Optimization of the Preparation Conditions for Activated Carbon from Locust Bean Pod (*Parkia biglobosa)* Using Factorial Design Approach

O. A. A. Eletta[1a], S. I. Mustapha[1b], J. O. Tijani[2], L. T. Adewoye[1c] and I. A. Mohammed[1d]
[1a,b,c,d]Department of Chemical Engineering, University of Ilorin, Ilorin
[2]Department of Chemistry, Federal University of Technology, Minna
[1a]modeletta@unilorin.edu.ng, [1b]mustapha.si@unilorin.edu.ng,
[2]adewoye.lt@unilorin.edu.ng, [1c]mohammed.ia@unilorin.edu.ng,
[1d]jimohtijani@futminna.edu.ng
Corresponding author:
modeletta@unilorin.edu.ng; Tel: +234 803 581 7674

Abstract

This study focused on the optimization of the preparation parameters (impregnation concentration, activation temperature and activation time) on the yield and adsorption potential of activated carbon (AC) prepared from Locust bean pod (*Parkia biglobosa)* using the chemical activation method based on factorial design. The two linear regression models developed from the factorial experimental design using Design Expert Software – 6.0.8 was used to determine the optimum production conditions required to provide a compromise between the AC yield and methylene blue (MB) removal efficiency from aqueous solution. The results demonstrated maximum AC yield of 41.6% and MB removal efficiency of 95.4% under the following optimum preparation conditions; H_3PO_4 impregnation concentration (60 %), activation temperature (444.4 ^oC) and activation time (30 min). This study showed that the experimental values obtained were in good agreement with the values predicted from the models under the applied conditions.

Keywords

Waste management, Locust bean pod, activated carbon, factorial design, chemical activation, yield

1. Introduction

Activated carbon (AC) is well known for its high adsorption capacity due to its exceptional high surface area, well developed internal porosity, wide spectrum of surface functional groups, and above all, high chemical and mechanical stability (Rivera-Utrilla *et al.*, 2011; Zhou *et al.*, 2015). Besides its uniqueness, commercial activated carbons have been successfully used to remove pollutants from various wastewaters; however, their large scale utilization is restricted due to high cost. This shortcoming has led to the search for less expensive activated carbon from unconventional, readily available materials such as, agricultural and industrial wastes (Demirbas, 2008; Ioannidou and Zabaniotou, 2007). In recent years, research efforts have been focusing on the preparation of activated carbon from agricultural lignocelluloses waste materials due to their abundance and availability at little or no cost. Some of these agricultural byproducts include bamboo waste (Reza *et al.*, 2014), water hyacinth leaves (Guerrero-Coronilla *et al.*, 2015), macadamia nut shells (Martins *et al.*, 2015), pomegranate peel (Ahmad *et al.*, 2014), rambutan peel (Njoku *et al.*, 2014), potato peels (Kyzas and Deliyanni, 2015), palm date seed (Islam *et al.*, 2015), almond shell and orange peel (Hashemian *et al.*, 2014), mung bean husk (Mondal *et al.*, 2015), *Ficus nitilda* (Ali and Alrafai, 2016), eggshell waste (Eletta *et al.*, 2016), rice husk, maize cob, cassava peels, yam peels, pawpaw peel among others among others.

In addition, *Parkia biglobosa* otherwise called African locust bean is a perpetual broad-leaved plant which belongs to the Fabaceae family and is widely found in the northern part of Nigeria (Farayola *et al.*, 2013). The plant produce several pulps which encapsulated the fruits and the seeds and studies have shown that over 200,000 tonnes of locust seeds are collected for locust bean on yearly basis (Olapade-Ogunwole *et al.*, 2011). Large quantities of locust bean pod/pulp waste is generated annually from the processing of the African locust bean fruit thereby constituting serious environmental nuisance due to the lack of proper solid disposal management.

Activated carbon (AC) can be prepared by either chemical or physical treatment with the former being a more commonly used process. This is due to its numerous advantages over the latter which include lower activation temperature, single step process for carbonization and activation, higher AC yield, and higher surface area. Others include, creation of a well-developed porous structure and easy recovery of the added chemical agents used for activation (Yahya *et al.*, 2015). The yield and quality of the prepared AC depend largely on the nature of initial material and the preparation conditions such as degree of impregnation, carbonization temperature and holding time (Abechi *et al.*, 2013; Campbell *et al.*, 2012). The production of quality activated carbon involves balancing the preparation conditions in order to obtain the desired yield and of higher adsorption capacity (Sahu *et al.*, 2010). Thus, there is need to establish the optimum conditions for the preparation of activated carbons with well-developed porosity and at the same time with high yield. In studying the effect of preparation conditions on quality characteristics, the utilization of an appropriate experimental design is considered necessary (Tan *et al.*, 2008). Factorial design (FD) has been found to be a useful tool to study the effects and interactions between two or more variables with minimum number of experiments, as well as to optimize the variables (Mohammed *et al.*, 2017). Optimisation of adsorption process conditions using response surface methodology has been widely documented (Adewoye *et al.*, 2017; Alam *et al.*, 2009; Auta and Hameed, 2011; Cronje *et al.*, 2011; Garba *et al.*, 2016; Gottipati and Mishra, 2010; Isoda *et al.*, 2014; Sahu *et al.*, 2010; Tan *et al.*, 2008). However, the application of full factorial experimental design in the optimization studies of activated carbon production process has not been well explored. To the best of the author's knowledge, no study has reported the optimization of the preparation parameters on the activated carbon develop from locust bean pod (*Parkia biglobosa*) using factorial design approach.

Therefore, this study focused on the optimization of the preparation conditions of activated carbon obtained from locust bean pod for the removal of methylene blue (MB) dye. The effects of preparation parameters; activation time, activation temperature and H_3PO_4 impregnation concentration were studied simultaneously to achieve high activated carbon yield and high MB percentage removal from aqueous solution using full factorial design

2. Materials and Methods

2.1 Materials

Methylene blue (MB) purchased from Sigma – Aldrich was used as an adsorbate. MB has a chemical formula of $C_{16}H_{18}ClN_3S$, with molecular weight of 319.85 g/mol. The chemical structure of MB is shown in Figure 1.

