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OPTIMIZATION OF ACTIVATED CARBON PRODUCTION FROM *BALANITES AEGYPTIACA* TRUNK FOR THE REMOVAL OF IODINE AND METHYLENE BLUE IN AQUEOUS SOLUTION

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ABSTRACT

This work focuses on optimizing the production of activated carbons (ACs) produced by chemical activation with orthophosphoric acid (H_3PO_4) from the trunk of *Balanites aegyptiaca (BA),* for use in the removal of iodine and methylene blue (BM) from aqueous solutions. Eleven (11) varieties of activated carbon were produced by varying the H_3PO_4 concentration (20, 25, and 30%), the impregnation ratio (24, 32, and 48%), the impregnation time (12, 16, and 24 h), and the pyrolysis temperature (400, 450, 500, and 600 °C). The ACs were characterized by pyrolysis yield, iodine value, methylene blue value, and textural analysis. The results show high pyrolysis yields (57.38 to 77.71%), iodine values between 934.19 and 987.14 mg/g, and BM adsorption capacities ranging from 611.83 to 617.01 mg/100 g. CAE-BA-25%-450°C-12h activated carbon exhibits the best iodine value (987.14 mg/g), while CAE-BA-25%-450°C-16h develops the best BET specific surface area (393.6 m^2/g) and the best BM adsorption capacity (617.01 mg/100 g).

Keywords: *Balanites Aegyptiaca*, Activated Carbon, Chemical Activation, Iodine Value, Methylene Blue, Optimization.

1 INTRODUCTION

The rapid increase in the world's population and the intensification of industrial activities have led to an exponential demand for water and, simultaneously, to increasing contamination of water resources. The textile, food processing, cosmetics, and paper industries are among the largest consumers of water and generate effluents laden with organic dyes and micropollutants, representing a major environmental challenge [**1**, **2**]. These organic dyes not only alter the color of the water but also hinder the penetration of sunlight, severely disrupting aquatic ecosystems [**3**].

Among the pollution control techniques developed sedimentation, filtration, reverse osmosis, oxidation, photocatalytic degradation, ion exchange, etc. adsorption on to activated carbon remains the most effective and economical method commonly used for removing organic and inorganic pollutants in solution. This success is due to the large specific surface area, surface chemistry, and pore size distribution of activated carbons [**4**]. The global activated carbon market has experienced exponential growth, increasing from $3.2 billion in 2020 to a projected $5.8 billion by 2030 [**5**].

However, commercial activated carbons derived from coal, animal bones, or apricot kernels remain expensive. Therefore, the development of low-cost activated carbons from local lignocellulosic biomass represents a sustainable and promising alternative. Various raw materials have been investigated in this regard: *Balanites aegyptiaca kernel shells*, Jatropha wood, coconut shells, olive pits, and cassava peels, among others [**6**, **7**].

Balanites *aegyptiaca* (BA), commonly known as "Adoua" in Niger, is a tree in the Zygophyllaceae family (*Balanites aegyptiaca (L.) Del. Zygophyllaceae*), widely distributed in the Sahelian and semi-arid regions of Africa and Asia. The kernel shell of this tree has already been studied in the field of adsorption [**8**, **9**]. The trunk, available in large quantities as wood waste, represents a little-explored lignocellulosic precursor for the production of activated carbon.

The present work aims to optimize the production conditions of activated carbons from the trunk of *Balanites aegyptiaca* by chemical activation with H_3PO_4, by evaluating the influence of the operating parameters (concentration of activating agent, ratio and time of impregnation, pyrolysis temperature) on the pyrolysis yield, the iodine value and the methylene blue value, in order to identify the optimal production conditions.

Figure 1 Wood of the trunk of Balanites aegyptiaca before and after crushing

2 MATERIALS AND METHODS

2.1 Collection and pretreatment of biomass

The wood from the trunk of *Balanites aegyptiaca* was collected at the Faculty of Agronomy (FA) of Abdou Moumouni University (UAM) in Niamey, Niger. The raw material was thoroughly washed with tap water and then oven-dried at 105 °C for 24 hours. It was then ground to particles smaller than 2 mm and dried again at 105 °C for 24 hours.

2.2 Immediate analysis of biomass

Immediate analysis of biomass consists of determining the moisture, ash, volatile matter, and fixed carbon content. In this work, the immediate analysis was carried out according to the AFNOR XP CEN/TS 14775 (ash), AFNOR XP CEN/TS 14774-3 (moisture), and XP CEN/TS 15148 (volatile matter) and fixed carbon standards.

