International Journal of Chemical Sciences
International Journal of Chemical Sciences
ISSN: 0972-768 X

Research | Vol 15 Iss 3

Equilibrium and Kinetic Studies for the Biosorption of Cd (II) in Aqueous Solution onto Almond Gum

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Received: June 16, 2017; **Accepted:** July 20, 2017; **Published:** July 22, 2017

Abstract

Batch adsorption process was carried out for the removal of cadmium (II) from aqueous solution using almond gum. The material was characterized using standard physio-chemical methods, SEM and FTIR studies. The adsorption isotherm was evaluated by means of Langmuir, Freundlich, Dubinin Radushkevich (D-R) and Temkin isotherm models. A maximum Langmuir adsorption capacity of 40.0 mg/g at 32°C was obtained for initial concentration of 10 mg/L, 20 mg/L, 30 mg/L and 40 mg/L. The order of the kinetics was also determined and adsorption of cadmium (II) followed second order kinetics which shows that physisorption may be involved in the process. Thermodynamic parameters (ΔG°, ΔH° and ΔS°) showed that the reaction to be endothermic, spontaneous and increasing randomness of the solid solution interface. The cost of almond gum is economically effective compared to commercial activated carbon.

Keywords:* *Almond gum; Temkin; Elovich; Desorption

Introduction

Nowadays, the environmental contamination is not the only concern of scientific community. Most of the human population is exposed to heavy metals through food, water, air or absorption on the skin [1]. Heavy metal pollution has turned out to be one of the most severe environmental problems because of the tendency to bio-accumulate in ecosystem [2]. Cadmium is a non-degradable and persistent metal [3], classified as priority pollutant by United States of Environmental Protection Agency [4]. Cadmium is released into the environment by industrial effluents from electroplating, pigments, plastic, battery and zinc refining industries [5], and finds its way into the natural water systems [6]. The drinking water guideline value recommended for this element by World Health Organization and American Water Works Association (AWWA) is 0.005 mg/L [7]. Cadmium, even in traces, may cause chronic health problems, lung edema, renal dysfunction [8-10], abdominal pain, convulsions, hypertension, loss of appetite, fatigue, sleeplessness hallucinations, headache [11], and osteoporosis respectively. Hence, removal of cadmium from water sources becomes important.

Many conventional techniques have been proposed such as oxidation, reduction [12], ion exchange, reverse osmosis, precipitation, solvent extraction, membrane technology, electrochemical treatment, flocculation, coagulation, and complexation [13-16]. These techniques have their own inherent limitations such as less efficiency, sensitive operating conditions, secondary sludge and costly for disposal [17]. Adsorption, on the other hand, is found to be the best technique for the remediation of heavy metal due to its simple design and affordability.

The last few decades have witnessed tremendous development of new adsorbents and modified performance of existing ones such as activated carbon [18], waste Fe (III)/Cr (III) hydroxide [19], sweet potato peel [20], guar gum silica nano composite, pomegranate peel [21], orange rind, *Crambe abyssinica* Hochst seeds, marine sponge *Cinachyrella tarentina* and banana peel [22-25].

Almond gum is produced by almond tree *(Prunus amygdalus,* Rosaceae family). Almond gum represents an abundant natural biomass worldwide. Almond gum is insoluble in aqueous solutions and could be used as adsorbent for the removal of cadmium (II) from waste water.

The objective of the study is to characterize the almond gum and find its feasibility for batch mode studies such pH, initial concentration, adsorbent mass and temperature on the treatment process. Langmuir, Freundlich, Dubinin Radushkevich and Temkin isotherm models were employed to quantify the biosorption equilibrium.

Material and Methods

Almond Gum was used as adsorbent which is commercially available in the local market of Kadapa town, Andhra Pradesh, India. The gum was ground and sieved for uniform particle size of 100 µm. The material was characterized using physio-chemical methods, SEM (Model JEOL ion sputtering device) and FTIR (Perkin Elmer Spectrum, two models, UK) respectively.

Preparation of cadmium nitrate stock solution

Cadmium nitrate [$Cd(NO_3)_2.4H_2O$], molecular weight is 308.47 g/mol, was supplied by Merck, India was used as adsorbate. The stock solution was prepared by dissolving requisite amount of cadmium nitrate in 1000 ml distilled water. The solutions were stored in brown glass bottles and used to prepare different working concentrations (10 mg/L, 20 mg/L, 30 mg/L and 40 mg/L).

