

Application of Response Surface Methodology for Preparation of Activated Carbon from Palmyra Palm Nut

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Abstract: Optimization for the preparation of activated carbon obtained from palmyra palm nut (PPN) was studied using response surface methodology. A three-variable Box-Behnken experimental design was employed to correlate the preparation variables (activation temperature, activation time and acid/precursor impregnation ratio) to the lead uptake from aqueous solution. The preparation variables for the production of activated carbon were optimized based on the percentage removal of Pb^{2+} from aqueous solutions. The adsorption tests were used to analyze the best activated carbon for Pb^{2+} adsorption under different conditions of temperature, time and acid/precursor ratio. Chemical activation method which employed phosphoric acid was used for the preparation of activated carbon. The result showed that the optimum conditions for preparing activated carbon from PPN for Pb^{2+} uptake were activation temperature of $500^{\circ}C$, activation time of 1hr and acid impregnation ratio of 1.5:1 (acid/precursor, wt basis) which resulted in 95.85% removal of Pb^{2+} from aqueous solution. The experimental result obtained was in agreement with the model prediction. The adsorption studies showed that activated carbon produced from PPN is an efficient adsorbent for Pb^{2+} removal from aqueous solution.

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1. Introduction

The term activated carbon defines a group of materials with highly developed internal surface area and porosity and hence a large capacity for adsorbing chemicals from gases or liquids (Patrick, 1995). These unique characteristics are responsible for its adsorptive properties, which are exploited in many different liquid-and gas-phase applications (Ruthven, 1984). Activated carbon is an amorphous material but has a microcrystalline structure. But this microcrystalline structure differs from that of graphite with respect to interlayer spacing, which is 0.335nm in the case of graphite and ranges between 0.34nm and 0.35nm in activated carbons (Bansal and Goyal, 2005). Activated carbons are amorphous solid adsorbents that can be produced from almost all carbon-rich materials, including wood, fruit stones, peat, lignite, anthracite, shells and other raw materials (Roque-Malherbe, 2007). Its unique adsorption properties result from its high- surface area, adequate pore size distribution, broad range of surface functional groups, and relatively high mechanical strength. Consequently, active carbons are used extensively for the removal of undesirable odour, colour, taste, and other organic and inorganic impurities from domestic and industrial wastewater, solvent recovery, air purification in inhabited places, restaurants, food processing and chemical industries; in the removal of colour from various syrups and

pharmaceutical products; in air pollution control from industrial and automobile exhausts; in the purification of many chemicals, pharmaceuticals, and food products; and in a variety of gas-phase applications(Bansal and Goyal, 2005).

The adsorption capacity of active carbons is strongly influenced by the chemical structure. The most important and common groups influencing the surface and adsorption properties of activated carbon are functional groups containing oxygen, and their presence enhances the adsorption of polar species (Roque-Marlherbe, 2007). These surface groups can be produced during the activation procedure and can also be introduced subsequently during preparation by an activated treatment. The different types of oxygen functional groups determine the acidic and basic character of the carbon surfaces. The acidic character is usually linked with surface complexes like carboxyl. The more important parameters that influence and determine the adsorption of metal ions from aqueous solutions are the carbon-oxygen functional groups present on the carbon surface and the pH of the solution (Bansal and Goyal, 2005).

In general, there are two main methods for the preparation of activated carbons, specifically, physical and chemical activation methods. Physical activation consists of a two-step procedure, performed at high temperature ($800-1000^{\circ}C$): carbonisation under oxidizing agents (carbon dioxide,

oxygen, or steam). Chemical activation involves the treatment of the initial material with a dehydrating agent, such as sulphuric acid, phosphoric acid, zinc chloride, potassium hydroxide, or other substances, at temperatures varying from 400 to 1000°C, followed by the elimination of the dehydrating agent by meticulous washing (Roque-Malherbe, 2007). Chemical activation is preferred over physical activation owing to the lower temperatures and shorter time needed for activating the material.

