Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2022.28892

276 (2022) 133–141
November

A study on the ability of processed squeezed bitter almond for the removal of cadmium ions from contaminated water

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Received 19 February 2022; Accepted 3 September 2022

ABSTRACT

Heavy metal ions and organic compounds are the most common chemical pollutants that pollute water. Natural water is contaminated with Cd ions at relatively low concentrations and neutralizing Cd polluted water is not only cumbersome but also a prohibitively expensive process. Cd unlike most organic contaminants is difficult to remove because they accumulate in the tissues of living organisms, do not biodegrade, and may be toxic. Adsorption was the most dominant method of Cd ions removal from water due to its low cost, eco-friendliness, high efficiency, and ease of operation however its efficiency depends on absorbent. In the present study processed squeezed bitter almond (PSBA) is abundantly available and used as a bio-adsorbent. PSBA was procured from the local almond oil factor, characterized, and critically examined on its properties, adsorption capacities, isotherms, kinetics, and thermodynamics before its use in the experiment. The Freundlich isotherm model was used and found to be the best to capture the equilibrium data. The stated value for the Langmuir bio-adsorption capacity is 59.52 mg/g. To examine the kinetic behavior of the process, pseudo-first-order and pseudo-second-order kinetic models were examined. The data were seeming to be better described by the pseudo-second-order model. The Cd ions bio-adsorption process employing PSBA is exothermic and spontaneous, according to the thermodynamic analysis.

Keywords: Bitter almond; Water treatment; Adsorption; Cadmium ions

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1. Introduction

Our world is currently facing a water crisis due to a scarcity of water resources as well as water pollution. Water quality is a major concern because it has a significant impact on human and animal health. These critical environmental pollution issues are caused by industrial and agricultural activities, urbanization, and the massive increase in the population [1]. Organic compounds and heavy metal ions are the most common chemical pollutants that pollute water. Heavy

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metals, unlike most organic contaminants, are difficult to remove because they accumulate in the tissues of living organisms, do not biodegrade, and are highly toxic [2].

Cadmium (Cd) is extracted as a by-product from zinc ore or sulfur cadmium ore. It is widely used in rechargeable batteries, the production of special alloys, the paint industry as a pigment and plating, and as a plastic stabilizer. It is also found in tobacco smoke [3,4]. A human who has absorbed this metal through inhalation or ingestion will experience acute and chronic intoxications [5]. Through various exposure pathways, the maximum carcinogenic risk of Cd was suggested to be 10^{-7} ppb for the individual [6]. As a result, more emphasis is to be placed on removing heavy metals such as Cd ions from drinking water. Natural water is contaminated with Cd ions at relatively low concentrations and neutralizing Cd polluted water is not a cumbersome but also prohibitively expensive process. So finding efficient procedures for Cd ion removal is critical.

Amongst various methods of Cd ions removal from water such as ion exchange [7], chemical precipitation [8], membrane filtration [9], and biological remediation [10]. Adsorption was the most dominant method due to its low cost, eco-friendliness, high efficiency, ease of operation, high reactivity with adsorbate, flexible design, and regeneration [11–14]. On the other side, it would be very costly to treat large quantities of contaminated water and discharge the Cd ions concentration at relatively low concentrations [15]. However, the selection of a suitable adsorbent is again a critical issue for its successful application in the adsorption process. Various adsorbents, including natural materials such as clay [16], diatomite [17], zeolites [18], aluminosilicates [19], biochar [20], and activated carbon [21], have previously been used to remove Cd ions from contaminated water. In addition, agricultural wastes such as peat [22], Setaria grass [23], mussel shell [24], bean-coat [25], *Anacardium occidentale* L [26], rice husk, wheat straw, and corncob [27] are also being used as efficient adsorbents. However, the use of these alternative adsorbents for green, cost-effective, and energy-efficient pollutant removal from wastewater requires easy separation and recovery is limited.

Almonds are a well-known variety of nuts, from a well-known indigenous tree in South Asia and the Middle East [28]. For instance, in Jordan, the cultivated area was about 1,280 hectares [29], with about 88,734 almond trees in 2017 [30,31], while the almond crop production was 6,800 tons annually [29].

Depending on the kind of tree, they can be either bitter or sweet. A ripen, dried bitter almond seed (*Prunus dulcis var. amara*) is rich in oils, proteins, vitamins, amino acids, and other nutrients [32]. Almond oil is obtained by mechanical pressing [33] has a yellow color and a characteristic bitter taste and flavor and odor, which is attributed due to hydrogen cyanide and benzaldehyde, respectively [34,35].

