



Research
Green Chemical Engineering—Article

Optimization, Kinetics, and Equilibrium Studies on the Removal of Lead(II) from an Aqueous Solution Using Banana Pseudostem as an Adsorbent

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ARTICLE INFO

Article history:

Received 2 January 2017

Revised 9 May 2017

Accepted 13 May 2017

Available online 13 June 2017

Keywords:

Banana pseudostem

Lead

Isotherm

Adsorption

Response surface methodology

ABSTRACT

Natural adsorbents such as banana pseudostem can play a vital role in the removal of heavy metal elements from wastewater. Major water resources and chemical industries have been encountering difficulties in removing heavy metal elements using available conventional methods. This work demonstrates the potential to treat various effluents utilizing natural materials. A characterization of banana pseudostem powder was performed using environmental scanning electron microscopy (ESEM) and Fourier-transform infrared (FTIR) spectroscopy before and after the adsorption of lead(II). Experiments were carried out using a batch process for the removal of lead(II) from an aqueous solution. The effects of the adsorption kinetics were studied by altering various parameters such as initial pH, adsorbent dosage, initial lead ion concentration, and contact time. The results show that the point of zero charge (PZC) for the banana pseudostem powder was achieved at a pH of 5.5. The experimental data were analyzed using isotherm and kinetic models. The adsorption of lead(II) onto banana pseudostem powder was fitted using the Langmuir adsorption isotherm. The adsorption capacity was found to be 34.21 mg·g⁻¹, and the pseudo second-order kinetic model showed the best fit. The optimum conditions were found using response surface methodology. The maximum removal was found to be 89%.

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1. Introduction

The effluents from chemical industries are released into the environment, causing severe water shortages and leading to many diseases due to the resulting environmental problems. Many conventional methods are available to reduce or remove heavy metals from effluents to some extent [1,2]. However, most undeveloped and developing countries are facing serious problems in the supply of good-quality drinking water to the public [3]. Drinking water quality is low due to the presence of heavy metal ions, which may cause enormous problems for human health and for ecological systems. Heavy metal pollution is greatly affecting natural fauna and flora [4–7]. Most of the pollution emitted by major lead-producing chemical process industries is composed of electroplating chemi-

cals, pharmaceutical chemicals, and electrochemicals. Lead is highly toxic in nature, and has an extremely negative effect on biodiversity. The lead-removal process is currently a challenging task for environmental scientists and engineers in terms of cost, effluent disposal, and safety concerns [1,8]. Various conventional water treatment methods such electroplating, precipitation, evaporation, membrane separation, ion exchange, coagulation, floatation, reverse osmosis, solvent extraction, membrane filtration, and adsorption, as well as different biological processes, are utilized for the recovery of metals from effluents [9,10]. Most of these conventional methods are highly laborious and energy consuming; in addition, their use is limited to batch processes. Hence, research focus should be aimed at continuous processes and at scaling up and meeting the requirements for secondary or tertiary sludge treatment.

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<http://dx.doi.org/10.1016/J.ENG.2017.03.024>

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To overcome the abovementioned difficulties, the development of an efficient, cost-effective, environmentally friendly, and continuous process for lead removal is extremely necessary. Based on a literature review, it was observed that agricultural residues have been employed as adsorbents in removing heavy metals. In general, the agricultural residues used for the removal of heavy metals were wheat, rice, oat, barley, and corn husk. At present, there is an increase in environmental awareness and in mandatory legal constraints for the discharge of effluent. These factors strongly promote the development of cost-effective alternative technologies [11,12]. Among the available conventional treatment methods, adsorption is a potential process for the removal of heavy metals from various industrial wastewaters, due to its utility for a wide range of pollutants, its overall capacity, and its selectivity. Recently, many researchers have shown an interest in the development of heavy metal removal through the utilization of a wide range of adsorbents that are good alternatives to commercial adsorbents [13–17].

The aim of this research work was to identify the optimal relation between variables including initial pH, adsorbent dosage, initial lead ion concentration, and contact time, when using banana pseudostem powder to adsorb lead(II). Using software, we conducted experiments to identify the optimum conditions for the removal of lead nitrate ($\text{Pb}(\text{NO}_3)_2$). Response surface methodology (RSM) and central composite design (CCD) were found to be the best statistical tools for optimization, as they predicted the best output with the lowest number of experiments.

