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Original Research Article

Modeling and Optimization of Cadmium and Lead Adsorption onto Natural *Pterocarpus santalinoides* Fruit

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Abstract

The presence of heavy metals in wastewater has raised concern in developing countries because of their impact on human health and environmental ecology. Therefore, this study aims to remove cadmium and lead ions from wastewater using natural *Pterocarpus santalinoides* fruit through the adsorption method. The Box-Behnken design (BBD) of the response surface methodology (RSM) method was adopted to optimize the process variables such as contact time (20-100 min), adsorbent dose (0.1-0.5 g), initial metal ion concentration (10-50 mg/l), and temperature (30-70 °C). The ANOVA results clearly indicate that the linear model was not sufficient to best predict the removal performance of cadmium ($R^2 = 0.4009$) and lead ($R^2 = 6353$). The optimum conditions for the maximum Cd (94.81%) and Pb (89.23 %) adsorption onto the adsorbent were achieved at contact time (42.16 min), adsorbent dose (0.25 g), initial metal ion concentration (21.52 mg/l), and temperature (37.00 °C). According to the findings of the present work, *Pterocarpus santalinoides* shows to be a potential eco-friendly and cheap adsorbent for the removal of Cd and Pb from aqueous solutions. The BBD-RSM actual and predicted values of the Cd and Pb ions response show non-significant correlation, suggesting poor agreement between the two, revealing that the BBD-RSM model applied is not effective for the relationship between the four parameters examined in the Cd and Pb ions removal process.

Keywords: *Pterocarpus santalinoides* fruit, Adsorption, heavy metal, response surface methodology, Box-Behnken design.

INTRODUCTION

Water is vital for the survival of all living creatures. It is important for consumption, irrigation, the industrial sector, and many other uses. Heavy metal pollution of water bodies is one of the major challenges in the developing countries because they tend to bioaccumulate and cause serious threats to human beings (Kwikima *et al.*, 2021). Heavy metals are the most prevalent pollutants found in water. Heavy metals have been identified as environmental contaminants at different concentrations and are released to the water sources through untreated industrial effluents, municipal wastewater, and agricultural runoff (Gupta *et al.*, 2021; Farhadi *et al.*, 2021). The major four heavy metals of individual health concern are cadmium, arsenic, lead, and mercury, respectively. The inability of the human body to metabolize these heavy metal ions is of great concern because of their detrimental effect on the human system and the environment (Simon *et al.*, 2022). The accumulation of heavy metals in living cells causes reduced organ growth, cancer, disruption of the body's nervous system, defense system, and death in severe cases (Tsade *et al.*, 2020; Gupta *et al.*, 2021.).

However, the main aim of wastewater treatment is the removal of contaminants, especially heavy metals, before being released into the ecosystem. Various predictable physical and chemical methods applied for removal of heavy metals from industrial wastewater include chemical precipitation, chemical oxidation/reduction, ion exchangers, co-precipitation, complexation, electrochemical remediation, adsorption, and reverse osmosis (Ummul *et al.*, 2023; Alalwan *et al.*, 2020). However, the disadvantages of these methods are low removal efficiency, sensitive operating conditions, and the generation of secondary sludge, which increases the operational cost (Neoh *et al.*, 2016). Among the aforementioned techniques, the adsorption process is generally considered an efficient and effective method for the eradication of heavy metals from wastewater (Ullah *et al*., 2020). The adsorption process is an environmentally friendly technique due to its low cost, flexibility, high selectivity, availability of waste biomass, simple operation, regeneration, and remarkable ability to remove toxic metal ions from their complexes (Ummul *et al*., 2023). Over the last few decades, various natural and synthetic materials from industrial or agricultural sources have been applied as adsorbents for the removal of heavy metals from wastewater. However, a number of these adsorbents do not fulfill the criteria of low cost, readily available, and high adsorption efficiency in the removal of toxic substances (Nleonu *et al*., 2023; Onyemenonu *et al*., 2023). Therefore, the search for highly efficient materials with a wide range of applicability and adsorption of a wide range of heavy metals is extremely important.

