RESEARCH PAPER

Efficiency of Chitosan-Grafted Poly (Carboxymethyl Cellulose-Co-Acrylamide) Nano Hydrogel for Cadmium (II) Removal: Batch Adsorption Study

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ARTICLE INFO

Article History:
Received 03 July 2024
Accepted 22 September 2024
Published 01 October 2024

Keywords:
CS-g-P(CMC-co-AM) nano hydrogel
Cadmium (Cd) ions
Batch adsorption
Kinetics

ABSTRACT

Presence of heavy metals (HMs) mainly cadmium (Cd (II)) ions in water cause serious environmental and health risks that needs to be addressed by some effective method. Current study involved the use of Chitosan-grafted Poly (Carboxymethyl Cellulose-Co-Acrylamide) Nano Hydrogel for Cd (II) ions removal from water. The prepared nano hydrogel was characterized via Fourier Transform Infrared (FTIR) spectroscopy, Field Emission Scanning Electron Microscopy (FESEM) and X-ray Diffraction (XRD). The results of these techniques revealed the presence of different types of functional groups in nano hydrogel structure that possess highly rough and amorphous texture. Adsorption study was conducted for investigating the effects of contact time, solution pH, nano hydrogel dose, and ionic strength of different salts (NaCl, KCl, $CaCl_2$) on Cd (II) removal. Findings of the study highlights maximum removal of 93.3% with adsorption capacity 37.3 mg/g in 90 minutes while using 200 mg/L Cd (II) solution of pH 10. Kinetic study showed fitness of adsorption process with pseudo second model with regression coefficient value equal to unity signifying a chemical adsorption mechanism. Overall, the prepared CS-g-P(CMC-co-AM) nano hydrogel possess adsorption potential for cadmium ions removal from water.

How to cite this article
Alshamusi Q., Hameed K., Taher A., Batool M., Jasim L. Efficiency of Chitosan-Grafted Poly (Carboxymethyl Cellulose-Co-Acrylamide) Nano Hydrogel for Cadmium (II) Removal: Batch Adsorption Study. J Nanostruct, 2024; 14(4):1122-1133. DOI: 10.22052/JNS.2024.04.013

INTRODUCTION

The extensive use of heavy metals (HMs) in modern world led to their increased levels in water sources resulting in deteriorating water quality and affecting all life forms on earth. Cadmium (Cd) is one of the toxic metals that enter water system via variety of routes such as galvanized pipes and metal fittings. When ingested, Cd is responsible for causing human organ dysfunction. Keeping in view the toxicity caused by Cd, Department of Environment, UK and European Economic Community placed this metal in red list of priority contaminants and black list of Dangerous Substance Directive respectively [1]. Researchers

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are now working on the development of effective and low-cost methods for removal of Cd (II) ions from water. Several physi-o-chemical and biological methods [2, 3] have been developed aiming to treat polluted water. However, high operational cost and secondary pollution associated with them make them ineffective for treating all kinds of pollutants in water [4-19]. Adsorption, however, an effective and efficient technique for water treatment takes precedence over all other conventional methods due to its simple design, low cost and easy availability of adsorbents.

In adsorption method, selection of adsorbents for efficient pollutant removal is a crucial step. Various kinds of natural and synthetic adsorbents have been developed and used till date for Cd (II) adsorption [1, 20-29]. This study, however, investigated the adsorptive removal of Cd (II) ions from water using poly (carboxymethyl cellulose-co-acrylamide) (CS-g-P(CMC-co-AM)) nano hydrogel as an adsorbent. The nano hydrogel was characterized by FTIR, FESEM, and XRD techniques. Swelling properties of prepared nano hydrogels were also investigated under variable pH of solution. Influence of adsorption factors as contact time, pH, adsorbent dosage, salt concentration was also studied. The nature of adsorption process was investigated by applying the time data on two different kinetic models namely pseudo first and pseudo second order kinetic models.

MATERIALS AND METHODS

Materials and chemicals used

The experimental reagents used in study were acrylamide (AM), chitosan, hydrochloric acid, Carboxymethyl cellulose sodium salt (CMC), ethanol, potassium persulfate, bis-acrylamide, acrylic acid, nitrogen gas, acetic acid, tetramethylethylenediamine (TMEDA), sodium chloride, potassium chloride, and calcium chloride. These chemicals were essential components in conducting batch adsorption experiments and analyzing the results.

Preparation of Cd (II) solution

A stock solution (500 mg/L) of cadmium (II) ions was prepared by dissolving 1.37 grams of cadmium nitrate tetrahydrate ($Cd(NO_3)_2 \cdot 4H_2O$) in distilled water. The solution was quantitatively transferred to volumetric flask (1000 mL) and diluted to final volume by addition of water. Working solutions of variable concentrations were subsequently prepared from this stock solution for further experimentation.

