Original Research Article

Optimum process parameters for activated carbon production from rice husk for phenol adsorption

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

Aim: The determination of optimum process parameters in the production of activated carbon from rice husk for the uptake of phenol from aqueous solution was the focus of this work.
Study design: The optimization was designed using response surface methodology.
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. Tetraoxophosphoric acid (H_3PO_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 ^oC, 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 m^2/s.
Conclusion: This work has optimized the process conditions for activated carbon production from rice husk for effective adsorption of phenol from wastewater.

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

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

One of the major drawback of the oil industry in Nigeria is the release of untreated wastewater containing pollutants into water bodies which the host communities use for drinking, cooking, washing, irrigation etc. This practice has not only endangered the immediate environment of the host communities but is also one of the reasons for the constant clashes between the host communities on one hand and the oil companies and the government on another hand.

Phenol is a major pollutant in the wastewater from the oil industry. This is because it has serious unpleasant effects especially on man both long term and short term. [1] reported that the consumption of water containing phenol can result in the damage of the capillaries in man which may lead to death. Even at low concentration, phenol is very harmful to human beings hence they are considered as priority pollutants [2-3]. Diarrhea, excretion of dark

urine, impaired vision etc are among other dangerous side effects of phenol [4]. Therefore, treatment of phenolic wastewater is one of the priority needs for the protection of the environment and for peace in the oil industry.

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

Rice husk is the chaff that is obtained from the milling of rice grains. It is relatively abundant and can be obtained as waste from the rice milling industries. Its potential as an adsorbent have been reported in adsorption processes. The ash of rice husk contains approximately high silica, which has porous structure and is lightweight, with high specific surface area. Annual rice production in Nigeria was estimated at over 5.8 million tons in 2017 [15]. Therefore, huge quantity of rice husk from the rice milling factories is usually discarded as waste annually. Activated carbon adsorbents produced from rice husk has been used in the adsorption of gasoline [16] and in the adsorption of lead (Pb) from car battery wastewater [17]. The adsorptive capacity of the adsorbent can be increased by carbonization. Different conditions of the process parameters used in the carbonization process affect the adsorptive potential of the activated carbon produced.

Optimization using response surface methodology can be used to determine the optimum conditions involved in a process [18]. It is different from the method of one factor at a time (OFAT) which involves keeping all other parameters constant while varying one factor. OFAT method uses a large number of experiments in determining the optimum condition. It is time consuming and does not show the interactive effects of the independent factors unlike optimization using response surface methodology (RSM). Design of experiment using RSM is an enhanced systematic experimentation that takes into consideration all the process parameters involved simultaneously [18].

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

2.0 MATERIALS AND METHODS

2.1 Preparation of raw materials

The rice husk was sourced from rice mills in Anambra State, Nigeria. It was washed with distilled water and sun dried. The phenol, tetraoxophosphoric acid (H3PO4), distilled water and other reagents were sourced from Chemical Engineering Laboratory in Nnamdi Azikiwe University, Awka, Anambra State.

2.2 Design of experiment

The experimental runs for the carbonization process were designed using central composite design of the RSM. Design Expert software version 10.0.7 was used to carry out the RSM analysis. This method uses a minimum number of experiments to optimize a process while analyzing the interaction between the parameters. The independent variables were percentage concentration of the tetraoxophosphoric acid, carbonization temperature and carbonization time. The dependent variable or the response was the percentage of phenol adsorbed. Table 1 shows the different levels of the independent variables that were used in the experiment. The distance of the star like points from the core point, that is, the alpha value was 1.68. The actual experimental design (in Table 3) consist of 20 runs made up of 8 core points, 6 star like points and 6 null points.
Statistical analysis of the model including the analysis of variance (ANOVA) was evaluated using the Design Expert software.

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 (^{o}C)	19	60	120	180	221
Carbonization time (minutes)	298	400	550	700	802

2.3 Carbonization process

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

2.4 Adsorption process

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

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

where Co is the initial concentration of phenol solution (mg/l), Ce is the equilibrium concentration of the phenol solution (mg/l).

2.5 Physical properties of the activated carbon

Some of the physical properties of the activated rice husk were determined using standard methods. A pH meter (Elico model L1 -120) was used to determine the pH. Fixed carbon iodine number, moisture content, volatile matter, ash content and porosity were determined

using the method reported by [14]. Water displacement method was used to determine the bulk density [19].

