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Highly microporous activated carbons from *Mangifera indica* residues: Optimization of preparation conditions using response surface methodology

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ARTICLE INFORMATION

ABSTRACT

Full factorial design (FFD) was applied to generate the matrix of experiments for optimizing the residence time (60–120 min), the activation temperature (600–800 °C) and the impregnation ratio (0.25 – 0.50) for the activated carbons preparation based *Mangifera indica* residues chemically activated. The three responses chosen namely the activated carbon yield (AcYd), the iodine number (ION) and the methylene blue number (MBN) were thoroughly studied and represented in all the experimental domain using the Minitab 16, Inc. and the Sigma16, Inc. software for regression analysis and response surface plots respectively. The results of optimization under the experimental domain revealed that the optimal conditions identified were 800 °C, 120 min and 0.50 for the activation temperature, the residence time and the impregnation ratio respectively. The aforementioned optimal conditions gave 22.7%, 1236 mg/g and 9.61 mg/g for the AcYd, ION and MBN respectively. The activated carbon prepared under this optimal condition conducted to the specific surface area of 831 m²/g as determined by the BET method, with predominance of micropores. Also, the *Mangifera indica* residues were found to be an effective precursor for the preparation of highly microporous activated carbons for depollution purpose.

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Graphical Abstract
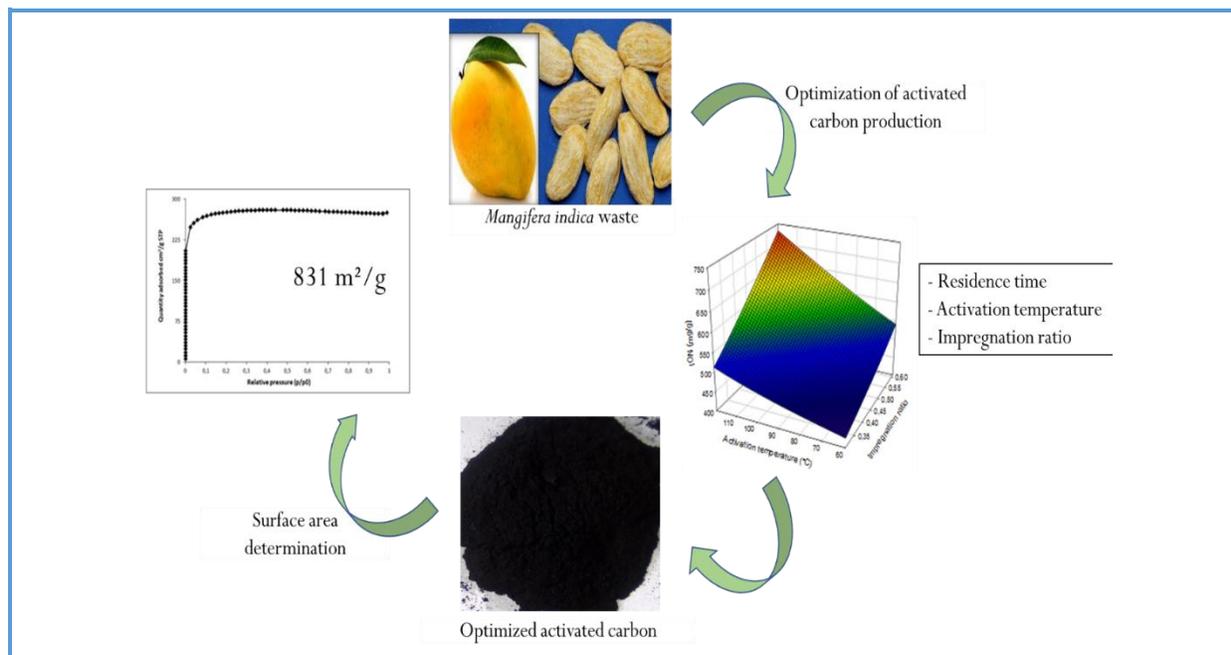
Introduction