All the chemicals and reagents used in the study were of analytical grade and distilled water was used to prepare all solutions.

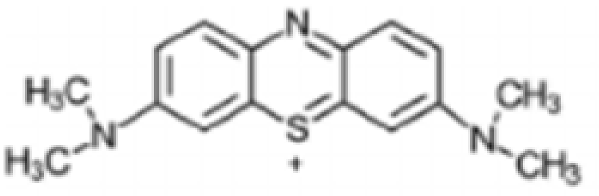


Figure 1 Chemical structure of methylene blue dye

2.2 Sample Collection and Preparation

The locust bean pod (LBP) used in this study was collected from different farm location within Oke-Oyi local government area in Ilorin, Kwara State to form composite out of which representative samples were used for the AC production. The collected samples were washed several times with deionised water to remove any adhering dirt present on the surface and thereafter air dried for one week. The dried samples were crushed and separated using sieve size of 225 µm. The powdered samples were then oven dried in a laboratory at 105 ^oC till a constant weight is attained.

2.3. Preparation of activated carbon from Locust bean pod

Twenty gram (20 g) each of the dried powdered locust bean pod was weighed into 250 mL beakers and 100 mL of each concentration of H_3PO_4 (0, 20, 40, 60, and 80 %) was added. The impregnation was carried out for 24 hr at room temperature to achieve good penetration of reagent into the interior of the precursor. Subsequently, the impregnated samples were then subjected to carbonization process in a chamber furnace (Carbolite CWP 12/13 Model) at specific temperature (350, 450, 550, and 650 ^oC) and reaction time (30 min and 120 min) respectively. The samples were subsequently cooled to room temperature. The yield of AC was determined using the relationship presented in Equation 1 (Auta and Hameed, 2011).

$$\text{Yield}(\%) = \frac{\text{Weight of LBPAC Prepared}}{\text{Weight of the LBP Used}} \text{ x } 100 \qquad (1)$$

2.4 Design of Experiments

Full factorial design (FFD) is widely used experimental designs at two-levels. These levels are referred to as 'high' and 'low' or '+1' and '-1', respectively. The total number of experiments for studying *k* factors at 2-levels is 2^k which is used to provide information about the main effects and interaction effects between the studied factors. In this study, a two-level and three-factor full factorial design (2^3 runs) was used to analyse the effect of main factors as well as the effect of the interactions between the preparation parameters on the yield and quality of LBPAC produced. The pink highlight to be deleted. It is already mentioned in section 2.2 The parameters studied were (i) H_3PO_4 impregnation concentration; (ii) activation temperature and (iii) activation time coded as A, B and C respectively. These three parameters together with their respective ranges were chosen based on information from literature (Gottipati, 2012). The levels and ranges of the studied factors are presented in Table 1.

Table 1 Experimental ranges and levels of the factors used in the FFD

Independent Variable (Factors)	Coded Symbol	Units	Range and Levels	
			-1	**+1**
H_3PO_4 Concentration	A	%	20	60
Activation Temperature	B	^oC	350	650
Activation Time	C	min	30	120

The experimental design matrix of all the factors in coded and actual values for all the experimental runs is shown in Table 2. The responses were expressed as the yield of LBPAC and percentage removal of MB.

The general empirical model developed for 2^3 factorial design in coded values is given as follows:

$$\%Y = X_0 + X_1A + X_2B + X_3C + X_4AB + X_5AC + X_6BC + X_7ABC \qquad (2)$$

Where Y is the predicted response (Yield of AC and Adsorption of MB), X_o is the global mean, X_i represents the other regression coefficients and A, B and C are the coded symbols for the studied factors.

Table 2 Factorial experimental design matrix with coded and real values

Run	Coded Values			Real Values			Yield of LBPAC, (%Y)	Removal of MB (%R)
	A	B	C	H_3PO_4 Concentration (%)	Activation Temp (°C)	Activation Time (min)		
1	-1	-1	-1	20	350	30	38.28	93.2
2	1	-1	-1	60	350	30	46.62	93.4
3	-1	1	-1	20	650	30	11.55	96.5
4	1	1	-1	60	650	30	30.34	98.0
5	-1	-1	1	20	350	120	33.02	92.7
6	1	-1	1	60	350	120	34.68	92.1
7	-1	1	1	20	650	120	1.2	98.9
8	1	1	1	60	650	120	11.86	98.4

2.5 Model Fitting and Statistical Analysis

Design Expert – 6.0.8 (Stat-Ease Inc., Minneapolis, USA) licensed statistical software was used for the regression analysis to fit the linear model equation and also for evaluation of the statistical significance of the model equation developed.

2.6 Adsorption Studies

Batch adsorption of MB on the developed activated carbon was carried out. Equal mass of 100 mg of the prepared AC was mixed with 100 ml of MB solutions with initial concentration of 100 mg/l in 16 sets of 250 ml Erlenmeyer flasks. Each mixture was kept in an isothermal shaker operating at 120 rpm and 30 °C until equilibrium was reached. Samples were taken out at regular intervals and residual concentrations of MB in the solution was determined using a double beam UV- Visible spectrophotometer (Shimadzu, Japan) at maximum wavelength of 650 nm. The percentage removal at equilibrium was calculated according to

$$\% \, Removal = \frac{(C_o - C_e)}{C_o} \times 100 \quad (3)$$

Where Co and C_e (mg/l) are concentrations of the MB dye at initial and at equilibrium, respectively.

2.7 Characterization of Activated Carbon

The surface functional groups of the precursor and the prepared activated carbon were determined using Fourier transform infrared radiation (FTIR) spectroscope (Perkin – Elmer) with resolution of 4cm^{-1} in the range of 4000 – 500 cm^{-1}. The surface morphology of the samples was examined using Zeiss Auriga HRSEM under the following operation conditions: current 10 mA, voltage 5 kV, aperture 0.4 mm and working distance 4 –10.4 mm. The microscope was operated with electron high tension (EHT) of 5kV for imaging.

