Preparation of Activated Carbons based *Balanites Aegyptiaca* Shells by Chemical Activation: Optimization Conditions Using the Methodology of Experimental Design

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ABSTRACT

The optimization conditions of preparation of activated carbons based *Balanites aegyptiaca* shells by chemical activation was investigated. The effects of three parameters of preparation namely, the activation temperature (600-800 °C), impregnation ratio (1:4-3:8) and residence time (60-120 min) were thoroughly studied on the activated carbon yield (Y_{ld} , Y_1), iodine number (ION , Y_2) and methylene blue number (MBN , Y_3) using the Methodology of Experimental Design (MED). The analysis of variance (ANOVA) under the experimental domain revealed that, the activation temperature of 800 °C, residence time of 02hrs and impregnation ratio of 1:2 were the optimum conditions of preparation leading to activated carbon yield of 23.0%, iodine number of 889.0 mg/g and methylene blue number of 9.7 mg/g. The polynomial equation showed that the three parameters were both synergetic and antagonistic on the responses retained. The higher values of iodine numbers obtained alongside the experimental matrix is an indication that the activated carbons so prepared were mainly microporous.

Keywords: Activated carbon, Adsorption, ANOVA, *Balanites aegyptiaca*, Optimization.

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I. INTRODUCTION

The management of huge amount of wastes is still a matter of concern to population confronted to these bigger discharges. This consequently caused environmental problem due to their accumulation which of course creates damages such as air, soils, and water pollution [1], [2]. The problem is more visible for the bigger producers of wastes like

agricultural industries of products transformation. For this reason, more interest is given to the valorization of these wastes which are produced and discharge in the environment [3]. It is interesting to know that the valorization does not consider these agricultural by-products like wastes properly which must be throw away, but consider them as a precursor to be exploited and gives an added value. The access to combustible for cooking purpose is still another problem

faced by many countries, these populations used agricultural residues like *Balanites aegyptiaca* shells as a source of energy, which of course favorite the production of green gas which are not good for the ozone layer. Therefore, it is advised to put in place ecological and economical techniques which are capable to valorize these wastes as adsorbents materials with high added value [3], [4]. The necessity to find sustainable adsorbents with zero damage to the environment are the economic and environmental challenges face by researchers nowadays. Thus, the elaboration of activated carbons based agricultural residues seems to be a good and right solution, since the activated carbons demand is still increasing around the world [5]. The conversion of agricultural residues into activated carbons is a way of valorization which has been proof and recognized industrially because applied to many residues such as mangoes seeds [6], rice husk, nuts shells, coconut shells [7], oil palm shells [3, 4, 8]; palm kernel shells [9], cola nut shells [10], [11]. The *Balanites aegyptiaca* residues are agricultural by-products that are used in reason of their lignocellulosic nature. *Balanites aegyptiaca* is a specie particularly abundant and available in sahelian regions and which have important utilization. Their fruits are used as foods for human beings and animals. All the parts of the *Balanites aegyptiaca* tree are used in traditional medicine for the treatment of many diseases like psychoses, epilepsy, rheumatisms, coughs, and rate [12]. This plant has been more studied in the domain of foods for human beings [13], animals [14] and also in the medical domain [15]. As, *Balanites aegyptiaca* shells are available and abundant and discharge in the environment, it was interesting, to find whether they can be converted into activated carbons for more used. Primarily, it is necessary for the production of activated carbons of good quality, to have deep knowledge on the precursor which is the starting material, particularly their physicochemical properties [3], [5]. The present work deals with the valorization of endocarp of the *Balanites aegyptiaca* fruits into activated carbons by chemical activation. As the literature well described, there are many methods of production of activated carbons such as physical activation, chemical activation even physicochemical activation [3], [5], [9], [10]. But amongst the aforementioned method, the chemical activation has the merit to create important porosity and avoid consumption of more energy compared to the other two methods [2]-[4]. The *Balanites aegyptiaca* is a ligneous plant which grows under various ecological conditions (100 mm to 1000 mm of rains per year), and it is abundant in arid and semi-arid regions in tropical Africa [16], this availability is one of the criteria of choice of activated carbons precursors [3]-[5]. Even though the *Balanites aegyptiaca* is consider as a versatile plant because of its various utilizations as showed above, and considering the fact that, researches are growing to find out good precursors for production of activated carbons, there are not much studies dealing with the utilization of *Balanites aegyptiaca* for the production of activated carbons as far as our knowledge is concerned [...]. Thus, the aim of this work is to use initial analysis like iodine and methylene blue numbers to show that, the *Balanites aegyptiaca* can be used for the production of activated carbons. To achieve this goal,

the methodology of experimental design which was used in literature for similar purpose [3]-[6], [17], [18] was deeply used in this work to investigate and find out the optimum conditions of preparation of outstanding activated carbons based *Balanites aegyptiaca* shells.