Synthesis and characterization of nano hydrogel CS-g-P(CMC-co-AM)

The prepared nano hydrogel i.e., CS-g-P(CMC-co-AM) was synthesized via preparation of two different solutions [30, 31]. Firstly, solution 1 was prepared a 1% of acetic acid (AAC), dissolve 0.5 g of chitosan (CS) in 20 mL of this solution and dissolving 6g acrylamide in 10 mL deionized water which was then combined with solution of acrylamide (6g in 10 mL water). A solution of N, N'-methylenebisacrylamide (MBA) was prepared by dissolving its 0.05g in 5 mL deionized water. This solution was then added to mixture and stirred for 15 min with simultaneous introduction of nitrogen gas. Followed by preparation of solution 1, next step is to prepare solution 2. For this, 0.5 g Carboxymethyl cellulose sodium salt was dissolved in 10 mL deionized water that was stirred continuously for 15 min. Afterwards, potassium persulfate (KPS) solution (0.1g in 5 mL water) and a TEMED solution (0.05g in 5 mL water) were

Fig. 1. Prepared CS-g-P(CMC-co-AM) nano hydrogel.

added to above mixture serially with continuous stirring. This was followed by addition of Solution 1 (in a dropwise manner) to Solution 2 at 50 °C while nitrogen gas was intermittently introduced for 30 sec. incubation of mixture was then carried out in a water bath at 60 °C for 2 hours that aid in completion of reaction. Obtained hydrogel was cut into smaller pieces that were then washed with water for one hour and oven-drying was done at 50 °C. The dried hydrogel was then ground into very fine particles having diameters varying from 50 to 250 nm. Fig. 1 shows prepared CS-g-P(CMC-co-AM) hydrogel.

The successful synthesis of nano hydrogel was confirmed by Fourier Transform Infrared (FTIR) spectroscopy (Shimadzu 8400s spectrophotometer within range of 500 to 4000 cm^{-1}), Field Emission Scanning Electron Microscopy (FESEM) (TESCAN MIRA3 at 25 kV) and X-ray Diffraction (XRD) analysis (Shimadzu XRD-6000, within 2θ range of 10° to 80°) that helps in analysis of functional groups [2, 32, 33] surface morphology [34, 35] and crystallinity of adsorbent [36] respectively.

Adsorption experiments

Batch adsorption study was carried out for investigating the effect of equilibrium time (0 min to 220 min), pH (1.2 to 10), adsorbent dose (0.001g to 0.08g) and concentration of different salts i.e., NaCl, KCl as well as $CaCl_2$ (0g/L to 0.2g/L). Temperature for all experiments was maintained at 20°C with shaking speed of 120 rpm, Cd (II) ions concentration of 200 mg/L and neutral pH (except pH study). The nano hydrogel dose used for each study was 0.05g and time of adsorption was 90 min unless otherwise specified. Once the adsorption equilibrium was achieved, % removal of adsorbate and adsorption capacity of adsorbent can be calculated using Eq. 1:

$$q_e = \frac{C_0 - C_e}{M} \times V \qquad (1)$$

where and refers to concentrations of Cd (II) ions in solution (mg/l) both before and after adsorption, and denote used volume (mL) of solution and adsorbent weight (g) correspondingly. For investigating effect of solution pH on swelling property of prepared nano hydrogel, 0.05g of it was immersed in solutions of varying pH i.e., 3 to 10. After immersion period, filtration of nano hydrogel was carried out for removing excess water and its weight was measured again. Afterwards, swelling ratios calculation was done by Eq. 2:

$$\text{Swelling ratio } (\%) = \frac{W_S - W_d}{W_d} \times 100 \qquad (2)$$

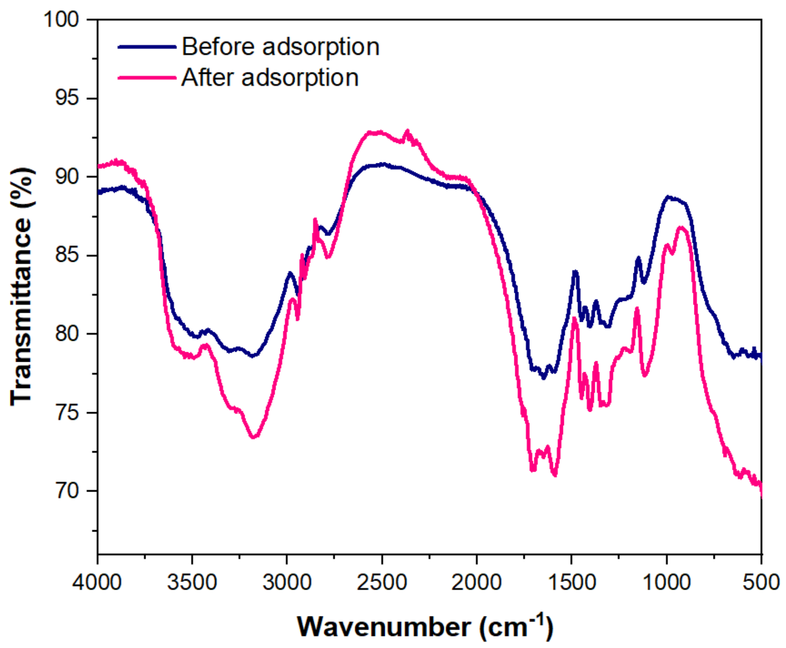


Fig. 2. FTIR results of prepared CS-g-P(CMC-co-AM) nano hydrogel both before and after Cd (II) ions adsorption.

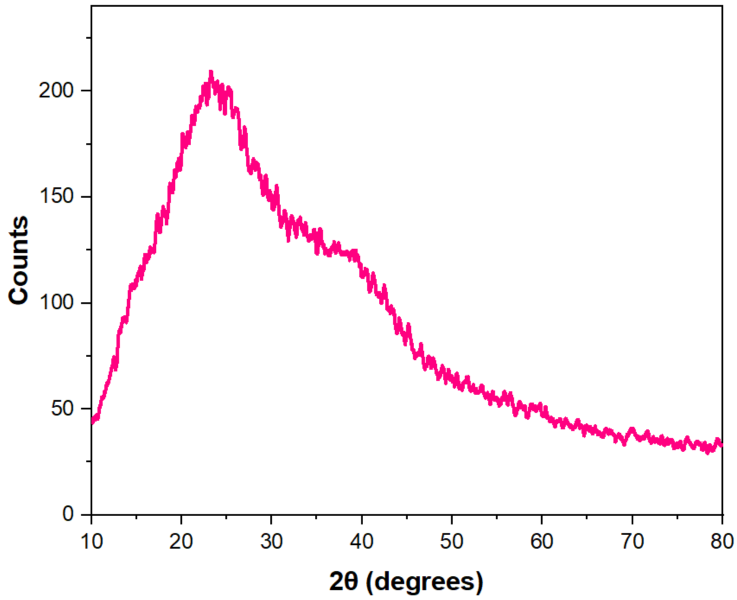


Fig. 3. XRD of prepared CS-g-P(CMC-co-AM) nano hydrogel.

