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Preparation and Characterization of Activated Carbons from Palm Nut Shells: Effects of Calcination Temperature on Porosity and Chemical Properties

Charly Mve Mfoumou[1,2*], Berthy Lionel Mbouiti[1,3], Spenseur Bouassa Mougnala[1,3], Pradel Tonda-Mikiela[1,2], Guy Raymond Feuya Tchouya[1,2]

[1]Laboratoire de Chimie des Milieux et des Matériaux Inorganiques (LC2MI), URCHI, Université des Sciences et Techniques de Masuku (USTM), Franceville, Gabon
[2]Département de Chimie, Faculté des Sciences, Université des Sciences et Techniques de Masuku (USTM), Franceville, Gabon
[3]Ecole Doctorale des Sciences Fondamentales et appliquées (ED-SFA), Laboratoire de Chimie des Milieux et des Matériaux Inorganiques (LC2MI), URCHI, USTM, Franceville, Gabon
Email: *charly.mvemfoumou@univ-masuku.org, *mvefoumou@gmail.com

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Abstract

Activated carbons (ACs) calcined at 400˚C, 500˚C, and 600˚C (AC-400, AC-500, and AC-600) were prepared using palm nut shells from Gabon as raw material and zinc chloride ($ZnCl_2$) as a chemical activating agent. Prepared ACs were characterized by physisorption of nitrogen (N_2), determination of diode and methylene blue numbers for studies of porosity and by quantification and determination of surface functional groups and pH at point of zero charge (pH_{pzc}) respectively, for studies of chemical properties of prepared ACs. Then, effects of calcination temperature (T_{cal}) on porosity and chemical properties of prepared ACs were studied. The results obtained showed that when the calcination temperature increases from 500˚C to 600˚C, the porosity and chemical properties of prepared ACs are modified. Indeed, the methylene blue and iodine numbers determined for activated carbons AC-400 (460 and 7.94 $mg{\cdot}g^{-1}$, respectively) and AC-500 (680 and 8.90 $mg{\cdot}g^{-1}$, respectively) are higher than those obtained for AC-600 (360 and 5.75 $mg{\cdot}g^{-1}$, respectively). Compared to the AC-500 adsorbent, specific surface areas (S_{BET}) and microporous volume losses for AC-600 were estimated to 44.7% and 45.8%, respectively. Moreover, in our experimental conditions, the effect of T_{cal} on the quantities of acidic and basic functional groups on the surface of the ACs appears negligible. In addition, results of the pH_{pzc} of prepared ACs showed that as T_{cal} increases, the pH of the adsorbents increases and tends towards

neutrality. Indeed, a stronger acidity was determined on AC-400 (pH_{pzc} = 5.60) compared to those on AC-500 and AC-600 (pH_{pzc} = 6.85 and 6.70, respectively). Also according to the results of porosity and chemical characterizations, adsorption being a surface phenomenon, 500˚C appears to be the optimal calcination temperature for the preparation of activated carbons from palm nut shells in our experimental conditions.

Keywords

Palm Nut Shells, Activated Carbon, Calcination Temperature, Porosity and Chemical Properties

1. Introduction

Natural or synthetic zeolites, modified clays and activated carbons (ACs) are among the porous materials used in scientific research as adsorbents in adsorption processes for air, gas and water treatments and in the removal of organic and mineral pollutants from aqueous media [1]-[9]. Among these porous materials, activated carbons are the most widely used in adsorption processes in aqueous media because of their textural, structural and chemical properties [9].

Concerning textural properties, in the structure of ACs we can find macropores with opening diameters > 50 nanometers (nm), mesopores with diameters between 2 - 50 nm, and micropores with pore diameters < 2 nm [10] [11]. This porous structure allows ACs to trap chemical molecules or species of different sizes [12]. The chemical properties of ACs are attributed mostly to acid (phenolic, carbonyl, carboxylic, etc.) and basic (pyrone, chromene, etc.) functional groups located on the surface of solids [13] [14]. These acidic and basic functions groups promote interactions between many ionic species and the surfaces of activated carbons [13]. Thus, these textural and chemical properties allow activated carbons to adsorb (trap) or remove ionic species or organic and inorganic molecules [9]-[13]. It is therefore important, when preparing activated carbons, to obtain ACs that have well structural (or physical) and chemical properties depending on the intended application.