2. Materials and methods

2.1. Preparation of adsorbent from banana pseudostem

Samples of banana pseudostem were collected from local agriculture fields. The collected banana pseudostem samples were cut into pieces with a size of 5 mm. Next, the pieces were thoroughly cleaned using normal water, and then thoroughly cleaned again using demineralized water, in order to remove mud and other foreign particles. Later, the material was air dried to remove surface free water and subjected to a hot air oven for 24 h at 105 °C. Finally, the material was sieved using a mesh size of 500 μm . It was collected and stored in an air-tight container for further experiments.

2.2. Stock solution preparation

Solutions with lead ion concentrations ranging from 10 ppm to 50 ppm were prepared using analytical-grade $\text{Pb}(\text{NO}_3)_2$. HCl and NaOH buffer solutions of 0.1 N ($1 \text{ N} = (1 \text{ mol}\cdot\text{L}^{-1}) \div 1$) were prepared to adjust the pH of the solutions.

2.3. Batch adsorption process

Experiments were conducted using batch adsorption processes in order to identify the optimum time required to achieve maximum adsorption for concentrations ranging from 10 ppm to 50 ppm at a fixed pH of 6. Banana pseudostem powder ($1 \text{ g}\cdot\text{L}^{-1}$) was used as the

adsorbent material, and the contact time was varied from 20 min to 120 min with intervals of 20 min. At each 20 min interval, samples were collected, filtered, and centrifuged at $2000 \text{ r}\cdot\text{min}^{-1}$ for 5 min. Lead ion concentrations in the obtained supernatant were analyzed using an atomic absorption spectrophotometer (AAS) (Varian, SpectrAA 200). The lead-uptake values were calculated in terms of percentages using Eq. (1) [18]. All experiments were conducted in duplicate.

$$\text{Removal}(\%) = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

where C_e denotes the lead ion concentration in solution at equilibrium and C_0 denotes the initial concentration of lead ion. The adsorption capacity (q_e) was calculated using Eq. (2).

$$q_e = (C_0 - C_e) \frac{V}{W} \quad (2)$$

where V is the volume of the aqueous solution (in L), and W is the mass of the adsorbent (in g).

2.4. Design of experiments for optimization

The experimental design examined three factors in the removal of $\text{Pb}(\text{NO}_3)_2$: initial pH, adsorbent dosage, initial lead ion concentration, and contact time. The coded and un-coded levels were designed using CCD, as shown in Table 1. Table 2 presents the sets of experiments that were obtained using CCD.

2.5. Analysis and optimization

The results were analyzed using RSM, and the combined effect of the three factors (initial pH, adsorbent dosage, and initial lead ion concentration) on the removal of lead nitrate was examined. The F -test was studied using analysis of variance (ANOVA). Surface plots were generated using the obtained results, run by RSM.

2.6. Characterizations

The Fourier-transform infrared (FTIR) spectra of the samples were recorded on a spectrophotometer (PerkinElmer, PE 1600) in a KBr medium (with a sample-to-KBr ratio of 1:5) across a wave number range of $400\text{--}4600 \text{ cm}^{-1}$. The surface morphology of the banana pseudostems before and after treatment with heavy metal ions was investigated using environmental scanning electron microscopy (ESEM) (FEI, Quanta 200) on a gold substrate.

3. Results and discussion

3.1. Point of zero charge

The point of zero charge (PZC) plays an important role in the characterization of the adsorbate, and explains the affinity of the adsorbate to an adsorbent surface. The PZC was determined as described in Ref. [17] for banana pseudostem powder over a pH range

Table 1
Experimental range and levels of initial pH, initial lead ion concentration, and adsorbent dosage in CCD.

Variable	Parameter	Level				
		$-\alpha$	-1	0	+1	$+\alpha$
x_1	Initial pH	1.29552	3	5.5	8	9.70448
x_2	Initial lead ion concentration ($\text{mg}\cdot\text{L}^{-1}$)	12.9552	30	55.0	80	97.0448
x_3	Adsorbent dosage ($\text{g}\cdot\text{L}^{-1}$)	0.318207	1	2.0	3	3.68179

that varied from 2 to 10. The PZC for banana pseudostem powder was observed at pH 5.5. This result reveals that at a pH below 5.5, the surface of the banana pseudostem powder shows positive charges, while at a pH above 5.5, the surface shows negative charges.

