ARTICLE

Adsorption of Pb(II) and Cd(II) by magnetic chitosan-salicylaldehyde Schiff base: Synthesis, characterization, thermal study and antibacterial activity

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Abstract

In this paper, new magnetic chitosan-salicylaldehyde Schiff base (ChS@Fe$_2$O$_3$) was prepared and characterized by FT-IR, DSC, XRD, VSM and SEM and utilized as an efficient sorbent for the removal of Pb(II) and Cd(II) ions from aqueous solution. Moreover, the effect of solution pH, Pb(II) and Cd(II) initial concentration, adsorbent dosage and contact time on the adsorption process was examined thoroughly and optimized. Also, the adsorbent reusability and adsorption mechanism were studied. The sorption capacity of Pb(II) and Cd(II) ions at optimum condition was found to be 135.8 and 114.8 mg/g, respectively. The adsorption efficiency of ChS@Fe$_2$O$_3$ remained above 88 and 72%, respectively for Pb(II) and Cd(II) ions. Finally, antibacterial activities evaluation of ChS@Fe$_2$O$_3$ were conducted against *S. aureus*, *B. cereus*, *E. coli* and *P. aeruginosa* bacteria strains. The results exhibited the high activity against all bacteria strains.

KEYWORDS

adsorption of Pb(II) and Cd(II), chitosan Schiff base, salicylaldehyde

1 | INTRODUCTION

In recent years, water pollution by organic dyes and heavy metal ions used in various industries is one of the major challenges.[1–5] Among the existing heavy metals ions, Lead(II) and cadmium(II) are among the best toxic materials for all life forms which can cause various diseases,[6–8] due to non-biodegradable nature, bioaccumulation and also stability of them. Therefore, the removal of these ions before entering the environment is essential.[6–8] So far, many methods have been used and developed for heavy metal ions removal from wastewater.[6–13] Among them, adsorption is one of the most promising, easy operation, economical and cheapest technique.[6–8,12,13] The most effective parameter in the adsorption process is the type of adsorbent used such as Cyclam-modified magnetic SBA-15, grapheme oxide modified with 2, 2′-dipyridylamine, mesoporous silica/dithizon and etc.[14–18] Chitosan, as a natural polysaccharide polymer,[19,20] due to its high amino and hydroxyl groups, biodegradability and non-toxicity, is the most suitable sorbent in the adsorption process.[21,22] Low solubility, non-porous structure and small surface area reduce the use of chitosan in different applications. To solve these problems and also improve its stability, flexibility and enhance absorption capacity, chitosan modified with the help of various additives such as aldehydes and nanoparticles by physical and chemical methods.[12,13,23–31] Recently, different magnetic chitosan Schiff bases reported as an efficient sorbents for Pb(II) and Cd(II) ions.[7,8,12,13,28–32] For example, thiosemicarbazide modified chitosan[31] could be adsorb Pb(II) and Cd(II) efficiently at pH solution of 4.3 and 6, respectively, with the maximum capacity sorption of 325.2 mg/g for Pb(II) and 257.2 mg/g for Cd(II). New Pb(II) sorbent of magnetic chitosan-(D-glucosimine methyl) benzaldehyde Schiff base (MCS-Sch) prepared by Shahraki and Delarami[32] and the maximum adsorption capacity obtained 121.95 mg/g. Yan et al rport the maximum sorption capacity value of 86.20 mg/g for Pb(II) ion using epichlorohydrin crosslinked chitosan Schiff base@Fe$_3$O$_4$.[6] Yuvaraja and Subbaiah reported the maximum monolayer sorption capacity of 104.16 mg/g for Pb(II) ion using magnetic chitosan-4-([pyridine-2-ylimino]methyl)benzaldehyde Schiff's base.[7] Liu et al,[33] prepared novel *N*-methylene phophonic acid chitosan with hyperbranched polyethyleneimine (NPCS-PEI). The maximum adsorption capacity of Cu(II) and Pb(II) on the NPCS-PEI are 276.12 and 645.16, respectively. Yang et al.[34] prepared chitosan-based bead by crosslinking as well as sulfhydryl modification reaction and studied its removal activity of Pb(II), Cu(II) and Cd(II). One of the most important advantages of magnetic chitosan Schiff bases, is their easily separated from solution using an external magnet and finally resulting the colorless and clear supernatant.[6–8,32]

Biological activity is other unique properties of modified chitosan.[23,26,27] Due to the presence of OH and NH$_2$ active groups, chitosan easily modified via coupling with aldehyde or ketone[35] to produce Schiff base compounds. They shows more inhibition potency against various types of bacteria.[35–37] Recently, Foroughnia et al[37] prepared new chitosan Schiff base-Fe$_2$O$_3$ nanocomposite and reported its antibacterial activity against various Gram-positive and Gram-negative bacteria.[37]

