

Preparation and Characterization of Activated Carbons from Asparagus Palm (*Laccosperma robustum*) Bark by Chemical Activation with H_3PO_4 and KOH

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Abstract

The present work deals with the preparation and characterization of activated carbons from the bark of the asparagus palm (*Laccosperma robustum*) by chemical activation with phosphoric acid and potassium hydroxide. The process was optimized on the basis of the analysis of the iodine number, methylene blue number and activated carbons yield as a function of the preparation parameters (concentration of the activating agents and the pyrolysis temperature). It emerges that the pyrolysis temperature and the concentration of activating agents influence the activated carbons preparation process. Their values were 500°C and 20% respectively for activated carbon with H_3PO_4 (ACP) and 700°C and 1.5% for activated carbon with KOH (ACK). The iodine numbers obtained were 850.26 mg/g for ACP and 865.49 mg/g for ACK. The methylene blue numbers obtained were 149.35 mg/g for ACP and 149.25 mg/g for ACK. The activated carbons yields obtained were 25% for ACP and 5.9% for ACK. The activated carbons prepared under optimal conditions have shown the pH of zero-point charge (pH_{ZPC}) of 4.4 and 7.0 for ACP for ACK respectively. The determination of the surface functions revealed that ACP had a strong acidic character while ACK had neutral character. The Fourier transformed infrared spectroscopy also showed the presence of different functional groups on the surface of the precursor and activated carbons.

Keywords

Activated Carbons, Asparagus Palm Bark, Chemical Activation, Phosphoric Acid, Potassium Hydroxide

1. Introduction

According to the socio-economic development, waste production becomes more important and their management therefore stands as a major problem of global concern. Indeed, biodegradable wastes are transformed by nature, whereas other wastes are not biodegradable or are difficult to transform accumulate and pollute nature [1]. Preparing activated carbons from these non-biodegradable wastes would help to solve this problem of discharge wastes, either partially or completely. There are several advantages including that are: 1) The valorization of waste as an available, renewable and cheap raw material; 2) The positive impact on reducing the cost price of the activated carbons obtained compared to the commercial ones which are too expensive; 3) A socio-economic impact on local population with the creation of non-polluting activities that generate employment; 4) A positive impact from an ecological and environmental point of view. The activated carbons (AC) are widely used on an industrial scale as adsorbents in the purification and separation of liquids and gases [2]. They can be obtained by carbonization and activation of precursors containing a high percentage of carbon and a low percentage of ash [2]. However, they are relatively expensive and not widely available in developing countries. The search for low-cost adsorbent materials with interesting physico-chemical characteristics for the preparation of activated carbons with high efficiency compared to commercial activated carbons is an important research topic [3]. Thus, many reports have appeared in recent years on the activated carbons preparation from a number of less expensive and carbon-rich plant materials such as kola edulis husks [4], coconut husks [5], sugarcane bagasse [6] or peanut husks [7]. It is in this perspective that the present work aims to valorize the bark of the asparagus palm into effective activated carbons for environmental sanitation. Gabon imports activated carbons, even though the country has considerable biomass and significant organic material waste. For example, asparagus palm (*Laccosperma robustum*) waste is widespread in the country. Only the bud (edible part) is widely traded and consumed by the population, while the bark of the asparagus palm is found in landfill sites, constituting abundant waste that is difficult to biodegrade and has not yet been subjected to any appropriate scientific study as far as our knowledge is concerned. The objective of this work is therefore to prepare activated carbons with a large adsorption surface area from these discarded barks, which can adsorb a varied number of pollutants contained in the water. Specifically, the impregnation and carbonization parameters will be varied by monitoring the evolution of the activated carbons yield, the iodine and methylene blue numbers.

The iodine number is a measure of the microporosity of activated carbon, thus, the higher its value, the greater the adsorption affinity of the material for small molecules [8], whereas, the methylene blue number makes it possible to measure the microporosity or even the microporosity of activated carbon [9]. Other characteristics such as the pH of zero-point charge, the determination of surface functional groups using the Boehm method and Fourier transform infrared spectroscopy were determined for activated carbons prepared under optimal conditions.

2. Materials and Methods

2.1. Sampling of Asparagus Palm Bark

The raw material consisting of asparagus peels was collected in Obia, Léconi-Lekori department, whose capital is Akiéni, in the South-Eastern region of Gabon in the Haut-Ogooué province. The asparagus peels were washed with tap water and then air-dried for three weeks. After drying, the asparagus peels were crushed and sieved to obtain particle sizes between 1 and 1.5 mm. The asparagus peels were washed again with distilled water and then dried in an oven at 110°C for 24 h.

2.2. Immediate Analysis

The immediate analysis of the asparagus peels consisted of determining the moisture content, ash content, volatile matter and fixed carbon.