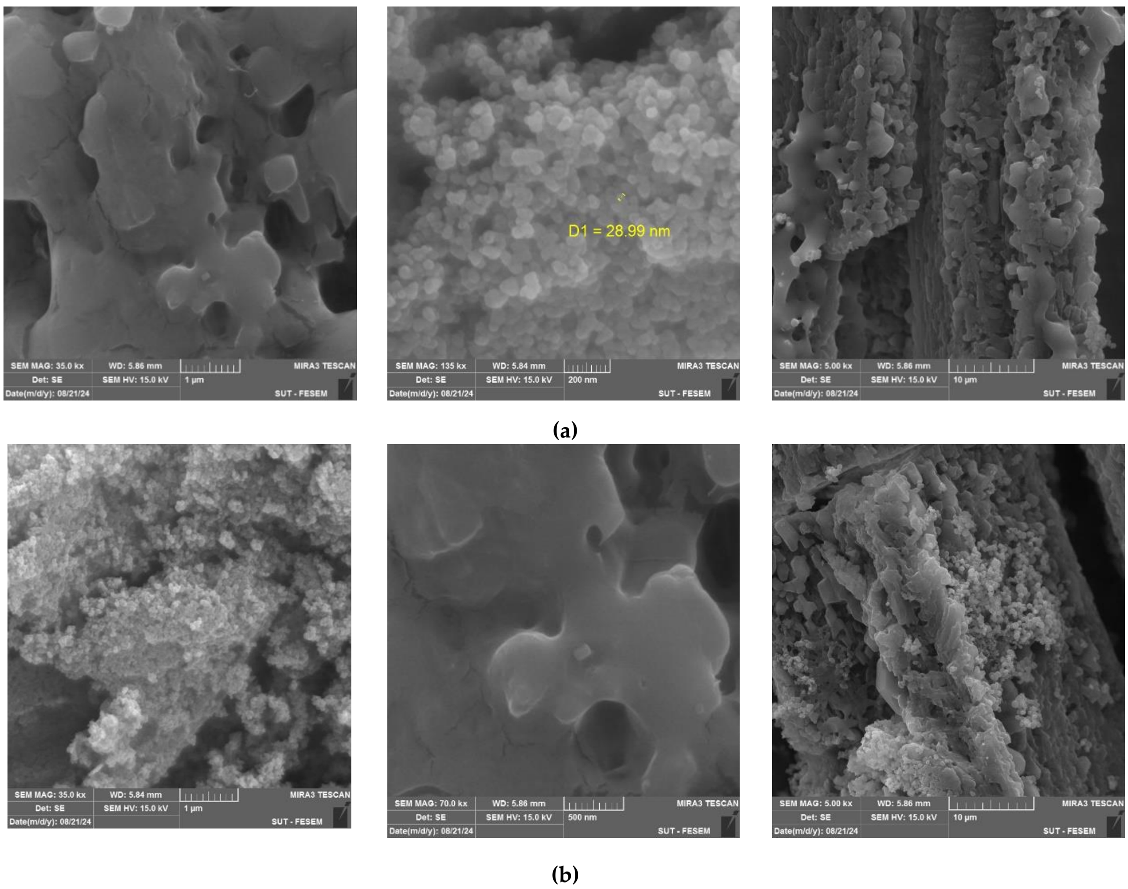


Fig. 4. FESEM of prepared CS-g-P(CMC-co-AM) nano hydrogel (a) before and (b) after adsorption.

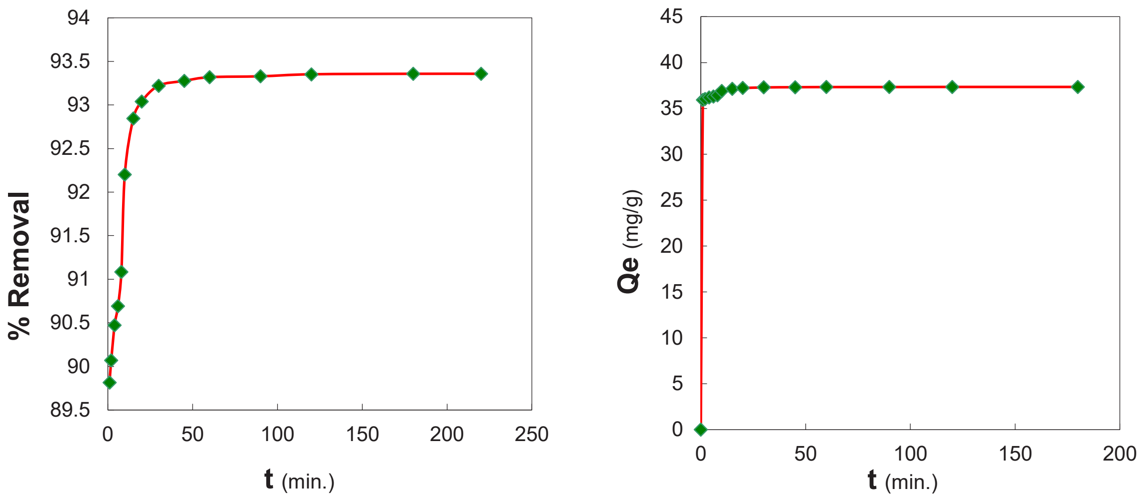
here, and refers to weight of nano hydrogel that gets swelled and weight before swelling (dried form) correspondingly.

Two different kinetic models namely pseudo first as well as pseudo second model applied to data obtained from kinetic study. Pseudo first order model assumes that adsorption rate has direct relation with number of available adsorption sites of adsorbent. In contrast, pseudo second model assumes that adsorption rate is directly related with square of available adsorption sites. Expressions for pseudo first and pseudo second order model are given in Eqs.3 and 4 respectively:

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

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e} \quad (4)$$

here (mg/g) and (mg/g) refers to adsorbate amount adsorbed at time (min) and at equilibrium,



(a) (b)

Fig. 5. Effect of time on adsorption of Cd (II) ions on prepared nano hydrogels in terms of (a) percentage removal and (b) (mg/ g).

Table 1. Effect of time on adsorption of Cd (II) ions on prepared nano hydrogels in terms of percentage removal as well as (mg/ g).

Time (min)	% Removal	q_e (mg/ g)
0	0	0
1	89.81462585	35.92585034
2	90.069161	36.0276644
4	90.47335601	36.1893424
6	90.69070295	36.27628118
8	91.08446712	36.43378685
10	92.20124717	36.88049887
15	92.8446712	37.13786848
20	93.04081633	37.21632653
30	93.22165533	37.28866213
45	93.27664399	37.3106576
60	93.3185941	37.32743764
90	93.32879819	37.33151927
120	93.35204082	37.34081633
180	93.35770975	37.3430839
220	93.35770975	37.3430839

correspondingly. Further, (1/min) and (g/mgmin) are rate constant for pseudo-first and second model correspondingly [37].