The actual biomass density was also determined using the test tube method. The immediate analysis was performed with particles smaller than 200 µm.

2.2.1 Humidity level

Porcelain crucibles were fired in a kiln at 1000 °C for 45 minutes. After cooling, the crucibles were left in a desiccator for 15 minutes. The mass **m** of the crucibles was weighed using a precision balance. A 2 g sample of biomass was then added to the crucibles, and the mass $\mathbf{m_1}$ was recorded. The entire assembly (crucible + biomass) was then placed in an oven at 105 °C for 24 h, and the mass $\mathbf{m_2}$ was weighed after cooling. The moisture content is given by equation 1:

$$(TH\%) = \frac{m_1 - m_2}{m_1 - m} \times 100 \qquad (\mathbf{1})$$

2.2.2 Volatile substances (loss on ignition)

, were heated in a furnace at 900 °C for seven (7) minutes. The mass $\mathbf{m^3}$ was weighed after these crucibles had cooled. Equation 2 gives the volatile matter content:

$$(MV\%) = \frac{m_2 - m_3}{m_2 - m} \times 100 \qquad (\mathbf{2})$$

2.2.3 Ash content

The ash content was determined by weighing the mass $\mathbf{M_1}$ **of** the empty crucible into which 1 g of biomass was introduced, then the **mass** $\mathbf{M_2}$ is noted. The whole thing was heated in a kiln at 800 °C until it turned to ash. The mass $\mathbf{M_3}$ was noted after the crucible had cooled. Equation 3 gives the ash content of the biomass:

$$(TC\%) = \frac{M_3 - M_1}{M_2 - M_1} \times 100 \qquad (\mathbf{3})$$

2.2.4 Fixed carbon rate

The fixed carbon content was determined by summing the percentages of moisture, volatile matter, and ash removed from 100. Fixed carbon is determined from equation 4:

$$(CF\%) = 100 - [(TH\%) + (MV\%) + (TC\%)] \qquad (\mathbf{4})$$

2.2.5 Actual density

The actual density ($\mathbf{g/cm^3}$) was determined by weighing the mass $\mathbf{m_1}$ of an empty 50 cm^3 test tube. The test tube was then filled with biomass, and the mass $\mathbf{m_2}$ **of the entire mixture was recorded. Equation 5 gives the** value of the actual density:

$$\rho = \frac{m_2 - m_1}{V} \qquad (\mathbf{5})$$

2.3 Production of activated carbons by chemical activation with H_3PO_4

Chemical activation with orthophosphoric acid (H_3PO_4) was carried out in two stages: impregnation of the biomass and pyrolysis of the impregnated material.

Solutions of 20%, 25%, and 30% H_3PO_4 were prepared by diluting an 85% stock solution. For each test, 15 g of pretreated biomass were mixed with 100 mL of the H_3PO_4 solution. The mixture was kept under magnetic stirring for 24 h at room temperature, then filtered and oven-dried at 105 ° C for 24 h. The operating parameters studied are presented in Table 1.

Pyrolysis was carried out in a muffle furnace (VOLCA MC 18 type, PROLABO) at 450 °C, with a heating rate of 10 °C/min and an isothermal holding period of 1 hour 30 minutes. After pyrolysis, the prepared activated carbons (ACs) were washed with distilled water, dried at 105 °C for 24 hours, and then stored in airtight containers.

Table 1 Parameters for optimizing the production of activated carbons

Biomass	Various parameter	Values tested
	H_3PO_4 concentration (%)	20, 25, 30
Balanites aegyptiaca	Impregnation ratio (%)	24, 32, 48
	Impregnation time (h)	12, 16
	Pyrolysis temperature (°C)	400, 500, 600

2.4 Pyrolysis efficiency

The pyrolysis efficiency (R) was calculated using the following formula:

$$R\ (\%) = \frac{\text{masse finale}}{\text{masse initiale}} x\ 100 \qquad (\mathbf{6})$$

2.5 Determination of the iodine value

The iodine value indicates the microporosity of activated carbon. It is defined as the amount of iodine (in mg) adsorbed per gram of activated carbon when the residual iodine concentration is 0.02 N. The method used is an adaptation of the CEFIC standard (1989) and the AWWA B 600-78 standard. The iodine adsorption capacity is given by:

$$Q_{I_2}(mg/g) = \frac{\left(C_0 - \frac{C_{thio} V_{thio}}{2V_{I_2}}\right) M_{I_2} x V_{ads}}{m_{CA}} \qquad (\mathbf{7})$$

With:

Q_{I_2} adsorption capacity or I_2 index ($en\ mg/g$);
C_0: concentration of the initial I_2 solution (in mol/L);
C_{thio}: the concentration of $Na_2S_2O_3$ (in mol/L);
V_{thio}: volume of $Na_2S_2O_3$ at equivalence (in mL);
V_{I_2}: the volume of iodine measured (in ml);
M_{I_2}: the molar mass of I_2 (in g/mol);
V_{ads}: the adsorption volume (in mL);
m_{CA}: mass of activated carbon used (in g).