Hydrogen cyanide and benzaldehyde are reported to have a beneficial effect on cancer patients at low doses [36,37], whereas a high dose can cause dizziness, heart palpitations, headache, and vomiting, and even lead to death [38,39]. The solid residual of almond after oil extraction containing benzaldehyde and hydrogen cyanide is often discarded as waste. To utilize this residual and alleviate environmental pollution, the proposed solution involves its uses as a bio-adsorbent for the removal of pollutants from aqueous solutions.

Based on the foregoing problems, the goal of this study is to investigate processed squeezed bitter almond (PSBA) as a promising alternative adsorbent for the removal of Cd ions from contaminated water, as well as to predict and compare adsorption performance using adsorption isotherm data modeling. Furthermore, investigate the effect of various parameters on maximum adsorption capacity, optimize adsorption mechanism pathways, express adsorbent capacities, and design adsorption systems.

2. Materials and methods

A stock solution of Cd ions (1,000 ppm) was prepared using cadmium nitrate ($Cd(NO_3)_2$) (Sigma-Aldrich) and nitric acid (HNO_3) (65%) from Riedel-de Haën (Puriss, Germany). For atomic absorption spectroscopy (AAS), a standard solution of Cd ions (1,000 ppm) from PanReac (Spain) was used AAS. The working solution pH was adjusted using 0.1 M HNO_3 and 0.2 M NaOH. PSBA's pH point of zero charge (pH_{PZC}) was determined using a Zeta-Meter 3.0 (Zeta-Meter Inc.). In this study, Milli-Q® deionized water was used.

PSBA was purchased from green fields factory (Jordan). Bitter almond was squeezed by a squeezing machine in factory to extract oil, what remained after the squeezing of bitter almond were used in the present work. PSBA samples were grinded and sieved to different particle sizes: <0.125, 0.125–0.212, 0.212–0.5, 0.5–1, and 1–2 mm.

2.1. Characterization of PSBA

The PSBA samples were first characterized in the laboratory at Taibah University. Using scanning electron microscopy (SEM) NOVA NANO (Thermo Fisher, USA) was for morphology, and an energy-dispersive X-ray spectroscopy (EDS) Superscan SSX-550 (Shimadzu, Japan) to identify the elements present in the samples. The PSBA samples were also characterized using Fourier-transform infrared spectroscopy (FTIR) Invenio (Bruker, Germany) and it's surface area was determined using Brunauer–Emmett–Teller (BET) (Micromeritics ASAP 2020, USA).

2.2. Cd ions removal experiments

The parameters that affect the removal of Cd ions from aqueous solutions were studied by batch experiments. Thermostat shaker incubator was used to control the temperature and shaking speed while removal of Cd. Several doses of PSBA with particle diameters of ranges 0.125–0.212, 0.212–0.5, 0.5–1.0 and 1.0–2.0 mm were mixed with 50.0 mL of certain concentration and pH of Cd ions solution. After a definite contact time between adsorbent and adsorbate, the exact volume of Cd ions solution was withdrawn then diluted to a certain volume with 2% v/v nitric acid to be in the range of AAS calibration curve.

2.3. Adsorption isothe+rm models

Langmuir and Freundlich, the two isothermal models were established to study the distribution of Cd ions between the adsorbent surface and solution at equilibrium [40].

2.3.1. Langmuir isotherm model

The Langmuir isotherm is represented by the following linear equation:

$$\frac{C_e}{Q_e} = \frac{1}{X_m K} + \frac{1}{X_m} C_e \tag{1}$$

where C_e (mg/L) is the concentration value of the adsorbed material at equilibrium; Q_e (mg/g) is the quantity of metal ions adsorbed per gram of adsorbent; K and X_m are the Langmuir equation constants representing the sorption rate and sorption capacity, respectively [41].

2.3.2. Freundlich isotherm model

The Freundlich model linear equation [42] is as follows:

$$\log Q_e = \log K_F + \frac{1}{n} \log C_e \tag{2}$$

where n and K_F stand for Freundlich equation constants, the value of n in the equation indicates how the sorption process is favorable, and K_F value represents sorption capacity of the adsorbent.

