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3 **Optimum process parameters for activated**

4 **carbon production from rice husk for phenol**

5 **adsorption**

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10 **ABSTRACT (ARIAL, BOLD, 11 FONT, LEFT ALIGNED, CAPS)**

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Aim: The determination of optimum process parameters in the production of activated carbon from rice husk for the purpose of treating wastewater containing phenol was the focus of this work.

Study design: The optimization was designed using response surface methodology.

Place and Duration of Study: Department of Chemical Engineering, NnamdiAzikiwe University Awka, Nigeria between August 2018 and May 2019.

Methodology: Central composite design (CCD) was used to generate the design matrix and analyze the result obtained. Carbonization temperature, percentage acid concentration and carbonization time were the factors considered. Tetraoxophosphate V acid (H_3SO_4) was employed in the activation process. The surface area was determined using the Brunauer-Emmet-Teller (BET) nitrogen adsorption method.

Results: The result indicated the optimum process conditions as carbonization temperature of 575 °C, time of 240 minutes and 45 percentage acid concentration. This gave 96.5% adsorption efficiency of phenol from aqueous solution. There was good agreement between the experimental values and the predicted values. The BET surface area of the activated carbon was 471.1 m²/g.

Conclusion: This work has optimized the process conditions for activated carbon production from rice husk for effective adsorption of phenol from wastewater.

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13 *Keywords:* Rice husk, activated carbon, optimization, response surface methodology, phenol

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15 **1. INTRODUCTION (ARIAL, BOLD, 11 FONT, LEFT ALIGNED, CAPS)**

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17 One of the major drawback of the oil industry in Nigeria is the release of untreated

18 wastewater containing pollutants into water bodies which the host communities use for

19 drinking, cooking, washing, irrigation etc. This practice has not only endangered the

20 immediate environment of the host communities but is also one of the reasons for the

21 constant clashes between the host communities on one hand and the oil companies and the

22 government on another hand.

23 Phenol is a major pollutant in the wastewater from the oil industry. This is because it has

24 serious unpleasant effects especially on man both long term and short term. [1] reported that

25 the consumption of water containing phenol can result in the damage of the capillaries in

26 man which may lead to death. Even at low concentration, phenol is very harmful to human
27 beings hence they are considered as priority pollutants [2-3]. Diarrhea, excretion of dark
28 urine, impaired vision etc are among other dangerous side effects of phenol [4]. Therefore,
29 treatment of phenolic wastewater is one of the priority needs for the protection of the
30 environment and for peace in the oil industry.

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32 Different techniques that have been reported for treatment of waste water include
33 electrocoagulation, biodegradation, solvent extraction, chemical oxidation, biological treatment,
34 phase transfer catalysis, adsorption, ion exchange etc. Adsorption has proved to be the most
35 effective and widely used method in treatment of waste water [5,4]. Adsorption is the
36 process where the adsorbate (in this case phenol) is attached on the surface of an
37 adsorbent and hence removed from the solution [6]. Adsorption is an effective method due to
38 its high treatment efficiency, low cost and the fact the it does not form harmful by-products
39 [7]. The adsorbent is usually activated carbon. Activated carbons have been employed in
40 water purification when removing both organic and inorganic pollutants from industrial
41 wastewater [8]. The drawback in adsorption method is the high cost of the activated carbon
42 which is used as the adsorbent [9] and most times, is imported in commercial quantities in
43 Nigeria. The over-dependence of our local industries on imported raw materials, activated
44 carbon inclusive, is currently the bane of the economy of developing countries such as ours
45 [10]. To curb this, the effectiveness of cheaper and abundant local materials has being
46 studied for use as adsorbents. They include oil palm fibre, saw dust, bamboo, kola nut shell
47 etc [11-14].

48 Rice husk is the chaff that is obtained from the milling of rice grains. It is relatively abundant
49 and can be obtained as waste from the rice milling industries. Its potential as an adsorbent
50 have been reported in adsorption processes. The adsorptive capacity of the adsorbent can
51 be increased by carbonization. Different conditions of the process parameters used in the
52 carbonization process affect the adsorptive potential of the activated carbon produced.
53 Optimization using response surface methodology can be used to determine the optimum
54 conditions involved in a process [15]. It is different from the method of one factor at a time
55 (OFAT) which involves keeping all other parameters constant while varying one factor.
56 OFAT method uses a large number of experiments in determining the optimum condition. It
57 is time consuming and does not show the interactive effects of the independent factors
58 unlike optimization using response surface methodology.