2.6 Determination of the methylene blue index

The methylene blue (MB) test is used to evaluate the mesoporosity and macroporosity of CA. A test solution of 1.944×10^{-5} mol/L was prepared from a stock solution of BM at 0.12 g/100 mL of distilled water. 100 mg of CA were mixed with 100 mL of this solution for 20 min and then filtered. The absorbance of the filtrate was measured at 662 nm by UV-Visible spectrophotometry. The adsorption capacity was calculated by:

$$Q_{BM} = \frac{(C_0 - C_r)\ x\ M\ x\ V}{m_{CA}} \qquad (\mathbf{8})$$

With:

Q_{BM}: CA adsorption capacity (in mg/g);
C_0: initial concentration of the MB solution (in mol/L);
C_r: residual concentration of the BM solution (in mol/L);
V: volume of the BM solution (in mL);
M: molar mass of BM;
m_{CA}: mass of adsorbent used (in mg)

2.7 Textural analysis (BET, BJH, DR)

Specific surface areas, volumes, and pore diameters were determined by nitrogen adsorption-desorption at 77 K, using the BET (Brunauer - Emmett -Teller), BJH (Barrett -Joyner - Halenda), and Dubinin-Radushkevich (DR) methods. These analyses were performed at the University of Zaria, Nigeria.

3 RESULTS AND DISCUSSION

3.1 Immediate biomass analysis

The immediate analysis revealed results grouped in Figure 2:

These results show a moisture content of 2.99%, an ash content of 1.09%, a volatile matter content of 85.69%, and a fixed carbon content of 10.23%. The actual biomass density was measured at 0.388 g/cm^3. However, the moisture content and ash content are the factors that influence the adsorption properties of activated carbon [**10]**. Thus, Gumus et *al.,* [**11**] reported that the moisture content should not exceed 6%, and the work of Sunanda et *al.,* [**12**] showed that

the ash content ranges from 1% to 20% depending on the nature of the precursor. Indeed, a high ash content decreases the specific surface area. This is explained by the simple fact that ash represents an inactive filler that reduces the performance of an adsorbent by clogging pores [**13**]. Thus, the lower the ash content, the better the CA [**14**].

