



Application of Response Surface Methodology for Preparation of Activated carbon from Melon Husk

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ABSTRACT

This study focuses on activated carbon preparation from melon husk via chemical activation using Box-Behnken design (BBD) of Response Surface Methodology (RSM). Potassium chloride (KCl) was used as activating reagent and it played an important role in enhancing the activated carbon porosity. Three input parameters and one response were evaluated via the software generated experimental design. The effects of three preparation parameters for activation of char produced from melon husk, which includes: activation temperature, activation time and activation concentration as well as one responses iodine number were investigated. The optimum conditions for preparation of activated carbon from char obtained from melon husk was at a activation temperature of 500 °C, activation time of 90 min and 1.0M activation concentration, which resulted in 1251.43mg/g of iodine number with desirability of 0.82. The predicted results were well corresponded with experimental results. This study is important in economical large scale activated carbon preparation for application of adsorption process for metal treatment in wastewater with minimum chemical and energy input.

Keywords: Activated carbon, Box-Behnken design, melon husk, Preparation, Response surface methodology

1 INTRODUCTION

Toxic metals, are dispersed to a large extent into the environment, as a result of industrial and technological expansion, population explosion and increased energy utilization through industrial effluents, organic wastes, refuse burning, and transport and power generation (Mahurpawar, 2015). The release of these heavy metals has rendered many waters unwholesome and posses an enormous threat to the environment and public health because of their toxicity, bioaccumulation in the food chain and persistence in nature (Mahurpawar, 2015). The menace of these heavy metals has resulted in the pressing need for effective removal. A number of technologies have been developed over the years to remove heavy metals from wastewaters, which include coagulation/flocculation process, membrane filtration, oxidation process, adsorption, reverse osmosis, ion exchange, solvent extraction (Tamrakar and Nair, 2017). These methods have several disadvantages like high cost, incomplete removal, low selectivity and high-energy consumption. Furthermore, among the mentioned treatment technologies, adsorption using activated carbon had been reported as the most technically and economically viable option (Onundi et al., 2010).

Activated carbon (AC) is defined as a carbonaceous material with large surface area and high porosity. The Iodine number is an imperative parameter to indicate the quality of carbon porosity by measuring the amount of iodine adsorbed (in milligrams) by 1 g of carbon using standard method of ASTM D4607-94. The percentage yield in activated carbon preparation plays a major role in continuous supply for application (Nurul-Shuhada *et al.*, 2016). The surface area and pore size distribution are essential factors in determination of AC performance. Activated carbons are made up of three major pores which include the macropores, mesopores and micropores (Guo and Lua, 2003). The macropores act as the entrance to the AC, while the mesopores and micropores are responsible for adsorption process determined by iodine number (Nurul-Shuhada *et al.*, 2016). Activated carbon (AC) has several applications, one of which is as an adsorbent for purification of water, air and many chemical products (Ash *et al.*, 2006).

A number of studies have revealed that a variety of agricultural waste materials have been used for the production of activated carbon such as such as corn cob (Tsai et al, 2001), hazelnut shell (Orkun et al, 2012) Jojoba seed (Tawalbeh et al, 2005), coconut shell (Olafadehan et al, 2012), hazelnut bagasse (Dermiral et al,2008), rice husk (Yalcin and Sevinc, 2000), groundnut shell (Hassan et al, 2010), coconut husk (Tan et al, 2008), Jatropha husk(Ramakrishnan et al, 2009), pistachionut(Lua and Yang, 2005), sugarcane bagasse jackfruit peel (Prahas, et al, 2008) and many others. Base on both environmental and the economical points of view, unique attention have been focused on the use of agricultural waste products as an alternative replacement for commercial activated carbon. The abundance and





availability of agricultural by-products make them excellent sources of raw materials for natural sorbents (Bernard and Jimoh, 2013).

Activated carbons are generally prepared by two different methods; chemical and physical treatment method (Al-Swaidan and Ahmad 2011). Both treatment methods are responsible in varying the sizes and the shapes of activated carbon (Imran,2010). Physical treatment method is a two step process involving, carbonization of the precursor material followed by activation step with steam or carbon dioxide of the char. The carbonization temperature usually ranges between 400 and 850°C. Whereas in chemical treatment method, the precursor material is impregnated with an activating reagent and followed by heating under an inert atmosphere at a range temperature range of 600-900 °C (Yahya et al, 2015).

