Environmental Health Engineering and Management Journal

Original Article

Environmental Health Engineering and Management

Removal of cadmium from aqueous solution using nano *Prosopis cineraria* leaf ash (NPCLA)

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Abstract

Background: Heavy metal pollution has become one of the most severe environmental problems, therefore, the present study was conducted to investigate removal of cadmium (Cd) from aqueous solution.

Methods: In the present study, nano *Prosopis cineraria* leaf ash (NPCLA) was used as an adsorbent for removing Cd from aqueous solution. Contact time, initial pH of the solutions, sorbent dosages, and initial Cd (II) concentration were considered as parameters affecting Cd removal efficiency. The experiments were designed by Design of Expert (DOE) software.

Results: It was revealed that an NPCLA dosage of 2.45 gL^{-1}, pH of 6.3, and initial Cd (II) concentration of 20.7 mgL^{-1} in contact time of 60 min was the optimum condition for removal of Cd (II) in the domain of experiments and at this optimum condition, the prediction value of removal efficiency was found 99.9%. Based on the results of kinetics experiments, the sorption system and experiment data of Cd (II) adsorption on the NPCLA followed the pseudo-second-order kinetic model. Freundlich, Langmuir, Brunauer-Emmett-Teller (BET), and Temkin isotherm models were investigated. The equilibrium adsorption data were fitted well with linearly transformed Langmuir isotherm with a correlation coefficient of $R^2 = 0.9877$, and the maximum sorption capacity of NPCLA was obtained to be 25.25 mgg^{-1}.

Conclusion: In laboratory conditions, NPCLA can remove Cd from aqueous solution with a high efficiency. Therefore, due to this plant's availability and cheapness, NPCLA can be considered a suitable option for producing adsorbents on industrial scales.

Keywords: Cadmium, Hydrogen-ion concentration, Nanostructure, Plant leaves, Kinetics

Citation: Pourjaafar M, Askari A, Salehi Sahl Abadi A, Anvaripour B, Nemati A, Rahimi SA, et al. Removal of cadmium from aqueous solution using nano *Prosopis cineraria* leaf ash (NPCLA). Environmental Health Engineering and Management Journal 2023; 10(2): 225–233. doi: 10.34172/EHEM.2023.25.

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Introduction

Heavy metals are elements having an atomic weight between 63.5 and 200.6, with a specific gravity greater than 5.0 (1). With the rapid development of various industries, direct or indirect discharge of wastes containing metals into the environment is increased, especially in developing countries, which have brought severe environmental pollution and threatened life. The disposal of industrial wastewater containing heavy metals in the environment has generated several concerns (2). All forms of heavy metal ions have lethal effects on life by entering the food chain through waste disposal in water channels. Among various metal ions, lead, mercury, cadmium (Cd), and chromium (VI) are on the top of the toxicity list. Nowadays, heavy metal pollution has become one of the most severe environmental problems. Cd can be extracted as a part of mineral stones. This heavy metal is used in various industries such as electroplating, paint production, battery making, etc (3,4). The annual production of Cd is approximately 20 000 tons. About 50% of Cd discharged into the waters is due to electroplating industry (4). The World Health Organization (WHO) has set a maximum guideline of Cd concentration to be 0.003 mg/L in drinking water. Therefore, Cd removal from

industrial wastewater due to its solidity and accumulation effects is vital before releasing it into the environment (5). Absorbed Cd accumulates in different body organs, including the kidney and liver, as the target organ, and in other parts, including the thyroid, gallbladder, etc (6). The most expansive methods used to remove Cd and heavy metals from the various industrial effluents are physico-chemical methods, such as chemical precipitation, ion exchange, electrochemical treatment, filtration and membrane separation, adsorption, etc. Conventional techniques have limitations, including less efficiency, sensitive operating conditions, secondary sludge, and costly disposal (7,8). Adsorption is an efficient approach for removing heavy metals from aqueous solution. The adsorption technique has several advantages, including flexibility in design and operation and producing high-quality treated effluent in many cases. In addition, adsorption is sometimes reversible so that a suitable desorption process can regenerate the adsorbents (9). Prosopis cineraria is a species of flowering tree, and it is aboriginal to arid portions of Western Asia and the Indian Subcontinent (10). In Iran, a lot of it is located in Khuzestan, especially in Abadan and Khorramshahr. In this study, the Prosopis cineraria was chosen as an adsorbent because of its low cost, abundance, and human safety. Also, nano *Prosopis cineraria* leaf ash (NPCLA) was used as an adsorbent for removing Cd from aqueous solution.

