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Activated carbon prepared from Shea Butter Husk by Box-Behnken response surface methodology

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Abstract

Activated carbon was prepared from shea butter husk with Potassium chloride (KCl) used as activating reagent. Box-Behnken design (BBD) tool, a subset of the Response Surface Methodology (RSM) was use to optimize the preparation parameters. The optimized variables used for activation of char obtained from shea butter husk after carbonization, were activation time, activation temperature and concentration of Potassium chloride (KCl) used. While iodine value was used as responses. The optimum iodine value of 1244.17mg/g was obtained at the best activating conditions of 600 ^{o}C, 90 min and 2.0M concentration. A desirability of 0.90 was obtained. The predicted results are in close range with the experimental results. The raw and prepared activated carbon were analyzed with the use FTIR and SEM. This study is important for cost-effective large scale activated carbon preparation for heavy metals treatment with the smallest amount of chemical usage and energy contribution.

Keywords: Char, Shea butter husk, activated carbon, Box-Behnken design, preparation, response surface methodology.

Introduction

One of the extensively used materials as adsorbent in many industry is activated carbon. Activated carbon also called activated charcoal are carbonaceous material that possess large surface area and high porosity[1]. The large specific surface area, large micro porosity with ample pore size distribution possessed by activated carbon makes it feasible to let large amount of adsorption[1]. Activated carbon has several applications such as removal of unwanted colour, taste, odour, air purification and for treatments of other impurities from domestic and industrial wastewater[2]. It is also used in the purification of several food products, chemicals and pharmaceuticals; and in several gas-phase applications[3].

Lately, because of its environmental benefits, activated carbon has turn out to be more attractive. Therefore, it becomes imperative to utilize inexpensive material source for activated carbon production. Currently, the use of low-cost agricultural and forestry wastes used for activated carbon production has drawn widespread interest, due to the profusion and accessibility of these agricultural wastes; making it a great source of raw materials for sorbents[4].

Several numbers of studies have disclosed that an ample number of waste materials from agriculture have been utilized for activated carbon making, such as walnut shells[5], barley husks[6], coconut shells[7], palm shells[8], rice husks[9], apricot stones[10], groundnut shell[11], durian shell[12], corn cob[13], rice bran[14], olive seed[15], jackfruit peel waste[16], Jatropha seed coat[17] and Tunisian olive waste cakes[18].

In this current study, a cheaply sustainable material, shea butter husk was selected and used to produce activated carbon. Shea butter tree (Vitellaria paradoxa), is a native tree species found in several countries in Sub-Saharan Africa. Shea butter tree belong to the family of Sapotaceae, mainly found in regions with about 400-1800 mm rainfall per year. Shea butter trees are abundant in Nigeria with an approximate output of two hundred and fifty thousand metric tonnes per annum[19]. Commercially shea butter is used as an ingredient in pharmaceutical, cosmetic and for household purposes such as skin moisturizer and for cooking[20]. Conventionally, it is used, for protecting the skin from harsh weather and sun, used as cream for dressing hair, used to ease aching and joint pains, used for curing swellings/wounds/ bruising and used for massaging children and pregnant women. Shea butter is also used for rashes, burns, ulcers, dermatitis and eczema treatment[21]. The numerous uses of shea butter makes the husk abundantly available to be converted into a useful material such as activated carbon.

In preparation of activated carbon two basic methods are widely employed, which includes the chemical and physical method[22]. The chemical and physical methods are accountable for the various sizes and shapes of activated carbon[23]. A two-step process is involved in the physical method of preparation of activated carbon, which consist of carbonization and subsequently activation of obtained char with carbon dioxide or steam. The carbonization temperature range in physical method is between 400-850^{o}C.While the chemical method involves a single step process, in which activating reagents are used for impregnation of the precursor material and heated under an inert atmosphere at 600-900^{o}C[24].

Activating agents like H_3PO_4, $ZnCl_2$, KOH, NaOH, K_2CO_3 and KCI are used for activation. Potassium chloride is preferred amidst the numerous available activating agents, due to less environmental pollution by Potassium chloride. The primary aim of this present work is to use shea butter husk for preparation of activated carbon using KCI as activator.

The RSM was engaged for optimization of the process preparation condition of activated carbon. RSM is generally used as a statistical approach, to learn of the interactive effects between multiple single factors[25]. Furthermore, the lesser number of experimental runs required for evaluation of several parameters and their interactions is a major advantage of RSM[26].

To achieve the most favorable processing conditions for shea butter husk–based activated carbon (SBAC), the BBD, a subset of RSM was employed. For this study, shea butter husk was chosen as precursor to be used and carbonization was done based on temperatures gotten from the thermo gravimetric analysis of the raw shea butter husk.

