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RESEARCH ARTICLE

STATISTICAL OPTIMIZATION OF PRODUCTION OF ACTIVATED CARBON DERIVED FROM OIL PALM EMPTY FRUIT BUNCH

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ABSTRACT

Statistical optimization study for the production of activated carbon derived from oil palm empty fruit bunch (EFB) was carried out using response surface methodology. Chemical activation method using phosphoric acid was employed. 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 Pb^{2+} from aqueous solutions. The optimum conditions for preparing activated carbon from EFB for Pb^{2+} adsorption were as follows: activation temperature of 800^0C, activation time of 2hr and acid impregnation ratio of 1.5:1 (acid/precursor, wt basis) which resulted in 90.28% uptake of Pb^{2+}. The experimental results obtained agreed satisfactorily with the model predictions. The result of adsorption studies showed that activated carbon produced from EFB is a good adsorbent for the removal of Pb^{2+} from aqueous solution.

INTRODUCTION

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. These unique characteristics are responsible for its adsorptive properties, which are exploited in many different liquid-and gas-phase applications (Ruthven, 1984). Activated carbons are the most widely used industrial adsorbent for removing contaminants pollutants from gaseous, aqueous and non aqueous streams, due in part to their uniquely powerful adsorption properties and the ability to readily modify their surface chemistry (Roque-Marherbe, 2007).

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). There are two main methods for the preparation of activated carbons. These are physical and chemical activation methods. Physical activation occurs in two stages – thermal decomposition or carbonisation of the precursor and controlled gasification or activation of the crude char. The carbonisation process causes some increase in porosity, although this is generally insufficient for practical use, and serves to modify the pore structure inherent to the precursor, as opposed to creating it (Patrick, 1995). During carbonization, most of the non-carbon elements such as oxygen, hydrogen, nitrogen and sulphur are eliminated as volatile gaseous products by the pyrolytic decomposition of the source raw material to produce a carbon skeleton possessing a latent pore structure (Bansal and Goyal, 2005; Ruthven, 1984). The activation process helps to develop further this structure by selective gasification of carbon, usually in temperature range of $600\text{-}1200^0C$ in steam, CO_2 or O_2 or mixtures of these. Physical activation method has been used by these researchers in preparation of activated carbons (Arol and Yalcin, 2002; Valix *et al*, 2004). 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 1000^0C, followed by the elimination of the dehydrating agent by meticulous washing (Roque-Malherbe, 2007). With chemical activation, relevant reactions involve attack on the cellulosic structures in the precursor. Water is eliminated and cross-linking and increased aromatization is promoted during carbonization. The effect of cross-linking is to bond otherwise volatile products into the precursor structure before reaching pyrolysis temperatures, with a consequential increase in the carbon yield and reduction in tar formation (Ruthven, 1984). These researchers have employed chemical method in producing activated carbon from agricultural raw materials (Wan Nik *et al*, 2006; Hameed *et al*, 2006; Sricharoenchaikul *et al*, 2007).

Recently, preparation of activated carbons from agricultural by products has been given serious attention 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).

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The oil palm mills generate many by-products and wastes beside the liquid wastes that may have a significant impact on the environment if they are not properly dealt with. The most common among these by-products is the empty fruit bunch (EFB). The EFB is a solid waste product of the oil palm milling process and has a high moisture content of approximately 55-65% and high silica content, from 25% of the total palm fruit bunch (Keu, 2005). The main components of EFB are 50.4% cellulose, 21.9% hemicellulose, 10% lignin and 0.5% ash (Umikalsom *et al*, 1997). The present study is aimed at optimizing the conditions for the preparation of activated carbon from oil palm empty fruit bunch for the removal of Pb^{2+} from aqueous solution using surface response methodology.