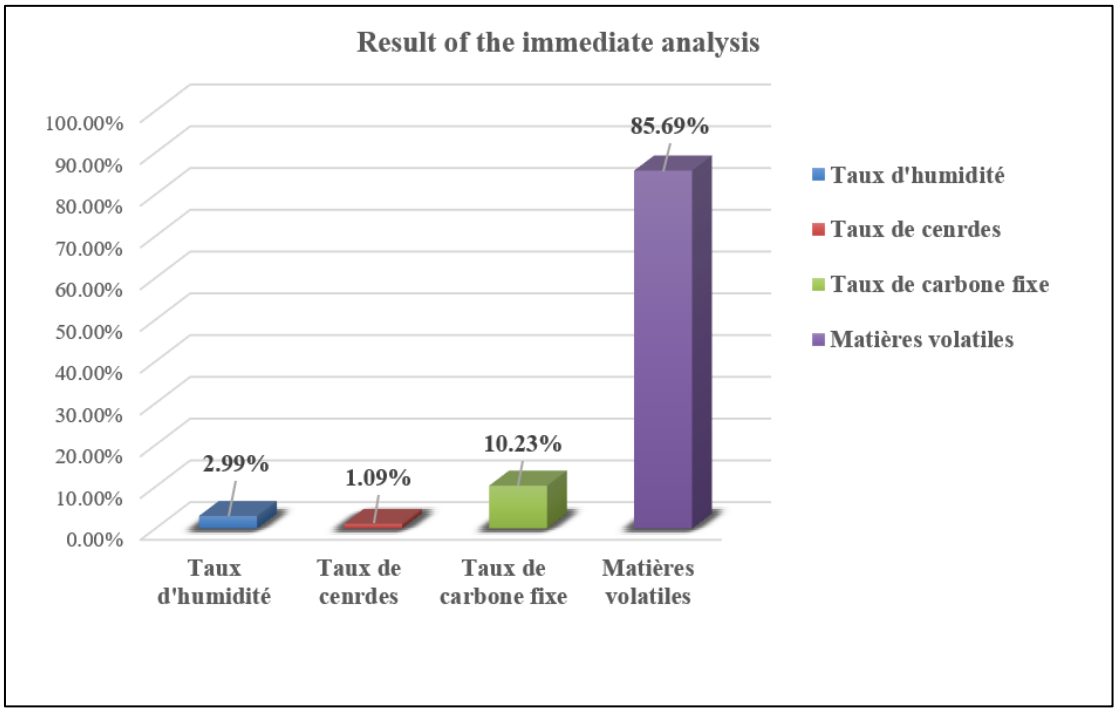

Figure 2 Result of the immediate analysis

3.2 Pyrolysis yield of processed activated carbons

The pyrolysis yield results for the different CAEs are presented in Table 2. The values obtained range from 57.38% to 77.71%, reflecting the high carbon retention during carbonization and confirming the suitability of *Balanites aegyptiaca trunk* as a precursor. These high yields can be attributed to the precursor's high lignin content, a thermally very stable compound.

Table 2 Pyrolysis yields of the activated carbons produced

Prepared Activated Carbon (CAE)	Pyrolysis efficiency (%)
CAE-BA-20%-450°C	63.02
CAE-BA-25%-450°C	57.38
CAE-BA-30%-450°C	70.07
CAE-BA-25%-450°C (ratio 24%)	66.17
CAE-BA-25%-450°C (ratio 32%)	65.57
CAE-BA-25%-450°C (ratio 48%)	66.04
CAE-BA-25%-450°C-12h	77.71 (max)
CAE-BA-25%-450°C-16h	67.11
CAE-BA-25%-400°C	76.12
CAE-BA-25%-500°C	73.78
CAE-BA-25%-600°C	65.29

Analysis of the results highlights that the concentration of the activating agent, the impregnation time, and the pyrolysis temperature significantly influence the yield. A decrease is observed at a H_3PO_4 concentration of 25 %, corresponding to an optimal concentration that promotes activation reactions and induces greater mass loss. The yield also decreases with increasing impregnation time (from 77.71% to 67.11%), due to increased diffusion of the activating agent within

the biomass structure, intensifying the production of volatile compounds. Similarly, an increase in the pyrolysis temperature reduces the yield (from 76.12% to 65.29%) through more extensive thermochemical degradation of lignocellulosic macromolecules. These trends are consistent with results reported in the literature for similar biomasses [**15**, **16**].

3.3 Iodine Index

The iodine index is an indicator of the microporosity of activated carbons. The results obtained (Table 3) show that all CAEs have high iodine indices, ranging from 934.19 to 987.14 mg/g, with the exception of CAE-BA-20%-450°C (934.19 mg/g) which is slightly below the SILEX INTERNATIONAL quality threshold set at 950 mg/g.

Table 3 Iodine Values of Processed Activated Carbons

Prepared Activated Carbon (CAE)	Iodine value (mg/g)
CAE-BA-20%-450°C	934.19
CAE-BA-25%-450°C	960.87
CAE-BA-30%-450°C	970.62
CAE-BA-25%-450°C (ratio 24%)	981.76
CAE-BA-25%-450°C (ratio 32%)	972.55
CAE-BA-25%-450°C (ratio 48%)	963.27
CAE-BA-25%-450°C-12h	987.14 (max)
CAE-BA-25%-450°C-16h	981.27
CAE-BA-25%-400°C	982.25
CAE-BA-25%-500°C	969.17
CAE-BA-25%-600°C	962.79

Increasing the H_3PO_4 concentration from 20 % to 30% leads to a rise in the iodine value (from 934.19 to 970.62 mg/g), confirming the role of the activating agent in the development of microporosity, in accordance with the work of Gueye [**7**] and Sanda [**8**]. Conversely, prolonged impregnation time and high pyrolysis temperatures tend to reduce the iodine value. Ceyhan et *al.,* [**17**] showed that excessive impregnation time can enlarge micropores, reducing their active proportion. Similarly, higher temperatures favor the formation of mesopores at the expense of micropores [**18**]. The maximum value of 987.14 mg/g was obtained with CAE-BA-25%-450°C-12h, identifying this CA as the most microporous among the samples studied. This CA is particularly suitable for the removal of small molecules.

