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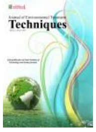

Efficient Cd (II) Ions Removal from Aqueous Solution Using Peganum Harmala and Prosopis Farcta Seed as Adsorbents

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Abstract

Cadmium is a highly toxic heavy metal and a dangerous pollutant to the environment. Anthropogenic activities are the main reasons for diffusing cadmium into water resources, soil, and air. In this study, the adsorption of cadmium from the aqueous phase has been investigated by the usage of biological adsorbents including Prosopis farcta and Peganum harmala seeds powder. The adsorption procedure was optimized in four steps: initial pH, contact time, adsorbent dosage, and initial concentration of cadmium. The cadmium concentration in solution was measured by the Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) method. The results of optimization tests showed 91% and 73% Cd (II) removal for pH = 6.5 and initial metal concentration of 20 mg/L for 20 g/L of Prosopis farcta at 45 min and 40 g/L of Peganum harmala at 30 min, respectively. In addition, the efficiency of the adsorption process improved when the cadmium concentration decreased in the initial solution. Finally, the adsorption process for both adsorbents complied with Freundlich isotherm and the pseudo-second-order kinetic.

Keywords: Adsorption, Cadmium contamination, Prosopis farcta, Peganum harmala, Isotherm, Kinetic models

1 Introduction

Currently, one of the most significant environmental problems in industrialized nations is the attendance of noxious heavy metals in the environment, especially in aqueous streams. The discharge of untreated industrial effluents containing metallic ions into the water bodies leads to this problem [1]. Owing to the ability of bioaccumulation in live tissues and the formation of non-biodegradable compounds, heavy metals can pose numerous environmental and human health risks [2]. Cadmium (Cd), as one of the most toxic heavy metals, exists in various industrial sources such as electroplating, paint pigments, alloy preparation, plastic manufacturing, ceramics, mineral processing, and battery manufacturing [3]. Cadmium exposure may cause both chronic and acute adverse disorders such as hypertension, anemia, cancer, lung insufficiency, testicular atrophy, bone degradation, Itai-Itai, liver and kidney damage, etc. [4,5]. The World Health Organization declared the maximum allowable concentration of cadmium in drinking water at 3 μg/L [6].

To eliminate such hazardous materials, many remediation strategies, based on physicochemical principles, have been recommended by researchers. Some of the most prevalent methods of heavy metal removal are ionic exchange, chemical precipitation, evaporation, electroplating, membrane filtration, and adsorption techniques [7,8]. These methods are expensive when cadmium is present in the wastewater at low concentrations, or when a very low concentration of cadmium in treated water is required. In addition, incomplete metal removal and sludge generation are the other disadvantages of these methods [9]. Thus, more cost-effective and environmentally benign technologies for metal remediation are still in demand. Bioremediation is one of the appropriate procedures for the removal of heavy metals from an aqueous solution. Bioremediation can be defined as the use of living organisms (plants, fungi, bacteria, etc.) or inactive, non-living biomass (biomass, inanimate tissues of animals, seeds, leaf, and wood) for adsorbing heavy metals from water solution. The mechanisms of uptake by living materials (bioaccumulation) and removal by non-living biomasses (biosorption) are entirely different. It seems that biosorption (use of dead materials) rather than bioaccumulation (use of living materials) has several advantages because there is no need for growing, no growth media is required and these materials are available as wastes or by-products. In addition, biosorbents can adsorb an extensive range of heavy metals with satisfactory performance, especially when the concentration of heavy metals in the solution is low. Selecting a proper, low-cost biosorbent with numerous metal-binding sites is the main stage in this procedure [10-12].

Different kinds of natural adsorbents have been used for the remediation of heavy metals [13-16]. Some examples including Alhajimaurorum seed [17], green coconut shell powder [18].

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Crataegus oxyacantha stone and Punica granatum seed [19], brown marine microalgae [20], leaves of Platanus orientalis plant [21], black gram husk [22], olive cake [23], banana peel [24], different species of seaweeds [25], and sugar cane bagasse [26], were used for adsorbing cadmium from aqueous solutions.

Prosopis farcta, also known as Syrian mesquite, is a species of the genus Prosopis. This perennial plant is widely distributed in North African areas, the West of the Middle East, Southwest of Asia, and the United States. It can easily endure hot and dry weather conditions or even years of drought and alkaline soil can't affect the resistance of this plant against the salt. It should be noted that this plant is considered to be a kind of invasive weed that is hard to eradicate. Important chemical components of the plant contain toxin, quercetin, tryptamine, lectin, alkaloids, and l-arabinose. The plant is being used as a folk medical treatment to cure cardiac or chest pain, colds, prostate and skin disorders. Additionally, it has other properties: anticancer, antidiabetic, antioxidant, antibacterial, antihyperlipidemic, cardioprotective, menstruation pain relief, energy-producing, fertility, wound healing, nano-particle synthesis, and hepatoprotective [27,28], Seeds of the plant are located in a dark brown, spongy sheath. They have soft cores with a yellowish-brown hard coating surrounding a very smooth surface [29,30].