59 Hence, the aim of this work is to use response surface methodology to optimize the
60 carbonization process parameters for optimum production of activated carbon from rice husk
61 that will be used in the treatment of wastewater containing phenol.

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65 **2.0 MATERIALS AND METHODS**

66 **2.1 Preparation of raw materials**

67 The rice husk was sourced from rice mills in Anambra State, Nigeria. It was washed with
68 distilled water and sun dried. The phenol, tetraoxophosphoric acid, distilled water and other
69 reagents were sourced from Chemical Engineering Laboratory in Nnamdi Azikiwe University,
70 Awka, Anambra State.

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72 **2.2 Design of experiment**

73 The experimental runs for the carbonization process were designed using central composite
74 design of the RSM. This method uses a minimum number of experiments to optimize a
75 process while analyzing the interaction between the parameters. The independent variables
76 were percentage concentration of acid, carbonization temperature and carbonization time.
77 The dependent variable or the response was the percentage of phenol adsorbed. Table 1
78 shows the different levels of the independent variables that were used in the experiment.

79 The distance of the star like points from the core point, that is, the alpha value was 1.68. The
 80 actual experimental design (in Table 3) consist of 20 runs made up of 8 core points, 6 star
 81 like points and 6 null points.
 82 Statistical analysis of the model including the analysis of variance (ANOVA) was evaluated
 83 using Design Expert software.

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Table 1: Factor levels of the independent variables

Independent variable	- α	-1	0	-1	- α
Percentage concentration of acid (%)	10	20	35	50	60
Carbonization temperature ($^{\circ}$ C)	19	60	120	180	221
Carbonization time (minutes)	298	400	550	700	802

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89 2.3 Carbonization process

90 The carbonization of the rice husk was carried out based on the design of experiment. The
 91 rice husks were broken into small pieces and dried in sunlight. This helps to reduce the
 92 moisture content of the sample. The dried sample was activated by mixing it with the
 93 required percentage concentration of the tetraoxophosphoric acid, H_3PO_4 and kept in an
 94 oven at 383K for 24 hours. Thereafter, the activated sample was washed severally with
 95 deionized water. The activated rice husk was placed in a furnace at the appropriate
 96 temperature and time (based on the experimental design) to undergo the carbonization
 97 process. The sample was cooled, ground using mortar and pistil and sieved using a mesh
 98 size of 75 μ m. The experiment was repeated with different percentage concentration of acid,
 99 carbonization temperature and time according to the experimental design. All produced
 100 activated carbons were properly labeled and used for the actual adsorption experiment.
 101 Response surface was used to determine the individual and interactive effects of the
 102 independent variables on the percentage of the phenol adsorbed.

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105 2.4 Adsorption process

106 Stock solution of phenol with concentration 100mg/l was prepared. 100ml of the phenol
 107 solution was placed on a magnetic stirrer set at 50 $^{\circ}$ C. 0.5g of the activated rice husk was
 108 introduced and the mixture allowed for about 60 minutes. Thereafter, the mixture was cooled
 109 and separated using centrifugation at 1,000 rpm for 20 minutes. The absorbance of the
 110 phenol was estimated using UV spectrophotometer at a wavelength of 250nm and then
 converted to concentration. The percentage adsorbed (%) was determined as follows;

$$111 \%Adsorbed = \frac{C_0 - C_e}{C_0} \quad (1)$$

112 where C_0 is the initial concentration of phenol solution (mg/l), C_e is the equilibrium
 113 concentration of the phenol solution (mg/l).

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116 2.5 Physical properties of the activated carbon

117 Some of the physical properties of the activated rice husk were determined using standard
 118 methods. A pH meter (Elico model L1 -120) was used to determine the pH. Fixed carbon
 119 iodine number, moisture content, volatile matter, ash content and porosity were determined
 120 using the method reported by [14]. Water displacement method was used to determine the
 bulk density [16].

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123 2.6 Surface area and Pore size distribution analysis

124 The BET nitrogen (N_2) adsorption-desorption isotherms was used to determine the surface
 125 area and micro pore volumes. The Quantachrome NOVA Win version 11.03 was used at
 77K using N_2 gas sorption analyzer. The total pore volume estimated using liquid volume of

126 adsorbate (N₂) at a relative pressure of 0.99 while the surface area was calculated from the
127 nitrogen adsorption isotherms by assuming the area of a nitrogen molecule was 0.162 nm².

