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ORIGINAL PAPER

Production and characterization of activated carbons from neem bark (Azadirachta indica)

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Abstract
The acquisition of activated carbon on the market remains a major concern for developing countries due to its non-availability on a large scale. The objective of this work is to determine the physical and chemical characteristics of activated carbons produced from neem bark for use. The preparation of activated carbons was carried out using the chemical method with phosphoric acid (H_3PO_4) as the activating agent at different concentrations. In order to evaluate the performance of these activated carbons, various parameters have been determined. Thus, the BET method by nitrogen adsorption was used to determine the specific surface area. The t-plot and BJH methods provided knowledge of the pore volumes of the different carbons. The pore topography and the elemental composition were determined by the SEM–EDS method. The surface functions were determined using the Boehm method. The preparation resulted in the carbons CA 25, CA 30 and CA 60. The specific surface area values obtained ranged from 305.10 to 666.38 m^2/g. Also, the results indicate a large number of pores on the surface of the carbons with volumes evolving with the increase in H_3PO_4 concentration. The results also indicate the acidic nature of the surface of each carbon. All these results show that the carbons produced from neem bark have very good characteristics for use in water treatment.

Keywords Activated carbon · Neem bark · Chemical activation · Characterization · Phosphoric acid

Introduction

Activated carbon (AC) has been used for long as absorbent characterized by, among other things, its high specific surface area, porous structure and thermostability (Chen et al. 2011). It is used in several fields, especially in water treatment (Guillossou 2019; Lengaye et al. 2019). However, commercial activated carbons on the market generally have high implementation costs and are therefore not always accessible on a large scale, especially for developing countries (Abo et al. 2020). It is therefore necessary to research alternative, biodegradable and natural precursors that can be used to produce activated carbons in large quantities and of good quality. Thus, in recent decades, the search for low-cost adsorbent materials with efficiency comparable to that of commercial activated carbons has been an important research topic (Khalfaoui 2012). As a result, much work has been done on the production of activated carbons from solid waste and local carbon-rich biomass (Haykiri-Acma et al. 2006; Madani et al. 2006; Trachi et al. 2014; Mamane et al. 2016; Unugul and Nigiz, 2020). In Côte d'Ivoire, activated carbons with a high adsorption capacity have already been obtained from new sources of production of activated carbons such as waste of vegetable origin. These include Makoré seed husks, Flamboyant fruits (Aboua 2013), coconut husks (Atheba et al. 2014), cocoa husks (Kouadio et al. 2019) and maize cobs (Abo et al. 2020). This work is part of this perspective of valorising the use of local biomass in order to produce activated carbon.

The source explored is neem bark (*Azadirachta indica*). Indeed the neem is a tree which develops in tropical and very hot zones and particularly in Côte d'Ivoire. It is found everywhere in the Côte d'Ivoire, in large quantities and is used in various fields. In addition, this material has proved to be an

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interesting precursor for the production of CA and its use for dye and heavy metal removal (Alau et al. 2010; Maheshwari and Gupta, 2014, 2015). Indeed, these authors have shown the good adsorption capacity of carbons from this precursor both in batch and column during the adsorption of methylene blue and heavy metals (Cu (II), Cr (VI) and Zn (II)). However, important aspects of the characterization such as BET surface area, iodine number, of pore topography and surface functions of neem bark charcoal have not been explored.

This study therefore proposes to produce carbon from this material and to determine its textural, chemical and physical properties. It will thus be a question of assessing the intrinsic capacities of the various carbons obtained in the light of their properties.

Materials and methods

Biological material

The neem of the non-systematic *Azadirachta indica* is a tree of the family Meliaceae native to eastern India (southern Himalayas). It is an angiosperm of the magnoliopsida class (Formed environment 2013). It is cultivated in tropical regions especially in Africa and is abundant in Côte d'Ivoire. Its bark is brown, vertically cracked, hard, heavy and reddish-brown (Montes-Molina 2008). In addition, the bark produces strong coarse fibres which are commonly braided into ropes.

Reagents and solvents

The reagents and solvents used were analytical grade. The commercial solution of phosphoric acid (H_3PO_4) of 85% purity was supplied by the Fluka Company. It was used to impregnate the skins at different concentrations (25, 30 and 60%). Hydrochloric acid (HCl), sodium hydroxide (NaOH), sodium thiosulphate ($Na_2S_2O_3$), sodium carbonate (Na_2CO_3), sodium bicarbonate ($NaHCO_3$) and sodium chloride (NaCl) with a purity of 99% were also supplied by Fluka.