3.2. Scanning electron microscopy analysis

An ESEM analysis was carried out to examine the surface characteristics of the banana pseudostem powder at a particle size of 500 μm . As shown in Fig. 1, the nature and surface of the material were found to be highly suitable for adsorption studies both before and after adsorption. The interfibrillar surface properties of banana pseudostem powder strongly increase the total absorption surface area for heavy metals.

3.3. Fourier-transform infrared analysis

Fig. 2 shows the FTIR analysis that was carried out on the banana pseudostem powder, including the absorptions bands before and after, at ranges from 3750 cm^{-1} to 2275 cm^{-1} , that were assigned

to stretching vibrations and to other polymeric associations of the hydroxyl groups. The symmetric stretching at 2900 cm^{-1} is associated with the $-\text{CH}_2$ groups that are present in polysaccharides. Angular deformations of the C–H linkages of aromatic groups were observed at 878 cm^{-1} and 775 cm^{-1} . The FTIR interpretation showed that the presence of peaks in the range of 1650–1337 cm^{-1} and 1243–1028 cm^{-1} were due to C=C, C=O, C–C, –OH, $-\text{CH}_2$, –CH, and C–O–C vibrations. The banana pseudostem powder FTIR results reveal the presence of various functional groups indicating hemicelluloses, cellulose, and lignin, suggesting an ethereal and aromatic character. The results showed that the banana pseudostem powder contained functional groups that created a suitable environment for interactions between the biosorbent and metallic ions [19].

3.4. Effect of initial pH

In the adsorption process, pH is a relevant parameter that provides suitable conditions for maximum adsorption at the adsorbent surface. Fig. 3 shows increased adsorption of lead nitrate before pH 6; the adsorption then decreases with an increase in pH. Adsorption

Table 2

Set of 20 experiments with combinations of three parameters.

No.	Initial pH	Initial lead ion concentration ($\text{mg}\cdot\text{L}^{-1}$)	Adsorbent dosage ($\text{g}\cdot\text{L}^{-1}$)	Lead nitrate removal efficiency (%)	
				Experimental	Predicted
1	3.0	30.00	1.00	63	66
2	8.0	30.00	1.00	71	67
3	3.0	80.00	1.00	80	75
4	8.0	80.00	1.00	58	63
5	3.0	30.00	3.00	79	72
6	8.0	30.00	3.00	72	75
7	3.0	80.00	3.00	72	74
8	8.0	80.00	3.00	70	65
9	1.3	55.00	2.00	71	73
10	9.7	55.00	2.00	67	65
11	5.5	12.96	2.00	72	72
12	5.5	97.04	2.00	71	71
13	5.5	55.00	0.32	71	69
14	5.5	55.00	3.68	74	76
15	5.5	55.00	2.00	90	88
16	5.5	55.00	2.00	89	88
17	5.5	55.00	2.00	89	88
18	5.5	55.00	2.00	87	88
19	5.5	55.00	2.00	89	88
20	5.5	55.00	2.00	89	88

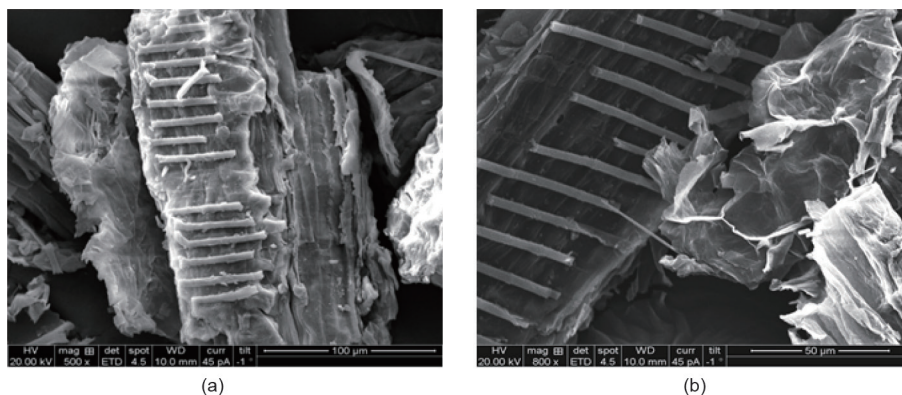


Fig. 1. ESEM photographs of banana pseudostem powder (a) before and (b) after adsorption of lead(II).

was carried out under fixed conditions with an adsorbent dosage of $1 \text{ g}\cdot\text{L}^{-1}$, a contact time of 60 min, and a lead ion concentration of 10 ppm, using a batch adsorption process.