Currently, natural agricultural waste has been studied by scholars as a viable adsorbent for the elimination of heavy metals from wastewater because of their good surface chemistry and well-developed permeable structure with a high specific surface area (Ahmed *et al*., 2023). The major objective of this work is to produce adsorbent materials that are beneficial for removing cadmium and lead from aqueous solutions. Moreover, the adsorption efficiency of natural *Pterocarpus santalinoides* fruit towards cadmium and lead ions will be analysed using the response surface methodology (RSM). The response surface methodology-based box-behnken design (RSM-BBD) method will be utilized to optimize cadmium and lead adsorption onto natural *Pterocarpus santalinoides* fruit. The various key variables studied were contact time, adsorbent dose, initial metal ion concentration, and temperature, respectively.

MATERIALS AND METHOD

All chemicals were of analytical grade and used without further pretreatment. Cadmium chloride ($CdCl_2$, 99%) and lead chloride ($PbCl_2$, 99%) were used in the present study. Deionized water was used throughout the experiments.

Design of Experiments using RSM-BDD in this study, the Box-Behnken design (BBD) of response surface methodology (RSM) was employed to optimize the processing variables estimating the regression model equation. Four experimental factors with three levels were selected as shown in Table 1. The contact time, adsorbent dose, initial metal ion concentration, and temperature were set as factors; percentage removal efficiency was set as a responsive variable. Based on factorial experimental design, 29 runs (Table 2) were generated from design expert software version 13 using BBD under the RSM approach.

Batch Adsorption Experiment

The adsorption experiments were performed in batch mode and on a laboratory scale. The effects of adsorbent dose (0.1-0.5 g/l), contact time (20-100 min), initial metal ion concentration (10-50 mg/l), temperature (30-70 ℃), and Cd and Pb ion removal were studied. For each adsorption study, 100 ml of Cd and Pb ions solution were studied based on BBD factors. The residual metal ion concentrations were directly measured using an atomic absorption spectrophotometer, as reported elsewhere (Onyemenonu *et al*., 2015).

The percentage removal of Cd and Pb adsorbed on the natural and activated carbon of *Pterocarpus santalinoides* fruit was obtained as follows (1) (Onyemenonu *et al*., 2023).

$$\%R = \frac{C_o - C_e}{C_o} \times 100 \qquad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where C_o is the initial metal ion concentration (mg/l), Ce is the equilibrium concentration of metal ion (mg/l).

Table 1: Box behnken design of experimental range and coded levels of the selected process variables

Variable (Factors)	Coding	Unit	Design range and coded level		
			-1	0	+1
Contact time	A	Min	20	60	100
Adsorbent dose	B	g/l	0.1	0.3	0.5
Initial metal ion concentration	C	Mg/l	10	30	50
Temperature	D	℃	30	50	70

RESULTS AND DISCUSSION

Interaction Effect of Selected Variables on the Removal of Cadmium and Lead by *Pterocarpus santalinoides* Fruit

Box-Behnken design created three-dimensional (3D) response surface plots against any two independent process variables, keeping the other process variables at their central (O) level in order to examine the interaction between the different process variables and their corresponding effects on the response (Cd and Pb removal efficiency). Figure 1a displays 3D surface plots demonstrating the interactions between processes between adsorbent dose and contact time for Cd and Pb adsorption and their corresponding output responses. The removal of cadmium shows no interactions between the process variables and their corresponding output responses, while the removal of lead shows six interactions, where none were statistically significant.

