Research Article

Enhanced heavy metals biosorption using chemically modified chitosan coated microwave activated sugarcane baggage ash composite biosorbents

Linh My Nguyen[1] ⊙ · **Tien Thuy Hoang Nguyen**[1] ⊙

Abstract

In recent years, toxic pollutants in industrial wastewater are seriously considered as one of the environmental problems due to the heavy metals present in it. Currently, biosorption has been strongly recommended by researchers as an efficient and economical technique to reduce the amount of heavy metals in the different industrial wastewater sources. Chitosan and sugar bagasse ash are the well-known biosorbents due to their low cost, available and effective in adsorption process. In this study, the composite biosorbent was prepared by mixing chitosan and sugar bagasse ash prepared by microwaving at a ratio of 1:5 (ASB-CBs) in acid condition. Adsorbents were characterized by SEM analysis method and FTIR infrared spectra. The parameters affecting the Pb(II) and Cd (II) adsorption such as pH, adsorption time, dose of adsorbent was studied by running the batch experiments. Research data show that the optimal pH for the biosorption process is 5.0. Experimental data is described by the Freundlich equation. Thermodynamic process is also studied in which the negative free Gibbs energy indicated the metal ions adsorption on the ASB-CBs was spontaneous and endothermic nature.

Keywords Chitosan · Microwave charcoal · Sugarcane baggage ash · Composite biosorbents · Heavy metal removal

1 Introduction

In recent years, toxic pollutants in industrial wastewater is seriously considered as one of the environmental problems due to the heavy metals present in it. These contaminants are not decomposable easily and seriously to affect human health through the food chain causing various diseases and disorders. Among these contaminants, lead and cadmium are recurrently found together, for example, in the wastewater of textile, dye, metal plating, batteries and paint formulation [1]. In potable water, the amount of acceptable limit of Pb(II) is 0.01 mg L^{-1} and for Cd (II) is 0.003 mg L^{-1}. These heavy metals may generate long-term harm in human beings, including lung fibrosis, dyspnea, anemia, muscle and joint pains, kidney damage,

high blood pressure, etc. [2]. Because of their poisonous nature and high risk of accumulation it presents a serious threat to the environment, it is significant to reduce the amount of Cd (II) and Pb(II) from the wastewater before being directly discharged into the environment. Currently, biosorption has been strongly recommended by researchers as an efficient and economical technique to reduce the amount of heavy metals present in the different industrial wastewater sources [3–6].

Chitosan is used as an adsorbent to remove heavy metals in many studies due to its low-cost, abundance, and high selectivity against contaminants [7, 8]. Chitosan can effectively adsorb various kinds of particles, including heavy metal ions based on the presence of hydroxyl (–OH) and amino (–NH$_2$) functional groups [9]. In order

✉ Linh My Nguyen, linhnm@hcmute.edu.vn | [1]Department of Environmental Technology, University of Technology and Education, HCMC, 264 Hoang Dieu 2 St, Linh Chieu Ward, Thu Duc District, Ho Chi Minh City, Vietnam.

to augment the acid resistance condition of the chitosan, the chemical crosslink process was conducted with some specific agents such as glutaraldehyde, acid citric and epichlorohydrin [10–12]. For this research, to improve the raw chitosan acid stability and its mechanical strength, glutaraldehyde was applied to crosslink it. In contrast, the crosslinked polymer network affected the functional groups on the chitosan surface; which lead to decrease of its adsorption abilities. Currently, sugar bagasse, considered as waste after the extraction sugar juice from crushed cane, has been reported as a potential, low-cost biosorbent based on an agriculture by-product. The constituent of sugar bagasse are hemicellulose and lignin which process many oxygen functional groups, for instance hydroxyl; carboxyl and phenol groups [13, 14]. In this regard, sugar bagasse was prepared into activated sugar bagasse ash via microwave radiation to enhance its adsorption capacity and immobilized on the crosslinked chitosan beads.

The purpose of this research is to evaluate the adsorption abilities of the composite material between glutaraldehyde-crosslinked chitosan and activated sugar bagasse ash (ASB-CBs) for reduction of Cd (II) and Pb(II) from liquid solutions. To evaluate the parameters affecting the biosorption process such as solution pH, contact time, and temperature, the batch experiments were conducted. The adsorption kinetics were analyzed by applying the pseudo-first-order and pseudo-second-order models. The mechanism of metal ions adsorption through the modified crosslinked chitosan beads (ASB-CBs) was also examined for further elucidation this biosorption process.