2.6 Surface area and Pore size distribution analysis

The BET nitrogen (N_2) adsorption-desorption isotherms was used to determine the surface 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 adsorbate (N_2) at a relative pressure of 0.99 while the surface area was calculated from the nitrogen adsorption isotherms by assuming the area of a nitrogen molecule was 0.162 nm^2.

2.7 Instrumental characterization of the activated carbon

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

3.0 RESULTS AND DISCUSSION

3.1 BET surface area and pore size distribution

The surface area was obtained using N_2 adsorption isotherm of the carbonized at 77K. The multipoint BET surface area was 471.7m^2/s while the single point BET surface area was 286.8m^2/s as seen in Table 2. The surface area was high as a result of the presence of excess pores that developed during the activation and carbonization process. The higher the surface area, the better the adsorption potentials of the adsorbent. The micropore volume was 0.179cm^2/g. These values are similar to those reported by [19,20]. The pore radius was 16.20A^o while the average pore width was 5.55 nm. The values obtained provide qualitative information on the adsorption mechanism and the pore structure of the carbon.

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^2/g)	0.179
Adsorption energy (KJ/mol)	4.162
Pore radius (A^o)	16.20

3.2 Physical properties of the activated carbon

Table 3 contains the physical properties of the adsorbent. The fixed carbon analysis gave a value of 10.14% which is not very high suggesting that the carbon content of rice husk is low. The moisture content was equally low (6.5%) as expected while the porosity index indicated 0.339. The iodine number was high at 461.84 mg/g which indicated high surface area. Iodine number is used as an index to investigate the internal structure and surface area of the activated carbon [21]. The high volatile matter (18.01%) and ash content (57.49%) suggested good properties of the activated carbon.

Table 3: Physical properties of the adsorbent

Property	**Quantitative value**
Bulk density (g/ml)	0.448
pH	6.8 $\pm$ 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

3.3 FTIR and SEM analysis

The FTIR analysis revealed the functional groups present in the rice husk as shown in Table 4. The chemical structure of the adsorbent is of vital importance in understanding the adsorption process. The wave number ranged from 3693.8 to 670 cm^{-1} with peaks from 96.68 to 83.45 cm^{-2}. They are instrumental in the adsorption of aromatic compounds. The –C=C- stretch indicates the presence of alkenes while the C-Cl stretch and vibration suggests the presence of alkyl halides. The coupled vibrations are appreciable due to the availability of various constituents [22]. This shows that the rice husk can be a good source of some hydrocarbons such as alcohols, alkenes, alkyl halides.

The SEM in figure 1 was obtained using the activated rice husk sieved at 200µm. The result was at a magnification of 1000x. It indicated that the texture and surface morphology of the activated carbon were characterized by rough surfaces. Interspatial pores were seen within the matrix of the adsorbent indicating good adsorption properties. The large pores observed is due to the fact the activating agents promote the contact area between the carbon and the activating agent.