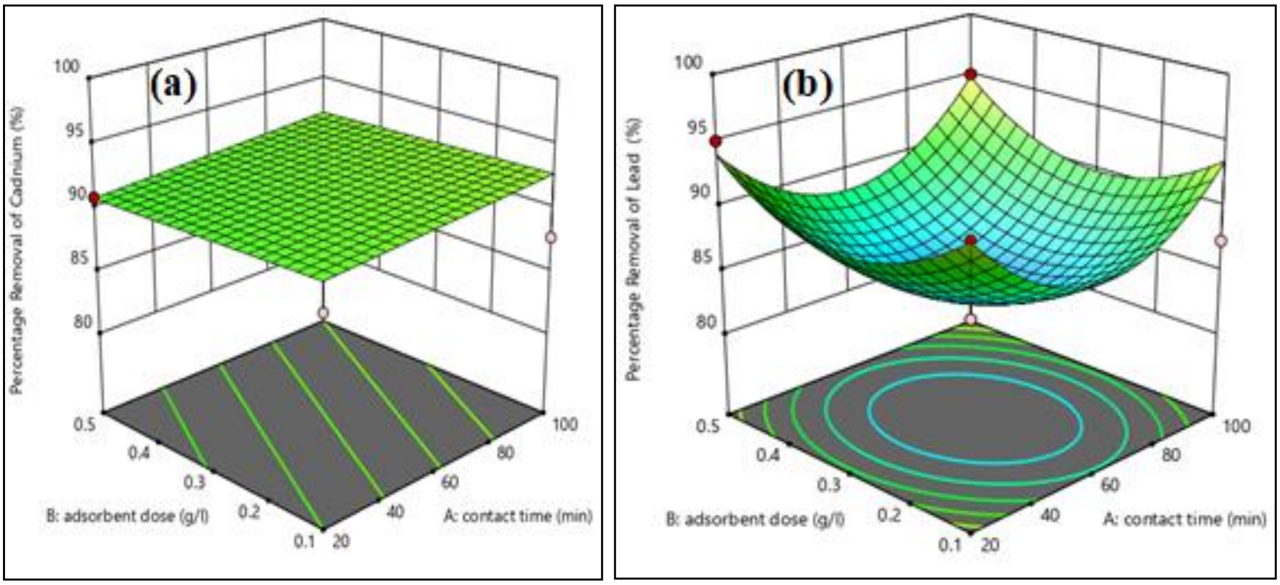

Figure 1: 3D surface plots of the effect of adsorbent dose and contact time for (a) Cd adsorption, (b) Pb adsorption

Table 2: Batch mode Cd adsorption process parameters of BBD used in RSM with the experimental and predicted values for the response

Model	Run	A: contact time (min)	B: adsorbent dose (g)	C: initial ion concentration (mg/l)	D: temperature (°C)	Actual: % Removal	Predicted: % Removal
13	1	60	0.1	10	50	97.58	96.87
1	2	20	0.1	30	50	89.23	91.44
9	3	20	0.3	30	30	95.97	93.01
27	4	60	0.3	30	50	89.81	91.76
4	5	100	0.5	30	50	90.88	92.08
3	6	20	0.5	30	50	90.88	90.80
12	7	100	0.3	30	70	89.69	90.51
24	8	60	0.5	30	70	89.69	89.54
29	9	60	0.3	30	50	89.81	91.76
8	10	60	0.3	50	70	80.57	85.07
5	11	60	0.3	10	30	95.97	98.44
26	12	60	0.3	30	50	89.81	91.76
22	13	60	0.5	30	30	98.41	93.33
6	14	60	0.3	50	30	80.57	88.86
10	15	100	0.3	30	30	99.63	94.29
23	16	60	0.1	30	70	89.69	90.18
15	17	60	0.1	50	50	90.88	87.29
18	18	100	0.3	10	50	95.97	97.19
11	19	20	0.3	30	70	95.97	89.22
7	20	60	0.3	10	70	99.79	94.66
16	21	60	0.5	50	50	80.57	86.65
2	22	100	0.1	30	50	87.72	92.72
14	23	60	0.5	10	50	98.41	96.23
20	24	100	0.3	50	50	95.97	87.61
19	25	20	0.3	50	50	90.88	86.32
28	26	60	0.3	30	50	89.91	91.76
17	27	20	0.3	10	50	89.23	95.91
21	28	60	0.1	30	30	97.58	93.97
25	29	60	0.3	30	50	89.91	91.76