Peganum harmala, commonly called Alharmal, is from the family Nitrariaceae and native to the Middle East and North Africa. This perennial, herbaceous and glabrous plant grows abundantly in semiarid and steppe areas and generally as wild species [31], Different parts of it have medicinal properties, although this plant is considered as a toxic and invasive plant. Several pharmacological effects have been reported for this plant including anti-inflammatory, anti-histaminic, antimalarial and insecticidal, anti-spasmodic, antileishmanial, anti-tumor, antibacterial, antiviral, anti-ulcer, antioxidant, antifungal, wound healing, and vasorelaxant effects [32]. The main phytochemical compounds identified in the Peganum harmala seeds are alkaloids such as harmaline ($C_{13}H_{14}N_2O$)(48%), harmine ($C_{13}H_{12}N_2O$)(38.4%), tetrahydroharmine ($C_{13}H_{16}N_2O$)(8.5%), (z)-octa-9-decenamide ($C_{18}H_{35}NO$)(1.02%) as well as derivatives of quinazoline [33]. The performance evaluation of Prosopis farcta and Peganum harmala seeds as bio-adsorbents to remove cadmium from aqueous solutions is the aim of the current study. The effects of four parameters including pH, adsorption time, amount of adsorbent, and the initial concentration of cadmium in solution were studied and optimized. Laboratory results conformity to isotherm models, Langmuir, Freundlich, and D-R, was investigated, and finally adsorption kinetics of the pseudo-first and pseudo-second-order for cadmium adsorption procedure by both adsorbents were analyzed.

2 Materials and methods

2.1 Materials

Prosopis farcta and Peganum harmala seeds were collected from the suburbs of Damghan, a city in Semnan Province, Iran. Separated seeds of the plants were washed with distilled water twice to purge the impurities. Then, they were dried at room temperature for 48 hours. To obtain the powder with a particle size less than 1 mm, the dried seeds were ground by a mortar grinder (RM 200, Retsch) and screened through a standard sieve no 18. Finally, powder of both Prosopis farcta and Peganum harmala seeds (PPFS and PPGS) was kept at room temperature available for the tests.

To prepare solutions with the required Cd (II) concentration, a stock solution of cadmium (100 mg/L) was diluted. This solution was obtained by dissolving cadmium nitrate ($Cd(NO_3)_2{\cdot}4H_2O$) with a purity of 95% (E. Merck) in deionized water. The pH was adjusted by either 0.1 M HNO_3 or 0.1 M NaOH solution and measured by a digital pH meter (YK-2001CT, Lutron).

2.2 Experimentation methods

The solutions were placed in a mechanical shaker for desired periods and agitated at 120 rpm. After each run, the sample was filtered through filter paper (Whatman, 0.45 μm) and ultimately, the cadmium concentration of the filtered sample was measured by ICP-OES (GBC Company, Australia).

All experiments were carried out three times and the final experimental data were reached by an average of them (triplicate experiment). The effects of four predominant parameters were examined. Initially, the performance of the process under various pH values (2, 3, 4, 5, 6, 7, and 8) was investigated for both adsorbents. It should be pointed out that the effect of pH for values higher than 8 was not studied, since the condition leads to precipitation of cadmium ions in the form of hydroxide compounds (Namasivayam and Ranganathan 1995). At the second step, the effect of contact time (5, 10, 15, 30, 45, 60, and 120 minutes) was scrutinized. Continuing the experiments, different samples with the mixture of 0.125, 0.25, 0.5, 0.75, 1, 1.5, 2, and 2.5 g of the adsorbents and 50 mL of the Cd solution (2.5, 5, 10, 15, 20, 30, 40 and 50 g/L) were tested to discover the adsorbent dose impact on the operation. Eventually, the effect of the initial concentration of cadmium was explored by varying its values (0.1, 0.5, 1, 10, 20, 40, 60, 80, and 100 mg/L). At each step, there was only one variable and the other factors were maintained at constant conditions.

The amount of cadmium ions adsorbed by the adsorbent, q_e (mg/g), and removal percentage (%) of the cadmium were calculated via equations 1 and 2 [34]:

$$q_e = \frac{V\,(C_i - C_e)}{m} \tag{1}$$

$$\mathrm{Re}\,moval\,(\%) = \frac{C_i - C_e}{C_i} \times 100 \tag{2}$$

where V is the volume of solution (L), C_i and C_e are the initial and equilibrium concentrations (mg/L), respectively and m represents adsorbent content (g).