The aim of this paper was designed to prepare new magnetic chitosan Schiff base ChS@Fe$_2$O$_3$ (Scheme 1) as an environmental friendly and reusability sorbent for removal of Pb(II) and Cd(II) ion from aqueous solution. In addition, antibacterial activity of ChS@Fe$_2$O$_3$ was studied against various bacteria strains.
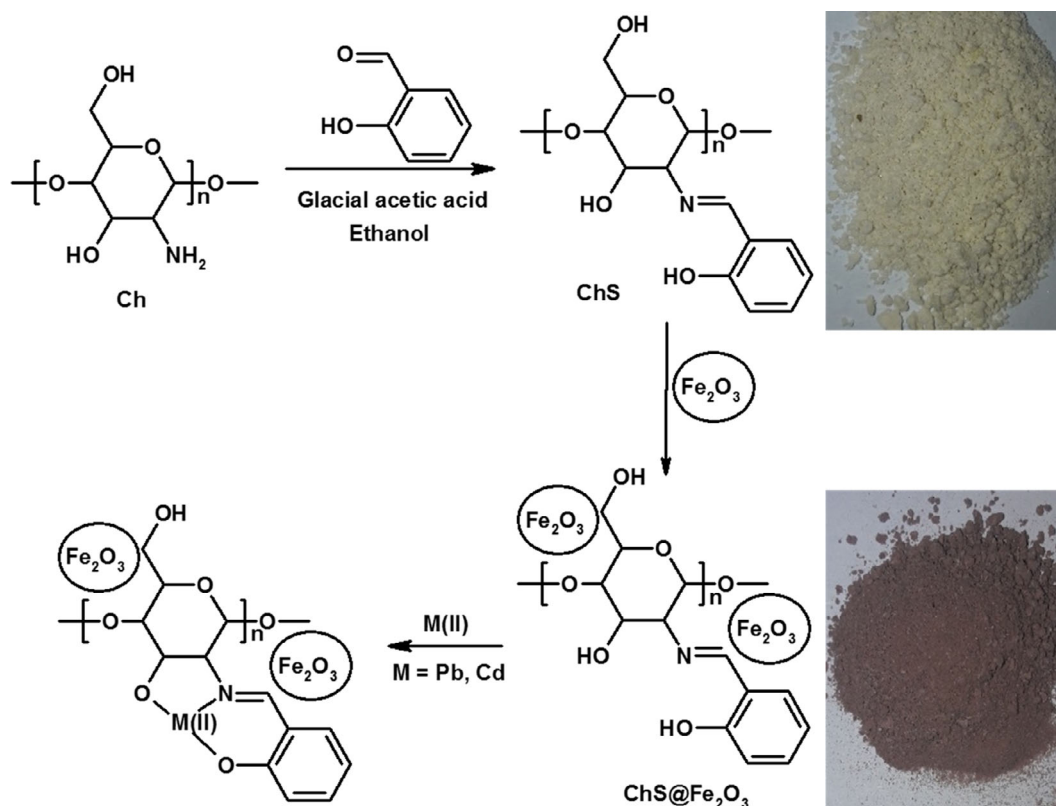
2 | EXPERIMENTAL

2.1 | Chemical and materials

Chitosan with low molecular weight was purchased from Sigma-Aldrich. Glacial acetic acid, ethanol, NaOH and 2-hydroxybenzaldehyde were purchased Merck. All regents used without further purification. FT-IR spectrum (400–4,000 cm^{-1}) was recorded with KBr disk using a Perkin-Elmer spectrophotometer. XRD pattern was recorded by Bruker AXS-D8 Advance diffractometer. FE-SEM images were carried out by TESCAN microscope model MIRA III (Czech Republic) with 10 kV electron beam. The magnetic behavior was analyzed with a vibrating sample magnetometer (VSM, Lake Shore Cryotronics-7,404). DSC curve was recorded by DSC analyzer model 60A, Shimadzu. The concentration of Pb(II) and Cd(II) ions in the solution was determined by using an atomic absorption spectrophotometer (Shimadzu AA-6300). Fe$_2$O$_3$ nanoparticles was synthesized according to the previous literature.[38,39]

2.2 | Synthesis of ChS@Fe$_2$O$_3$

1 g of chitosan was dissolved in 25 ml of 5% glacial acetic acid solution under stirring at 70°C for 0.5 hr. The 10 ml ethanolic solution of 2-hydroxybenzaldehyde was added and the mixture was stirred for about 6 hr. In the following, 0.25 g of Fe$_2$O$_3$ nanoparticles[38,39] was added and the mixture was sonicated for 1 hr followed by stirring for another 24 hr at 70°C. After that, the mixture is added into a dilute NaOH solution to product precipitated faster. Finally, desired product was filtered, washed several times using cold water and dried under vaccum for 24 hr. The as-prepared brown product was named as ChS@Fe$_2$O$_3$.

SCHEME 1 The procedure for the synthesis of ChS@Fe$_2$O$_3$ and the mechanism of adsorption of Pb(II) and Cd(II) onto ChS@Fe$_2$O$_3$ sorbent

2.3 | Adsorption studies

All experiments were performed in a series of 50 ml Erlenmeyer flasks containing of ChS@Fe$_2$O$_3$ and 25 ml of metal ions solution. The effects initial metal ions concentration (30, 50, 70, 90 and 110 ppm), agitation time (0–120 min), sorbent dosage (0.1, 0.3, 0.5, 0.7 and 0.9 mg/ml), and pH (1–9) has been considered. The pH solutions were adjusted by using 0.1 M HCl or NaOH. The mixture in flasks were agitated using mechanical shaker with as speed of 150 rpm and the final concentration of metal ions in the filtered was analyzed using atomic absorption spectrophotometer at every 15 min. The removal percentage (%) ad adsorption capacity (q_t) have been determined using the following equations, where, C_i and C_f were the initial and final Pb(II) concentration (mg/L), respectively. V is the volume of solution (L) and M is the mass sorbent (g).

$$R\,(\%) = \frac{(Ci - Cf) \times 100}{Ci} \tag{1}$$

$$q_t\,(\text{mg/g}) = \frac{(Ci - Cf) \times V}{M} \tag{2}$$

2.4 | Antibacterial studies

Agar-well diffusion method was applied for screening the antimicrobial activities.[22] The solutions of all compounds were prepared and then incubated at 37°C for 24 hr. Then, the plates were taken out.[22,24,37]