Batch adsorption experiment

For batch method, 50 ml of the cadmium (II) solution with the known initial concentration and a known amount of the adsorbent were taken in a 250 ml conical flask. This mixture was agitated at 32°C by keeping the conical flask in orbital shaker at a constant speed of 180 rpm. The Cadmium (II) solution was separated from the adsorbent by centrifugation at 10,000 rpm for 20 min and the absorbance was determined using double beam UV visible spectrophotometer (LabIndia

3000+UV) at 575 nm, by the Xylenol orange colorimetric method [26]. The percentage removal of Cadmium (II) and equilibrium adsorption uptake, q_e (mg/g), was calculated using the following relationships:

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

$$\text{Amount adsorbed } (q_e)=\frac{(C_0-C_e)}{w}\times V \quad (2)$$

Where C_0 is the initial adsorbate concentration (mg/L), C_e is the equilibrium adsorbate concentration (mg/L), V is the volume of the solution (L) and w is the mass of the adsorbent (g).

Desorption study

After adsorption, the adsorbate-loaded adsorbent was separated from the solution and the supernatant was drained out. The adsorbent was gently washed with distilled water at various pH values. Then, they were agitated for the equilibrium time and the desorbed solution was estimated.

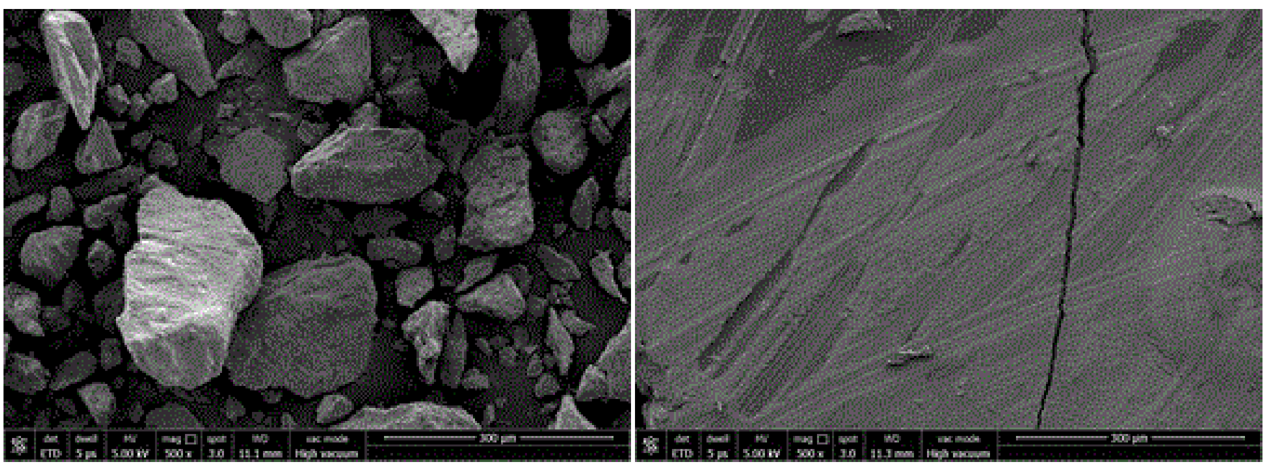
Results and Discussion

Characterization of the adsorbent

Almond gum is an acidic polysaccharide composed on dry weight by 2.45% of protein, 0.85% of fats and 92.36% of carbohydrates with traces of mannose, rhamnose and glucuronic acid respectively [27]. The physio-chemical characteristics of almond gum are given in TABLE 1. The surface morphology of the adsorbent material by SEM shown in FIG. 1 (a) and 1(b) revealed that pores at the surface of almond gum were filled by the cadmium (II) ions adsorption. A similar observation with respect to the porous structure with large surface area on to the orange and (*Citrus sinensis) (L.) osbeck* for cadmium biosorption has been reported. The X-Ray Diffraction of the almond gum exhibit broad peaks and absence of a sharp peak that revealed the amorphous structure. An FTIR spectrum revealed that the peaks observed at 2940 cm^{-1} and 2942 cm^{-1} (before and after biosorption) was associated with the stretching vibrations of C-H bond of methyl, methylene and methoxy groups. Furthermore, from FIG. 2 (a) and 2(b) it was observed that there are shifts in C-H stretching vibrations (2147 cm^{-1} to 2155 cm^{-1}). The same findings were also being reported [25]. This shift in the vibrations indicates the inference of the Cd (II) bonding onto the biosorbent. The results of the characterization are shown in TABLE 1.