MATERIAL AND METHODS

Preparation of activated carbon

Oil palm empty fruit bunches were obtained from Ichida women co-operative oil processing mill, Ichida, Anambra State, Nigeria. The raw material was washed several times using de-ionized water to remove all traces of impurities, oil, dirt, dust, etc. The raw material was dried in the sun to remove excess water until constant weight was obtained. The sun- dried raw material was 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 120^0C for 24hours. One step activation of the samples was performed in KGYV Budapest muffle furnace. The samples (20g each) were carbonized for 1hr, $1^1/_2$hrs and 2hrs at 500^0C, 650^0C and 800^0C 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 6.7, filtered with Whatman No.1 filter paper and then dried in the oven at 110^0C 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 (Table 2) was used for the production of activated carbon. 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, ^{0}C	500	650	800
Carbonization time, hour	1	1.5	2
Acid concentration/raw material ratio (wt)	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 ^{0}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 900^0C 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 ($Pb(N0_3)_2$), and de-ionized water. The reagents were of high grade. The sample $Pb(N0_3)_2$ was dried in an oven for 2hrs at 105^0C to remove moisture. 1.6g of $Pb(N0_3)_2$ 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 3hrs, 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 Absorption Spectrophotometer at 217.0nm wavelength. The percentage adsorbed was calculated from equation 1.

$$\%\ Adsorbed = [(C_o - C_e)/C_o]\ x\ 100 \qquad \text{...........1}$$

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).

RESULTS AND DISCUSSION

Characterization of activated carbon

Physico-chemical characteristics of activated carbon derived from oil palm empty fruit bunch are shown in Table 3. Ash content obtained in this work was 4.0%. The ash content is well below the typical ash content values of 8-12% obtained by Yenisoy-Karakas *et al.* (2004). Ash content is the measurement of the amount of mineral (e.g. Ca, Mg, Si and Fe) in activated carbon (Alam *et al.*, 2008). 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). Consequently low surface area is observed in carbons with high ash content (Pendyal *et al.*, 1999). Moisture content of 3.22% was obtained for activated carbon. Maheswari *et al* (2008) obtained moisture content of 4.33% in their work. Usually moisture content decreases as the temperature increases. The value of volatile matter obtained for activated carbon was 22.42%. 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 950^0C.

The value of fixed carbon obtained for the activated carbon is 73.58%. 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 950^0C. The bulk density obtained was 0.58g/cm^3. 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. 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). The iodine number of 802.45mg/g was obtained in this work. 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). 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 750^0C 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). The carbon pH of the activated carbon is 6.8 which suggests weakly acidic surface properties. 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. 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. Surface area of 850.11m^2/g was obtained for the activated carbon. Castro *et al.* (2008) obtained surface area ranging from 780 to 1100m^2/g.

Table 3. Physico-chemical characteristics of activated carbon derived from EFB

Properties	Values
pH	6.7
Bulk density, g/cm^3	0.58
Iodine number, mg/g	802.45
Moisture content, %	3.22
Volatile matter, %	22.42
Ash content, %	4.0
Fixed carbon, %	73.58
Surface Area, m^2/g	850.11

Preparation of activated carbon

To evaluate the preparation of activated carbon, a three-variable Box-Behnken experimental design was employed. The activation temperature and retention time plus the phosphoric acid /precursor impregnation ratio for the production of activated carbons were optimized on the basis of 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. Minitab Release (Version 15.0) was employed for the statistical analysis of the data obtained. Table 4 shows predicted values and experimental results for percentage removal of Pb^{2+}. The result of the Minitab output is given in Table 5. It can seen that the optimum percentage removal, 90.24% for experimental and 90.28% for predicted values, was obtained for activated carbon produced at temperature of 800^0C, 2hr of activation and concentration of 1.5:1 (acid/EFB ratio, wt/wt %). The result of analysis of variance (ANOVA) for the response surface quadratic model is shown in Table 5. Equation 1 gives the resultant model

$$Y = -30.6 + 0.172A + 14.62B + 8.56C - 0.000075A^2 - 3.51B^2 - 1.76C^2 + 0.00533AB - 0.00090AC + 0.030BC \tag{1}$$

The significance of each of the coefficients in the equation was determined by F-test and P values which in turn are necessary to understand the pattern of the mutual interactions between the test variables (Shrivastava *et al*, 2008). P values, F values, T values, coefficient of determination, R^2 and adjusted coefficient of determination, R^2 (adj.) are given in Table-4.The smaller the P-values and the larger the magnitude of F-test value, the higher the significance of corresponding coefficient (Alam *et al*, 2008). P values less than 0.05 was used to check the significance of each model term.The fitness of the model equation was expressed by the coefficient of determination, R^2, which was found to be 100%. R^2 indicates the fraction of the total variables of response variable that has been explained by the predicator variables. The greater the value of R^2, the better the fit and the more effective the estimated regression equation for estimation and prediction (Canavos and Koutrouvelis, 2009). The greater the magnitude of a T-value, the greater the relative accuracy of estimating the corresponding coefficients