3 | RESULTS AND DISCUSSION

3.1 | Characterization

FT-IR spectra of Fe$_2$O$_3$, ChS, and ChS@Fe$_2$O$_3$ were recorded in the range of 400–4,000 cm^{-1} shows in Figure 1. In the spectrum of Fe$_2$O$_3$ (Figure 1a), two peaks appeared 467 and 553 cm^{-1} represented the Fe-O vibration.[38] ChS showed three peaks at 3425, 1692 and 1,580 cm^{-1} assigned to the stretching vibration of —OH, —C=N— and —CH$_2$- groups, respectively (Figure 2b).[6-8,37] In the FT-IR spectrum of ChS@Fe$_2$O$_3$ (Figure 1cv), the peak at 3427 cm^{-1} assigned to the OH stretching vibration.[6-8,37] The sharp peak appeared at 1680 cm^{-1} assigned to the imine (—C=N—) group and the peak at 1578 cm^{-1} assigned to the C—H stretching bending of CH$_2$ groups.[7] The C—O—C

FIGURE 1   FT-IR spectrum of
(a) Fe_2O_3, (b) ChS and (c) ChS@Fe_2O_3
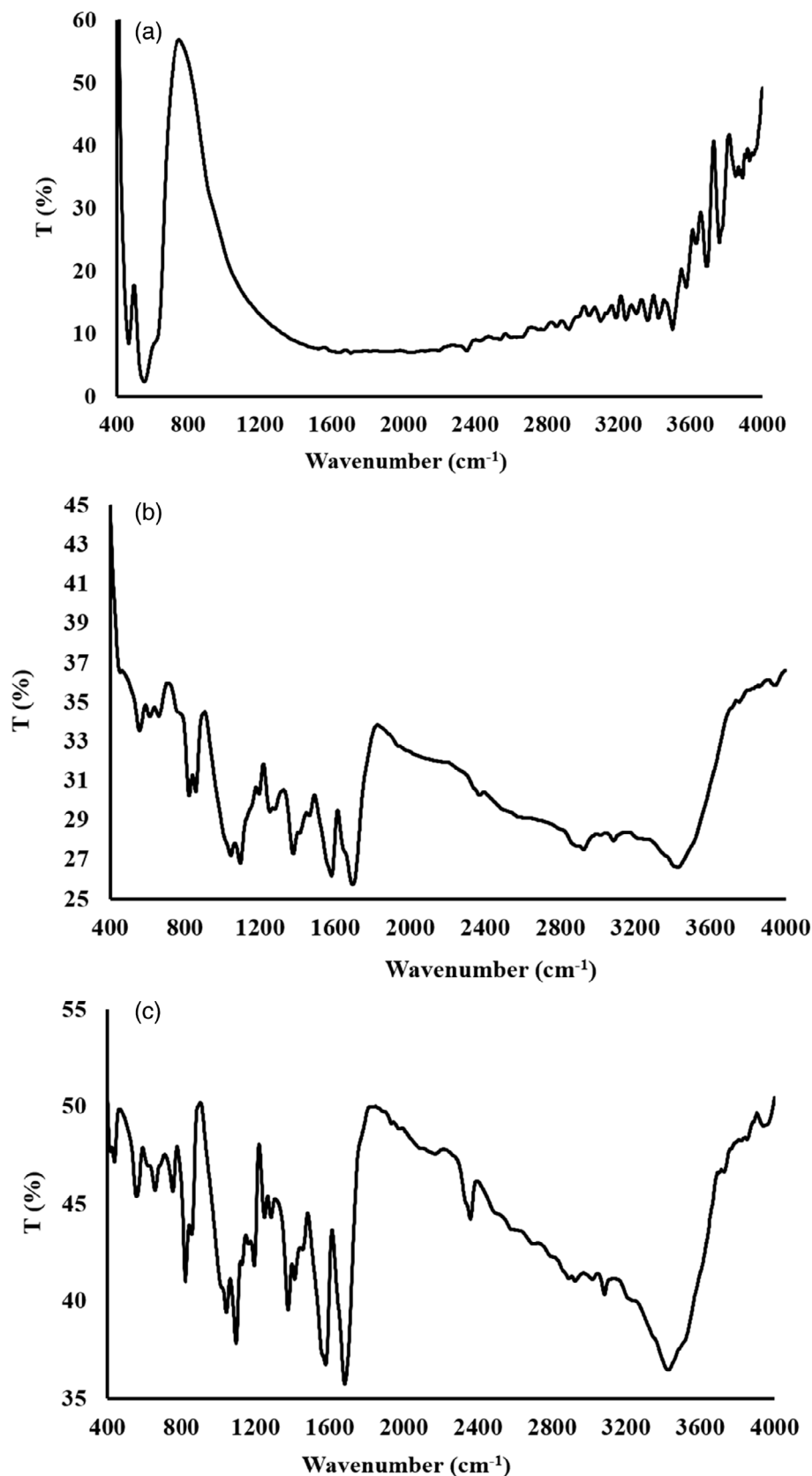
stretching band appeared at 1095 cm^{-1}[7] and the C—H out of plane bending of aromatic group appeared at 821 cm^{-1}.[40,41]