2.2.1. Moisture Content

The moisture content was determined by simple oven drying according to the standard method (ASTM D1763-84). An empty crucible was weighed (m_o). A mass of 8 g (m_i) of sample was weighed into a crucible and the whole was placed in the oven at 110°C for 24 h. After removing from the oven, the assembly was cooled to room temperature in a desiccator and reweighed (m_1). Weighing was carried out until a constant mass was obtained. The moisture content was calculated from Equation (1):

$$\text{Moisture content (\%)} = \frac{(m_i - (m_1 - m_o)) \times 100}{m_i} \quad (1)$$

m_o : mass of the empty crucible before drying (g); m_i : mass of the sample before drying (g); m_1 : mass of the whole (crucible + sample) after drying (g).

2.2.2. Ash Content

The ash is the grey or whitish residue from the complete incineration of solid biofuels to a constant mass. The ash content was determined according to the standard method (ASTM D2866-11). An empty crucible with lid was weighed (m_o). A mass of 1 g (m_i) of sample was weighed into the crucible and the assembly was placed in an oven set at 650°C for 4 h. After cooling to room temperature, the assembly was weighed again (m_1). The ash content was calculated from

Equation (2):

$$\text{Ash content (\%)} = \frac{(m_1 - m_o) \times 100}{m_i} \quad (2)$$

m_o : mass of the empty crucible before carbonization (g); m_i : mass of the sample before carbonization (g),

m_1 : mass of the whole (crucible with lid + sample) after carbonization (g).

2.2.3. Volatile Matters Content

The volatile matters are the gases and vapors released from the sample during the heating process. The volatile matters content was determined according to the standard method (ASTM D5832-98). An empty crucible with lid was weighed (m_o). A mass of 1 g (m_i) of sample was weighed into a crucible and the whole was placed in an oven set at 900°C for 7 min. After cooling to room temperature, the assembly was reweighed (m_1). The volatile matter content was calculated from Equation (3):

$$\text{Volatile matters content (\%)} = \frac{(m_i - (m_1 - m_o)) \times 100}{m_i} \quad (3)$$

m_o : mass of empty crucible before carbonization (g); m_i : mass of the sample before carbonization (g); m_1 : mass of the whole (crucible with lid + sample) after carbonization (g).

2.2.4. Fixed Carbon Content

The fixed carbon content was calculated from Equation (4):

$$\text{Fixed carbon (\%)} = 100 - \text{Moisture content} - \text{Volatile matters} - \text{Ash content} \quad (4)$$

2.3. Activated Carbons Preparation

Due to the greater energy consumption required by physical activation, but also its difficult and costly implementation, the activated carbons preparation was carried out by chemical activation using two different activation reagents that were phosphoric acid and potassium hydroxide. The activated carbons were prepared according to a protocol taken from the work of Merlin *et al.* (2017) [10]. Thus, 20 g of biomass were impregnated with phosphoric acid and another 20 g were impregnated with potassium hydroxide by varying the impregnation ratio (mass of activating reagent/mass of precursor) and carbonization temperature. The concentration varied from 10% to 40% for phosphoric acid and the impregnation ratio from 1 to 2.5 for potassium hydroxide. After suitably mixing the precursor with the different activating agents for 02 hrs, the whole mixture was dried in an oven at 110°C for 24 hrs. The impregnated sample was then placed in crucibles with lids and the whole set was placed in a muffle furnace where carbonization was carried out at different temperatures (400°C, 500°C, 600°C and 700°C) for 1 h at a heating rate of 10°C/min. After the carbonization, the carbonized product obtained was cooled and washed several times with distilled water in order to remove the residues of the different activating agents.

The washing was carried out until a pH of between 6 and 7 was obtained. After washing, the activated carbons obtained were put in the oven at 110°C for 24 hrs for complete drying. Then after, removed ground and sieved to obtain particles with a diameter of less than 75 µm. Activated carbons activated with phosphoric acid were designated ACP and those activated with potassium hydroxide ACK.

2.4. Optimization of Activated Carbon Preparation Conditions

The process was optimized on the basis of the analysis of the activated carbons yield, the iodine and methylene blue numbers as a function of the preparation parameters such as the concentration of the activating agents and the carbonization temperature.

2.4.1. Determination of the Iodine Number

The iodine value is an important parameter for characterizing the adsorption capacity of carbons. It evaluates the adsorption capacity of very small molecules and is defined as the quantity, in milligrams, of iodine adsorbed by 1 g of activated carbon in an iodine solution whose normality is 0.02 N [9]. Thus, 0.1 g of activated carbon was brought into contact with 20 mL of a 0.01 M iodine solution in a 250 mL Erlenmeyer flask. The mixture was stirred for 30 min and then filtered. After filtration, a 10 mL volume of this filtrate was titrated with a 5×10^{-3} M sodium thiosulphate solution ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in the presence of starch as an end-of-reaction indicator. The iodine number (IN) was obtained from Equation (5):

$$\text{IN}(\text{mg/g}) = \frac{(C_i - C_f) \times V}{m} \quad (5)$$

C_i : concentration of iodine before adsorption (mol/L); C_f : concentration of iodine after adsorption (mol/L); V : volume of the solution (L); m : mass of activated carbon (g).