2.3 Adsorption kinetics

Chemical kinetics expresses the reaction rate until the equilibrium is reached, whereas chemical equilibrium does not give any information about the sorption reaction pathways during the time. Sorption kinetics largely depends on physiochemical properties of the sorbent material and experimental conditions. The prediction of kinetic models performs a fundamental role in designing efficient adsorption systems. Reaction progress and sorption mechanism can be described via various sorption models [35,36]. The pseudo-first and pseudo-second order [37], kinetics are two types of well-known models represented in equations 3 and 4 in linear forms, respectively:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \tag{3}$$

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

where q_e and q_t are the amounts adsorbed (mg/g) at equilibrium and at time t (min). Also, k_1 (1/min) and k_2 (g/(mg.min)) present the rate constants of the first and second-order equilibriums, respectively. Using the experimental data, acquired for different contact times, plots of log(q_e-q_t) and t/q_t against t were drawn. The constant parameters of both kinetic models were determined and correlation coefficients, R^2, were calculated for laboratory conditions according to the plots [38,39].

2.4 Adsorption isotherms

Adsorption isotherms can interpret the adsorption equilibrium data and clarify the specific relationship between the absorbed amount of the adsorbate and its concentration in the solution at equilibrium. Normally, three known isotherms of Langmuir [40, 41], Freundlich [42], and Dobinin–Radushkevich (DR) [43], are employed to explain the adsorption phenomenon. Linear forms of Langmuir, Freundlich, and DR relations are represented as equations 5, 6, 7:

$$\frac{c_e}{q_e} = \frac{1}{bq_{\max}} + \frac{c_e}{q_{\max}} \tag{5}$$

$$Lnq_e = Lnk_f + \frac{1}{n} Lnc_e \tag{6}$$

$$Lnq_e = Lnq_{\max} - k_{DR}\varepsilon^2 \tag{7}$$

$$\varepsilon = RTLn(1 + \frac{1}{c_e}) \tag{8}$$

where c_e is the equilibrium concentration of metal ions in aqueous solution (mg/L), q_e is the amount of cadmium adsorbed at equilibrium (mg/g), q_{max} is the sorption capacity of the sorbent (mg/g). b defines the Langmuir isotherm constant (L/mg), n (dimensionless) and k_f ((mg/g)/(mg/L)^n) are the Freundlich isotherm constants and k_{DR} is the DR isotherm constant (mol^2/J^2). ε, named Polanyi potential, is calculated via equation 8. T is the absolute temperature (K), and R is the gas constant (J/(mol.K)) [44, 45]. The essential characteristic and further analysis of Langmuir isotherm can be acquired in terms of R_L, a dimensionless constant, generally known as separation parameter. It indicates the isotherm shape and predicts the desirability of the adsorption system. According to equation 9, R_L values, between 0 and 1, signify favorable adsorption, while $R_L>1$ represents an unfavorable adsorption rate. c_0 in the equation defines the initial solute concentration (mg/L) [46].

Dimensionless parameter n, in Freundlich isotherm (equation 6), specifies the distribution of ions adsorbed onto the adsorbent surface. If it lies between 1 and 10, the adsorption process is beneficial, but as n is approaching 1, it demonstrates that heterogeneity of the adsorbent surface is less important [10].

Another factor in Dobinin–Radushkevich isotherm, calculated according to equation 10, determines the type of adsorption mechanism. Mean biosorption energy parameter (E) for values less than 8 kJ/mol introduces physical multilayer process, and for values between 8 and 16 kJ/mol implies chemical monolayer adsorption dominance [47].