TABLE 1. **Physical and chemical characteristics of the adsorbent.**

pH zpc	5.4
Bulk density (g/ml)	0.856
Mechanical moisture content (%)	9.8
Ash content (%)	9.42
pH	4.79
EC (µs/cm)	520
Water soluble content (%)	19.7
Acid soluble content (%)	27.42
Sodium (Na) (ppm)	408.86
Potassium (K) (ppm)	340.36
Calcium (Ca) (ppm)	1637.33
Phosphate (ppm)	4.81
Decolorizing power (mg/g)	225
Specific gravity	3.448



A B

FIG. 1. **SEM pictures of almond gum (a) before and (b) after Cd (II) biosorption.**

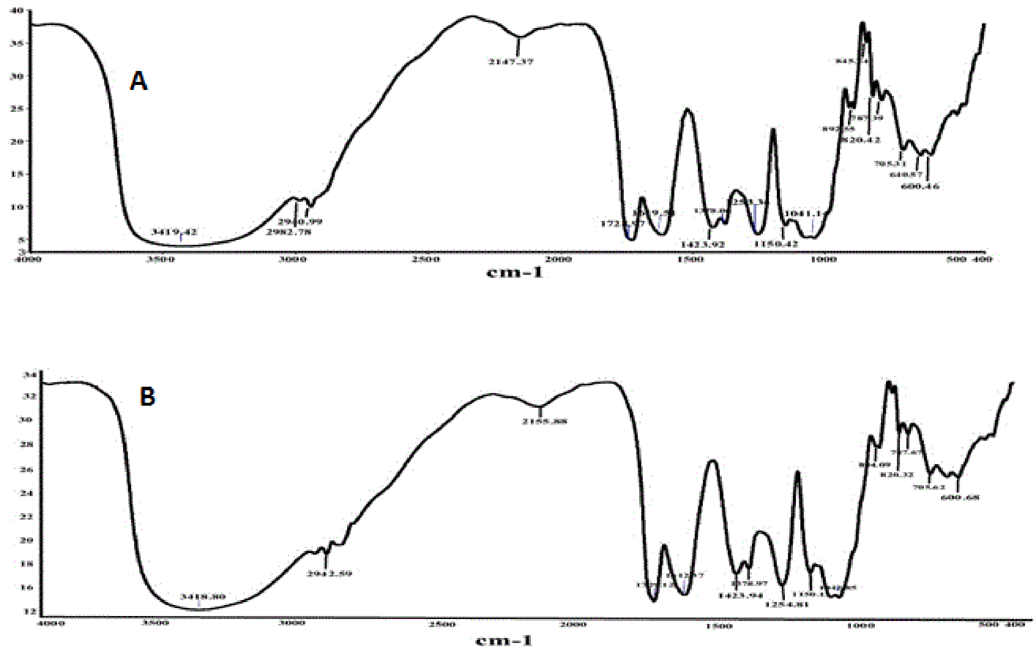


FIG. 2. **FTIR spectra of almond gum (a) before and (b) after biosorption of Cd (II).**

Effect of contact time and initial Cd (II) concentration

The biosorption efficiency of the cadmium ions was performed as a function of contact time and initial concentration. The adsorption rate was relatively fast initially and adsorption equilibrium was achieved within 40 min for 20 mg/L of cadmium concentration. The amount of Cd (II) adsorbed onto almond gum increased from 8.03 mg/g to 25.83 mg/g with the increase in concentration from 10 mg/L to 40 mg/L. Similar findings have been reported by several authors for the adsorption of cadmium removal from aqueous solution by pine cone biochar, *Prunus avium* leaves [28] respectively.

Adsorption kinetics

The uptake of Cd (II) by the almond gum has been modeled using first order, pseudo second order and Elovich respectively. The pseudo-first-order rate equation is [29]:

$$\frac{dq_t}{d_t} = k_1\left(q_e - q_t\right) \qquad (3)$$

Integrating equation (3) for the boundary conditions t=0 to t=t and q=0 to q=q gives

$$\ln\left(q_e - q\right) = k_1 t \qquad (4)$$

Where, q and q_e are the grams of solute adsorbed per gram of adsorbent at any time and at equilibrium, respectively, and k_1 is the rate constant of first-order adsorption.