3. Results and Discussion

3.1 Development of Regression Model Equation

The individual run of the 2^3 factorial experimental designs was carried out and the values of the responses (% yield and % MB removal) were evaluated and presented in Table 2. The percentage yield obtained was in the range of 1.2 – 46.62% while the percentage removal of MB was in the range of 92.1 – 98.9%. Full factorial design (FFD) was used to develop the linear regression model for analysis of the correlation between the yield, MB adsorption and the preparation variables of LBPAC. The final resultant model in terms of coded factors with the exclusion of the insignificant terms for activated carbon yield (Y_1) and adsorption of methylene blue (Y_2) are as follows:

$$Y_1 = 25.94 + 4.9A - 12.21B - 5.75C + 2.43AB - 1.85AC - 1.45A\text{BC} \quad (4)$$

$$Y_2 = 95.05 + 0.97A + 2.45B + 0.82C - 0.90AC + 0.33BC + 0.20ABC \quad (5)$$

Where A is H_3PO_4 impregnation concentration, B is activation temperature and C is the activation time.

It can be observed from both equation (4) and (5), that the magnitude of coefficients of term B was larger than the coefficients for A and C. This indicates the activation temperature exerted greater significant effect on the LBPAC yield than the individual or combined effect of acid concentration and activation time.

3.2 Statistical Analysis of Variance (ANOVA)

The ANOVA results for the developed linear regression models (equations 4 and 5) are presented in Tables 4 and 5. The p-value represents the probability value used to determine the statistically significant effects in the factorial model (Gottipati, 2012). Model terms having p-value less than 0.05 are statistically significant for a 95% confidence level. As shown in Tables 4 and 5, the models suggest that, the responses are significant with p-value of 0.023 and 0.016 for the LBPAC yield and MB adsorption, respectively.

Table 4 - ANOVA results for LBPAC yield

Source	**Sum of Squares**	**Degree of Freedom**	**Mean Square**	**F - Value**	**p - Value**
Model	1742.94	6	290.49	1105.31	0.0230[a]
A	194.54	1	194.54	740.22	0.0234[a]
B	1191.94	1	1191.94	4535.33	0.0095[a]
C	264.85	1	264.85	1007.73	0.0200[a]
AB	47.29	1	47.29	179.93	0.0474[a]
AC	27.42	1	27.42	104.32	0.0621[b]
BC	16.91	1	16.91	64.33	0.0790[b]
Residual	0.26	1	0.26		
Total	1743.20	7			

[a] significant at 95 % confidence level
[b] not significant at 95 % confidence level

Table 5 - ANOVA results for methylene blue adsorption

Source	**Sum of Squares**	**Degree of Freedom**	**Mean Square**	**F - Value**	**p - Value**
Model	68.72	6	11.45	2290.50	0.0160[a]
A	7.60	1	7.60	1521.00	0.0163[a]
B	48.02	1	48.02	9604.00	0.0065[a]
C	5.44	1	5.44	1089.00	0.0193[a]
AC	6.48	1	6.48	1296.00	0.0177[a]
BC	0.85	1	0.85	169.00	0.0489[a]
ABC	0.32	1	0.32	64.00	0.0792[b]
Residual	0.005	1	0.005		
Total	68.72	7			

[a] significant at 95 % confidence level
[b] not significant at 95 % confidence level

Furthermore, from the ANOVA of response surface linear model for AC yield (Table 4), the terms A, B, C and AB were significant while AC and BC were insignificant terms in the LBPAC yield model. In this case, the model term B (activation temperature) with F-value of 4535.33 and p-value of 0.023 exerted the most significant effect on the LBPAC yield. For the case of the ANOVA results for response surface linear model for MB adsorption (Table 5), the model F-value of 2290.50 implied that the model was significant as well. In this instance, all the terms (A, B, C, AC and BC) were significant except for the term ABC that was found to be insignificant to the response.

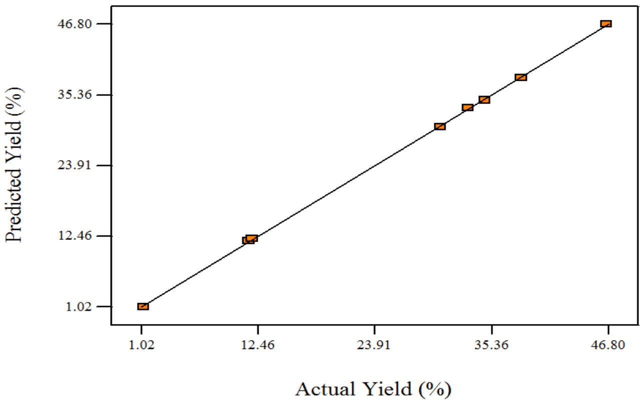


Figure 2 The predicted versus actual plot of yield of LBPAC

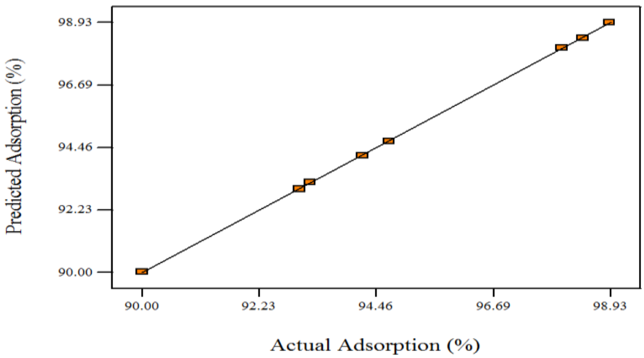


Figure 3: The predicted versus actual plot of adsorption of methylene blue

The quality of the regression models developed was evaluated based on the correlation coefficient value. The R^2 values of 0.99 obtained for both yield and methylene blue adsorption was relatively high (close to unity), indicating a good agreement between the predicted and experimental data (Figures 2 and 3). The closeness of this value to unity support the adequacy of the models as well as the reliability of the data distribution in the experiment (Auta and Hameed, 2011).