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129 2.7 Instrumental characterization of the activated carbon

130 A JOEL scanning electron microscope model JSM 6400 was used to carried out the
131 scanning electron microscope (SEM) ananalysis while a Shimadzu Fourier Transform Infrared
132 Spectrophotometer (FTIR) 8400S was used to identify the functional groups present in the
133 activated rice husk.

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135 3. RESULTS AND DISCUSSION

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138 3.0 RESULTS AND DISCUSSION

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140 3.1 BET surface area and pore size distribution

141 The surface area was obtained using N₂ adsorption isotherm of the carbonized at 77K. The
142 multipoint BET surface area was 471.7m²/s while the single point BET surface area was
143 286.8m²/s as seen in Table 2. The surface area was high as a result of the presence of
144 excess pores that developed during the activation and carbonization process. The higher the
145 surface area, the better the adsorption potentials of the adsorbent. The micropore volume
146 was 0.179cm³/g. These values are similar to those reported by [17,16]. The pore radius was
147 16.20A^o while the average pore width was 5.55 nm. The values obtained provide qualitative
148 information on the adsorption mechanism and the pore structure of the carbon.

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150 Table 2: BET surface area analysis of the activated rice husk

Property	Quantitative value
Multipoint BET surface area (m ² /s)	471.67
Single point BET surface area (m ² /s)	286.8
Average pore width (nm)	6.247
Micropore volume (cm ³ /g)	0.179
Adsorption energy (KJ/mol)	4.162
Pore radius (A ^o)	16.20

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152 3.2 Physical properties of the activated carbon

153 Table 3 contains the physical properties of the adsorbent. The fixed carbon analysis gave a
154 value of 10.14% which is not very high suggesting that the carbon content of rice husk is
155 low. The moisture content was equally low (6.5%) as expected while the porosity index
156 indicated 0.339. The iodine number was high at 461.84 mg/g which indicated high surface
157 area. Iodine number is used as an index to investigate the internal structure and surface
158 area of the activated carbon [18]. The high volatile matter (18.01%) and ash content
159 (57.49%) suggested good properties of the activated carbon.

160

161 Table 3: Physical properties of the adsorbent

Property	Quantitative value
Bulk density (g/ml)	0.448
pH	6.8 ± 0.2
Ash content (%)	57.49
Iodine Number (mg/g)	461.84
Moisture content (%)	6.5
Porosity(η)	0.339
Volatile matter (%)	30.82

Fixed Carbon (%)	10.14
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3.3 FTIR and SEM analysis

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The FTIR analysis revealed the functional groups present in the rice husk as shown in Table

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4. The chemical structure of the adsorbent is of vital importance in understanding the

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adsorption process. The wave number ranged from 3693.8 to 670 cm^{-1} with peaks from

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96.68 to 83.45 cm^{-2} . They are instrumental in the adsorption of aromatic compounds. The –

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C=C- stretch indicates the presence of alkenes while the C-Cl stretch and vibration suggests

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the presence of alkyl halides. The coupled vibrations are appreciable due to the availability

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of various constituents [19]. This shows that the rice husk can be a good source of some

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hydrocarbons such as alcohols, alkenes, alkyl halides.

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The SEM in figure 1 was obtained using the activated rice husk sieved at 200 μm . The result

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was at a magnification of 1000x. It indicated that the texture and surface morphology of the

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activated carbon were characterized by rough surfaces. Interspatial pores were seen within

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the matrix of the adsorbent indicating good adsorption properties. The large pores observed

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is due to the fact the activating agents promote the contact area between the carbon and the

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activating agent.

