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Biosorptive removal of lead and cadmium ions from aqueous solution: The use of carrot residues as low cost non-conventional adsorbent

[Sıvı çözeltilerden kurşun ve kadmiyumun biyoemilim ile uzaklaştırılması: Düşük maliyetli, modern adsorban olan havuç artıklarının kullanımı]*

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ABSTRACT

Objectives: Carrot residue as low cost, non-conventional adsorbent was used for biosorptive removal of lead and cadmium from the aqueous solution.

Methods: A batch adsorption method was experimented for biosorptive removal of lead and cadmium ions from the aqueous solution.

Findings: Experimental data fitted well to both Langmuir and Freundlich isotherm models. The calculated q_{max} for lead and cadmium was 0.522 mg/g and 0.421 mg/g respectively. Thermodynamic parameters like ΔG, ΔH and ΔS showed spontaneity endothermic nature of adsorption for cadmium ion and exothermic nature for the lead ions. Nearly 93% removal of lead and 92% removal of cadmium were observed in about 35 minutes at pH ≅ 3 and 5 respectively, under the batch test conditions.

Conclusion: Carrot residue was investigated as low cost non-conventional adsorbent for the sorption of cadmium and lead ions from the contaminated water under various experimental conditions like pH, dosage amount and contact time.

Key Words: Carrot residue, adsorbent, lead, cadmium.

Conflict of Interest: There is no conflict of interest among the authors who contributed to the present study.

ÖZET

Amaç: Sıvı çözeltilerden kurşun ve kadmiyumu biyoemilim ile uzaklaştırmak için düşük maliyetli ve modern adsorban olan havuç artıkları kullanılmıştır.

Yöntem: Kurşun ve kadmiyumun sıvı çözeltilerden biyoemilim ile uzaklaştırılması için toplu adsorbsiyon metodu denenmiştir.

Bulgular: Deneysel bulgular Langmuir ve Freundlich'in izoterm modeline uymaktadır. Kurşun ve kadmiyum için hesaplanan q_{max} sırasıyla 0.522 mg/g and 0.421 mg/g olarak bulunmuştur. ΔG, ΔH and ΔS gibi termodinamik parametreler adsorpsiyonun kadmiyum iyonları için endotemik, kurşun iyonları için ekzotermik olduğunu ortaya koymaktadır. Kurşun iyonlarının %93'ünün, kadmiyum iyonlarının ise %92'sinin 35 dakikada sırasıyla pH @ 3 ve 5'de uzaklaştığı gözlenmiştir.

Sonuç: Düşük maliyetli ve modern adsorban olan havuç artıklarının, kirlenmiş sularda bulunan kadmiyum ve kurşun iyonlarını pH, doz miktarı ve temas süresi gibi değişik deneysel koşullar altında tutma özelliği incelenmiştir.

Anahtar Kelimeler: Havuç artıkları, adsorban, kurşun, kadmiyum

Çıkar Çatışması: Yazarların çıkar çatışması bulunmamaktadır.

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Introduction

Contamination of aquatic environment with heavy and toxic metals is a complex problem and their removal requires much attention [1-3]. Their concentration gets accentuated through bioaccumulation via food chain in living tissues, causing various diseases and physiological disorders [2, 3]. Hence the safe and effective disposal of contaminated water containing heavy metals like lead and cadmium is always remained a challenge to the industrialists and environmentalists [4].

Lead get introduced into natural water from a variety of sources such as lead acid batteries, lead smelting, tetraethyl lead, mining, electroplating, ammunition and the ceramic glass industries. The tolerance limit for discharge of lead into drinking water is 0.05 mgL^{-1} and in land surface waters is 0.1 mgL^{-1}. Excess of lead ions in drinking water causes diseases like anemia, encephalopathy and hepatitis. Lead ions have an affinity for ligands containing thiol and phosphate groups. Like calcium, excess lead ions inhibit the biosynthesis of heme, causing damage both to the kidney and liver. However, lead can remain immobilized for years in human body and is very difficult to detect its metabolic disorder [2, 3].

Cadmium is found in many domestic products like tobacco, phosphate fertilizers, polyvinyl chloride, rechargeable cells, petrol, commercial oils and is believed to cause pulmonary emphysema and bone diseases like osteomalacia and osteoporosis [5].