Table 3: Batch mode Pb adsorption process parameters of BBD used in RSM with the experimental and predicted values for the response

Model	Run	A: contact time (min)	B: adsorbent dose (g)	C: initial ion concentration (mg/l)	D: temperature (°C)	Actual: % Removal	Predicted: % Removal
13	1	60	0.1	10	50	96.85	90.50
1	2	20	0.1	30	50	88.77	94.04
9	3	20	0.3	30	30	96.49	93.19
27	4	60	0.3	30	50	82.89	83.80
4	5	100	0.5	30	50	95.02	94.82
3	6	20	0.5	30	50	95.02	93.95
12	7	100	0.3	30	70	86.94	87.10
24	8	60	0.5	30	70	86.94	88.11
29	9	60	0.3	30	50	82.89	83.80
8	10	60	0.3	50	70	81.29	85.00
5	11	60	0.3	10	30	96.49	97.85
26	12	60	0.3	30	50	82.89	83.80
22	13	60	0.5	30	30	98.59	95.23
6	14	60	0.3	50	30	81.29	89.42
10	15	100	0.3	30	30	99.23	99.50
23	16	60	0.1	30	70	86.94	88.37
15	17	60	0.1	50	50	95.02	91.63
18	18	100	0.3	10	50	96.49	98.17
11	19	20	0.3	30	70	96.49	93.09
7	20	60	0.3	10	70	92.83	89.77
16	21	60	0.5	50	50	81.29	84.51
2	22	100	0.1	30	50	87.36	93.50
14	23	60	0.5	10	50	98.59	98.84
20	24	100	0.3	50	50	96.49	88.44
19	25	20	0.3	50	50	95.02	91.41
28	26	60	0.3	30	50	82.89	83.80
17	27	20	0.3	10	50	88.77	94.88
21	28	60	0.1	30	30	96.85	93.75
25	29	60	0.3	30	50	87.43	83.80

Box Behnken Design (BBD) and Analysis of Variance (ANOVA)

BBD-based RSM was used to examine the effects of four effective processing variables on the Cd and Pb adsorption processes, such as contact time, adsorbent dose, initial metal ion concentration, and solution temperature. The BBD-designed experimental matrix for the studied variables, along with the experimental and predicted responses is tabulated in Tables 2 and 3, respectively. The relationship between the independent parameters and metal ion removal (Cd and Pb) was expressed by the following quadratic model equations 2 and 3, respectively.

$$\text{Cd R\%} = +91.76 + 0.6417A - 0.3200B - 4.79C - 1.89D \;\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

$$\text{Pb R\%} = +83.80 + 0.0808A + 0.3050B - 3.30C - 3.13D + 0.3525AB - 1.56AC - 3.07AD - 3.87BC - 0.4350BD + 0.9150CD + 6.07A^2 + 4.21B^2 + 3.36C^2 + 3.35D^2 \;\ldots\ldots\ldots\ldots (3)$$

The regression equation optimizes process parameters, showing Cd and Pb adsorption influenced by the process variables, with positive terms indicating a synergistic effect on the experimental responses, whereas negative terms reveal an antagonistic effect on the process output (Bekele *et al*., 2024; Chima *et al*., 2023).