Production of activated carbons

The protocol adopted for the preparation of activated carbons (AC) was inspired by those used by Ounas et al., (2009) and Gueye et al. (2011) who produced activated carbons from plant biomass. After acquiring the biological material, the bark was washed with plenty of tap water to remove impurities such as sand and dust. Then the barks were dried in an oven at 110 °C for 24 h before the production of the activated carbons by the chemical method. The carbons were prepared by impregnating, in reactors, 150 g of neem bark with 150 mL of phosphoric acid (H_3PO_4) at different concentrations (H_3PO_4 25%, H_3PO_4 30%, H_3PO_4 60%) for 24 h, kept under agitation. The matter obtained was dried at 110 °C/24 h and then calcined at 550 °C for 1h30min. Then the resulting carbons were washed, dried, crushed and finally sieved to have a diameter of between 2 and 2.4 mm which was used for the rest of the work.

Characterization of activated carbon

Yield

Yield is an important quantitative characteristic for activated carbons. It reflects the loss of mass during the preparation of the coal. The method used for its determination consisted of differential weighing of a known quantity of the starting material ($m = 20$ g). The expression of the mass yield is given by the relation (1):

$$\text{Yield (\%)} = \frac{m_2}{m_1} \times 100 \tag{1}$$

With m_1: initial mass before carbon preparation; m_2: final mass after carbon preparation.

Ash rate

This is the inorganic, inert, amorphous and unusable part that affects the quality of the coal. The protocol adopted for the determination of ash rate is the one used by Khalfaoui (2012). A mass of 1 g (M_1) of dry activated carbon was placed in the furnace at 650 °C for 3 h.

After drying, the carbon was weighed again (M_2). The calculation of the ash rate is given by the relation (2):

$$\text{Ash rate(\%)} = \frac{M_2}{M_1} \times 100 \tag{2}$$

With M_2: mass of carbon after calcination at 650 °C; M_1: mass of carbon before calcination.

Humidity rate

The humidity rate represents the residual water contained in the coal. It gives an idea of the actual mass of carbon that will be in contact with the effluent. The protocol adopted for the determination of the humidity rate is the one used by Khalfaoui (2012). A mass of 1 g (m_1) of coal was introduced into a porcelain capsule and placed in an oven at 110 °C for 24 h. After drying, the coal was weighed again (m_2). The humidity rate is calculated by the relation (3):

$$\text{Humidity rate (\%)} = \frac{(m_1 - m_2)}{m_1} \times 100 \quad (3)$$

With m_1: initial mass of carbon; m_2: mass of carbon after drying at 110 °C.

Iodine number

The iodine number is one of the methods used to measure the adsorption capacity of carbon. The iodine number is the amount in milligrams of iodine adsorbed per gram of carbon in an aqueous solution with an iodine normality of 0.02 N. The protocol used in this work follows the one proposed by Girgis and El-Hendawy (2002).

A mass of 0.5 g of charcoal previously oven-dried at 110 °C was placed in a beaker to which 15 mL of iodine solution of 0.1 N was added. The mixture was stirred for 4 min and then filtered through filter paper.

Two drops of the starch solution was added to 10 mL of the filtrate, which was then dosed with a volume of sodium thiosulphate of 0.1 N. The iodine number expressed in mg/g is given by the relation (4):

$$\text{Iodinenumber}\left(\frac{m_g}{g}\right) = \left(\frac{(C_0 - \frac{C_n \times V_n}{2V_{I_2}}) \times M_{I_2} \times V_{\text{abs}}}{m}\right) \quad (4)$$

With C_n: Concentration of sodium thiosulphate (mol / L); V_n: Volume of sodium thiosulphate; VI_2: Volume of iodine measured: V_{abs}: Adsorption Volume; MI_2: Molecular weight of iodine (254 g/mol): m: Mass of activated carbon (g).

Specific surface area and pore volume: BET and t-plot methods

The BET method is based on the adsorption of nitrogen on activated carbon. According to the BET method a degassing was carried out under vacuum at a temperature of 120 °C for 24 h. The adsorption isotherm allows access to the specific surface area which was calculated according to the BET method in its linear form indicated by the relation 5:

$$\frac{P/P_0}{Q(1 - P/P_0} = \frac{1}{Q_m C} + \frac{C-1}{Q_m C} \cdot P/P_0 \quad (5)$$

With Q: amount adsorbed at pressure P; Qm: amount of gas necessary to cover 1 g of adsorbent with a single layer of gas; C: BET constant.