3. Results and discussions

3.1. PSBA characterization

3.1.1. FTIR spectra

Two PSBA samples were characterized by FTIR to find out the functional groups that participated in Cd ions adsorption. One sample was analyzed before Cd ions adsorption and the second one after that. Fig. 1 illustrates the functional groups that are present in the samples. It is shown that the broad bands between 3,260–3,500 cm^{-1} which indicates the presence of stretching vibration bands of the O–H bond of hydroxyl groups that accompanying with the N–H bond of amino groups [43,44].

Stretching vibrations bands of aliphatic CH_3 and CH_2 presented at two strong sharp peaks at 2,926 and 2,855 cm^{-1}, respectively. It is well known that the strong sharp peak at 1,740 cm^{-1} stands for carbonyl group, and the peak at 1,660 cm^{-1} indicates the presence of alkene C=C. Furthermore, the results show that there was a minor difference in the hydroxyl and amino groups bands after adsorption. This might be attributed to the binding of Cd ions to hydroxyl and amino groups.

3.1.2. SEM, EDS and surface area (BET) characterizations

BET analysis results showed a relatively small surface area for the used adsorbent with 2.127 m^2/g. Characterization of surface morphology using SEM (Fig. 2) indicated that PSBA surface has low porosity and rough surface which synchronizes with surface area.

EDS analysis results exhibited that PSBA is composed of oxygen, carbon, and nitrogen elements which harmonized with FTIR analysis spectra. There also presents a relatively small amount of potassium element (Table 1).

3.2. Factors affecting Cd ions removal

3.2.1. Contact time

Fig. 3 shows the percentage of Cd ions removal using PSBA vs. contact time. The PSBA possess the ability to remove Cd ions proficiently. Within a short period of time

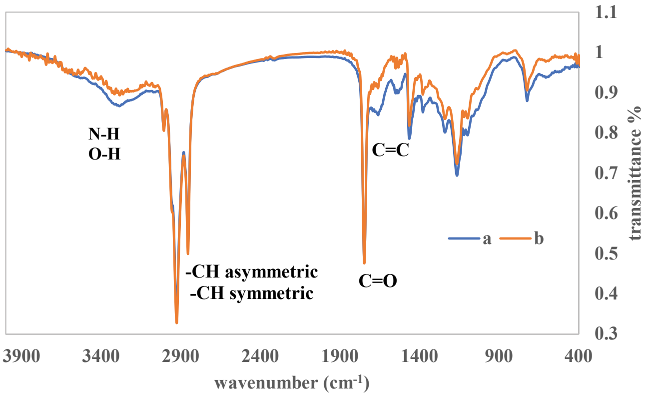

Fig. 1. FTIR spectra of PSBA: before Cd ions adsorption (a) and after Cd ions adsorption (b).

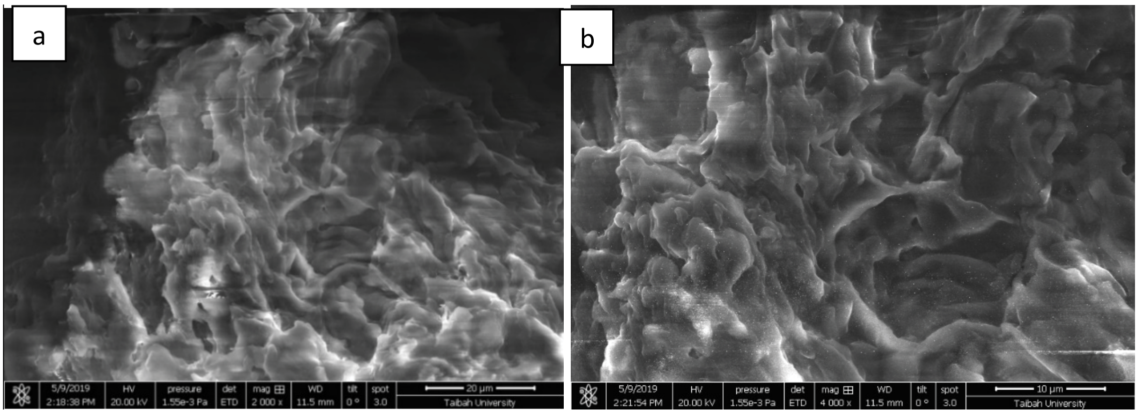

Fig. 2. Scanning electron microscopy image of PSBA powder; scale bar, 20 μm (a) and 10 μm (b).