2.4.2. Determination of the Methylene Blue Number

The methylene blue number, expressed in mg/g, is defined as the quantity in mg of methylene blue adsorbed by 1 g of activated carbon. It indicates the capacity of activated carbon to adsorb medium-sized molecules [9]. Thus, 0.1 g of activated carbon was brought into contact with 30 mL of a 500 mg/L methylene blue solution in a 250 mL Erlenmeyer flask. The mixture was stirred for 4 h and then filtered. After filtration, the solution was determined by UV-visible spectrophotometer at a maximum wavelength of 664 nm. The value of the methylene blue number (MBN) was obtained from Equation (6):

$$\text{MBN}(\text{mg/g}) = \frac{(C_o - C_e) \times V}{m} \quad (6)$$

C_o : initial concentration of the methylene blue solution (mg/L); C_e : residual concentration of methylene blue at equilibrium (mg/L); V : volume of methylene blue solution (L); m : mass of activated carbon (g).

2.4.3. Determination of Activated Carbons Yield

The activated carbons yield reflects the mass loss of the precursor during the preparation of the activated carbon. It was determined from Equation (7):

$$\text{AC Yield}(\%) = \frac{(\text{mass of activated carbon}) \times 100}{\text{mass of precursor}} \quad (7)$$

2.5. Characterization of Activated Carbons

2.5.1. Determination of the pH of Zero-Point Charge

The pH of zero-point charge (pH_{ZPC}) defines the pH at which the overall surface charge of a solid material is zero. Below the pH_{ZPC} value, the surface carries a positive charge and above the pH_{ZPC} the surface is negatively charged [8]. Solution of 0.1 M sodium chloride was prepared at different pH values (2 to 10). The pH values were adjusted with a pH meter using 0.1 M sodium hydroxide and 0.1 M hydrochloric acid solutions. Then 0.1 mg of activated carbon was mixed with 30 mL of each solution in 250 mL Erlenmeyer flasks. The mixture was stirred for 72 h and then filtered with filter paper. After filtration, the pH was measured again. The pH_{ZPC} value was determined by plotting $\Delta\text{pH} = \text{pH}_f - \text{pH}_i = f(\text{pH}_i)$.

2.5.2. Determination of Surface Functional Groups

The determination of the surface functional groups was carried out by the Boehm method, which corresponds to the acid-base titration. The basic groups are determined as a whole, while the acid groups are determined separately. A mass of 0.1 g of activated carbon was mixed with 30 mL of each of the aqueous solutions of sodium hydroxide (NaOH), sodium carbonate (Na₂CO₃), potassium bicarbonate (KHCO₃) and 0.1 N hydrochloric acid (HCl). Each solution was stirred for 72 h and then filtered with filter paper. Then, 5 mL of the filtrate of each solution was determined. The basic solutions were determined by hydrochloric acid and the acidic solution by sodium hydroxide. Phenolphthalein and helianthin were used as indicators.

In addition, **Table 1** and **Table 2**, below, present respectively the chemical reagents and the apparatus used for the realization of the various experiments.

2.5.3. Fourier Transform Infrared Spectroscopy

The Fourier transform infrared spectroscopy is a very important analysis for the study of functional groups on the surface of a material. It is based on the irradiation of a sample with infrared (IR) radiation whose absorbed adsorption wavelengths are characteristic of the different chemical groups or atoms present on its surface. The IR analyses were carried out using a BRUKER infrared spectrometer with retention of 4 cm⁻¹ in the 4000 - 400 cm⁻¹ range. The samples and the potassium bromide (KBr) were pre-dried at 100 °C for 24 hours before analysis. The KBr and samples were prepared using the “disc technique” by using a finely ground mixture of 0.25 mg sample with 100 mg KBr. The pellets thus formed by uniaxial compression were placed in the sample holder of the apparatus for

Table 1. Chemical reagents.

Name	Chemical formula	Origine	Purity (%)
Phosphoric acid	H ₃ PO ₄	Prolab	85
Hydrochloric acid	HCl	Prolab	30
Starch	C ₆ H ₁₂ O ₆	Prolab	100
Methylene blue	C ₁₆ H ₁₈ ClN ₃ S	Prolab	100
Sodium carbonate	Na ₂ CO ₃	Prolab	100
Helianthine	C ₁₄ H ₁₅ N ₃ O ₃ S	Prolab	100
Sodium hydroxide	NaOH	Prolab	100
Potassium hydroxide	KOH	Prolab	100
Potassium hydrogen carbonate	KHCO ₃	Prolab	100
Iodine (granules)	I ₂	Prolab	100
Potassium iodide	KI	Prolab	100
Phenolphthalein	C ₂₀ H ₁₄ O ₄	Prolab	100
Congo red	C ₃₂ H ₂₂ N ₆ O ₆ S ₂ Na ₂	Prolab	100
Sodium thiosulfate	Na ₂ S ₂ O ₃ ·5H ₂ O	Prolab	98

Table 2. Apparatus.

Apparatus	Brand	Precision
Magnetic Stirrer	Edmund buhler gmbh	1%
Electronic balance	Golden-Mettler USA	0.001 g
Electric grinder	TRF 300 Super-Trapp	
Oven Blue	Blue Pad	1 °C
Muffle Oven	Carbolite Gero	1 °C
Filter paper	Whatman 1	110 mm
Spectrophotometer UV-Visible S23A	Techmel USA	2 nm
Spectrometer	Bruker	4 cm ⁻¹
Sieves	ASTM	1 mm et 75 μm

analysis. The results of the transmittance versus wavenumbers are read directly from the screen. Subsequently, the spectrogram obtained was analyzed to identify the functional groups on the surface of the samples.