Second order kinetic model [30]:

$$\frac{dq_t}{d_t} = k_2\left(q_e - q_t\right)^2 \quad (5)$$

Where, q_e and q_t are the adsorption capacity at equilibrium and at time t, respectively (mg/g). For the boundary conditions to q_t=0 to q_t=q_t at t=0 to t=t; the integrated form of equation (5) becomes:

$$t/q=1/k_2q_e^2+t/q_e \quad (6)$$

If the pseudo-second order kinetics is applicable, the plot of t/q versus t of equation (6) should be linear relationship from which q_e and k_2 can be determined from the slope and intercept of the plot. The calculated equilibrium values obtained from second order were very close to the experimentally determined values (q_e) with the correlation coefficient (R^2) being 0.999 for 30 mg/L initial Cd (II) concentration and were thus higher than the R^2 values of first order kinetic (TABLE 2). The applicability of this model implies that the rate limiting step may be chemisorption involving valency forces through sharing or exchange of electrons between adsorbate and adsorbent [30]. Obtained results are in accord with those similar studies of cadmium and lead adsorption onto mesoporous activated carbon, sorption of Cd (II) by sweet potato [31].

The Elovich model equation is generally expressed as [32]:

$$q = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\beta}\ln t \quad (7)$$

The values of β and α are calculated from the slope (1/β) and the intercept [ln (αβ)/β] of the plot and are listed in TABLE 2.

Equilibrium isotherms

The Langmuir isotherm has been one of the most widely used isotherms applied to pollutant biosorption process. The model can be written in the form as:

$$C_e/q_e=1/Q_ob+C_e/Q_o \quad (8)$$

TABLE 2. **Kinetic model constant values for Cd (II) adsorption by almond gum.**

Model	**Kinetic parameters**	**Initial cadmium (II) concentration (mg/L)**			
		10	20	30	40
Pseudo-first order	$q_e$exp (mg/g)	8.038.03	13.97	19.24	25.83
	K_1 (min^{-1})	0.359	0.106	0.085	0.023
	q_e cal (mg/g)	0.072	0.711	6.834	1.235
	R^2	0.996	0.998	0.995	0.997
Pseudo-second-	q_e cal (mg/g)	8.22	14.10	19.42	25.64

order	K_2 (min^{-1})	0.846	0.384	0.420	0.293
	R^2	0.995	0.998	0.999	0.999
Elovich	α	5.23×10^2	8.09×10^3	2.09×10^6	9.92×10^6
	β	1.049	0.801	0.881	0.716
	R^2	0.995	0.996	0.994	0.994

The Langmuir isotherm assumes that the surface has homogeneous binding sites, equivalent adsorption energies and no interaction between adsorbed species and its relevant parameters are Langmuir constant "b" which refers to the energy constant related to the heat of adsorption capacity and Q_o which represents the maximum loading capacity of the adsorbent [30]. The adsorption capacity, Q_o was found to be 40.0 mg/g (TABLE 3).

A characteristic of the Langmuir isotherm can be expressed using a dimensionless constant called the separation factor R_L as:

$$R_L=1/(1+bC_o) \tag{9}$$

Moreover, the R_L values decreased as the initial Cd(II) concentration increased, suggesting that the adsorption process was more favorable at higher initial Cd(II) concentrations, which was also proved by the intensity factor of Freundlich model since n>1, implying that adsorption intensity was favorable at high concentrations and much less at lower concentrations. The maximum monolayer loading capacity (Q_o) obtained for the biosorption of Cd (II) ion from aqueous solution was compared to other adsorbents reported in the literature are presented in TABLE 4.

The Freundlich isotherm assumes a heterogeneous adsorption surface and active sites with different energy. The linear form is given as eqn. (10).

$$\text{Log } q_e=\log k_f+1/n \log C_e \tag{10}$$

Where k_f denotes approximate adsorption capacity and n is related to intensity of adsorption. TABLE 3 shows the obtained k_f and n. The correlation coefficient values and the plot q_e vs. C_e indicated that the Freundlich isotherm gave a good fit to the biosorption process.

Dubinin-Radushkevich (D-R) isotherm approach assumes that there is a surface area where the adsorption energy is homogeneous. The linear form of the equation is:

$$q_e=q_m\exp(-B\varepsilon^2) \tag{11}$$

The values of q_m, B, E and regression coefficient (R^2) are listed in TABLE 3. The importance of the E value is as follows: E<8 kJ/mol (physical adsorption); E=8-16 kJ/mol (ion exchange); E=20-40 kJ/mol (chemical adsorption). The estimated value of E was found to be 35. 71 kJ/mol, which indicates that the adsorption of Cd (II) ions by almond gum is a chemical adsorption process.