Martials and Methods
Experimental methodology
Cd $(No3)2.4H_2O$, HNO_3, and NaOH were provided from Merck productions. Standard Cd (II) stock solution was prepared by dissolving an appropriate amount of Cd $(No3)2.4H_2O$ in distilled water. PH adjustment of solutions was made using dilute or concentrated 0.1N NaOH and 0.1N HNO_3 solutions (11). All the reagents were used without further purification. The elements of NPCLA and particle size were defined using scanning electron microscopy (SEM) with the energy-dispersive X-ray spectroscopy (SEM/EDX) technique (Jeol JSM-6700F). The chemical composition was distinguished by the X-ray diffraction technique (XRD: EQUINOX-3000 diffractometers, USA) (12). The functional groups and surface properties of NPCLA were carried out by Fourier transform infrared spectroscopy instrument (FTIR vertex 70 Bruker, Germany) in the wave number range of 4000–400 (13). The surface area measurement for NPCLA was determined by the Brunauer–Emmett–Teller (BET) method using BELSORP (mini (II), Japan) instrument by nitrogen adsorption-desorption measurements (14). The adsorbent was dried by an oven (Fan Azmagostar Co., Iran). Next, the Cd (II) concentration in the centrifuged solution was determined using an atomic adsorption flame spectrometer (Analytic Jena AAS 5FL, Germany).

PH was measured using a pH meter (Jenway 3510). The mixing rate was measured by an adsorption flame spectrometer (Analytic Jena AAS 5FL, Germany). A statistical package Design–Expert (version 10.0.0, State-Ease, Minneapolis) was used to design and analyze experiments (15).

Adsorbent development
Prosopis cineraria leaves were collected, then, they were washed several times with distilled water to remove dust and soluble impurities, then, dried in an oven for 24 hours at 105°C and burned at 500°C for 5 hours. After burning, the obtained ashes were rewashed with distilled water to remove impurities. Afterwards, they were washed with 0.001M HCL to remove all of the remaining salts precipitates and washed again with distilled water remaining acids. In the next step, they were kept in the oven at 85°C for 24 hours to evaporate organic impurities. Finally, the product was put in 1M HNO_3 at room temperature for 5 hours.

Adsorption studies
Different Cd (II) concentrations (20, 60, and 100 ppm) were prepared. Sorption batch experiments were conducted in batch adsorption isotherm mode at room temperature (20 ± 2°C). The batch sorption experiments were directed in a shaker (yellow line-MSEB model, Germany) at a constant agitation rate (250 rpm) for a constant time (60 minutes). The influence of the contact time parameter on the adsorption process was studied in the kinetic experiments to obtain the equilibrium time. All experiments were carried out with 50 mL of Cd (II) solutions in a 100 ml Erlenmeyer flask. According to the experimental design, 50 mL of predicted initial Cd (II) concentration was poured into a 100 mL Erlenmeyer flask. The pH was controlled at a defined value by adding 0.1 mol/L nitric acid or 0.1 mol/L sodium hydroxide solutions. The appropriate dosage of NPCLA based on the experiment design prediction was added to the mixture solution. Next, the mixture was mounted on the shaker at a constant agitation speed (250 rpm) for one hour at room temperature (20 ± 2°C). After reaching to 60 min, the adsorbent was separated by centrifuging the solution below 11000 rpm for 10 min, and then, solution concentration was analyzed using a flame atomic absorption spectrophotometer (FAAS).