In this work, activated carbon was prepared from melon husk. By utilizing melon husk into activated carbon will decrease the cost of waste disposal and also converted this waste into value-added product. Currently no study has been done on application of response surface methodology (RSM) approach for preparation of activated carbon from melon husk. Response surface method is a set of statistical techniques for designing experiments, building models, evaluating the effects factors and searching for the optimum conditions (Nwabanne and Igbokwe, 2012).

2. MATERIALS AND METHODS

2.1. PREPARATION OF ACTIVATED CARBON

Melon seed husk were obtained from Kpakungu in Minna, Niger State. The husk samples were washed with ordinary tap water to remove possible foreign materials present (dirt and sands). Washed samples were sun dried for 2-5 days and then crushed with a mortar and pestle to reduce the size The reduced size was subjected to carbonization in a muffle furnace at different temperatures range (300 °C, 350 °C,400 °C, 450 °C, 500 °C, 550 °C and 600 °C) obtained from the thermo gravimetric analysis of the raw melon husk and at a constant time of 15mins to obtain the best temperature for carbonization and subsequently time was varied from 15mins to 60mins to obtain the best time for carbonization. 25gram of the carbonized sample was mixed in 50ml of different concentrations of potassium chloride (1.0M KCI, 2.0M KCI and 3.0M KCI) and kept for 24hours to soak. The impregnated samples were then dried at a temperature of 100 °C for 30 mins in an oven. Activation of the carbonized samples involves the use of statistical design to optimize the activation parameters (temperature, time and concentration) performed in a muffle furnace at different temperatures of 400 °C, 500 °C and 600 °C and at different time of 30min, 45min and 60min and concentrations of 1.0M, 2.0M and 3.0M according to design of experiment in Table 6.0. Factor levels of the independent variables for activation are shown in Table1.0. After cooling down to room temperature, the final activated carbon was repeatedly washed with hot distilled water until the wash water attained a pH 7. The washed carbon was dried in oven at 110°C for 8 hours, and finally kept in tightly closed bottles.

TABLE 1.0: EXPERIMENTAL DESIGN WITH VARIABLES AND LEVELS FOR ACTIVATION

Variable Factor	Lower level	Middle level	Upper level
	(-1)	(0)	(+1)
Temperature	400	500	600
Concentration	1	2	3
Time	30	60	90

2.2 CHARACTERIZATION OF CHAR SAMPLE FROM MELON HUSK 2.2.1. DETERMINATION OF MOISTURE CONTENT

The moisture content of the sample was determined using thermal drying method. 1.0g of the char sample from melon husk was weighed and placed in washed, dried and weighed crucible. The crucibles was placed in an oven and dried at 105°C to constant weight for 4h (Rengaraj, 2002). The percentage moisture content (%MC) was computed as shown by equation 1.

$$M.C = \frac{M - m}{M} \times 100 \tag{1}$$

Where, M.C = Moisture content of carbonized sample, M = Original mass of sample before heating and m = final mass of sample after heating.

2.2.2. ASH CONTENT

1.0g of the char sample from melon husk was weighed and placed in the crucibles and transferred into the muffle furnace at $900~^{\circ}\mathrm{C}$ for 1~h. The crucibles (with content) were allowed to cool in desiccators and then weighed to obtain the weight of the ash. The ash content was





expressed as percentage of the oven dry weight (Dara,1991). The percentage ash content (%AC) was computed as shown by equation 2.

$$A.C = \frac{Weight of \ ash}{Weight of \ sample} \times 100$$
 (2)

2.2.3 VOLATILE CONTENT DETERMINATION

The volatile content (V.C) was determined in accordance to the description reported by Fapetu, (2000). A mass of the char sample obtained melon husk was heated for about 850 0C for 10 min in a muffle furnace. The crucible and its content was retrieved and cooled in a desiccator. The difference in weight was recorded and percentage volatile content (VC) computed as shown by equation 3.

$$V.C = \frac{w_o - w_f}{w_o} \times 100 \tag{3}$$

Where, V.C₌Volatile matter content, $W_{f} = difference$ in weight (g) and Wo O₌ riginal weight of carbon.

