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Evaluation of the Performance of Ethylene Di-Amine Tetra-Acetic Acid Modified Activated Carbon for Lead ion Adsorption from Palm Kernel Shell

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

There is increasing research on the adsorption of lead because its use in industrial processes has resulted in various forms of environmental contamination and negative human health issues. Currently, researchers have intensified their search for low-cost adsorbents like activated carbon produced from nonfossil sources. This study is focused on the use of Ethylene Di-Amine Tetra-Acetic Acid for the adsorption of Lead ions. It was conducted to optimize the process variables in the production of Palm Kernel Shell Activated Carbon modified with Ethylene Di-Amine Tetra-Acetic Acid. A 2^3 three-level Central Composite Design was used to develop a statistical model for the optimization of the time (10-130) X_1, pH (5.0 – 7.0) X_2, and adsorbent dose (0.4 -5.0g)X_3. Data obtained from RSM on activated carbon production were subjected to ANOVA and analyzed using a second-order polynomial equation. The extent of lead ion removal by Ethylene Di-Amine Tetra-Acetic Acid activated carbon from aqueous solution was 96.30% at the solution pH of 7.2, contact time of 70 minutes, and an adsorbent dose of 2.1g/L. The Langmuir isotherm model was in good agreement with the experimental data.

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Keywords: Adsorption; optimization; activated carbon; palm kernel shell; response surface methodology; bioadsorbent; lead ion.

NOMENCLATURE

EDTA : Ethylene Di-Amine Tetra-Acetic Acid
PKS : Palm Kernel Shell;
AC : Activated carbon;
PKS-AC : Palm kernel shell - Activated carbon

1. INTRODUCTION

The environment is contaminated by different factors which create widespread environmental problems due to their adverse effects. Some heavy metals present in wastewater constitute serious environmental problems because of their inability to degrade. Lead is a naturally occurring heavy metal that is toxic. Possible sources of contamination of lead include mining, smelting, manufacturing, and recycling activities. It is used in the manufacturing of pigments, paints, jewelry, ammunition, crystal, glassware e.t.c. In addition, it has been observed that more than three-quarters of the global lead consumption is used in the manufacturing of lead-acid batteries for vehicles. Moreover the contamination may be from drinking water delivered through lead pipes [1,2,3]. To purify and keep the environment clean, the development of optimization tools to produce highly efficient and effective adsorbents such as activated carbon.

Biomass-based activated carbon has received considerable attention due to its excellent characteristics such as inexpensiveness, good absorption behaviour, and potential to reduce strong dependence towards nonrenewable precursors [4,5]. Activated Carbon (AC) is one of the most effective adsorbents owing to its well-developed porous structure, large active surface area, and good mechanical properties [6]. Researchers are intensifying their search on AC produced from nonfossil sources like lignocellulosic waste from agriculture which are abundant and largely available [7,8]. Activated carbon is mostly used because its chemical and physical properties can be designed and adjusted according to the required applications. Thus, this has increased the demand for activated carbon and as well as the cost. This fact has prompted new frontiers in the production of low-cost activated carbon. Recently, researchers have been reported for preparing cheaper and readily available precursors for activated carbon from agricultural wastes such as bamboo, bagasse, groundnut shells, peanut shells, coconut shells, etc [9].

A large amount of palm kernel shells are produced as waste in Nigeria. Previous works in this area include the use of palm kernel shells, natural ferric oxide, tea leaves, groundnut husks, cocoa shells, e.t.c as adsorbents for the removal of heavy metals from waste water [10]. The palm kernel shell (PKS) of oil palm is useful as an adsorbent for the removal of heavy metal ions, because the organic compounds within it are capable of adsorption of metal ions through biosorption mechanisms. PKS is a sustainable agricultural waste produced in millions of tons every year. The disposal of large quantities of PKS causes adverse effects on the environment as it is disposed of by burning resulting in a lot of smoke [11].