Optimization
Optimization of Cd (II) removal in the domain of experiments was done by Design of Expert (DOE) software (Response Surface Methodology, RSM technique). The result indicates that an NPCLA dosage of 2.45 gL^{-1}, pH of 6.3, and initial Cd (II) concentration of 20.7 mg in contact time of 60 minutes is the optimum condition for removing Cd (II) in the domain of experiments.

Kinetic experiments

Kinetic studies were done to study the effect of time on Cd (II) removal rate by NPCLA. For this step, the same amount of NPCLA was used and kept in contact with the standard solution for different time intervals (20, 30, 60, 120, and 240 minutes). 300 mL of Cd $(No3)2.4H_2O$ with 20.7 mg/l Cd (II) concentration was taken in a 300 ml volumetric flask pH was controlled at optimized pH (for adjusting, 0.1 M HNO_3 or 0.1 M NaOH were used). The solution was added from volumetric flask to 6 Erlenmeyer flasks (50 mL to each Erlenmeyer flask). To each Erlenmeyer flask, 0.12 g of sorbent was added and kept in contact with the solution for the stated time intervals. The speed of the shaker was set to 250 rpm at room temperature (25 ± 5°C). The adsorbent was separated by centrifuging the solution below 11 000 rpm for 10 min, and then, solution concentration was analyzed using a FAAS. The complaisance between experimental data and the model predicted values was expressed by correlation coefficients (R^2) used to determine the best suitable kinetic model.

Results

NPCLA characteristics

Based on the BET analysis, the specific surface area and mean pore diameter NPCLA was found to be 5.1862 (m^2 g^{-1}) and 39.45 (nm), respectively. The mean pore diameter is between 2 and 50 nm, as a result, the pores are mesoporous. According to XRD analysis, NPCLA chemical compounds almost are $CaCO_3$, Ca_3SiO_5, $Ca_2P_2O_7$, SiO_2 and Ca_2SiO_4. Figure 1 shows the FTIR spectra of the NPCLA. The figure shows a hydroxyl group such as carboxylic acids (R-CO-OH and C=C-C-OH) at 3424 cm^{-1}, alkanes (RCH_2CH_3) at 3000 and 2896 cm^{-1},

phosphonic acid ((O=) PO-H) at 2609 cm^{-1}, phosphine and silane at 2358 cm^{-1}, oxygen functional groups such as C=O stretching in carboxylic groups, and carboxylate moieties at 1778 and 1797 cm^{-1}, carboxylic acids, alkenes and aromatics at 1600 cm^{-1}, sulfate (S=O) at 1428 cm^{-1}, and finally, C-O and P=O at 1200 cm^{-1}.

Figure 2 indicates the field emission scanning electron microscopy (FE-SEM) images of NPCLA particles. It was revealed that the nanoparticles have a high affinity to congregate. Also, it was observed that NPCLA particles have porous structures, which are sufficient for the adsorption process.

Figure 3 displays the FE-SEM analysis of NPCLA particles size. As shown in this figure, the size of NPCAL is around 17 nm.

The percentage of components of NPCLA was calculated by EDX analysis. Table 1 shows the EDX atomic ratio.