$$R_L = \frac{1}{1 + bc_0} \tag{9}$$

$$E = \frac{1}{\sqrt{2k_{DR}}} \tag{10}$$

3 Results and discussion

3.1 The Effect of pH

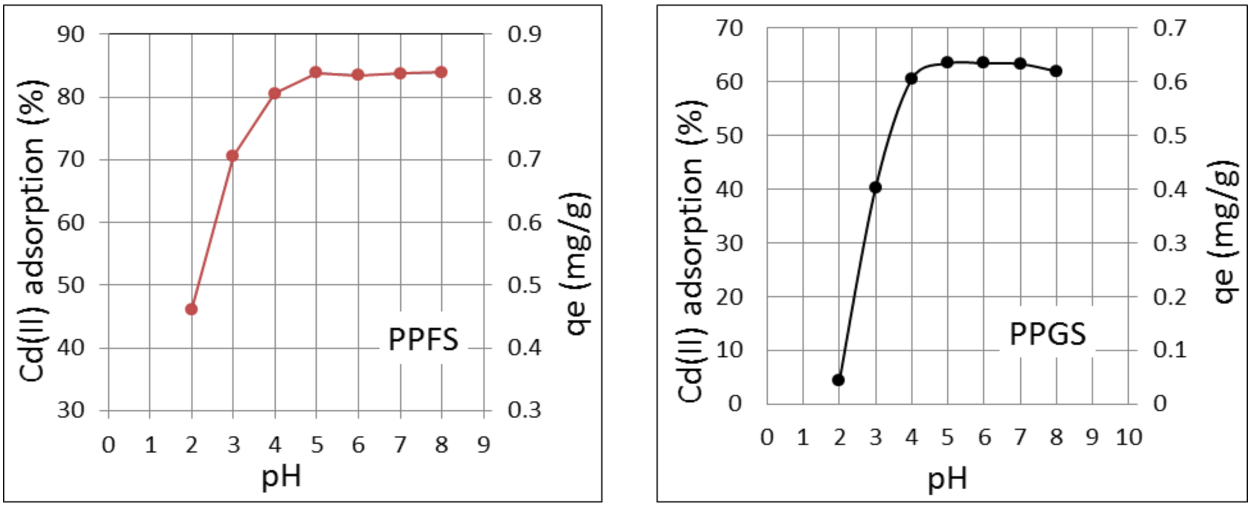
The solution pH can strongly affect the sorption process. As figure 1 illustrates, the cadmium uptake increased when pH was raised, and the highest adsorption rate occurred at pH = 5 which continued to pH almost equal to 7. The maximum rate of adsorption for PPFS and PPGS reached 84% and 63%, respectively. In this research, pH equal to 6.5, which is in the normal range for pH of water, was considered as the optimum pH for cadmium adsorption from the aqueous phase by both PPFS and PPGS adsorbents. In acidic pH, a competition appears between Cd (II) cations and H^+ or H_3O^+ ions for negative binding sites on the adsorbent surface which is the reason for the lower adsorption rate. By shifting toward an alkaline medium, H^+ and H_3O^+ ions decrease in solution and metal cations find the opportunity to occupy the negative ligands of the sorbent until saturation occurs. Furthermore, in acidic pHs, H^+ causes electrostatic repulsive forces on the adsorbent surface which can prevent the adsorption of positive cadmium ions [48].

3.2 The effect of contact time

The effect of contact time on the removal of Cd (II) was studied according to figure 2. As it can be seen, the biosorption capacity of cadmium by both adsorbents increased with contact time. The slope of the plots indicates rapid adsorption at first minutes until it gained its maximum level. These values of adsorption for PPFS and PPGS were obtained at 91% for the time of 45 min and 63% for the time of 30 min respectively, and thereafter adsorption process was roughly stabilized. This behavior can be elucidated by further vacant sites available on the surface area of the adsorbent in the beginning. In a prolonged time, adsorption sites are exhausted, and hence the removal of sorbate approaches equilibrium [49]. The PPFS adsorbent demonstrated a slight adsorption reduction from 45 to 60 minutes (about 4%); nevertheless, it attained its rate of sorption at 120 minutes again. Although the reduction was negligible, it could result from the desorption process. This kind of reduction was not observed for PPGS which was due to differences in adsorption mechanisms. The results of this stage were used for calculations related to the adsorption kinetics.

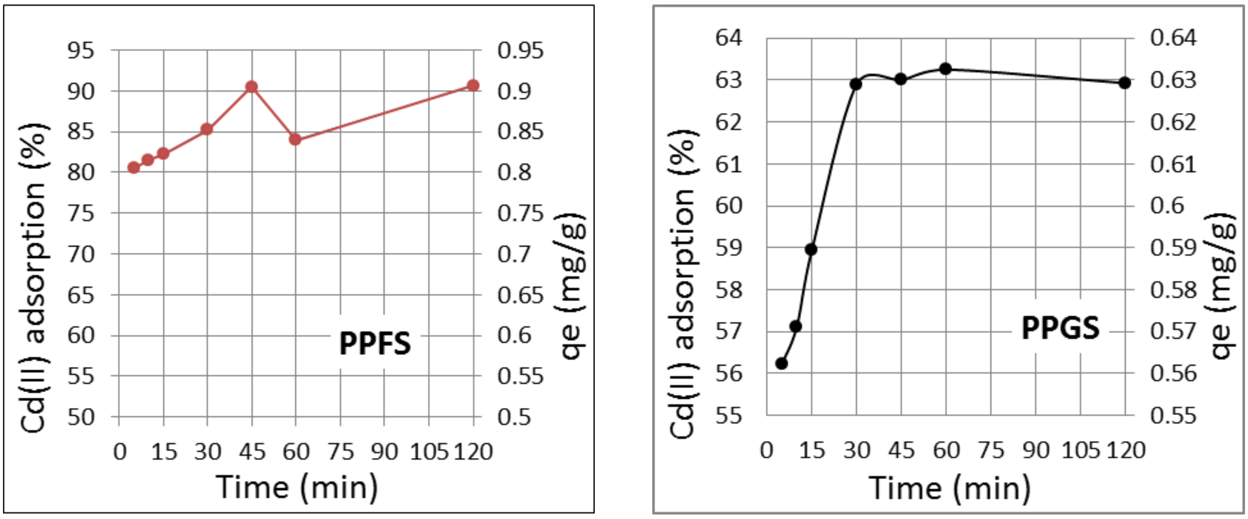
3.3 The effect of adsorbent dose on the volume of solution

The influence of adsorbent dose on the removal of Cd (II) ions has been shown in figure 3. The outcomes confirmed that the adsorption percentage rose rapidly by adding the dose of the adsorbent. The maximum adsorption percentages, 91%, and 73% were achieved in 20 g/L and 40 g/L of adsorbent dose, for PPFS and PPGS respectively. This increase can be attributed to more void cell walls, available to adsorb metal ions in higher adsorbent doses. Afterward, the increment in adsorbent did not play role in enhancing the adsorption percentage, since the active Cd (II) ions were adsorbed before.