XRD patterns of Fe_2O_3 and ChS@Fe_2O_3 were recorded from $2\theta = 10$ to 70, represented in Figure 2. In the XRD of Fe_2O_3 (Figure 2a), the characteristic peaks

appeared at $2\theta \approx 24.12, 33.18, 36.63, 41.22, 49.76, 54.55$, and $63.49°$ could be assigned to (012), (104), (110), (113), (024), (116), and (214) planes of rhombohedral of hematite (JCPDS no. 33–0664).[38,39] ChS@Fe$_2$O$_3$ showed two peaks at $2\theta \approx 13$ and $20°$, confirm the preparation of chitosan Schiff base.[6–8,31,32] Also, in XRD pattern of ChS@Fe$_2$O$_3$, the characteristic peaks of Fe$_2$O$_3$ was observed indicates that the crystal structure of Fe$_2$O$_3$ was not destroyed after preparation of ChS@Fe$_2$O$_3$.[32] The average particle size (D) of Fe$_2$O$_3$ nanoparticles was calculated using Debye-Scherer following Equation,[39] where λ is the X-ray wavelength, β is the FWHM and θ is diffraction angle. The as-synthesized Fe$_2$O$_3$ nanoparticles showed the average particle size of 33.5 nm.

$$D = \frac{0.94\,\lambda}{\beta\cos\theta} \tag{3}$$

Surface morphology is an important parameter for adsorption capacity of heavy metal ions such as Pb(II), Cd(II) and Cu(II).[31,40,42–44] Figure 3 shows the SEM images of Fe$_2$O$_3$ and ChS@Fe$_2$O$_3$. As seen in Figure 3a, the morphology of Fe$_2$O$_3$ is irregular with well narrow size distribution. However, the agglomeration of Fe$_2$O$_3$ nanoparticles was also found. The morphology of ChS@Fe$_2$O$_3$ (Figure 3b) shows that Fe$_2$O$_3$ nanoparticles distributed inside and/or on the surface of ChS. The surface morphology of ChS@Fe$_2$O$_3$ nanocomposite showed highly porous and is rougher.[32]

The magnetic property of Fe$_2$O$_3$ and ChS@Fe$_2$O$_3$ nanocomposite was studied using a VSM and represented in Figure 4. Hysteresis loops of pure Fe$_2$O$_3$ and ChS@Fe$_2$O$_3$ nanocomposite were observed between ±15 KOe and shown a maximum magnetization saturation (M$_s$) of 14.66 and 9.55 emu/g, respectively, confirmed the

M$_S$ value of ChS@Fe$_2$O$_3$ was lower than the M$_S$ value of pure Fe$_2$O$_3$, due to the decrease of Fe$_2$O$_3$ percentage in ChS@Fe$_2$O$_3$[45] and also depended to size effect and particle crystallization.[7,8] Furthermore, the M$_s$ value of ChS@Fe$_2$O$_3$ nanocomposite could ensure easy application for Pb(II) and Cd(II) removals[23] and simple separation from aqueous solution using an external magnetic and finally resulting the clear and colorless supernatant.[6–8,45]
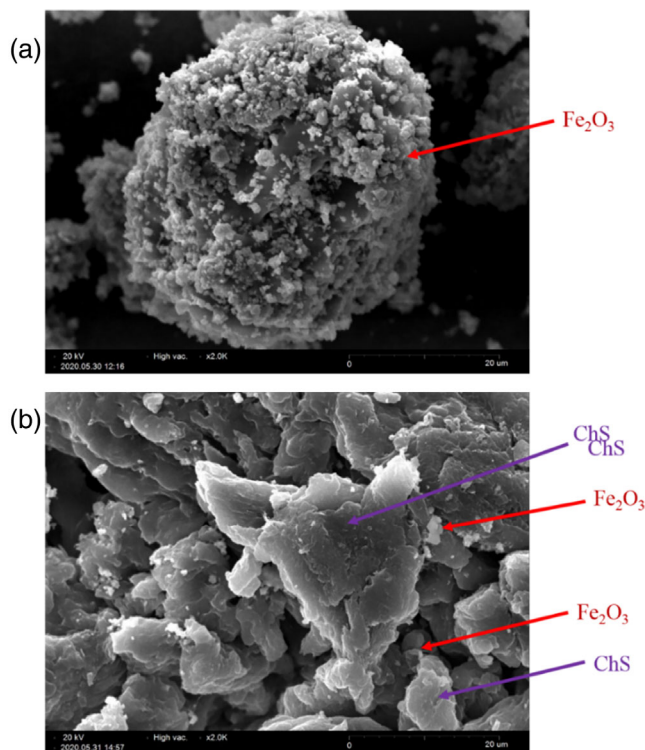

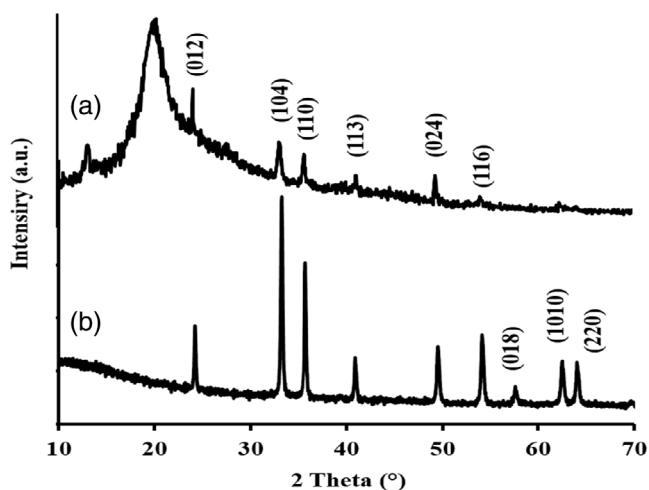FIGURE 3 SEM images of (a) Fe$_2$O$_3$ and (b) ChS@Fe$_2$O$_3$