2 Materials and methods

2.1 Materials

The standard solution of Pb(II) and Cd (II) (1000 mg L^{-1}) was provided by Merck Company (Germany). All reagents as well as chitosan used in this research were analytical graded and supplied by Sigma Aldrich. The sugar bagasse used in this work was collected from the Plantation Thanh Long (Tay Ninh, Vietnam). The sugar bagasse was dried, ground and sieved to the size from 0.1 to 0.15 mm for subsequent experiments.

2.2 Preparation of microwave activated sugar bagasse ash

Sugar Bagasse Ash (SBA, 10 g) was mixed with 10 mL of 91% H$_3$PO$_4$ in a porcelain beaker. The mixture was put into microwave model SHARP R-20A1(S)VN for 5 min at 500 W. The product was left to room temperature then centrifuged to wash until pH of solution was at 6.0–6.5.

The final result was dried in oven at 80 °C in 3 h to obtain the activated sugar bagasse ash (ASB) which will be used to modified on the crosslinked chitosan beads later.

2.3 Composite biosorbents preparation

Composite biosorbents were formulated by mixing chitosan powder (5 g) with activated sugar bagasse ash (1 g) dissolved into acetic acid (7%) (240 mL) and left for 16 h. The slurry was poured into an alkali coagulating solution (H$_2$O: methanol: NaOH = 4:5:1, w/w/w) (200 mL), the result are beads that have an average diameter of 3.5 mm. The products (ASB-CBs) were washed away by using distilled water to remove the residual NaOH [15–17]. In order to increase the acid resistance ability of the composite adsorbents, the wet ASB-CBs were mixed with a solution of glutaraldehyde (fixed at 1:2) within 16 h. The final beads were rinsed multiple times to remove the residual glutaraldehyde using distilled water. The end result (ASB-CBs) was dehydrated at 80 °C in an oven for 12 h. SEM, FTIR were analyzed to investigate the characteristic of biosorbent.

2.4 Adsorption experiments

The batch adsorption tests were carried out to evaluate the different parameters affected such as biosorbent dose, pH, agitated time, initial concentration of heavy metals on the removal efficiency of metal ions. The affecting of pH parameter was studied with the initiate pH from 3.0 to 8.0. The effect of contact time was carried out at 303 K and 200 rpm. The ASB-CBs (2 g L^{-1}) was stirred with a metal ions solution (100 mL). After been completely mixed by using a shaker incubator (model LM-570RD) at 303 K and 200 rpm, the residual concentration of metal ions was measured using an atomic absorption spectrophotometer (Spectra AA 220, FS Varian). Different concentrations of metal ions ranging from 10 to 250 mg L^{-1} were examined under the optimal temperature, pH, agitation time and biosorbent dose that have been found in the earlier tests.

The load of a single metal ion adsorbed on the surface of the adsorbent at equilibrium and time, q$_e$ and q$_t$ respectively, in mg g^{-1} were estimated based on the equations as follow:

$$q_e = \frac{(C_0 - C_e) \times V}{m} \tag{1}$$

$$q_t = \frac{(C_0 - C_t) \times V}{m} \tag{2}$$

where C$_0$, C$_e$, and C$_t$ are the initial concentrations of metal ion, at equilibrium and at time t in mg L^{-1}, correspondingly,
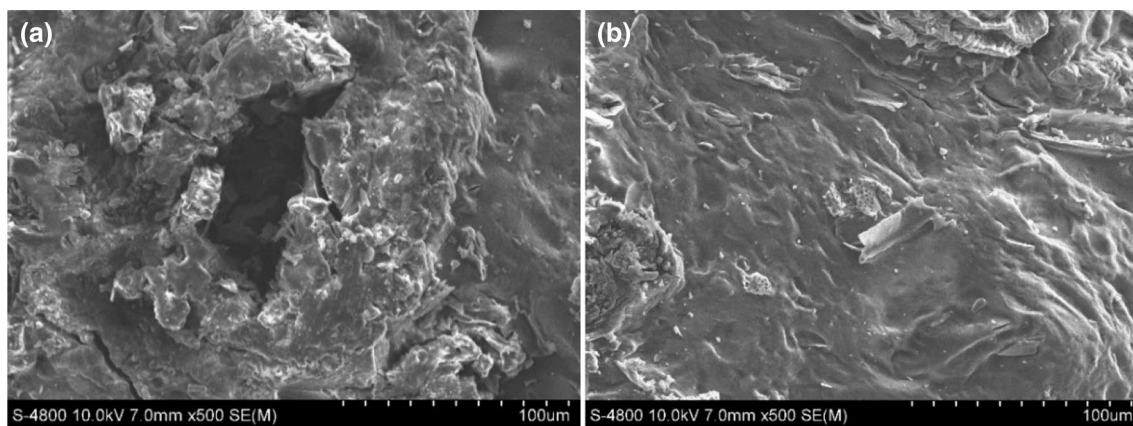
Fig. 1 SEM images of the **a** ASB-CBs, **b** metal ions-adsorbed ASB-CBs
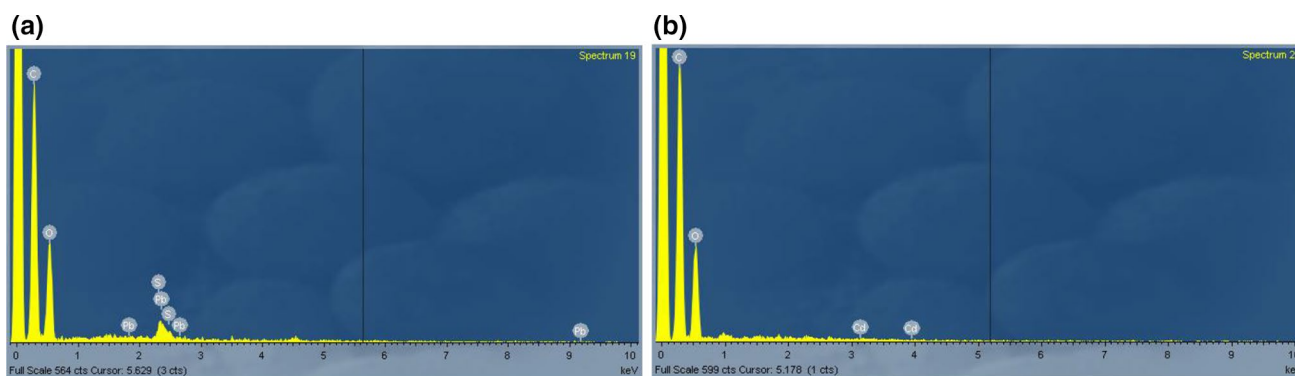

Fig. 2 EDX images of the **a** Pb(II)-adsorbed ASB-CBs, **b** Cd (II)-adsorbed ASB-CBs

V represents the solution volume (L), and m symbolizes the adsorbent dose used (g).

3 Results

3.1 Characterization of the biosorbents

The surface morphology of the biosorbents (ASB-CBs) before and after metal ions adsorption was studied using SEM analysis. The SEM images of ASB-CBs before and after metal uptake, as illustrated in Fig. 1a, b, display significant structure changes. The ASB-CBs surfaces are rough, dense and porous texture. However, after contact with Pb(II) and Cd (II), the porous texture disappeared and turn into smooth and tighter structure. The EDX images (Fig. 2a, b) show that Pb(II) and Cd (II) are well-adsorbed onto the composite materials.

The infrared spectrum analyzes of CBs, ASB-CBs, Pb-adsorbed ASB-CBs and Cd-adsorbed ASB-CBs all had widespread igniting scales at $4000 \div 3000 \text{ cm}^{-1}$ indicating
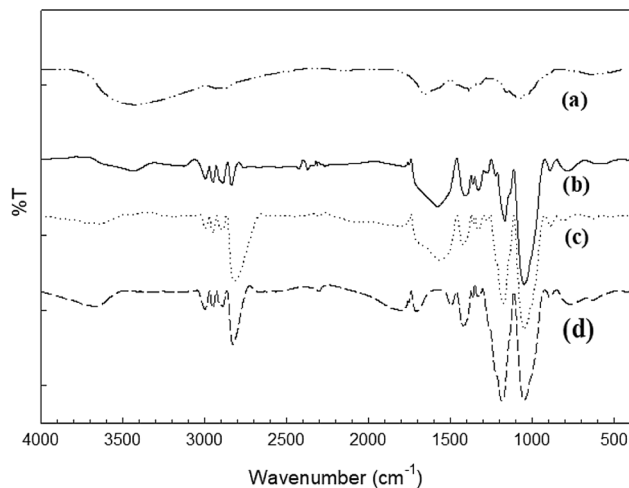