2.2.4. FIXED CARBON CONTENT

This was conducted in accordance to the procedure in ASTM D 121 (2001). The Fixed carbon content was determined by subtracting the sum of percentage composition of moisture content, volatile content and moisture content from 100. The value obtained was the amount of fixed carbon content in the sample expressed as percentage.

2.2.5. CHAR YIELD

The char yield is determined by weight of final product after carbonization/activation divided by initial weight of sample (Fapetu,2000). The percentage char yield is given by equation 4.

$$Y_{ch} = \frac{W_{ch}}{W_o} \times 100 \tag{4}$$

Where, y_{ch} char yield, w_{ch} weight of final product and w_{o} == original weight of sample

2.2.6. IODINE NUMBER:

This was conducted in accordance to the procedure in ASTM D 4607-94(2006). 1 g of the activated carbon samples were transferred to a clean, dry 250 ml Erlenmeyer flask. 10 ml of 5 wt % hydrochloric acid solution was pipetted and add into the flask. The flasks

were closed and calmly swirled until the carbon was completely wet. The contents of the flasks were boil gently for $30 \pm 2s$ to eradicate any sulphur which may interfere with the results. The flasks were removed from the hot plate and cooled to room temperature. 100 ml of 0.1 N iodine solution was added to the flasks and shook vigorously for 30±1s. The contents of the flasks were filtered. 50 ml of the filtrate were taken in 250 ml Erlenmeyer flask and titrated against standardized 0.1 N sodium thiosulphate solutions until the solution turned pale yellow. 2 ml of the starch indicator was added to this solution and the titration was continued with sodium thiosulfate until the solution turned colourless. The volume of sodium thiosulphate used was recorded and the amount of iodine adsorbed per gram of activated carbon dosage was calculated by equation 5.

$$\frac{X}{M} = \frac{(A - (DF)(B)(S))}{M}$$

$$A = (N_1) \times 12693$$

$$B = (N_2) \times 126.93$$

$$DF = \frac{I + H}{F}$$
(5)

Where, X/M is iodine adsorbed per gram of activated carbon (mg/g), S is volume of sodium thiosulfate used (ml) and M is amount of activated carbon used (g), N_1 is the normality of iodine (N), N_2 is the normality of sodium thiosulphate (N), DF is dilution factor, I is volume of iodine (ml), H is 5 % hydrochloric acid used (ml), and F is filtrate (ml).

2.3. THERMO GRAVIMETRIC ANALYSIS (TGA)

In order to determine general decomposition characteristics of the agricultural waste during carbonization and activation, the sample was subjected to thermo gravimetric analysis (Perkin Elmer Pyris TG/DTA). The volatile evolution during carbonization of the samples was monitored by TGA.

2.4. SCANNING ELECTRON MICROSCOPY (SEM)

In order to observe the surface morphology of the activated carbon, a scanning electron microscopic analysis of the activated sample was employed in this study. SEM images were recorded using JEOL JSM-6300F field emission.

3.0. RESULTS AND DISCUSSION

Thermogravimetric analysis (TGA) and derivative thermo gravimetric (DTG) analysis of melon seed husk. Generally, there are three major stages of decomposition





indicated by the TGA curve. On the other hand, DTG curves provide a more detailed representation of the mechanism of thermal decomposition of the samples over the entire temperature range. The weight change of a sample is recorded as a function of temperature. Table 2.0 summarizes the changes that occurred on the TGA/DTG curves presented in Figure 1.0.

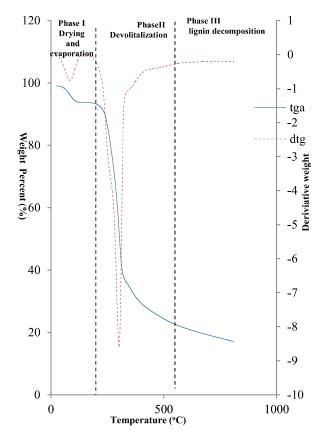


Figure 1.0: A plot of TGA and DTG for Melon seed husk

TABLE 2.0: TGA ANALYSIS OF MELON HUSK

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Sample	Sample Temp		Temp	Deg	T_P		
	range of	(%)	range of	(%) in			
	M.C (%)		deg in	stage			
			stage II	II			
Melon	25.11-	5.28	153.02-	59.01	300.95		
husk	153.02		346.99				

Where, Temp range of M.C=temperature range of moisture, Temp range of deg = temperature range of degradation and T_P = peak of degradation.