(a) (b)

Figure 1: Effect of initial pH on efficiency of cadmium adsorption capacity by the use of adsorbents, a) PPFS, b) PPGS, (adsorbent dose = 20 g/L, cadmium concentration = 20 mg/L, and contact time = 60 min)



(a) (b)

Figure 2: Effect of contact time on efficiency of cadmium adsorption capacity by the use of adsorbents, a) PPFS, b) PPGS (adsorbent dose = 20 g/L, cadmium concentration = 20 mg/L, and pH = 6.5).

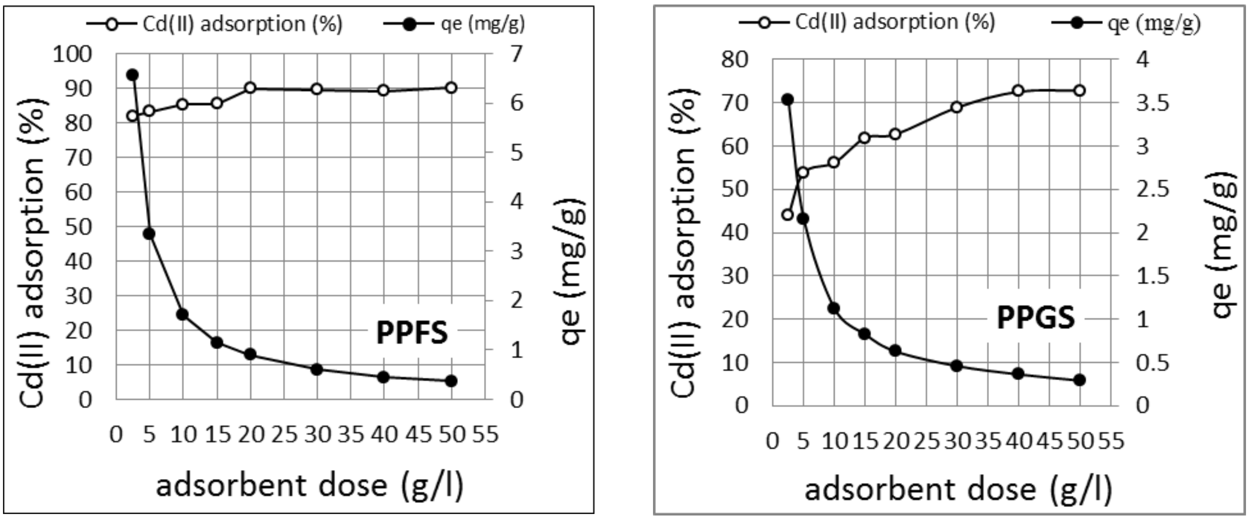
The adverse process of adsorption percentage and adsorption capacity denotes that by the adsorbent dose increase, the ratio of cadmium mass adsorbed onto the adsorbent is reduced, and the adsorbent surface remains unsaturated [50].

3.4 The effect of initial concentration of cadmium

The initial cadmium concentration in an aqueous solution governs the sorption rate. Figure 4 reveals the inverse relationship of initial concentration with adsorption percentage. According to the plot, in low initial concentrations (near to 0.5 mg/L) adsorption percentage for PPFS and PPGS was found to be 98% and 88%, respectively. With the increase in initial concentration, the adsorption decreased until an initial concentration of 100 mg/L when it reached 80% and 57% removal for PPFS and PPGS, respectively. Similar results were achieved by earlier researchers [51]. Constant numbers of pores on the sorbent surface confine the process, particularly when the metal concentration increases. The results of this stage were used for calculations related to the adsorption isotherms.