From the statistical results obtained, it was shown that the developed models were adequate to predict the AC yield and MB adsorption within the range of variable studied. Thus, the model can be used to navigate the design space at 95% confidence level (Rashid *et al*., 2011).

3.3 Pareto Plot

Pareto plot is a useful tool for visualising the relative size of effects of main factors and the effects of interaction among the factors (Gottipati, 2012). A

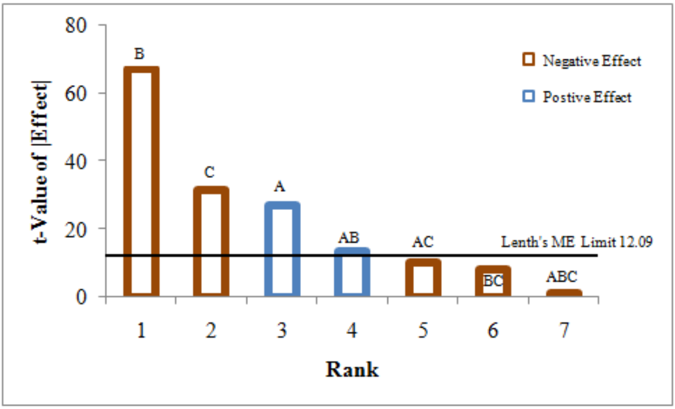


Figure 5: Pareto plot for effects of individual factors and interactions for LBPAC yield

length's benchmark is drawn to indicate that the factors which extend past this line are potentially important. The effects of all the factors and their interactions are shown in Figures 5 and 6 for AC yield and MB adsorption, respectively. The t-values of effect are obtained by dividing the model coefficients by standard error. Effects above lenth's limit are possibly significant terms in the model. For the case of AC yield (Figure 5), the main factors namely H_3PO_4 concentration (A), activation temperature (B), activation time (C) and interactions such as AB significantly influenced the response. Other terms such as AC and BC were added to the model because of the importance of their effect contributions. In the case of MB adsorption (Figure 6), the terms AC and BC together with the main factors (terms A, B and C) significantly influenced the response. For both responses, activation temperature (B) was found to have the greatest effect on the response while impregnation concentration (A) and activation time (C) showed almost similar effects on the responses, which were less significant when compared with the activation temperature.

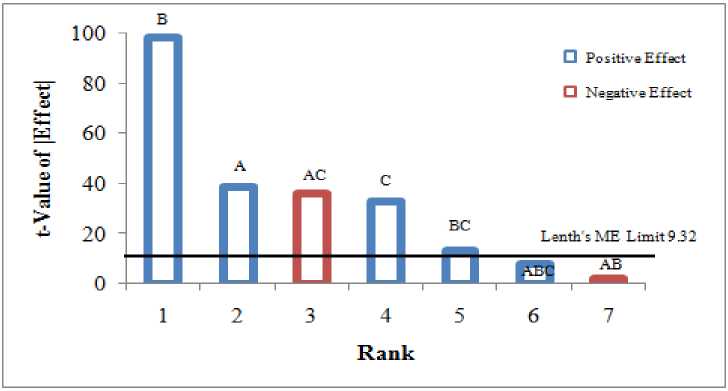
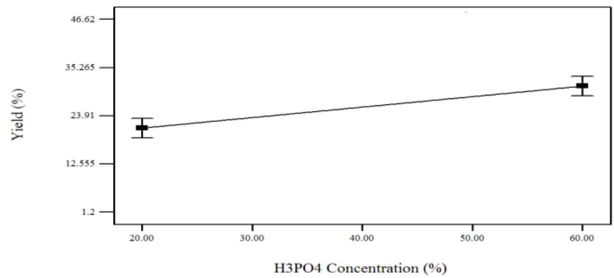


Figure 6: Pareto plot for effects of individual factors and interactions for MB adsorption

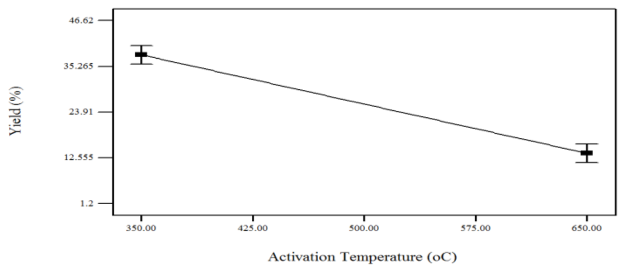
3.4 Activated Carbon Yield

Based on the ANOVA result (Table 4) and pareto plot (Figure 5), all the three variables were found to be significant on the response for activated carbon yield, with the activation temperature imposing the greatest effect (with highest F-value of 4535.33). The impregnation concentration and activation time have almost similar effects on this response and these were less significant compared to activation temperature. The effect of these main factors on the yield of LBPAC was studied as they were found to have significant effects on the production of activated carbon. Figure 7a showed that the phosphoric acid impregnation concentration had a positive effect and the percentage yield of LBPAC increased with increase in H_3PO_4 impregnation concentration. On the other hand, activation temperature and activation time negatively affected the yield of LBPAC as shown in Figures (7b and 7c) respectively. From the Figure 7b, it can be clearly seen from the nature of the slope of the plot that the activation temperature exerted the most effect on the yield of LBPAC in the studied range compared to the other factors.

(a)



(b)



(c)

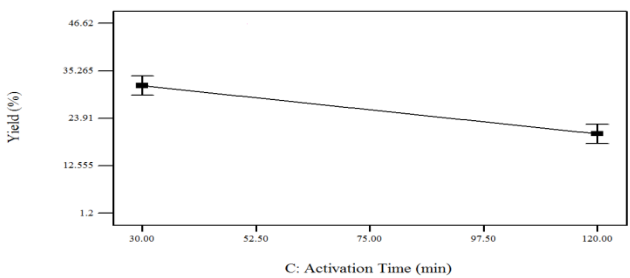


Figure 7: Effect of Main Factors on Yield of LBPAC
(a) H_3PO_4 concentration, % (b) Activation temperature, ^oC (c) Activation time, min

The combined interaction effects of impregnation concentration and activated temperature was studied and was found to have significant effect on the yield of LBPAC. Figure 8 shows the three dimensional response surfaces which was constructed to show the interaction effects of the impregnation concentration and activation temperature on the yield of LBPAC.