In fact, several studies have been carried out on the influence of pyrolysis (calcination) temperature on structural and chemical properties, under a variety of experimental conditions [15]-[19]. For example, the work of Kouassi Brou *et al.* [15] on the physico-chemical characterization of ACs obtained by pyrolysis at 400 and 600˚C, showed that specific surface areas (S_{BET}) and microporous volumes increased when increasing the pyrolysis temperature. They also observed an increase in the pH of prepared ACs and obtained mainly materials with a microporous structure. The results obtained by Bouchemal *et al.* [16] on the effect of calcination temperature on ACs impregnated with $ZnCl_2$ and calcined between 500 ˚C and 600˚C under an inert atmosphere (N_2), showed also an increase in both the S_{BET} and microporous volumes respectively when the temperature

increased from 500˚C to 600˚C.

On the other hand, activated carbons are prepared from carbon-rich raw materials. Wood, coconut shells, and other carbon-rich plant-based materials are among the sources of raw materials used [6] [10] [20]. Waste from the food industry, such as palm nut shells, can be recovered by being used as source of raw material in the preparation of activated carbons [21]. In Gabon, in order to diversify the economy, the company Olam Palm Gabon has been created for the development of the palm oil sector. Today, Olam Palm Gabon operates on 300,000 hectares of forests and more than 4500 oil palm plantations [22]. Thus, in the production of palm oil, significant wastes from palm nut shells are produced and are responsible for significant environmental pollution, particularly visual pollution. As a result, the recovery of these wastes is becoming very necessary.

In literature, very few studies have featured on the effects of calcination temperature (pyrolysis) on structural and chemical properties of ACs prepared from industrial wastes, in order to determine the optimal experimental conditions for obtaining ACs, when calcined in an air atmosphere, for being used as molecular sieves in the removal of organic or inorganic pollutants in aqueous media.

Indeed, this work aimed to evaluate the influence of calcination temperature on porosity and chemical properties of activated carbons prepared from palm nut shells of Gabon as raw material and zinc chloride ($ZnCl_2$) as activating agent. This will enable to determine, in our experimental conditions, the optimal pyrolysis temperature for the obtention of activated carbons with best structural and chemical properties.

2. Experimental

2.1. Preparation of Activated Carbons

Activated carbons (ACs) were prepared using palm nut shells collected in the city of Franceville, in the Haut-Ogooué region of Gabon. The entire preparation procedure of the ACs was reported in a recent study [8]. However after impregnation at a $ZnCl_2$ solution to 1 M (1:1 ratio ($ZnCl_2$: Acros Organics, purity > 98.5%) and drying, the solids were activated at 400˚C, 500˚C, and 600˚C for 1 h and 30 minutes (min) with a heating rate of $5^{\circ}C \cdot min^{-1}$. The activated carbons obtained were cooled, rinsed with 0.1 M HCl (HCl: Emsure, purity: 33%) and washed with distilled water until the pH of the residual water being equal to 6.5. Then dried in the oven for 48 h at 100˚C. The activated carbons obtained at 400˚C, 500˚C, and 600˚C were indexed AC-400, AC-500, and AC-600, respectively (**Figure 1**).