Environmental pollution remains a problem of global concern and actualized topic of interest. This problem of pollution is crucial notably in its ecological aspect, thus some international organization in charge of environmental questions such as the United Nation Funds for Environment (UNFE), the environmental protection agency (EPA) just to cite the few are mobilized to find different means to reduce the pollution in all the part of the ecosystem. Some factors such as the human growth, increase of agricultural and industrial activities which consequently discharge huge quantities of wastes of various types are incriminated in the expansion of pollution. Therefore, the challenge is a recall to researchers to propose sustainable methods of managing the wastes to preserve our environment safety. According to a report of the Food and Agriculture Organization Statistics, the West African countries produce huge amount of mangoes for consumption purpose and principally for juice industries [1]. This kind of production is appreciable in terms of food supplies in one hand and the poverty alleviation in another hand. But besides all these above considerations, there is a problem behind notably that of the zero-information given to the destination taken by the residues of these mangoes after its food used. The major preoccupation is to joint ideas to find solution of these mangoes residues by giving them second life in terms of their reuse for example in the production of efficient adsorbents like activated carbons. In the context of sustainable development, the valorization of local biomass based agricultural and industrial waste particularly mangoes residues retained our attention. Transforming these mangoes residues into activated carbons will be an added value to these wastes which is a direct response to the environmental pollution due to their discharge [2]. Firstly, the choice put on the *Mangifera indica* residues was justified by its

lignocellulosic nature favorable for the activated carbons production [3] and secondly, because of the availability of the *Mangifera indica* residues quantities discharged in the neighborhood of juice industries production. Additionally, their abundance and their good physicochemical properties such as high carbon and low ash contents which are the main criteria for choosing activated carbons precursors. Various starting biomass wastes have been used to prepare activated carbons, some of them include, oil palm shells [4–6], olive cakes [7, 8], bamboo [9], Egusi seed shells [10] cola nuts shells [11], Fox nut shells [12], cocoa pods [13], coconuts shells [14], Balanites Aegyptiaca shells [15], plastics wastes mixed with oil palm shells [16]. The scientific literature is also intensified in the activated carbons preparation methods, namely, the chemical, physical and physicochemical activations [5, 10, 16, 17]. Amongst the aforementioned activation methods, the chemical activation seems to be more appreciated because of its low operation conditions such as the temperature around 400 to 700 ℃ comparably to the physical activation, which takes place at high temperature up to 1000 °C [7, 17, 18]. As all the method of activation has a disadvantage, that of the chemical activation is only limited by the washing step during the removal of the excess unreacted activating reagent used. The physical activation has also interesting aspect like its aptitude to promote various type of pores and the non-contamination of final activated carbon produced due to its operation conditions with no need of chemical reagents for activation [7, 17, 18]. Its only disadvantage remains the high energy consumption which of course is not advice according to the restriction of earth temperature raised. The classical technique of activated carbons production which is known to be factor to factor variation, needs many

experiments, consumes too much reagents and waste of times [19, 20]. To avoid this way of activated carbons preparation which works as a black box, the present work and some of our previous works [5, 6, 19, 20] have put an emphasis on the way of optimizing the activated carbons preparation conditions using the methodology of experimental design (MED). The MED is used to better organize the different experimental runs that conduct the scientific research or industrial studies and to study the influence between the parameters of the preparation conditions [10, 13, 21]. There are many types of experimental design adapted to the different cases found by researchers [19, 20, 22]. Amongst the existing design, the full factorial design is used to reduce the total number of experiments in order to obtain a global optimization of a system. Also, it helps to study simultaneous effects of all the factors during the optimization process [23]. Hence, the present work has two main objectives, the first one is to show the feasibility of transforming *Mangifera indica* residues which are considered as waste material into outstanding activated carbons material and secondly to find out the optimum preparation conditions of activated carbons based *Mangifera indica* residues by chemical activation using the response surface methodology.

Experimental

Sample collection

The *Mangifera indica* residues were collected about a market in the city of Ouagadougou, Burkina Faso, they were washed, sun dried, then dried in an oven at 105 °C. They were grinded and sieved to the particle sizes between 1.6 to 3.5 mm retained for the preparation of activated carbons. Whereas the particles sizes of 1 mm diameter were used for immediate analysis, and those of diameter less

than 212 μm for the determination of biomass constituents.