II. MATERIALS AND METHODS

A. Sample Collection

The *Balanites aegyptiaca* residues were sampled by an association called Tiipaalga in Burkina Faso. This association collected the *Balanites aegyptiaca* fruits in order to use the seeds for the oil production. The *Balanites aegyptiaca* shells were obtained by sampling the fruits directly on the tree and also by collecting the shells that animals leave after eating and digesting the seeds. The *Balanites aegyptiaca* shells were washed, dried, grinded and sieved into particles sizes ranged between 1.00–3.15 mm.

B. Experimental Methods

10.0 g of KOH were dissolved in 250 mL conical flasks with distilled water. In another 500 mL conical flask, the prepared solution of KOH and a known mass of *Balanites aegyptiaca* shells at the desired impregnation ratio (v:w) were mixed. The mixture was shaken during 2 hours and at the temperature of 25 °C. Once the time of impregnation was reached, the mixture was filtrated and dried in an oven overnight. After, the impregnation step, the activation process was done in a Carbolite furnace at the pyrolysis temperature varying between 600–800 °C under nitrogen gas flow for a residence time ranging from 60 to 120 min. When the activation process was achieved, the activated carbons obtained were washed with a 0.1 N hydrochloric acid solution, followed with distilled water until the pH range of 6–7. The obtained activated carbons based *Balanites aegyptiaca* shells were dried at 105 °C for 24 hours. Finally, after drying, the samples were grinded and sieved to the particles size less than 0.212 mm.

TABLE 1: THE FACTORS THAT AFFECT THE PREPARATION OF ACTIVATED CARBON AND THEIR DIFFERENT LEVELS

Factor	Low level value		High level value
X ₁ : Activation temperature (°C)	600	700	800
X ₂ : Residence time (min)	60	90	120
X ₃ : Impregnation ratio	1/4	3/8	1/2

C. Methodology of Experimental Design

The Methodology of Experimental Design (MED) is a statistical technique for modeling and analysis of problems in which a response of interest is influenced by several factors [3], [4], [18]. The MED is utilized to optimize the effective parameters with a minimum number of experiments, as well as to analyze the interactions between the parameters [3], [4]. The three factors investigated in the present work (Table1) were coded as the activation temperature (X₁), impregnation ratio (X₂) and the residence time (X₃). The three responses analyzed were the activated carbons yield, Yld (%); iodine

number, ION (mg/g); and methylene blue number, MBN (mg/g). Each response was used to develop a model which correlates the responses to the three coded factors using a polynomial equation given by the following equation:

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X_2 + a_{13}X_1X_3 + a_{23}X_2X_3 \quad (1)$$

where, Y is the predicted response, a_0 is a constant coefficient, a_i is a linear coefficient; a_{ij} is an interaction coefficient; X_1 , X_2 and X_3 , are the coded values of the factors. The Full Factorial Design (FFD) using three factors was used to generate an experimental matrix of twelve experiments including four experiments at the center of the studied domain. The experiments at the center points were used to determine the experimental error and to verify the reproducibility of experimental data. This FFD has been chosen, because it helps to observe the influence of different factors on the responses studied. The experimental data were analyzed using a statistical software design expert named NEMROD, for regression analysis to fit the equations developed and also to evaluate the statistical significance of the equations obtained.

D. Activated Carbons Characterizations

Prior characterizations of activated carbons consisted of the determination of the yield, iodine, and methylene blue numbers. Of course, the sample of activated carbon have been grinded in the porcelain mortar and sieved at the particles sieved of 212 μm as described above.

1. The activated carbons yield

The determination of the activated carbons yield helps to estimate the quantities of activated carbons that can be obtained from an initial mass of precursor. Practically, it is to determine the ratio of the weight percent of carbon obtained after pyrolysis of the initial mass of precursor used. It is calculated from the following formula:

$$R = \frac{m}{m_0} \times 100 \quad (2)$$

where,

m and m_0 are the mass of carbon obtained after pyrolysis and the mass of initial biomass used respectively (g).