Analysis of variance (ANOVA) was conducted statistically to confirm the significance of the effects of the investigated variables and the quality of the obtained model equation for the adsorption of Cd and Pb. The statistical results given in Tables 4–6, respectively, show that the P-value and F-value of cadmium were statistically significant, whereas the lead P-value and F-value were statistically non-significant for the quadratic model. The model Fisher's F-value of 4.02 implies the model for Cd removal was significant, whereas 1.74 implies the model for Pb removal was not significant. There were 1.24 % and 15.54 % chance that an F-value of the quadratic model these large could occur due to noise in Cd and Pb percentage removal, respectively. P-values less than 0.05 indicate the model terms that are significant. In the present study, all the process parameters involved in Pb removal were non-significant model terms, whereas only the initial metal ion concentration in cadmium removal shows a significant model term. Model term values greater than 0.10 indicate a non-significant model. The probability of rejecting a null hypothesis depends on the P-value. The lack of fit F-value of 8089.20 of cadmium removal entails the lack of fit was significant, whereas the lack of fit F-value of 9.34 of lead removal entails the lack of fit was non-significant relative to the pure error. There are 0.01 % and 2.27 % chances that a lack of fit F-value these large possibly will occur due to noise for Cd and Pb removal. A non-significant lack of fit indicates well, whereas a significant lack of fit is bad. Moreover, the P-value of lack of fit was less than 0.05 in Cd and Pb removal. This implies that the model fits the experimental data and the independent process parameters have a nonsignificant effect on the experimental responses.

On the other hand, the R^2 of the model (Table 6) were 0.4009 and 0.6353 for Cd and Pb removal respectively, revealing that the obtained model had a low correlation between the response and the investigated variables. The model's nonsignificance is observed in their low adjusted coefficients of determination and prediction, with an adequate precision greater than 4, indicating the potential of BBD for predicting responses of Cd and Pb adsorption. Only the square terms of contact time in lead removal significantly impacted the process with a P-value of 0.0120.

The relationship between the normal percentage probability against externally studetized residuals, residuals versus run number, residual vs. predicted value, and predicted response against experimental data were analyzed in Figs. 2a-d for Cd and 3a-d for Pb in order to evaluate the agreement between the optimization model and the experimental data. The normal probability plot of residuals in Figures 2a and 3a reveals that the errors follow a normal distribution, attesting to the empirical model assumptions (Bekele *et al*., 2024). Figure 2b and 3b show that the majority of the data points are uniformly distributed over the range of -3.54581 to +3.54587 for Cd and -3.93041 to +3.93041 for Pb, failing between the lower and upper bounds of outlier detection. The distribution of predicted values against actual values shown in Figures 2c and 3c shows that the residual values were dispersed randomly with no discernible trend. The residual and predicted values of the Cd and Pb removal responses, as seen in Figures 2d and 3d, have a non-significant relationship, suggesting poor agreement between the actual and predicted values. This implies that the BBD-RSM model applied did not effectively represent the fitted correlation between the four parameters studied in the Cd and Pb adsorption processes.

Table 4: ANOVA of response surface quadratic model for Cd adsorption

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	324.84	4	81.21	4.02	0.0124	Significant
A-contact time	4.94	1	4.94	0.2443	0.6256	Not significant
B-adsorbent dose	1.23	1	1.23	0.0608	0.8074	Not significant
C-initial metal ion concentration	275.62	1	275.62	13.63	0.0011	significant
D-temperature	43.05	1	43.05	2.13	0.1575	Not significant
Residual	485.36	24	20.22			
Lack of Fit	485.35	20	24.27	8089.20	< 0.0001	significant
Pure Error	0.0120	4	0.0030			
Cor Total	810.20	28				

