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Optimization Of The Production Of Activated Carbon Synthesized From *Zizyphus Mauritiana* (L.) Lam. Shells And Determination Of Iodine And Methylene Blue Indices

Ousmaila SANDA MAMANE [1,2], Mahamane Nassirou AMADOU KIARI[2,3*], Rabilou SOULEY MOUSSA[2], Maman Mousbahou MALAM ALMA[2] And Ibrahim NATATOU[1,2]

[1]*Department Of Basic Sciences,National School Of Engineering And Energy Sciences/ University Of Agadez, Niger*
[2]*Department Of Chemistry, Faculty Of Science And Technology, Materials, Water And Environment Laboratory Abdou Moumouni University, Niger*
[3]*Laboratory Of Industrial Processes Of Synthesis, The Environment And New Energies (LAPISEN), Institut National Polytechnique Félix Houphouët-Boigny, African Center Of Excellence For The Recovery Of Waste Into High Value-Added Products (CEA-VALOPRO), Yamoussoukro, Côte d'Ivoire BP 1093*

Abstract
The objective of this work is to optimize the production of activated carbon synthesized from Zizyphus Mauritiana (L.) Lam. shells and to determine the iodine and methylene blue indices. The shells of the selected biomass are impregnated with a solution of orthophosphoric acid (H PO) and sulfuric acid (H SO). The activation and pyrolysis parameters (concentration effect, impregnation rate, impregnation time, and pyrolysis temperature) were studied. The iodine and methylene blue indices were determined using the CEFFIC method. The results obtained show that the various tests carried out for the production of activated carbon yielded reproducible mass yields ranging from 17.3 to 56.6%. The best yields were obtained at an impregnation time of 9 hours (56.6%) in the case of H_3PO_4 activation. Regardless of the activating agent considered, it was noted that when the pyrolysis temperature was high, the yield decreased. All parameters studied have a real influence on iodine indices. In general, the adsorption capacities of iodine I_2 increase with the concentration of the activating agent and the impregnation rate in the case of H_3PO_4. Activated carbons prepared by H_3PO_4 activation developed the best iodine adsorption capacities (990.6 and 965.2 mg g^{-1} respectively for an impregnation time of 6 hours and concentrations of 40 and 50%). The methylene blue indices ranged from 402.5019 to 691.3629 mg.100 g $^{-1}$ for H SO and H PO . The discoloration rates of the methylene blue solution vary from 57.07 to 98.02% for a concentration of 10% H_2SO_4 and a pyrolysis temperature of 600°C.
Keywords : *Optimization, Activated Carbon, Zizyphus Mauritiana (L.) Lam, Iodine, and Methylene Blue.*

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I. Introduction

In recent years, the use of activated carbon as an adsorbent has been the subject of numerous patents, generating growing interest in both industry and the medical field [1–3]. The possible applications of this material are extremely varied [4–9]. They mainly exploit its physicochemical properties and specific surface area [10,11]. The various characteristics of AC justify its wide range of applications [12]. In the medical field, AC is used as a chelating agent in a large number of cases of poisoning, in digestive purification, and in particular to lower total cholesterol levels [13]. It is also used in the decolorization of sweeteners (glucose, sucrose, and derivatives from the starch industry), organic acids from fermentation processes, amino acids, and vitamins [12,14,15]. Activated carbon consists mainly of carbonaceous material with a porous structure [5,16,17]. This structure is generally obtained after a high-temperature carbonization stage of "lignocellulosic" biomass. There are various types of AC with specific surface areas ranging from 100 to 2500 m^2 g^{-1} [18]. Despite the availability of biomass in the sub-region, African countries continue to import large quantities of activated carbon (AC) for various applications, including industrial wastewater treatment, ore processing, etc. It therefore seems necessary to develop and characterize ACs from local lignocellulosic biomass, particularly the shells of *Zizyphus mauritiana* (L.) Lam. (Magaria) kernels, through chemical activation with orthophosphoric acid and sulfuric acid. The selected biomass comes from wild trees that are widespread in Niger and produce seasonal fruits consumed by the population. The pits of these fruits end up in municipal landfills as urban waste.