Operating variables effect
Effect of pH on Cd (II) removal efficiency

As an essential and influential factor for the adsorption process, pH was studied over a pH range of 4.0 to 10.0 (Figure 4). It was found that Cd binding to NPCLA was pH-dependent, and the maximum sorption of Cd to NPCLA was found to have an initial pH of 6.37. At low pH, the number of available H^+ ions is high, and hydrogen (H^+) ions are likely to compete with Cd (II) ions for binding sites on NPCLA surfaces. Therefore, H^+ ions hinder the approach of Cd (II) ions from reaching the sorption sites because of the offensive force. So, sorption of Cd (II) to NPCLA increased with the increase of the initial pH from 4.0 to 6.37 since the repulsive force and contest of Cd (II) and H^+ decreased. On the other hand, removal efficiency at higher initial pH (pH > 6.37) was
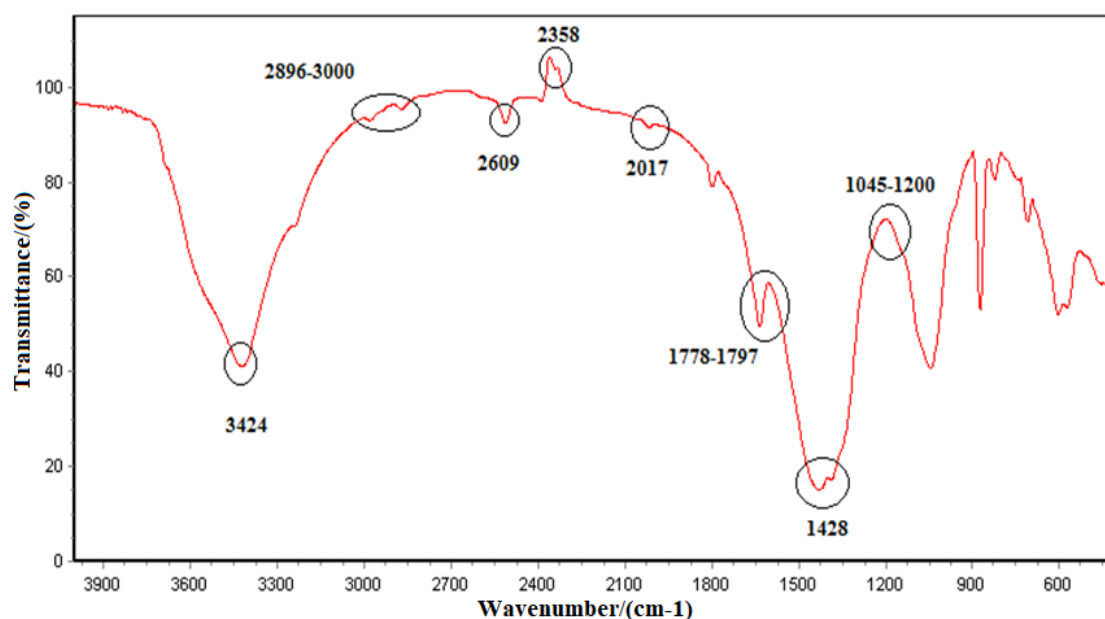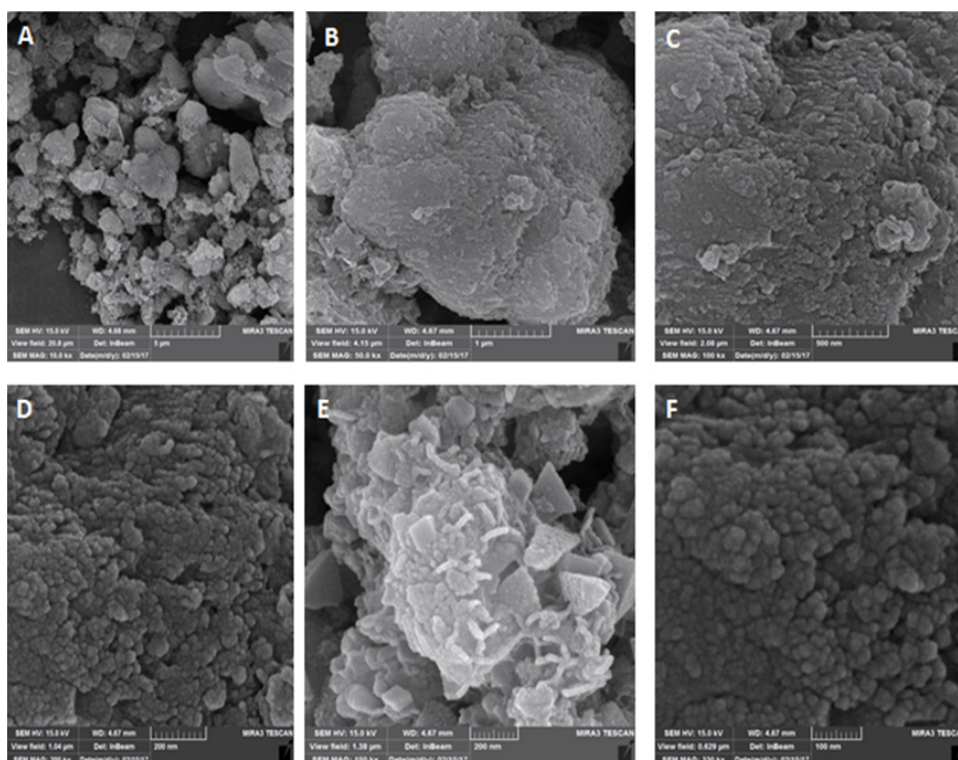