Table 5: ANOVA of response surface quadratic model for Pb adsorption

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	699.15	14	49.94	1.74	0.1554	Not significant
A-contact time	0.0784	1	0.0784	0.0027	0.9590	Not significant
B-adsorbent dose	1.12	1	1.12	0.0389	0.8464	Not significant
C-initial metal ion concentration	130.81	1	130.81	4.56	0.0508	Not significant
D-temperature	117.25	1	117.25	4.09	0.0627	Not significant
AB	0.4970	1	0.4970	0.0173	0.8971	Not significant
AC	9.77	1	9.77	0.3406	0.5688	Not significant
AD	37.76	1	37.76	1.32	0.2704	Not significant
BC	59.83	1	59.83	2.09	0.1706	Not significant
BD	0.7569	1	0.7569	0.0264	0.8733	Not significant
CD	3.35	1	3.35	0.1168	0.7376	Not significant
A^2	238.84	1	238.84	8.33	0.0120	significant
B^2	115.07	1	115.07	4.01	0.0649	Not significant
C^2	73.20	1	73.20	2.55	0.1324	Not significant
D^2	72.93	1	72.93	2.54	0.1331	Not significant
Residual	401.42	14	28.67			
Lack of Fit	384.93	10	38.49	9.34	0.0227	significant
Pure Error	16.49	4	4.12			
Cor Total	1100.57	28				

Table 6: Analysis of variance (ANOVA) results for the response Cd and Pb quadratic models

Parameters	Cd Adsorption	Pb Adsorption
Standard deviation	4.50	5.35
Mean	91.76	90.83
Coefficient of variance (CV, %)	4.90	5.90
Coefficient of determination (R^2)	0.4009	0.6353
Adjusted R^2	0.3011	0.2705
Predicted R^2	0.0724	1.0380
Adequate precision	7.1619	4.0769