In recent years, the preparation of activated carbons from several agricultural by products has been emphasized due to the growing interest in low cost activated carbons from renewable biomass, especially for applications concerning treatment of drinking water and wastewater (Castro et al, 2000). Several works have been carried out on the preparation of activated carbons from sawdust, ficus religiosa leaves, dead biomass, coconut shell, rice husk, and nypa fruticans shoot (Ahmad et al, 2008; Qaiser et al, 2007; Geu et al, 2006; Sekar et al, 2004; Ong et al, 2007; Wankasi et al, 2004). The present study is aimed at optimizing the conditions for the preparation of activated carbon from palmyra palm nut for the removal of Pb²⁺ from aqueous solution using surface response methodology. Response surface method is a collection of statistical techniques for designing experiments, building models, evaluating the effects factors and searching for the optimum conditions (Shrivastava et al, 2008).

2. Material and Methods

Preparation of activated carbon

Palmyra palm nuts were obtained from Nnamdi Azikiwe University, Awka, Nigeria. The palm nuts were washed with de-ionized water and dried in the sun. The sun-dried raw materials were ground into fine particles using a Jencod grinding machine (model Y1600) and sieved to a particle size of 300µm. 400g of ground raw material was impregnated with concentrated ortho-phosphoric acid at different ratios of acid to raw material (1:1, 1.5:1 and 2:1) on weight basis as shown in Table 2. The impregnated samples were dried in a Memmert oven at 120°C for 24hours. One step activation of the samples was performed in KGYV Budapest muffle furnace. The samples (20g each) were carbonized for 1hr, 1½hrs and 2hrs at 500°C, 650°C and 800°C according to design of experiment in Table 2. After cooling to the ambient temperature, the samples were weighed in order to determine the yield of activated carbon from the materials. The samples were washed with de-ionized water several times until pH 6.8, filtered with Whatman No.1 filter paper and then dried in the oven at 110°C for 8 hours. The dried samples were pulverized, sieved and then stored in air tight bottles ready for use. A three-variable Box-

Behnken experimental design was employed for the production of activated carbon as represented in Table 2. Factor levels of the independent variables for the production of activated carbon are shown in Table 1.

Table 1: Factor levels of the independent variables for the production of activated carbon.

Independent variables	Low level (-1)	Medium level (0)	High level (+1)
Temperature, °C	500	650	800
Carbonization time, hour	1	1.5	2
Acid concentration/raw material ratio (w/w)	1	1.5	2.0

Table 2: A three-variable Box-Behnken experimental design for the production of activated carbon

Run	Coded values	Natural values		
		Temp of activation °C	Activation time (hour)	Impregnation ratio Acid: raw material
1	- - 0	500	1	1.5
2	- + 0	500	2	1.5
3	+ - 0	800	1	1.5
4	+ + 0	800	2	1.5
5	- 0 -	500	1.5	1
6	- 0 +	500	1.5	2
7	+ 0 -	800	1.5	1
8	+ 0 +	800	1.5	2
9	0 - -	650	1	1
10	0 - +	650	1	2
11	0 + -	650	2	1
12	0 + +	650	2	2
13	0 0 0	650	1.5	1.5
14	0 0 0	650	1.5	1.5
15	0 0 0	650	1.5	1.5

Characterization of activated carbon

The pH of the carbon was determined using standard test of ASTM D 3838-80 (ASTM, 1996). Moisture content of activated carbon and raw materials was determined using ASTM D 2867-91 (1991). The bulk density of the activated carbon was determined according to the tamping procedure by Ahmedna et al (1997). The volatile content was determined by weighing 1.0g of sample and placing it in a partially closed crucible of known weight. It was then heated in a muffle furnace at 900°C for 10mins. The percentage fixed carbon was determined as 100 – (Moisture content + ash content + volatile matter). The iodine number was determined based on ASTM D 4607-86 (1986) by using the sodium thiosulphate volumetric method. The specific surface area of the activated carbon was estimated using

Sear's method (Al-Qadah and Shawabkah, 2009 and Alzaydien, 2009) by agitating 1.5g of the activated carbon samples in 100ml of diluted hydrochloric acid at a pH = 3. Then a 30g of sodium chloride was added while stirring the suspension and then the volume was made up to 150ml with deionized water. The solution was titrated with 0.1N NaOH to raise the pH from 4 to 9 and the volume, V recorded. The surface area according to this method was calculated as $S = 32V - 25$. Where, S = surface area of the activated carbon, V = volume of sodium hydroxide required to raise the pH of the sample from 4 to 9.