3. Results and Discussion

3.1. The Results of the Immediate Analysis

The results of the immediate analysis are presented in **Table 3**.

Table 3. Immediate analysis.

Parameters	Values (%)
Moisture content	2.31
Volatile matter	85.85
Ash content	1.60
Fixed carbon	10.24

From **Table 3**, it can be seen that the precursor has low moisture content (2.31%) and low ash content (1.6%) while the volatile matter content (85.85%) is high. The fixed carbon content is 10.24%. According to other authors, the limit value for moisture content should not exceed 6% [11]. Moreover, the ash content of activated carbons is generally in the range of 1% to 20%, depending mainly on the nature of the precursor [12]. As an adsorbent, the lower the ash content, the better the activated carbon [10] [13]. These results suggested that the asparagus bark is a good precursor for the activated carbons preparation.

3.2. The Results of Fourier Transform Infrared Analysis

The Fourier transform infrared analysis made it possible to determine the functional groups likely to be present on the surface of asparagus barks. The FTIR spectrum of asparagus bark is shown in **Figure 1**.

It can be seen from **Figure 1**, that the peak at 3297 cm^{-1} corresponds to the elongation of the O-H bond of hydroxyl groups (alcohols or phenols) [14]. The peak at 2918 cm^{-1} corresponds to the C-H bond of aliphatic radicals [15]. The peak at 1717 cm^{-1} corresponds to the C=O bonds of carboxylic acid functions [8]. The peaks at 1634 and 1508 cm^{-1} could correspond to the C=O bonds of carbonyl groups (aldehydes or ketones) and the C=C bonds of monosubstituted alkenes [16]. The peak at 1419 cm^{-1} corresponds to the C=C elongation vibration of aromatic rings [16]. The peaks at 1241 and 1022 cm^{-1} correspond to the axial deformation of C-O bonds in carboxylic acids, alcohols, esters and ethers [15].

3.3. Influence of the Operating Parameters on the Activated Carbons Yield

The activated carbons yield is an important measure of the feasibility of producing activated carbon from a given precursor under given conditions. It is an indicator of the performance of an activated carbon preparation method, especially on an industrial scale.

3.3.1. Case of Activated Carbons Obtained by Impregnation with H_3PO_4

The influence of the pyrolysis temperature (**Figure 2(a)**) and the concentration (**Figure 2(b)**) on the activated carbons yield is shown in **Figure 2**.

Figure 2 shows that the yield values range from 43.35% to 39.7% for 10% activated ACP, from 27% to 21.5% for 20% activated ACP, from 29.5% to 22.5%

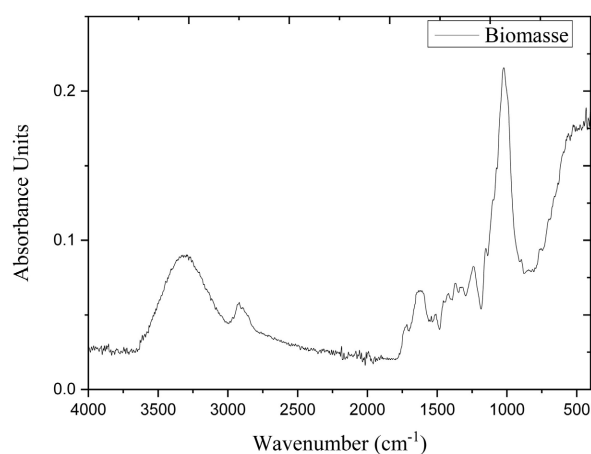


Figure 1. FTIR spectrum of asparagus peels.

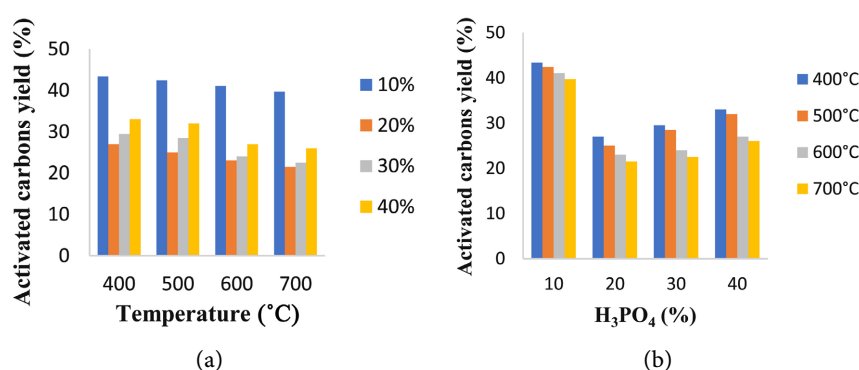


Figure 2. Influence of carbonization temperature and concentration on activated carbons yield.