They constitute abundant agri-food waste that is more or less difficult to biodegrade in tropical countries. The use of these pits in this work has a double advantage: on the one hand, it produces activated carbon and, on the other hand, it recycles waste to give it added value.

II. Materials And Methods

Plant Material

The plant material used to produce activated carbon is the shells of *Zizyphus Mauritiana* (L.) Lam. kernels. This selected biomass comes from a fruit and vegetable market in the city of Niamey (Niger) called KATAKO. The fruits (Figure 1) were first peeled, then the pits were collected, washed thoroughly with water, and dried in the sun for five days. Next, the pits were crushed using a rotary jaw crusher (Figure 2) to separate the shells from the kernels (Figure 3). The shells, which are the precursors, were then ground using a ball mill (Figure 4), sieved, and only particles with a particle size between 0.8 and 2 mm were retained (Figure 5) for the production of activated carbon and dried in an oven at 105°C for 24 hours (h).

Figure 1. Fruit of *Zizyphus mauritiana* (L.) Lam.

Figure 2. Rotary jaw crusher

Figure 3. Shells and almonds of *Zizyphus mauritiana* (L.) Lam.

Figure 4. Ball mill

Figure 5. Particles with a particle size between 0.8 and 2 mm

Production of activated carbon

The synthesis of activated carbon is carried out in three stages [1–3,12,19,20].Table 1 shows the experimental conditions for the production of activated carbon.

Table 1 Conditions for the synthesis of activated carbons

Steps	Experimental protocol
Impregnation	16 g of pretreated biomass and 100 mL of activating agent solution (H PO or H SO) were added. The resulting mixture was stirred for 15 hours on a magnetic stirrer at atmospheric pressure and room temperature.
Pyrolysis	The dry sample obtained after impregnation was placed in a programmable high-temperature muffle furnace. The oven temperature was gradually increased to the pyrolysis temperature (450°C) at a heating rate of 2.5°C/min, with an isothermal plateau of 1 hour 30 minutes at the end of heating, representing the residence time in the oven.
Purification	At the end of the process, the activated carbon obtained is cooled and then washed thoroughly with hot distilled water until the pH reaches between 6.5 and 7 to remove impurities, then dried in an oven at 105°C for 24 hours.

To optimize the performance of CAs, the effects of certain activation and pyrolysis parameters were studied to determine the optimal conditions for activated carbon production. This study was conducted under conditions where all other parameters remained constant. Table 2 shows the effect of activation parameters.

Table 2. Optimization parameters for CAE development

	Parameters	
***Zizyphus Mauritiana* (L.) Lam**	Concentration of the activating agent (%)	10 20 30 40 50
	Impregnation rate (%)	24 32 40 48 56
	Imprégnation time (h)	3 6 9 12 15
	Pyrolysis temperature (°C)	300 400 500 600 700

The Activated Carbon Products (ACPs) are cooled and stored in airtight bottles until characterization and application tests are performed.

Characterization of Activated Carbon Products

Mass yield after pyrolysis

The mass yield represents the ratio of the mass of activated carbon (m_{AC}) to the mass of biomass (m_B) used. The mass was measured using a precision balance. The mass yield is determined by equation (1):

Iodine index

The procedure used is that of the Waste Study Center, which is an adaptation of the CEFIC (European Chemical Industry Council) 1989 method and AWWA B 600 – 78 standard [1–3,19,20].