3.5. Effect of adsorbent dosage

Adsorbent dosage was observed to play an important function in the adsorption of lead nitrate. With a higher dosage, more active sites are present, increasing the interactions with metal ions, and therefore increasing the adsorption of metal ions. In the present work, the selected adsorbent dosages ranged from $0.2 \text{ g}\cdot\text{L}^{-1}$ to $1 \text{ g}\cdot\text{L}^{-1}$. The fixed parameters were an initial pH of 6, a contact time of 60 min, and an initial lead ion concentration of 10 ppm. As shown in Fig. 4, the adsorbent dosage of $1 \text{ g}\cdot\text{L}^{-1}$ resulted in the maximum removal of lead nitrate.

3.6. Effect of initial lead ion concentration and contact time

The combined effect of initial lead ion concentration and contact time for the maximum adsorption of lead nitrate was studied. The percentage removal of lead nitrate was identified by selecting an initial lead ion concentration from 10 ppm to 50 ppm at intervals of 10 ppm, and varying the contact time from 20 min to 120 min. The adsorbent dosage and pH were fixed at $1 \text{ g}\cdot\text{L}^{-1}$ and 6, respectively. As Fig. 5 shows, the percentage adsorption of lead nitrate decreased with an increase in ion concentration, and the percentage adsorption of lead nitrate increased with an increase in contact time.

3.7. Adsorption isotherms

The adsorption capacity was studied using different adsorption isotherm models—namely, the Langmuir and Freundlich models. The experimental adsorption data were correlated with these two models. Most of the literature studies show that the Langmuir adsorption isotherm is extensively used for adsorption that occurs at specific sites of a homogenous adsorbent. Adsorption cannot take place once the adsorbate molecule occupies a site, due to equilibrium being reached. Eq. (3), the Langmuir linear equation, describes the saturation of the monolayer curve:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0} \quad (3)$$

where C_e (in $\text{mg}\cdot\text{L}^{-1}$) denotes the concentration equilibrium of the lead ions present in the solution at equilibrium; q_e (in $\text{mg}\cdot\text{g}^{-1}$) is the adsorption capacity; Q_0 (in $\text{mg}\cdot\text{g}^{-1}$) is the maximum adsorption capacity; and b is the Langmuir adsorption constant (in $\text{L}\cdot\text{mg}^{-1}$). The intercept plotted can be used to calculate the plots of C_e/q_e against C_e that give a straight line. From this, it is possible to identify Q_0 and b . The favorability or unfavorability of the adsorption system can be predicted by the equilibrium parameter R_L , which is a dimensionless constant that is an essential characteristic of the Langmuir model. The equilibrium parameter is determined using Eq. (4) [18–21]:

$$R_L = \frac{1}{1 + bC_0} \quad (4)$$

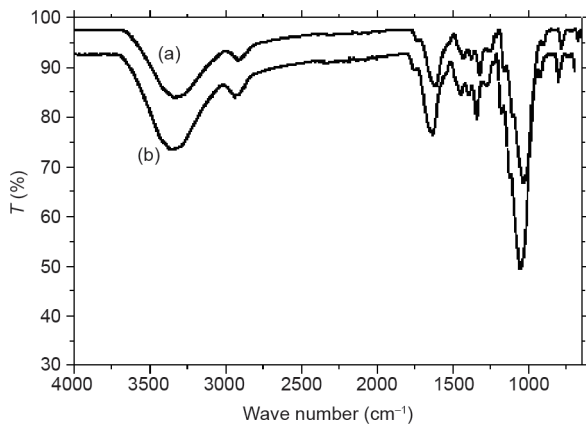


Fig. 2. FTIR spectra of banana pseudostem powder (a) before and (b) after adsorption of lead(II).

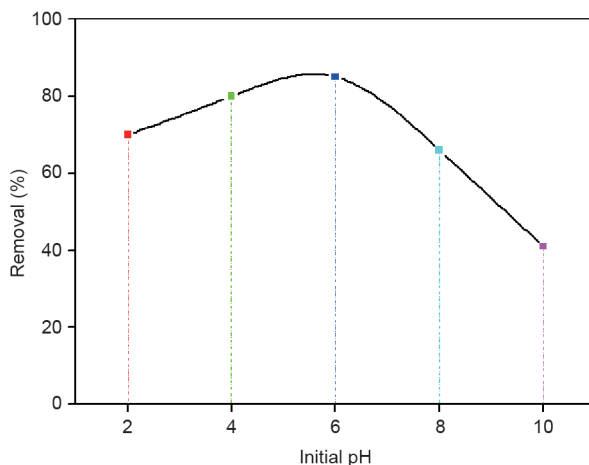


Fig. 3. Effect of initial pH on adsorption.