Figure 1. FTIR spectra of the NPCLA
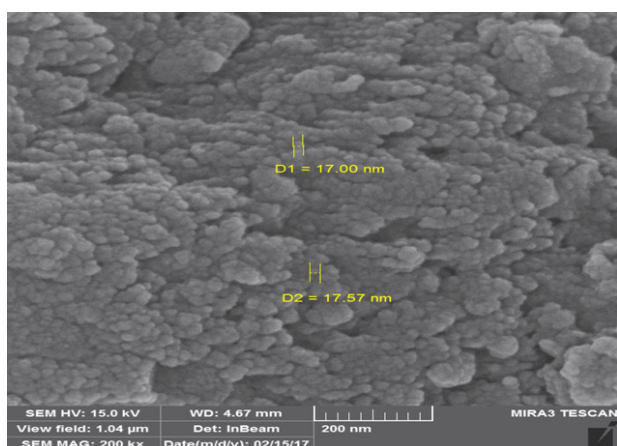
Figure 2. FE-SEM analysis of NPCLA structure particles

Figure 3. FE-SEM analysis of NPCLA particles

Table 1. NPCLA element analysis by EDX

Element	$W_t\%$	$A_t\%$
O	66.08	80.67
Na	0.21	0.18
Mg	5.63	4.52
Si	2.01	1.40
P	3.23	2.04
S	0.88	0.54
K	0.44	0.22
Ca	21.49	10.32
Fe	0.33	0.11

declining due to the precipitation of Cd (II) ions. Thus, hydroxide anions formed a Cd hydroxide precipitate.

Sorbent dosage (NPCLA) effect on Cd (II) removal efficiency
Adsorbent dosage is another critical parameter that determines an adsorbent's capacity for a given initial concentration of the adsorbent. This item was studied (Figure 5) for removal of Cd ions from aqueous solutions by varying the amount of NPCLA mass in order to vary mass ratio while keeping the other parameters constant (NPCLA dosage in the range of 1.0 g to 3.0 gL^{-1} at pH = 7.0 and initial Cd (II) concentration of 60 mg).