Table 1. Adsorption capacity (mg/g) of lead by different adsorbents Adopted from [12]

Adsorbent	pH	Lead (IT)
Natural ferric oxide		230.0
Tea leaves		78.7
Clay		0.12
Groundnut husk		39.3
Sphagnum moss peat		30.7
Olive mill residue	5.6	21.56
Sago waste	2-5.5	46.64
Acc	2-10	30.45
Carex peat	7.8	83%
Cocoa shells	2	6.2
Bone powder	3 and 5	29.5 and 53.3
Active carbon	3 and5	32.7 and 41
Nile rose plant powder	3 and 5	19.7 and 27.4
Commercial carbon	3 and5	9.8 and 27.3
Palm shell activated carbon	3 and5	82.0 and 95.2

Currently, the focus is shifting to the use of low cost materials because the economy of the process depend on the cost of the adsorbent materials [12]. Table 1 shows the adsorption capacities for different adsorbents.

In this research work, Ethylene Di-Amine Tetra-Acetic Acid (EDTA) is used in activating the PKS-AC bio sorbent for the adsorption of lead. Previous research showed that EDTA has not been used for lead adsorption. This research is also geared towards the use of RSM for optimizing the parameters in producing AC by using palm kernel shells as low-cost, readily available adsorbents for heavy metal removal.

2. MATERIALS AND METHODS

2.1 Carbonization

Palm kernel shells obtained from Okada, Edo state, were thoroughly washed with running water three times and distilled water to eradicate possible sand and dirt according to [13]. It was sun-dried for 24 hours and then oven-dried at temperature 105°C for 4 hours and ground with a mechanical grinder and sieved with a mesh sieve of 250µm. The carbonization process was achieved by weighing a batch of 156g of sample in clean silica crucibles and heated in a muffle furnace (Mechanical Lab) at 600°C for 3hour according to [14].

2.2 Activation Process

156 g of the carbonized palm kernel shells was weighed into a 500ml beaker and mixed with 200cm^3 of 1.0 M EDTA. The content of the beaker was boiled on a hot plate with stirring at intervals using a stirring rod until the water evaporated and then subjected to thermal activation in a muffle furnace at 400°C for 2 hours. The activated carbon was then washed with distilled water to remove excess salt and dried at 105°C in an oven.

2.3 Preparation of Aqueous Solutions of Lead

100 mg of heavy metal Pb, H_2SO_4 was added to 500ml distilled water and boiled for 1 hour on a hot plate. After boiling, add distilled water to make it up to 1000ml.

2.4 Adsorption Study

100ml of the prepared aqueous solution was measured and transferred into a beaker after which the pH was measured. The prepared solution was transferred into a conical flask, and the required mass of AC was added to the solution. The mixture was stirred using a magnetic stirrer for a required period. The decanted solution was stored as a filtrate in plastic bottles for Atomic Absorption Spectroscopy (AAS) analysis. The AAS was used to analyze the concentrations of the Pb metal ions present in the filtrate according to [15]. The amount of Pb ions adsorbed by the adsorbent was evaluated using:

$$q_t = \left(\frac{C_o - C_t}{W}\right) V \quad (1)$$

The mass balance equation is used to determine the adsorptive capacity (q_e) from [16].

$$q_e = \frac{(C_o - C_g)V}{W} \quad (2)$$

Where C_0 and C_t = the initial and final concentration of the heavy metals present in the wastewater before and after adsorption, for a time t (mg/L). C_e = concentration of heavy metals in wastewater when equilibrium is attained, mg/L

V = volume of wastewater used, ml

W = mass (g) of the adsorbent used

The percentage of metal ions removed was obtained using [17].

$$R\ (\%) = \frac{(C_o - C_t)}{C_o} * 100 \quad (3)$$

Where R (%) is the ratio of differences in metal concentration before and after adsorption

2.5 Design of Experiment (DOE)

This is a systematic method to determine the relationship between the factors affecting a process and the output of that process. A central composite design was used. Central composite design is a factorial design useful in response surface methodology, building a second-order model for the response variable without the need to use a complete three-level factorial experiment.

The investigation of adsorption parameters in the process are as follows:

2.5.1 Effect of pH

his was done to determine the efficiency of the adsorbent, the pH of wastewater is one of the imperative factors governing the adsorption of

metal ions. Therefore, it is cheaper and easier to control the pH. In this study, a pH of 4 - 10 was used as estimated in DOE.

2.5.2 Effect of Adsorbent Dose

The dose of adsorbent strongly affects the sorption phenomenon. This may be due to the availability and accessibility of surface active sites resulting from an increased dose of adsorbent under specific conditions. The range of the adsorbent used in this project is 0.4 – 5.0g, and it is agitated to the required mass in the solution as estimated by DOE and decanted.