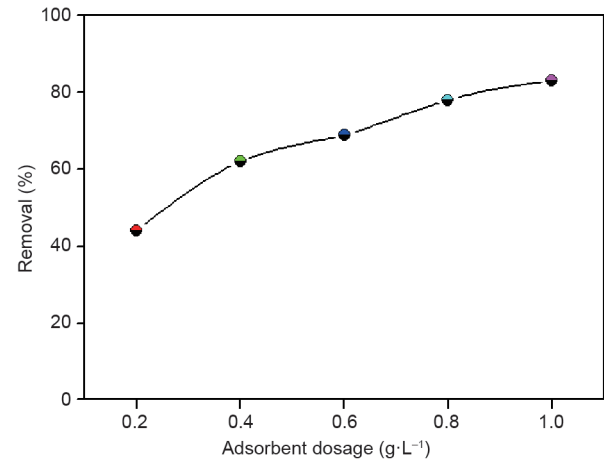


Fig. 4. Effect of adsorbent dosage on adsorption.

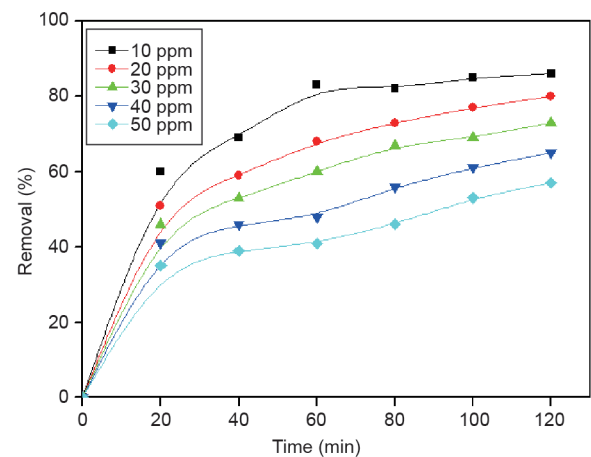


Fig. 5. Effect of initial lead ion concentration and contact time on adsorption.

where C_e denotes the initial concentration. This parameter suggests the type of isotherm, which may be irreversible ($R = 0$), favorable ($0 < R < 1$), or unfavorable ($R > 1$). The Langmuir adsorption isotherm was obtained by plotting a graph of C_e/q_e versus C_e . The values of Q_o and b for the adsorption of lead ions, as determined from Fig. 6, were $34.21 \text{ mg}\cdot\text{g}^{-1}$ and 0.22 , respectively. The regression coefficient was observed to be 0.9997 for the Langmuir adsorption isotherm.

The Freundlich adsorption isotherm model is a commonly used model that describes a heterogeneous adsorption process. On a heterogeneous surface, the adsorption process takes place through a multilayer adsorption mechanism. The Freundlich adsorption isotherm is expressed by Eq. (5).

$$q_e = K_F C_e^{1/n} \tag{5}$$

Eq. (6) gives the rearranged, linear equation [18–21]:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \tag{6}$$

where q_e is the adsorption capacity (in $\text{mg}\cdot\text{g}^{-1}$) and C_e is the lead ion concentration in solution (in $\text{mg}\cdot\text{L}^{-1}$) at equilibrium. For the Freundlich adsorption, the physical constants are K_F (the Freundlich adsorption constant) and n (the adsorption intensity constant). The slope is n and the intercept is K_F ; as shown in Fig. 7, these values were found to be 2.25 and 8.024 , respectively, with a Freundlich adsorption isotherm regression of 0.97 . The obtained data show that the Langmuir adsorption isotherm suits the situation better than the Freundlich adsorption isotherm.

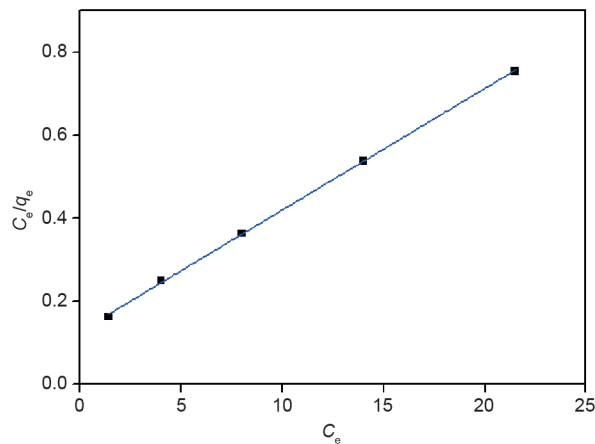


Fig. 6. Langmuir adsorption isotherm of lead ion in aqueous solution.