From the ANOVA result, three main factors (temperature (A), time (B), and impregnation ratio, C) together with 2 second-other main effects (A^2 and B^2) and one interaction effect (AB) are significant model terms since their P values are less than 0.05. Consequently, the model was reduced by elimination of insignificant terms to achieve a more desirable model as shown in equation 2.

$$Y = -30.6 + 0.172A + 14.62B + 8.56C - 0.000075A^2 - 3.51B^2 - 1.76C^2 + 0.00533AB \tag{2}$$

Table 4. Experimental and Theoretical values for percentage removal of Pb^{2+}

	Temp of activation ^{0}C	Activation time(hour)	Impregnation ratio Acid: raw material	Experimental (% Rem.)	Predicted (% Rem.)
1	500	1	1.5	58.48	58.44
2	500	2	1.5	65.46	65.26
3	800	1	1.5	81.66	81.86
4	800	2	1.5	90.24	90.28
5	500	1.5	1	60.66	60.84
6	500	1.5	2	63.67	63.73
7	800	1.5	1	85.26	85.20
8	800	1.5	2	88.00	87.82
9	650	1	1	70.17	70.03
10	650	1	2	72.78	72.76
11	650	2	1	77.62	77.63
12	650	2	2	80.26	80.40
13	650	1.5	1.5	76.78	76.52
14	650	1.5	1.5	76.10	76.52
15	650	1.5	1.5	76.69	76.52

It can be seen that the mathematical coefficients of B, C and B^2 are higher than others. This shows that time and concentrations of acid have higher effect on adsorption. All the single factors have positive signs, indicating that increasing temperature, time and concentration increased the amount of lead adsorbed. The negative signs indicate negative effect on the uptake of lead from aqueous solution.

The 3D surface plots and contour plots for the production of activated carbons were generated from the Minitab software and presented in Figures 1-3 and 4-6 respectively. The contour plots were studied for determination of optimum values of the combination of the independent variables. From Figures 1-6, increasing the temperature, time of activation and acid impregnation ratio to 1.5 increased the percentage of Pb^{2+} adsorbed.

Table 5. Minitab output for preparation of EFB based on Pb^{2+} adsorption

Predictor	Coefficient	StDev	T-value	P-value	
Constant	-30.642	4.896	-6.26	0.002	
A	0.17154	0.01023	16.77	0.000	
B	14.631	2.518	5.81	00.002	
C	8.560	2.518	3.40	0.019	
A^2	-0.00007496	0.00000710	-10.56	0.000	
B^2	-3.5067	0.6391	-5.49	0.003	
C^2	-1.7567	0.6391	-2.75	0.040	
AB	0.005333	0.002047	2.61	0.048	
AC	-0.000900	0.002047	-0.44	0.679	
BC	0.0300	0.6141	0.05	0.963	
S = 0.307031	R-Sq = 100.0%	R-Sq (adj) = 99.9%		R-Sq (pred) = 99.71%	
Analysis of Variance					
Source	DF	SS	MS	F-value	P-value
Regression	9	1318.27	146.47	1553.8	0.000
Error	5	0.47	0.09		
Total	14	1318.74			

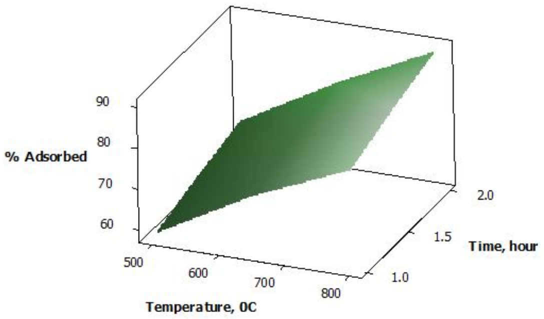

Figure 1. 3D surface plot for production of EFB for Pb^{2+} adsorption (Temperature/Time)