2.5.3 Effect Contact time

This determines the optimum time for the removal of the metal ions (adsorption). The time interval used in this study is 10 - 130 minutes, it is agitated for the required period estimated by the DOE.

3. RESULTS AND DISCUSSION

3.1 Properties of Palm Kernel Shell Activated Carbon (PKS-AC)

The adsorption efficiency of activated carbon is directly related to the total surface area of the carbon such that the larger the surface area, the higher the adsorption efficiency of the carbon [18] and hence an important attribute while considering the selection of adsorbents in the separation process. From the data shown in Table 2, the total surface area produced in the laboratory had a large total surface area of 1038 m^2/g. Ash content is a measure of minerals as impurities in the carbons mainly derived from the carbon precursor.

Table 2. Characteristics of prepared PKS - AC

Property	Value
Ash Content, wt%	0.1
Total surface area, m^2g	1038
Particle size, μm	250
pH	7.1

3.2 Effects of Process Parameters on Metal Adsorption

Adsorption was carried out simultaneously with varied parameters as obtained by the software (DESIGN EXPERT version (8.0.6) using Response surface methodology (RSM). It is observed that the total metal ions removal was affected by contact time, initial pH, pollutant concentration, and adsorbent dose. In this study, all these were been explored to evaluate a treatment technology for lead removal from synthetically simulated solutions.

3.3 Effect of Contact Time

The contact time determines the optimum time for the removal of the metal ions (adsorption). The lead removal from aqueous solution was studied as a function of contact time in the range of 10 to 130 minutes at about 50mg/L initial metal concentration, 7.16 pH, and 30ºC ambient temperature. The effect of contact time on the lead removal is shown in Fig. 1, wherein it was observed that the rate of lead removal was higher at the beginning until 65mins, thereafter, the adsorption rate became very slow, after 92.3 min, it decreased. The difference in the degree of adsorption may be because initially, the sites on the surface of the adsorbent were vacant and the solute concentration gradient was high. As a result, the extent of lead removal decreased with an increase in contact time.

3.4 Effect of pH

The pH is one of the major factors governing the adsorption of metal ions. Therefore, it is cheaper and easier to control the pH. From Figs. 1 and 2, a pH of 7.20 was considered as the optimum pH for lead ion removal for this work, which agrees with [19].

It is a known fact that the pH of the solution has a significant effect on the uptake of the metal ions because it determines the surface charge of the adsorbent, the degree of ionization, and the specification of the adsorbate. The pH also has a large effect on the efficiency of the electrocoagulation process since the pH changes during the process depending on the anode material and the initial pH value of the treated solution. From Fig. 1, the percentage adsorption was observed to increase with the increase in pH with an optimal point of pH 7.2. Minimum adsorption was shown at high pH, contrary to the fact that lower pH favors adsorption which could be because of the presence of higher mobility and higher concentration of H+ ions. The optimum result was obtained at almost neutral pH due to the surface of the adsorbent becoming more positively charged at high H+ concentration. [20] observed that the maximum adsorption was within the pH range 6 to 8, which

might be due to partial hydrolysis of metal ions. From the results, it was clear that the metal uptake was 96.30% at an optimum pH of 7.

3.5 Effect of Adsorbent Dose

The results of the adsorbent dose on the percentage removal of Pb on the activated carbon are reflected in Fig. 3. It is worthy of note that the metal uptake changed slightly with adsorbent dose 1.8g/L to 2.4g/L and then started decreasing continuously which attributed to increased adsorbent surface area and the availability of more sites for adsorption. However, at 2.7g/L adsorbent dose, the metal ions adsorbed per unit weight of adsorbent decreased, this could be because at the higher adsorbent dose the solution ion concentration drops to a lower value of q (amount adsorbed) indicating that the adsorption sites remained unsaturated as agreed by [18]. Based on these results, 2.15g/L was taken as the optimum adsorbent dose for the experiments.

"Sequential Model Sum of Squares [Type I]": Select the highest order polynomial where the additional terms are significant and the model is not aliased (Table 3).

"Lack of Fit Tests": Want the selected model to have insignificant lack-of-fit (Table 4).

"Model Summary Statistics": Focus on the model maximizing the "Adjusted R-Squared" and the "Predicted R-Squared" (Table 5).

The Model F-value of 9.84 implies the model is significant. There is only a 0.07% chance that a "Model F-value" this large could occur due to noise.