Initial Cd (II) concentration effect on Cd (II) removal efficiency
Metal removal efficiency closely depends on the initial concentration of metal ions. Figure 6 shows the effect of the initial concentration of Cd (II) by varying the initial concentration of Cd (II) while keeping the other parameters constant (initial concentration of Cd (II) in the range of 20 $mg.L^{-1}$ to 100 $mg.L^{-1}$ at pH = 7.0 and NPCLA dosage of 2 $g.L^{-1}$). The removal efficiency increases as the initial concentration of Cd decrease (initial concentration was increased from 20 to 100 $mg.L^{-1}$ and the removal efficiency decreased from 95.2 to 68.5%), respectively due to the saturation of adsorption sites. In other words, at a low initial concentration of Cd (II), more sites that are binding are available on the adsorbent surface. When the initial concentration increases, the Cd (II) ions compete for binding sites on the NPCLA surface.
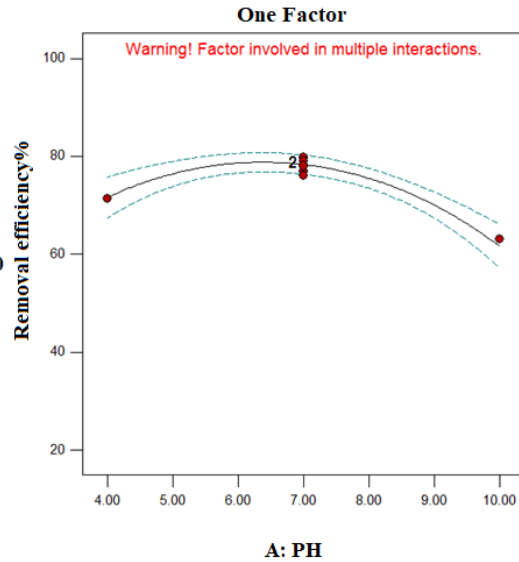
Figure 4. Cd (II) removal efficiency versus various pHs from 4.0 to 10.0
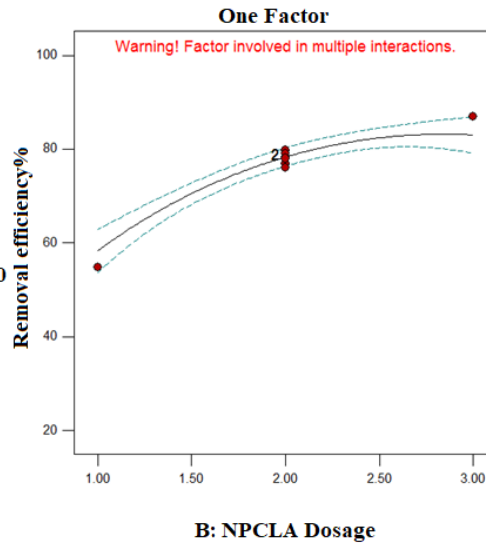
Figure 5. Cd (II) removal efficiency versus various NPCLA dosage from 1.0 to 3.0 g/L
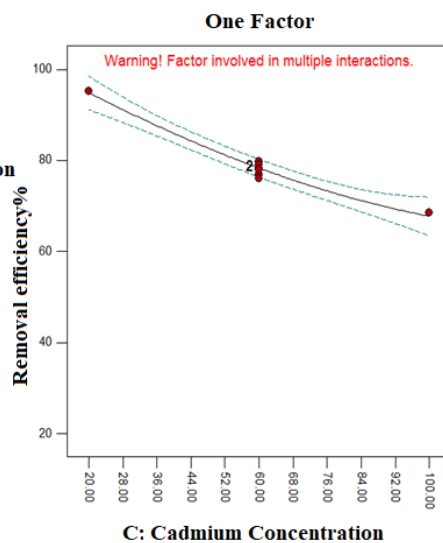

Figure 6. Cd (II) removal efficiency versus various initial Cd (II) concentrations

Combined effect of pH and NPCLA dosage on removal efficiency

The combined effect of pH and NPCLA dosage results on the percent of removal are shown in Figures S1 and S2 (See Supplementary file 1). Three-dimensional (3D) graphs help to have a better quality understanding of the process, but contour graphs help to have a better quantity understanding. A contour plot is a version of 3D plots compressed on the X_1-X_2 plane. Based on Figure S1, the yellow region (plane X_1-X_2) is a contour plot indicated in Figure S2.

Figure S2 displays that by increasing pH from 4.0 to 6.37, the Cd (II) removal efficiency is increased, and at pH values higher than 6.37, the removal efficiency decreases. Also, the removal efficiency increased as NPCLA dosage increased. As shown in the figures, the slope of removal efficiency versus NPCLA dosage is more than the slope of the removal efficiency versus pH in the domain of experiments. The figures indicate that NPCLA dosage changes are more important than pH changes.

Combined effect of NPCLA dosage and initial concentration on removal efficiency

The results of the combined effect of NPCLA dosage and initial Cd (II) concentrations on the percent of removal are shown in Figures S3 and S4. These figures show that removal efficiency decreased with increasing Cd (II) initial concentrations. Also, removal efficiency has a direct relationship with NPCLA dosage.

Equilibrium contact time studies

A contact time study was carried out to obtain the required time to reach equilibrium for further adsorption. Figure S5 shows the plot of the removal rate of Cd by the adsorbent.