2.2. Characterizations of Porosity and Chemical Properties

2.2.1. Porosities of ACs

1) N_2 physisorption

Data on the surface areas and pore volumes were carried out using a Micromeritics TRISTAR 3000 instrument following method described in the work of Belin *et al.* [2]. About 100 mg of activated carbon sample was pretreated during

1 h at 90˚C, and 10 h at 350˚C successively. Physisorption isotherms of nitrogen (N_2) were carried out at −196˚C. The specific surface areas of samples were evaluated by means of the Brunauer, Emmett and Teller (BET) theory [23]. Microporous and mesoporous volumes were determined by t-plot method of De Boer and Dubinin-Radushkevich equations respectively [24]-[26].

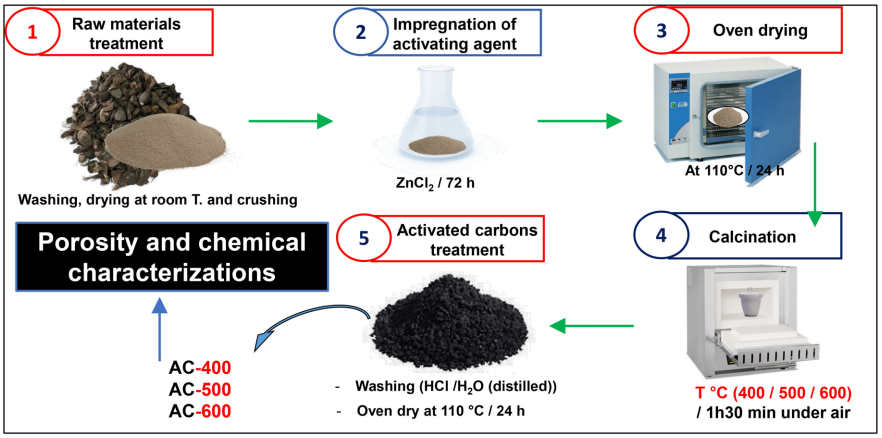

Figure 1. Process of preparation of activated carbons from palm nut shells.

2) Iodine number

The iodine number test was performed on prepared activated carbons based on the ASTM D4607-94 method [27]. 10 mL of hydrochloric acid (1 N) was mixed with different masses of activated carbon (0.1, 0.3, and 0.5 g), stirred and boiled for 30 seconds. Once the mixture cooled, 50 mL of an iodine solution (0.02 N (Fisher Scientific)) was added. The mixture was then stirred vigorously for 30 seconds and then vacuum-filtered. The filtrate was then titrated with a sodium thiosulfate solution, $Na_2S_2O_3$ (0.1 N ($Na_2S_2O_3$: Labosi, purity > 99%)), until stained. 2 mL starch (1 g·L^{-1}) were then added to the titrated filtrate and then the titration was continued until the solution was completely discolored.

The residual concentration of iodine is given by the following relationship (2):

$$C_r = \frac{C \cdot V_{eq}}{V} \tag{1}$$

With, C_r: residual concentration of I_2 (N); C: concentration of $Na_2S_2O_3$ (N); V_{eq}: volume of $Na_2S_2O_3$ (mL) and V: volume of titrated filtrate (mL).

The residual concentration of I_2 should be between 16×10^{-4} N and 8×10^{-3} N. Thus, the amount of iodine adsorbed is given by the following formula (2):

$$Q_{I_2} = \frac{\left[A - \left(DF \cdot B \cdot V_{eq}\right)\right]}{m_{CA}} \tag{2}$$

where, A is the concentration of I_2 (mol·L^{-1}) × Molar mass of iodine (g·mol^{-1}); B is the concentration of $Na_2S_2O_3$ (mol·L^{-1}) × Molar Mass of Iodine (g·mol^{-1}), DF is the dilution factor ($\frac{\left(V_{I_2} + V_{HCl}\right)}{V_{\text{Filtrate}}}$) and m_{AC} is activated carbon mass (g).

By plotting the curve $Q_{I_2} = f(C_r)$, the iodine number is then the value of Q_{I_2} ($mg \cdot g^{-1}$) corresponding to a residual iodine concentration of 0.04 N.