From Figure 1.0, the first stage of decomposition was from 25.11-153.02°C; with 5.28% weight loss indicates removal of moisture from melon seed husk. The second stage involves exothermic reactions which ensure the

removal of volatiles (devolatization), from rapid thermal decomposition of hemicelluloses and cellulose and partly lignin, which is a major step in the conversion process of the wastes to carbon. A major weight loss occurs in this stage, it is also known as the region of active pyrolysis. The degradation temperature range for melon seed husk was from 153.02-346.99°C, as shown in Figure 1.0; which indicates the removal of volatiles such as hemicelluloses, cellulose and partly lignin, with 59.01% weight loss. The derivative peak temperature (Tp) shown by the DTG curve was at 300.95°C, which indicates the point of highest rate of change on the weight loss curve. The last stage involves the endothermic decomposition of lignin, which occur at a wider temperature range (starting from about 250-800°C), due to steady decomposition of the remaining heavy component mainly from lignin. Since moisture was removed from the first stage of TGA curve, the highest weight loss occur at stage two with the peak of degradation for melon was at 300.90 °C and the third stage involves lesser weight loss with removal of the remaining volatile matters. Base on the provided information from TGA/DTG curve, temperature was varied (optimized) from 300°C - 600 °C for carbonization of melon seed husk.

3.1. EFFECT OF CARBONIZATION TEMPERATURE AND Time CHAR PRODUCED FROM MELON HUSK

Carbonization temperature and time are important parameters which affect the quality and quantity of carbon produced. Tables 3.0 and 4.0, summarizes the effect of carbonization temperature and time on char produced. Increase in carbonization temperature promote the release of volatile mater as well as enhancing the condensation and collapsing reactions of organic matters in the sample to become char with predominantly fixed carbon . The volatile matter content of 89.90% obtained at 300 °C from char produced from melon husk was decreased to 32.45% at $600\,^{\circ}\text{C}$.





TABLE 3.0: CHARACTERIZATION OF CARBONIZED MELON (CM) PRODUCED AT DIFFERENT TEMPERATURE AND CONSTANT TIME

Temp	T	V.C	A.C	M.C	F.C	Y
(°C)	(min)	(%)	(%)	(%)	(%)	(%)
300	15	89.90	4.00	1.01	5.09	43.10
350	15	74.23	6.45	3.00	16.32	23.90
400	15	60.61	7.00	1.00	31.39	21.70
450	15	57.14	8.00	2.00	32.86	20.50
500	15	49.49	9.00	1.00	40.51	18.90
550	15	40.00	24.40	4.00	31.60	13.00
600	15	32.45	37.50	1.78	28.27	4.60
	(°C) 300 350 400 450 500 550	(°C) (min) 300 15 350 15 400 15 450 15 500 15 550 15	(°C) (min) (%) 300 15 89.90 350 15 74.23 400 15 60.61 450 15 57.14 500 15 49.49 550 15 40.00	(°C) (min) (%) (%) 300 15 89.90 4.00 350 15 74.23 6.45 400 15 60.61 7.00 450 15 57.14 8.00 500 15 49.49 9.00 550 15 40.00 24.40	(°C) (min) (%) (%) (%) 300 15 89.90 4.00 1.01 350 15 74.23 6.45 3.00 400 15 60.61 7.00 1.00 450 15 57.14 8.00 2.00 500 15 49.49 9.00 1.00 550 15 40.00 24.40 4.00	(°C) (min) (%) (%) (%) (%) 300 15 89.90 4.00 1.01 5.09 350 15 74.23 6.45 3.00 16.32 400 15 60.61 7.00 1.00 31.39 450 15 57.14 8.00 2.00 32.86 500 15 49.49 9.00 1.00 40.51 550 15 40.00 24.40 4.00 31.60

Where, Temp = Temperature, T= Time, V.C = Volatile content, A.C = Ash content, M.C = Moisture content, F.C = Fixed carbon, Y = Char yield.

