in Microwave-assisted preparation and adsorption performance of activated carbon from biodiesel industry solid reside. The same phenomenon is ascribed when the biomass/impregnation solution goes from 1g/15ml to 1g/20ml with a decrease of iodine number.

When the time of impregnation is 6 hours, increasing concentration of the activating agent from 20% to 60% indicated an increase in iodine number from 398 to 476 mg/g or from 360 to 502 mg/g respectively with the ratio biomass/impregnation solution of 1g/15ml or 1g/20ml. The values obtained are lower than those found at 2 hours of impregnation. This phenomenon can be explained by the fact that at 2 hours, transition of micropores-mesopores into macropores is completely achieved, and then iodine number decreases gradually at 6 hours. But, small increasing of iodine number at 6 hours of impregnation with concentration of activating agent can be explained by creation of new micropores available to iodine from ultramicropores.

The iodine number of a commercial activated carbon indexed F100 prepared by CHEMV society from bitu Coal is found at 838 ± 13 mg/g. The best iodine number of the activated carbon obtained is 629± 13 mg/g. Then, better results can be obtained if the carbonization process was carried out by loading dried chips of Moabi into a tubular furnace[19] and heating to a carbonization temperature of 700 °C under purified N_2 flow (150 cm^3/min), with heating rate and residence time respectively at 1°C/min and 2 hours.

CONCLUSION

This work highlighted the feasibility of chips of Moabi as a promising precursor for the manufacture of activated carbon by chemical activation. Firstly, we have done the physicochemical and morphological characterisation of the raw material. Then, the methodology of the experimental research was used to optimize parameters (carbonization temperature, heating rate, residence time, concentration of the activating agent, time of impregnation, impregnation ratio) influencing the preparation of activated carbon. Test on iodine number are carried out to examine the effectiveness of activated carbon obtained. The values of iodine number of the activated carbon obtained are compared favorably to those of commercial activated carbons. The study of the parameters influencing the preparation of activated carbon through the methodology of the experimental research allowed determining the optimums conditions of chemical activation of the chips of Moabi with NaOH solution. The optimum parameters of the carbonization found in this study are as follows: 700°C of carbonization temperature, 1°C/min of heating rate and 2 hours of residence time. The optimum parameters of activating agent (NaOH) are as follows: 20% of concentration, 2 hours of time of impregnation and 1g/15ml of ratio biomass/impregnation.

Future studies should examine the characterisation of the activated carbon obtained into a determination of its BET surface area and pore size distribution.

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Table I: Chemical composition of Moabi

Sample	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Extractable (%)	Ashes (%)
Moabi	40,5±0,3	14,9±0,3	29,6±1,0	13,9±0,1	0,50±0,04

Table II: Physical properties of Moabi

Sample	Humidity rate (%)	Density of dry wood (%)	Average pore volume (%)	Moabi density at 12% humidity[16]
Moabi	12,2±0,2	0,718±0,006	52,1±0,4	0,820 – 0,940

Table III: Infrared bands characteristic of Moabi

Moabi (present study)	Oak[13]	Okoumé[14]	Wood[15]	
3409	3355	3418	3300	O-H bonded (shear): Polysaccharides
2925	2900	2901	2900	C-H (symmetric stretching): Polysaccharides.
1734	1734	1734	1740	C = O ester groups (symmetric stretch) acetylated xylans.
1597	1605	1593	1600	C = C aromatic ring (symmetric stretch): lignin, tannins.
1507	1510	1507	1505	C = C aromatic ring (symmetric stretch): lignin, tannins.
1463	1455	1465	1460	CH of methyl and methylene (asymmetric stretching): lignin, polysaccharides
1425	1425	1420	1425	Deformation in the plane of vibration and aromatic CH groups: lignin, polysaccharides
1374	1371	1374	1370	Deformation in the plane of groups C-H2: Polysaccharides
1332	1318	1320	1325	Aromatic C-O vibration units syringyl and deformation in the plane of CH2 groups: lignin, polysaccharides.
1240	1230	1240	1240-1230	Deformation of acetyl (xylan) and vibration syringyl units: lignin, polysaccharides.
1160	1163	1162	1160	COC vibration of hemicelluloses and cellulose (asymmetric stretching) polysaccharides
1124		1125		COC vibration glucose (asymmetric stretching)
1106	1110	1113	1110	OH vibration cellulose and hemicelluloses: polysaccharides
1049	1036	1033	1050	CO vibration cellulose and hemicelluloses: polysaccharides
898	898	896	895	Vibration glycosidic bonds: polysaccharides
818	806	802	810	Trisubstituted aromatic ring vibration: conifer lignins
662		662	680	C-OH deformation vibration in the plane: Cellulose

Table IV: Experimental domain for carbonization process

	Factor	Number of level	Level
U1	Carbonization température	2	600°C
			700°C
U2	Heating rate	2	1°C/min
			3°C/min
U3	Residence time	2	2 hours
			5 hours

Table V: Parameters used for the preparation of the charcoals

Test	Final température	Heating rate	Residence time at the final temperature	Charcoals of Moabi	
				Samples	Iodine number (mg/g) ± 13
1	600°C	3°C/min	2 hours	MO62	127
2	700°C	3°C/min	2 hours	MO72	152
3	700°C	1°C/min	2 hours	MO72*	159
4	700°C	1°C/min	5 hours	MO75*	108

Table VI: Experimental domain for activation process

	Factor	Number of level	Level
U1	concentration of reagent NaOH	3	20%
			40%
			60%
U2	Time of the impregnation	2	02 hours
			06 hours
U3	Impregnation ratio of biomass/reagent(NaOH)	2	1g/15ml
			1g/20ml