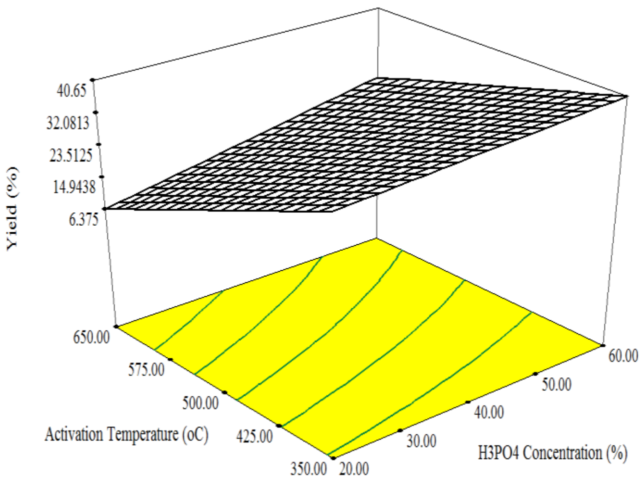


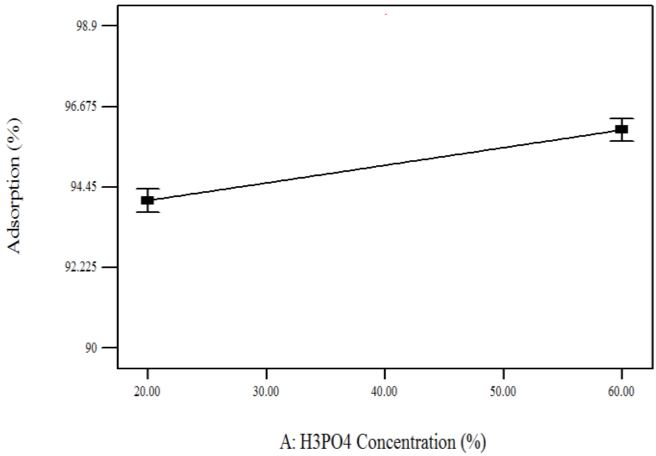
Figure 8: 3D Response Surface Plot of LBPAC Yield (Effect of impregnation concentration and activation temperature, time = 30 min)

The results obtained were in agreement with the workdone by AhmadAlrozi (2010) where activation temperature was found to have the greatest effect on the yield of activated carbon whereas impregnation ratio and activation time were less significant compared to activation temperature. Overall weight loss was found to increase with increasing temperature as a result of intensifying dehydration and elimination reactions (Tan *et al.*, 2008). An increase in activation temperature would release increasing volatiles from the cross-linked framework generated by activating agent (H_3PO_4) used, thereby resulting in decrease of the activated carbon yield (Mi *et al.*, 2015). The yield was positively affected by the impregnation concentration where increasing the impregnation concentration increased the carbon yield. This increase is most likely due to the formation of large molecules in the structure of activated as a result of condensation (polymerization) reactions promoted usually by the effect of the chemical agent (Sahu *et al.*, 2010).

3.5 Adsorption of Methylene Blue

For methylene blue adsorption, among all the factors being considered, activation temperature with highest F-value of 9604 was found to impose the greatest effect on the adsorption (see Table 5 and Figure 6).

(a)



(b)

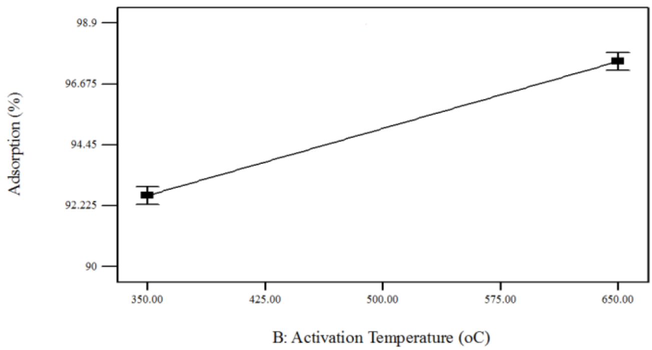


Figure 9 Effect of main factors on methylene blue adsorption (a) H_3PO_4 concentration, % (b) Activation temperature, ^oC

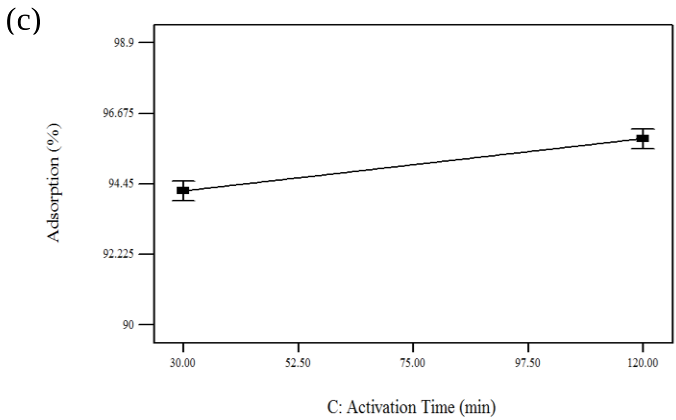


Figure 9 Contd
(c) Activation time, min

The effect of the factors on methylene blue adsorption were studied and all the three variables had positive effect on MB adsorption. (Figures 9). From Figure 9c, it can be observed from the nature of the slope of the plot that the activation time exerted the least effect on MB adsorption.