The plot of $\left(\frac{(P/Po)}{Q(1-P/Po)}\right)$ as a function of P/P_o is a straight line with slope a $= \frac{C-1}{Q_m C}$ and intercept b $= \frac{1}{Q_m C}$ which allows the constants $Q_m = \frac{1}{a+b}$ and $C = 1 + \frac{a}{b}$. to be determined. The S_{BET} specific surface area is calculated from the following equation:

$$S_{\text{BET}} = \frac{N \times A_i \times V_m 10^{-20}}{m \times V_M}$$

With N: Avogadro number; A_i: area occupied by an adsorbate molecule (0.162 Å^2 for N^2); V_m: volume of vapour necessary to completely cover the surface of the solid with a monomolecular layer of adsorbate (cm^3/g of adsorbent); m: mass of the solid analysed; V_M: molar volume (22,414 cm^3/mol).

The t-plot method is the most commonly used method for analysing microporous carbon isotherms. This method, consisted in bringing the volume of adsorbed gas per gram of solid (in cm^3/g) to the relative pressure P/Po as a function of the statistical thickness t (in Å) of the layer adsorbed on the non-porous reference solid at this same relative pressure. The t-plot method is calculated by the formula proposed by Lippens and Boer (1965):

$$t = \frac{eP_0}{P_0 - P}$$

Surface functions: Boehm method

The measurements are carried out according to the Boehm method (1966), which allows us to quantify its various functions and gives us information on the chemical composition of the surface of the carbon. It consisted of mixing 0.5 g of activated carbon with 50 mL of 0.1 M aqueous solutions NaOH, Na_2CO_3, $NaHCO_3$ and HCl.

These solutions were stirred for 24 h and then filtered. The dosage of 10 mL of the filtrate was carried out either with hydrochloric acid (HCl) or with and 0.1 M soda (NaOH). Subsequently, the determination of the functional groups was carried out by the following calculations:

GI = [$NaHCO_3$]; GII = [Na_2CO_3]—[$NaHCO_3$] and GIII = [NaOH]—[Na_2CO_3].

With GI = strong carboxylic; GII = weak carboxylic and lactonic; GIII = phenolic.

pH zero point charge (pHzpc)

The protocol adopted was inspired by those used by Ramon et al. (1999) and Nebaghe (2016). Solutions with 0.1 M NaCl and a pH between 2 and 10 were first prepared. Then 0.1 g of dry activated carbon was put together with 20 mL of different solutions. The mixture was stirred for 48 h and afterwards new pH readings were taken. The curve representing final pH = f (initial pH) was plotted. The pH_{ZpC} then corresponds to the pH of the solution for which the curve crosses the first bisector (final pH = initial pH).

Topography and elemental composition: SEM–EDS method

Topography and elemental composition were determined by the SEM–EDS method. Before the samples were introduced into the SEM, they were pre-treated. The samples were finely ground in a porcelain mortar and sieved on a 250 µ. Next, 10 mg of the sieving residue was spread on a pad primed with double-sided adhesive carbon which was then fixed on a tray. Finally, the sample tray was mounted on the plate of the SEM chamber for microanalysis.

Results and discussion

Activated carbons obtained

The three activated carbons (ACs) obtained after preparation are named CA 25, CA 30 and CA 60. After production, these ACs were crushed, sieved and conditioned for the various characterization tests.

Yield, humidity rate, ash rate and iodine number of prepared ACs

Table 1 presents the results of various characterization tests on prepared carbons.

The table shows the rates of yield of 46.02%, 45.23% and 42.48%, respectively, for CA 25, CA 30 and CA 60 carbons.

There is a small difference between the values. However, a downward trend in yield is observed as the concentration of the activating chemical increases. The results of the ash rate of the various carbons indicate a rate of 2% for CA 60 carbon and rates of 11 and 14% for CA 30 and CA 25 carbon. As regards the iodine number, the values given in the table are 671.41, 689.19 and 693.08 mg/g for CA 25, CA 30 and CA 60 carbons, respectively. The results for these three parameters show that the increase in phosphoric acid induces a decrease in yield and ash rate and an increase in iodine number.