Fig. 3 FTIR spectra of the **a** CBs, **b** ASB-CBs, **c** Pb-adsorbed ASB-CBs and **d** Cd-adsorbed ASB-CBs

functional groups –OH, –NH$_2$, –COO and C–H aromatics. In particular, the presence of strong intensities at 1647.10 cm^{-1}, 1658.67 cm^{-1} (Fig. 3b) and 1642.31 cm^{-1}, 1639.59 cm^{-1} has a combined carboxylate functional group NH$_2$ [18]. The 1544.88 cm^{-1} spectrum corresponds to the quaternary amine group. Peaks at 1317.29 cm^{-1} and 1380.94 cm^{-1} in ASB-CBs showed C–H groups. The adsorption peaks have a weak intensity of 669.29 cm^{-1} and 570.89 cm^{-1} due to the metal oxide bonding of M–O [19]. Some FTIR spectral absorption bands after adsorption of metal ions decreased the intensity indicating that the shift to low-spectrum bands indicated that there was a linkage between the functional groups and the metal oxide as illustrated in Fig. 3c, d). Changes in the absorption spectra after metal ions adsorption showed the successful preparation of absorbable material. Functional substituent groups on the materials include the –OH, –CH$_2$, –CH$_3$, –COO, –NH$_2$, –COOH, –C–H, –CH$_2$.

3.2 Effect of pH

The pH of the solution significantly impacts the adsorption capacity, Fig. 4 illustrates the effect of the initial pH on the quantity of metal ions adsorbed onto ASB-CBs. The amount of removed metal ions increased with the increasing of pH from 3 to 5. The maximum adsorption capacity was found at pH 5.0 in which Pb(II) biosorbed is 11.79 mg g^{-1} and Cd (II) is 12.14 mg g^{-1} at initial metal ions concentration of 25 mg L^{-1}. Nevertheless, as the pH increments over 7.0, the biosorption volume reduces due to the precipitation of the metal hydroxide. As a result, pH 5.0 was chosen as the optimum pH for subsequent experiments. Also, many previous studies report pH 5.0–6.0 as the optimum pH for heavy metals adsorption onto biosorbents [4, 20–22].
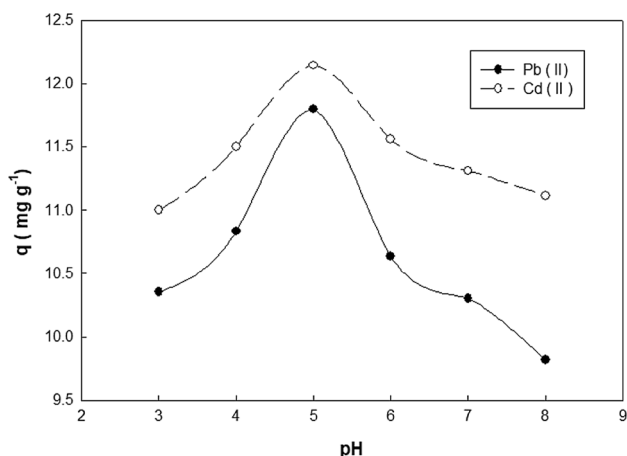
3.3 The biosorbent dose effect on metal ions biosorption

A significant parameter that highly affects the biosorption capacity is the dose of adsorbent. The adsorption tests were carried out with the range of ASB-CBs between 0.5 and 5 g L^{-1} under the same conditions, for instance temperature, pH, initial metal ions concentration and agitating time. The Fig. 5 illustrates the adsorption capacity effect of ASB-CBs. By increasing the absorbent dose, the amount of metal ions absorb on ASB-CBs decreases. Due that the adsorption sites have been saturated on the surface of the biosorbent when the dose is incremented, no more adsorption occurs after the equilibrium point [12].