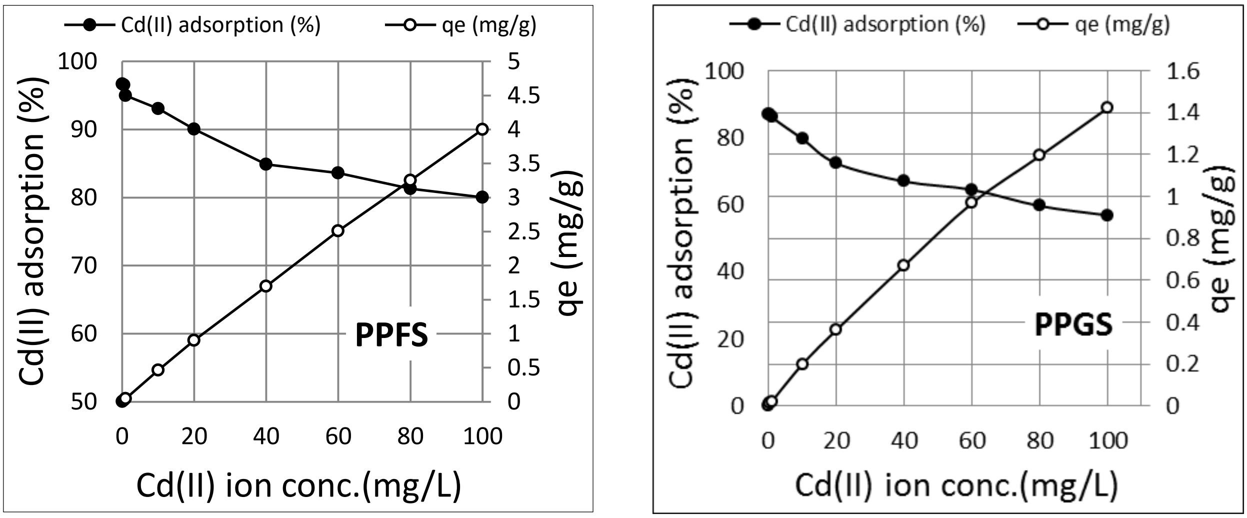
3.5 Relation between initial and equilibrium pH of the solution

Besides the above experiments, a correlation between the initial and equilibrium pH of the solution was investigated. As can be observed in figure 5, the initial acidic pH of the solution led to a strong increase in the ultimate pH. On the other hand, as we moved toward the alkaline area, the ultimate pH dropped in solution. Typically, it can be concluded that during the adsorption process the equilibrium pH tended to hold neutral pH. Indeed, the adsorption of some H^+ ions by the adsorbent in an acidic solution made the equilibrium pH go up. In the same way, the adsorption of some OH^- ions by the adsorbent in basic solution caused the equilibrium pH to go down. In other words, the adsorbent showed buffer characteristics [15].



(a) (b)

Figure 3: Effect of adsorbent dose on the efficiency of cadmium adsorption capacity by the use of adsorbents, a) PPFS, b) PPGS, (contact time PPFS and PPGS are 45 and 30 min respectively, cadmium concentration = 20 mg/L, pH = 6.5)



(a) (b)

Figure 4: Effect of initial cadmium concentration on the efficiency of cadmium adsorption capacity by the use of adsorbents, a) PPFS, b) PPGS, (contact time for PPFS and PPGS are 45 and 30 min, adsorbent dose for PPFS and PPGS are 20 g/L and 40 g/L respectively, pH = 6.5)

3.6 Adsorption kinetics

Coefficients of both pseudo-first and pseudo-second-order kinetic models have been manifested in table 1 and related plots have been illustrated in figure 6 as well. Correlation coefficients disclose that adsorption of cadmium followed the pseudo-second-order kinetic model with a correlation coefficient of $R^2 = 0.99$ for both PPFS and PPGS. It shall be noted that the pseudo-first-order model was applied only for determining K_1 coefficient (mass transfer coefficient in design) and q_e obtained from this model was not reliable. In contrast, the difference between q_e obtained from the pseudo-second-order kinetic and experimental q_e was insignificant [52].

3.7 Adsorption isotherms

Employing the experimental data, the linear form of Langmuir, Freundlich, and DR equations were studied (figures 7-9). Moreover, the constant and correlation coefficients of the isotherms have been reported in table 2. Based on the presented information, the maximum capacity of adsorbents in single-layer adsorption (q_{max} in Langmuir isotherm) was determined to be 4.610 and 1.765 mg/g for PPFS and PPGS, respectively. The separation coefficient, R_L, was calculated 0.056-0.98 and 0.13-0.99 for PPFS and PPGS adsorbent, respectively (equation 9). For both adsorbents, the R_L parameter was less than 1 which denoted the Langmuir isotherm as a favorable adsorption process. The adsorption magnitude constant, n, in Freundlich isotherm was obtained 1.319 and 1.272 for PPFS and PPGS, respectively. Since they were little more than 1, it could be concluded that the adsorption process was suitable and heterogeneity of adsorbent surface was not important on the adsorption process. Owing to the mean biosorption energy values, E parameter, which were less than 8 kJ/mol (0.03162 and 0.02887 kJ/mol for PPFS and PPGS, respectively), it could be inferred that the cadmium adsorption process by these adsorbents had a physical and multilayer nature. The correlation coefficients equal to 0.99 suggested that Freundlich isotherm was the best-fitting isotherm. A Comparison of maximum amounts of adsorbent capacity (q_{max} (mg/g)) between the adsorbents used in this study (PPFS and PPGS) and the other studies is performed in table 3.