for 30% activated ACP and from 33% to 26% for 40% activated ACP when the temperature varies from 400°C to 700°C. In all cases, the percentage of activated carbons yield decreases with increasing pyrolysis temperature and the high yield values were obtained at low temperature (10°C). This decrease could be explained by the loss of volatile matter under the effect of temperature, in the presence of H₃PO₄. Indeed, the transformation of precursors into activated carbon involves the loss of oxygen (O) and hydrogen (H) atoms in the form of volatile compounds (H₂O, CO, CO₂, CH₄) [10]. In addition, the low rate of decrease in activated carbons yield could mean the formation and stabilization of activated carbons [10]. On the other hand, increasing the concentration of phosphoric acid increases the activated carbons yield. This can be explained by the fact that H₃PO₄ is a dehydrating agent that delays thermal decomposition, limiting the loss of volatile matter and leading to the formation of a rigid carbon matrix [17]. The difference observed could be explained by a certain loss of mass of activated carbons during washing.

3.3.2. Case of Activated Carbons Obtained by Impregnation with KOH

The influence of the pyrolysis temperature (Figure 3(a)) and the impregnation rate (Figure 3(b)) on the activated carbons yield is shown in Figure 3.

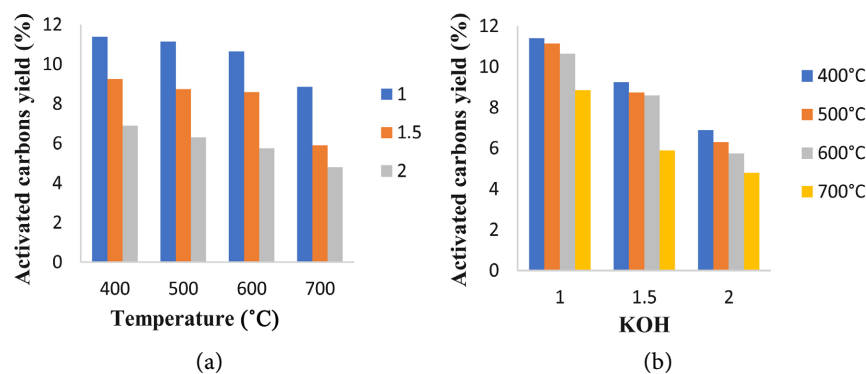


Figure 3. Influence of temperature and ratio on mass yield.

Figure 3 shows that the activated carbons yield values range from 11.4% to 8.85% for ACK activated at 1, from 9.25% to 5.9% for ACK activated at 1.5, from 6.9% to 4.8% for ACP activated at 2 when the temperature is increased from 400°C to 700°C. It is observed that the percentage of activated carbons yield decreases with the increase of the pyrolysis temperature and the impregnation ratio. This decrease could be explained by the loss of volatile matter under the effect of temperature in the presence of KOH, as previously with H_3PO_4 . Furthermore, the activated carbons yield decreases with the increase of the impregnation ratio. This decrease could be explained by a continuous release of tars inside the pores. These results are consistent with the results obtained by other authors [14].

3.4. Influence of the Operating Parameters on the Iodine Number

The iodine number allows the microporosity of the activated carbon to be estimated. Thus, the higher its value, the greater the adsorption affinity of the material for small molecules.

3.4.1. Case of Activated Carbons Obtained by Impregnation with H_3PO_4

The influence of the pyrolysis temperature (**Figure 4(a)**) and of the concentration (**Figure 4(b)**) on the iodine number is shown in **Figure 4**.

It can be seen from **Figure 4** that in all cases the increase in temperature from 400°C to 700°C and in concentration from 10% to 40% leads to an increase in the amount of iodine until it reaches a maximum value and then decreases. In the case of temperature, the increase in the amount of iodine as a function of temperature can be explained by the fact that the increase in temperature leads to the elimination of the volatile materials contained in the precursor [8]. This elimination leads to an increase in the adsorption capacity of the activated carbons and therefore the iodine number. On the other hand, the decrease in the value of the iodine number beyond temperatures of 500°C and 600°C can be explained by the fact that part of the micropores formed has been destroyed. This decrease in the number of micropores is accompanied by a reduction in the adsorption capacity of activated carbons [8]. In the case of concentration, the

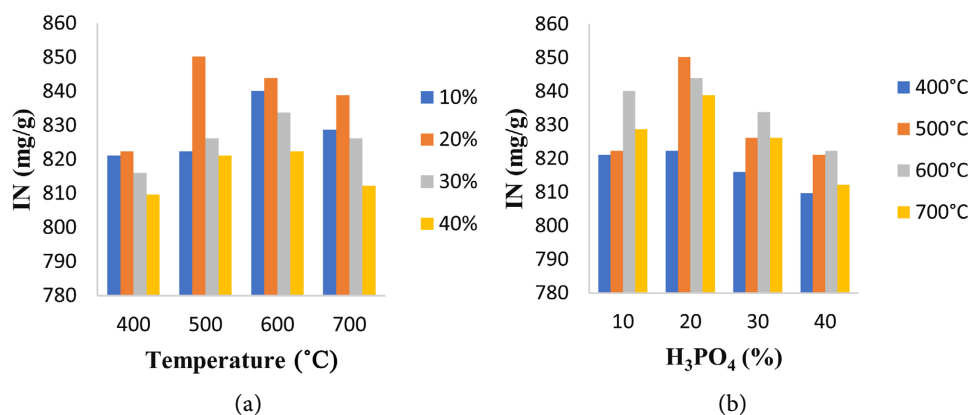


Figure 4. Influence of temperature and concentration on the iodine number.