The Temkin isotherm is represented as:

$$q_e = B \ln A + B \ln C_e \qquad (12)$$

Where, C_e is the concentration of the adsorbate at equilibrium (mol/L), q_e is the amount of adsorbate adsorbed at equilibrium (mol/g), RT/h=B; and A and h are constants. The adsorption data can be analyzed according to eqn. (12). A plot of q_e vs. ln C_e enables the determination of the constants A and B.

FIG. 3 presents different adsorption isotherms along with the experimental data. In order to compare the validity of isotherms, a normalized deviation, Δq (%), was calculated using the following equation:

$$\Delta q(\%) = 100 \times \sqrt{\frac{\sum\left[\left(q_e^{\exp} - q_e^{cal}\right)/q_e^{\exp}\right]^2}{(n-1)}} \qquad (13)$$

Where, superscripts ‘exp’ and ‘cal’ are the experimental and calculated values, respectively, and ‘n’ is the number of measurements. From the results it was found that values of Δq obtained from Langmuir [Δq (%)=3.5], Freundlich [Δq (%)=5] and Temkin [Δq (%)=7.0] were lower compared to D-R [Δq (%)=47.4] adsorption isotherm models. It shows that the equilibrium data fit better with Langmuir, Freundlich and Temkin isotherms over the entire range of concentrations compared to D-R and isotherm.

TABLE 3. **Isotherm parameters for the biosorption of Cd (II) onto almond gum.**

Langmuir constants			
C_o (mg/L)	**Q_o (mg/g)**	**b (L/mg)**	**R_L**
10			0.998
20	-	-	0.997
30	40.0	0.106	0.995
40			0.997
Freundlich constants			
C_o (mg/L)	kf ($mg^{1-1/n}L^{1/n}$ g^{-1})	n	R^2
10	1.242	0.546	0.999
20	0.034	0.345	0.999
30	2.393	0.265	0.999
40	0.02	0.379	0.996
Dubinin-Radushkevich constants			
C_o (mg/L)	qm (mg/g)	E (kJ/mol)	R^2
10	297.98	35.71	0.996
Temkin constants			
A (L/mg)	B (mg/g)	-	R^2
1.09	13.68	-	0.999

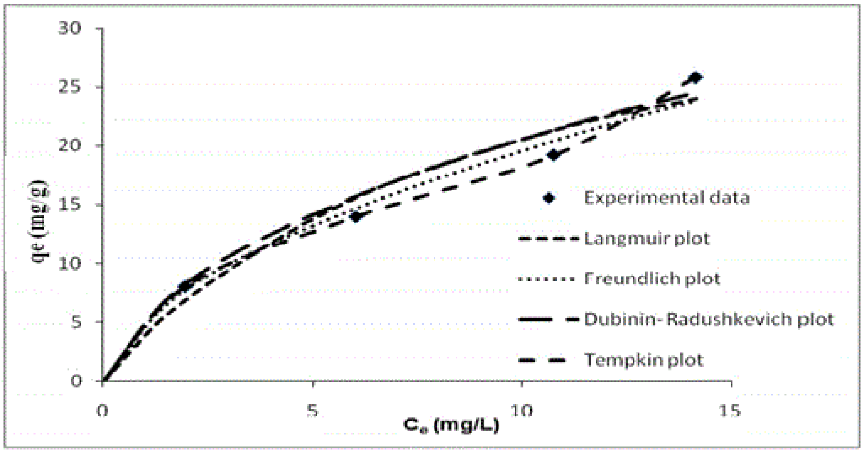


FIG. 3. **Adsorption isotherms of Cd (II) ions.**

TABLE 4. **Comparison of the maximum adsorption capacities for Cd (II) by other adsorbents.**

Adsorption	**q_{max}(mg/g)**	**References**
Sun flower (*Helianthus annuus*)	14.9	35
Wheat bran	15.71	36
Techtona grandis L.f	23.20	18
Fe (III)/Cr (III) hydroxide	3.08	19
Durian peel	18.5	37
Coconut copra meal	5.1	38
Papaya wood	17.22	39
Cashew nut shell	22.11	40
Coffee grounds	15.65	41
Sweet potato peels	18. 9	20
Pomegranate peel	18.52	21
Commercial activated carbon	27.3	30
Mustard biomass	33.56	42
Crambe abyssinica Hochst seeds	19. 34	23
Banana peels	5.71	2
Almond gum	40.0	-