Table 1
Energy-dispersive X-ray spectroscopy results of PSBA

Element	Weight %	Atom %
C	37.96	45.18
N	9.60	9.80
O	48.97	43.75
K	3.47	1.27
Total	100.00	100.00

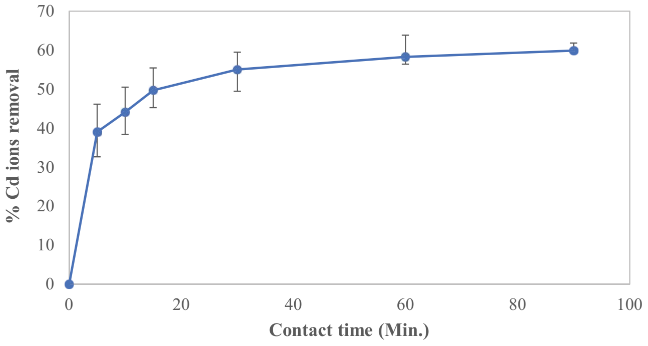

Fig. 3. Study of contact time parameter on Cd ions removal (mean ± RSD) (50 mL, 50 ppm) using PSBA (0.2 g, 0.212–0.5 mm) (120 rpm, pH 5, 25°C).

30 min, the percentage of Cd ions removal reached approximately 60%. After 30 min of contact time, there was no significant change in the percentage of Cd ions removal observed.

3.2.2. Effect of Cd ions solution pH

The pH range in this study was limited to 2 to 5, as higher pH values may result in metal hydroxide formation, especially at high metal concentrations. PSBA at pH values 4 and 5, resulted in a high percentage removal for Cd ions (60%). However, using lower pH values resulted in a significant decrease in Cd ions removal (Fig. 4).

The pH_{PZC} of PSBA is about 3.6 which indicates that the PSBA's surface was positively charged at solution pH below 3.6, and negatively charged at solution pH above 3.6. This observation of the present study might help in understanding the possible mechanism of the adsorption process. The maximum adsorption was achieved at pH 5, and a negative charge is present on the surface of PSBA, causing better Cd cations adsorption through the electrostatic attraction. The higher the pH than pH_{PZC}, the greater the density of negative ions on the surface of PSBA which will permit more adsorption. The lower the pH than pH_{PZC}, means that the PSBA's surface was positively charged. This causes competition between H^+ ions and Cd cations for adsorption locations as well as the repulsion of Cd cations, resulting in the reduction of the adsorption process.

3.2.3. Effect of PSBA dose

Results of adsorption capacity show that a 0.2 g dose of PSBA exhibited the highest performance compared to higher and lower doses with 7.49 mg·Cd/g PSBA (Fig. 5). It can be concluded that it is the optimum dose for the experimental conditions of the batch experiment.

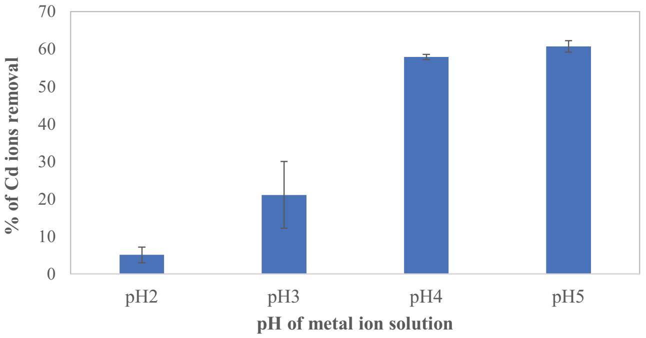

Fig. 4. Effect of adsorbate solution pH on Cd ions removal (mean ± RSD) (50 mL, 50 ppm) using PSBA (0.2 g, 0.212–0.50 mm) (120 rpm, 60 min, 25°C).

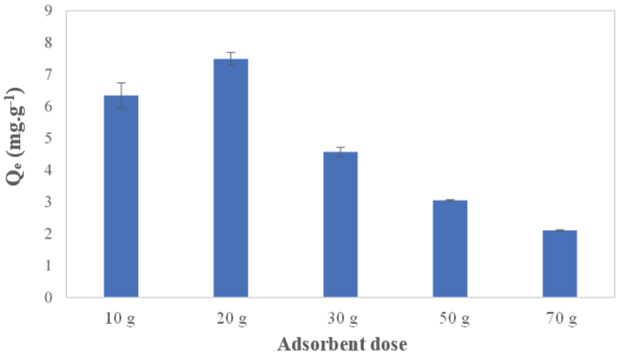

Fig. 5. Effect of PSBA dose (0.212–0.50 mm) on Cd ions removal (mean ± RSD) (50 mL, 50 ppm, pH 5) (120 rpm, 60 min, 25°C).