Activated carbons preparation

For the activated carbons preparation, 20.0 g of the dried *Mangifera indica* residues were added to a conical flask of 250 mL containing potassium hydroxide solution of 40 g/L concentration at the desired impregnation ratio. This mixture was shaken during 2 hours at temperature around 50 °C. When the impregnation time was reached, the mixture was filtrated, then the solid part was oven dried at 105 °C overnight. The dried samples were carbonized in furnace at the heating rate of 10 °C/min at required activation temperature under the nitrogen gas during the various residence times. When the carbonization was achieved, the furnace was allowed to cool to room temperature, then the samples were removed and first washed with HCl (0.1 N) then with the distilled water until the pH ranged between 6 and 7. The last step consisted of drying the washed samples in an oven at 105 °C overnight. Finally, the activated carbons obtained were grinded (75 μm) and kept in the desiccator for further experiments.

Experimental design

Based on the literature review and some preliminary analysis which were not presented here, three parameters which were supposed to have significant effects on the characteristics of activated carbons were investigated that were: the residence time (X_1), activation temperature (X_2) and impregnation ratio(X_3). To study the effects of these three factors on the quality and the quantity of activated carbons produced, three responses were studied that were: the activated carbon Yield, Y_1 (%); Iodine number, Y_2 (mg/g) and Methylene blue number, Y_3 (mg/g). The experimental matrix for the full

factorial design was generated using the Minitab 16 Inc Software. The responses studied (Y_i) are linked with coded values of different factor by a regression model equation as follows:

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

Where, a_o is a constant coefficient, a_1, a_2 and a_3 are linear coefficients, a_{12}, a_{13} and a_{23} are the interaction coefficients; X_1, X_2 and X_3 are the coded values of the activated carbon preparation variables. The experimental data were analyzed using a statistical software Minitab (Minitab 16 Inc.) for regression analysis to fit whether the equation developed and also to evaluate the statistical significance of the equation obtained [6]. The response surface plots were generated using SigmaPlot11 software (Systat. Software, Inc.) to describe the relationships between the variable against the responses chosen.

Activated carbon yield

The activated carbon yield (AcYd) is an important quantitative characteristic for the activated carbon production. It gives an idea of the weight loss of the precursor during the pyrolysis step. The activated carbon yield can be calculated by the relation below:

$$AcYd(\%) = \frac{m_i - m_f}{m_i} \times 100 \qquad (2)$$

Where, m_i and m_f are the initial and final mass (g) after pyrolysis of material respectively.

Iodine number

The iodine number (ION) is defined as the quantity in milligrams of iodine adsorbed by one gram of activated carbon. The activated carbon reacts with the iodine solution which the action is to reveal the micropores presence. For this purpose, 20.0 mL of iodine solution of concentration 0.09 mol/L was mixed with 0.2 g

of activated carbon, then mixed for 4 min. Thereafter the mixture was filtrated, and 10.0 mL of the filtrate was collected then titrated using a sodium thiosulfate pentahydrate solution 0.1 N. At the end point, the iodine number, ION (mg/g) was calculated by the following relation.

$$ION = \frac{[20 - V_{thio}] \times 25.4}{m_{CA}}$$ (3)

Where, V_{thio}: is the volume (mL) of sodium thiosulfate solution and m_{CA} the mass (g) of activated carbon.

Methylene blue number

The methylene blue number (MBN) is the volume of methylene blue which can be decolorized by 0.1 g of activated carbon. This index helps to show the ability of activated carbon to adsorb the medium and large molecules in aqueous medium. The experiments were done on a spectrophometer, Miltonroy Company Spectronic 20 D. For this purpose, 0.1 g of activated carbon was mixed with 100 mL of methylene blue solution of 10 mg/L during 4 hrs. After the centrifugation the residual methylene blue concentration was determinated using a Miltonroy Company Spectronic 20D spectrophometer, at the maximum adsorption wavelength was 664 nm. The methylene blue number, MBN (mg/g) was calculated using the following relation:

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

Where, C_i and C_r: are the initial and residual concentration (mg/g) of methylene blue solution; m_{CA} the mass (g) of activated carbon and V the volume (L) of methylene blue solution.

Determination of specific surface area of activated carbon obtained at optimized conditions

The adsorption-desorption isotherms of nitrogen were done using ASAP 2020, Micrometrics equipment. The specific surface area of activated carbon was calculated on the basis that, the cross section of nitrogen adsorb on the surface of activated carbon is 0.162 nm². Before the analysis, the samples were degasified at the temperature of 350 °C to remove all impurities on the surface.