2. Iodine number

Iodine number is a test which gives an indication on the microporosity of activated carbons, in another words, the measurements of the capacity to adsorb molecules of small sizes (less than 10 Å) [19]. Iodine was considered as probe molecule for assessing the adsorption capacity of adsorbent for solutes of molecular sizes <10 Å. The iodine number is defined as the milligrams of iodine adsorbed by 1g of carbon. The iodine adsorption was determined using the sodium thiosulfate volumetric method (ASTM D4607-94, 2006) [20]. The iodine number was estimated by mixing the activated carbon with 0.02 N iodine solution shaken occasionally and then by titration of the solution against $\text{Na}_2\text{S}_2\text{O}_3$ as well described in our earlier work [3]. The iodine number, ION (mg/g) is calculated from the following formula:

$$\text{ION} = \frac{25.4 \times (20 - V_{\text{thio}})}{m_{\text{AC}}} \quad (3)$$

where,

V_{thio} is the volume of $\text{Na}_2\text{S}_2\text{O}_3$ solution (mL).

m_{CA} is the mass activated carbon (mg).

3. Methylene blue number

This index gives an indication on the capacity of activated carbon to adsorb molecules medium and large sizes (between 15-28 Å) [19]. Methylene blue (MB) was considered as probe molecule for assessing the adsorption capacity of adsorbent for solutes of molecular sizes >15 Å. The methylene blue number is the milligrams of methylene blue adsorbed by 1g of carbon [21]. The concentration of methylene blue was measured by using a beam UV-visible spectrophotometer Miltonroy Company Spectronic 20D at the maximum absorbance wavelength of 664 nm. The methylene blue number MBN (mg/g) was calculated using the following formulae:

$$MBN = \frac{(C_0 - C_f)}{m_{\text{AC}}} \times V \quad (4)$$

where,

V is the volume of methylene blue solution de (mL).

C_0 and C_f are initial and final concentration of the MB solution (mg/L) respectively.

m_{CA} is the mass activated carbon (mg) in contact with methylene blue solution.

III. RESULTS AND DISCUSSION

A. Immediate Analysis of *Balanites aegyptiaca* Shells

The aim of this immediate analysis was to determine the contents of main constituents of *Balanites aegyptiaca* shells used. The results of different analysis are compiled in the Table 2. The results of immediate analysis revealed that *Balanites aegyptiaca* shells have a low moisture and ash contents but, have high content in fixed carbon and volatiles matters. Additionally, the *Balanites aegyptiaca* shells revealed their character of lignocellulosic material according to the lignin, cellulose and hemicellulose contents. Thus, their thermochemistry valorization in activated carbons is interesting compared to others works in the literature [3], [4], [7], [11], [18].

TABLE 2: IMMEDIATE ANALYSIS OF MAIN CONSTITUENTS OF *BALANITES AEGYPTIACA* SHELLS

Components	Weight percent (%)
Moisture	4.86
Volatiles matters	80.19
Ash	4.21
fixed carbon	15.60
Extract	9.66
Hemicellulose	33.88
Lignin	15.17
Cellulose	41.29

B. Analysis of the Experimental Design

The combination of different factors is provided by the experimental design, such that the observation of influence (inner and interaction effects) of these different factors on the

three responses studied and the determination of optimum condition of preparation of activated carbons be possible. The experiments done help to determine the responses. The results obtained after execution of the matrix are interpreted using the ANOVA. The model predicted, is analyzed by the values of correlation coefficients. The indicators of validity of the model are R^2 and R^2 -adjusted which values must be closed to unity to confirm the fitting of the model postulated. For the polynomial equation given the different responses in function of the factors investigated, a positive sign in front of the coefficients indicates synergistic effects, whereas negative sign indicates antagonistic effects.

TABLE 3: EXPERIMENTAL MATRIX AND THE DIFFERENT RESPONSES OBTAINED

N°	Activation temperature (X_1)	Residence time (X_2)	Impregnation ratio (X_3)	Y_1 (%)	Y_2 (mg/g)	Y_3 (mg/g)
1	600	120	1:2	26.55	165.10	3.03
2	800	120	1:4	22.51	774.70	9.81
3	800	60	1:2	24.93	476.25	4.95
4	600	60	1:2	27.64	209.55	2.84
5	800	60	1:4	23.47	527.05	3.49
6	600	120	1:4	25.12	215.90	4.18
7	600	60	1:4	26.12	177.80	3.63
8	800	120	1:2	23.00	889.00	9.70
9	700	90	3:8	25.35	374.65	4.01
10	700	90	3:8	25.31	488.95	3.74
11	700	90	3:8	24.60	361.95	5.55
12	700	90	3:8	24.18	374.65	6.55

Y_1 = response of activated carbon yield.

Y_2 = response of iodine number.

Y_3 = response of methylene blue number.