Materials and methods

Materials: Husk of shea butter were obtained from Kpakungu in Minna, Niger State, Nigeria. Ordinary tap water was used to wash the husk samples, to take out possible foreign materials present (sands and dirt). The washed samples were exposed to sunlight and dried for 2-5 days. The sundried samples were crushed with a Mechanical Crusher to diminish the size and sieved.

Figure-1 shows the method employed for activated carbon preparation from shea butter husk.

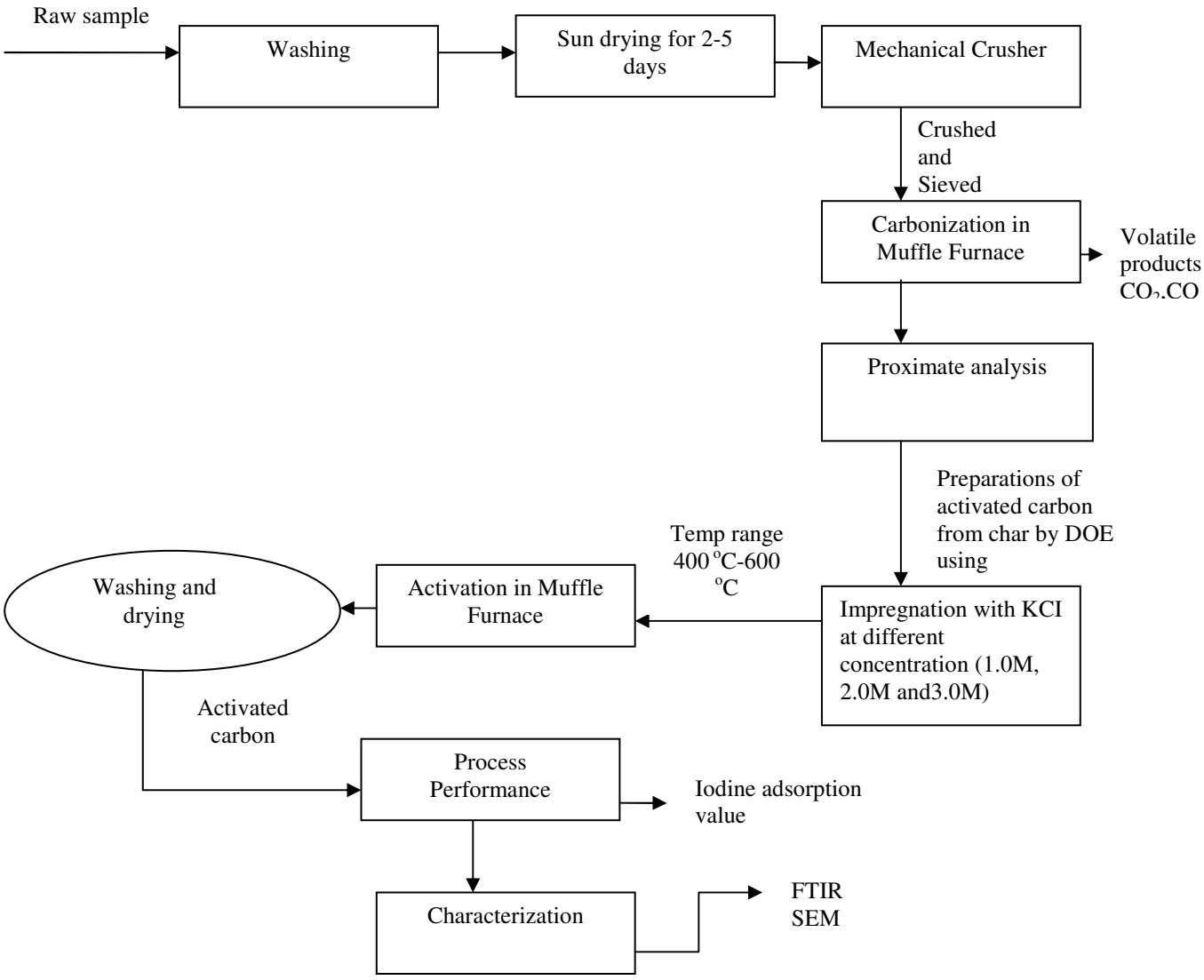

Figure-1: Method employed for activated carbon preparation from shea butter husk.