3) Methylene blue number

The method used was the same as that proposed by the European Centre of Chemical Industry Federations (ECCIF) [28]. 25 mL of methylene blue (M.B) solution (120 $mg \cdot L^{-1}$ (M.B: Alp'Osmose, purity > 98.2%)) were put in contact with 0.1 g of activated carbon and then stirred (200 rpm) for 20 minutes. The mixture was then filtered on Büchner and the residual concentration of methylene blue was determined by colorimetry using a Thermo Scientific Evolution 60S spectrophotometer, using a previously established methylene blue calibration curve, at a wavelength λ = 664 nm.

The methylene blue number is given by the following relationship (3):

$$Q_{BM} = \frac{\left(C_0 - C_f\right)V}{m_{AC}} \tag{3}$$

where, Q_{BM} is the methylene blue number ($mg \cdot g^{-1}$); C_0 is the initial concentration of methylene blue ($mg \cdot L^{-1}$); C_f is the residual concentration of methylene blue ($mg \cdot L^{-1}$); m_{AC} is the activated carbon mass (g) and V is the volume of the methylene blue solution in (L).

2.2.2. Chemical Properties of ACs

1) pH at point of zero charge (pH_{pzc})

The pH at point of zero charge (pH_{pzc}) of activated carbons were obtained based on the acid-base titration method described by Amola [29] and reproduced in in a recent study [8]. The pH_{pzc} of activated carbons are given by the point of intersection between the experimental curves and the theoretical curves of $pH_{final} = f(pH_{initial})$.

2) Surface Functional Groups

The quantification of surface functional groups of activated carbons was performed using the Boehm method [30], with little modifications applied in a recent study [8]. The number of equivalents ($m_{eq} \cdot g^{-1}$) or concentrations ($mmol \cdot g^{-1}$) of acid or basic surface functional groups were calculated using the following Equation (4):

$$n\left(\text{mmol} \cdot \text{g}^{-1}\right) = \frac{C \times \left(V_b - V_s\right) \times 1000}{m} \tag{4}$$

where C is the concentration of NaOH (Emsure, purity > 99%) or HCl ($mol \cdot L^{-1}$); V_b and V_s are the equivalent volumes of the blank and sample (L) respectively; m is the mass of activated carbon (g) and 1000 is the conversion factor (mmol).

3. Results and Discussion

The study of the influence of calcination temperature (T_{cal}) was carried out after characterizations of porosity and chemical properties of prepared activated carbons (AC-400, AC-500, and AC-600).

3.1. Effect of T_{cal} on Porosity

The study of the effect of calcination temperature on porosity was conducted based only on data from the methylene blue and iodine numbers and those obtained from the N_2 physisorption study of prepared ACs.

Iodine number is an important parameter in the characterization of activated carbons [23]. Indeed, it provides information on the ability of activated carbons to trap or adsorb small molecules and therefore provides information on the microporosity of the material [31] [32]. **Figure 2** shows the iodine number values obtained on activated carbons calcined to 400˚C, 500˚C, and 600˚C (AC-400, AC-500, and AC-600 respectively). Regardless of the calcination temperature applied, the prepared ACs developed micropores in their structures. The iodine number obtained vary between 360 - 680 $mg{\cdot}g^{-1}$. In contrast to the work of Huang *et al.* [31], the results in **Figure 2** indicate an increase in the iodine number as the calcination temperature (T_{cal}) increases from 400˚C to 500˚C and a decrease as T_{cal} increases from 500 ˚C to 600˚C. Indeed, the values of the iodine number obtained on AC-400 and AC-500 (460 and 680 $mg{\cdot}g^{-1}$ respectively) are higher than those obtained on AC-600 (360 $mg{\cdot}g^{-1}$).