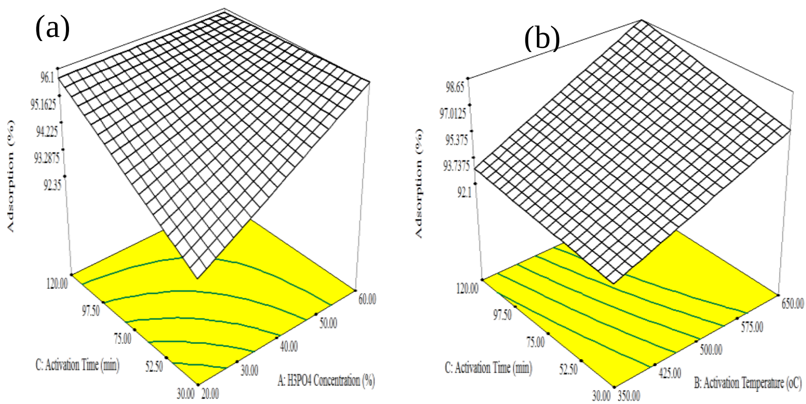


Figure 10: 3D Response surface plot of methylene blue adsorption (a) Effect of impregnation concentration and activation time, temp = 500 ^oC (b) Effect of temperature and activation time, impregnation conc. = 60%)

Figure 10 shows the three-dimensional response surfaces of the two interaction terms (AC and BC) that were found to have significant effect on MB adsorption (see Figure 6). Figure 10a shows the response surface plot for the combined interaction effects of impregnation concentration and activation time at constant activation temperature while Figure 10b shows the three – dimensional surface plot for the combined interaction of activation temperature and activation time at constant impregnation concentration. A maximum adsorption of methylene 98.9% was determined at constant impregnation concentration of 60%.

3.6 Process Optimization

The economic viability of the production of commercial activated carbons depends on the carbon yield and the adsorption capacity. One of the main goal of this study was to find the optimum process conditions at which LBP activated carbons produced should have high carbon yield and a high methylene blue adsorption. However, as a result of the differences in the interest region of factors, it is difficult to optimize both these responses under the same conditions. For instance, when the adsorption capacity increased, carbon yield will decrease and vice versa.

Therefore, process optimization was carried out using Design Expert software version 6.0.8 (STAT-EASE Inc., Minneapolis, USA) in order to compromise between these two responses. The best solution with the highest desirability for optimization within the experimental range of the studied independent variables was selected and verified (Adewoye *et al.*, 2017; Araromi *et al.*, 2017).

The optimum LBPAC was obtained by using H_3PO_4 concentration (60%), activation temperature (444.4 ^oC) and activation time (30 min) resulting in 95.4% removal of methylene blue and 41.6% of activated carbon yield.

3.7 Validation of Model

The model validation was carried out under predicted conditions by the developed model and the predicted values were found to agree satisfactorily with the experimental values (Table 6), with an error of 1.56% and 1.81% for the activated carbon yield and methylene blue adsorption, respectively.

Table 6 Model validation

H_3PO_4 Conc., A (%)	Activation Temp. B (°C)	Activation Time, C (min)	LBPAC Yield, Y_1 (%)		MB Adsorption Y_2 (%)	
			Predicted	Experimental	Predicted	Experimental
60	444.4	30	41.60	42.26	95.40	96.75

3.8 Characterization of Optimally Prepared AC

3.8.1 Surface Functional Groups

The FTIR spectra of the locust bean pod and prepared activated carbon are shown in Figure 11. From the spectrum obtained for raw locust bean pod, the band at 2929.82 cm^{-1} was attributed to $(C-H)$ in methyl vibrations group. The sharp band located around 2370 cm^{-1} was ascribed to $(C\equiv N)$ stretch. The peaks at 1713.98 cm^{-1} and 1036.41 cm^{-1} are associated with the $(C=O)$ and $(C-O)$ vibrations group. The FTIR spectrum obtained for the activated carbon as shown in Figure 11 had its $(O-H)$ vibrations in hydroxyl groups at broad peak of 3008.97 cm^{-1}. A medium peak assigned as alkyne with stretches of $(C\equiv C)$ was found around 2350.12 cm^{-1}.

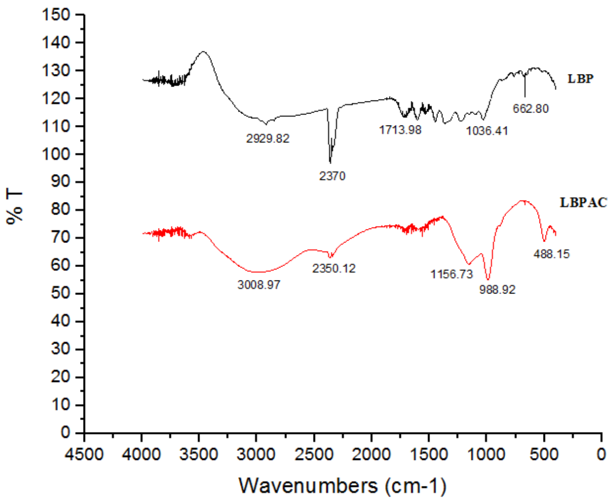
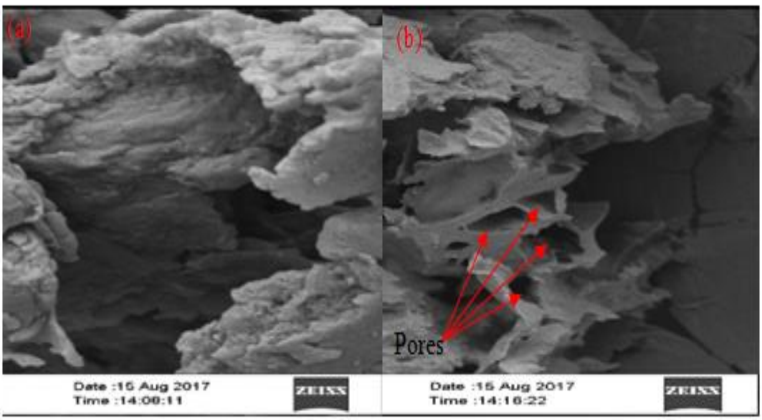


Figure 11 FT-IR for Raw locust bean pod (LBP) and locust bean pod activated carbon (LBPAC)

The band located at about 1690 cm^{-1} was attributed to carbonyl group $(C = O)$ stretching from tertiary amine. The presence of $(O - H)$, alkyne stretches of $(C \equiv C)$, and the carbonyl group $(C = O)$, confirms the presence of carboxylic functional groups in the prepared activated carbon. The $(C - O)$ stretching attributed possibly to phenol was found at 1156.73 cm^{-1}. Absorption due to $(C - H)$ groups occurred at 988.92 cm^{-1}. The numerous functional groups present in the prepared activated carbon are the important sites responsible for the adsorption activities (Adewoye *et al.*, 2017; Mustapha *et al.*, 2017).

