A comparative study on Fe(III)-chitosan and Fe(III)-chitosan-CTAB composites for As(V) removal from water: preparation, characterization and reaction mechanism

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Abstract
Fe(III)-chitosan and Fe(III)-chitosan-CTAB composites were prepared using an ionotropic gelation method. Various techniques were used to analyze the morphology, structure, and property of the adsorbents, including SEM, EDS, FT-IR, XPS, and zeta potential. Compared with Fe(III)-chitosan, Fe(III)-chitosan-CTAB was more effective for As(V) adsorption at a wide range of pH (3.0–8.0). The adsorption of As(V) onto Fe(III)-chitosan and Fe(III)-chitosan-CTAB could reach equilibrium in 20 min, and their maximum adsorption capacities were 33.85 and 31.69 mg g⁻¹, respectively. The adsorption kinetics was best described by the pseudo-second-order model ($R^2 = 0.998$ and 0.992), whereas the adsorption isotherm was fitted well by the Freundlich model ($R^2 = 0.963$ and 0.987). The presence of $H_2PO_4^-$ significantly inhibited the adsorption of As(V) onto Fe(III)-chitosan and Fe(III)-chitosan-CTAB, and humic acid also led to a slight decrease in As(V) adsorption by Fe(III)-chitosan-CTAB. Over 94% of As(V) at the initial concentration of no more than 5 mg L⁻¹ was removed from real water by the two adsorbents. 1% (w/v) NaOH solution was determined to be the most suitable desorption agent. Fe(III)-chitosan and Fe(III)-chitosan-CTAB still maintained their initial adsorption capacities after five adsorption–desorption cycles. Based on different characterization results, both electrostatic attraction and surface complexation mechanisms played important roles in As(V) adsorption on Fe(III)-chitosan and Fe(III)-chitosan-CTAB.

Keywords Adsorbent · Adsorption · Arsenic · Cationic surfactant · Toxic metal

Introduction
Arsenic (As) is a common occurring environmental pollutant that enters aquatic ecosystems through both geological processes and anthropogenic activities (Smedley and Kinniburgh 2002). It has attracted widespread attention because of its toxicity and carcinogenicity (Clancy et al. 2013). The toxicity of As is related to its chemical form (Sarwar et al. 2021). Inorganic As (iAs) tends to be more toxic than organic As (oAs) (Li et al. 2018). iAs generally exists in two predominant oxidation states, arsenate [As(V)] and arsenite [As(III)]. As(III) is the major species in reductive environment, while As(V) is the predominant species under oxidative environment. Unfortunately, an estimated more than 296 million people worldwide, which include 200 million from Asia (India, Bangladesh, China, Vietnam, Cambodia, Pakistan, and Nepal), are potentially exposed to iAs contamination above the World Health Organization (WHO) guideline value of iAs in drinking water (0.01 mg L⁻¹) (Shaji et al. 2021). Given the risk of chronic iAs exposure via drinking water, it is necessary to develop proper remediation technologies.

Many technologies have been applied to the remediation of iAs-contaminated water, including electrocoagulation, chemical precipitation, membrane filtration, adsorption, ion exchange, and bioremediation (Dadwal and Mishra 2017). Among these technologies, adsorption is an easy but effective and economical method (Gadd 2009). Over the last decades, a variety of natural and synthetic
adsorbents has been used for the removal of iAs from water. Owing to their easy availability and low cost, natural materials such as zeolite, clay, chitosan, and agricultural waste were used as adsorbents to remove metal ions (Gupta et al. 2021). Chitosan, a unique cationic polysaccharide, has received attention as a renewable, nontoxic, and biodegradable chelating agent for some metal ions (Bakshi et al. 2020). It could provide distinctive adsorption functions because of the existence of active hydroxyl (−OH) and amino (−NH₂) groups in its molecular structure (Liu et al. 2013). However, chitosan is only soluble in few organic acids and dilute inorganic acids, such as acetic acid and dilute hydrochloric acid (HCl), which limits its utilization for a specific application. The adsorption capacity and selectivity of chitosan are also still unsatisfactory (Alves and Mano 2008). Surface modification of chitosan is thus required to overcome these limitations.