Activated carbon preparation: The crumpled shea butter husk samples were placed in a clean crucible and placed in a muffle furnace, subjected to carbonization at different temperatures. Classical method of optimization was used to get the best temperature for carbonization at a constant time of 15 min and afterward time was optimized from 15 min to 60 min to attain the best time for carbonization. 25 grams of the char obtained after carbonization was mixed in 50ml of potassium chloride solution prepared at concentrations and allowed for 24hours at room temperature to soak. The obtained samples were oven dried for 30min at 100^{o}C. The dried samples were transferred into a muffle furnace and activated, at temperatures and time determined by the design of experiment (Box-Behnken design) at 5C/min heating rate. The obtained sample were transferred into a desiccator to cool. Thereafter the sample was carefully rinsed using 0.1 M of HCI and distilled water to eliminate the residual salt present, until the pH of filtrate reached 7.

Proximate analysis: The proximate analysis of the obtained char from shea butter husk was carried out.

Moisture content: The thermal drying method was hired to determine the moisture content of the sample. 1.0g of the obtained char sample derived from Shea butter husk was transferred to a dried crucible. The content of crucibles was taken in to an air tight oven and dried at 105^{o}C for 4h. The moisture content of char sample derived from shea butter husk was evaluated with Equation-1.

$$M.C = \frac{W_i - W_f}{W_i} x\ 100 \quad (1)$$

Where, *M.C*= Moisture content of char sample derived from shea butter husk, W_i= initial weight of char before heating and W_f = final weigth of char after heating.

Ash Content: This was performed in accordance to the ASTM D 2017 (1998). 0.3 g of the obtained char from shea butter husk was put into in a weighed crucible, which was then moved into a muffle furnace were it was incinerated at 760^{o}C for 1 h. It was then transferred into a desiccator and allowed to cool. The cooled sample was then weighed. Equation-2 was utilized to compute the ash content of char obtained from shea butter husk.

$$A.C = \frac{W_a}{W_c} x\ 100 \quad (2)$$

Where: A.C$_=$ Ash content, Wa = Weight of ash and Wc = Weight of char.

Volatile content: The volatile matter content of char derived from shea butter husk was determined by measuring 1.0g of char and transferred to a clean crucible and heated at 850^{o}C in a muffle furnace for 10 min. The crucible was removed from the muffle furnace and moved into a desiccator to cool. The percentage volatile content (VC) was calculated with Equation-3.

$$V.C = \frac{W_o - W_d}{W_o} x\ 100 \quad (3)$$

Where, V.C = Volatile matter content, Wo = original weight of char and Wd = difference in weight (g).

Fixed carbon content: The ASTM D 121 (2001) method was adopted in determination of fixed carbon content of char from shea butter husk. The summation of moisture content, volatile content and ash content was deducted from 100, to give the fixed carbon content of the sample.

Char yield: Final weight of sample after carbonization of sample divided with initial weight of sample to give the char yield. The char yield is expressed in Equation-4.

$$Y = \frac{W_{ck}}{W_o} x\ 100 \quad (4)$$

Where, y = char yield, w_o = original sample weight before carbonization and w_{ch} = weight of final product after carbonization.

Process performance: The activated carbon obtained was evaluated base on its adsorption capacity via the iodine number.

Iodine number: The iodine value was estimated using the procedure of the ASTMD 4607-94 (2006). 1.0 g of the obtained activated carbon was weighed and transferred into a dry, clean Erlenmeyer flask. A pipetted 10 ml of5 wt % of HCI was added to the clean Erlenmeyer flask. To destroy sulphur which might meddle with the results, the substance in the flasks were boiled mildly for 30s. The content of the flasks was then left to cool. 100 ml of 0.1 N iodine solution was measured and added to the substances in the flasks. It was then shook briskly for 30s. The substances in the flasks were filtered off. 50 ml of the filtrate was measured and transferred into an Erlenmeyer flask. 0.1 N sodium thiosulphate solutions was used to titrated against 50 ml of the filtrate till the solution was pale yellow. 2ml of starch indicator was measured and added to the pale yellow solution and titration with sodium thiosulfate was further continued on till the solution turned colourless. The sodium thiosulphate volume used for the whole titration process was noted and the quantity of iodine adsorbed for each gram of activated carbon dosage was worked out using Equation-5.

$$\frac{Y}{M} = \frac{(A-((DF)(B)(S))}{M} \quad (5)$$

$A = (N_1) \times (12693)$
$B = (N2) \times (126.93)$
$DF = (1 + H) / F$

Where, the iodine adsorbed for each gram of actuated carbon is represented by X/M (mg/g), DF represent the dilution factor, S represent the sodium thiosulfate volume utilized (ml), M is the quantity of activated carbon utilized (g), the normality of iodine

(N), is represented by N_1,sodium thiosulphate normality is represented by N_2, I is volume of iodine used (ml), H represent the 5% of HCI utilized (ml), and F is the filtrate (ml).