FIGURE 2 XRD patterns of (a) Fe$_2$O$_3$ and (b) ChS@Fe$_2$O$_3$
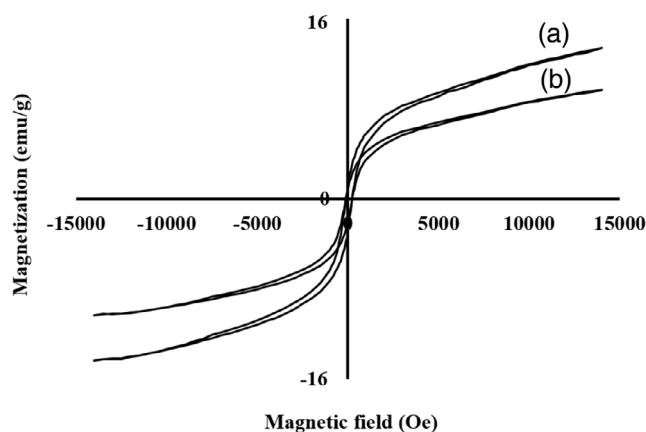

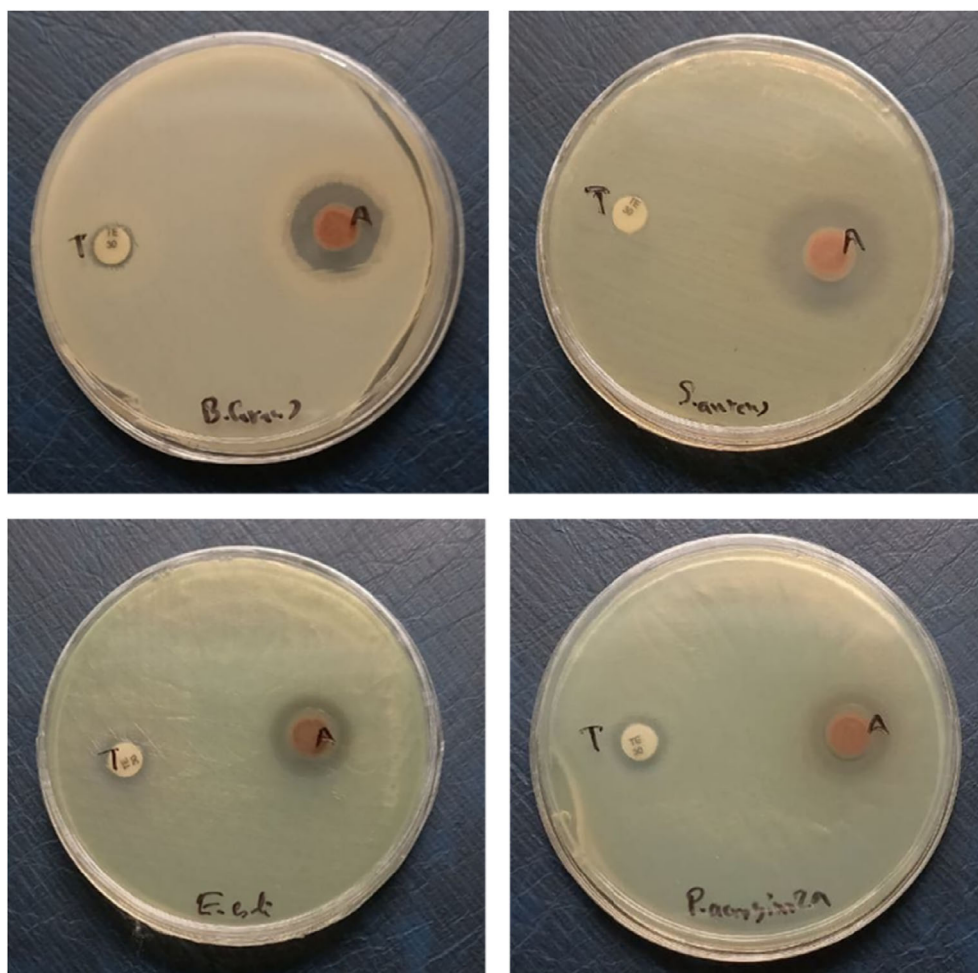FIGURE 4 VSM analysis of (a) Fe$_2$O$_3$ and (b) ChS@Fe$_2$O$_3$

FIGURE 5 Antibacterial activity of ChS@Fe₂O₃ nanocomposite (A) and tetracycline (T)

3.2 | Antibacterial results

The antibacterial activity of ChS@Fe₂O₃ was studied against different pathogenic bacteria (*S. aureus*, *B. cereus*, *E. coli* and *P. aeruginosa*) (Figure 5). The title compound ChS@Fe₂O₃ showed a weak antibacterial activity against *P. aeruginosa* and *E. coli*, while showed a good antibacterial activity against *S. aureus* and *B. cereus*. The order of antibacterial effect of this compound at the presence of bacterial strains is as follows:

S. aureus > *B. cereus* > *E. coli* > *P. aeruginosa*

3.3 | Pb(II) and Cd(II) ions adsorption

The solution pH is one the most important parameters on the sorption capacity of the metal ions from aqueous solution because of the protonation of the functional active group of sorbent and also the speciation of metal ions in solution.[6–8,31,32] In this paper, pH solution range of 1–9 was selected to the sorption of initial concentration of 70 ppm for Pb(II) and Cd(II) metal ions, 0.5 mg/ml of sorbent and shaking time of 90 min.
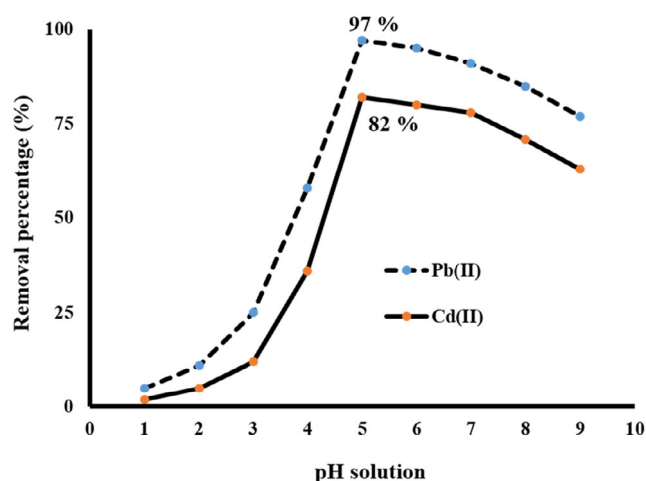

FIGURE 6 Removal percentage of Pb(II) and Cd(II) as a function of pH solution (initial concentration: 70 ppm; sorbent dosage: 0.5 mg/ml and shaking time: 90 min)

As seen in Figure 6, increases of pH from 1 to 5, the removal percentage increased and achieved to maximum values of 97 and 82% for Pb(II) and Cd(II) respectively.[6–8,32]
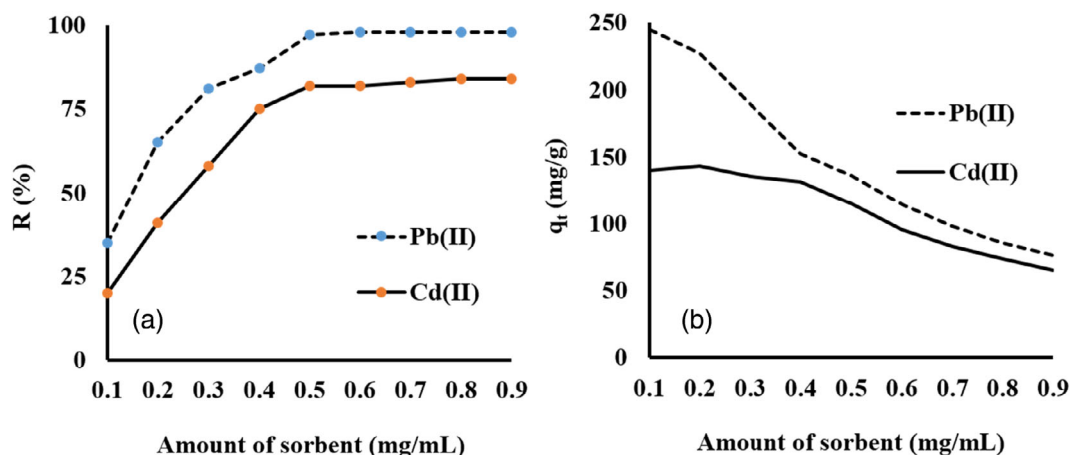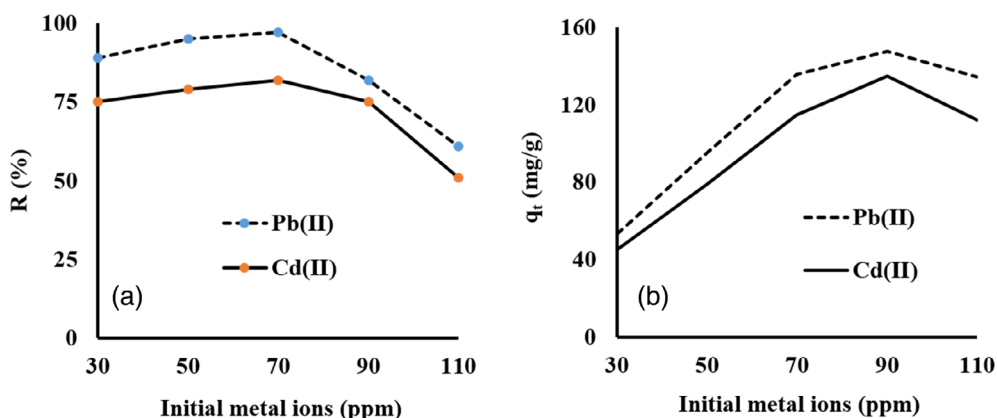
FIGURE 7 (a) Removal percentage (%) and (b) adsorption capacity (mg/g) of Pb(II) and Cd(II) as a function of sorbent concentration (initial concentration: 70 ppm; pH solution: 5 and shaking time: 90 min)

FIGURE 8 (a) Removal percentage (%) and (b) adsorption capacity (mg/g) of Pb(II) and Cd(II) as a function of initial metal ions (sorbent dosage: 0.5 mg/ml; pH solution: 5 and shaking time: 90 min)

At low pH (or high concentration of H^+), the active sites on the surface of sorbent was blocked using protonation and cannot coordinated to the Pb(II) and Cd(II) ions. At this condition, the low physical adsorption of Pb(II) and Cd(II) ions cause the removal of them at about 5 and 2% at solution pH of 1. By increasing of pH to 5, deprotonation of active sites of sorbent was happened and coordinated to metal ions and form polymeric complexes. After that, increase of pH solution (pH ≥ 6) cause the precipitation of metal ions because of $M(OH)^+$ and/or $M(OH)_2$ formation.[6–8,32] At pH of 9, the percentage removal of Pb(II) and Cd(II) found to be 77 and 63%, respectively. According to these results, pH solution of 5 was selected to investigate the effects of other parameters.[7,8]