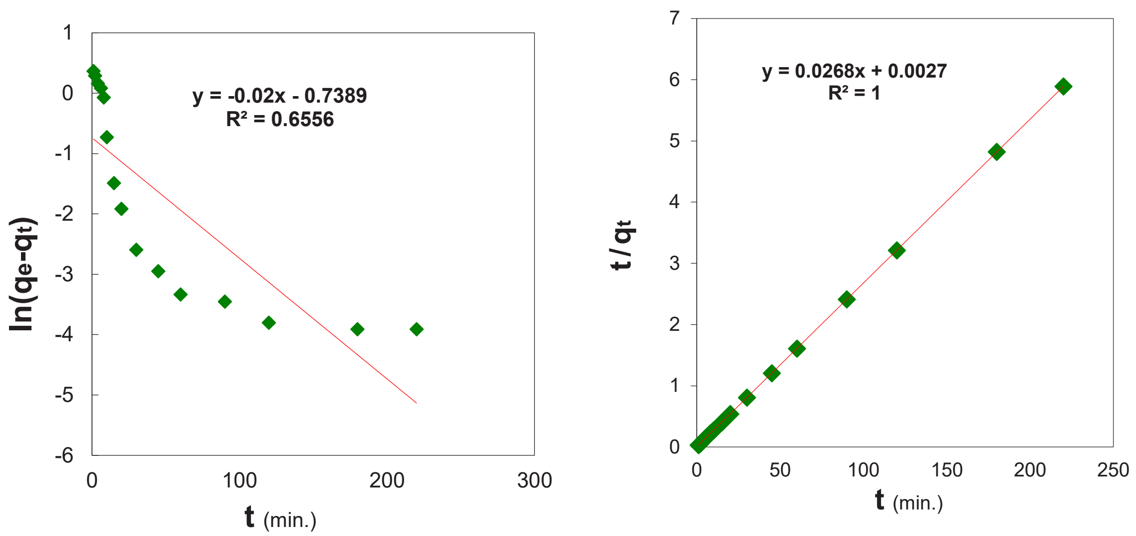
RESULTS AND DISCUSSION

Characterization study

The FTIR spectra of CS-g-P(CMC-co-AM) nano hydrogel before and after Cd (II) ion adsorption revealed significant changes, providing insights into adsorption mechanism. Before adsorption (Fig. 2), the broad peak at 3400-3200 cm^{-1} indicated the presence of hydroxyl and amide groups. After adsorption, a shift in these peak suggested interactions with Cd (II) ions. Furthermore, the peak at 2900 cm^{-1}, associated with C-H stretching, remained relatively unchanged. However, significant changes were observed in 1750-1500 cm^{-1} region [5, 17, 19, 38, 39]. Results revealed that peaks related to C=O stretching of carboxylate groups undergo shifting in both intensity and position highlighting the presence of strong interactions between adsorbent functional groups and Cd (II) ions. Peaks around 1000-1200 cm^{-1}, attributed to C-O stretching, also undergo changes, that further corroborate the contribution of hydroxyl as well as carboxylate groups in Cd (II) ions removal. This variation in peaks highlight the contribution of hydroxyl, carboxylate, and amide functional groups of prepared nano hydrogels for cadmium ions adsorption [38, 40-46].

The crystallographic study of nano hydrogel (Fig. 3) revealed the presence of broad peak present nearly at 2θ of 20°. The peak corresponds to an amorphous structure of nano hydrogel due to the lack of long-range crystallinity. The highly amorphous nature of the nano hydrogel is mainly responsible for Cd (II) ions adsorption [47].

The morphological analysis of nano hydrogel is shown in Fig. 4 highlighting the presence of numerous pores and heterogeneity in its structure. This porous structure is responsible



(a) (b)

Fig. 6. Plot of (a) pseudo first and (b) pseudo second model for adsorption of Cd (II) ions onto prepared nano hydrogels.

Table 2. Parameters calculated from pseudo first and pseudo second models. Experimental (mg/g) = 37.34 mg/g.

Model	Pseudo-first order	Pseudo-second order
q_e (mg/g)	0.477	37.31
R^2 values	0.6556	1
Constant	0.02	0.266
	K_1(1/min)	K_2 (g/mg · min)

for providing numerous adsorption sites for Cd (II) ions adsorption thereby results in improving its adsorption capacity. Study revealed that after adsorption of Cd (II) ions, the structure of nano hydrogel transit from heterogeneous to homogeneous one signifying saturation of active sites present on adsorbent surface with adsorbed Cd (II) ions [10, 48].