The analysis of the Table 3 above, showed that the activated carbons yield varied from 22.51 to 27.64%. The iodine numbers varied from 165.10 to 889.00 mg/g and the methylene blue from 2.84 to 9.81 mg/g. It can be notice that the higher values of activated carbons yield are obtained for the activation temperature of less than 600 °C and residence time less than 60 min, whereas, the lower values of activated carbon yield are obtained at 800 °C and 120 min for activation temperature and residence time respectively. This can be explained by the fact that, there are rupture of chemical bonds in the biomass network with the increase of activation temperature, which conducted to the release of volatiles matters and thus reducing progressively the mass of the samples. The similar trends have been observed in our earlier works [3], [4]. The longer the residence time the higher the time of reaction. The higher values of iodine and methylene blue numbers are obtained for higher activation temperatures and residence time (800 °C and 120 min) whereas, the lower values iodine and methylene blues are obtained at activation temperatures and time of residence which are lower (600 °C and 60 min). In fact, the increase of activation temperature conducts to the opening and the widening of pores, which consequently increase the adsorption iodine and methylene blue molecules. The higher values of iodine number (between 165.10 and 889.00 mg/g) compared to the lower values of methylene blue (between 2.84 and 9.81mg/g) indicated clearly that the activated carbons have the capacity to adsorb molecules with the particle's sizes less than 2 nm. Therefore, the porosity of activated carbons obtained are predominated

by micropores and this assertion can be confirmed by rigorous equipment of surface analysis.

C. Statistical Analysis

The synthesis of ANOVA (Table 4) gives the probabilities of each factor and their coefficient of interaction when the two factors are taken together in function of the different responses. The Table 4 below showed that for the activated carbons yield (Y_1), the factors having a probability less than 0.05 with a confidence interval of 95%, were the activation temperature (X_1), impregnation ratio (X_2) and residence time (X_3). For the iodine (Y_2) and methylene blue (Y_3) numbers, the factors having a probability less than 0.05 were the activation temperature (X_1), the residence time (X_3) and the interaction between these two factors (X_1X_3). This means that the above factors cited were significantly different of zero at the confidence level of 95% and therefore, only these factors have significant effects on different responses where they are linked.

TABLE 4: ANALYSIS OF VARIANCE OF RESPONSES OBTAINED

	Probability		
	Y_1	Y_2	Y_3
X_1 : Activation temperature	0.0003	0.0001	0.0050
X_2 : Impregnation ratio	0.0136	0.8164	0.8528
X_3 : Residence time	0.0126	0.0155	0.0109
X_1X_2	0.573	0.0144	0.0183
X_1X_3	0.4701	0.6685	0.3232
X_2X_3	0.4512	0.6685	0.5462
R^2	95.53	96.46	91.20
R^2 -adjusted	0.17	92.21	80.63

Additionally, the analysis of Table 5, also showed that for the three responses studied, R^2 and R^2 -adjusted have the values greater than 80%, exception done for the activated carbon yield where the R^2 -adjusted was 0.17. The different values of R^2 indicated that the adjusted model explained respectively 95.53% of the variability of activated carbons yield; 96.46% of variability of iodine numbers and 91.20% of variability of methylene blue numbers. This consideration conducted to the conclusion that the model predicted is validated.

D. Mathematical Modeling of the Responses and Analysis of Effects of Different Factors

The expressions of different responses are given in equations below:

$$Y_1 = 24.8973 - 1.43875X_1 - 0.623625X_2 + 0.611625X_3 - 0.098575X_1X_2 - 0.128125X_1X_3 - 0.134X_2X_3 \quad (5)$$

$$Y_2 = 419.629 + 237.331X_1 + 81.7563X_2 + 5.55625X_3 + 83.3438X_1X_2 + 10.3187X_1X_3 + 10.3188X_2X_3 \quad (6)$$

$$Y_3 = 5.12208 + 1.78362X_1 + 1.47538X_2 + 0.073125X_3 + 1.29113X_1X_2 + 0.410125X_1X_3 + 0.242125X_2X_3 \quad (7)$$