By increasing of sorbent dose, the active sites for coordination to metal ions have been increased, then removal percentage of metal ions from aqueous solution were increased.[6–8,32,41] In this paper, the effect of sorbent dose has been studied by changing of sorbent from 0.1 to 0.9 mg/ml and represent in Figure 7. At low concentration of sorbent,

saturation of active site of sorbent was happened very soon and the residual metal ions in the solution is large.[8] Increase of sorbent can provide the more number of active sites, then providing the increase of removal percentage of metal ions.[6–8] Maximum of removal percentage observed for using of 0.5 mg/ml concentration of sorbent. After that, using of high concentration of sorbent (> 0.5 mg/ml), the number of unoccupied active sites on the surface of sorbent cannot contribute to the removal of metal ions and also cause aggregation of sorbent.[32]

The effect of initial concentration of Pb(II) and Cd(II) ions on removal percentage using as-prepared ChS@Fe$_2$O$_3$ nanocomposite was evaluated and represented in Figure 8. Maximum percentage removal is achieved at 70 ppm. As seen in Figure 8, the removal percentage of Pb(II) and Cd(II) ions with ChS@Fe$_2$O$_3$ sorbent increase when initial concentration increase from 30 to 70 ppm, because of a larger driving force to overcome mass transfer.[8,26] At low metal ion concentration (≤ 70 ppm), coordination of them to the high active sites
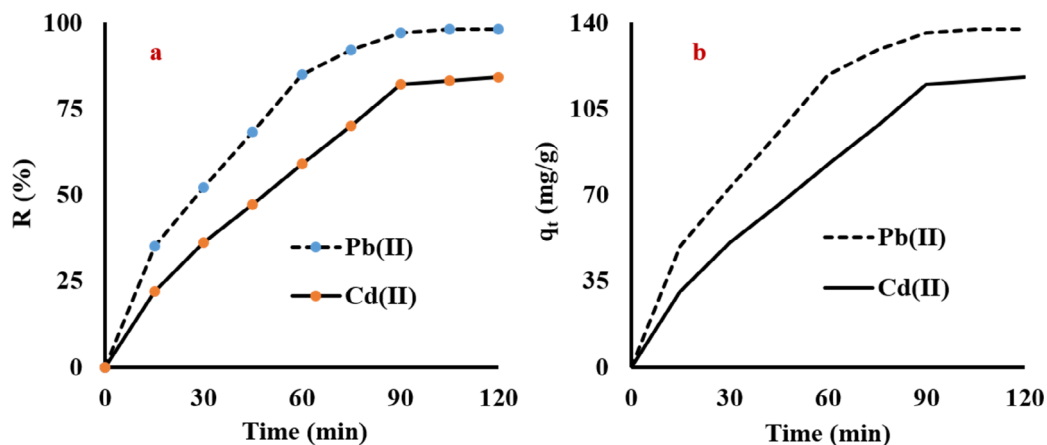
FIGURE 9 (a) Removal percentage (%) and (b) adsorption capacity (mg/g) of Pb(II) and Cd(II) as a function of shaking time (initial concentration: 70 ppm; sorbent dosage: 0.5 mg/mL; and pH solution: 5)

TABLE 1 Comparison of adsorption capacity of chitosan based sorbents on Pb(II) and Cd(II) removal

	Adsorption capacity (mg/g)		
Sorbents	Pb(II)	Cd(II)	References
Thiosemicarbazide modified CS	325.2	257.2	[31]
N-Ch-Sal	123.67	63.11	[40]
CSB@Fe$_3$O$_4$	83.33	—	[8]
MCS-Sch	121.95	—	[32]
ECCCSB@Fe$_3$O$_4$	86.20	—	[6]
ZnO@chitosan	476.1	135.1	[43]
CS-Fe$_2$O$_3$	214.9	204.3	[44]
Chitosan/nanocellulose	252.6	—	[46]
Xanthin/cellulose	134.4	—	[47]
NPCS-PEI	645.16	—	[33]
DMSA-CS	273.7	183.1	[34]
MCS-ITMB	134.10	—	[45]
ACSSB@ZnO	55.55	—	[41]
N-Ch-Sal	123.67	63.71	[48]
ChS@Fe$_2$O$_3$	135.8	114.8	This paper

of ChS@Fe$_2$O$_3$ sorbent to prepared coordination polymer was easy.[6] At high metal ion concentration (>70 ppm), due to the saturation of the active sites of ChS@Fe$_2$O$_3$ sorbent the removal percentage of Pb(II) and Cd(II) decreased[6] and/or remains unaffected.[7]

Shaking time is an important parameters to removal percentage of metal ions using sorbents.[6–8,30,31] All of reports confirm that the removal percentage on sorbent increased with increasing shaking time. As seen in Figure 9 equilibrium was reached at 90 min and afterwards, there is no any significant adsorption. Removal of Pb(II) ion is faster than Cd(II) ion because of the size of Pb(II) is larger than Cd(II) and can easily coordinated to the active sites on the surface of ChS@Fe$_2$O$_3$ sorbent.[31]

The adsorption capacity of ChS@Fe$_2$O$_3$ for Pb(II) and Cd(II) at optimal condition were found to be 135.8 and 114.8 mg/g, respectively. Table 1 compared the performance of ChS@Fe$_2$O$_3$ sorbent with other chitosan derivatives in different experimental conditions. Different adsorption capacities for various sorbents reported in Table 1 predicts the Pb(II) and Cd(II) removal was affected on the type, surface structure and also functional groups on the sorbents.[6–8,37,40]