These results could be explained by a significant removal of impurities within the pores with the increase in acid concentration. Also there is a huge loss of volatile matter as the acid concentration increases. Some researchers have obtained similar results by activation with phosphoric acid (Tan et al. 2008; Gharib and Ouedemi 2005; Sido-Pabyam et al. 2009). With regard to the humidity rate, there is almost no variation, with rates of 13 and 14% for CA 25, CA 30 and CA 60 carbons as shown in the table. These values are very close to those of Gueye et al. (2011), who obtained a value of 12.87% for carbons prepared from jatropha shell.

Table 1 Results of the various characterization tests for CA 25, CA 30 and CA 60 carbons

Activated carbon	Yield (%)	Ash rate (%)	Humidity rate (%)	Iodine number (mg/g)
CA 60	42.48	2	14	693.08
CA 30	45.23	11	13	689.19
CA 25	46.02	14	13	671.41

BET surface area and porosity

Figure 1 shows the results of the nitrogen (N_2) adsorption isotherms for the three activated carbons.

The three adsorption isotherms curves in Fig. 1 have the same shape and are according to the IUPAC Type I classification (Weber and Smith 1986). Figure 2 shows the plot of the transform of the BET equation, the lines obtained allow the determination of Q_m (amount of gas necessary to cover 1 g of adsorbent with a single layer of gas) and C (BET constant). Their knowledge makes it possible to determine the specific surface area.

The various results are recorded in Table 2. It summarizes parameters such as micropore and mesopore area, total surface area and pore volume.

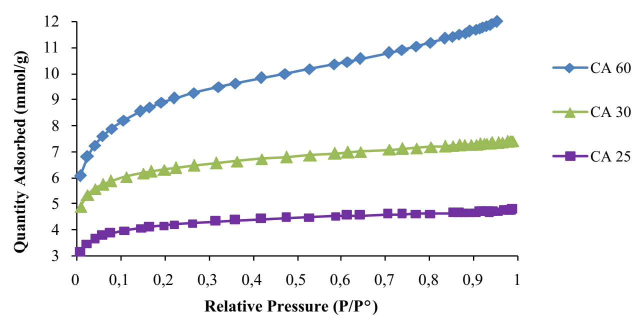

Fig. 1 Nitrogen adsorption isotherms curves for CA 60, CA 30 and CA 25

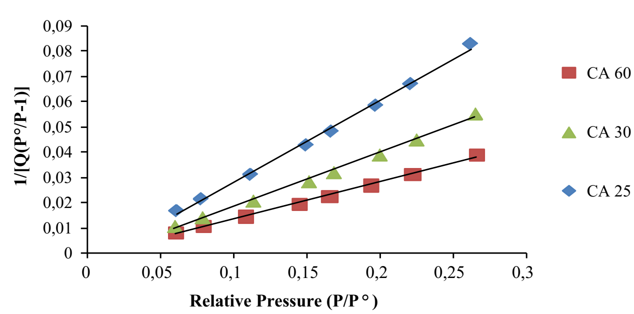

Fig. 2 Plot of the transform of the BET equation for CA 60, CA 30 and CA 25

Table 2 Specific surface area and pore volume results

Identified parameters	Methods	CA 60	CA 30	CA 25
Total surface area S_{BET} (m^2/g)	BET	666.38	460.65	305.10
Microporous surface area (m^2/g)	t-Plot	293.80	315.67	206.12
External surface area Sext (m^2/g)	t-Plot	372.57	144.98	98.97
Mesoporous surface area (m^2/g)	Adsorption BJH	208.31	108.40	71.34
Total pore volume (cm^3/g)	Single point adsorption	0.42	0.26	0.16
Microporous volume (cm^3/g)	t-Plot	0.15	0.16	0.10
Mesoporous volume (cm^3/g)	Adsorption BJH	0.20	0.08	0.05
	Desorption BJH	0.23	0.05	0.04

The specific surface area values for CA 60, CA 30 and CA 25 carbons shown in Table 2 are 666.38, 460.65 and 305.10 m^2/g, respectively. A significant increase in surface area and pore volume can be observed as the concentration of the activating agent increases. Indeed, the specific surface area of activated carbons increases from 305.10 to 666.38 m^2/g, i.e. an increase of 25.23% when the concentration of phosphoric acid (H_3PO_4) increases from 25 to 60%.