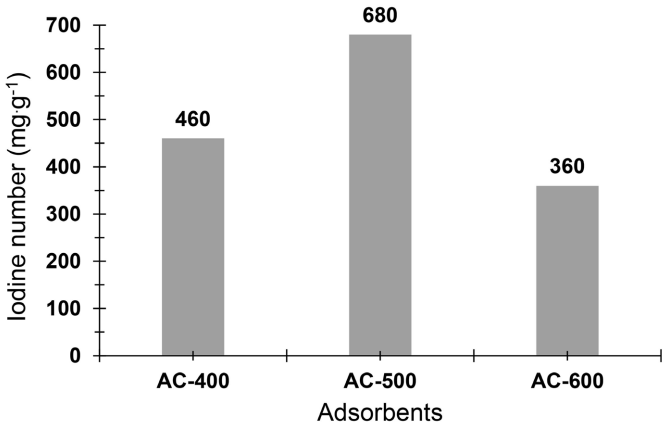

Figure 2. Iodine number obtained on prepared activated carbons AC-400, AC-500, and AC-600.

In our experimental conditions, it seems that at 500˚C (AC-500) we develop more adsorption sites favorable to interactions with iodine and/or microporosity likely to trap/adsorb small molecules, compared to AC-400. In addition, when T_{cal} increases from 500˚C to 600˚C, a loss of adsorption sites or microporous surface is observed. This loss of microporous surface could be related to collapse of the microporous structure at 600˚C or clogging of the microporous due to ash formation during calcination at this temperature [31].

Figure 3 shows the methylene blue number values obtained on adsorbents studied. Prepared ACs appear to have developed mesopores within their structures but very negligible compared to micropores (**Figure 2**). Indeed, the values of the methylene blue number range between 5.72 and 8.90 $mg{\cdot}g^{-1}$. As in the case of the iodine number, the methylene blue number also increases as the calcination temperature

(T_{cal}) increases from 400˚C to 500˚C and decreases from 500˚C to 600˚C (Figure 3). There is also a loss of adsorption sites or mesoporous surfaces, on AC-600, favorable to the trapping of big molecules, such as methylene blue, when T_{cal} = 600˚C.

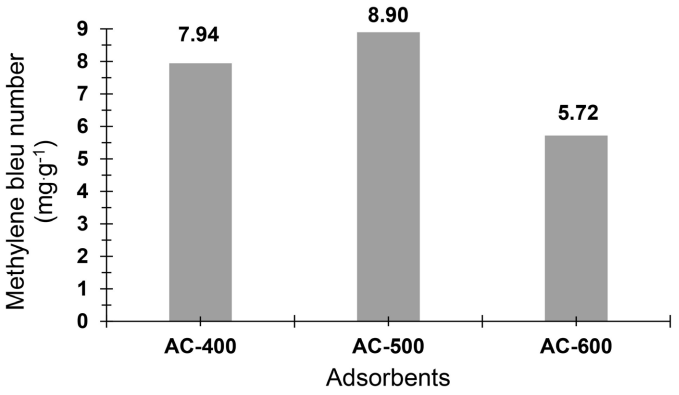

Figure 3. Methylene blue number obtained on prepared activated carbons AC-400, AC-500, and AC-600.

In our experimental conditions it appears that, regardless of T_{cal}, the prepared ACs seem to develop mainly a microporous structure. This parameter appears to be related to the activation agent ($ZnCl_2$) used in the ACs activation process. Although little is known about the mechanism of this process, it is known in the literature that $ZnCl_2$ used as an activating agent promotes pore creation during the calcination process in the preparation of ACs [33]-[35].

The results obtained are in agreement with the work of Molina-Sabio *et al.* [36], who used $ZnCl_2$ as an activating agent in the preparation of activated carbons. Similarly, they observe a loss of porosities when the carbonization temperature is above 500˚C.

Figure 4 shows the N_2 physisorption isotherms obtained on the prepared ACs. The isotherms obtained are type I, characteristic of microporous materials [37], regardless of T_{cal}.