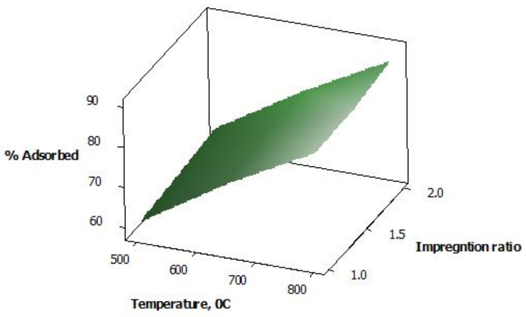

Figure 2. 3D surface plot for production of EFB for Pb^{2+} adsorption (Temperature/Impregnation ratio)

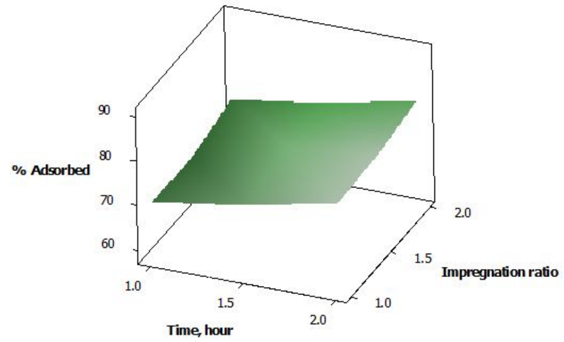

Figure 3. 3D surface plot for production of EFB for Pb^{2+} adsorption (Time/impregnation ratio)

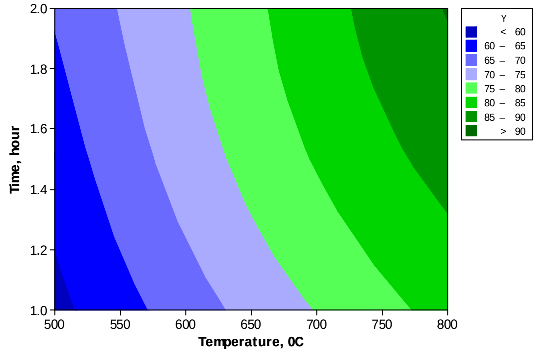

Figure 4. Contour plot for production of EFB for Pb^{2+} adsorption (Temperature/Time)

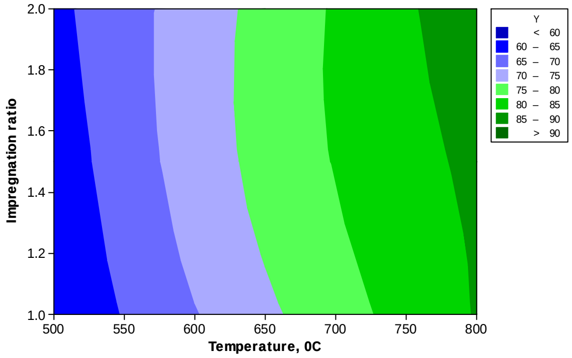

Figure 5. Contour plot for production of EFB for Pb^{2+} adsorption (Temperature/Impregnation ratio)

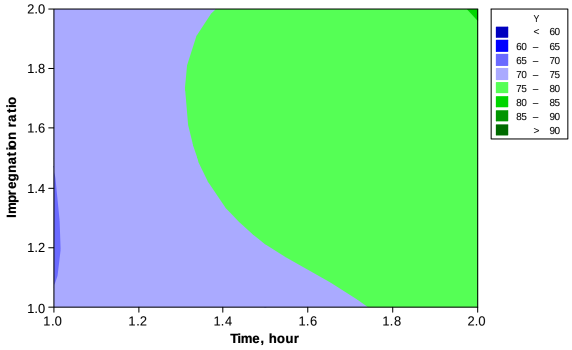

Figure 6. Contour plot for production of EFB for Pb^{2+} adsorption (Time/impregnation ratio)

Conclusion

Statistical optimization of production of activated carbon obtained Box-Behnken experimental design. Phosphoric acid as an activating agent was used for preparing the activated carbon at different activating conditions. It is seen that the quality of the activated carbon was dependent on the preparation conditions. The optimum condition for preparing activated carbon for lead (11) removal from aqueous solution was obtained at activation temperature of 800^0C, activation time of 2hr and acid/precursor impregnation ratio of 1.5:1 (wt basis). The experimental results obtained agreed with the model predictions.

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