In a 100 mL beaker, 0.2 g of activated carbon powder (particle size less than 0.1 mm) was mixed with 20 mL of 0.02 N I_2 solution. The mixture was stirred for 4 to 5 min and then filtered through ashless filter paper. Next, a 10 mL volume of the filtrate was taken and titrated with a 0.1 N sodium thiosulfate (Na S O) solution. Phenolphthalein was used as a color indicator. The iodine index is given by equation (2)

Where, : adsorption capacity of I_2; C_0: initial concentration of the I_2 solution (in mol/L); C_{thio}: concentration of $Na_2S_2O_3$ (in mol/L); V_{thio}: volume of Naso at equivalence (in mL); V_1 : volume of iodine titrated (in mL); MI : molar mass of I (in g/mol); Vads: adsorption volume (in mL); m_{ca}: mass of adsorbent used (in g).

Methylene Blue (MB) Index

The procedure used is that of the CEFIC method [1–3,12,19–21]. In practice, it is expressed in mg/100g of charcoal.

BM adsorption was performed by introducing 0.1 g of CA into a 250 mL Erlenmeyer flask with 100 mL of the standard BM analysis solution. The mixture was stirred for 20 min. After this contact time, it was filtered through filter paper and the residual MB concentration was determined using a UV-visible spectrophotometer at a wavelength of 620 nm. Thus, equation (3) gives the Methylene Blue index.

With : adsorption capacity of CA (in mg/g); Ci: initial concentration of BM solution (in mol/L); Cr: residual concentration of BM solution (in mol/L); V: volume of BM solution (in mL); M: molar mass of BM; m: mass of adsorbent used (in g)

III. Results

Mass yield after pyrolysis

The results of the mass yields of activated carbons produced by H_3PO_4 activation are shown in Table 3.

Table 3. Mass yield after pyrolysis/H_3PO_4 activation

Biomass	Parameters		Mass of CAE (g)	Yield (%)
Zizyphis mauritiana	Concentration (%)	10	8.79	43.95
		20	9.74	48.7
		30	8.68	43.4
		40	9.52	47.6
		50	9.27	46.35
	Imprégnation rate (%)	24	5.31	44.25
		32	7.02	43.87
		40	8.96	44.8
		48	10.55	43.95
		56	12.57	44.89
	Imprégnation time (H)	3	10.78	53.9
		6	10,66	53.3
		9	11,32	56.6
		12	9.18	45.9
		15	9.56	47.8
	Pyrolysis temperature (°C)	300	11.18	55.9
		400	10.3	51.5
		500	8.1	40.5
		600	9.62	48.1
		700	9.22	46.1

Table 4 shows the mass yield results for activated carbons produced by H_2SO_4 activation.

Table 4. Mass yield after pyrolysis/H_2SO_4 activation

Biomass	Parameters		Mass of CAE (g)	Yield (%)
		10	8.35	41.75
		20	7.58	37.9

Zizyphis mauritiana	Concentration (%)	30	7.44	37.2
		40	6.7	33.5
		50	6.88	34.4
	Imprégnation rate (%)	24	3.37	28.08
		32	5.98	37.37
		40	7.47	37.35
		48	8.55	35.62
		56	10.26	36.64
	Imprégnation time (H)	3	6.34	31,7
		6	6.88	34,4
		9	6.89	34.45
		12	6.7	33.5
		15	7.24	36.2
	Pyrolysis temperature (°C)	300	9.3	46.5
		400	7.24	36.2
		500	3.46	17.3
		600	4.24	21.2
		700	4.02	20.1

Iodine index

The iodine index characterizes the areas accessible to any particle smaller than or equal to the size of the I_2 molecule, as well as their adsorption capacity, particularly the mini-micropores accessible to small particles responsible for tastes and odors. The iodine index results for activated carbons produced by H_3PO_4 activation are shown in Table 5.