Results and Discussion

Characterization of the Mangifera indica residues

The biomass valorization depends on its thermochemical transformation, nature and properties. The immediate analysis (moisture, ash, volatiles matter, and fixed carbon contents) help to determine, analyze, and see the behavior of the biomass during the pyrolysis process. The results obtained are represented in the Table 4 below. The immediate analysis showed that the *Mangifera indica* residue has a low ash (3%) and fix carbon contents of 15%. This observation indicated that, the *Mangifera indica* residues are good precursor to prepare carbon with a low ash content. These results are similar with those obtained with the activated carbon based mangoes seeds [24]. The biomass quantification constituents' helps to understand the mechanism involved in the biomass conversion and to estimate the energetic potential of a particular biomass. For this purpose, it was interesting to know the composition in main constituents (extracts, cellulose, hemicelluloses, and lignin) obtained by wet analysis of *Mangifera indica* residues which are compiled in the Table 1. The analysis of the chemical composition of *Mangifera indica* residues showed that, the cellulose content was greater than the hemicellulose content, this trend can be attributed to the crystalline structure of *Mangifera indica* residues used. It

can be noticed, a small value of lignin content. It's well known that the lignin contains a major part of fix carbon, this part where the energetic content was the greater one, thus this small value of lignin content indicated that, the *Mangifera indica* residues will has weak activated carbon yield. This same results have been observed by Hernandez and Gonzalez [24].

Analysis of the experimental design

A regression model equation has been developed by the Minitab 16 Inc. software, in order to analyze the correlation between the studied variables (activation temperature (X_1); residence time (X_2) and impregnation ratio (X_3)) and the responses (activated carbon yield (Y_1), iodine number (Y_2) and methylene blue number (Y_3)). The four trials at the center were done to determine experimental error and to verify the reproducibility of experimental results. The experimental design matrix which included the experimental and predicted responses investigated in this work are given in the Table 2.

Table 1. Proximate and chemical analysis results of *Mangifera indica* residues

Proximate analysis (%)		Chemical analysis (%)	
Moisture	2	Extracts	17
Ash	3	Lignin	15
Fix carbon	15	Hemicellulose	26
Volatile matter	80	Cellulose	42

Table 2. Experimental design matrix and the responses obtained

Run N°	Factors			Y_1 (%)		Y_2 (mg/g)		Y_3 (mg/g)	
	X_1 (min)	X_2 (°C)	X_3	Exp. value	Pre. value	Exp. value	Pre. value	Exp. value	Pre. value
1	60	600	1/4	25.60	26.56	269.92	238.38	1.08	1.37
2	120	600	1/4	26.75	25.79	227.64	259.18	3.78	3.49
3	60	800	1/4	22.90	21.94	811.58	843.12	9.72	9.43
4	120	800	1/4	22.65	23.61	1236.08	1204.54	9.61	9.90
5	60	600	1/2	35.15	34.19	101.11	132.65	1.31	1.02
6	120	600	1/2	28.85	29.81	82.45	50.91	1.84	2.13
7	60	800	1/2	24.80	25.76	747.99	716.45	9.58	9.87
8	120	800	1/2	24.80	23.84	943.81	975.35	9.62	9.33
9	90	700	3/8	26.91	27.09	538.29	530.18	5.54	5.54
10	90	700	3/8	26.98	27.09	537.80	530.18	5.56	5.54
11	90	700	3/8	26.65	27.09	525.60	530.18	5.56	5.54
12	90	700	3/8	27.82	27.09	519.02	530.18	5.50	5.54

The activated carbon yield varied from 22.65 to 35.15%, whereas iodine numbers were ranged from 227.64 to 1236.08 mg/g and the methylene blue numbers varied between 1.08 and 9.72 mg/g. These results showed that the highest values of iodine and methylene blue numbers are obtained from activated carbons prepared at activation temperature of 800 °C and the lower values at lower temperature of activation of 600 °C. All responses were used to develop mathematical regression equations of the models showing interactions which correlated each response with the three coded variables. These model equations in coded values for yield (Y_1), iodine (Y_2) and methylene blue number (Y_2) are obtained by replacing each

coefficient in equation (1) to obtain equations (5) to (7).