Kinetic modeling

The results of contact time study in terms of % removal as well as adsorption capacity are shown in Fig. 5 and Table 1 highlighting the direct relation of adsorption time with Cd (II) ions removal. With increasing time from 1 min to 220 min, there was an increment in both percentage removal (from 89.81% to 93.357%) and adsorption capacity (increased from 35.92 mg/g to 37.343 mg/g). This can be better explained by accessibility of free active sites on adsorbent that allows maximum number of ions to get adsorb on adsorbent surface. However, no significant change was observed in Cd (II) ions adsorption after 90 min and 220 min where removal percentage was 93.32% and

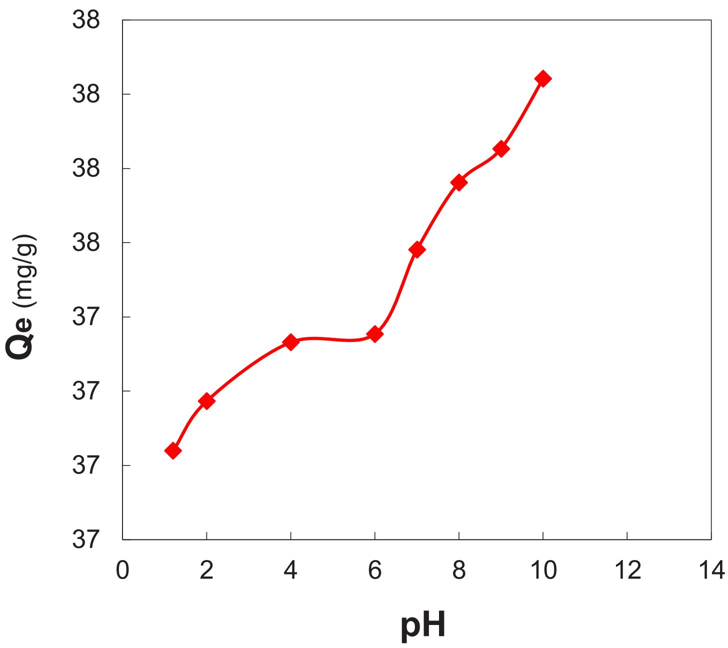


Fig. 7. Effect on pH on adsorption of Cd (II) ions on prepared nano hydrogel.

Table 3. Effect on pH on adsorption of Cd (II) ions on prepared nano hydrogel.

pH	q_e (mg/ g)
1.2	37.03922902
2	37.17301587
4	37.33174603
6	37.35442177
7	37.58117914
8	37.76258503
9	37.85328798
10	38.0414966

93.35% and adsorption capacity observed to be 37.33 mg/g and 37.343 mg/g respectively. Due to this slight variation in results, 90 minutes was selected as the optimal contact time [5].

The results of kinetic study for pseudo first order and pseudo second order model are shown in Fig. 6a and 6b respectively. It was observed that there exists greater difference between calculated (0.02 mg/g) as well as experimental [37.34 mg/g) adsorption capacities for pseudo first model. Additionally, value of regression coefficient also observed to be lesser i.e., 0.6556 when compared with the results of pseudo second order model. These finding revealed the unsuitability of pseudo first model with the studied data. Owing to R^2 = 1 and less variation between experimental q_e (37.34 mg/g) and calculated q_e (37.31 mg/g), as summarized in Table 2, pseudo second order

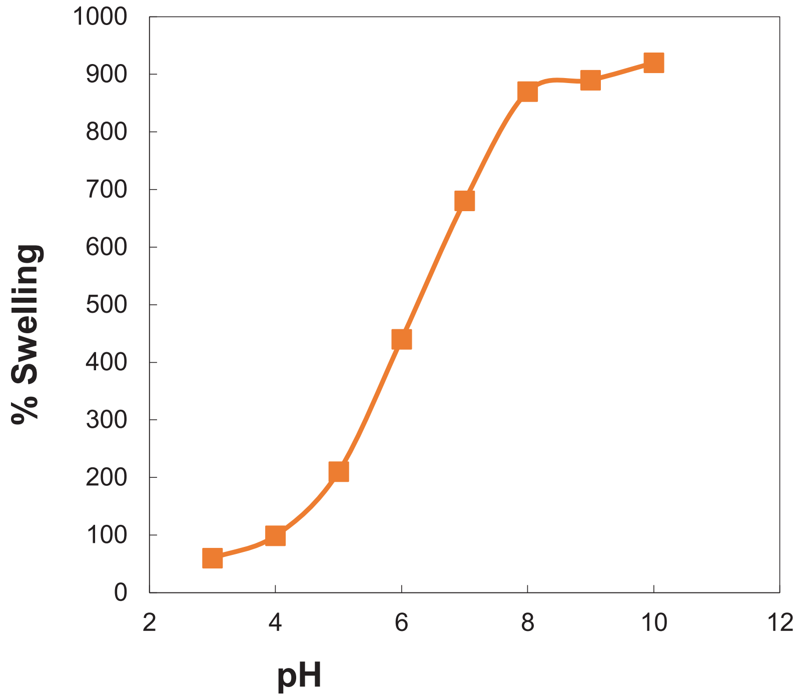


Fig. 8. Effect of pH on swelling ratio of prepared nano hydrogel.