Table 5. I_2 adsorption capacity (mg g ^{1}) / H PO activation

Biomass	Parameters		Volume of Na S O at equivalence (mL)	I_2 adsorption capacity (mg g ^{1})
Zizyphis mauritiana	Concentration (%)	10	0.9	787.4
		20	0.4	914.4
		30	0.3	939.8
		40	0.2	965.2
		50	0.2	965.2
	Imprégnation rate (%)	24	0.2	965.2
		32	0.7	838.2
		40	0.5	889.0
		48	0.7	838.2
		56	0.4	914.4
	Imprégnation time (H)	3	0.55	876.3
		6	0.1	990.6
		9	0.3	939.8
		12	0.3	939.8
		15	0.35	927.1
	Pyrolysis temperature (°C)	300	1.6	609.6
		400	0,8	812.8
		500	0.4	914.4
		600	0.3	939.8
		700	0.3	939.8

Table 6 shows the iodine index results for activated carbons produced by activation with H_2SO_4.

Biomass	Parameters		Volume of Na S O at equivalence (mL)	I_2 adsorption capacity (mg g ^{1})
		10	2	508
		20	1.7	584.2

Zizyphis mauritiana	Concentration (%)	30	1.5	635
		40	1.2	711.2
		50	1.3	685.8
	Imprégnation rate (%)	24	1.2	711.2
		32	1.4	660.4
		40	1.1	736.6
		48	1	762
		56	1.2	711.2
	Imprégnation time (H)	3	1.1	736.6
		6	1.1	736.6
		9	1.1	736.6
		12	1	762
		15	1.2	711.2
	Pyrolysis temperature (°C)	300	2.2	457.2
		400	1.9	533.4
		500	1	762
		600	1.2	711.2
		700	0.5	889

BM index

The BM index, expressed in mg g^{-1}, represents the adsorption capacity of medium-sized molecules on activated carbons developed to evaluate mesopores and macropores. The results of the BM indices of activated carbons developed by H_3PO_4 activation are summarized in Table 7.

Table 7. BM adsorption capacity (mg.100g^{-1}) / H_3PO_4 activation

Biomass	Parameters		Absorbance of residual solutions	BM adsorption capacity	Rate (%)
Zizyphis mauritiana	Concentration (%)	10	0.097333333	623.6261701	88.42458668
		20	0.057333333	657.2146533	93.18713174
		30	0.087666667	631.7433869	89.57553507
		40	0.086333333	632.863003	89.73428657
		50	0.077666667	640.1405077	90.76617133
	Imprégnation rate (%)	24	0.071333333	645.4586842	91.52024097
		32	0.059	655.8151331	92.98869236
		40	0.082	636.5017553	90.25022895
		48	0.053666667	660.2935976	93.62369837
		56	0.045	667.5711022	94.65558314
	Imprégnation time (H)	3	0.048	665.051966	94.29839226
		6	0.046666667	666.1715821	94.45714376
		9	0.040333333	671.4897586	95.21121339
		12	0.065333333	650.4969566	92.23462273
		15	0.044666667	667.8510063	94.69527101
	Pyrolysis temperature (°C)	300	0.196666667	540.2147704	76.59759977
		400	0.070666667	646.0184922	91.59961672
		500	0.026666667	682.9658237	96.83841629
		600	0.016666667	691.3629444	98.02905256
		700	0.018	690.2433283	97.87030105

The results of the methylene blue indices of activated carbons produced by activation with H_2SO_4 are shown in Table 8.

Table 8. BM adsorption capacity (mg.100g^{-1}) /Activation

Biomass	Parameters	Absorbance of residual solutions	BM adsorption capacity	Extraction rate (%)