3.4 Kinetic of adsorption process

In order to investigate the adsorption rate and the step which control the rate reaction, such as chemical reaction or mass transfer, kinetic models are used. The pseudo-first-order and the pseudo-second-order kinetic models were employed to determine the data obtained from the tests. The pseudo-first-order kinetic model equation is:

$$\ln \left(q_e - q_t \right) = \ln q_e - k_1 t \tag{3}$$

where k_1 is the first-order rate constant (1 min^{-1}) and t is the contact time (minutes). The pseudo-second-order kinetic model equation is:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e} \right) t \tag{4}$$

where k_2 is the second-order rate constant (g mg^{-1} min^{-1}).

Fig. 4 The effect of pH on the adsorption capacity of metal ions on the ASB-CBs (at 303 K) and an initial concentration of metal ions (25 mg L^{-1})
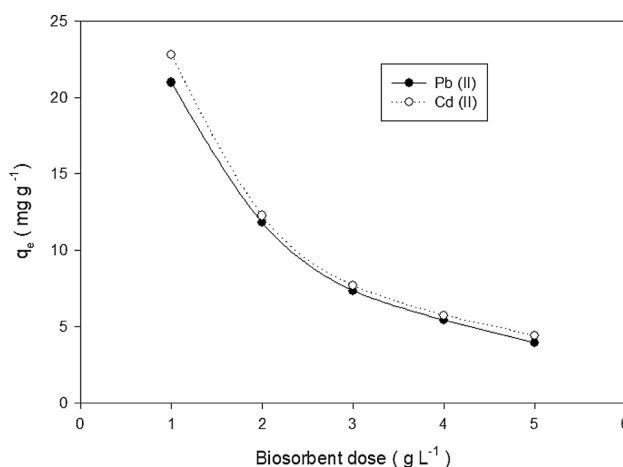

Fig. 5 Effect of adsorbent dose on the removal of metal ions on the ASB-CBs (at 303 K), pH 5.0, and an initial concentration of metal ions (25 mg L^{-1})
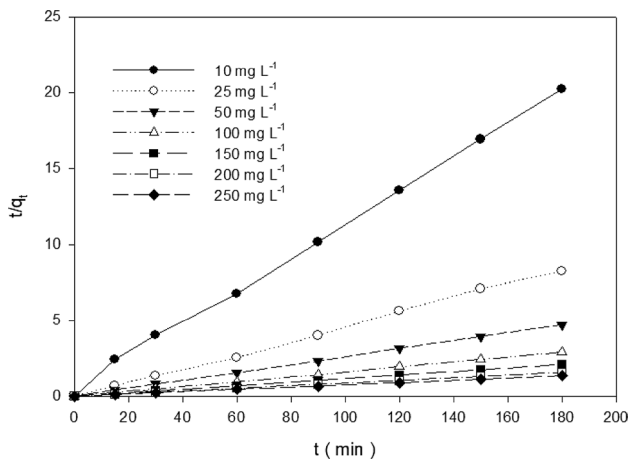
Fig. 6 Graph of the pseudo-second-order model for the adsorption of Pb(II) on the ASB-CBs (303 K), pH 5.0, and different initial concentrations of Pb(II)
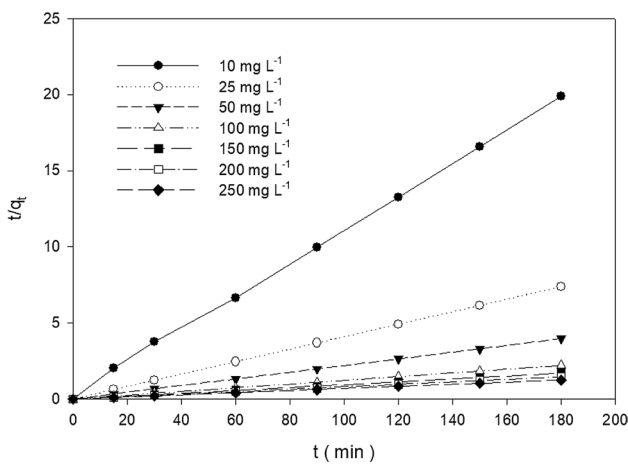

Fig. 7 Graph of the pseudo-second-order model for the adsorption of Cd (II) on the ASB-CBs (303 K), pH 5.0, and different initial concentrations of Cd (II)

The value of k_1 was estimated by the plot between ln $(q_e - q_t)$ and t (not shown), and the correlation coefficient (r^2) was also found. The plot of (t/q_t) versus t (Figs. 6, 7) was used to determine k_2 and r^2.