These toxic heavy metal ions should be removed at the source to manage water pollution and subsequent metal accumulation in the food chain. Conventional and expensive methods for their removal are chemical precipitation, chemical oxidation, chemical reduction, ion exchange, filtration, electrochemical treatment and evaporation. All these procedures have significant disadvantages like the incomplete removal, high-energy requirements and production of toxic sludge or waste products that also require further disposal.

Alternatively, low cost methods for heavy metal removal have been developed in the last decade. A great effort has also been contributed to develop new adsorbents like activated carbons [6, 7], hydroxyapatite [8–11], silica [12], zeolites [13, 14], polymers [15], clays [16], mango and banana peels [17, 18].

Carrots are primarily used for edible purposes. From the last few decades, carrot juice is globally used as public favorite natural drink. During juice production, a large number of carrot residues are produced as waste product. This residue or carrot pomace has no further economic or commercial value and advantage of using this as an adsorbent includes cheap, quick and easy heavy metal separation from an aqueous media. The adsorptive properties of this residue can be attributed to the presence of carboxylic, phenolic and other functional groups, which exist in either the cellulosic matrix or in the materials associated with cellulose such as hemicellulose, lignin and also in the peptide.

Literature study indicates that Nasernejad et al. used carrot residues to remove chromium, copper and zinc [19]. Guzel et al. used this residue to remove manganese, cobalt, nickel and copper [20]. Bhatti et al. used to remove both trivalent and hexavalent chromium ions from aqueous media [21]. To the best of authors' knowledge, no study has been reported so far for the adsorption of lead and cadmium ions on carrot residue.

In the present manuscript, systematic biosorption of lead and cadmium from aqueous solution by the batch adsorption technique has been developed. Adsorption characteristic has been evaluated as a function of pH, time of contact, adsorbent dose and temperature. The equilibrium data were examined using Langmuir and Freundlich isotherm models. The adsorption mechanism was also investigated in terms of thermodynamics.

Materials and Methods

Preparation of adsorbent

Carrot residue, the fibrous by-product of carrot milling operation, was obtained from a nearby carrot-juice processing shop in a large quantity. Carrot residue was dried overnight at 80 °C in an oven. The dried residue was ground and sieved with a 60-mesh US standard sieve to get homogenous particle sized material.

Preparation of stock solution and standards

For preparation of 1000 ppm stock solution of each metal ion, 1.596 g of Pb(NO3)2 and 2.74 g of Cd(NO3)2. 4H2O were dissolved separately in one liter volumetric flasks and final volumes were made up to the mark with deionised water. The standard solutions of lead and cadmium (5 to 50 ppm) were prepared by successive dilution of their respective stock solutions.

Batch adsorption procedure

The adsorption experiments were carried out at room temperature i.e. 25 ºC at the desired pH value, contact time and adsorbent dosage level using the necessary adsorbent in a 100 ml conical flask containing 50 ml of 50 ppm test solutions of lead and cadmium ions, respectively. The required amount of dried carrot residue adsorbent was added and contents in the flask were shaked for the desired contact time in an electrical shaker at the speed of 100 rpm. The time required for reaching the equilibrium condition was estimated by drawing samples at regular intervals of time till equilibrium was reached. The contents of the flask were filtered through Whattman # 01 filter paper and the filtrate was analyzed for lead and cadmium ions concentration using atomic absorption spectrophotometry. The percentage removal of these metal ions was calculated by following equation.

$$\% \text{ Removal} = \frac{(C_o - C_f)}{C_o} \times 100 \qquad (1)$$

Where C_i and C_f are the initial and final concentration of metal ions (mg/L).

The amount of lead and cadmium adsorbed at equilibrium per unit mass of the adsorbent material is q_e (mg/g), which is calculated by using the following equation:

$$q_e = \frac{(C_o - C_f)}{m} \times V \qquad (2)$$

Where 'V' is the volume of the solution in liter and 'm' is the mass of the dry adsorbent material in grams.

Effect of adsorbent dose

For investigating the effect of adsorbent dose on the removal of metals ions, experiments were conducted by agitating 50 ml of 50 ppm solution of Pb^{2+} and Cd^{2+} containing different amount of carrot residue adsorbent ranging (0.5 – 4.5 g) with the difference of 0.5 g for a period of 10 minutes. The effect for time of contact on lead was studied by using 50 ml of 50 ppm of Pb^{2+} ions solution containing 1.5 g dose of carrot residues adsorbent and agitated for the time ranging from 5 to 45 minutes with the difference of 5 minutes interval. Similar procedure was repeated for Cd^{2+} with 3.0 g of adsorbent dose. Effect of **a)** biosorbent dose, **b)** contact time, and **c)** pH for sorption of lead and cadmium ions on carrot residue is given in Figure 1.