Adsorbate preparation and adsorption study

The reagents used were lead nitrate salt ($Pb(NO_3)_2$), and de-ionized water. The reagents were of high grade. The sample ($Pb(NO_3)_2$) was dried in an oven for 2hrs at $105^{\circ}C$ to remove moisture. 1.6g of $Pb(NO_3)_2$ was dissolved in 1000ml of de-ionized water to get the stock solution of 1000g/l. A known weight of activated carbon was added to 100ml of the 100mg/l of adsorbate in a conical flask and placed on a magnetic stirrer. The adsorption tests were performed at fixed parameters (contact time of 2hrs, initial ion concentration of 100mg/l, adsorbent dose of 0.5g, pH of 6 for Pb^{2+} and agitation speed of 200rpm). After adsorption is complete, the solution was filtered using Whatman no.1 filter paper. The residual Pb^{2+} concentrations of the effluent were determined using Atomic adsorption Spectrophotometer at 217.0nm wavelength. The percentage adsorbed was calculated from equation 1.

$$\% \text{ Adsorbed} = [(C_o - C_e) / C_o] \times 100 \dots\dots 1$$

Where, C_o and C_e are the metal concentrations (mg/l) at initial and any time t, respectively, and V the volume of the solution (l).

3. Results and Discussion

Characterization of activated carbon

Physico-chemical characteristics of activated carbon derived from palmyra palm nut are shown in Table 3. Good activated carbons must have low ash content. Typical ash content of activated carbons is around 5 – 6 %. A small increase in ash content causes a decrease in adsorptive properties of activated carbons. High ash content is undesirable for activated carbon since it reduces the mechanical strength of carbon and affects adsorptive capacity. The presence of ash has been shown to inhibit surface development (Valix et al 2004). Ash content obtained in this work is 3.40%. The ash content is well below the typical ash content of 12% obtained by Maheswari et al (2008). Usually moisture content

decreases as the temperature increases. Moisture content of 4.10% was obtained for activated carbon. Maheswari et al (2008) obtained moisture content of 4.33% in their work.

The value of volatile matter obtained for activated carbon was 18.14%. Lua and Guo (1999) studied preparation and characterization of chars from oil palm waste and obtained % volatile matter ranging from 74.86 to 4.08% between 450 to $950^{\circ}C$. The value of fixed carbon obtained for PPN activated carbon is 78.56%. Sivakumar and Palanisamy (2009) prepared activated carbon from Euphorbia Antiquorum L. and obtained 57.94% for fixed carbon. Lua and Guo (1998) reported values ranging from 23.7 to 87.13% within 450 to $950^{\circ}C$. The raw material, the particle size range used and the degree of activation affect bulk density, which does have an effect on adsorption per unit volume (Yemisoy-Karakas et al, 2004). $0.61g/cm^3$ was obtained as bulk density. Ahmad et al (2008) studied comparative adsorption studies for the removal of copper (II) from aqueous solution using different adsorption and obtained values ranging from 0.32 to $0.62cm^3$. Bulk density of $0.48g/cm^3$ was obtained by Sivakumar and Palanisamy, 2009.