The Percentage yield is also an imperative parameter to know the extent of carbonization process. It was observed that percentage yield decreases with increase in temperature. The low yield at high temperature was essentially due to the devolatization of the raw material upon heating, giving a corresponding increase to the fixed carbon content. The yield of char from melon husk was reduced from 43.10% at 300 °C to 4.60% at 600 °C. The highest fixed carbon content of 40.51%, with a ash content of 9.00% was obtained at temperature of 500 °C from char produced from melon husk, Hence 500 °C was chosen as the best carbonization temperature for melon husk.

The effect of different carbonization time on char produced from melon husk as shown in Table 4.0. Longer carbonization time does not only reduce the quantity of carbonized product (yield of carbon) the nature, pore size and shape but the activity would also be attenuated. Such effects are most probably due to excessive burning /oxidation and collapse of pore structures which are predominate at longer resident time and higher temperatures.

TABLE 4.0: CHARACTERIZATION OF CARBONIZED MELON HUSK (CM) PRODUCED AT DIFFERENT TIME

Temp (°C)	T (min)	V.C (%)	A.C (%)	M.C (%)	F.C (%)	Y (%)
500	15	49.49	9.00	1.00	40.51	20.30
500	30	48.45	11.00	3.00	37.55	18.00
500	45	45.36	12.05	3.00	39.59	16.90
500	60	44.90	14.00	2.04	39.06	12.60

Temp = Temperature, T= Time, V.C = Volatile content, A.C = Ash content, M.C = Moisture content, F.C = Fixed carbon, Y = Char yield

Also when time and temperature is increased, the ash content is increased which blocks the pores, In addition, ash does not contribute significantly to surface area and its presence can reduce the surface area of carbon. Based on the stated reason, the best carbonization time of 15min was chosen with the highest fixed carbon content of 40.51%, yield of 20.30% and lowest ash content of 9.00% compared to ash of 11.00%12.05% and 14.00% obtained at 30,45 and 60mins respectively; which had higher ash content and lower yield.

3.2. ACTIVATION OF CHAR PRODUCED FROM MELON HUSK

A three-variable Box-Behnken experimental design was employed to evaluate the activation of char produced from melon husk. The activation temperature and retention time plus the concentration of potassium chloride solution used for impregnation for the production of activated carbons were optimized. The Iodine tests were used to analyze the best activated carbon produced based on the highest iodine value. The software used for the statistical analysis was Minitab Release (Version 17). Table 5.0, shows the experimental results and predicted values for the iodine test using activated carbon prepared from char of melon husk activated with Potassium chloride (KCI).





TABLE 5.0: EXPERIMENTAL AND PREDICTED VALUES OF IODINE NUMBER OF KCL ACTIVATED CARBON FROM MELON HUSK BY BOX-BEHNKEN DESIGN

Runs	Temperature	Conc.	time	Iodine Number	
	(°C)	mole	(min)	Experimental Values	Predicted values
				(mg/g)	(mg/g)
1	600	2	30	1238.86	1237.77
2	400	1	60	1231.04	1232.07
3	500	2	60	1235.79	1234.41
4	500	2	60	1230.21	1234.41
5	400	2	90	1235.51	1238.66
6	500	3	30	1239.70	1241.95
7	600	2	90	1223.50	1227.90
8	400	2	30	1230.76	1228.43
9	500	3	90	1233.00	1229.84
10	500	1	90	1251.43	1246.60
11	500	1	30	1233.56	1234.13
12	600	3	60	1228.81	1226.88
13	500	2	60	1233.00	1234.41
14	400	3	60	1229.93	1227.59
15	600	1	60	1233.28	1231.35

The final model equation used for predicted values of iodine number is given by equation 6.0.

$$Y = 1057.0 + 0.591 \times A + 10.05 \times B + 0.755 \times C - 0.000494 \times A^{2} + 0.00413 \times C^{2} - 0.001676 \times AC - 0.2048 \times BC$$
(6)

Where, y = iodine value, A =Temperature, B=concentration and C= time.

The highest experimental iodine value of 1251.43mg/g, with predicted value of 1246.60 mg/g was obtained at a temperature of 500 °C, 1.0M concentration and 90mins, which was chosen as best conditions for production of activated carbon.

The interactive effect of activation temperature, time and concentration was discussed using a three- dimensional respond surface (3D surface respond) and two-dimensional contour plots at optimum condition (500,1.0M and 90mins) with the highest iodine value of

1251.43mg/g. A three- dimensional response surface (3D response surface) and two- dimensional contour was plotted as shown in Figure 2(a-b)- 4.0(a-b).