Effect of pH

The effect of pH on lead removal was studied by using 50 ml of 50 (mg/L) solution of Pb^{2+} (adjusted to initial pH 1-9) and agitated with 1.5 g of carrot residues adsorbent for 35 minutes. Similar procedure was repeated for Cd^{2+} solution with 3.0 g of adsorbent dose with the 30 minutes contact time.

Thermodynamic effect

The thermodynamic effect was studied by agitating 50 ml of 50 ppm of Pb^{2+} solution with 1.5 g of carrot residues adsorbent dose at temperatures 30, 40, 50, 60, 70 and 80 ºC for a time period of 35 minutes. Similar procedure was repeated for Cd^{2+} solution with 3.0 g of adsorbent dose for a time period of 30 minutes.

Adsorption isotherms study

For the study of adsorption isotherms, five 100 ml measuring flasks containing 10 to 50 ppm Pb^{2+} and Cd^{2+} solution were used independently. 1.5 g carrot residues for lead and 3.0 g for cadmium were added to each flask independently. pH of each Pb^{2+} sample solution was adjusted to 3 and 5 for Cd^{2+} sample solution. Each flask was agitated for 35 minutes for lead and cadmium respectively. All samples were filtered and filtrates were subjected to atomic absorption spectrophotometer for the determination of lead and cadmium ions concentration. A graph was plotted according to Langmuir and Freundlich isotherm equations and are shown in Figure 2a and 2b respectively. Other parameters were calculated from slope and intercept of straight line.

Results and Discussion

Effect of adsorbent dosage

The effect of variation in adsorbent dosages on the removal efficiency of lead and cadmium ions i.e. 0.5-6.0 g/50 ml of solution was studied. Observation shows that removal efficiency for both lead and cadmium ions were raised with increasing adsorbent dosage. Maximum removal efficiency for lead and cadmium ions was 91.32% and 89.9% respectively. In the case of lead, adsorption increases from 88.05 – 91.32% when the adsorbent dose is increased from 1.0 to 3.0 and then become almost constant. While in the case of cadmium, adsorption increases from 67.6 – 89.9% for 1.0 to 6.0 g of adsorbent dose and after that equilibrium was established. Hence the percentage removal decreases with increase in adsorbent dosage. The increase in adsorption percentage with the rise in adsorbent dosage is due to increase in active sites on the adsorbent that makes an easy penetration of the metal ions on the available sorption sites [22]. The decrease in percentage removal after equilibrium is due to less available sites. Therefore, in next experiments 3.0g/50mL and 6.0g/50mL were selected as optimum adsorbent dosage for lead and cadmium ions respectively as shown in Figure 1a.

Effects of contact time

The contact time is one of the important parameters for successful usage of adsorbents in practice. Figure 1b shows the effect of contact time on the removal efficiency of lead and cadmium ions using 3.0g/50 ml and 6.0g/50 ml carrot residues respectively. The removal efficiency increases gradually up to 92.56% for lead and 90.77% for cadmium ions with the increase in contact time up to 35 minutes. Therefore, this optimum contact time was selected for further experiments.

Effect of pH

The effect of pH on the removal efficiency of lead and cadmium ions is studied from pH 1–10. The maximum removal efficiency was found to be 92.6% for lead at pH 3 and 93.8% for cadmium ions at pH 5 respectively and is shown in Figure 1c.