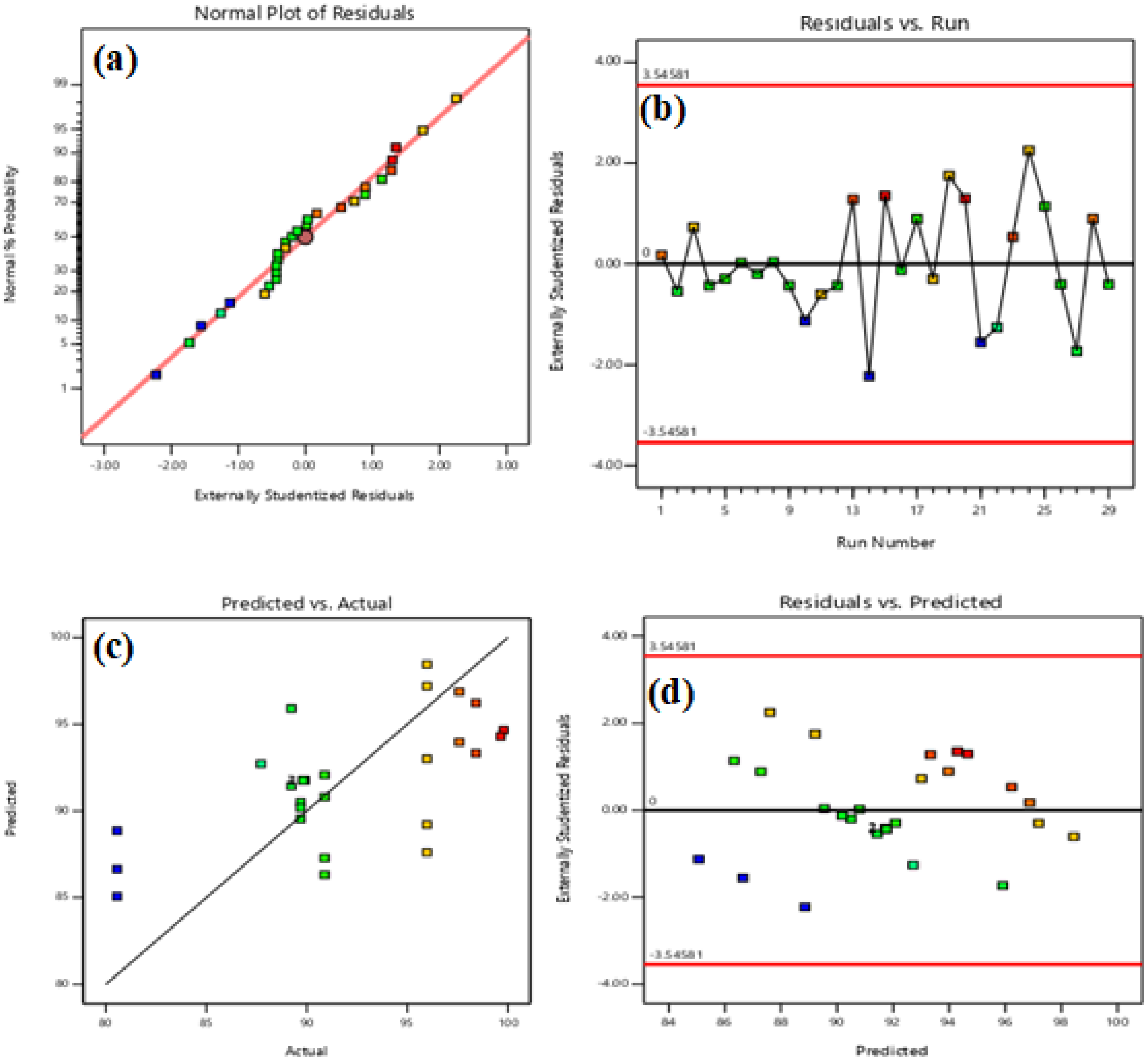

Figure 2: (a) Normal plot of residuals, (b) residuals vs run number, (c) predicted vs actual value (d) residuals vs predicted value of the fitted models for Cd adsorption

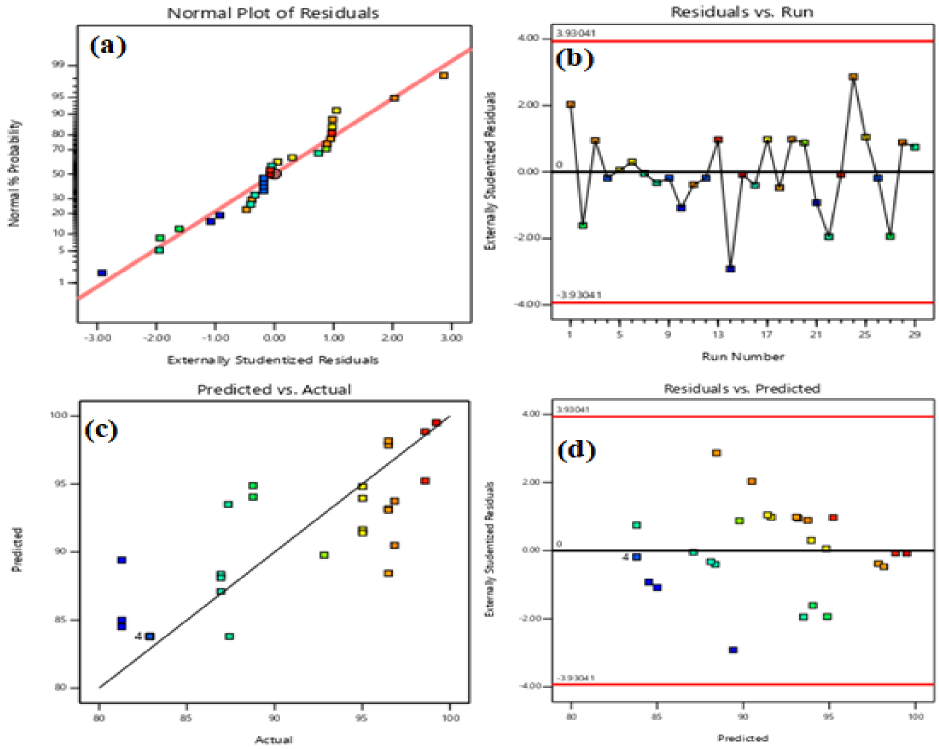

Figure 3: (a) Normal plot of residuals, (b) residuals vs run number, (c) predicted vs actual value (d) residuals vs predicted value of the fitted models for Pb adsorption