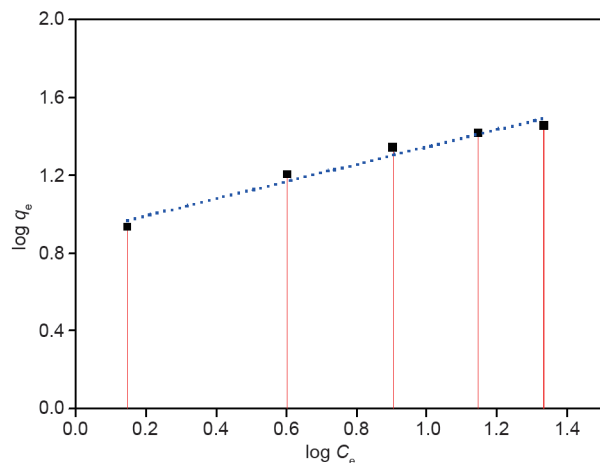


Fig. 7. Freundlich adsorption isotherm of lead ion in aqueous solution.

3.8. Adsorption kinetics

Here, the adsorption kinetics are explained using two models: pseudo first- and pseudo second-order kinetic models. Banana pseudostem powder was used to remove lead nitrate while varying the parameters of initial pH, contact time, adsorbent dosage, and initial lead ion concentration. The experimental data were correlated with the pseudo first- and second-order kinetic models. Eq. (7) describes the pseudo first-order kinetic model [18–21].

$$\log(q_e - q_t) = \log q_e - \frac{K_1 t}{2.303} \tag{7}$$

where K_1 denotes the reaction rate constant (min^{-1}) for the pseudo first-order kinetic model and q_t is the amount of lead ions adsorbed at time t . We then plotted the graph of $\log(q_e - q_t)$ versus t , as shown in Fig. 8.

The pseudo second-order kinetic model was selected based on the equilibrium of the adsorption capacity, as shown in Eq. (8) [18–21].

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \tag{8}$$

where K_2 denotes the reaction rate constant ($(\text{g}\cdot\text{mg}^{-1})\cdot\text{min}^{-1}$) for the pseudo second-order kinetic model. A graph was plotted of t/q_t ($\text{min}\cdot(\text{g}\cdot\text{mg}^{-1})$) versus t (min), as shown in Fig. 9.

Table 3 reports the results as a comparison of the pseudo first-order and pseudo second-order kinetic models based on linear regression values. Based on the regression values, the pseudo second-order

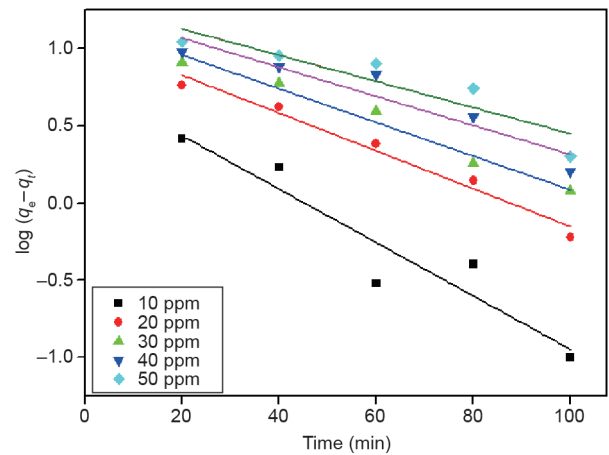


Fig. 8. Pseudo first-order kinetic model for lead ion adsorption.