At low pH, carboxylic and amino functional groups of carrot residue adsorbate are protonated [23], thus active sites are less available for metal ion binding and so lead and cadmium ions are less absorbed. Whereas, at higher pH, carboxylic and amino functional groups are

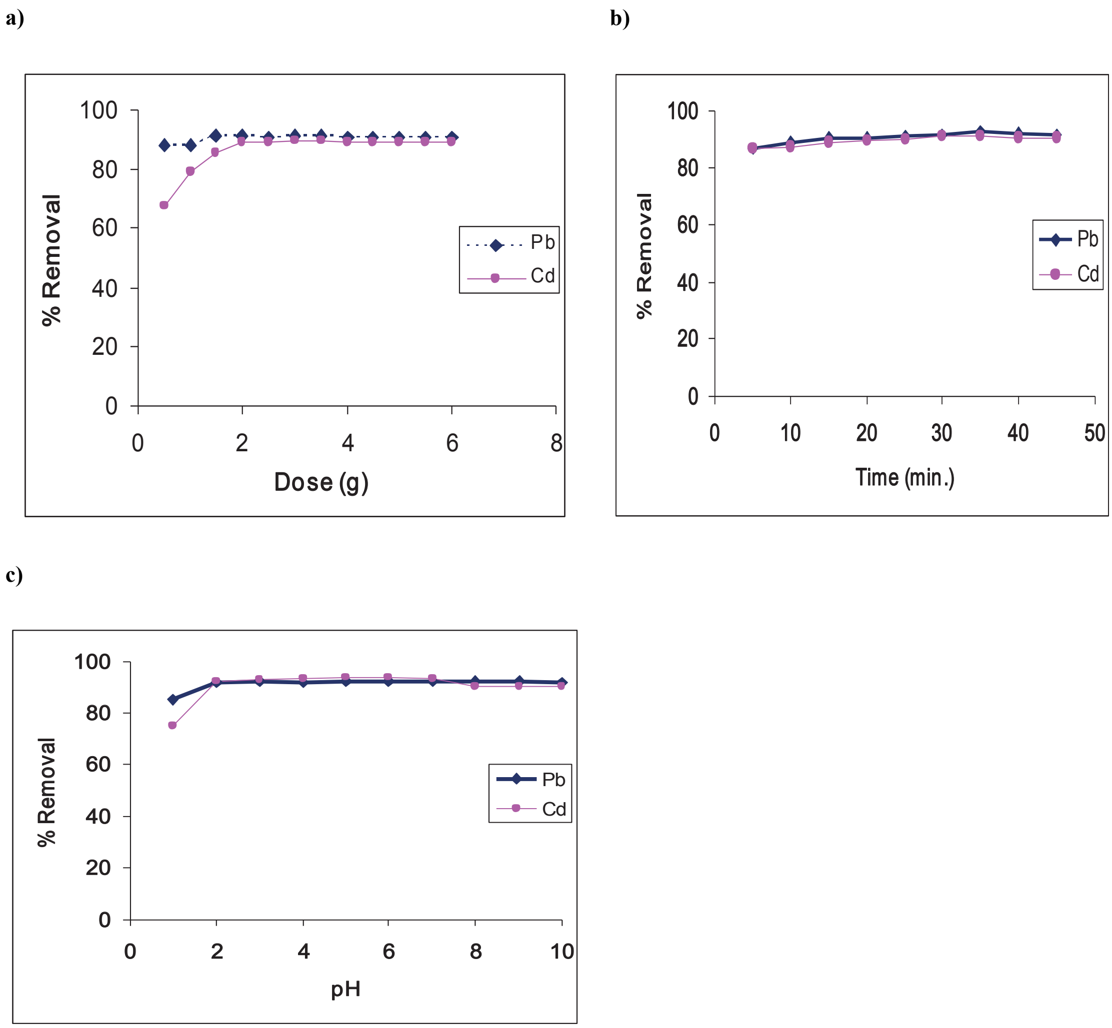

Figure 1. Effect of a) biosorbent dose, b) contact time, and c) pH for sorption of lead and cadmium ions on carrot residue