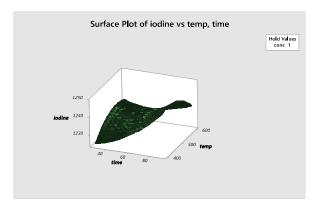
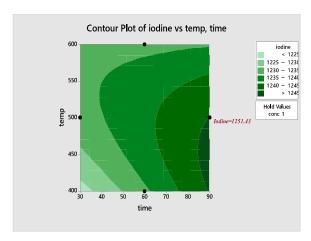


Figure 2(a) Surface Plot of Iodine verse Temperature and Time



 $\label{eq:Figure 2(b): Contour Plot of Iodine verse Temperature} and \ Time$

The 3D response surface and its relevant contour plot shown in Figures 2(a) and (2b), represent the interaction between temperature and activation time with fixed concentration (1.0M). The response shows the prominent effect between temperature and activation time on the iodine value. As shown in Figure 2(a), the iodine value increased with increase in activation time (beyond 80mins) and as temperature increased to 500°C. The increase in activation temperature and time further develop the undeveloped pore structure in the char and also enhance the existing pores thus creating higher surface area and higher porosities. At a higher temperature of 600 °C, the iodine value decreased, as a result of adjacent pore walls and micropores been destroyed. The contour plot shown in Figure 2(b) relates





the response variable (iodine value) to continuous variables (temperature and time). The contour with the darkest green colour at the inner right corner identified by a design point indicates the contour point where iodine value was highest (1251.43mg/g), at 500 °C and 90mins. The 3D response surface and the contour plot demonstrated that there were considerable interactions between temperature and time on the obtained iodine value of activated carbon. It is evident from Figure (2a) and (2b) that the highest iodine value was obtained at temperature of 500 °C and at time of 90mins, hence the optimum condition for production of activated with better surface area was at temperature of 500°C,1.0M concentration and 90mins.

The 3D response surface and its relevant contour plot shown in Figure3(a) and(b) represent the interaction between activating concentration against activation time with temperature fixed (at 500 °C), shows that the interaction between time and concentration was significant.

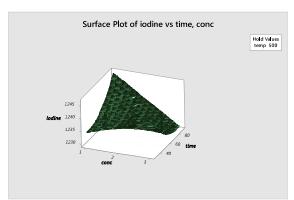


Figure 3(a): Surface Plot of Iodine verse Time and Concentration

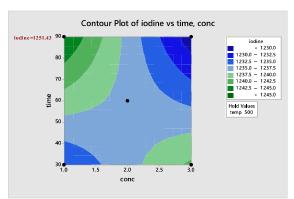


Figure 3 (b): Contour Plot of Iodine verse
Time and Concentration

The iodine value increased at lower activation concentration of 1.0M with an increase in activation time as shown by Figure 3(a). Activation at higher

concentrations of 2.0M and 3.0M with KCI, led to wider pores of the activated carbon (i.e., the micropore structure of activated carbon deteriorated due excess activation), as evident by the low iodine values. The contour plot showed in Figure 3(b) relates the response variable (iodine value) to continuous variables (concentration and time.) The contour with the darkest green colour at the upper left corner identified by a design point indicates the contour point, where iodine value was highest (1251.43mg/g) at 1.0M and 90mins. Iodine value increased as you move from the lower right to the upper left corner of the plot. That is, iodine value increases as you simultaneously reduce concentration and increase time. This plot suggests that you can maximize iodine value at a time of 90mins and a concentration of 1.0M.

The 3D response surface and its relevant contour plot shown in Figures 4(a) and 4(b) represent the interaction between activating concentration against temperature with time fixed (at 90min), shows that the interaction between concentration and temperature was significant.