Experimental design: To get the best conditions for carbonization of shea butter husk, single-factor optimization method was employed. After production of char from shea butter husk with single-factor experimental method, the BBD experimental method was further used for optimization of the activation process. The software used for the statistical analysis was Minitab Release (Version 17). For development of BBD, the required number of experiments (N) was expressed by Equation-6.

$$N = 2k (k - 1) + C_0 \quad (6)$$

Where k represents the number of factors and C_0 stands for the number of central points.

The predicted response (Y) is expressed by the model equation given in equation 7.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} (x_i)^2 + \sum_{i=1}^{k-1} \sum_{j=2}^{k} \beta_{ij} x_i x_j \quad (7)$$

Where β_0, is the model constant term when x_i is zero for each parameter, β_i is linear term, β_{ii} represent the quadratic term, β_{ij} represent the interactive term, while xi and x_j are variables of parameters affecting the process being treated). In this work, BBD was utilized to estimate the interactions and effects of process parameters (carbonization temperature, concentration used for activation and activation time). The levels and ranges of the independent variables are shown in Table-1.

Characterization of shea butter husk and activated carbon:
TGA analysis: The thermal behavior of the raw shea butter husk was studied by the thermogravimetric analysis (TGA). TGA 4000 Perkin-Elmer thermal analyzer was used. The weight loss of the shea butter husk as a function of temperature was measured by the thermal analyzer. The shea butter husk was subjected to pyrolysis in the influence of N_2 flow (35ml/min) at a heating rate of 20^0C/min.

Table-1: BBD factors and levels used for activation.

Factor	Lower level (-1)	Middle level (0)	Upper level (+1)
Temperature (^{o}C)	400	500	600
Concentration (M)	1	2	3
Time (Min)	30	60	90

FTIR analysis: Shimadzu 8300 spectrometer was used to perform the spectral analysis using the potassium bromide pellet technique in the range of wave number of 400-40001/cm.

SEM analysis: The surface morphology of the shea butter husk and activated carbon was obtained using Scanning electron microscopic (JEOL JSM-6300F). A gold sputtering device was used to coat the samples with gold for the purpose of conductivity and clear visibility. Afterward, the coated sample were placed on the sample holder for analysis.

Results and discussion

TGA analysis: Figure-2 shows the obtained TGA result and summarized in Table-2.

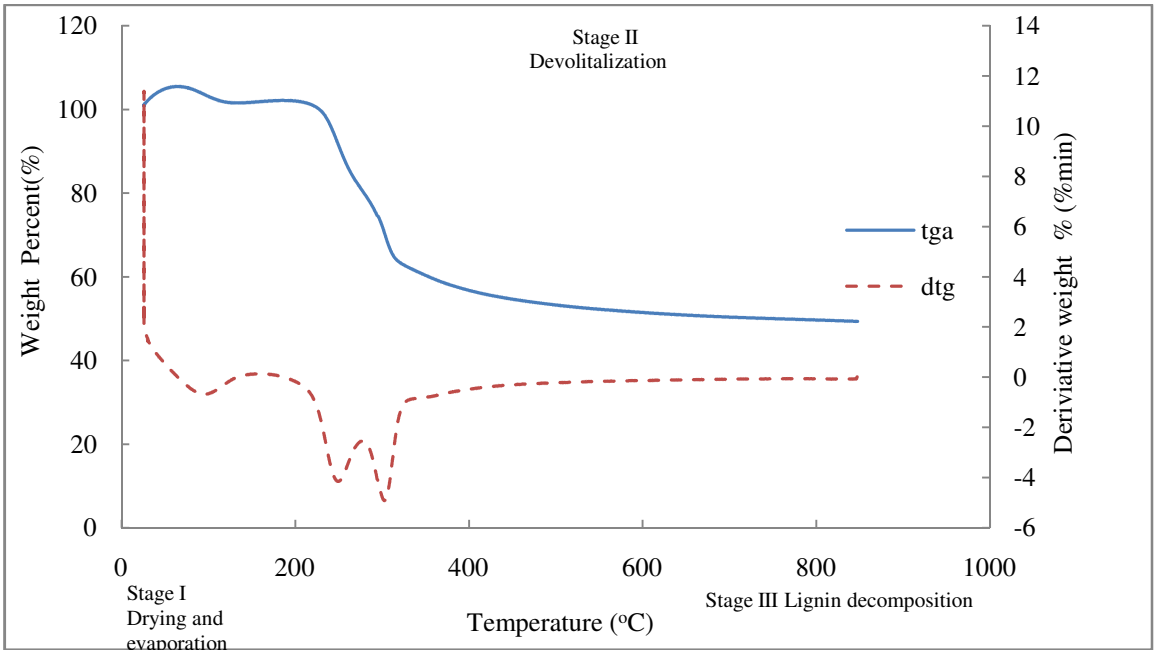

Figure-2: The thermo gravimetric analysis of raw shea butter husk.