3.2.4. Effect of PSBA particle size

The adsorption capacity results depicted in Fig. 6 shows that the particle size range (0.212–0.500 mm) is considered the optimum particle size with an adsorption capacity of 7.6 mg·Cd/g PSBA, however, no significant difference between higher and lower diameter were observed, except for the largest size of 1.0–2.0 mm which showed a sharp decrease in adsorption capacity that comes from smaller surface area in contact to Cd ions solution compared to other studied PSBA particle sizes.

3.3. Adsorption isotherm study

Langmuir and Freundlich isothermal models used in this study are presented in Fig. 7 and Table 2. The modeling results demonstrated that the data of Cd ions adsorption on the surface of PSBA fit to the linear Freundlich isotherm model better than the non-linear Langmuir model, at a higher correlation coefficient (R^2). This implies that Cd ions adsorption on PSBA is heterogeneous multilayer adsorption. Furthermore, the $1/n$ value is less than one (0.7624), indicating that the adsorption process is favorable

[45,46]. Furthermore, according to X_m value in (Table 2), the adsorption capacity of PSBA for Cd ions is 59.52 mg/g.

3.4. Adsorption kinetics studies

Pseudo-first and second-order kinetic models were applied in order to figure out the mechanism of sorption kinetics of Cd ions on PSBA surface is either the physical or chemical mechanism [47,48]. The following equation expresses the linearized form of pseudo-first-order kinetic model:

$$\log\left(Q_e - Q_t\right) = \log Q_e - \frac{k_1 t}{2.303} \tag{3}$$

where Q_t is the adsorption capacity at a certain time t (g/g) and k_1 is the pseudo-first-order rate constant (h^{-1}). Calculated $Q_{e,\text{calc}}$ and k_1 values were obtained from the slope and intercept of the plot of log (Q_e–Q_t) vs. time, respectively, in (Fig. 8a).

Eq. (4) expresses the linearized form of pseudo-second-order kinetic model:

$$\frac{t}{Q_t} = \frac{1}{\left(K_2 Q_e^2\right)} + \left(\frac{t}{Q_e}\right) \tag{4}$$

where k_2 is the pseudo-second-order rate constant of adsorption (g/mg·min). $Q_{e,\text{calc}}$ and k_2 represents the slope and intercept of the plot t/Q_t vs. time (t), respectively, (Fig. 8b).

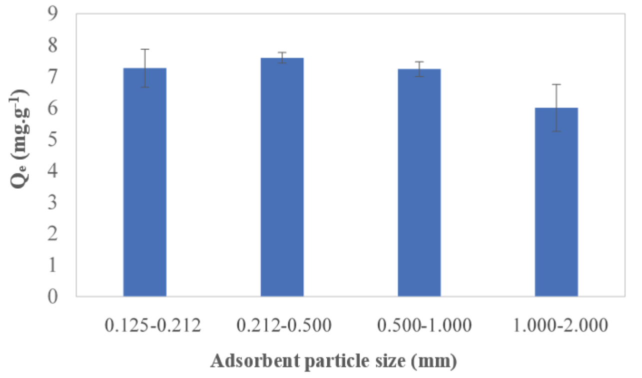

Fig. 6. Effect of PSBA particle size (0.20 g) on Cd ions removal (mean ± RSD) (50 mL, 50 ppm, pH 5) (120 rpm, 60 min, 25°C).

The results of both (Fig. 8) and (Table 3) illustrate that pseudo-second-order kinetic model had a higher correlation coefficient (R^2) value and a lower difference between $Q_{e,\text{calc}}$ and $Q_{e,\text{exp}}$ as compared to pseudo-first-order model. Accordingly, it can be concluded that the adsorption of Cd ions fits pseudo-second-order kinetic model. Consequently, this may also mean that adsorption on PSBA is chemisorption [49]. However, the chemisorption is a relative rate limiting step for Cd ions uptake onto PSBA; this is probably due to the valence forces involved through an exchange of electrons or sharing between Cd ions and composite binding sites [50].