increase in the quantity of iodine could be attributed to impregnation with phosphoric acid, which inhibits the formation of tar and favors the release of volatile matter to produce more micropores [18]. These micropores favor the adsorption of small molecules like iodine. Hence it increases. Furthermore, all ACP achieved maximum iodine content at 20%. But when the concentration is higher than 20%, the amount of iodine decreases. This decrease can be explained, on the one hand, by excessive dehydration and the destruction of the micropores by making them larger due to the very high oxidizing capacity of H₃PO₄ [19] and, on the other hand, by a possible formation of phosphate, via the interaction between the excess of H₃PO₄ and the inorganic elements present in the precursor, under the action of pyrolysis and would lead to the obstruction of the pores, thus preventing further volatilization [19]. The maximum iodine number (850.26 mg/g) was obtained at a temperature of 500°C for a H₃PO₄ concentration of 20%.

3.4.2. Case of Activated Carbons Obtained by Impregnation with KOH

The influence of the pyrolysis temperature (Figure 5(a)) and the impregnation ratio (Figure 5(b)) on the iodine index is represented in Figure 5.

The results in Figure 5 show that the amount of iodine increases with increasing temperature while with concentration it increases to a maximum value before decreasing. In the case of temperature, the amount of iodine in all activated carbons reaches maximum values at 700°C. The effect of temperature can be explained by the fact that the increase in temperature leads to the elimination of the volatile matter contained in the precursor [20]. In the case of concentration, this could be explained by the creation of micropores during impregnation with the base, which favors the adsorption of small molecules such as iodine [20]. Moreover, the maximum iodine quantity was obtained at 1.5 for all the carbons. But beyond 1.5, there is an enlargement of the micropores and this enlargement is unfavorable to the adsorption of small molecules [21]. The maximum iodine value (865.49 mg/g) was obtained at a temperature of 500°C for a H₃PO₄ concentration of 20%.

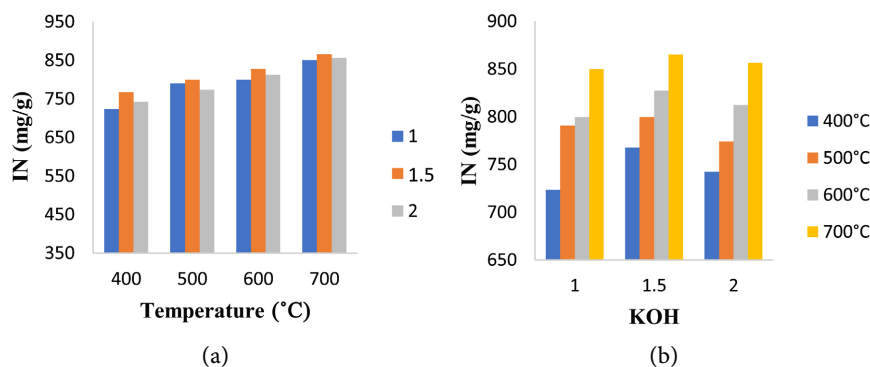


Figure 5. Influence of temperature and impregnation ratio on the iodine number.

3.5. Influence of the Operating Parameters on the Methylene Blue Number

3.5.1. Case of Activated Carbons Obtained by Impregnation with H_3PO_4

The influence of the pyrolysis temperature (**Figure 6(a)**) and of the concentration (**Figure 6(b)**) on the methylene blue number is represented by **Figure 6**.

From **Figure 6**, it can be seen that the amount of methylene blue increases proportionally with increasing temperature and concentration. Increasing the temperature leads to a continuous release of the volatiles contained in the precursor, while increasing the concentration enhances the catalytic oxidation of the precursor causing the micropores to enlarge into mesopores [22]. The higher the percentage of mesopores, the higher the adsorption of methylene blue. These results are in perfect correlation with those found for the iodine value, which showed the appearance of mesopores for temperatures and concentrations higher than 500°C and 20% respectively.

3.5.2. Case of Activated Carbons Obtained by Impregnation with KOH

The influence of the pyrolysis temperature (**Figure 7(b)**) and the impregnation ratio (**Figure 7(a)**) on the methylene blue number is represented by **Figure 7**.

Figure 7 shows that the amount of methylene blue increases proportionally with increasing temperature and concentration. As with H_3PO_4 seen previously, increasing the temperature leads to a continuous release of the volatiles contained in the precursor while increasing the ratio enhances the catalytic oxidation of the precursor causing the enlargement of the micropores into mesopores. The best parameters for the preparation of ACP and ACK are summarized in **Table 4** below.