Table VII: Parameters used for the preparation of the activated carbon

Test	Samples	IMPREGNATION			Iodine number (mg/g) ± 13
		Duration (h)	Concentration	Ratio biomass/ impregnation solution	
1	2MON207-1	2	20%	1g/15ml	629
2	2MON407-1	2	40%	1g/15ml	546
3	2MON607-1	2	60%	1g/15ml	362
4	6MON207-1	6	20%	1g/15ml	398
5	6MON407-1	6	40%	1g/15ml	438
6	6MON607-1	6	60%	1g/15ml	476
7	2MON207-2	2	20%	1g/20ml	559
8	2MON407-2	2	40%	1g/20ml	508
9	2MON607-2	2	60%	1g/20ml	438
10	6MON207-2	6	20%	1g/20ml	360
11	6MON407-2	6	40%	1g/20ml	381
12	6MON607-2	6	60%	1g/20ml	502

Abbreviation of the sample is: *X*MON*Y*07-*Z*
Where *X* is the duration of impregnation, MO: Moabi, N: NaOH, *Y: Y0% of concentration*, 7: 700°C, Z: the impregnation ratio
Each value represents the mean of three replications ± Standard Dispersion (SD)

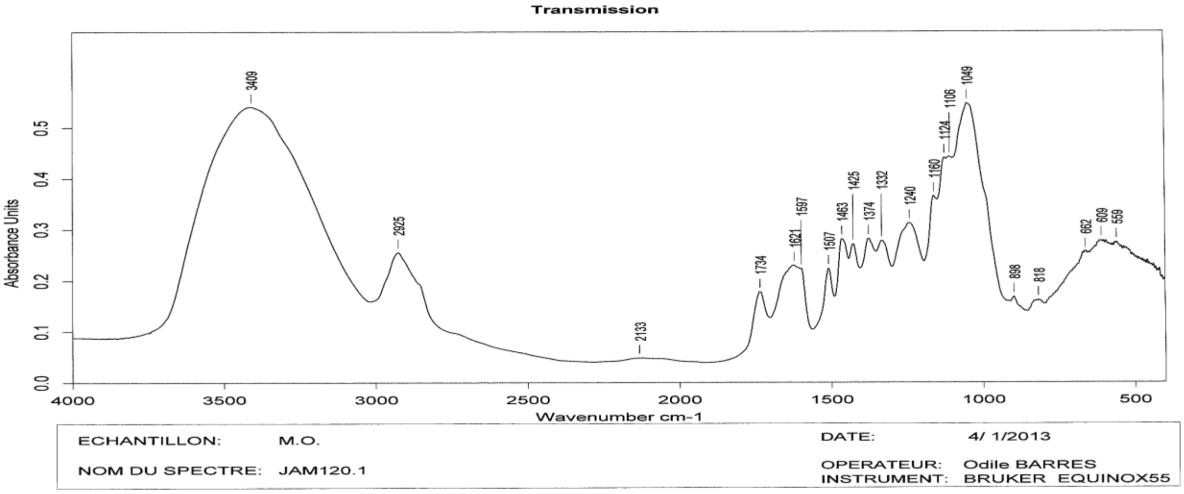

Figure I: FTIR Spectrum of sawdust wood of Moabi

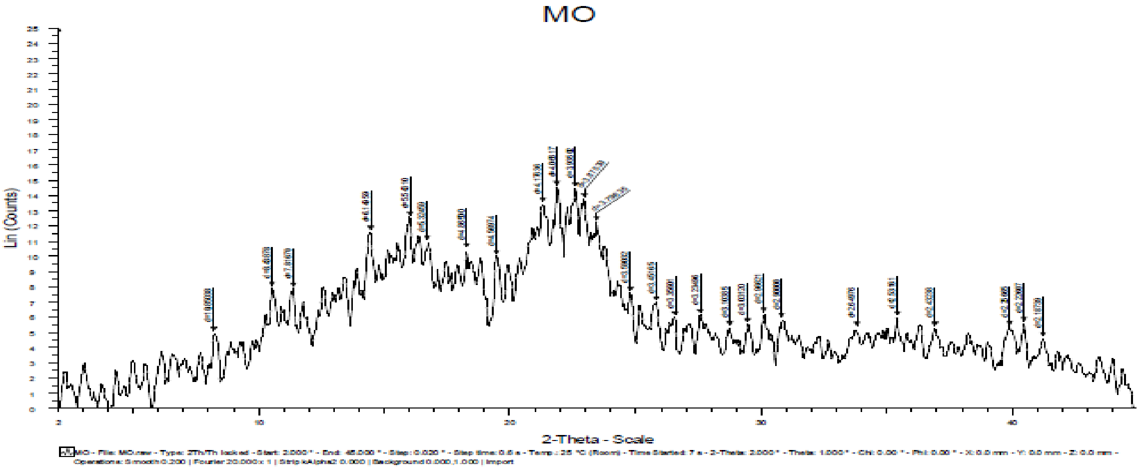

Figure II: Wood of Moabi diffractogram

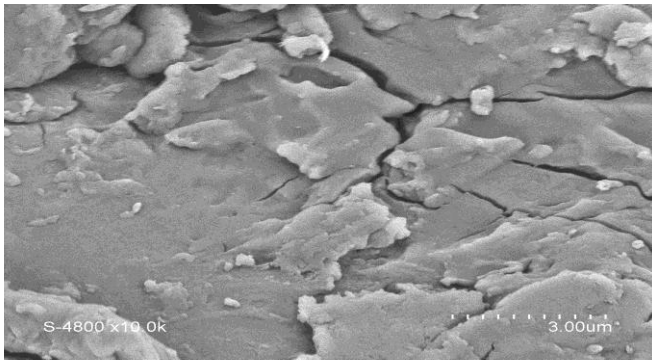

Figure III: Scanning electron microscopy of moabi