Activated carbons with iodine numbers of about 550mg/g can be attractive for waste water treatment from the user's viewpoint (Dai et al, 1999). The iodine number value is an indication to surface area of the activated carbon (Abdullah et al, 2001). The iodine number of 785.78mg/g was obtained in this work. In their work, production of activated carbon from palm-oil shell by pyrolysis and steam activation in a fixed bed reactor, Vitidsant et al (1999) obtained maximum iodine number of 766.99mg/g at $750^{\circ}C$ and 3hrs. Castro et al (2008) obtained iodine number between 608 and 746mg/g. Each 1.0mg of iodine adsorbed is ideally considered to represent $1.0m^2$ of activated carbon internal area (Al-Quodah and Shawabkah, 2009). Surface area of $820.37m^2/g$ was obtained for the activated carbon. Castro et al (2008) obtained surface area ranging from 780 to $1100m^2/g$. The pH of activated carbon can be defined as the pH of a suspension of carbon in distilled water. The chemical nature of the carbon surfaces were globally inferred from the acidity or pH of the carbon. The carbon pH of the activated carbon is 6.8 which suggest weakly acidic surface properties. Similar result was obtained by Ahmed et al (2008). Valix et al (2004) obtained pH between 6.4 and 7.4 for activated carbon prepared from bagasse.

Table 3: Physico-chemical characteristics of activated carbon derived from palmyra palm nut.

Properties	Values
pH	6.8
Bulk density, g/cm ³	0.61
Iodine number, mg/g	785.78
Moisture content, %	4.10
Volatile matter, %	18.14
Ash content, %	3.40
Fixed carbon, %	78.56
Surface Area, m ² /g	820.37

Preparation of activated carbon

A three-variable Box-Behnken experimental design was employed to evaluate the preparation of activated carbon. The activation temperature and retention time plus the phosphoric acid /precursor impregnation ratio for the production of activated carbons were optimized based on the percentage removal of Pb²⁺ from aqueous solutions. The adsorption tests were used to analyze the best activated carbon for Pb²⁺ adsorption under different conditions of temperature, time and acid/precursor ratio. The software used for the statistical analysis is Minitab Release (Version 15). Table 4 shows predicted values and experimental results for percentage removal of Pb²⁺. The result of the Minitab output is given in Table 5. It can be seen from Table 4, that the optimum percentage removal, 95.85% for experimental and 95.73% for predicted values, was obtained for activated carbon produced at temperature of 500°C, 1hr of activation and concentration of 1.5:1 (acid/PPN ratio, wt/wt %). The result of analysis of variance (ANOVA) for the response surface quadratic model is shown in Table 5. The Resultant model is given by equation 1.

$$Y = 122 - 0.146A + 15.7B + 14.9C + 0.00052A^2 - 3.55B^2 - 4.66C^2 - 0.00057AB - 0.00207AC + 1.09BC \quad (1)$$

The P values were used as a tool to check the significance of each of the coefficients, which in turn are necessary to understand the pattern of the mutual interactions between the test variables (Shrivastava et al, 2008). The larger the magnitude of F-test value and the smaller the magnitude of P-values, the higher the significance of corresponding coefficient (Alam et al, 2008). Values of P less than

0.05 indicate that the model terms are significant. The fitness of the model equation was expressed by the coefficient of determination, R². R² indicates the fraction of the total variables of response variable that has been explained by the predictor variables. The greater the value of R², the better the fit and the more effective the estimated regression equation (Canavos and Koutrouvelis, 2009). The greater the magnitude of a T-value, the greater the relative accuracy of estimating the corresponding coefficients

From the result of analysis of variance, three main factors (temperature (A), time (B), and impregnation ratio, C) along with 3 second-order main effects (A², B² and C²) are significant model terms. Consequently, the model was reduced by elimination of insignificant terms to achieve a more desirable model as shown in equation 2.

$$Y = 122.08 - 0.147A + 15.92B + 15.05C + 0.00053A^2 - 3.615B^2 - 4.705C^2 \quad (2)$$

The elimination of insignificant terms did not affect the adequacy of the model based on the correlation coefficient (R²), adjusted correlation coefficient and predicted correlation coefficient for the reduced model which is 100% each as shown in Table 6. It can be seen that the mathematical coefficients of B and C are higher than others. This shows that time and concentrations of acid have higher effect on adsorption. The single factors (B and C) have positive signs, indicating that increasing time and concentration increases the amount of lead adsorbed while decreasing temperature increases amount of lead adsorbed. The second order terms (B² and C²) had negative sign, indicating negative effect on adsorption of lead.