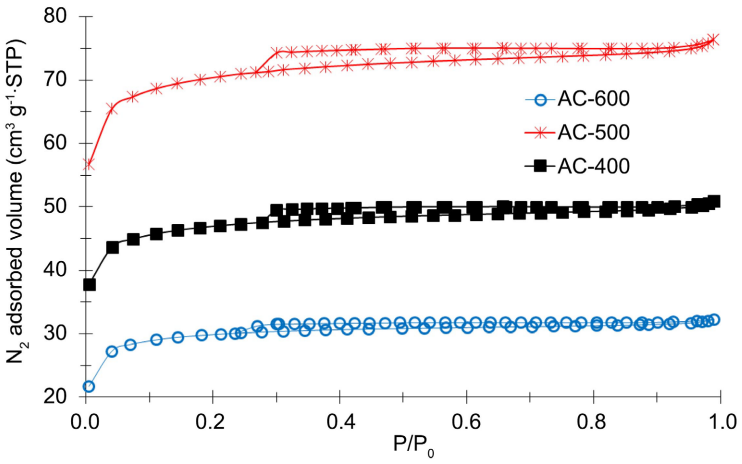

Figure 4. Isotherms of N2 physisorption obtained on prepared activated carbons AC-400, AC-500, and AC-600.

Table 1 summarizes the specific surface areas (S_{BET}), average diameters and pore volumes obtained after exploitation of isotherms of N_2 physisorption of studied adsorbents.

Table 1. Structural properties obtained by N2 physisorption on prepared activated carbons AC-400, AC-500 and AC-600.

Adsorbents	S_{BET} ($m^2 \cdot g^{-1}$)	Average Pore size (nm)	V_{micro} ($cm^3 \cdot g^{-1}$)	V_{meso} ($cm^3 \cdot g^{-1}$)	$V_{total\ pore}$ ($cm^3 \cdot g^{-1}$)
AC-400	182.4	1.70	0.071	0.003	0.077
AC-500	210.3	1.72	0.085	0.004	0.092
AC-600	116.2	1.68	0.046	0.002	0.049

S_{BET}: specific surface BET. V micro: microporous volume. V_{meso}: mesoporous volume.

According to results, a decrease in S_{BET} and porous volumes is observed when the T_{cal} increases from 500˚C to 600˚C and an increase from 400˚C to 500˚C. Indeed, the S_{BET} and microporous volume obtained on AC-600 (116.2 $m^2 \cdot g^{-1}$ and 0.046 $cm^3 \cdot g^{-1}$ respectively) are lower than those obtained on AC-400 (182.4 $m^2 \cdot g^{-1}$ and 0.077 $cm^3 \cdot g^{-1}$ respectively) and AC-500 (210.3 $m^2 \cdot g^{-1}$ and 0.085 $cm^3 \cdot g^{-1}$ respectively). The results of the N_2 physisorption study are in agreement with those obtained in the quantification of the methylene blue and iodine number. Indeed, prepared ACs have predominantly microporous structures with negligible mesoporosity. On the other hand, the average pore sizes obtained on the adsorbents studied are comparable (Table 1). Therefore, there is no effect of calcination temperature on pore size in prepared ACs.

Based on the study of the porosities of prepared ACs, adsorption being a surface phenomenon, AC-500 adsorbent has the best surface and pore volume properties (Table 1) for use in solid adsorption experiments. It appears, in our experimental conditions, that 500˚C is the maximum calcination temperature to be applied in the preparation of ACs. Above 500˚C, a loss of surface area and micropores volume would be possible.

3.2. Effect of T_{cal} on Chemical Properties

In order to evaluate the effect of calcination temperature on the chemical properties of prepared ACs, acidic and basic surface functional groups have been quantified and pH at point of zero charge (pH_{pzc}) determined.

The pH_{pzc} is used to determine the acid-base character of prepared ACs and to evaluate the overall charge of the material according to the pH of the media [38] [39]. The surfaces of the activated carbons will be charged + or – when the pH of the treated media is < and/or > at pH_{pzc} [18] [29]. **Figure 5** shows the pH_{pzc} values determined on prepared activated carbons.