The kinetic parameters of the biosorption process for Pb(II) and Cd (II) are listed in Tables 1 and 2, correspondingly. The r^2 values in both metal ions adsorption clarified that the experimental data used, fitted with the pseudo-second-order model ($r^2 > 0.9995$). These results validate that the pseudo-second-order model can indicate that the rate-limiting step in metal ions biosorption on the ASB-CBs is the chemisorption.

3.5 Equilibrium modeling

The following equation described the linear form of the Freundlich model [23, 24].

$$\ln q_e = \ln K_F + (1/n) \ln C_e \qquad (5)$$

where K_F is a constant indicating the relative adsorption capacity of the biosorbent (mg$^{1-(1/n)}$ L$^{1/n}$ g^{-1}), and n is a constant indicating the adsorption intensity.

The values of K_F and n found from the graph of ln q_e and ln C_e at different temperatures are reported in Tables 3 and 4. The n value indicates the trend of the adsorption process; if $n = 1$, a linear adsorption occur; if $n < 1$, it indicates that is a chemical process; if $n > 1$, it indicates that is a physical process [25]. The obtained correlation coefficient r^2 is larger than 0.998, indicating the feasibility of the application of the Freundlich equation. In this research, the value of n is found between 0.7 and 0.8 that is almost equal to 1.0, demonstrating not only chemical but also physical process of metal ions biosorption on the ASB-CBs.

In order to reveal the adsorption behavior on the surface [16, 26, 27], the Langmuir isotherm model was applied. The linearized Langmuir equation is:

$$\frac{C_e}{q_e} = \left(\frac{1}{K_L \times q_{max}} \right) + \left(\frac{1}{q_{max}} \right) \times C_e \qquad (6)$$

where q_{max} is the maximum quantity of adsorption (mg g^{-1}), and K_L is the Langmuir constant (L mg^{-1}).

The values of K_L and q_m found from the graph C_e/q_e and C_e, listed in Tables 3 and 4, compares the experimental data with the ones fitted by the Langmuir equation (Eq. 6) at different temperatures.

Table 1 The kinetic parameters of the adsorption of Pb(II) on the ASB-CBs (at 303 K) and pH 5.0

C_0 (mg L^{-1})	Pseudo-first-order model		Pseudo-second-order model		
	k_1 (1 min^{-1})	r^2 (–)	k_2 (g mg^{-1} min)	q_e (mg g^{-1})	r^2 (–)
25	1.85	0.9856	0.0336 ± 0.0005	8.4 ± 0.6	0.9990
50	1.76	0.9452	0.0134 ± 0.0003	15.2 ± 1.5	0.9979
100	0.99	0.9234	0.0118 ± 0.0004	28.1 ± 1.7	0.9966
150	0.76	0.9080	0.0103 ± 0.0002	41.2 ± 2.1	0.9984
200	0.63	0.9452	0.010 ± 0.0002	52.1 ± 2.4	0.9995
250	0.52	0.9051	0.0051 ± 0.0001	56.4 ± 2.3	0.9990

Table 2 The kinetic parameters of the adsorption of Cd (II) on the ASB-CBs (at 303 K) and pH 5.0

C_0 (mg L^{-1})	Pseudo-first-order model		Pseudo-second-order model		
	k_1 (1 min^{-1})	r^2 (–)	k_2 (g mg^{-1} min)	q_e (mg g^{-1})	r^2 (–)
25	1.65	0.9243	0.04336±0.0005	12.4±0.6	0.9990
50	1.46	0.9532	0.0345±0.0003	22.0±1.5	0.9981
100	1.29	0.9183	0.0219±0.0004	39.3±1.7	0.9943
150	1.11	0.8993	0.0131±0.0002	45.2±2.1	0.9979
200	1.05	0.9012	0.0105±0.0002	59.1±2.4	0.9995
250	0.87	0.9192	0.0101±0.0001	63.4±2.3	0.9990