3.5. Adsorption thermodynamic studies

Values of adsorption thermodynamic parameters (ΔG, ΔH, ΔS) were obtained employing van't Hoff equation. Fig. 9b shows a plot of log(Q_e/C_e) vs. ($1/T$). The values of enthalpy (ΔH) and entropy (ΔS) were calculated from the slope of the plot and the intercept, respectively.

The thermodynamic data for the removal of Cd ions by PSBA (Table 4) represents that as the temperature increased, the (ΔG) value also increased. This represents reaction spontaneity decreases as temperature increases. Moreover, data in Table 4 shows that enthalpy (ΔH) had a negative value which indicates that adsorption of Cd ions is an exothermic process. Additionally, entropy ΔS had a negative value reflecting the decreasing entropy, this reveals a decrease in the degree of freedom of adsorption of Cd ions onto PSBA. One explanation is that the molecules before adsorption can move in three dimensions but as they get adsorbed on the surface, the motion of molecules is restricted toward

Table 2
Langmuir and Freundlich isotherm constants for the adsorption of Cd ions

Langmuir isotherm model		
X_m	K	R^2
59.52	6.56×10^{-3}	0.3529
Freundlich isotherm model		
K_F	$1/n$	R^2
0.738	0.7624	0.8949

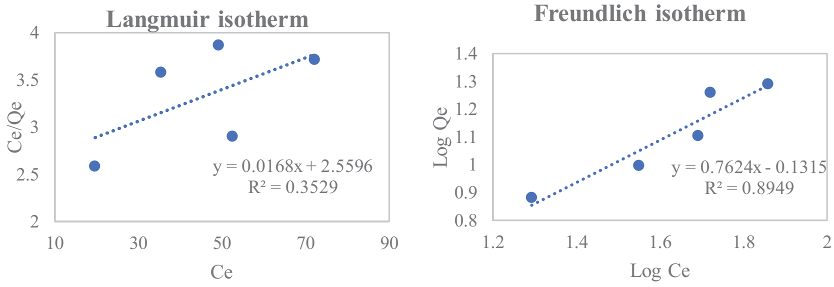

Fig. 7. Langmuir and Freundlich isotherms of Cd ions adsorption, 0.20 g PSBA, 25–150 mg/L initial concentrations (50 mL, pH 4, 120 rpm, 60 min, 25°C).

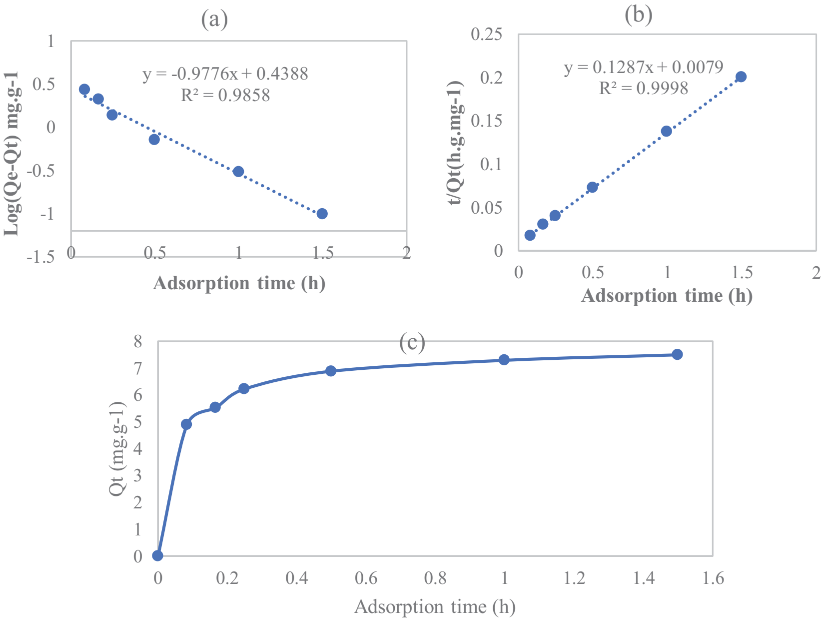

Fig. 8. Pseudo-first-order kinetic model (a), pseudo-second-order kinetic model (b), and effect of time on the adsorption capacity of Cd ions on PSBA (c).

the surface and their disorderness decreased resulting in the decrease in entropy [51]. Similar results were also observed by Karaoglu et al. [52], Iqbal and Ashiq [53], and Purkait et al. [54], and consistent with the kinetics studies which approved that the adsorption process on PSBA is chemisorption. Therefore, the process is irreversible [55], the adsorption mechanism involves valence forces and chemical bonds [56]. Thus the Cd ions once adsorbed, they can neither diffuse along nor desorb from the PSBA surface, they are strongly bonded.