Table 4. Effect of pH on swelling ratio of nano hydrogel.

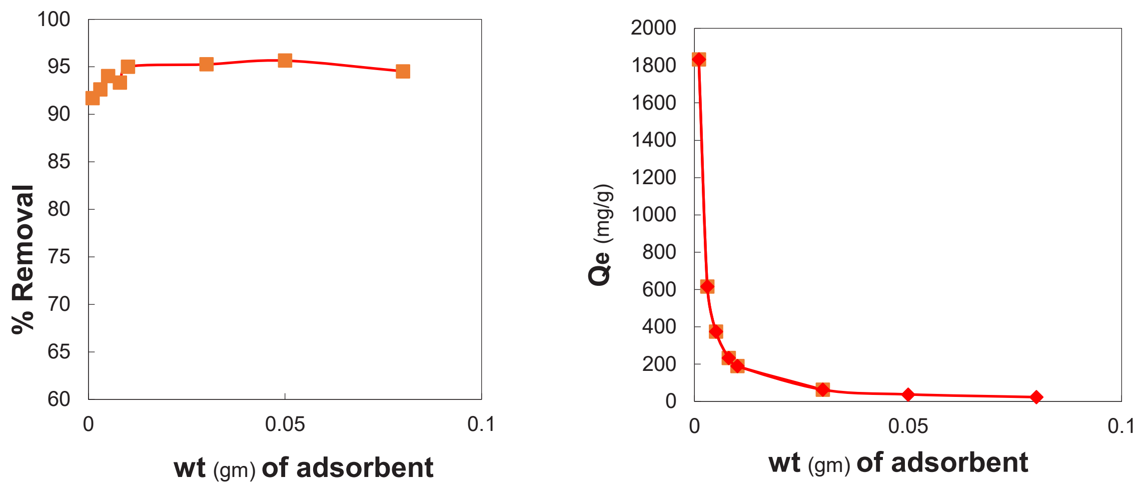
pH	Swelling %
3	60.0
4	99.0
5	210.0
6	440.0
7	680.0
8	870.0
9	890.0
10	920.0

model fits best to studied adsorption process. The fitness of pseudo second model confirmed the chemical adsorption between adsorbent surface and Cd (II) ions [49].

Effect of solution pH

Solution pH is an important parameter that affect the charge of both adsorbent surface and adsorbate thereby affecting the overall adsorption capacity of adsorbent. To optimize best pH for study, experiments were carried out by varying pH (Fig. 7 and Table 3). Results showed that by increasing pH from acidic to basic, there was an increment in adsorption capacity of nano hydrogel. Adsorption capacity of 37.03 mg/g and 38.04 mg/g was observed by increasing pH from 1.2 to 10 respectively. Reason behind this was that at lower solution pH, i.e., acidic pH, the surface of adsorbent become positively charged that result in causing repulsions between positive Cd (II) ions and positively charged adsorbent surface. However, increasing pH from acidic to basic results in inducing native charge on adsorbent surface that thereby results in adsorbing cationic Cd (II) ions via electrostatic attraction [9].

Swelling property of prepared nano hydrogel was investigated by varying solution pH from 3 to 10 (Fig. 8 and Table 4). It was observed that at lower solution pH i.e., 3, minimal swelling i.e., 60% can be observed. This swelling was increased to 920% when solution pH was 10. The reason behind this behaviour is that at lower pH values, water absorption was less due to the compact structure of nano hydrogel. However, an increase in solution pH allow for greater water uptake thus results in remarkable increase in swelling ratio [35].



(a) (b)

Fig. 9. Influence of nano hydrogel dosage on adsorption of Cd (II) ions in terms of (a) % removal and (b) (mg/ g)

Table 5. Influence of nano hydrogel dosage on adsorption of Cd (II) ions in terms of % removal and (mg/ g).