Values of "Prob > F" less than 0.0500 indicate the model terms are significant.

In this case A, A^2 are significant model terms.

Values greater than 0.1000 indicate the model terms are not significant.

If there are many insignificant model terms (not counting those required to support hierarchy),

Model reduction may improve your model.

The "Lack of Fit F-value" of 1.47 implies the lack of fit is not significant relative to the pure error. There is a 34.15% chance that a "Lack of Fit F-value" this large could occur due to noise.

Std. Dev.	2.84	R-Squared	0.8986
Mean	5.70,	Adj R-Squared	0.8073
C.V. %	49.78	Pred R-Squared	0.4815
PRESS	412.19	Adeq Precision	10.997

The "Pred R-Squared" of 0.4815 is not as close to the "Adj R-Squared" of 0.8073 as one might normally expect. This may indicate a large block effect or a possible problem with your modelsand/or data. Things to consider are model reduction, response transformation, outliers (Tables 6- 7).

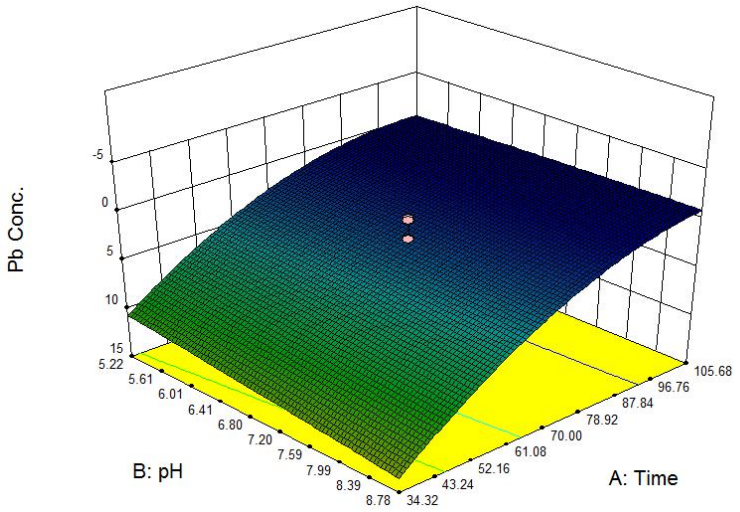

Fig. 1. 3D response surface of the effects of pH and time on metal lead ion concentration