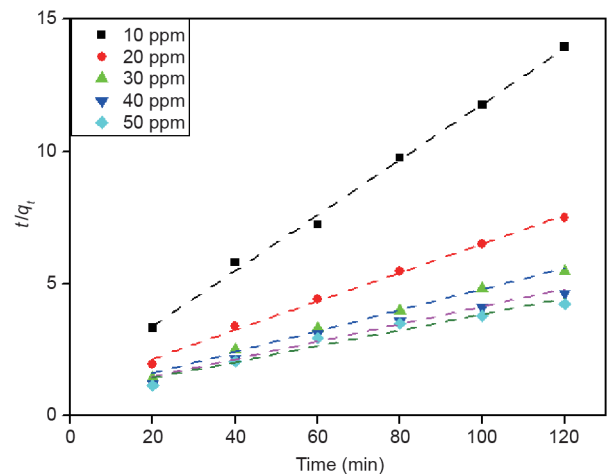


Fig. 9. Pseudo second-order kinetic model for lead ion adsorption.

kinetics were found to suit the obtained kinetics better than the pseudo first-order kinetics.

3.9. Process optimization by response surface methodology

The optimization of the process conditions affecting the percentage of lead nitrate removal was carried out for the maximum extraction of lead nitrate from an aqueous solution using the CCD method. Software runs were performed for 20 experiments with three different combinations of initial pH, initial lead ion concentration, and adsorbent dosage, as shown in Table 2.

A detailed analysis was done on the effects of the interactions between initial pH, initial lead ion concentration, and adsorbent dosage on the percentage of lead nitrate removal from an aqueous solution. The Design-Expert (Stat-Ease, Inc., Minneapolis, USA) software was used to determine the optimum conditions based on the results obtained from the regression and graphical analysis. The multiple regression data were analyzed using Design-Expert software. The parameters were estimated using response variables under certain

considerations. The test results showed a second-order polynomial equation for the removal of lead nitrate:

$$Y_1 = 88.81 - 2.18x_1 - 0.49x_2 + 1.91x_3 - 3.12x_1x_2 + 0.63x_1x_3 - 1.62x_2x_3 - 6.86x_1^2 - 5.98x_2^2 - 5.63x_3^2 \tag{9}$$

where Y_1 is the response variable in terms of the removal of lead nitrate; x_1 (initial pH), x_2 (initial lead ion concentration), and x_3 (adsorbent dosage) are coded values for the independent variables. The results were fitted with a second-order kinetic model. The goodness of fit for Eq. (9) was correlated with the coefficient of determination (R^2) in order to measure the total variations; the coefficient of correlation (R) explains the correlation between the experimental and predicted values from the model. The experimental results show that $R^2 = 0.8992$ for the removal of lead nitrate. The results explain 89.92% of the total variability in the responses. The R for lead nitrate removal is 0.9482, where the closer the value of the R is to unity, the better. Table 4 shows the ANOVA results. A P value of less than 0.05 indicates that the model was fit.

Fig. 10 shows the combined effect of a three-dimensional response surface (initial pH, initial lead ion concentration, and adsorbent dosage) on the removal of lead nitrate. The optimum result of an adsorbent dosage of 2.5 g·L⁻¹ was obtained by a combined study of initial pH and initial lead ion concentration. At lower pH and lower lead ion concentration, the percentage adsorption increases. The

Table 3 Parameters of the kinetic models.

Model	Initial lead ion concentrations (ppm)				
	10	20	30	40	50
Pseudo first-order					
K_1 (min ⁻¹)	0.039	0.028	0.025	0.022	0.017
q_e (mg·g ⁻¹)	6.05	11.81	15.03	18.00	19.78
R^2	0.8981	0.9751	0.9772	0.8931	0.8336
Pseudo second-order					
K_2 ((g·mg ⁻¹)·min ⁻¹)	0.0090	0.0030	0.0020	0.0013	0.0011
q_e (mg·g ⁻¹)	9.5	18.0	25.0	30.0	33.0
R^2	0.9969	0.9962	0.9938	0.9729	0.9571

Table 4 Analysis-of-variance results.

Source	Sum of squares	Degree of freedom	Mean square	F	P
Model	1599.92	9	177.77	9.92	0.0007
Residual	179.28	10	17.93		
Total	1779.20	19			