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Table 4: FTIR analysis result of carbonized rice husk

Wave Number (cm^{-1})	Peak area (cm^{-2})	Bond source	Compound
3693.8	96.68	O-H vibration	Alcohols
3272.6	90.66	-C=C- stretch	alkenes
2922.2	88.05	-C=C- stretch	alkenes
2855.1	85.08	O-H bending	Carboxylic acid
2209.9	95.58	O-H vibration	Alcohols
1640.0	889.48	O-H bending	Carboxylic acid
1233.7	90.23	C-H vibration	alkanes
853.6	87.83	C-Cl stretch	Alkyl halides
670.9	83.45	C-Cl stretch	Alkyl halides

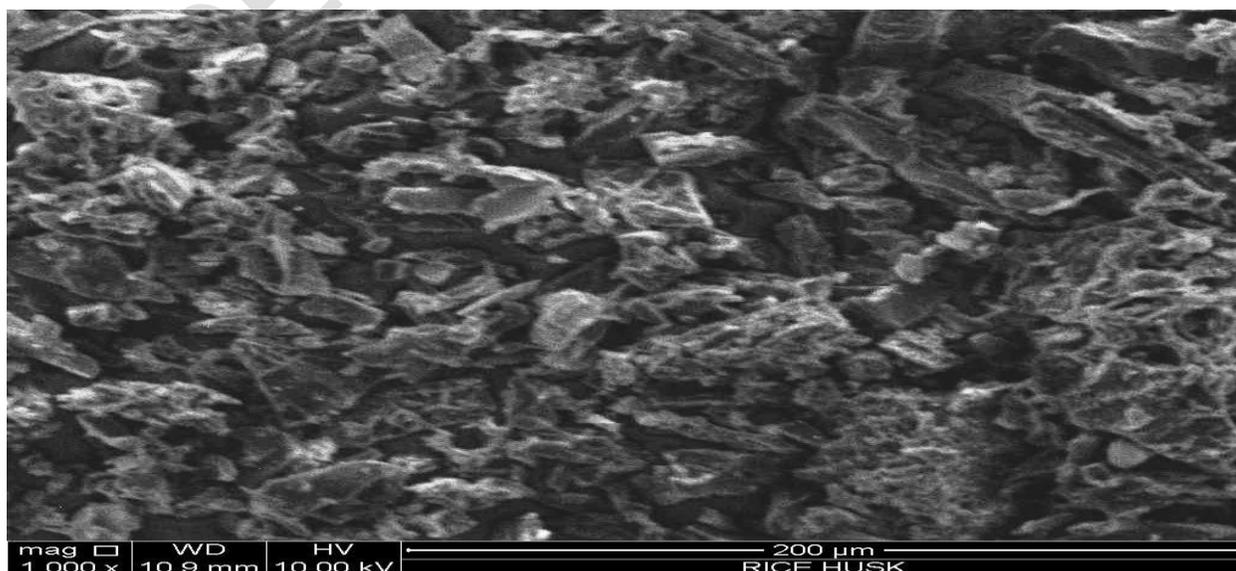
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Figure 1: SEM image of the activated carbon

3.4 Optimization process

The model summary statistics for the adsorption efficiency of phenol is presented in Tables 5. The model summary values suggested that a quadratic model best fitted the optimization process. The R-squared values for the quadratic and cubic models have the best values of 0.9909 and 0.9936 respectively when compared to that of other models (2FI and linear). The R-Squared is usually a measure of how efficient the variability in the actual response values can be explained by the experimental variables and their interactions. The cubic model is always aliased because the CCD does not contain enough runs to support a full cubic model. Aliases are false signals of any sort present hence the quadratic model was suggested.

Table 5: Model Summary Statistics

Source	Std. Dev.	R-squared	Adjusted R-sq	Predicted R-sq	PRESS	Remark
Linear	20.48	0.1550	-0.0035	-0.4966	11887.43	Not suggested
2FI	22.44	0.1757	-0.2047	-0.8298	14533.60	Not suggested
Quadratic	6.01	0.9546	0.9137	0.6717	2607.29	Suggested
Cubic	6.43	0.9688	0.9012	-5.2957	50005.71	Aliased

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The ANOVA in table 6 was used to analysis the result and validate the adsorption model. The lack of fit test and the adequacy of the regression models were equally performed. A significance level of 5% was used hence P-values greater than 0.05 are considered insignificant while those at 0.05 or less are significant. Hence, only the interactions of AB, AC and C² are insignificant. The model F-value of 23.35 implies that the model is significant agreeing with the P-value being less than 0.0001. The P values check the significance of the factors and equally help to understand the pattern of the mutual interactions between the test variables [20]. The R² value of 0.9546 is in close agreement with the adjusted R² value of 0.9137.