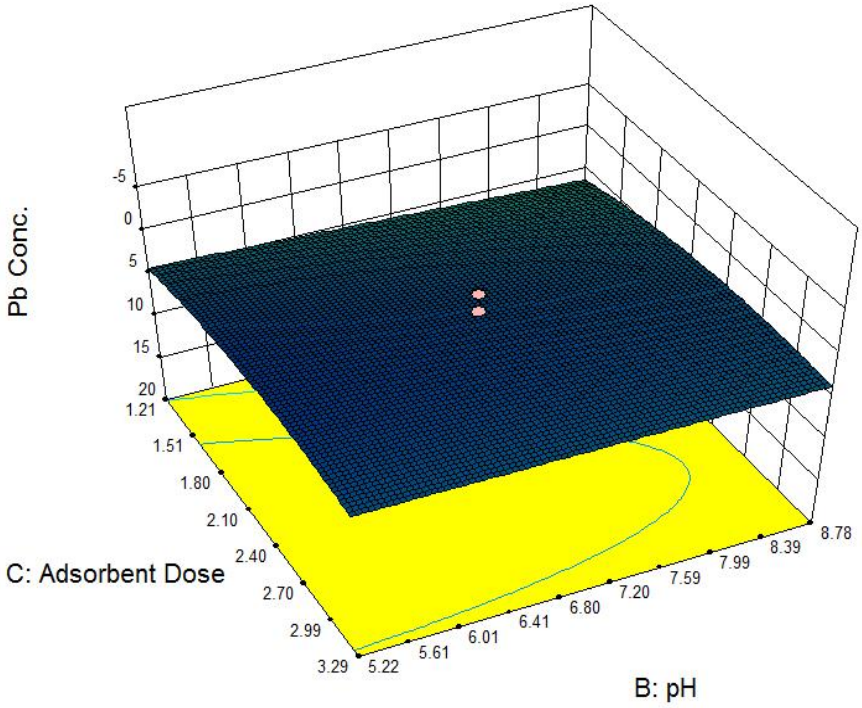

Fig. 2. 3D response surface of the effects of adsorbent dose on lead with pH

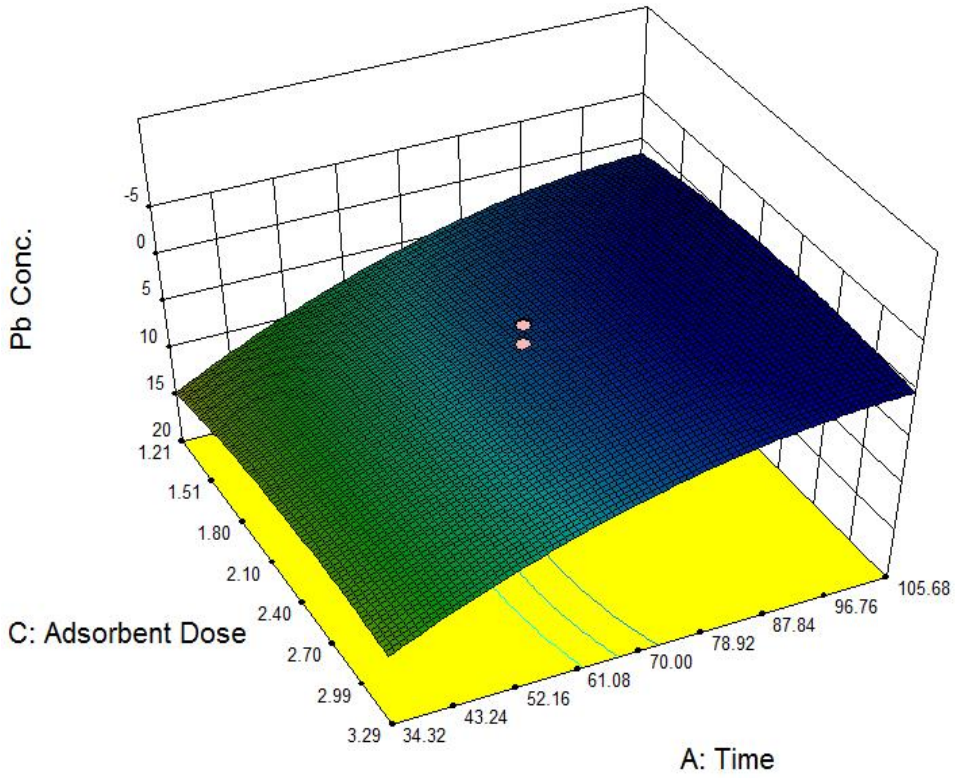

Fig. 3. 3D response surface of the effects of adsorbent dose on lead time

Table 3. Analysis of variance (ANOVA) for the response surface quadratic model Response 1 Pb Conc. Transform: None (Sequential Model Sum of Squares [Type I])

Source	Sum of Squares	df	Mean Square	F Value	p values Prob > F
Mean vs Total	650.83	1	650.83		
Linear vs Mean	565.62	3	188.54	13.15	0.0001
2FI vs Linear	8.12	3	2.71	0.16	0.9220
Quadratic vs 2FI	140.66	3	46.89	5.81	0.0145
Cubic vs Quadratic	18.84	4	4.71	0.46	0.7656
Residual	61.80	6	10.30		
Total	1445.86	20	72.29		

Table 4. Lack of fit tests

Source	Sum of Squares	df	Mean Square	F Value	p values Prob > F
Linear	196.76	11	17.89	2.74	0.1380
2FI	188.64	8	23.58	3.61	0.0868
Quadratic	47.99	5	9.60	1.47	0.3415
Cubic	29.15	1	29.15	4.46	0.0883
Pure Error	32.66	5	6.53		

Table 5. Model summary statistics

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	Press
Linear	3.79	0.7114	0.6573	0.5445	362.14
2FI	4.13	0.7216	0.5932	0.0909	722.79
Quadratic	2.84	0.8986	0.8073	0.4815	412.19
Cubic	3.21	0.9223	0.7538	-7.1407	6472.14