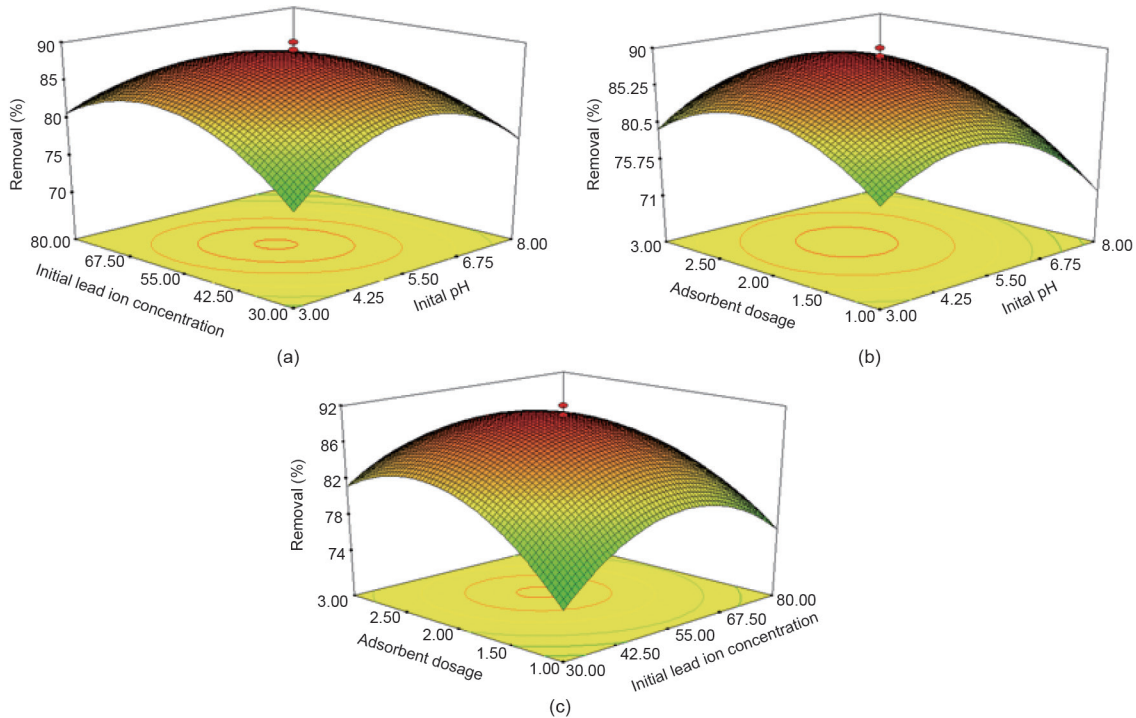


Fig. 10. Surface plots for the effect of different parameters on lead nitrate removal efficiency. (a) Initial lead ion concentration and initial pH; (b) adsorbent dosage and initial pH; (c) adsorbent dosage and initial lead ion concentration.

optimum result of an initial lead ion concentration of 54 mg·L⁻¹ was obtained by a combined study of initial pH and adsorbent dosage. Increasing the pH results in a decrease in the percentage adsorption, whereas lower pH and high dosage result in better percentage adsorption. A combined study of the effect of adsorbent dosage and initial lead ion concentration on lead removal was performed at a constant pH of 5.2. An increase in adsorbent dosage and initial lead ion concentration results in a decrease in percentage adsorption. The experimental results demonstrate that a lower lead ion concentration and a higher dosage lead to the maximum removal of lead nitrate. The maximum removal of lead nitrate was found to be 89%.

4. Conclusions

The removal of lead(II) using banana pseudostem powder was studied, and the optimum parameters affecting the efficiency of lead(II) removal were obtained. The surface morphology was examined using ESEM, and the molecular functional groups were examined using FTIR spectroscopy. The combined effect of three parameters—initial pH, initial lead ion concentration, and adsorbent dosage—was studied. The results are explained by three-dimensional responses. The results showed that maximum lead(II) removal occurred at pH 5.2, with an initial lead concentration of 54 mg·L⁻¹ and an adsorbent dosage of 2.5 g·L⁻¹. The maximum removal of lead(II) was 89%. This methodology successfully explains the optimization of the initial pH, initial lead ion concentration, and adsorbent dosage. Response surface graphs assisted in understanding the interaction of these three parameters. The Langmuir adsorption isotherm was found to be the best-fitting model for the removal of lead(II) by banana pseudostem powder. The kinetic model was better explained using the pseudo second-order model. This experimental work establishes banana pseudostem powder as an inexpensive adsorbent that can be considered as an alternative for the removal of heavy metals during wastewater treatment.

Acknowledgements

This research was supported by the Department of Chemical Engineering and Biotechnology at the Siddaganga Institute of Technology, Tumkur, Karnataka, India.

Compliance with ethics guidelines

Shridhar S. Bagali, Bychapur S. Gowrishankar, and Aashis S. Roy declare that they have no conflict of interest or financial conflicts to disclose.

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