Zizyphis mauritiana	Concentration (%)	10	0.360666667	402.5019895	57.07116501
		20	0.105	617.1883775	87.51176554
		30	0.193333333	543.0138106	76.99447853
		40	0.136333333	590.8773991	83.78110524
		50	0.176666667	557.0090119	78.9788723
	Imprégnation rate (%)	24	0.079	639.0208916	90.60741983
		32	0.039333333	672.3294707	95.33027702
		40	0.14	587.7984548	83.34453861
		48	0.235333333	507.7459033	71.99380621
		56	0.143333333	584.9994145	82.94765985
	Imprégnation time (H)	3	0.042666667	669.5304304	94.93339826
		6	0.029333333	680.7265915	96.52091329
		9	0.051666667	661.9730217	93.86182562
		12	0.041	670.9299505	95.13183764
		15	0.162	569.3247891	80.72513882
	Pyrolysis temperature (°C)	300	0.187	548.3319871	77.74854816
		400	0.212333333	527.0592811	74.73226962
		500	0.072666667	644.3390681	91.36148947
		600	0.038666667	672.8892787	95.40965277
		700	0.040666667	671.2098546	95.17152552

IV. Discussion

The various tests carried out for the production of activated carbon yielded reproducible mass yields. These ranged from 17.3 to 56.6%. The mass yield results obtained (Tables 3 and 4) show that acid concentration, impregnation rate, impregnation time, and pyrolysis temperature are parameters that influence mass yields. Thus, the best yields are obtained at an impregnation time of 9 hours (56.6%) in the case of H_3PO_4 activation (Table 3). However, in the case of H_2SO_4, the best yields are obtained at a temperature of 300°C (46.5%) (Table 4). In general, the yield increases as the impregnation rate increases, while it decreases as the impregnation time and temperature increase. In the case of H_3PO_4, the mass yields are more reproducible compared to H_2SO_4 activation. This is consistent with the results found by [1,19] for the effect of orthophosphoric acid concentration. This could be explained by the fact that H_3PO_4 acid is an activating agent, thereby delaying the thermal decomposition of lignocellulosic biomass while limiting the loss of volatile matter, which would lead to the formation of a rigid carbon matrix [22]. In the case of activation by H_2SO_4, the yield decreases as the concentration increases. Overall, the yields of activated carbons prepared by H_2SO_4 activation (Table 4) are low compared to those obtained by H_3PO_4 activation (Table 3). Regardless of the activating agent considered, it can be seen that when the pyrolysis temperature is high, the yield decreases (Tables 3 and 4). Authors [23] made the same observations on ACs made from sorghum grains by activation with H_3PO_4. They explained this by the fact that when the pyrolysis temperature is high, the biomass degrades, producing a large amount of volatile matter and a small amount of activated carbon. This situation is a classic phenomenon in thermochemistry; under the effect of an increase in heat or the duration of its exposure, there is a greater loss of macromolecules (cellulose, hemicellulose, and lignin) that make up the biomass.

The iodine index results compiled in Tables 5 and 6 range from 457.2 to 990.6 mg g^{-1}. These results show that all the parameters studied have a real influence on the iodine indices. In general, the adsorption capacities of iodine I_2 increase with the concentration of the activating agent and the impregnation rate in the case of H_3PO_4 (Table 5). Activated carbons prepared by H_3PO_4 activation developed the best iodine adsorption capacities (990.6 and 965.2 mg g^{-1} respectively for an impregnation time of 6 hours and concentrations of 40 and 50%) (Table 3) compared to H_2SO_4 activation (Table 6). Several authors have shown that the iodine index increases with the concentration of H_3PO_4, as demonstrated by [24] for the activation of shea cake and cottonseed cake. For the impregnation rate, [12] studied the effect of the concentration of the activating agent H_3PO_4 on the I_2 adsorption capacity of CAs prepared from olive pits. He showed that the iodine index increases with the impregnation ratio. This was observed overall in this work. In the case of H_2SO_4 activation, the best iodine adsorption capacity (889 mg g ^{1}) is obtained at a pyrolysis temperature of 700 °C, followed by 762 mg g ^{1} for an impregnation rate of 48%, an impregnation time of 12 hours, and a pyrolysis temperature of 500 °C (Table 6). In both cases of activation, the increase in temperature significantly increases the adsorption capacity of I_2. These results confirm the work of [24]. The increase in temperature significantly promotes the development of micropores, leading to an increase in the adsorption capacity of activated carbons. Below 400°C, the pores are not well developed. However, above 600°C, some of the micropores formed are destroyed. This decrease in the number of micropores is accompanied by a significant reduction in adsorption capacity. Several studies in the literature have obtained similar results [25], for example, activated pistachios, coconut shells, coconut kernels, and date pits using orthophosphoric acid and observed an increase in the iodine index when the carbonization temperature was between 400 and 550°C. However, other research groups [17,26] have noted that the iodine index increases significantly as the temperature rises. We therefore believe that a temperature of 450°C would be more suitable for carbonization. SILEX INTERNATIONAL's technical