The pH_{pzc} values obtained vary between 5.60 - 6.85. The pH_{pzc} increases as T_{cal} increases and tends towards neutral pH values. Indeed, the pH_{pzc} obtained on AC-500 and AC-600 (6.85 and 6.70 respectively) are close to the pH value = 7 and

higher than the pH_{pzc} of the AC-400 adsorbent (5.60). Based on these results and in our experimental conditions, the pH_{pzc} of prepared ACs tends towards neutrality as the calcination temperature increases. However, it appears that regardless of T_{cal}, prepared ACs retain an acidic character, in particular the adsorbent AC-400 (Figure 5).

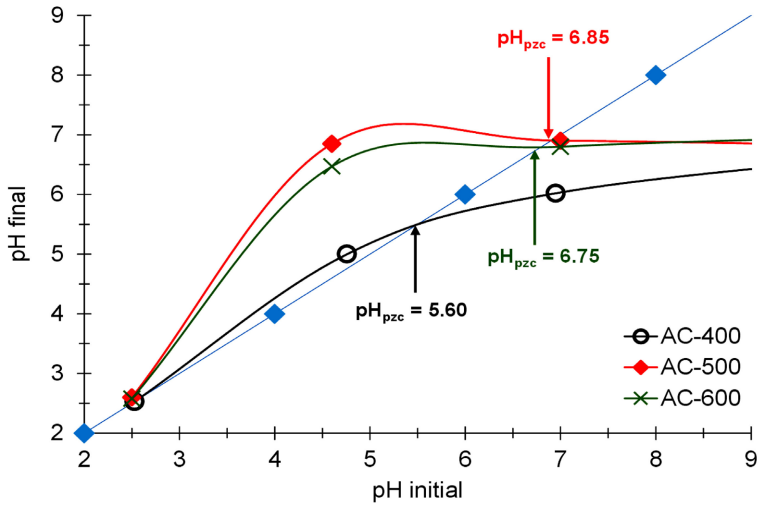

Figure 5. Values of pH_{pzc} determined on prepared activated carbons AC-400, AC-500 and AC-600 using acid-base titration method [29].

Figure 6 shows the concentrations of the acidic (carboxylic, phenolic, etc.) and basic (pyrones, chromenes, etc.) surface functional groups quantified on AC-400, AC-500, and AC-600. Acidic functional groups are predominant on the surface of prepared ACs. Indeed, their concentrations vary between 2.80 - 3.58 $mmol \cdot g^{-1}$ and those of the basic functional groups between 0.21 - 0.83 $mmol \cdot g^{-1}$. These results are in agreement with those of pH_{pzc} (Figure 5). Prepared adsorbents are mainly acidic in character. In the work of Zue Mve *et al.* [40], by preparing of activated carbons with $ZnCl_2$ as an activating agent, they also obtained the majority of groups of acidic functions on the surface of the solids. Therefore, it appears that $ZnCl_2$ promotes the activation of acidic functional groups on the surfaces of prepared ACs. The works of Thouraya *et al.* [41] has also revealed an increase in acidic surface functional groups when the activating agent is acidic. This study also that showed that the results of acidic and basic functional groups on the surface of ACs obtained by Boehm titration were in agreement with those obtained by FTIR spectroscopy. According to results of this study, the wide transmittance band appearing at 3423 cm^{-1} is related to the OH stretching vibration mode in alcohol and phenol. The peak at 2359 cm^{-1} could be attributed to CN stretching. Bands appearing between 1622 and 1543 cm^{-1} are ascribed to C–C vibrations in aromatic rings. The band between 1340 - 1360 cm^{-1} may be attributed to the aromatic CH and carboxyl–carbonate structures. The bands located at 1111 cm^{-1} and 1052 cm^{-1} are related to C–O stretching in alcohols and phenols. The bands observed between 1000 cm^{-1} and 500 cm^{-1} are due to the out-of-plane deformation

mode of C–H for alkene aromatic rings.