There has recently been growing concern about the chemical modification of chitosan to improve its favorable properties. Both cross-linking and grafting are considered as the most useful methods to improve native properties of chitosan, such as enhancing complexation property and increasing the adsorption capacity (Saheed et al. 2021; Nourmohammadi et al. 2021). Iron (Fe) is a commonly used metal ion for ionic cross-linking of chitosan. The crosslinked Fe(III)-chitosan has exhibited superior performance towards removing Cr⁶⁺ from aqueous solution with pH 2.0 (Demarchi et al. 2015). Fe(III)-chitosan synthesized by Shinde et al. (2013) showed better adsorption efficiency for As(V) than La(III)-chitosan. Chemical modification of magnetic chitosan composites is also well studied in the previous literature (Baghban et al. 2017). Furthermore, chitosan modification utilizing ionic surfactants (e.g., cetyltrimethyl ammonium bromide (CTAB) and sodium dodecyl sulfate (SDS)) also could make adsorbents with large adsorption capacity, high selectivity, and good stability. It has been reported that the chitosan beads impregnated with 0.05% CTAB had higher adsorption capacity for Congo red than the normal chitosan beads (Chatterjee et al. 2009). This was partly due to the electrostatic interaction between CTAB and Congo red, and partly because of the hydrophobic interaction between the cationic surfactant and dye. Nevertheless, little literature is available on the utilization of cationic surfactant CTAB to improve the removal efficiency of anionic As(V) by Fe(III)-chitosan.

In this study, Fe(III)-chitosan and Fe(III)-chitosan-CTAB composites were synthesized, characterized, and applied for anionic As(V) removal. As(V) adsorption property of the two adsorbents was investigated based on different factors like pH, contact time, initial As(V) concentration, and coexisting ions. The experimental data were fitted with typical kinetic and isotherm models to evaluate the adsorption mechanism. In addition, the regeneration of the adsorbents was used repeatedly up to five cycles for examining their reusability.

Materials and methods

Materials

Chitosan (80–95% degree of deacetylation), CTAB (≥ 99.0%), sodium hydroxide (NaOH, 99.9%), ferric trichloride hexahydrate (FeCl₃·6H₂O, ≥ 99.0%), HCl (36.0–38.0%), calcium chloride dihydrate (CaCl₂·2H₂O, ≥ 99.0%), sodium nitrate (NaNO₃, ≥ 99.0%), and magnesium chloride hexahydrate (MgCl₂·6H₂O, ≥ 99.0%) were obtained from Sinopharm Chemical Reagent Co., Ltd. Sodium sulfate (Na₂SO₄, ≥ 97.0%), hydrogen peroxide (H₂O₂, 30wt.% in H₂O), and nitric acid (HNO₃, 70%) were acquired from Macklin Biochemical Co., Ltd. Arsenic trioxide (As₂O₃, 99.95–100%) was provided from Shanghai Chemical Reagent Factory. All solutions were prepared using ultrapure water (18.2 MΩ cm⁻¹, Direct-Q3, Millipore SAS, Molsheim, France). A stock solution of 1000 mg L⁻¹ As(V) was prepared via dissolving 0.66 g of As₂O₃ in NaOH solution, oxidizing it with excess H₂O₂ and then diluting to 500 mL using ultrapure water. Simulated wastewaters containing varying concentrations of As(V) were prepared via diluting the stock solution in ultrapure water.

Preparation of adsorbents

Fe(III)-chitosan and Fe(III)-chitosan-CTAB composites were prepared using an ionotropic gelation method (Lobo et al. 2020). First, 1.0 g of chitosan powder was dissolved in 50 mL of FeCl₃ solution (4% w/v) and magnetically stirred at room temperature for 5 h. The resulting gel was named as Fe(III)-chitosan. Subsequently, 1.0 g of CTAB was added into the Fe(III)-chitosan gel and stirred for 3 h to obtain a Fe(III)-chitosan-CTAB gel. Then, each gel was slowly dropped into the NaOH solution (2 M) and stirred for 1 h. After stirring, each suspension was centrifuged for 5 min at 5000 rpm for no less than three times. The precipitate was washed with ultrapure water several times and then dried at 60 °C. Finally, Fe(III)-chitosan and Fe(III)-chitosan-CTAB were ground, sieved through a 100-mesh sieve, and then stored in a desiccator for further experiments.