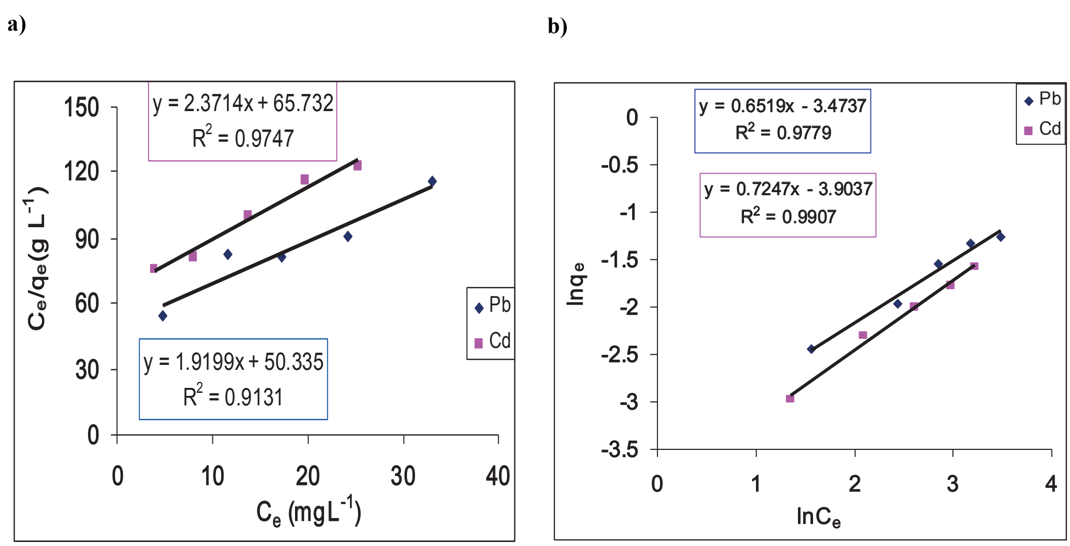

Figure 2. Adsorptions isotherms; Langmuir a), Freundlich b) for sorption of lead and cadmium ions on carrot residue

deprotonated and active sites are abundantly available for metal ion binding, so lead and cadmium ions are more adsorbed on carrot residues.

Adsorption isotherm models

Adsorption equilibrium data is very important to optimize the parameters of an adsorption system. It is also helpful to provide sufficient physicochemical information to understand the mechanism of adsorption. The sorption capacity of an adsorbent can also be described by equilibrium sorption isotherm, which is characterized by definite constants whose values express the surface properties and affinity of adsorbent material. In this study, Langmuir and Freundlich isotherm models are selected to fit the experimental data.

The Langmuir model assumes that adsorption occurs at specific homogeneous sites on the adsorbent and is used successfully in many monolayer adsorption processes.

This model can be written as follows;

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \left(\frac{1}{q_{max} \times K_L}\right)\frac{1}{C_e} \quad (3)$$

Where q_e is the equilibrium metal ion concentration on the adsorbent (mg/g), C_e is the equilibrium metal ion concentration in the solution (mg/L), q_m is the monolayer adsorption capacity of the adsorbent (mg/g), and K_L is the Langmuir adsorption constant (mg/L) relating the free energy of adsorption in which a plot of C_e/q_e versus C_e gives a straight line of slope = $\frac{1}{q_{max} \times K_L}$ and intercept = $\frac{1}{q_{max}}$. The values of linear correlation coefficients (R^2) strongly support the fact that metal–adsorbent adsorption data closely fit the model [24].

A linear relationship is shown between the amount of lead and cadmium ions (mg) sorbed per unit mass of *carrot residue* (g) against the concentration of lead and cadmium ions in the remaining solution (mg/L). The values of linear correlation coefficients R^2 and Langmuir parameters are given in Table 1. These results indicate that the adsorption of the metal ions onto *carrot residue* fitted well with the Langmuir model. The maximum adsorption capacity (q_m) was found to be 0.522 mg/g for lead ion and 0.421 mg/g for cadmium ion. The K_L value was found to be 0.058 L/mg for lead ions and 0.036 L / mg for cadmium ions.

The Freundlich model can be applied for non-ideal sorption on heterogeneous surfaces and multilayer sorption. The Freundlich model is described as;

$$\ln q_e = \ln K_f + \frac{1}{n}(\ln C_e) \quad (4)$$

Where K_f is a constant relating the adsorption capacity and 1/n is an empirical parameter relating the adsorption intensity, which varies with the heterogeneity of the material. The values of K_f and 1/n were found to be 32 and 1.533 for lead and 51.28 and 1.37 for cadmium metal ions respectively. The value of 'n' is greater than 1 indicates that the adsorption of lead and cadmium on carrot residues is favourable. Freundlich parameters are given in Table.1

Thermodynamic parameters for the adsorption system

Thermodynamic parameters for the adsorption of lead and cadmium ions on carrot residue such as heat of adsorption enthalpy change (ΔH°), entropy change (ΔS°) and free energy change of adsorption (ΔG°) were calculated by using the following equations.