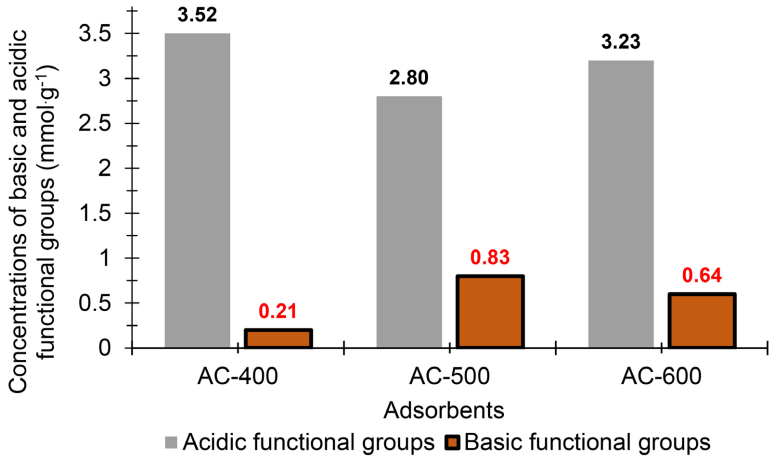

Figure 6. Concentrations of basic and acidic surface functional groups obtained on prepared activated carbons AC-400, AC-500, and AC-600 using Boehm titration method [25].

The results do not show significant variation in the acid and basic functional groups of prepared ACs as T_{cal} increases (**Figure 6**). However, on AC-500, there was a slight decrease in the amounts of acid surface functional groups (2.80 mmol·g^{-1}), compared to amounts obtained on AC-400 and AC-600 (3.52 and 3.23 mmol·g^{-1} respectively).

Thus, in our experimental conditions, the calcination temperature has a negligible effect on acidic and basic surface functional groups of studied materials. This parameter appears to be related to the activation agent used.

4. Conclusions

The objective of this work was to prepare activated carbons (ACs), by the chemical method, at different calcination temperatures (T_{cal}), 400˚C, 500˚C, and 600˚C (AC-400, AC-500, and AC-600) using palm nut shells as raw material and the zinc chloride ($ZnCl_2$) as a chemical activating agent. The prepared ACs were characterized by nitrogen (N_2) physisorption, the determination of iodine and methylene blue numbers for porosity studies, and the quantification and determination of surface functional groups and pH at the point of zero charge (pH_{pzc}) for chemical property studies. Then, effects of calcination temperature (T_{cal}) on porosity and chemical properties of prepared ACs were performed.

The results obtained show that when the calcination temperature reaches 600˚C, the porosity and chemical properties of the prepared ACs are modified. Indeed, methylene blue and iodine numbers determined on AC-400 (460 and 7.94 mg·g^{-1}, respectively) and AC-500 (680 and 8.90 mg·g^{-1} respectively) are higher than those obtained on AC-600 (360 and 5.75 mg·g^{-1}, respectively). Compared to AC-500 adsorbent, specific surface areas (S_{BET}) and microporous volumes losses on AC-600 were estimated at 44.7% and 45.8%, respectively. Moreover, in our

experimental conditions, the effect of T_{cal} on the quantities of acidic and basic functional groups on the surface of the ACs appears negligible. In addition, results of the pH at point of zero charge (pH_{pzc}) of the prepared ACs show that as T_{cal} increases, the pH of the adsorbents increases and tends towards neutrality. Indeed, a stronger acidity was determined on AC-400 (pH_{pzc} = 5.60) compared to those on AC-500 and AC-600 (pH_{pzc} = 6.85 and 6.70, respectively). Also based on the results of porosity and chemical characterizations, adsorption being a surface phenomenon, 500˚C appears to be the optimal calcination temperature in the preparation of activated carbons from palm nut shells in our experimental conditions.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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