3.6. The Analysis of the Fourier Transformed Infrared Spectroscopy of ACP and ACK

The Fourier transformed infrared analysis was used to determine the functional groups likely to be present on the surface of ACP and CAK and their FTIR spectra are given in **Figure 8** and **Figure 9** respectively.

The spectrum in **Figure 8** shows that the peaks at 3297 and 2918 cm^{-1} that were present on the surface of the precursor disappear. This disappearance can

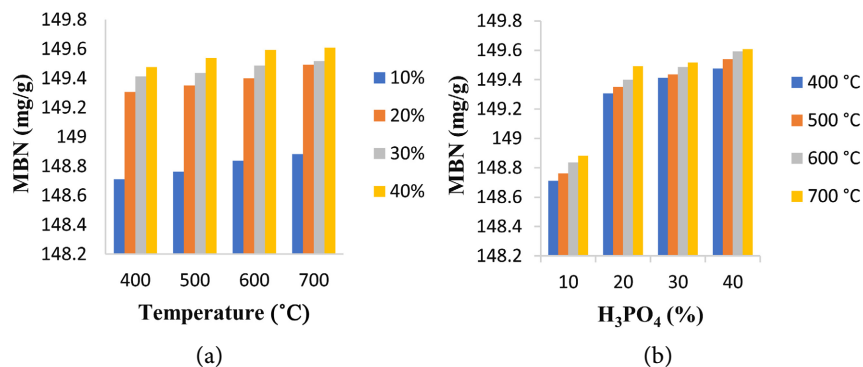


Figure 6. Influence of temperature and concentration on the methylene blue number.

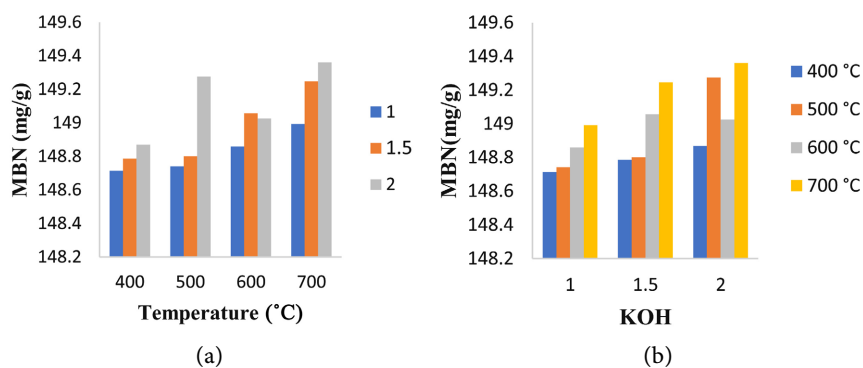


Figure 7. The influence of concentration and ratio on the methylene blue number.

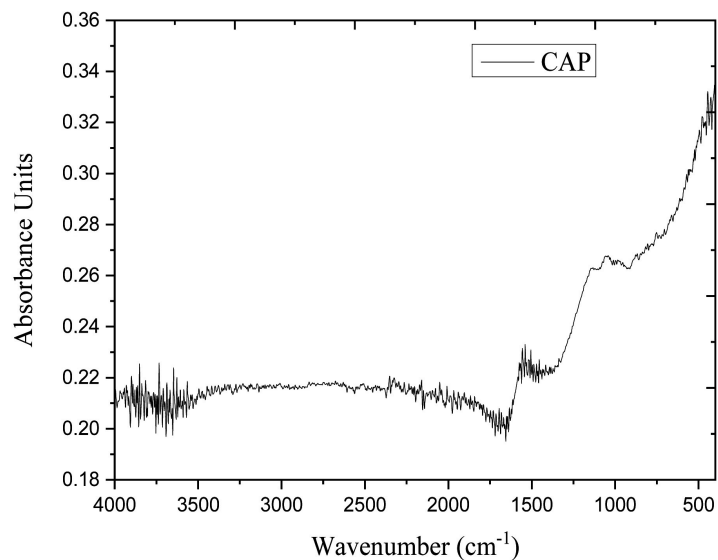


Figure 8. FTIR spectrum of ACP.

be explained by the loss of volatile matter under the effect of temperature. Then, there is a peak at 2325 cm⁻¹ corresponding to the O=P-OH bond. The peak at 1539 cm⁻¹ corresponds to the C=C bonds of the aromatic rings [22]. The peak at 1042 cm⁻¹ is attributed to the O-C bond of P-O-C. The peaks at 867 and 752 cm⁻¹ correspond to the O-C bond of P-O-C.

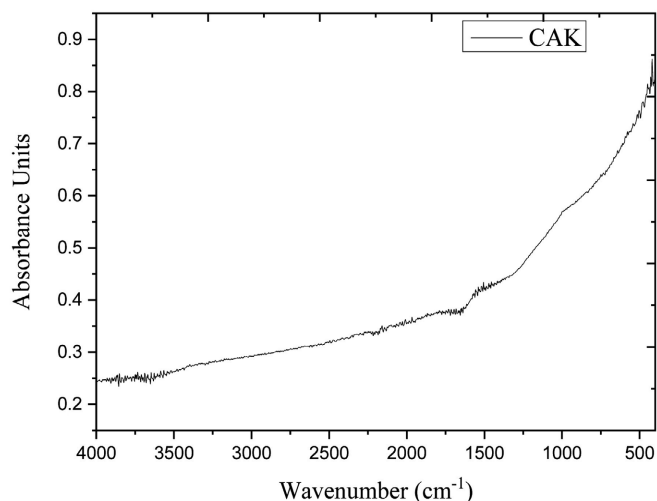


Figure 9. FTIR spectrum of ACK.