The same applies to the external surface area, which goes from 98.97 to 372.57 m^2/g, i.e. an increase of 44.38% for H_3PO_4 concentrations ranging from 25 to 60%. From these values, it can be seen that the S_{BET} area and the external surface area increase with H_3PO_4 concentration. Also, the total pore volume increases from 0.16 to 0.42 cm^3/g, an increase of 39.95% for concentrations ranging from 25 to 60%. These results can be explained by the fact that as the acid concentration increases, the surface pores are cleaned of impurities. This situation induces a greater release of the space occupied by mesopores and macropores and therefore a larger external surface area, thus confirming the results obtained for the iodine number. Furthermore, for CA 60 carbons, the microporous volume is smaller than the mesoporous volume, unlike CA 30 and CA 25 carbons. This could be explained by the fact that as, the quantities of H_3PO_4 increase, the pores on the outer surface of the carbon develop. This situation leads to an important development of the mesopores so that the total volume and the mesoporous volume increase strongly.

Many authors who have worked on activated carbon prepared from lignocellulosic materials have shown that the amount of phosphoric acid plays a role in the development of porosity (Suárez et al. 2001; Molina and Rodriguez 2004). For Jagtoyen and Derbyshire (1998) phosphoric acid undergoes a reaction within the internal structure of cellulose to produce a depolymerisation leading to an increase in pore volume and thus an expansion of the overall volume. In addition, Molina and Rodriguez (2004) have shown that when the amount of H_3PO_4 acid used for activation increases, the volume to which it has access and the content of various associated polyphosphates are also increased. Thus, this increase leads to a large pore volume with enlarged pore size, which therefore favours the formation of mesopores. These results are also comparable to those obtained by Reffas (2010) for carbons obtained from coffee grounds for different concentrations of H_3PO_4.

Surface functions and pH zero point charge (pHzpc)

Table 3 gives the values of the different surface functions of the three carbons (CA 60, CA 30 and CA 25) and the corresponding pH zero point charge.

An average total acidity of 8 meq/g is noted compared to an average total basicity of 2.72 meq/g for all coals. The activated carbons obtained therefore have a strong acidic character in general due mainly to the carboxylic, phenolic and lactonic groups present. The carboxyl group represents approximately 53% of the surface functions for CA 60 and CA 30. On the other hand, for CA 25 carbon, the carboxylic and phenolic groups each represent 49.50% and 50.49% of the surface functions.

The presence of both acidic and basic groups gives these activated carbons a better efficiency with regard to the chemical nature of the pollutant to be treated.

Table 3 Results of the surface functions (Boehm titration) and pHzpc

Activated carbons	Carboxylic function (meq/g)	Phenolic function (meq/g)	Lactonic function (meq/g)	Basic Total (meq/g)	Total acid (meq/g)	Character on the surface	pHzpc
CA 60	4.35	2.02	1.78	3.57	8.15	Acid	4.60
CA 30	4.28	2.40	1.58	3.38	8.26		4.60
CA 25	3.47	3.63	1.14	1.21	7.01		5.00

The pHzpc value given in Table 3 is 4.6 for CA 30 and CA 60, and 5 for CA 25 carbon. These acidic pHzpc values thus corroborate the results of the surface functions.

Topography pore and elemental composition: SEM–EDS method

The SEM–EDS method was used to describe the morphology, microstructures and elemental composition for CA 60 and CA 30 carbons with the best BET surfaces. Figures 3 and 4 illustrate the results obtained.

Figure 3 shows that there is an influx of pores on the surfaces of CA 60 and CA 30 carbons. It is observed that the number of pores increases with the concentration of the activating agent.

Figure 4 shows the spectrum results at the surface composition level.

The overall composition reveals a high amount of carbon and slightly less oxygen for both carbons.

The presence of these two atoms confirms the organic nature of the activated carbons used in this study. However, it is noted that the oxygen spectrum for the CA 30 carbon is slightly higher than that of the CA 60 carbon. The EDS also detected the presence of heteroatoms such as calcium, sulphur and phosphorus in identical proportions in both coals. The presence of phosphorus may be due to the phosphorus atoms bound to the surface of the oxygen. Note the presence of platinum in both carbons and the presence of aluminium only in the CA 30. On the other hand, other elements such as potassium and magnesium of plant origin are only found at the CA 30 level. The very low presence of these elements is explained by the fact that H_3PO_4 did not allow their total elimination.