The first stage of decomposition was from 26.06-124.05°C; with 1.96% weight loss, which indicates the removal of moisture from shea butter husk. Exothermic reactions take place at the second stage, with the removal volatiles (devolatization) from cellulose, hemicelluloses and partially lignin. This is the key step in conversion of the wastes to carbon, a notable weight loss occurs at this stage. The degradation temperature range for shea butter husk was from 124.50-345.61°C, as shown in Figure-2; with 41.78% weight loss recorded.

The second stage involves the removal of volatiles (devolatization) by the swift thermal breakdown of hemicelluloses, cellulose and partly lignin. A prominent weight loss take place at this stage. The degradation temperature range for shea butter husk was from 124.50-345.61°C, as shown in Figure-2; with 41.78% weight loss. The derivative peak temperature (Tp) depicted via the DTG curve was at 297.90°C. Endothermic decomposition of lignin occurred at the third stage, which took place at a broader temperature range (beginning from around 250-800^{o}C), as a result of steady decomposition of the lingering heavy component primarily from lignin.

Holding to the fact that moisture content was mainly removed from the first stage of TGA curve, with notable loss weight loss occurring at stage two and lesser weight loss at the third stage of TGA curve, a temperature range of 300^{o}C-600^{o}C was chosen for carbonization of shea butter husk as shown in Table-3.

Influence of carbonization temperature and time on char obtained from Shea butter husk: The quantity and quality char produced are affected by carbonization temperature and time. The influence of carbonization temperature and time on char produced from shea butter husk is presented in Tables-3 and 4.

The release of volatile matter present in shea butter to become char with primarily fixed carbon occur due to increase in carbonization temperature. At a temperature of 300^{o}C the volatile matter content of 89.83% was obtained from char of shea butter husk, which further decreased to 44.40% as temperature increased to 600^{o}C.

The percentage yield is also a very important parameter that tells the extent of carbonization process. Increase in carbonization temperature lead to decreases in percentage yield of char. The char yield obtained from shea butter husk at 300^{o}C was 59.70% which was reduced to 21.60% as carbonization temperation increased to 600^{o}C. However, the highest fixed carbon content of char obtained from shea butter husk was 25.42% at a temperature of 500^{o}C. Hence the best carbonization temperature of 500^{o}C was chosen and used for production of char. The obtained results was also in agreement with the work of [27] were the fixed carbon of rice husk char produced at 600^{o}C increased from 26.37% to 38.88% at 1000^{o}C.

The quantity of char resulting from shear shea butter husk was reduced with longer carbonization time. Also the shape, pore size and nature char is altered at longer carbonization time, which occur as a result of undue burning/ oxidation and also as a result of collapsing of pore structures. Hence 30 min was picked as the best time for carbonization since the best fixed carbon was obtained at 30mins.

Activation of char produced from shea butter husk: For activation of char derived from shea butter husk, the BBD was used for optimization of the activation process parameters (activation time, activation temperature and concentrations of potassium chloride solution used).

The experiment in the design matrix shown in Table-5 was performed, with iodine value used as response. The obtained iodine value of the developed activated carbon from shea butter husk ranges from 1218.48 to 1244.17 mg/g.

Table-2: TGA analysis of Shea butter husk.

Sample	Temp range of MC (%)	MC (%)	Temp range of deg in stage II	Deg (%) in stage II	T_P
Shea butter husk	26.06-124.50	1.96	124.50-345.61	41.78	297.90

Table-3: Proximate analysis of char from shea butter (CS) produced at different temperature and constant time.

Temp (^{o}C)	T (min)	VC (%)	AC (%)	MC (%)	FC (%)	Y (%)
300	15	89.83	3.01	2.01	5.15	59.70
350	15	83.36	6.00	3.00	7.64	36.00
400	15	79.54	14.56	3.02	2.88	25.70
450	15	72.63	18.00	5.00	4.37	25.10
500	15	50.00	22.48	2.10	25.42	23.40
550	15	48.50	28.50	2.76	20.24	22.72
600	15	44.40	37.20	2.00	16.40	21.60

Table-4: Proximate analysis of char from shea butter husk (CS) produced at different time.