3.8.2 Surface Morphology

Figure 12(a) and (b) respectively shows the scanning electron micrograph (SEM) of the raw locust bean pod and the locust bean pod activated carbon prepared at optimum conditions. Very few pores were observed on the surface of the raw locust bean pod as shown in Figure 12a. However, after phosphoric acid impregnation at optimum preparation conditions, many large pores were developed on the surface of the LBP activated carbon (Figure 12b) resulting in AC with large surface area and high adsorption of methylene blue. NethajiSivasamy (2014) reported similar observation in their work of preparing activated carbon from walnut shell.



(a) (b)

Figure 12: Scanning electron micrographs (a) LBP (b) LBP activated carbon

4. Conclusions

The preparation of activated carbon from locust bean pod, where the effects of H_3PO_4 concentration, activation temperature and activation time on the yield of AC and MB removal from aqueous solution were investigated using Full Factorial Design. It was found that the experimental values obtained for the activated carbon yield and methylene blue adsorption were in close agreement with the predicted values. The optimum AC preparation conditions were obtained using 60% H_3PO_4 concentration, 444.4 ^oC activation temperature and 30 min activation time resulting in 95.4% of methylene blue adsorption and 41.60% of activated carbon yield. Activation temperature was found to exert greatest influence on both the yield of activated carbon and the methylene blue adsorption. Thus, the present study demonstrates the effectiveness of using factorial design approach in determining the optimum conditions for the production of locust bean pod activated carbon.

References

Abechi, S. E., Gimba, C. E., Uzairu, A. and Dallatu, Y. A. (2013). Preparation and characterization of activated carbon from palm kernel shell by chemical activation. Res. J. Chem. Sci., 3(1), 54-61.

Adewoye, L. T., Mustapha, S. I., Adeniyi, A. G., Tijani, J. O., Amoloye, M. A. and Ayinde, L. J. (2017). Optimization of nickel (II) and chromium (III) removal from contaminated water using sorghum bicolor. Nigerian Journal of Technology, 36(3), 960-972.

Ahmad, M. A. and Alrozi, R. (2010). Optimization of preparation conditions for mangosteen peel-based activated carbons for the removal of Remazol Brilliant Blue R using response surface methodology. Chemical Engineering Journal, 165(3), 883-890.

Ahmad, M. A. Puad, N. A. A., and Bello, O. S. (2014). Kinetic, equilibrium and thermodynamic studies of synthetic dye removal using pomegranate peel activated carbon prepared by microwave-induced KOH activation. Water Resources and industry, 6, 18-35.

Alam, M. Z., Ameem, E. S., Muyibi, S. A. and Kabbashi, N. A. (2009). The factors affecting the performance of activated carbon prepared from oil palm empty fruit bunches for adsorption of phenol. Chemical Engineering Journal, 155(1), 191-198.

Ali, I. H. and Alrafai, H. (2016). Kinetic, isotherm and thermodynamic studies on biosorption of chromium (VI) by using activated carbon from leaves of Ficus nitida. Chemistry Central Journal, 10(1), 36.

Araromi, D. O., Alade, A. O., Bello, M. O., Bakare, T., Akinwande, B. A., Jameel, A. T. and Adegbola, S. A. (2017). Optimization of Oil Extraction from Pitanga (Eugenia Uniflora L) Leaves Using Simplex Centroid Design (SCD). Separation Science and Technology(just-accepted).

Auta, M. and Hameed, B. (2011). Optimized waste tea activated carbon for adsorption of Methylene Blue and Acid Blue 29 dyes using response surface methodology. Chemical Engineering Journal, 175(1), 233-243.

Campbell, Q., Bunt, J., Kasaini, H. and Kruger, D. (2012). The preparation of activated carbon from South African coal. Journal of the Southern African Institute of Mining and Metallurgy, 112(1), 37-44.

Cronje, K., Chetty, K., Carsky, M., Sahu, J. and Meikap, B. (2011). Optimization of chromium (VI) sorption potential using developed activated carbon from sugarcane bagasse with chemical activation by zinc chloride. Desalination, 275(1), 276-284.

Demirbas, A. (2008). Heavy metal adsorption onto agro-based waste materials: a review. Journal of Hazardous Materials, 157(2), 220-229.

Eletta, O. A. A., Ajayi, O. A., Ogunleye, O. O. and Akpan, I. C. (2016). Adsorption of cyanide from aqueous solution using calcinated eggshells: Equilibrium and optimisation studies. Journal of Environmental Chemical Engineering, 4(1), 1367-1375. doi:https://doi.org/10.1016/j.jece.2016.01.020

Farayola, C., Okpodu, V. and Oni, O. (2013). Economic Analysis of Locust Beans Processing and Marketing in Ilorin, Kwara State, Nigeria. International Journal of Agricultural Research, Innovation and Technology, 2(2), 36-43.