The heavy metal ions removal using various chitosan based sorbents happened by polymeric complex formation using reaction of active sites on the sorbent and metal ions.[31,32,40] In this work, removal of Pb(II) and Cd(II) ions may be take place through both physical and
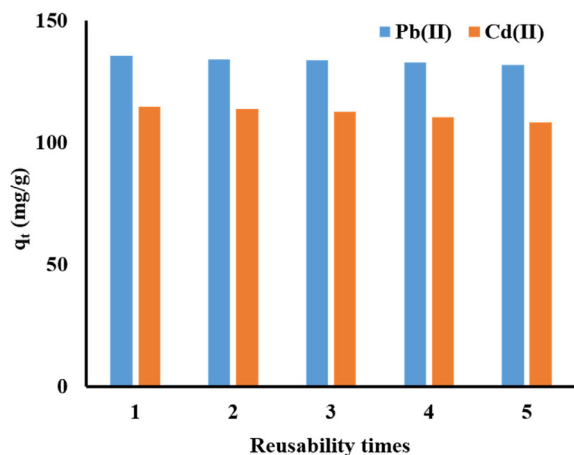
FIGURE 10 The effect of reusability times of ChS@Fe$_2$O$_3$ on adsorption capacity (mg/g) of Pb(II) and Cd(II) (initial concentration: 70 ppm; sorbent dosage: 0.5 mg/ml; shaking time: 90 min and pH solution: 5)

FIGURE 11 The effect of other ions on adsorption capacity (mg/g) of Pb(II) and Cd(II) (initial concentration: 70 ppm; sorbent dosage: 0.5 mg/ml; shaking time: 90 min and pH solution: 5)
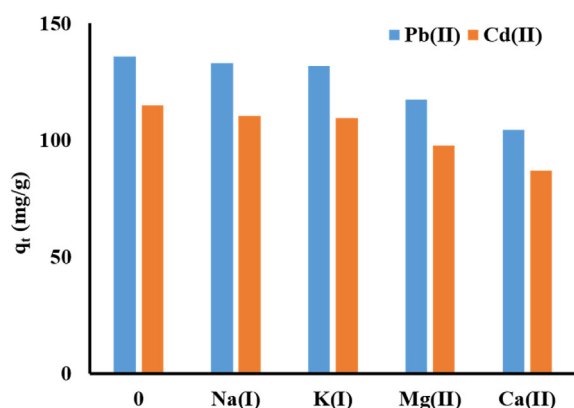
chemical adsorption.[33,34] However, complexation is favorite mechanism for removal of metal ions.[41] The chemical adsorption mechanism is proposed in Scheme 1.

To justify the use of the synthesized adsorbent is economical and feasible, the removal of Pb(II) and Cd(II) ions by the as-synthesized ChS@Fe$_2$O$_3$ as adsorbent after several uses was investigated (Figure 10). Adsorbent recovery after use was carried out using 0.1 M HCl solution.[44] As seen in Figure 10, after 5 adsorption–desorption cycles, the maximum of removal Pb(II) and Cd(II) ions remained 88 and 72%, respectively, indicating that the ChS@Fe$_2$O$_3$ is well adsorbent for Pb(II) and could be used repeatedly.[43,44,46,49]

Finally, the effect of other cations such as Na(I), K(I), Ca(II) and Mg(II) on Pb(II) and Cd(II) ions adsorption is

shown in Figure 11. As seen in this Fig, monovalent Na(I) and K(I) ions have little effect on adsorption capacity of Mg(II) on Pb(II), due to the lower tendency of these ions to coordinate with the active groups of ChS@Fe$_2$O$_3$. While, divalent Mg(II) and Ca(II) cations decrease the adsorption capacity. The effect of Ca(II) is higher than Mg(II), due to the its ionic radius is larger than that of Mg(II) and it coordinates better with active groups on the ChS@Fe$_2$O$_3$. A similar result has been reported using cellulose xanthate as adsorbent.[47]

4 | CONCLUSIONS

In summary, a new magnetic chitosan based sorbent for the removal of Pb(II) and Cd(II) was synthesized and characterized. XRD and FT-IR results confirm the preparation of ChS@Fe$_2$O$_3$ nanocomposite. VSM predict the ferromagnetic property. Antibacterial activity of ChS@Fe$_2$O$_3$ show that the well activity on all bacterial strains with order as follows: *S. aureus* > *B. cereus* > *E. coli* > *P. aeruginosa*. The optimal conditions for sorption performance of ChS@Fe$_2$O$_3$ were pH = 5, shaking time = 90, sorbent dose of 0.5 mg/ml, and initial metal ions concentration of 70 ppm. The maximum sorption capacity for Pb(II) and Cd(II) found to be 135.8 and 114.8 mg/g, respectively. ChS@Fe$_2$O$_3$ shows well reusability and good sorbent at the presence of monovalent Na(I) and K(I) cations for removal of Pb(II) and Cd(II) ions. However, divalent Mg(II) and Ca(II) cations reduce the adsorption capacity of Pb(II) and Cd(II) ions. This study showed that the ChS@Fe$_2$O$_3$ could be a promising sorbent materials for toxic and stable Pb(II) and Cd(II) ions from aqueous solution. Therefore, this adsorbent can be useful for effective and efficient removal of other heavy metal ions such as Hg(II), Cu(II), Cr(III) and Zn(II).

CONFLICT OF INTEREST
The authors declare that there is no conflict of interest.

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