Temp (^oC)	T (min)	VC (%)	AC (%)	MC (%)	FC (%)	Y (%)
500	15	50.00	22.48	2.10	25.42	23.40
500	30	40.15	20.85	3.00	36.00	22.3
500	45	37.90	28.00	3.40	30.70	20.5
500	60	32.72	35.41	2.81	29.06	19.4

Table-5: Experimental design matrix and response.

Runs	Temperature	Concentration	Time	Iodine Number	
	(^oC)	mole	(min)	Experimental Values (mg/g)	Predicted values (mg/g)
1	600	2	30	1227.97	1229.69
2	400	1	60	1218.48	1216.10
3	500	2	60	1230.21	1229.88
4	500	2	60	1229.09	1229.88
5	400	2	90	1224.62	1223.36
6	500	3	30	1226.86	1224.24
7	600	2	90	1244.17	1240.96
8	400	2	30	1221.83	1225.51
9	500	3	90	1227.41	1228.8
10	500	1	90	1223.22	1227.74
11	500	1	30	1224.62	1223.18
12	600	3	60	1226.30	1228.06
13	500	2	60	1230.76	1229.88
14	400	3	60	1225.18	1228.98
15	600	1	60	1235.23	1234.82

Regression model representation: The BBD was used to study the influence of the independent variables as shown in Table-1. The responses (Iodine adsorption value) is shown in Table-5. Minitab Release (Version 17) was employed to carry out the Multiple regression analysis and obtain a polynomial equation (Equation-8).

Equation 8.0, gave the final model equation used for predicted values of iodine number.

$$Y= 1175.9 + 0.0656\ A + 35.64B - 0.483C - 3.89\ B^2 - 0.0391AB + 0.001118\ AC \quad (8)$$

The predicted values were obtained using Equation-8.

Statistical analysis of variance: To define the importance and fitness of the model, ANOVA via BBD was carried out as shown in Table-6.

The model terms A, B^2 and AB in Table-6 were statistically significant, while B, C and AC are insignificant. The R^2 value of the model response (Y) was 0.8635, suggesting that the equation had a significant degree of fit of the mode. The predicted versus actual plots for response Y is shown in Figure-3.

Table-6: ANOVA for the regression equation.

Source	Sum of squares	Degree of freedom	Mean square	F-Value	P-Value
Model	443.181	6	73.864	8.44	0.004*
Linear	280.522	3	93.507	10.68	0.004*
A-Temp	237.184	1	237.184	27.09	0.001*
B-Conc	2.205	1	2.205	0.25	0.0629
C-Time	41.132	1	41.132	4.70	0.062
Square	56.628	1	56.628	6.47	0.035
B^2	56.628	1	56.628	6.47	0.035*
2-way interaction	106.031	2	53.016	6.06	0.025*
AB	61.074	1	61.074	6.98	0.030*
AC	44.957	1	44.957	5.13	0.053
Error	70.045	8	8.756		
Lack of fit	68.596	6	11.433	15.78	0.061
Pure Error	1.449	2	0.724		
Total R^2	513.226 0.86	14			

Significant at $p < 0.05$.

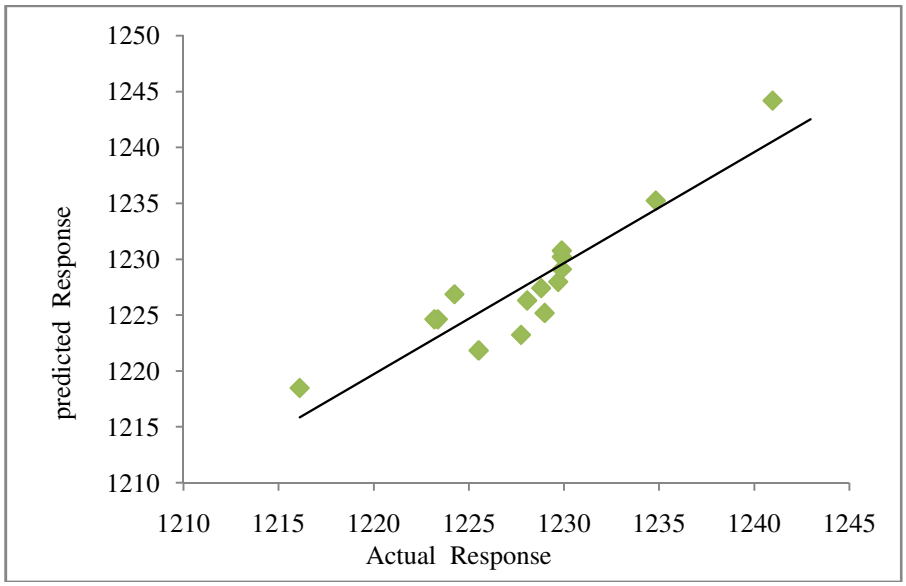

Figure-3: Comparison between actual response and predicted response for iodine adsorption value.