3.3 Numerical Optimization and BBD Model Validation

Numerical optimization of Cd and Pb removal was analyzed using BBD-RSM methods; the highest removal efficiency of Cd and Pb was prioritized as a desirable aim while optimizing the operational parameters. The optimal variables of contact time = 42.16 min, adsorbent dose = 0.25 g, initial metal ion concentration = 21.52 mg/l, and temperature = 37.00 °C, and optimization results of Cd and Pb removal are shown in Table 7 and Figure 4, respectively. The maximum removal efficiency of cadmium and lead achieved was 94.81% and 89.23 %, respectively. The calculated function accurately explains the experimental model and the intended conditions where the desirability value is 1.00.

Table 7: Optimal conditions of the selected factors for Cd and Pb adsorption onto *Pterocarpus santalinoides*

Optimized process parameters				Predicted removal efficiency (%)		
Contact time (min)	Adsorbent dosage (g)	Initial ion concentration (mg/l)	Temperature (°C)	Cd	Pb	Desirability
42.157	0.252	21.521	37.005	94.81	89.23	1.000

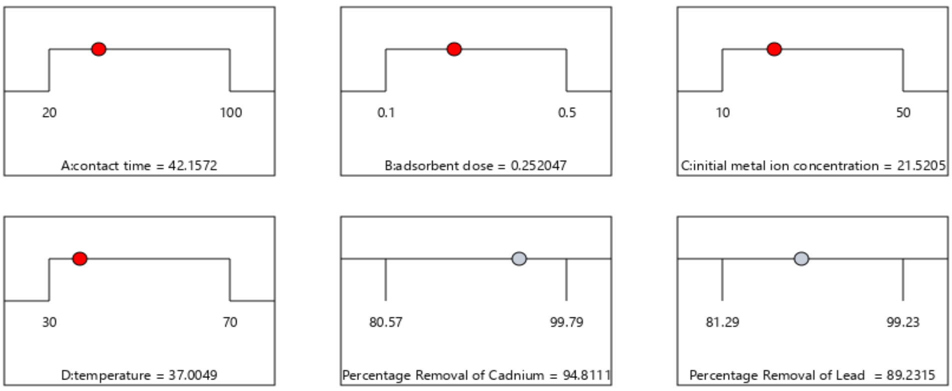

Figure 4: Numerical optimization model parameters for Cd and Pb adsorption by natural *Pterocarpus santalinoides* fruit

CONCLUSION

The suitability of natural *Pterocarpus santalinoides* fruit for the adsorption-based approach in the removal of Cd and Pb ions from aqueous solutions was investigated. Using the RSM-BBD method, the impact of various process variables, such as contact time, adsorbent dose, initial metal ion concentration, and temperature, on the Cd and Pb removal with natural *Pterocarpus santalinoides* was examined. Based on the model results of the analysis of variances (ANOVA), it was observed that the linear model, which has a low correlation coefficient, explains the removal efficiency maximum removal effectiveness of 91.81 % and 89.23 % were achieved for Cd and Pb, respectively, at a contact time of 42.16 min, an adsorbent dose of 0.25 g, an initial metal ion concentration of 21.52 mg/l and temperature of 37.00 ^{0}C. According to the findings in this study, natural *Pterocarpus santalinoides* fruit can serve as a potential low-cost adsorbent for the removal of Cd and Pb from aqueous solutions. The BBD-RSM adopted in the optimization of the process revealed that the statistical model applied represents a poor relationship between the four parameters examined in the Cd and Pb removal process.

Data Availability

The authors declare that the data supporting this finding are available from the corresponding author on reasonable request.

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Conflict of Interest: The authors declare no conflict of interest.

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