$$\Delta G^o = \Delta H^o - T\,\Delta S^o \quad (5)$$

$$\Delta G^o = -RT \ln K_c \quad (6)$$

$$\ln K_c = \frac{-\Delta H^o}{RT} + \frac{\Delta S^o}{R} \quad (7)$$

Where, $K_c = C_s/C_e$, where C_s is solid phase concentration at equilibrium and C_e is the equilibrium concentration in solution (mg/L), R is the gas constant (8.3143J/mol K), and T is the temperature in Kelvin.The values of ΔH° is calculated from the slope and of ΔS° is from the intercept of linear plots of $\ln K_c$ versus 1/T as given in the Figure 3. The values of thermodynamic parameters ΔH°, ΔG° and ΔS° are given in Table 2. The negative value of ΔG° at different temperatures indicate the spontaneous nature of adsorption. As the temperature increases, the ΔG° values increases, indicating more driving force and hence resulting more adsorption capacity at higher temperatures. The positive values of ΔH° and ΔS° confirms the endothermic nature and the increased randomness at the solid/solute interface during the adsorption of lead and cadmium ions.

Kinetic parameters

The Lagergren's kinetics equation has most widely been used for the adsorption of an adsorbate from an aqueous solution. The Lagergren model may not reflect the true nature of kinetic study. It is applied due to its simplicity and good fit [25]. The model is based on the assumption that rate is directly proportional to the number of free sites.

$$\log(q_e - q_t) = \log q_e - \frac{k}{2.303}(t) \quad (8)$$

$\text{Log}(q_e\text{-}q_t)$ vs. t was plotted to evaluate this kinetic model and to determine the rate constant 'k'. From values of R^2 (Figure 4a and Table 3), it is observed that the Lagergren's model is not fitted well for the sorption of lead and cadmium on carrot residue.

The second order model is based on the biosorption followed by second order mechanism nearby the rate

Table 1. Adsorption isotherms parameters for biosorption of lead and cadmium ions on carrot residue

Metals	Langmuir isotherms			Freundlich isotherms		
	q_m	K_L	R^2	K_F	n	R^2
Lead	0.522	0.038	0.913	32.00	1.53	0.977
Cadmium	0.421	0.036	0.974	51.26	1.37	0.990

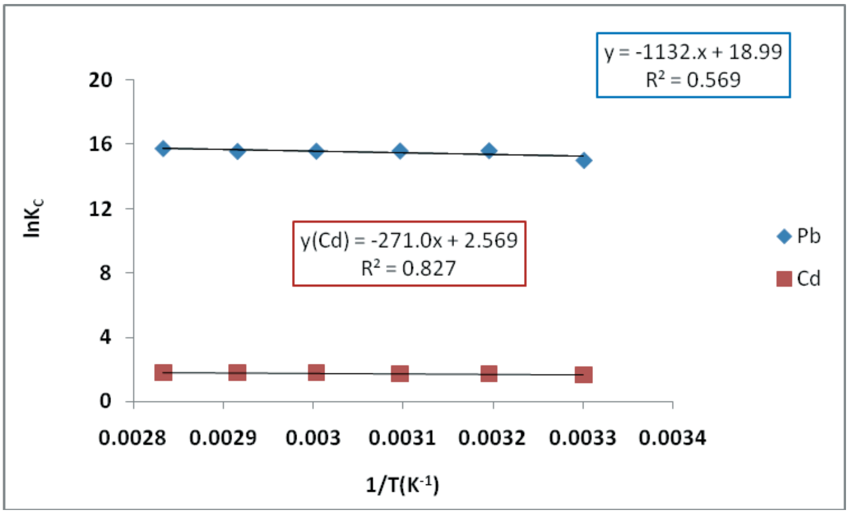

Figure 3. Thermodynamic parameters for biosorption of lead and cadmium ions on carrot residue

Table 2. Thermodynamic parameters in $kJmol^{-1}$ for biosorption of lead and cadmium ions on carrot residue

T (Kelvin)	1/T (Kelvin)		lnK_C for Cd	lnK_C for Pb
303	0.00303		1.663836	15.00957
313	0.00313		1.703466	15.62092
323	0.00323		1.721425	15.60585
333	0.00333		1.796185	15.59179
343	0.00343		1.776966	15.57852
353	0.00353		1.779687	15.76123
Metals	ΔG^o (kJ/mol)		ΔS^o (kJ/mol)	ΔH^o (kJ/mol)
	303K	353K		
Lead	-41.90	-51.20	0.157	9.41
Cadmium	-38.13	- 44.42	0.021	0.157

a) b)