When a positive sign is in front of a coefficient, this means that the factor has a synergetic effect on the response, consequently, its increase conducts to the increase of the

response and inversely. Whereas a negative sign in front of the coefficient indicates an antagonistic effect, thus its increase causes the decrease of the response concerned and inversely. According to the polynomial equations above, for *Yld* (Y_1) only the impregnation ratio has a synergetic effect. For *ION* (Y_2), all the factors have synergetic effect, which of course shows that, all the factors favored the production of micropores on the adsorbent. For *MBN* (Y_3), all the factors have synergetic effect exception for the impregnation ratio and the interaction coefficient of activation temperature and impregnation ratio. This trend can be explained by the fact that, the impregnation with KOH generally promote the formation of micropores. But it can be noticed that the *MBN* is too low with *Balanites aegyptiaca* activated with KOH. This observation is similar to the one observed in our earlier work, where the oil palm shells was impregnated in one hand by KOH and H_3PO_4 . It was clearly demonstrated that, oil palm shells activated with KOH was mostly adsorbed Iodine rather than methylene blue [3], [4]. The analysis of the Pareto diagrams for the activated carbons yield (Fig. 1a) revealed that, the activation temperature was the factor having the significant effect followed by the residence time and the impregnation ratio which have moderated. This significance of activation temperature on the activated carbon yield can be explained because, the increase in activation temperature would involve the release of volatiles matters as the result of intensifying dehydration and elimination reactions as well as increasing the C-KOH reaction rate, thus the decrease in activated carbons yield [3], [22]-[24]. Additionally, when the activation temperature increased, the gasification reactions allowed the liberation of the entrances of the pores blocked by amorphous matters, thus Baçaoui *et al.* observed the same trends of decreasing in activated carbons yield in the work dealing with olive-wastes cake optimizations conditions of preparation [5]. It can be also observed that, increasing activation temperature and impregnation ratio conducted to increase the carbon burn off, this weight loss was attributed to huge elimination reactions [3], [24], which affected significantly *Balanites aegyptiaca* shells and then reduced the yield. The interactions effects (X_2X_3 , X_1X_3 and X_1X_2) have low values indicating that the factors put together do not have significant effects the activated carbon yield.

The Pareto diagrams for the iodine number (Fig. 1b) shows that the activation temperature have the most significant effect whereas the impregnation ratio and the interactions (X_2X_3 , X_1X_3) have not significant effects. But the interaction (X_1X_2) has a moderate significant effect on iodine number. The Pareto diagrams for the methylene blue (Figure 1c) revealed that, the activation temperature, residence time and their interaction (X_1X_2) have the most significant effect. On contrary, the impregnation ratio and the interactions (X_2X_3 and X_1X_3) have the low effects thus they are not significant for the adsorption of methylene blue.

Fig. 1. Pareto graphics of (a) activated carbon yield, (b) iodine and (c) methylene blue number

E. Responses Surfaces

At each point of the experimental domain corresponds a response, thus for the set of points of the experimental domain investigated corresponds a set of responses called responses surface. Therefore, it is possible to predict the values that the different of responses will have inside the experimental domain studied. On the response's surfaces represented below, it can be noticed the evolution of different responses in function of the three factors. Fig. 2 shows the both synergetic effects of activation temperature and residence time on the iodine (Fig. 2a) and methylene number (Fig. 2b).

Fig. 2. Response surface of (a) iodine number and (b) methylene blue number in 3D.

F. Optimum Conditions Obtained

The optimum values together with the corresponding values of factors which allow to obtain these values are summarized in Table 5 below. These values resulted from the superposition of the three responses surfaces Fig. 2 above. The zone of interest was determined, and it was in that zone where the optimum conditions were found. To the optimum conditions obtained to be valid to the model predicted, these optimum conditions have been triplicated and the results of these replications were compared with the values predicted by the model. This operation of optimization has succeeded regarding the difference between the experimental and the theoretical values less than 5%.

TABLE 5: OPTIMUM VALUES OF RESPONSES

Responses	Optimum Values	Activation Temperature (°C)	Residence time (min)	Impregnation ratio
Y_1 (%)	27.73	600	60	1:2
Y_2 (mg/g)	848.25	800	120	1:2
Y_3 (mg/g)	9.77	800	120	1:2

IV. CONCLUSION

The factorial plan used helped to determine the optimum conditions for the preparation of activated carbons. The activation temperature and the residence time found to have significant effects on all the three responses investigated. Additionally, the best values of activated carbons yield were obtained for the lowers values of activation temperature and residence time of 600 °C and 60 min respectively. On the contrary, for the iodine and methylene numbers, the best values are obtained at higher values of 800 °C and 120 min for the activation temperature and residence time respectively. The higher values of iodine numbers obtained between 165.1-889 mg/g comparatively to the lower values of methylene blue numbers obtained between 2.84-9.81mg/g is a primary proof that, the activated carbons based *Balanites aegyptiaca* activated with KOH reagent can be used in the adsorption of molecules of small sizes. Thus, the *Balanites aegyptiaca* is a promising precursor for the preparation of microporous adsorbent which can be useful for the sequestration of micropollutants.

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