The 3D surface plots and contour plots for the production of activated carbons are shown in Figures 1-3 and 4-6 respectively. The contour plots were studied to find optimum values of the combination of the independent variables. Each contour curve represents an infinite number of combinations of 2-test variables. It is seen from Figures 1-6 that decreasing the temperature and time of activation and acid impregnation ratio to 1.5 increased the percentage of Pb²⁺ adsorbed.

Table 4: Experimental and Theoretical values for percentage removal of Pb²⁺

Run	Temp of activation °C	Activation time (hour)	Impregnation ratio Acid: raw material	Experimental (% Rem.)	Predicted (% Rem.)
1	500	1	1.5	89.76	89.29
2	500	2	1.5	95.85	95.73
3	800	1	1.5	66.28	66.40
4	800	2	1.5	72.20	72.67
5	500	1.5	1	90.32	90.43
6	500	1.5	2	93.55	94.04
7	800	1.5	1	67.63	67.15
8	800	1.5	2	71.48	71.37
9	650	1	1	73.47	73.83
10	650	1	2	77.22	77.20
11	650	2	1	79.62	79.64
12	650	2	2	84.46	84.10
13	650	1.5	1.5	80.81	80.75
14	650	1.5	1.5	81.08	80.75
15	650	1.5	1.5	81.35	80.75

Table 5: Minitab output (ANOVA) for preparation of PPN based on Pb²⁺ adsorption

Predictor	Coefficient	St Dev	T-value	P-value	
Constant	122.41	10.18	12.03	0.000	
A	-0.14609	0.02127	-6.87	0.001	
B	15.743	5.236	3.01	0.030	
C	14.929	5.236	2.85	0.036	
A ²	0.00005174	0.00001477	3.50	0.017	
B ²	-3.553	1.329	-2.67	0.044	
C ²	-4.663	1.329	-3.51	0.017	
AB	-0.000567	0.004256	-0.13	0.899	
AC	0.002067	0.004256	0.49	0.648	
BC	1.090	1.277	0.85	0.432	
S = 0.638379	R-Sq = 99.8%	R-Sq (adj) = 99.5%	R-Sq (pred) = 98.19%		
Analysis of Variance					
Source	DF	SS	MS	F-value	P-value
Regression	9	1181.01	131.22	322.00	0.000
Error	5	2.04	0.41		
Total	14	1183.05			

Table 6: Minitab output for preparation of PPN based on Pb²⁺ adsorption (Reduced)

Predictor	Coefficient	StDev	T-value	P-value	
Constant	122.078	0.374	326.08	0.000	
A	-0.147125	0.000923	-159.32	0.000	
B	15.9200	0.1924	82.73	0.000	
C	15.0475	0.1924	78.20	0.000	
A ²	0.00005283	0.00000071	74.60	0.000	
B ²	-3.61500	0.06374	-56.72	0.000	
C ²	-4.70500	0.06374	-73.82	0.000	
S = 0.0306186	R-Sq = 100.0%	R-Sq (adj) = 100.0%	R-Sq (pred) = 100.0%		
Analysis of Variance					
Source	DF	SS	MS	F-value	P-value
Regression	6	1175.05	195.84	208897.68	0.000
Error	8	0.01	0.00		
Total	14	1175.06			

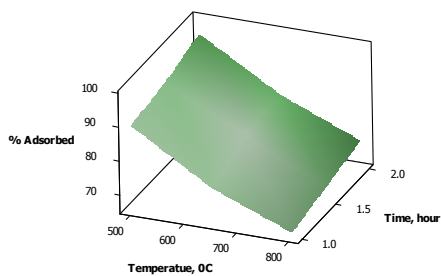


Figure 1: 3D surface plot for production of PPN for Pb²⁺ adsorption (Temperature/Time)