$$Y_1 = 26.4 - 0.3X_1 - 2.27X_2 - 1.58X_3 + 0.23X_1X_2 + 0.52X_1X_3 + 0.57X_2X_3 \quad (5)$$

$$Y_2 = 545.4 + 69.9X_1 + 382.2X_2 + 83.7X_3 + 85.1X_1X_2 + 25.6X_1X_3 + 5.2X_2X_3 \quad (6)$$

$$Y_3 = 5.7 + 0.39X_1 + 3.8X_2 + 0.23X_3 - 0.41X_1X_2 + 0.25X_1X_3 - 0.19X_2X_3 \quad (7)$$

The validity of these equation models is linked on the value of the correlation coefficient (R^2) which must be close to unity [23]. Thus, the experimental and predicted values are closed and then the model is validated. In the present case, R^2 and adjusted R^2 are all closed to unity for all the responses. It is important to note that the adjusted R^2 value is the correlation coefficient obtained after removing the non-significant coefficients from the model. R^2 values of yield, iodine and methylene numbers were found to be 0.9199, 0.9938 and 0.9944 respectively indicating the variability of 91.99 %, 99.38% and 99.44% of the response variable.

Analysis of variance and response surface analysis of the activated carbon yield

The ANOVA for the activated carbon yield, Y_1 was summarized in the Table 3.

According to the ANOVA, the activation temperature (X_2) and the impregnation ratio (X_3) are the significant factors influencing the yield since their *p*-value are both less than 0.05 (Table 3). These observations are also confirmed by the Pareto char (Figure 1).

Table 3. ANOVA for the activated carbon yield

Source	Df	Seq SS	Adj SS	Adj MS	F-value	P-value
X_1	1	3.645	3.6450	3.6450	1.78	0.253
X_2	1	56.180	56.1800	56.1800	27.46	0.006**
X_3	1	30.811	30.8113	30.8113	15.06	0.018**
X_1X_2	1	3.001	3.0012	3.0012	1.47	0.292
X_1X_3	1	6.480	6.4800	6.4800	3.17	0.150
X_2X_3	1	7.220	7.2200	7.2200	3.53	0.133
Residual Error	4	8.182	8.1822	2.0456		
Lack of Fit	1	7.411	7.4112	7.4112	28.84	0.013**
Total	11	116.655				

R^2: 0.9199 ; R^2 adjusted : 0.8071

Df Degrees of freedom
Seq SS Sequential Sum of Squares
Adj SS Adjusted Sum of Squares
Adj MS Adjusted Mean Squares
** Most significant

Figure 1. Pareto chart of the standardized effects of the activated carbon yield (Y_1)
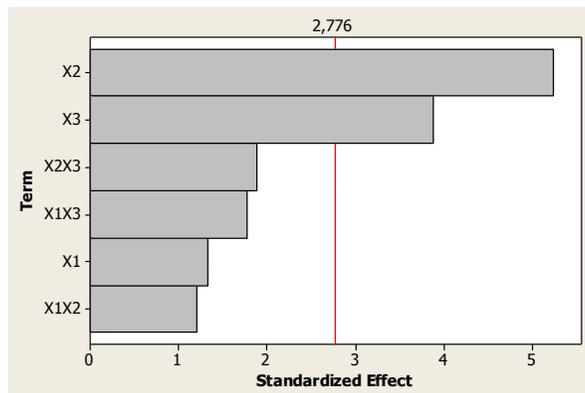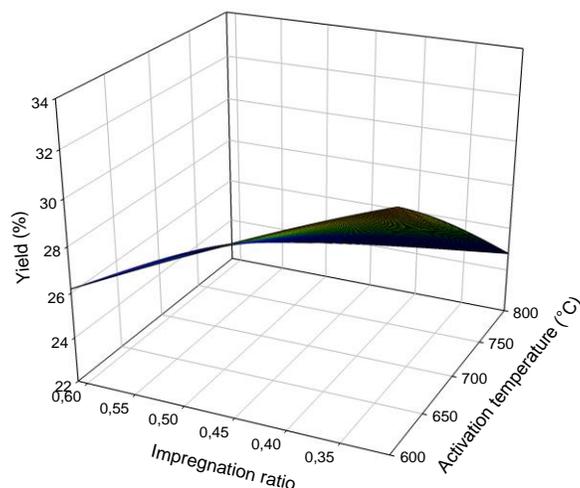
Figure 2. Response surface plot for the effect of activation temperature and impregnation ratio on activated carbon yield (Y_1)