Table 6. ANOVA for response surface quadratic model Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p values Prob > F
Model significant	714.39	9	79.38	9.84	0.0007
X_1-Time	551.05	1	551.05	68.33	< 0.0001
X_2-pH	5.06	1	5.06	0.63	0.4467
X_3-Adsorbent Dose	9.51	1	9.51	1.18	0.3031
X_1X_2	6.27	1	6.27	0.78	0.3988
X_1X_3	0.77	1	0.77	0.095	0.7638
X_2X_3	1.08	1	1.08	0.13	0.7220
X_1^2	122.21	1	122.21	15.15	0.0030
X_2^2	0.081	1	0.081	9.990E-003	0.9224
X_3^2	27.07	1	27.07	3.36	0.0968
Residual	80.64	10	8.06		
Lack of Fit not significant	47.99	5	9.60	1.47	0.3415
Pure Error	32.66	5	6.53		
Cor Total	795.03	19			

Table 7. Interaction of the process parameters

Coefficient Factor	Standard Estimate	95% CI df	95% CI Error	Low	High
Intercept	2.73	1	1.16	0.15	5.31
X_1-Time	-6.35	1	0.77	-8.06	-4.64
	1.00				
X_2-pH	0.61	1	0.77	-1.10	2.32
	1.00				
X_3-Adsorbent Dose	-0.83	1	0.77	-2.55	0.88
	1.00				
X_1X_2	-0.88	1	1.00	-3.12	1.35
	1.00				
X_1X_3	0.31	1	1.00	-1.93	2.55
	1.00				
X_2X_3	-0.37	1	1.00	-2.60	1.87
	1.00				
$X_1$2	2.91	1 0.75	1.25	4.58	1.02
$X_2$2	0.075		0.75	-1.59	1.74
	1.02	1			
$X_3$2	1.37		0.75	-0.30	3.04
	1.02	1			