Characterization of adsorbents

The surface morphologies of the adsorbents were investigated via scanning electron microscopy (SEM) (Zeiss...
Gemini 300, Carl Zeiss AG, Oberkochen, Germany) operated at 3.0 kV. Surface elemental composition of the adsorbents was confirmed using energy dispersive X-ray spectroscopy (EDS) (X-Max, Oxford Instruments, Abingdon, UK). Fourier transform infrared spectra were obtained with FT-IR spectrometer (Nicolet iS50, Thermo Fisher Scientific Inc., Madison, WI, USA) using KBr pellets in the range 4000–500 cm⁻¹. X-ray photoelectron spectroscopy (XPS) was recorded on ESCALAB Xi+ instrument (Thermo Fisher Scientific Inc., Waltham, MA, USA) with a monochromatic Al-Kα X-ray source (hv = 1486.60 eV) at 12.5 kV. The zeta potential was observed at pH 3.0–10.0 using Zeta-sizer Nano ZS90 analyzer (Malvern Pananalitical, Malvern, Worcestershire, UK).

**Adsorption experiments**

Batch adsorption experiments were performed in a 50-mL centrifuge tube via mixing 0.8 g L⁻¹ adsorbent with 25 mL As(V) solution at 25 °C and 250 rpm in triplicate. The effect of pH on As(V) adsorption was evaluated by adjusting the initial pH of As(V) solution (10 mg L⁻¹) to 2.0–10.0. The pH was adjusted to an appropriate value via adding negligible amount of 0.1 M NaOH or HCl. After 5 h adsorption reaction, aliquots of the adsorption solution were taken outside the centrifuge tube and centrifuged to isolate the adsorbents. The supernatant was passed through a 0.22-μm pore size membrane filter. The residual As(V) concentration in the supernatant was determined using Agilent 7700× ICP-MS (Agilent, Tokyo, Japan). The operating parameters of ICP-MS were presented in Table S1. The removal efficiency (R%) and adsorption capacity (qₜ (mg g⁻¹)) of As(V) were calculated as follows:

\[
R\% = \left(\frac{C_0 - C_t}{C_0}\right) \times 100
\]

\[
q_t = \frac{(C_0 - C_t)V}{m}
\]

where \(C_0\) (mg L⁻¹) and \(C_t\) (mg L⁻¹) are the initial and residual concentrations of As(V), respectively, \(V\) (L) is the solution volume, and \(m\) (g) is the mass of adsorbent. \(q_e\) (mg g⁻¹) can be acquired by replacing \(C_0\) in Eq. (2) with the residual As(V) concentration at equilibrium (\(C_e\)).

Adsorption kinetic experiments were conducted by using 10 mg L⁻¹ As(V) solutions at optimal pH via taking samples at intervals during the 5-h period. The pseudo-first- and pseudo-second-order kinetic models were employed to predict the adsorption kinetics. The best-fit model was selected based on the correlation coefficient (R²) value for regression. These two kinetic models are given as follows:

\[
q_t = q_e (1 - e^{-k_1 t})
\]

\[
q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t}
\]

where \(q_e\) (mg g⁻¹) and \(q_t\) (mg g⁻¹) are the amounts of As(V) adsorbed at equilibrium and at time \(t\) (min), respectively, \(k_1\) (min⁻¹) and \(k_2\) (g mg⁻¹ min⁻¹) are the pseudo-first- and pseudo-second-order rate constants.