Effect of pH

The effect of pH on the biosorption of Cd (II) using almond gum, separate set of batch equilibrium adsorption experiments was conducted modifying the pH from 3.0 to 8.0 and illustrated in FIG. 4. The maximum biosorption was observed at pH 7.0 for Cd (II). The pH point of zero charge for the almond gum was found to be 5.4. At $pH>pH_{ZPC}$, the adsorbent is negatively charged and the adsorbate species are positively charged. Obviously, a negatively charged surface site did favor the adsorption of cationic Cd (II) ions because of electrostatic attraction. At pH 8, cadmium exists mainly as Cd $(OH)^+$ and thus adsorbed at negatively polarized surface sites. To avoid the possible precipitation of Cd $(OH)_2$, the pH studies should not be performed at pH>8. Similar trends have been reported for the adsorption of Cd (II) in other adsorbents [33,5,34,19].

Desorption studies

Desorption studies help recycle the adsorbent and to recover the metal. The percent desorption increased with decrease in pH and being capable of desorbing 26% (FIG. 4). In acidic conditions, H^+ ions protonate the adsorbent surface leading to the desorption of the positively charged Cd ions [35-37].

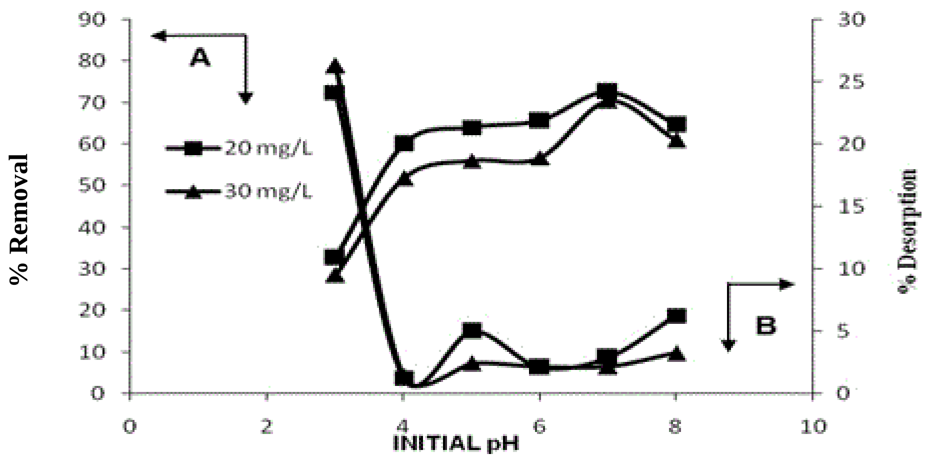


FIG. 4. **Effect of pH and desorption on removal of Cd (II) ions.**

Thermodynamics of biosorption

Thermodynamic parameters such as the change in standard free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) of adsorption were calculated using the following equations [19,23]:

$$\Delta G° = -RT \ln K_c \qquad (14)$$

Where R is gas constant (8.314 J/mol/K), b is equilibrium constant (L/mol) and T is the temperature in (K):

$$\log K_{c=} (\Delta S°/2.303 \times R) - (\Delta H°/2.303 \times RT) \qquad (15)$$

The values of ΔH and ΔS were determined from the slope and intercept of van’t Hoff plots log K_c vs. 1/T [38-41]. Positive values of ΔH° listed in TABLE 5 suggest the endothermic nature of biosorption. The negative values of ΔG° indicate spontaneous nature of adsorption of Cd (II) onto almond gum [42,43]. The positive values of ΔS° show the increased randomness at the solid/solution interface during the biosorption of Cd (II) ion by almond gum.

TABLE 5. **Thermodynamic parameters.**

Temperature (°C)	K_c	ΔG° (KJ/mol)	ΔH°(KJ/mol)	ΔS° (J/mol/K)
32	4.082	-3.544	-	-
40	2.013	-1.82	32.42	95.69
50	1.960	-1.808	-	-

Conclusion

In this work almond gum, an inexpensive and efficient adsorbent are used for the removal of Cd (II) from aqueous solution. Results indicate that the pseudo second order kinetic model showed good correlation with experimental data and the equilibrium data was satisfied well with the Freundlich and Temkin isotherms equation. Biosorption increases with pH until the cadmium (II) ions are precipitated as cadmium hydroxide. Temperature studies showed that biosorption was endothermic and spontaneous in nature.

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