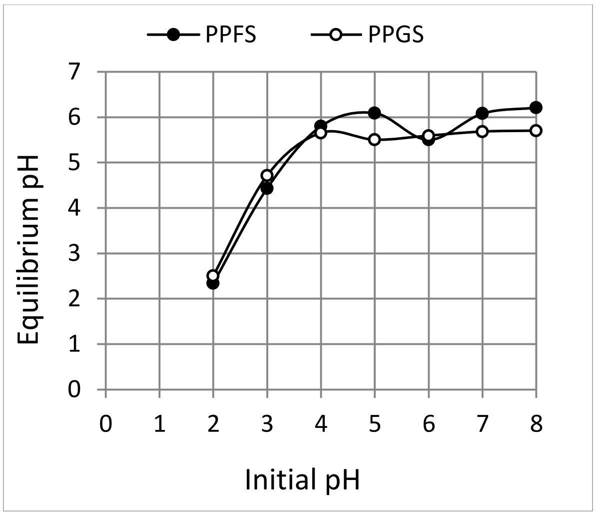
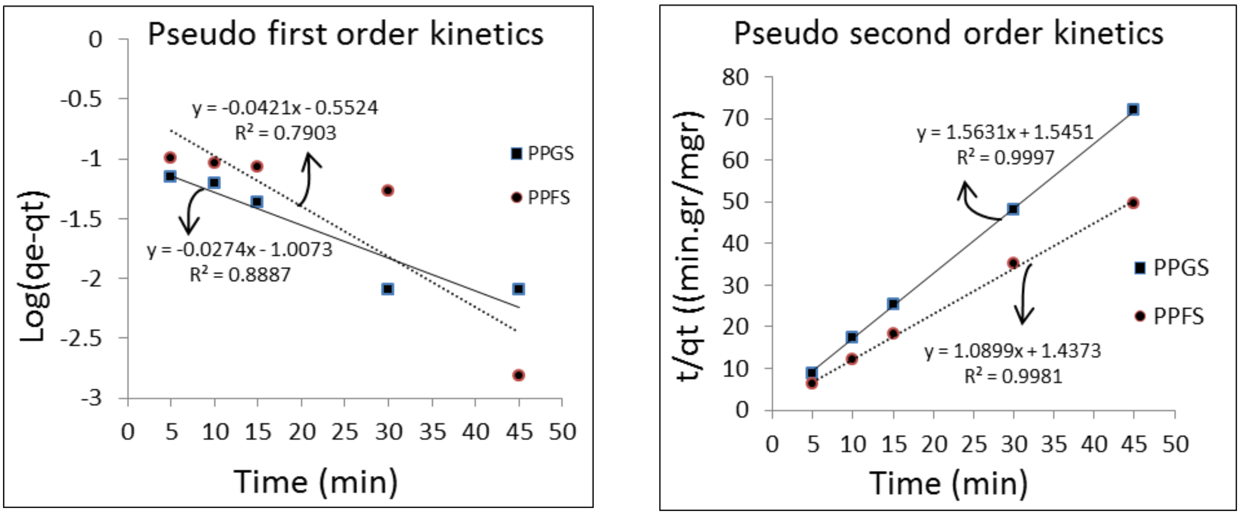


Figure 5: Initial pH versus equilibrium pH of solution for both adsorbents (adsorbent dose = 20 g/L, cadmium concentration = 20 mg/L, and contact time = 60 min)



(a) (b)

Figure 6: Pseudo-first order and pseudo-second order kinetic models PPSF and PPGS, (adsorbent dose = 20 g/L, pH = 6.5 and cadmium concentration = 20 mg/L)

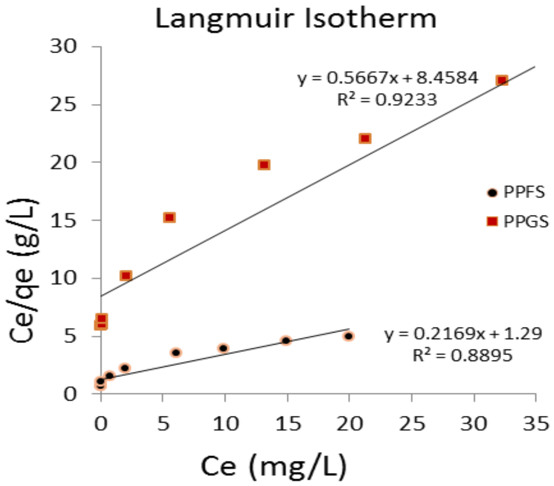


Figure 7: Langmuir isotherm model for the cadmium adsorption onto PPFS and PPGS

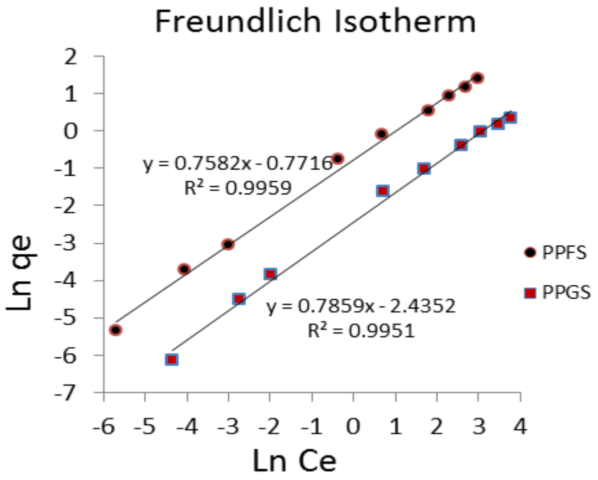


Figure 8: Freundlich isotherm model for the cadmium adsorption onto PPFS and PPGS