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

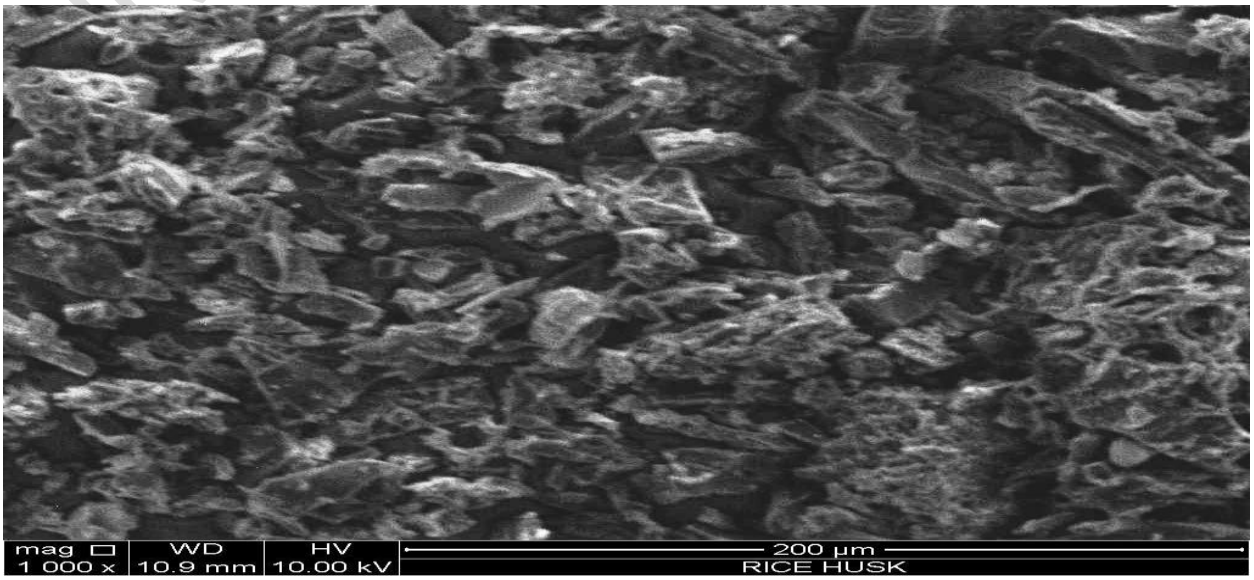

Figure 1: SEM image of the activated carbon

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

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 C^2 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 [23]. The R^2 value of 0.9546 is in close agreement with the adjusted R^2 value of 0.9137.

Table 6: ANOVA of the optimization process

Source	Sum of 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			

Std. Dev. = 6.01; Mean = 54.26; C.V. = 11.07%; PRESS = 2607.29
R-Squared = 0.9546 Adj R-Sq = 0.9137; Pred R-Sq = 0.6717; Adeq Precision = 21.210

3.5 Optimum model equation

The generated model equation for the adsorption process in terms of coded factors is

Percentage Adsorbed (%) = $+62.97 - 6.52A + 4.43B + 5.28C + 0.43AB - 1.67AC + 4.20BC - 18.96A^2 + 6.37B^2 - 0.17C^2$ (1)

The positive sign of a factor indicates that there will be increase in the response when there is an increase in the factor while negative sign will lead to decrease in the response [24]. Increase in carbonization temperature will show the most significant increase in the response on the account that its coefficient is highest.

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

Percentage Adsorbed (%) = $+62.97 - 6.52A + 4.43B + 5.28C + 4.20BC - 18.96A^2 + 6.37B^2$ (2)

3.6 Comparism of predicted and experimental values

A comparism of the actual experimental response and the predicted response are given in Table 7. The result of the experimental runs in the optimization process indicated that the best carbonization conditions are at an acid concentration of 45%, carbonization time of 240.9 minutes and carbonization temperature of 575 ^{o}C. This gave the highest adsorption efficiency of 96.6% of phenol adsorbed from the phenol solution. The result equally revealed that the three factors optimized have great effect on the production of activated carbon.

The close correlation between the actual experimental response and the predicted response confirms the suitability of the quadratic model used the analysis.

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

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 striaght line of the plot thereby confirming the good relationship between the experimental values and the predicted values of the response and the adeqency of the suggested model in predicting the response variables in the experimental values.