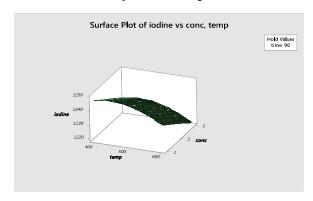


Figure 4(a): Surface Plot of Iodine verse Concentration and Temperature

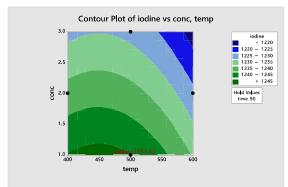


Figure 4(b): Contour Plot of Iodine verse Concentration and Temperature

As shown in Figure 4(a), the rise in iodine value was towards 1.0M concentration at a temperature of 500°C,





which was further emphasized by the contour plot. The darker regions of the contour plot in Figure 4(b) indicate areas with higher iodine values. The highest iodine value (1251.43mg/g) identified by the design point was at 1.0M concentration and at a temperature of 500°C. Higher concentrations of activation (2.0M and 3.0M with KCI) and temperature, led to lower iodine values due to destruction of micropores.

Design expert was further used to predict the optimum iodine value of activated carbon from melon husk as shown in Figure 5.0.

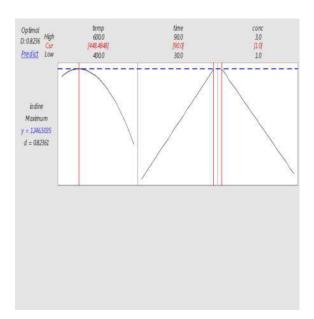


Figure 5.0: Graphical representation of numerical optimization of parameters at optimal condition

Optimization of the parameters was accomplished by multiple response method called desirability. The confirmatory experiment showed iodine value of 1251.43mg/g under optimal conditions (500 °C, 90mins 1.0M) compared with the of iodine value of 1246.50mg/g obtained by the model. This indicates the suitability and accuracy of the model. The composite desirability was 0.82.

3.3. CHARACTERIZATION OF ACTIVATED CARBON

3.3.1. Surface Morphology: Scanning Electron Microscope (SEM) was used to observe the surface morphology of the activated carbon as shown in Figure 6(a) and (b). The SEM results show that the surface activated carbon contained well-developed pores. The large irregular structure of pores in the activated carbon external surface indicated the macropores, with linkage of

mesopores and micropores rooting inside the internal structure of AC. Formation of the pores corresponded to the formation of metallic potassium (K) through activation process using KCl as activating agent. During the activation process, the C– KCl reaction rate was increased, thus resulting in carbon 'burn off', thereby developing good pores on the sample, which would have more surface area available for adsorption. The SEM image was further analyzed using image J software as shown in Figure 7 (a) and (b), to extract, show and count the actual pores present in the SEM micrograph.



Figure 6(a): SEM of KCI Activated Carbon at 100,000X (in nm)

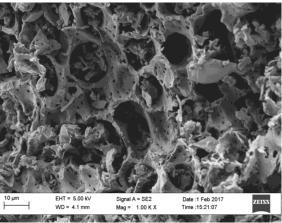


Figure 6(b): SEM of KCI Activated Carbon at 1000X (in μm)





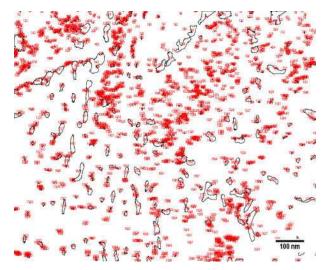


Figure 7(a): Analyzed SEM Image in nm by Image J Software

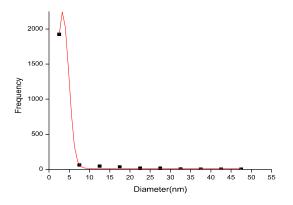


Figure 7(b): Pore Size distribution

From the analyzed SEM image by Image J, shown by Figure 7(a) and 7(b), fewer pores were larger than 10nm while majority of the pore size distribution were less than 5nm with a frequency above 1500 as evident from Figure 7(b). The total pore counted by Image J software, was 2108 pores with an average pore size of 3.34nm, which implies that the pores are mainly meso porous. Also a pore area of 8393.99nm² was obtained from image J analysis.

Conclusion

The preparation of activated carbon from melon husk was done using both classical optimization process (for carbonization of melon husk) and response surface methodology(for activation of the char from melon husk). Chemical activation was done using potassium chloride at different activating conditions obtained from the experimental design. The optimum conditions for preparing activated carbon from char obtained from melon husk was at activation temperature of 600 °C,

activation time of 90 min and 1.0M activation concentration. The experimental iodine values obtained are in close agreement with the model predicted iodine values.

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