Table 3 The isotherm parameters of the biosorption process of Pb(II) on the ASB-CBs at pH 5.0

T (K)	Langmuir isotherm			Freundlich isotherm		
	K_L (L mg^{-1})	q_{max} (mg g^{-1})	r^2 (–)	K_F (mg$^{1-(1/n)}$ L$^{1/n}$ g^{-1})	n (–)	r^2 (–)
298	0.023	117.2	0.9313	3.475	0.349	0.9959
303	0.045	142.8	0.9019	4.188	0.461	0.9968
308	0.087	169.0	0.9512	5.961	0.733	0.9931
313	0.0117	185.1	0.9415	10.191	0.769	0.9980

Table 4 The isotherm parameters of the biosorption process of Cd (II) on the ASB-CBs at pH 5.0

T (K)	Langmuir isotherm			Freundlich isotherm		
	K_L (L mg^{-1})	q_{max} (mg g^{-1})	r^2 (–)	K_F (mg$^{1-(1/n)}$ L$^{1/n}$ g^{-1})	n (–)	r^2 (–)
298	0.037	76.9	0.9004	1.824	0.261	0.999
303	0.049	77.5	0.8901	1.447	0.1605	0.9971
308	0.064	78.1	0.9428	1.58	0.1987	0.9911
313	0.081	84.0	0.9518	2.078	0.3176	0.9982

$$q_e = \frac{q L_{emax}}{1 + K_L q_{max}} \tag{7}$$

The maximum amount of Pb(II) and Cd (II) adsorption are 185.18 mg g^{-1} and 84.03 mg g^{-1} at 313 K, respectively. The ASB-CBs in this study show improvement and promising performance for metal ions removal compare with other biosorbents in previous studies.

3.6 Thermodynamic

The Gibbs energy (ΔG) (kJ mol^{-1}), the enthalpy change (ΔH) (kJ mol^{-1}), and the entropy change (ΔS) (J mol^{-1} K) of an adsorption process can be estimated by [28]

$$\Delta G = -RT \ln K_L \tag{7}$$

where T is the absolute temperature (K) and R is the universal gas constant (8.314 J mol^{-1} K^{-1}). The values of ΔH and ΔS can be obtained from the slope and intercept of the graph of $\ln K_L$ against $1/T$ based on the van't Hoff equation

Table 5 The thermodynamic parameters of the biosorption of Pb(II) on the ASB-CBs at pH 5.0

T (K)	ΔG (kJ mol^{-1})	ΔH (kJ mol^{-1})	ΔS (J mol^{-1} K)
298	−5.67	19.49±0.9	111.1±8.9
303	−6.04		
308	−8.09		
313	−9.62		

$$\ln K_L = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \tag{8}$$

Tables 5 and 6 show the thermodynamic parameters of the adsorption process of Pb(II) and Cd (II) on the ASB-CBs. The negative ΔG values which indicated the metal ions adsorption on the ASB-CBs was spontaneous. The values of ΔH and ΔS were 19.49 kJ mol^{-1} and 111.1 J mol^{-1} K^{-1}, correspondingly in case of Pb(II); 15.9 kJ mol^{-1} and 110.1 J mol^{-1} K^{-1} in case of Cd (II). The positive enthalpy change (ΔH) reveals the endothermic nature. The entropy change value is positive, indicating

Table 6 The thermodynamic parameters of the biosorption of Cd (II) on the ASB-CBs at pH 5.0

T (K)	ΔG (kJ mol^{-1})	ΔH (kJ mol^{-1})	ΔS (J mol^{-1} K)
298	-7.47	15.9 ± 0.7	110.1 ± 7.8
303	-6.33		
308	-7.01		
313	-8.57		

increased randomness at the surface of the ASB-CBs during the process [29].

Compliance with ethical standards

Conflict of interest We hereby pledge that this study is a true scientific research of my own, conducted under my research group. The references in the manuscript are from scientific papers, books have been verified and published widely. We promise to quote the source clearly in the reference list. The research results in this project are done by the group itself strictly, honestly and not overlapping with other topics.

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