Optimization

Optimization of Cd (II) removal in the domain of experiments was done by DOE software (Response Surface Methodology; RSM technique). As shown in Table 2, an NPCLA dosage of 2.45 gl^{-1}, pH of 6.3, and initial Cd (II) concentration of 20.7 mg in contact time of 60 min is the optimum condition for removing Cd (II) in the domain of experiments. At this optimum condition, the maximum value of removal efficiency occurred. At this optimum

Table 2. Optimum experiments results

pH	NPCLA dosage (gL^{-1})	Cd (II) concentration (mgL^{-1})	Removal efficiency (%)
			99.3
			99.6
6.3	2.45	20.7	99.7
			99.7
			99.7

condition, the prediction value of DOE software for removal efficiency is 99.9%. It should be noted that to ensure the results, each experiment was repeated 5 times.

Sorption kinetics

Based on Table 3, the correlation coefficient for pseudo-first-order is 0.9935 and the theoretical value of q_e (q_e, cal) is 12.195 mg/g; it has a high difference from the experimental value (q_e, exp = 8.421 mg/g). Furthermore, the second-order kinetic model is 0.9973, and also, the calculated value of q_e (8.857 mg/g) agrees very well with the experimental value. These results suggest that the sorption system and experimental data of Cd (II) adsorption on the NPCLA followed the pseudo-second-order kinetic model.

Sorption isotherm

The Langmuir, Freundlich, BET, and Temkin equations were used for modeling the adsorption isotherms. Table 4 shows the results of the experiments.

Comparison of the R^2 values for various model isotherms reveals that the Langmuir model best fits the experimental data for Cd (II) adsorption.

Sorption cost

Table S1 shows a comparison of maximum Cd adsorption capacities for different adsorbents. The NPCLA adsorbent also shows an adequate adsorption capacity compared to the adsorption capacity of various low-cost adsorbents for Cd removal. Thus, NPCLA shows a suitable locally available and cost-effective alternative sorbent material to remove Cd from wastewater (16-24).

Discussion

One of the most critical parameters affecting the metal uptake capacity of adsorbent materials is the pH value of the metal solutions. The pH affected the adsorption property concerning Cd and was less influenced by the ionic strength (25). The results of the present study are significantly consistent with the results of similar studies (Figure 4). Studies by Minnu et al (16), Gupta et al (26), Rao et al (19), and Garg et al (27) show that at lower pH, H$^+$ions competed with Cd (II) ions for the surface binding-sites of the adsorbent and the metal retention in such condition was not significant. At pH < 6, the divalent form of the Cd (II) ions were dominant species; hydroxide forms were present at pH values higher than 6. Therefore, it can be said that at pHs about 6 is predominantly by the adsorption process it is controlled. At pHs above 6, this value can be expected to increase with the deposition of Cd hydroxide. According to Figure 5, it is clearly shown that by increasing the adsorbent dosage from 1 to 3.0 g/L, the removal efficiency increases. Increasing the adsorption dosage increases the number of available adsorption sites to improve the removal efficiency. So, the removal

Table 3. Adsorption kinetic parameters of Cd (II) on NPCLA

Kinetic model		Pseudo-first order model			Pseudo-second order kinetic model			
C0 (mg/L)	q_e, exp (mg/g)	q_e, cal (mg/g)	K_1 (min⁻¹)	R^2	q_e, cal (mg/g)	K_2 (g/mg.min)	H (mg/g.min)	R^2
20.7	8.421	12.195	0.0779	0.9935	8.857	0.013	1.013	0.9973

Table 4. The Freundlich, Langmuir, BET, and Temkin parameters for Cd (II) sorption on NPCLA