According to Figure 2, an increase of the activation temperature from 600 to 800 °C conducted to a decrease of the activated carbon yield from 32 to 25%. Other authors observed the same trend on the values of activated carbon yield for precursors impregnated with KOH [24]. It can be noticed that the thermal treatment in inert atmosphere with previous impregnation of the biomass conducted to the degradation of the microstructure, thus an important weight loss. The activation temperature had a negative effect on the activated carbon yield (-2.46), because at high temperature, there was important volatilization of compounds and above 700 °C, the activated carbon yield remained constant. This observation was in accordance with the work done by Gueye *et al*. [25], who worked with the Jatropha wood and the groundnuts shells impregnated phosphoric acid and potassium hydroxide. The impregnation ratio was less significant on the activated carbon yield. It can be observed on the Figure 4 that, as the impregnation ratio increased, the activated carbon yield decreased. During the impregnation step with KOH solution, particularly with the concentrated solutions,

the presence of hydroxides ions which are nucleophiles species caused the fragmentation and the solubilization of the main components of the biomass thus the important weight loss observed [25]. The same results have been obtained by others authors who worked with coconuts shells [26]. For highly concentration solution, the weight loss was due to the increase of release of volatiles matters consequence of the deshydratation and removal reactions. The above response surface graph (Figure 2) illustrated the different evolution.

Iodine number

The ANOVA for the iodine number, Y_2 was summarized in the Table 4.

The ANOVA revealed that, the factor shaving a probability less than 0.05 are the residence time (0.012) and activation temperature (0.000). The impregnation ratio (0.006) and the interaction between the residence time and activation temperature (0.006) with activation temperature having the most significant effect on the iodine number.

As seen in Figure 3, all the three factors have synergetic effects on the response Y_2.

Table 4. ANOVA for the iodine number

Source	Df	Seq SS	Adj SS	Adj MS	F-value	P-value
X_1	1	39113	39113	39113	19.02	0.012
X_2	1	1169180	1169180	1169180	568.51	0.000***
X_3	1	56089	56089	56089	27.27	0.006**
X_1X_2	1	58014	58014	58014	28.21	0.006**
X_1X_3	1	5256	5256	5256	2.56	0.185
X_2X_3	1	219	219	219	0.11	0.761
Residual Error	4	8226	8226	2057		
Lack of Fit	1	7957	7957	7957	88.62	0.003 **
Total	11	1337436				

R^2: 0.9938; R^2 adjusted: 0.9831

[Df] Degrees of freedom;
[Seq SS] Sequential Sum of Squares
[Adj SS] Adjusted Sum of Squares
[Adj MS] Adjusted Mean Squares
**Most significant
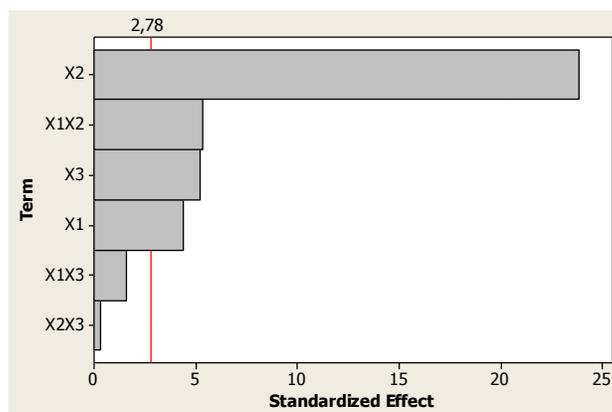
Figure 3. Pareto chart of the standardized effects for the iodine number (Y_2)

As can be seen in Figure 4, there was an increase of Y_2 from 82 mg/g to 1236 mg/g. This implied on the adsorbent surface, the presence of mixture of pores namely the micropores and mesopores. This assertion goes in the same point of view with the work done by Juang *et al.* [27], who studied the role of microporosity on the phenol removal and concluded that, this type of adsorption can only be possible on one type of pores. Moreover. The results obtained indicated that, the activated carbon with impregnation ratio of 0.5 (experiments 2 and 4) had high iodine numbers compared to those with impregnation ratio of 0.25 (experiments 6 and 7) which of course had lower values of iodine numbers. This difference of iodine number against the impregnation ratio can be explained by the effect of the impregnation of the biomass with KOH and during the pyrolysis step, which favorites the volatilization volatiles compounds which inhibited the tar formation [28]. The microporosity of samples impregnated with KOH was mainly due to the intercalation of potassium in the carbon structure. At high concentration, the effect of metallic salt in the formation of the porosity was more pronounced [28].