description of the quality criteria for activated carbon states that the iodine index must be greater than 950 mg/g [3]. Many of the carbons produced by H_3PO_4 activation have met this performance criterion. They would therefore have the ability to adsorb small molecules such as those responsible for tastes and odors [5]. These results are similar to those obtained by Siragi et al. in[1,19].

The results of the MB indices obtained for the different activated carbons in this study are summarized in Tables 7 and 8. They indicate the adsorption capacities and MB extraction rates. The results obtained show that the methylene blue indices vary from 402.5019 to 691.3629 mg.100 g^{-1} for H_2SO_4 and H_3PO_4. These results correspond to a discoloration of the methylene blue solution with rates ranging from 57.07% to 98.02% for a concentration of 10% H_2SO_4 (Table 7) and a pyrolysis temperature of 600°C (Table 8). Activated carbons prepared by activation with H_3PO_4 acid develop better adsorption capacities under the operating conditions used (Tables 7 and 8). In general, the BM adsorption capacity developed by activated carbons increases with the concentration of the activating agent, the impregnation time, and the pyrolysis temperature; the maximum values are obtained at a temperature of 600 °C. The activated carbon that developed the best BM adsorption capacity was obtained with 25% H_3PO_4 and a pyrolysis temperature of 600°C. Similar results have been reported in the literature by [27] on activated carbon obtained from bitter almond shells and by [12] on activated carbon obtained from olive pits. It is reported that the Methylene Blue index is a parameter that can be used to evaluate the mesopores (2 nm < diameter < 50 nm) and/or macropores (diameter > 50 nm) necessary for the adsorption of medium-sized molecules [17,28]. In the case of activation by H SO , the BM adsorption capacity values obtained are relatively low compared to those obtained by activation with H PO (Tables 7 and 8).

V. Conclusion

At the end of this work, the following information can be retained:

- The various tests carried out for the production of activated carbon yielded reproducible mass yields. These vary from 17.3 to 56.6% ;
- The best yields were obtained at an impregnation time of 9 hours (56.6%) in the case of H_3PO_4 activation ;
- In the case of H_3PO_4, the mass yields are more reproducible than in the case of H_2SO_4 activation ;
- Regardless of the activating agent considered, it is noted that when the pyrolysis temperature is high, the yield decreases ;
- All the parameters studied have a real influence on the iodine indices. In general, the adsorption capacities of iodine I_2 increase with the concentration of the activating agent and the impregnation rate in the case of H_3PO_4 ;
- Activated carbons prepared by H_3PO_4 activation developed the best iodine adsorption capacities (990.6 and 965.2 mg g^{-1} for impregnation times of 6 hours and concentrations of 40 and 50%, respectively) compared to H_2SO_4 activation ;
- A temperature of 450°C would be more suitable for carbonization ;
- Many of the carbons produced by H_3PO_4 activation met this performance criterion ;
- The results obtained show that the methylene blue indices vary from 402.5019 to 691.3629 mg.100 g ¹ for H_2SO_4 and H_3PO_4 ;
- Methylene blue solution decolorization rates vary between 57.07% and 98.02% for a concentration of 10% H_2SO_4 and a pyrolysis temperature of 600°C ;
- The activated carbon with the best BM adsorption capacity was obtained with 25% H_3PO_4 and a pyrolysis temperature of 600°C.

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