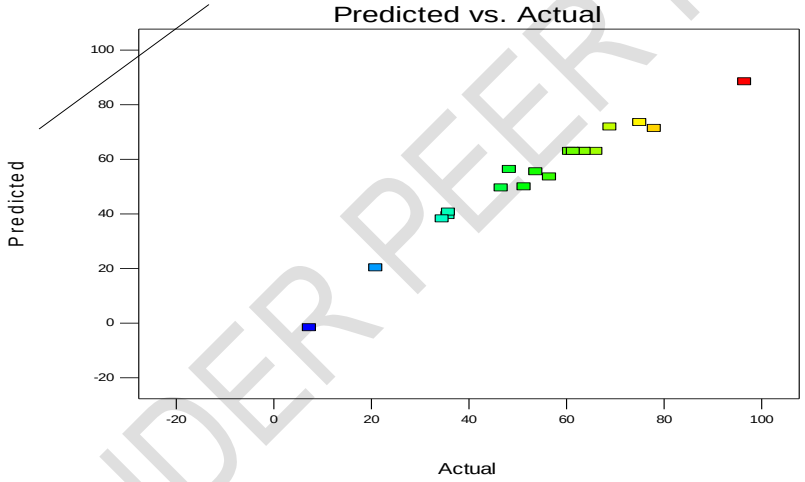

Figure 2: The Predicted vs Actual plot of the optimization process

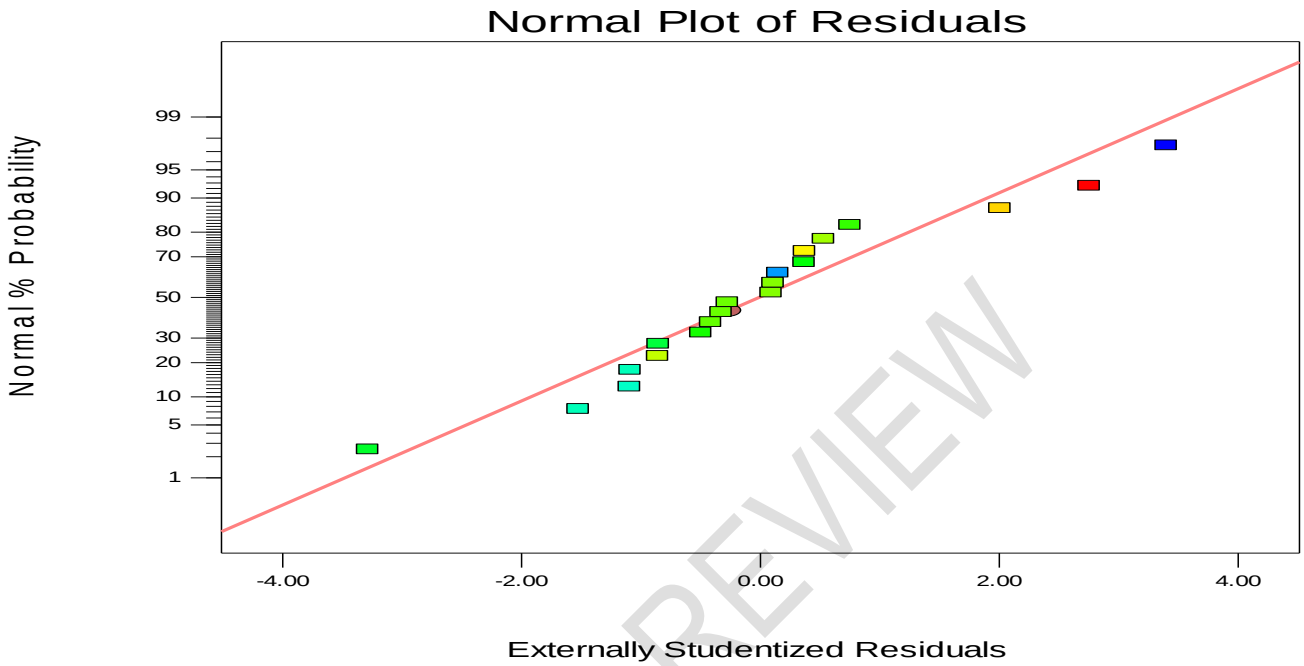

Figure 3: Normal plot of Residuals of the optimization process

3.8 3-D response surface plots

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

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

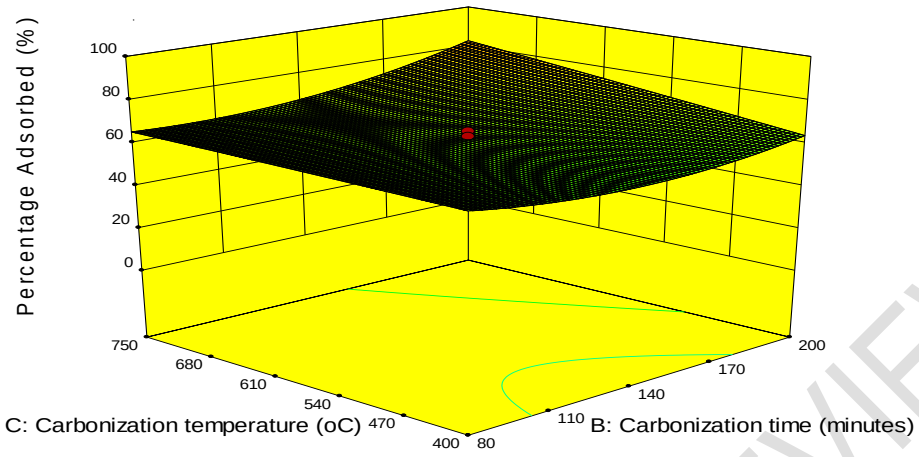

Figure 4: Interactive effects of temperature and time

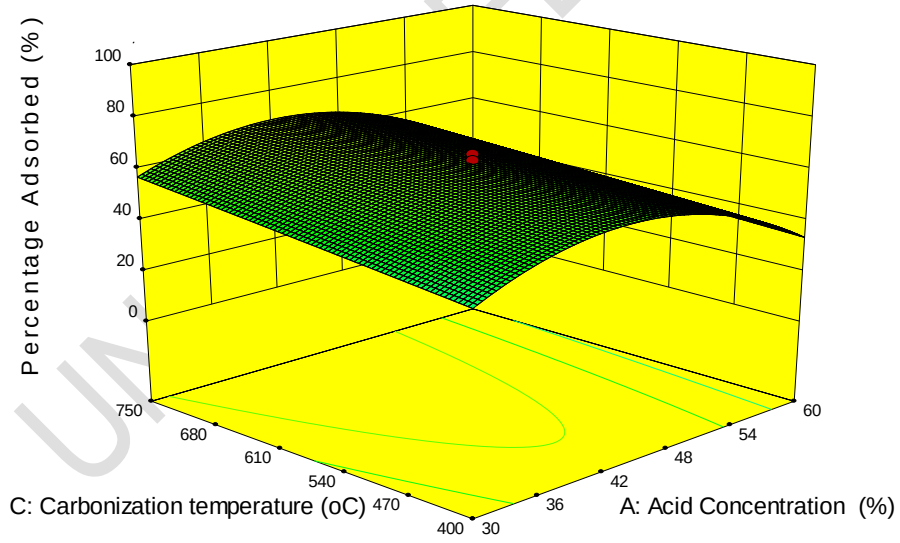

Figure 5: Interactive effect of temperature and concentration

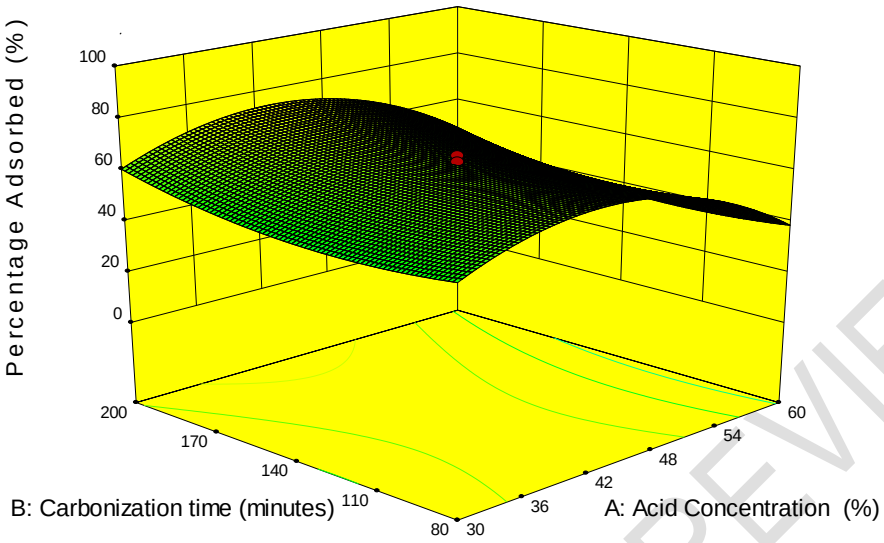

Figure 6: Interactive effects of time and concentration

An adsorption capacity of 38.2 mg/g was obtained in this work for phenol adsorption using the activated rice husk produced. This is more 26.26 mg/g reported in phenol removal using sewage sludge based adsorbent modified with H_2SO_4 [25] and 25.0 mg/g obtained in phenol adsorption using multiwalled carbon nanotubes [26]. However, it is much less than 91 to 112.5 mg/g reported in phenol adsorption using Latana camara [27].

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 ^{o}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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