Table 6: ANOVA of the optimization process

Source	Sum squares	Df	Mean square	F value	p-value (Prob>F)
Model	7582.08	9	842.45	23.35	< 0.0001
A-Acid Concentration	580.95	1	580.95	16.10	0.0025
B-Carbonization time	268.53	1	268.53	7.44	0.0213
C-Carbonization temperature	381.26	1	381.26	10.57	0.0087
AB	1.45	1	1.45	0.040	0.8454
AC	22.44	1	22.44	0.62	0.4485

BC	141.12	1	141.12	3.91	0.0761
A ²	5180.95	1	5180.95	143.62	< 0.0001
B ²	585.03	1	585.03	16.22	0.0024
C ²	0.41	1	0.41	0.011	0.9169
Residual	360.73	10	36.07		
Lack of Fit	339.73	5	67.95	16.18	0.0042
Pure Error	21.00	5	4.20		
Cor Total	7942.81	19			

217 Std. Dev. = 6.01; Mean = 54.26; C.V. = 11.07%; PRESS = 2607.29
 218 R-Squared = 0.9546 Adj R-Sq = 0.9137; Pred R-Sq = 0.6717; Adeq Precision =
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224 3.5 Optimum model equation

225 The generated model equation for the adsorption process in terms of coded factors is
 226 Percentage Adsorbed (%) = +62.97 – 6.52A + 4.43B + 5.28C + 0.43AB – 1.67AC + 4.20BC
 227 – 18.96A² + 6.37B² - 0.17C²
 228 (1)

229 The positive sign of a factor indicates that there will be increase in the response when there
 230 is an increase in the factor while negative sign will lead to decrease in the response [21].
 231 Increase in carbonization temperature will show the most significant increase in the
 232 response on the account that its coefficient is highest.

233 Since a significant level of 5% was used, all factors with P-values greater than 0.05 are
 234 eliminated giving the final model equation as

$$235 \text{Percentage Adsorbed (\%)} = +62.97 - 6.52A + 4.43B + 5.28C + 4.20BC - 18.96A^2 + 6.37B^2 \quad (2)$$

239 3.6 Comparism of predicted and experimental values

240 A comparism of the actual experimental response and the predicted response are given in
 241 Table 7. The result of the experimental runs in the optimization process indicated that the
 242 best carbonization conditions are at an acid concentration of 45%, carbonization time of
 243 240.9 minutes and carbonization temperature of 575 °C. This gave the highest adsorption
 244 efficiency of 96.6% of phenol adsorbed from the phenol solution. The result equally revealed
 245 that the three factors optimized have great effect on the production of activated carbon.
 246 The close correlation between the actual experimental response and the predicted response
 247 confirms the suitability of the quadratic model used the analysis.
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249 Table 7: Experimental and predicted responses of the optimization process

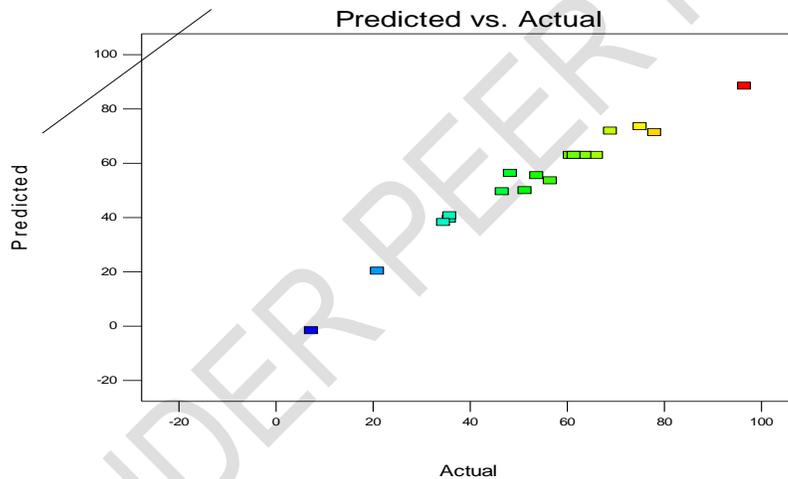
Standard order	Acid Conc	Carbonization time (mins)	Carbonization temperature	Experimental Response	Predicted Response
1	30	80	400	51.3	49.9
2	60	80	400	35.7	39.4
3	30	200	400	46.6	49.6
4	60	200	400	35.8	40.7
5	30	80	750	53.7	55.5
6	60	80	750	34.5	38.2
7	30	200	750	68.9	71.9
8	60	200	750	48.3	56.4
9	19.7731	140	575	20.9	20.3