Garba, Z. N., Bello, I., Galadima, A, and Lawal, A. Y. (2016). Optimization of adsorption conditions using central composite design for the removal of copper (II) and lead (II) by defatted papaya seed. International Journal of Modern Science, 2(1), 20 - 28. doi:10.1016/j.kijoms.2015.12.002

Gottipati, R. (2012). Preparation and characterization of microporous activated carbon from biomass and its application in the removal of chromium (VI) from aqueous phase. (PhD. Thesis), Department of Chemical Engineering, National Institute of Technology, Rourkela, Odisha, India.
.
Gottipati, R. and Mishra, S. (2010). Process optimization of adsorption of Cr(VI) on activated carbons prepared from plant precursors by a two-level full factorial design. Chemical Engineering Journal, 160(1), 99-107. doi:http://dx.doi.org/10.1016/j.cej.2010.03.015

Guerrero-Coronilla, I., Morales-Barrera, L. and Cristiani-Urbina, E. (2015). Kinetic, isotherm and thermodynamic studies of amaranth dye biosorption from aqueous solution onto water hyacinth leaves. Journal of environmental management, 152, 99-108.

Hashemian, S., Salari, K. and Yazdi, Z. A. (2014). Preparation of activated carbon from agricultural wastes (almond shell and orange peel) for adsorption of 2-pic from aqueous solution. Journal of Industrial and Engineering Chemistry, 20(4), 1892-1900.

Ioannidou, O. and Zabaniotou, A. (2007). Agricultural residues as precursors for activated carbon production—a review. Renewable and Sustainable Energy Reviews, 11(9), 1966-2005.

Islam, M. A., Tan, I., Benhouria, A., Asif, M. and Hameed, B. (2015). Mesoporous and adsorptive properties of palm date seed activated carbon prepared via sequential hydrothermal carbonization and sodium hydroxide activation. Chemical Engineering Journal, 270, 187-195.

Isoda, N., Rodrigues, R., Silva, A., Gonçalves, M., Mandelli, D., Figueiredo, F. C. A. and Carvalho, W. A. (2014). Optimization of preparation conditions of activated carbon from agriculture waste utilizing factorial design. Powder Technology, 256, 175-181.

Kyzas, G. Z. and Deliyanni, E. A. (2015). Modified activated carbons from potato peels as green environmental-friendly adsorbents for the treatment of pharmaceutical effluents. Chemical Engineering Research and Design, 97, 135-144.

Martins, A. C., Pezoti, O., Cazetta, A. L., Bedin, K. C., Yamazaki, D. A., Bandoch, G. F. and Almeida, V. C. (2015). Removal of tetracycline by NaOH-activated carbon produced from macadamia nut shells: kinetic and equilibrium studies. Chemical Engineering Journal, 260, 291-299.

Mi, T., Chen, L., Xin, S.-Z., and Yu, X.-m. (2015). Activated carbon from the Chinese herbal medicine waste by H3PO4 activation. Journal of Nanomaterials, 2015.

Mohammed, I., Bankole, M., Abdulkareem, A., Ochigbo, S., Afolabi, A. and Abubakre, O. (2017). Full Factorial Design Approach to Carbon nanotubes synthesis by CVD method in Argon environment. South African Journal of Chemical Engineering.

Mondal, S., Sinha, K., Aikat, K. and Halder, G. (2015). Adsorption thermodynamics and kinetics of ranitidine hydrochloride onto superheated steam activated carbon derived from mung bean husk. Journal of Environmental Chemical Engineering, 3(1), 187-195.

Mustapha, S. I., Adewoye, L. T., Aderibigbe, F. A., Alhaji, M. H., Adekola, M. I. and Tijani, I. A. (2017). Removal of Lead and Chromium from Aqueous Solution onto Flamboyant (Delonix regia) Pod Activated Carbon. Nigerian Journal of Technological Development, 14(2), 56-66.

Nethaji, S. and Sivasamy, A. (2014). Removal of hexavalent chromium from aqueous solution using activated carbon prepared from walnut shell biomass through alkali impregnation processes. Clean Technologies and Environmental Policy, 16(2), 361-368. doi:10.1007/s10098-013-0619-1)

Njoku, V., Foo, K., Asif, M. and Hameed, B. (2014). Preparation of activated carbons from rambutan (Nephelium lappaceum) peel by microwave-induced KOH activation for acid yellow 17 dye adsorption. Chemical Engineering Journal, 250, 198-204.

Olapade-Ogunwole, F., Olawuyi, S. and Akinniran, T. (2011). Economic analysis of locust bean processing and marketing in Iwo local government, Osun state. International Journal of Applied Agriculture and Apiculture Research, 7(1), 54-63.

Rashid, U., Anwar, F., Ashraf, M., Saleem, M. and Yusup, S. (2011). Application of response surface methodology for optimizing transesterification of Moringa oleifera oil: Biodiesel production. Energy conversion and Management, 52(8), 3034-3042.

Reza, R. A., Ahmaruzzaman, M., Sil, A. K. and Gupta, V. K. (2014). Comparative adsorption behavior of ibuprofen and clofibric acid onto microwave assisted activated bamboo waste. Industrial and Engineering Chemistry Research, 53(22), 9331-9339.

Rivera-Utrilla, J., Sánchez-Polo, M., Gómez-Serrano, V., Alvarez, P., Alvim-Ferraz, M. and Dias, J. (2011). Activated carbon modifications to enhance its water treatment applications. An overview. Journal of Hazardous Materials, 187(1), 1-23.

Sahu, J., Acharya, J. and Meikap, B. (2010). Optimization of production conditions for activated carbons from Tamarind wood by zinc chloride using response surface methodology. Bioresource technology, 101(6), 1974-1982.

an, I., Ahmad, A. and Hameed, B. (2008). Optimization of preparation conditions for activated carbons from coconut husk using response surface methodology. Chemical Engineering Journal, 137(3), 462-470.

Yahya, M. A., Al-Qodah, Z. and Ngah, C. Z. (2015). Agricultural bio-waste materials as potential sustainable precursors used for activated carbon production: a review. Renewable and Sustainable Energy Reviews, 46, 218-235.

Zhou, Y., Zhang, L and Cheng, Z. (2015). Removal of organic pollutants from aqueous solution using agricultural wastes: a review. Journal of Molecular Liquids, 212, 739-762.