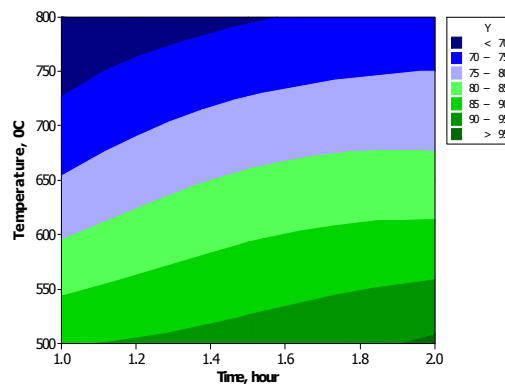


Figure 4: Contour plot for production of PPN for Pb²⁺ adsorption (Temperature/Time)

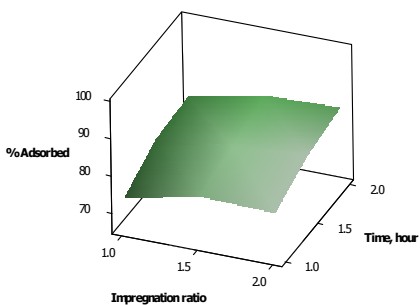


Figure 2: 3D surface plot for production of PPN for Pb²⁺ adsorption (Time/Impregnation)

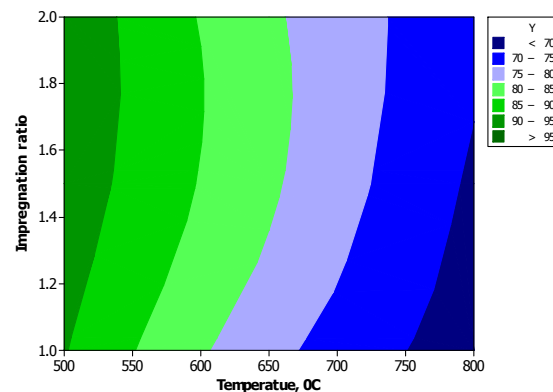


Figure 5: Contour plot for production of PPN for Pb²⁺ adsorption (Temperature/Impregnation ratio)

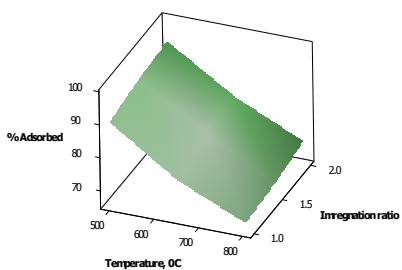


Figure 3: 3D surface plot for production of PPN for Pb²⁺ adsorption (Temperature/Impregnation)

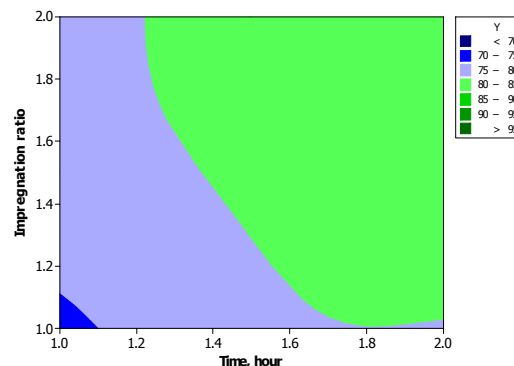


Figure 6: Contour plot for production of PPN for Pb²⁺ adsorption (Time/impregnation ratio)

Conclusion

Statistical optimization of production of activated carbon from nipa palm nut was carried using response surface methodology. Chemical activation using phosphoric acid at different activating conditions was used for preparing the activated carbon. The quality of the carbon was dependent on the preparation conditions. The optimum condition for preparing activated carbon for lead(II) removal from aqueous solution was obtained at activation temperature of 500°C, activation time of 1hr and acid impregnation ratio of 1.5:1 (acid/precursor, wt basis). The experimental results obtained agreed with the model predictions.

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