Table 4. Optimum parameters for the preparation of activated carbons.

Parameters	Adsorbents	
	ACP	ACK
Concentration (or ratio)	20%	1.5
Temperature (°C)	500	700
Activated carbons yield (%)	25	6.3
IN (mg/g)	850.26	865.49
MBN (mg/g)	149.35	149.25

The spectrum in **Figure 9** shows that the peaks at 3297 and 2918 cm^{-1} which were present on the surface of the precursor have disappeared. This disappearance can be explained by the loss of volatile matter under the effect of temperature as seen previously in the spectrum of H_3PO_4 . The peak located at 1731 cm^{-1} corresponds to the C=O bonds of the carboxylic acid functions. The peak at 1539 cm^{-1} corresponds to the C=C bonds of the aromatic rings.

3.7. Determination of the pH_{zpc} of ACP and ACK

The pH_{zpc} values of activated carbons are presented in **Figure 10**.

The results of **Figure 10** show that the pH_{zpc} value of ACP is 4.4. This value reflects the acidity of this carbon by the predominance of acid groups on its surface. On the other hand, the value of the pH_{zpc} of ACK is 7 which reflects an equality between the acid and basic groups.

3.8. Determination of Surface Functional Groups

The determination of the acid and basic functional groups on activated carbons was carried out using the Boehm titration method. It is based on the determination of the acid-base strength of the surface functions. The results obtained are reported in **Table 5**.

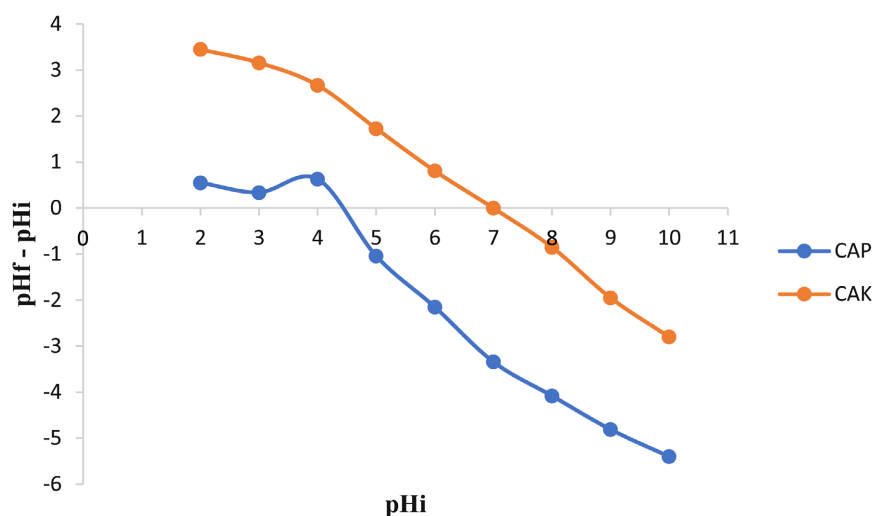


Figure 10. The graph of the pH_{zpc} of activated carbons.

Table 5. Functional groups on the surface of ACP and ACK.

Activated carbons	Quantities (mmol/g)				
	Carboxylic	Lactonic	Phenolic	Acidic	Basic
ACP	0.625	0.5625	1.5625	2.75	1.00
ACK	0.625	0.0625	1.5625	2.25	2.25

The results in **Table 5** show that the ACP surface is predominantly composed of acidic groups with an amount of 2.75 mmol/g while the amount of basic group is 1 mmol/g. This result is in agreement with those obtained with the FTIR spectrum and the pH_{zpc}. On the other hand, there is equality between the amount of acidic and basic groups on the surface of ACK. This result is in agreement with those of the pH_{zpc} and the FTIR spectrum.

4. Conclusion

The aim of the present work was to prepare and characterize activated carbons from asparagus palm bark by chemical activation with phosphoric acid and potassium hydroxide. This study shows that the pyrolysis temperature and the concentration of activating agents influence the activated carbons preparation process. The values obtained were 500°C and 20% for ACP and 700°C and 1.5 for ACK. The iodine numbers obtained were 850.26 mg/g for ACP and 865.49 mg/g for ACK. The methylene blue numbers obtained were 149.35 mg/g and 149.25 mg/g for ACP and ACK respectively. The activated carbons yields obtained were 25% for ACP and 5.9% for ACK. The functions on the surface of ACP are acidic in nature and their pH_{zpc} is 4.4. The functions on the surface of ACK were neutral with a pH_{zpc} of 7. The results obtained showed that the barks of the asparagus palm could be good precursors for the preparation of activated carbons.

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Conflicts of Interest

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

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