3.4 Methylene blue index

The methylene blue index indicates the adsorption capacity of the meso- and macropores. The results presented in Table 4 show high and relatively similar adsorption capacities, ranging from 611.83 to 617.01 mg/100 g, corresponding to removal rates of 98.40 to 99.23%.

Table 4 Adsorption capacities of methylene blue in the prepared activated carbons

Prepared Activated Carbon (CAE)	Adsorption capacity BM (mg/100g)
CAE-BA-20%-450°C	613.73
CAE-BA-25%-450°C	615.81
CAE-BA-30%-450°C	615.63
CAE-BA-25%-450°C (ratio 24%)	611.83
CAE-BA-25%-450°C (ratio 32%)	614.94
CAE-BA-25%-450°C (ratio 48%)	616.32
CAE-BA-25%-450°C-12h	616.67

CAE-BA-25%-450°C-16h	617.01 (max)
CAE-BA-25%-400°C	615.47
CAE-BA-25%-500°C	616.67
CAE-BA-25%-600°C	613.73

The adsorption capacity of BM increases proportionally with the H_3PO_4 concentration, the impregnation ratio, and the impregnation time, confirming the observations of Muhammad et *al.,* [**19**] and Ridzuan et *al.,* [**20**]. However, excessively high pyrolysis temperatures tend to reduce this capacity, probably due to partial destruction of the meso-macroporous porous matrix. A notable value of 616.67 mg/100 g is obtained at 500 °C. The maximum capacity of 617.01 mg/100 g is achieved with CAE-BA-25%-450 °C-16 h, corresponding to a BM removal rate of 99.23%. It also exhibits the best BET surface area (393.6 m^2/g) and the best mesoporous volumes. This CA is optimal for the adsorption of medium and large molecules, such as dyes.

3.5 Textural analysis of activated carbons

Table 5 presents the specific surface areas and pore volumes of the main samples, determined by different methods. The results show a progressive improvement in textural characteristics throughout the processing stages.

Table 5 Specific surface areas of the main samples (m^2/g)

Sample	**BET (m^2/g)**	**Langmuir (m^2/g)**	**BJH (m^2/g)**	**DR (m^2/g)**
BA-brute	173.1	404	229.4	215.5
Non-pyrolyzed impregnated BA	196.6	1406	218.0	207.8
BA-brute pyrolyzed	218.2	776.8	263.1	243.8
CAE-BA-25%-450°C	282.6	986.6	350.0	323.8
CAE-BA-25%-450°C-16h	393.6	880.2	509.5	500.7

The specific surface area of BET increases from 173.1 m^2/g for raw BA to 393.6 m^2/g for CAE-BA-25%-450°C-16h, representing a 127% increase. Impregnation slightly increases the BET surface area (196.6 m^2/g), but pyrolysis leads to a significant increase through the elimination of volatile compounds and the development of porosity. Chemical activation at 25% followed by pyrolysis at 450°C with an impregnation time of 16 h results in an improvement in all specific surface areas. The BJH surface area increases from 229.4 m^2/g (raw BA) to 509.5 m^2/g for CAE-BA-25%-450°C-16h, confirming the excellent mesoporous development. The BET and DR models exhibit very satisfactory correlation coefficients (R^2> 0.98), indicating adsorption dominated by multilayer and microporous filling. These results are consistent with the work of Lua and Yang [**21**] on the carbonization of plant biomass.

4 CONCLUSION

This study demonstrated the potential of using *Balanites aegyptiaca* trunk as *a* lignocellulosic precursor for the production of high-performance activated carbons by chemical activation with orthophosphoric acid (H_3PO_4). The results of the immediate analysis (low ash content of 1.09%, high volatile matter content of 85.69%) confirm the suitability of this local biomass for producing high-quality activated carbons. Among the eleven varieties of activated carbons, CAE-BA-25%-450°C-12h and CAE-BA-25%-450°C-16h also exhibited satisfactory pyrolysis yields (77.71% and 67.11%, respectively) and improved iodine and methylene blue values (987.14 mg/g and 617.01 mg/100g, respectively).

Looking ahead, studies on the application of these CAs for the removal of other micropollutants (heavy metals, pesticides, pharmaceuticals) as well as comparison with other adsorbents are envisaged.

STATEMENTS ON COMPLIANCE WITH ETHICAL STANDARDS

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Disclosure of conflict of interest

The authors declare no conflicts of interest regarding this manuscript.

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