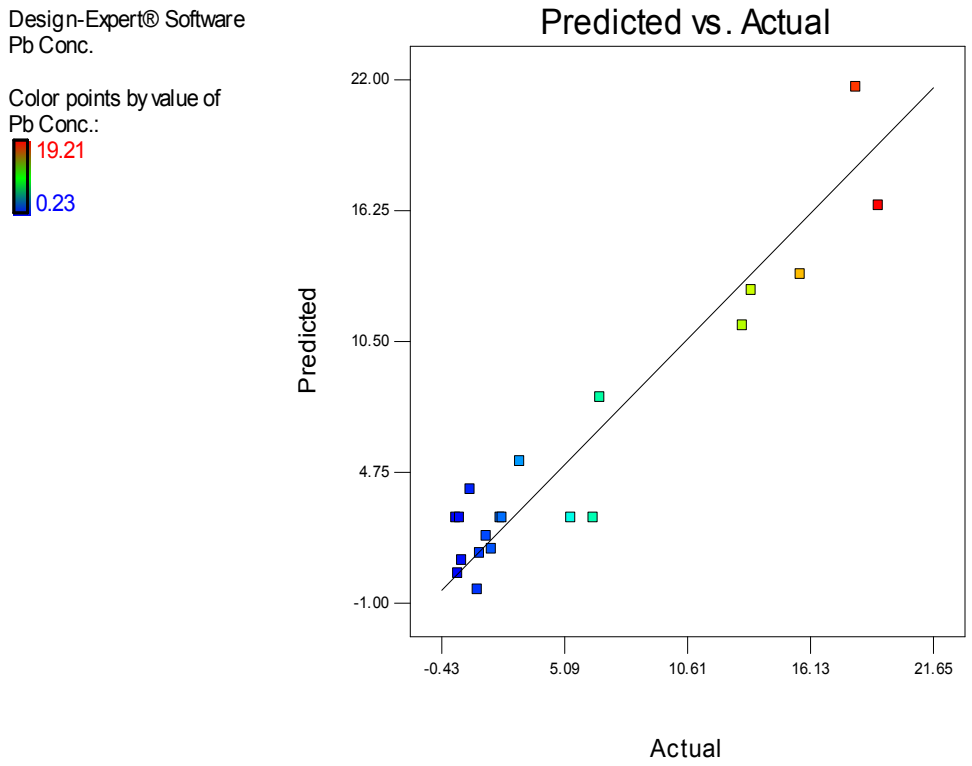

Fig. 4. Parity plot of predicted against actual metals ions Pb^{2+}concentration

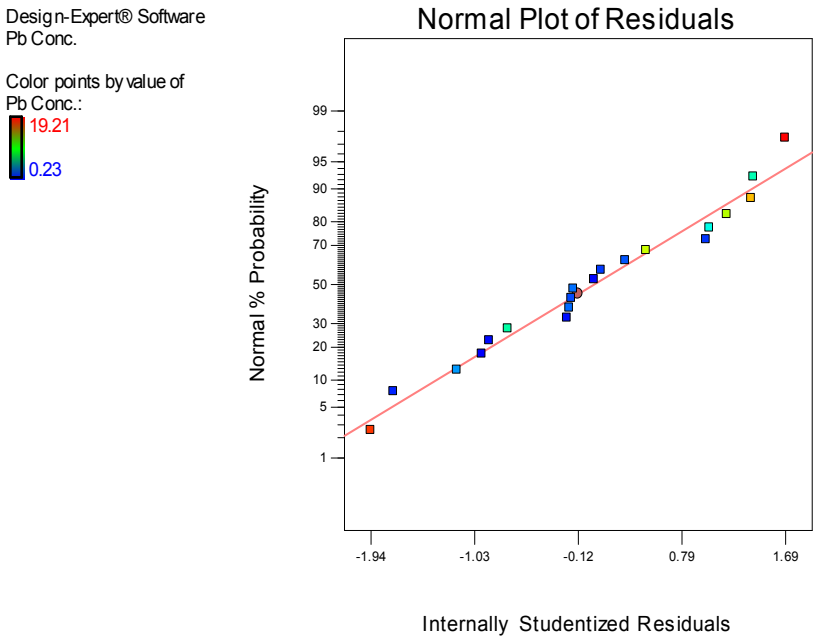

Fig. 5. Normal plot of residuals for the response

"Adeq Precision" measures the signal-to-noise ratio. A ratio greater than 4 is desirable. Your Ratio of 10.997 indicates an adequate signal. This model can be used to navigate the design space.

Final Equation in Terms of Coded Factors:

$$Y_{Pb\ Conc.} = +2.73 - 6.35X_1 + 0.61X_2 - 0.83X_3 - 0.88X_1X_2 + 0.31X_1X_3 - 0.37X_2X_3 + 2.91X_1^2 + 0.075X_2^2 + 1.37X_3^2$$

Final Equation in Terms of Actual Factors:

$$Pb\ Conc. = +24.76114 - 0.41980*Time + 1.43118*pH - 5.69648*Adsorbent\ Dose - 0.013906*Time*pH + 8.35059E\text{-}003*Time*Adsorbent\ Dose - 0.19799*pH*Adsorbent\ Dose + 2.28791E\text{-}003*Time^2 + 0.023497*pH^2 + 1.26579*Adsorbent\ Dose^2$$

Parity Plot

The relationship between the predicted and actual metal ions concentrations is shown in Fig. 4, which was plotted by the software. It can be observed from the plot that the data points are distributed near the straight line. This is an indication that there is a high correlation (R^2 value close to unity), which means that the data fit well with the model and convincingly is a good estimate of response for the system in the ranges studied. This further indicates that the model could be employed as the signature model for predicting response over the independent input variables.

Normal Plot

Fig. 5 shows the normal plot of residuals for the responses. The residual gives the difference between the observed value of a response measurement and the value that is fitted under the theorized model [21]. A closer examination of this plot shows that most of the data points are closed to the line, having some points scattered. This is expected with normal data. From the observed distribution, it could thus be inferred that the data were normally distributed.

In line with the bioadsorption model and mechanism in [22], this work fitted well with the Langmuir Isotherm with an R^2 value of 0.8978.

4. CONCLUSION

In this study, it was deduced that the effects of parameters such as adsorbent dose, adsorbent contact time, and pH on Pb metal ion transfer

separation have been investigated. Based on the investigation of lead ion removal by EDTA activated carbon, the results showed that it is capable of removing metal ions from aqueous solution to the extent of 96.30% at a solution pH of 7.2, contact time of 70 minutes, and an adsorbent dose of 2.1g/L. This will be beneficial to manufacturers of lead batteries, effluent treatment, and other related industries. Furthermore, based on the value of R^2 calculated, which is equivalent to 0.8978, the Langmuir isotherm model was found to be in good agreement with the experimental data on the adsorptive behavior of lead ions. The adsorption results are in line with the linear and quadratic model representation, which is evident from the models for optimization of lead ions.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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