Adsorbent dose (g)	% Removal	Q_e (mg/g)
0.001	91.70805	1834.161
0.003	92.62642	617.5094
0.005	94.01345	376.0538
0.008	93.35771	233.3943
0.01	95.00737	190.0147
0.03	95.2585	63.50567
0.05	95.65533	38.26213
0.08	94.53458	23.63365

Effect of nano hydrogel dose and salt concentration

Effect of adsorbent dose on adsorption of Cd (II) both in terms of % removal and adsorption capacity were studied and results are presented in Fig. 9 and Table 5. It was observed that increasing nano hydrogel dose from 0.001g to 0.05g, there was an increment in % removal i.e., from 91.70% to 95.65% respectively. This was mainly due to the increased availability of active sites for adsorption of Cd (II) ions with an increase in adsorbent dose. However, with further increase in adsorbent dose to 0.08g, a decrease in % removal i.e., 94.53% was observed that might be due to agglomeration effect of adsorbent particles at higher dose. In terms of adsorption capacity, an inverse relation with an increase in adsorbent dose was observed i.e., by increasing dose from 0.001g to 0.08g, there was a continuous decrease in adsorption capacity from 1834.14 mg/g to 23.63 mg/g respectively. The reason of this trend was that with an increased % removal of Cd (II) ions, the availability of free actives sites for more adsorption decreased (since maximum adsorption sites are occupied by the adsorbate) thereby results in decreasing adsorption capacity [12, 35].

Adsorption capacity of prepared nano hydrogels was also investigated in presence of different salts i.e., NaCl, KCl and $CaCl_2$. Findings of study (Fig. 10 and Table 6) revealed that the adsorption capacity of adsorbent increased with an increase in

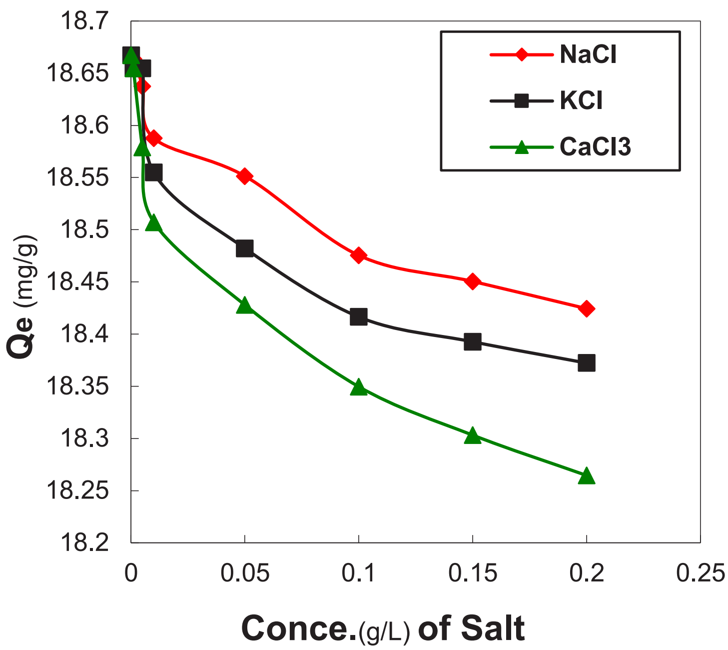


Fig. 10. Effect of different salts on adsorption of Cd (II) ions on prepared nano hydrogel.

Table 6. Effect of different salts on adsorption of Cd (II) ions on prepared nano hydrogel.

Cd (II) ions C_o	Salt concentration (g/L)	Q_e (mg/g)		
		NaCl	KCl	$CaCl_2$
200	0	18.66701	18.66701	18.66701
200	0.001	18.66361	18.65454	18.65465
200	0.005	18.63753	18.65454	18.57868
200	0.01	18.58764	18.55476	18.50726
200	0.05	18.55136	18.4822	18.42789
200	0.1	18.4754	18.41644	18.34956
200	0.15	18.45045	18.39263	18.30317
200	0.2	18.42438	18.37222	18.26451

concentration of all salts being studied. However, the maximum results were obtained with NaCl when compared with KCl and $CaCl_2$ due to the competitive effect of Cd (II) ions with ions of KCl and $CaCl_2$.

CONCLUSION

Results of the study highlight the adsorptive potential of prepared poly (carboxymethyl cellulose-co-acrylamide) (CS-g-P(CMC-co-AM)) nano hydrogel to adsorb Cd (II) ions from water. Characterization of prepared nano hydrogels revealed presence of ionic functional groups within the nano hydrogel, chiefly hydroxyl, carboxylate, and amide groups that are mainly responsible for adsorption of Cd (II) ions on its heterogeneous and amorphous surface. Results of batch adsorption studies showed that adsorption efficiency increased with an increase in contact time and equilibrium was achieved within 90 minutes at higher pH levels (i.e., 10). The process followed pseudo-second kinetic model, suggestive of chemically controlled mechanism. The study highlights that the CS-g-P(CMC-co-AM) nano hydrogel is a low-cost, efficient adsorbent for removing cadmium ions from water.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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