Conclusion

The objective of this study was to prepare and determine the characteristics of activated carbons produced from neem bark. The production of activated carbons with different concentrations of phosphoric acid made it possible to obtain three types of activated carbons, namely CA 25, CA 30 and AC 60. The characterization of the different carbons shows a greater decomposition of the bark as the concentration of the activating agent increases. Also, the results of the ash rate thus demonstrate the good quality of the various carbons

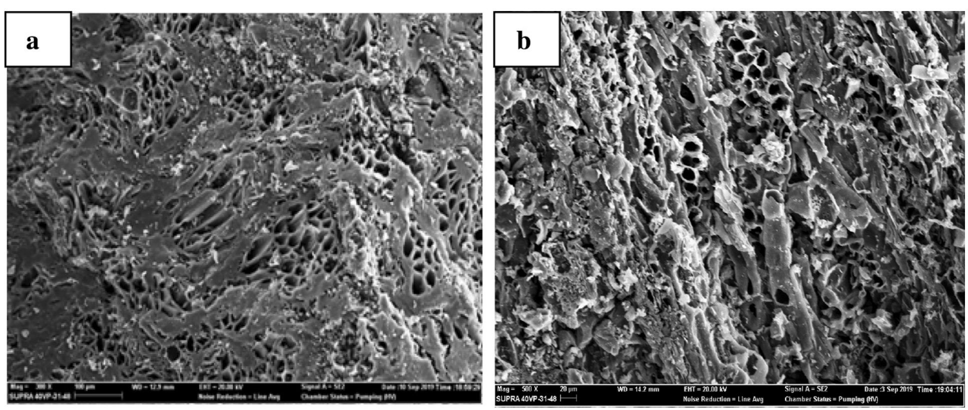

Fig. 3 Overview of the external morphology of CA 60 (**a**) and CA 30 (**b**) carbons

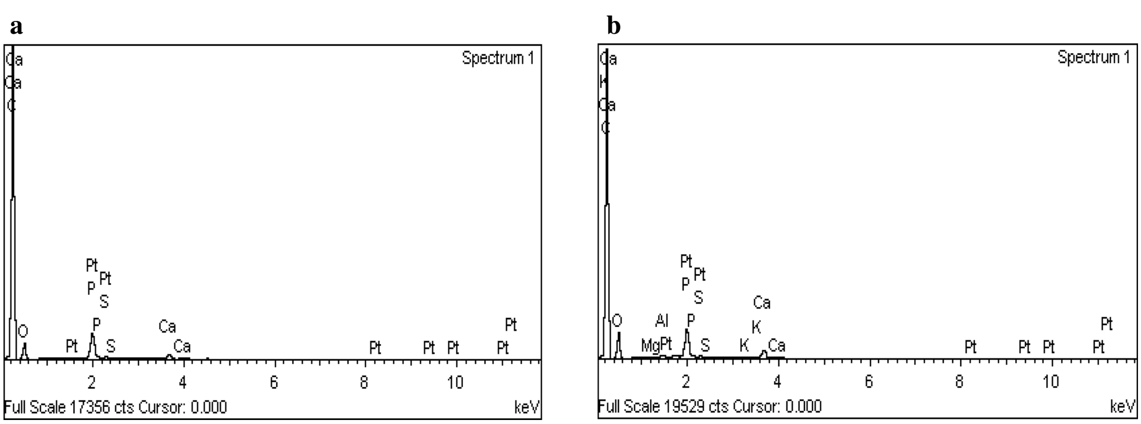

Fig. 4 EDS spectrum of activated carbons (a- AC 60 and AC 30)

obtained, in particular the CA 60, whose rate is 2%. The results also made it possible to obtain a microporosity of the carbons due to the high iodine number values. The specific surface area measured using the BET method indicates the evolution of the specific surface area as the concentration of phosphoric acid increases, with values ranging from 305.10 to 666.38 m^2/g. The study of acid–base titration has shown that the surface of the carbons has an acidic characteristic with a strong presence of carboxylic and phenolic groups. The study of the topography showed that in addition to the presence of carbon and oxygen, other elements were detected such as calcium and sulphur, which are heteroatoms. Finally, activated carbons produced from neem bark have interesting characteristics and can therefore be used for various purposes, particularly in the field of water decontamination.

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Declaration

Conflict of interest The authors declare that they have no conflict of interest.

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