10	70.2269	140	575	7.3	-1.6
11	45	39.0924	575	75	73.5
12	45	240.908	575	96.5	88.5
13	45	140	280.686	56.5	53.6
14	45	140	869.314	78	71.4
15	45	140	575	61.1	62.9
16	45	140	575	63.5	62.9
17	45	140	575	62.6	62.9
18	45	140	575	63.6	62.9
19	45	140	575	60.6	62.9
20	45	140	575	61.4	62.9

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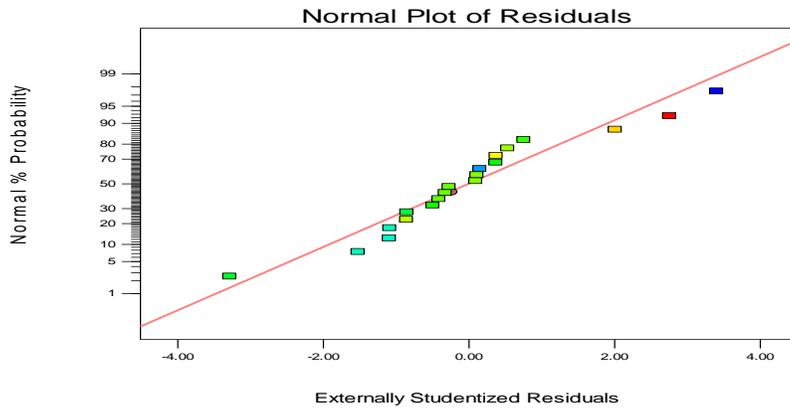
3.7 Error graphs

The Predicted vs Actual plot in figure 2 and the Normal plot of Residuals in figure 3 were used to determine if the residuals follow a normal distribution. It is assumed to have followed a normal distribution as the points closely aligned to the straight line of the plot thereby confirming the good relationship between the experimental values and the predicted values of the response and the adequacy of the suggested model in predicting the response variables in the experimental values.



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Figure 2: The Predicted vs Actual plot of the optimization process



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268 Figure 3: Normal plot of Residuals of the optimization process

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271 3.8 3-D response surface plots

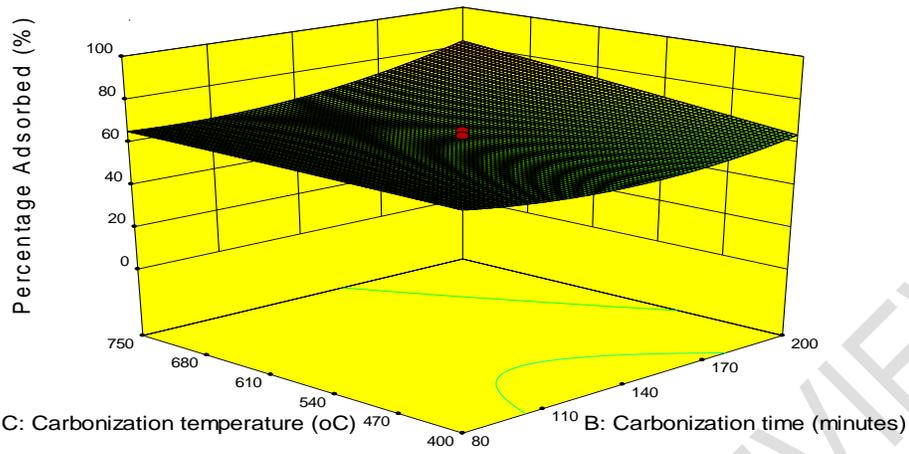
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273 The 3-D response surface plots are graphical representation of the interactive effects of any
274 two variables factors. Response surface estimation serves as a function of two factors at a
275 time, maintaining other factors at fixed levels. This is more helpful in understanding both the
276 main and the interaction effects of those two factors. These plots can be easily obtained by
277 calculating from the model, the values taken by one factor where the second varies with
278 constraint of a given response value. The response surface curves were plotted to
279 understand the interaction of the variables and to determine the optimum levels of each
280 variable for maximum response.