Isotherm models	Isotherm Parameters							
	K_F	N	q_m	K_L	K_B	b_T	a_T	R^2
Freundlich	11.1609	4.5809						0.9368
Langmuir			25.2525	0.5706				0.9877
BET			0.04		409.8361			0.9678
Temkin						944.0847	198.939	0.9432

efficiency increases as the absorbent dosage increases. These results are correct when compared to the adsorbent load needed to achieve similar removals. For instance, Gupta et al (26) reported that a 10 g/L dose of the bagasse fly ash was optimum for almost complete Cd removal from a 14 mg/L Cd solution. Sanchooli Moghaddam et al (20) reported that the maximum Cd uptake was observed at about 4 g/L for Saxaul Tree Ash, with an initial concentration of 30 mg/L. According to Figure 6, when initial Cd concentration is decreased, removal efficiency increases, which is consistent with the results reported by Oyewo et al (28) and Alameen and Majeed (29). As shown in Figure S5, in the first 60 minutes, the removal efficiency reached its maximum value, indicating the high ability and speed of NPCLA in removing Cd ions. As time increases, ions become more likely to contact with the functional groups in the adsorbent structure (adsorption sites), and the adsorption rate increases. The study of Gupta and Nayak on removing and recovering Cd from aqueous solutions by adsorbents newly prepared orange peel and Fe_2O_3 nanoparticles indicates that the highest removal efficiency was in the beginning times, which is consistent with the results of the present study (30). Gatabi et al used carbon nanotube magnetic sorbents to effectively remove Cd and achieved similar results at the contact time study stage, stating that the reaction rate is initially rapid and equilibrates over time (31). A study by Badruddoza et al on removing heavy metals by cyclodextrin-Fe_3O_4 polymer nanocomposite shows that the quasi-quadratic kinetic model is suitable for Cd adsorption and its correlation coefficient is 0.999 (32). Comparison of correlation coefficients obtained by plotting the curves of each isotherm can be a good criterion for expressing the matching of equilibrium absorption data and the studied isotherms. The results of isothermal models showed that the adsorption system and Cd (II) adsorption experimental data on NPCLA follow the quasi-second-order kinetic model (Table 3) and have high correlation coefficients for Freundlich, Langmuir, BET, and Temkin isotherms. We calculated the correlation coefficients for mentioned models 0.9368, 0.9877, 0.9678, and 0.9432,

respectively (Table 4). Therefore, it can be concluded that data modeling is more consistent with the Langmuir model. In the study of Boparai et al on removing Cd by zero iron nanoparticles, the isothermal models of Langmuir, Freundlich, Temkin, and Dubinin-Rashkovich showed that the adsorption of Cd follows the Langmuir model better, which is consistent with the results of the present study (33). Singh et al investigated removal of Cd from aqueous solutions by silica-modified acrylamide nanocomposites and reported that the Cd uptake data follow the Langmuir model. The correlation coefficients for Langmuir and Freundlich isotherms in this study are 0.99 and 0.88, respectively (34). The results of the present study indicate that the NPCLA has a high adsorption capacity that can be used as an effective adsorbent to remove Cd under optimal conditions.

Conclusion

According to the results of the present study, NPCLA is an effective and low-cost adsorbent. By increasing the initial pH from 4 to 6.37, removal efficiency increases because adsorbent surfaces are more negatively charged, and competition decreases. At pH 6.37, removal efficiency decreases because of the precipitation of Cd. The removal efficiency increases with increasing NPCLA dosage because surface area and availability of active sites are more activated. By increasing Cd (II) initial concentrations, removal efficiency decreases because adsorption sites are saturated. The optimum conditions for removal of Cd (II) were calculated at an NPCLA dosage of 2.45 gL⁻¹, pH of 6.3, and initial Cd (II) concentration of 20.7 mgL⁻¹ in a contact time of 60 min. At these conditions, Cd (II) removal efficiency is 99.9%. The pseudo-second-order kinetic model accurately described the adsorption kinetics. The Cd (II) adsorption results showed that the Langmuir isotherm displayed a better fit than the other isotherms.

of Technology. The authors also thank people who participated in this study.

Competing interests
The authors declare that they have no conflict of interests.

Ethical issues
This study was approved by the Ethics Committee of OICO under the 4000821/027 registration number. Ethical issues (e.g., plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, redundancy, etc) have been completely observed by the authors.

Supplementary files
Supplementary file 1 contains Figures S1-S5 and Table S1.

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