From the plot shown by Figure-3, majority of the data points were closely disseminated close to the straight line, which signified a remarkable link amongst the experimental and predicted values of the responses.

Effect of interaction of factors on response: The collaborating impact of activation temperature, activation time and concentration of KCl used for activation was further discussed by means of a three-dimensional respond surface (3D) on the obtained activated carbon from shea butter husk at optimum conditions (600, 2.0M and 90mins), which was achieved with two parameters varied at a time, while the other variable was kept constant. The interaction between activation temperature (A) and activation time(C) with iodine value as a response at constant activation concentration of 2.0M is demonstrated in Figure-4(a).The iodine value increased with increase in activation temperature from 400^{o}C to 600^{o}C as time increased beyond 80mins.

Figure-4(b) showed the significant interaction between concentration of KCl used for activation(B) and activation time(c) with activation temperature held constant (at 600^{o}C). The iodine value increased when the activation concentration increased up to 2.0M, with an increase in activation time (beyond 80min), while activation at higher concentration of 3.0M with KCI, caused wider opening of the pores on the activated carbon, which led to lower iodine value at 3.0M. Figure-4(c) shows the interaction between activation temperature (A) and concentration of KCl used for activation (B). The iodine value increased with increase in concentration up to 2.0M as temperature rises from 400^{o}C-600^{o}C.

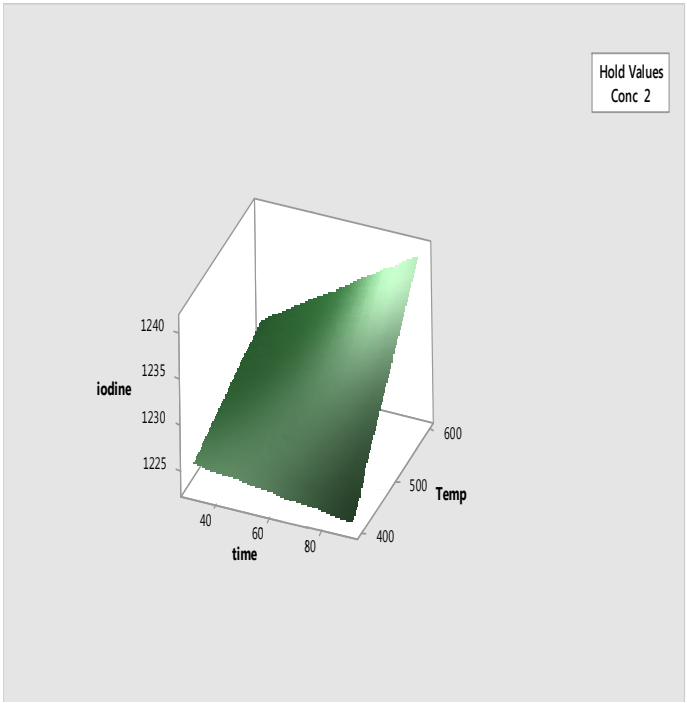

Figure-4a: Effect of activation temperature and activation time.

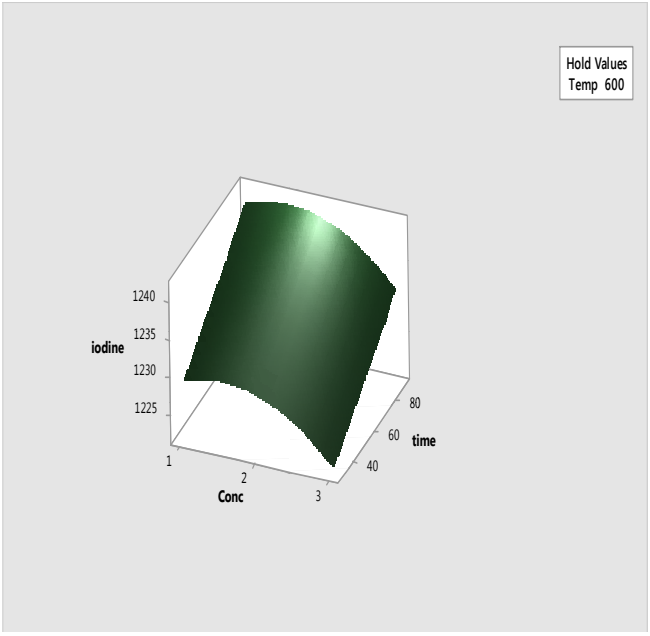

Figure-4b: Effect of activating agent concentration and activation time.