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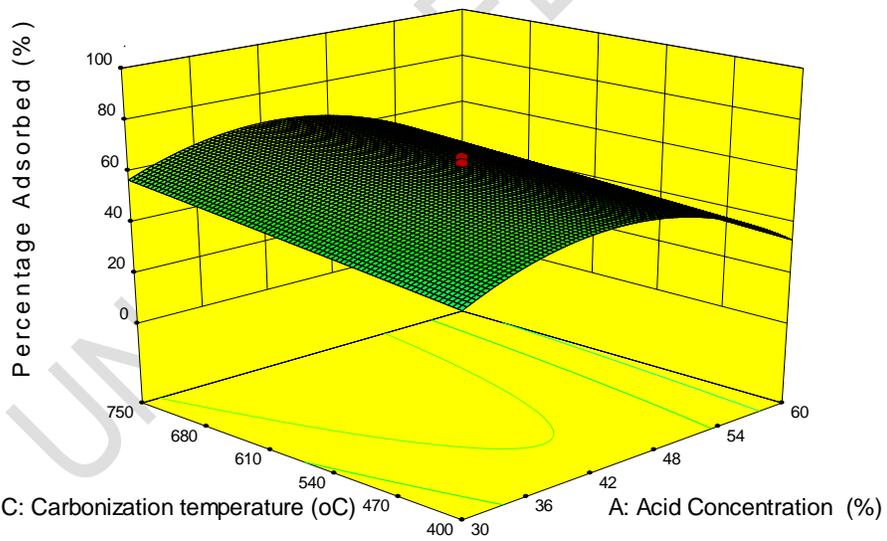
282 The nature of the response surface curves shows the interaction between the variables. The
283 elliptical shape of the curve indicates good interaction of the two variables and circular shape
284 indicates no interaction between the variables. There was a relative significant interaction
285 between every two variables, and there was a maximum predicted efficiency as indicated by
286 the surface confined in the smallest ellipse in the contour diagrams.

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Figure 4: Interactive effects of temperature and time



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Figure 5: Interactive effect of temperature and concentration

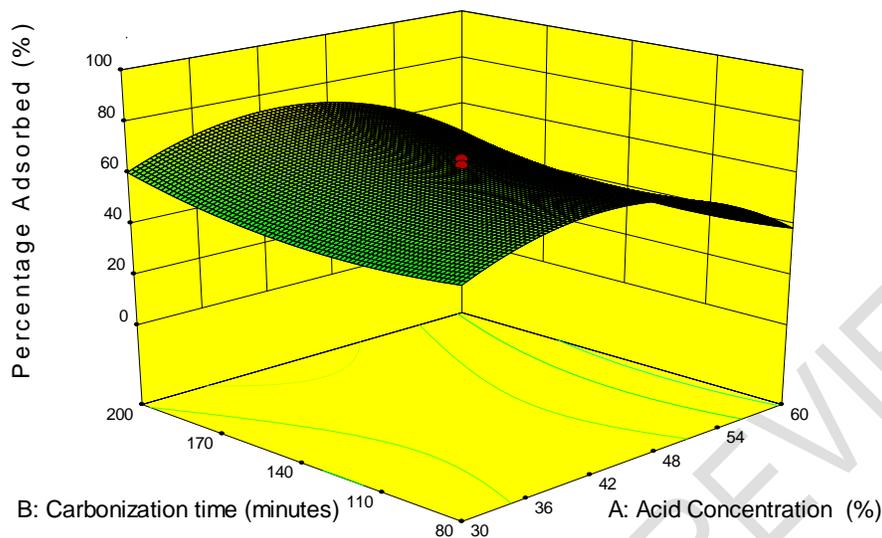


Figure 6: Interactive effects of time and concentration

4. CONCLUSION

The process parameters for activated carbon production from rice husk were optimized using response surface methodology for the treatment of phenolic wastewater. BET multi-point surface area of the activated carbon was high indicating favourable applicability of the adsorbent in the adsorption processes. Maximum adsorption efficiency of 96.5% was obtained at carbonization time of 240 minutes, carbonization temperature of 575 °C and at acid concentration of 45%. A quadratic model with a high correlation coefficient was suggested in describing the interactive effects of the process parameters. This study has shown that activated carbon can be produced from rice husk at optimum process conditions for the uptake of phenol from wastewater.

COMPETING INTERESTS

The authors have declared that no competing interest exists.

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