Table 3
Pseudo-first-order and pseudo-second-order kinetic models for the adsorption of Cd ions on PSBA

Pseudo-first-order kinetic model			
$Q_{e,\exp}$ (mg/g)	$Q_{e,\text{calc}}$ (mg/g)	k_1 (h^{-1})	R^2
7.50	2.74	2.25	0.9833
Pseudo-second-order kinetic model			
$Q_{e,\exp}$ (mg/g)	$Q_{e,\text{calc}}$ (mg/g)	k_2 (g/mg·h)	R^2
7.50	7.77	2.09	0.9996

$$\log\left(\frac{Q_e}{C_e}\right) = -\frac{\Delta H}{2.303RT} + \frac{\Delta S}{2.303R} \tag{5}$$

Table 4
Thermodynamic data for the removal of Cd ions by PSBA

Metal ions	Temperature (K)	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (J/mol)	R^2
Cd	298	2.259	–9.33	–38.89	0.8386
	308	2.648			
	318	3.037			

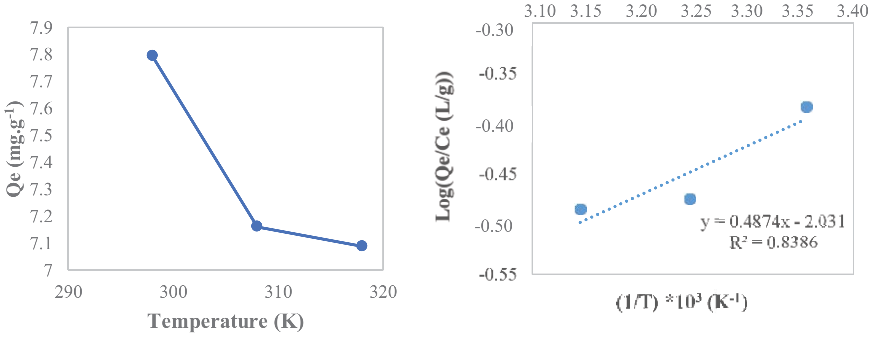

Fig. 9. Adsorption capacity of PSBA vs. temperature (a) and van't Hoff plot (b).

Table 5
Comparison of various adsorbents used to adsorb Cd ions from aqueous solutions

Adsorbents	Maximum capacity (mg/g)	References
Starch modified montmorillonite	4.2	[57]
Cladophora biomass	12.07	[58]
Orange peel	63.35	[59]
Corncobs	8.99	[60]
Rice husk	16.7	[61]
Sugar beet pulp	24.39	[62]
Banana peel	35.52	[63]
Pine bark	14.16	[64]
Olive mill solid residue	4.525	[65]
NImp-Alg-25 wt.% CS, Imp-Alg-25 wt.% CS	15.62, 22.24	[66]
PSBA	59.52	Present work

In the present study, PSBA showed high performance for Cd ions removal compared to other studied adsorbents according to data in Table 5.

4. Conclusion

PSBA was a low-cost, bio-adsorbent used in this study to remove Cd ions from an aqueous solution. FTIR and EDS analysis confirmed PSBA's can play the role of an adsorbent. The maximum removal capacities for investigated PSBA were achieved with 0.2 g of PSBA and particle size of 0.212–0.500 mm after 30 min of contact time. The adsorption of Cd ions was affected by pH, pH of 5 provides the highest efficiency.

The equilibrium data fits best into Freundlich adsorption isotherm which shows the heterogeneity of adsorbent multilayers. All n values were greater than one, indicating favorable adsorption of Cd ions. In addition, the adsorption data fitted best to a pseudo-second-order kinetic model, adsorption process assumed chemisorption. Thermodynamic parameters indicated that the adsorption process is spontaneous and exothermic in nature.

PSBA is a natural, green, renewable, and environmentally friendly resource. It could be an alternative to synthetic, non-degradable adsorbents that use expensive technologies to build, use and dispose of them off.

Acknowledgment

The Authors extend their appreciation to the Dean of Scientific Research at Imam Mohammad Ibn Saud Islamic University for funding this work through research group no. RG-21-09-81. In addition, we thank both Jerash University and Isra University.

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