Methylene blue number

The ANOVA for the methylene blue, Y_3 was summarized in the Table 5. The analysis of the mathematical model of Y_3 showed that the coefficients of the residence time (0.39), activation temperature (3.81) and impregnation ratio (-0.22) had opposite effects of methylene blue number. The activation temperature had the most important effect as represented by Pareto char shown in the Figure

5. This behavior of activation temperature was predicted because as the activation temperature increased, it conducted to the widening of pores, thus the increase of methylene blue adsorption [2]. This trend had been observed by others authors [5, 6].

Also the Figure 6 showed that the methylene blue numbers decreased as the activation temperature increased while there was an increase observed with an increase of the residence time.
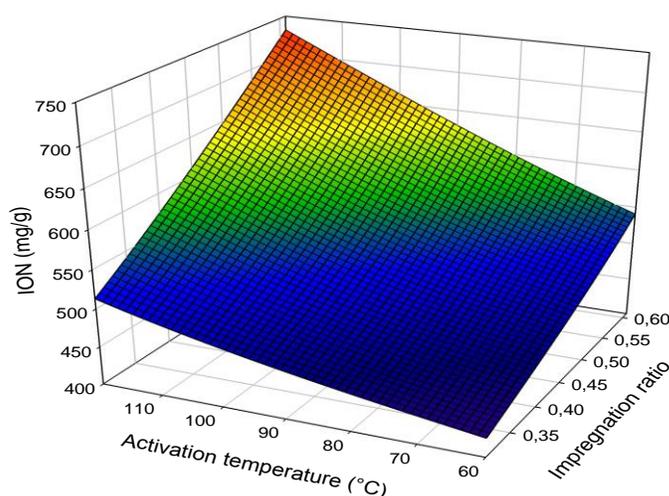
Figure 4. Response surface plot for the effect of activation temperature and impregnation ratio on the iodine number (Y_2)

Table 5. ANOVA for the methylene blue number

Source	Df	Seq SS	Adj SS	Adj MS	F-value	P-value
X_1	1	1.248	1.248	1.248	7.39	0.053
X_2	1	116.434	116.434	116.434	689.77	0.000***
X_3	1	0.423	0.423	0.423	2.51	0.189
X_1X_2	1	1.361	1.361	1.361	8.06	0.047 ***
X_1X_3	1	0.510	0.510	0.510	3.02	0.157
X_2X_3	1	0.312	0.312	0.312	1.85	0.246
Residual Error	4	0.675	0.675	0.169		
Lack of Fit	1	0.673	0.673	0.673	841.00	0.000***
Total	11	121.169				

R^2 : 0.9944 ; R^2 adjusted : 0.9847

[Df] Degrees of freedom
[Seq SS] Sequential Sum of Squares
[Adj SS] Adjusted Sum of Squares
[Adj MS] Adjusted Mean Squares
**Most significant
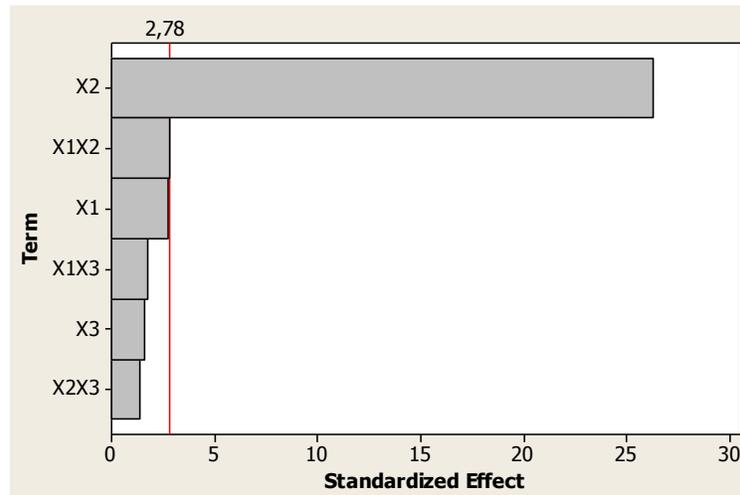
Figure 5. Pareto chart of the standardized effects for the methylene blue number (Y_3)
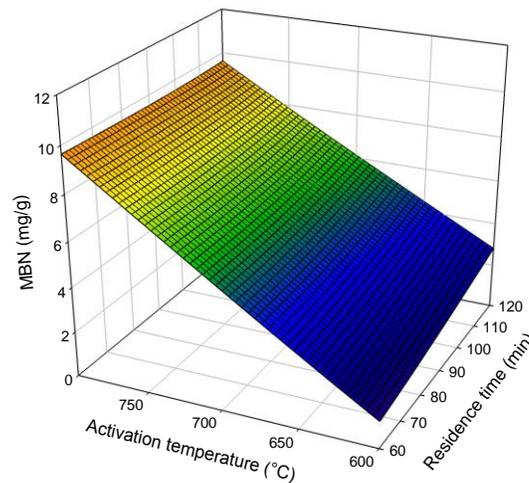