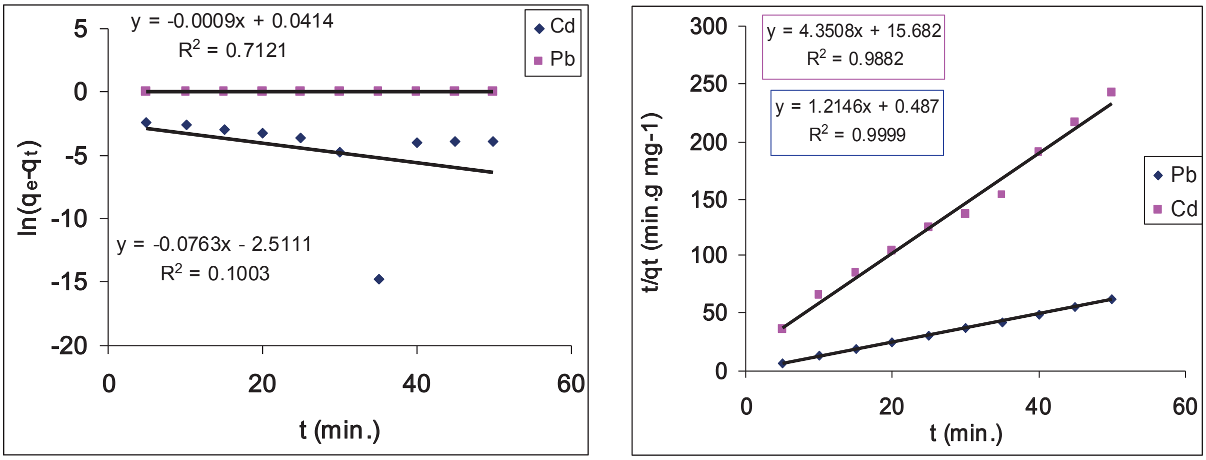

Figure 4. Pseudo first a), and second b), order kinetics for sorption of lead and cadmium ions on carrot residue

Table 3. The adsorption rate constant for adsorption of lead and cadmium ions on carrot residue

Metals	Pseudo-first order				Second order			
	k_1 (min^{-1})	qe(cal.) (mg/g)	qe(Exp.) (mg/g)	R^2	k_2 (g/mg min)	qe(cal.) (mg/g)	qe(Exp.) (mg/g)	R^2
Lead	0.0009	1.04	0.822	0.712	3.00	0.822	0.823	0.999
Cadmium	0.0763	0.0712	0.228	0.100	1.20	0.229	0.228	0.988

of sorption is proportional to the square of number of unoccupied sites [26] and can be represented as equation 7.

$$\frac{t}{q} = \frac{1}{K_f q_e^2} + \frac{1}{q_e}(t) \quad (9)$$

Where q_e and t are the adsorption capacity (mg/g) at equilibrium and at time 't' and K_f is the rate constant of the second order model for the adsorption process (gmg^{-1} min^{-1}). Values of K_f and q_e can be calculated from the plot of t/qt against 't' as shown in Figure 4b and are given in Table 3. These results show that the adsorption of lead and cadmium ions on carrot residue has followed the second-order kinetic model at all time intervals. The correlation coefficient R^2 values for the second order kinetic plots are higher as shown in Figure 4b. But in case of pseudo-first-order, calculated q_e values did not agree well with experimental q_e values and the value of the correlation coefficients was low as given in Table 3.

Conclusions

It is evident from this study that adsorption of lead and cadmium ions is highly dependent on amount of adsorbent, contact time and pH. The kinetic studies reveal that second order model is the best fit . This adsorption data is also fitted well for Langumir and Freundlich isotherms from which it can be concluded that the *carrot residue* has important potential for the removal of lead and cadmium ions from aqueous solutions. The endothermic nature of adsorption is indicated by thermodynamic parameters. Positive value of ΔH° showed that adsorption is favourable at higher temperature. It can be concluded that carrot residue is an environment friendly, biodegradable, low cost and potential biosorbent for the removal of heavy metals like lead and cadmium.

Conflict of Interest

There is no conflict of interest among the authors who contributed to the present study.

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