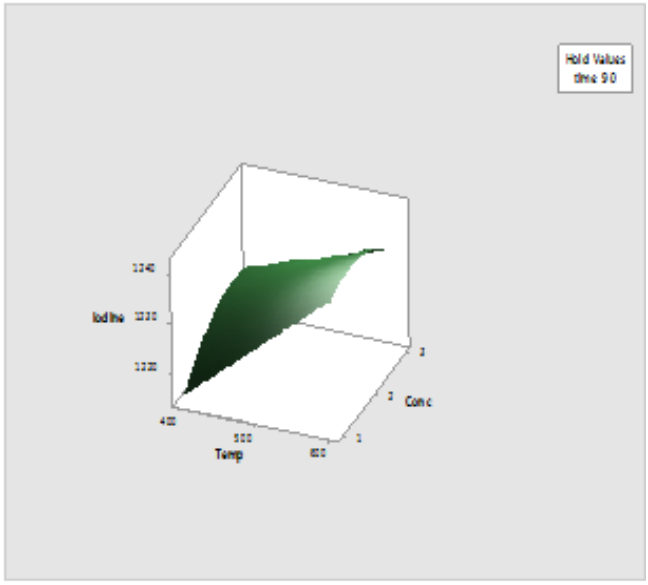

Figure-4c: Effect activation temperature and activating agent concentration at contant activation time (90min).

Characterization of samples: Surface morphology: The raw shea butter husk and activated carbon produced at optimum condition was viewed using scanning SEM as shown in Figure-5(a) and (b). The SEM results of the raw husk surface showed no pores but the activated carbons produced, contained pores. Creation of the pores present in activated carbon was due to metallic potassium formation (K) as a result of activation with KCl. Through the activation process, the C–KCl reaction rate was increased, leading to carbon 'burn off, hence developing good pores on the sample, which will more surface area available for adsorption.

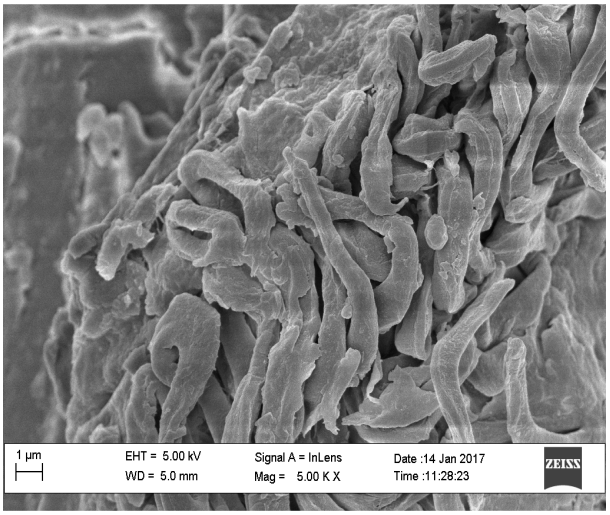

Figure-5a: SEM image of shea butter husk.

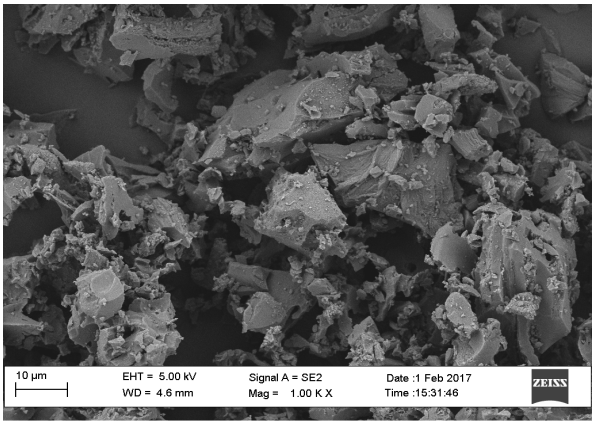

Figure-5b: SEM image of activated carbon.

Conclusion

Activated carbon was prepared from shear butter husk by means of chemical activation using KCI as activating agent. The BBD of RSM was employed for optimization of activation parameters (activation temperature, activation concentration and activation time). Iodine adsorption value was selected as the response. The best conditions of activated carbon obtained from char of shea butter husk were activation temperature of 600^{o}C, 2.0M concentration of KCl used for activation and activation time of 90 min; which possessed the highest iodine adsorption value of 1244.17mg/. The experimental iodine values are in close accord with predicted iodine values obtained from the model. This research established that shea butter husk can be employed for preparation of activated carbon.

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