Figure 6. Response surface plot for the effect of activation temperature and residence time for the methylene blue number(Y3)

Optimum conditions

In accordance with our desirability which was to produce an activated carbon in quantity and quality, the values of parameters leading to the optimum condition were found to be 120 min; 800 °C and 1/2 for the residence time, activation temperature and impregnation ratio respectively which conducted to 22.7%; 1236 mg/g and 9.61 mg/g for the activated carbon yield, iodine and methylene blue number respectively.

The specific surface area of the sample of activated carbon obtained in the optimum conditions was then assessed using nitrogen adsorption. The adsorption isotherm was compared to the information sent to the pores structure of an adsorbent, the adsorption temperature, physical, and chemical characteristics [29]. Figure 7 presents the adsorption isotherm obtained for the *Mangifera indica* activated carbon residues prepared under optimum conditions.
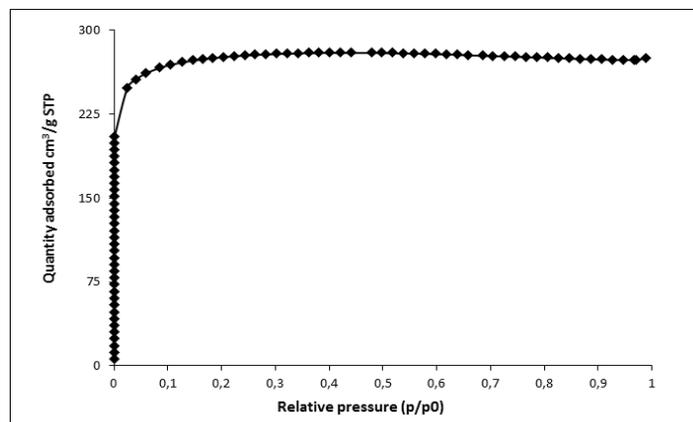
Figure 7. Adsorption isotherm of *Mangifera indica* activated carbon residues

According to the IUPAC classification, the adsorption isotherm obtained (Figure 1) was of type I, which corresponded in the first approximation to the distribution of micropores on the adsorbent surface. At the beginning of the isotherm, it represented the filling of micropores and at higher pressure where there was the slope, it represented the adsorption on the mesopores and the external surface of the adsorbent. The total surface area calculated by the BET method at low relative pressure (0.02 – 0.3) was found to be 831 m²/g.

Conclusions

The experimental data obtained revealed that, the activated carbon based *mangifera indica* residues had a specific surface area of 831 m²/g. The BET adsorption isotherm demonstrated a microporous structure with the little mesoporous structure of activated carbon. Whereas the activation temperature of 600 °C, residence time of 60 min and impregnation ratio of 1:4 provided a higher value of activated carbon yield of 32%, which matched singularly the activated carbon in quantity but not the quality. The optimum conditions of the activated carbon both in quantity and quality were designed by the following conditions: 800 °C, 120 min and 0.50 for the activation temperature, residence time and impregnation ratio respectively. These optimum conditions conducted to 22.7%, 1236 mg/g and 9.61 mg/g for the AcYd, ION and MBN respectively. It was also found that, the *Mangifera indica* residues is a promising precursor for the preparation of outstanding activated carbon for the removal of pollutants for depollution purpose.

Disclosure Statement

No potential conflict of interest was reported by the authors.

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