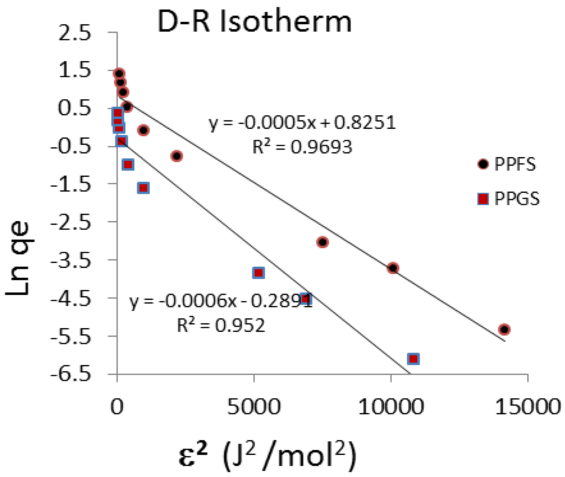


Figure 9: DR isotherm model for the cadmium adsorption onto PPFS and PPGS

Table 1: Calculated kinetic parameters for pseudo-first and second-order models for the adsorption of cadmium using PPFS and PPGS

Kinetic model	Constant coefficient of the model	PPFS	PPGS
Pseudo-first order	K_1 (1/min)	0.097	0.063
	q_e (mg/g)	0.28	0.098
	R^2	0.790	0.888
Pseudo-second order	K_2 (g/mg.min)	0.827	1.58
	q_e (mg/g)	0.917	0.64
	R^2	0.998	0.999
Experimental q_e (mg/g)		0.905	0.625

Table 2: Isotherm constants and calculated regression coefficients of adsorption isotherms for cadmium adsorption onto PPFS and PPGS

Isotherms	Parameters of isotherm	PPFS	PPGS
Langmuir	q_{max} (mg/g)	4.610	1.765
	b (L/mg)	0.168	0.067
	R^2	0.8895	0.923
Freundlich	k_f ((mg/g).(L/mg)n)	0.462	0.087
	n	1.319	1.272
	R^2	0.996	0.995
DR	q_{max} (mg/g)	2.282	0.749
	k_{DR} (mol^2/J^2)	0.0005	0.0006
	R^2	0.969	0.952

Table 3: Comparison of adsorption capacities of Cd (II) between various adsorbents

Adsorbents	q_{max} (mg/g)	Reference	Adsorbents	q_{max}(mg/g)	Reference
Moringa Oleifera	1.06	[53]	Walnut tree	4.51	[57]
Spruce Wood	2	[54]	Punica granatum seed	5	[17]
Rice husk ash	3.02	[55]	Alhajimaurorum seed	3.75	[18]
Raw corn stalk	3.39	[56]	Prosopis farcta seed	4.610	Current study
Almond shell	7	[57]	Peganum harmala seed	1.765	Current study

4 Conclusion

Our present investigation elicited that PPFS and PPGS have potential applications for cadmium adsorption from the aqueous phase as alternative and low-cost adsorbents. The adsorption percentages were achieved 91% and 73% at pH = 6.5 with 20 mg/L of initial cadmium concentration for the usage of 20 g/L and 40 g/L of PPFS and PPGS adsorbents in 45 min and 30 min, respectively. Adsorption kinetics for both adsorbents satisfactorily fitted the pseudo-second-order kinetic model. In an analysis of three isotherms, Langmuir, Freundlich, and DR, adsorbents strongly obeyed the Freundlich isotherm and therefore, a physical and a multilayer process was dominant.

Ethical issue

Authors are aware of and comply with, best practices in publication ethics specifically about authorship (avoidance of guest authorship), dual submission, manipulation of figures, competing interests, and compliance with policies on research ethics. Authors adhere to publication requirements that submitted work is original and has not been published elsewhere in any language. Also, all procedures performed in studies involving human participants were in accordance with the ethical standards of the institutional and/or national research committee and with the 1964 Helsinki declaration and its later amendments or comparable ethical standards. All procedures performed in this study involving animals were following the ethical standards of the institution or practice at which the studies were conducted.

Competing interests

The authors declare that no conflict of interest would prejudice the impartiality of this scientific work.

Authors' contribution

All authors of this study have a complete contribution for data collection, data analyses, and manuscript writing.

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