Adsorption isotherms experiments were determined with As(V) in the concentration range of 1–100 mg L⁻¹ for 2 h at optimal pH. Both Freundlich and Langmuir isotherm models were adopted to fit the experimental data. These models are given by the following equations:

\[
q_e = \frac{q_mC_k C_e}{1 + K_L C_e}
\]

\[
q_e = K_F C_e^{1/n}
\]

where \(q_e\) (mg g⁻¹) is the adsorption capacity at equilibrium, \(q_m\) (mg g⁻¹) is the maximum adsorption capacity, \(C_e\) (mg L⁻¹) is the residual As(V) concentration at equilibrium, \(K_F\) [(mg g⁻¹) (L mg⁻¹)⁻¹/n] is the Freundlich constant related to adsorption capacity, \(K_L\) (L mg⁻¹) is the Langmuir constant related to the affinity of adsorption sites, and \(n\) is the dimensionless Freundlich intensity parameter.

The effects of co-existing ions (Mg²⁺, Ca²⁺, NO₃⁻, SO₄²⁻, HCO₃⁻, and H₂PO₄⁻, 1 mmol L⁻¹) and dissolved organic matter (HA, 10 mg L⁻¹) were studied at optimal pH for 2 h using As(V) concentration of 10 mg L⁻¹. Three water samples from different sources (Xiang River water, Hou Lake water, and tap water) were used to evaluate the As(V) removal performance of adsorbents in different water matrices. Because of their extremely low concentrations of As(V) (see Table S2), these samples were spiked to final concentrations of 0.1–10 mg L⁻¹ with As(V) solution.

**Desorption and regeneration experiments**

The desorption and regeneration behaviors of adsorbents were investigated to evaluate their reusability. The influence of the concentration of desorbing agent (NaOH solution) on the adsorption capacity of regenerated adsorbents was studied. After obtaining the optimal NaOH concentration, an adsorption–desorption experiment was performed over five cycles. The adsorption experiment was conducted with 0.2 mg of adsorbents to treat 10 mg L⁻¹ of As(V) solution at 25 °C and optimal pH for 2 h. The As(V)-loaded adsorbents were then eluted down with a 1% (w/v) NaOH solution for 3 h. The adsorbents were collected via centrifugation,
washed with ultrapure water, dried at 60 °C, and reused for the next cycle.

**Results and discussion**

**Characterization of adsorbents**

**Surface morphology and elemental composition**

The morphology of Fe(III)-chitosan and Fe(III)-chitosan-CTAB powders exhibited irregular particles with a particle size of 10 to 150 μm (Fig. 1a–d). The surface of Fe(III)-chitosan was rough with some fibrous particles attached. After modification with CTAB, more small particles were observed on the surface of Fe(III)-chitosan-CTAB, creating a much rougher surface compared with Fe(III)-chitosan. This may be due to the hydrophobic interaction between some hydrophobic moieties of chitosan and CTAB tails when the concentration of CTAB reached the critical micelle concentration (Zhao et al. 2008). The elemental composition of Fe(III)-chitosan-CTAB was analyzed by EDS. As shown in Fig. 1e, five elements of C, N, O, Fe, and Br were present onto the surface of Fe(III)-chitosan-CTAB, where Br came from CTAB. It was suggested that CTAB was successfully loaded onto Fe(III)-chitosan.

**FT-IR spectra**

The FT-IR spectra verified the existence of surface functional groups of adsorbents. Figure 2 reports the FT-IR spectra of chitosan, Fe(III)-chitosan, and Fe(III)-chitosan-CTAB before and after As(V) adsorption. For chitosan, the FT-IR spectrum exhibits the typical absorption bands at 3450, 3280, 2851, 1653, 1580, 1418, 1376, 1258, 1150, 1058, 1027, and 896 cm⁻¹. The characteristic broad absorption band at 3450 cm⁻¹ is assigned to the stretching vibration of the –OH group in absorbed water molecules. The absorption band at 3280 cm⁻¹ can be ascribed to the stretching vibration of N–H, and the other band at 1580 cm⁻¹ is in good agreement with the bending vibration of N–H in amide II (Jiang et al. 2021; Matet et al. 2013). The three bands at 2851, 1418, and 1258 cm⁻¹ can be corresponded to the symmetric and asymmetric stretching vibrations of –CH₂ (Matet et al. 2013). The characteristic band appeared at 1653 cm⁻¹ is related to the –C=O stretching vibration in amide I. The band at 1376 cm⁻¹ corresponds to the C–N stretching vibrations in the amine group.
vibration in amide III (Kloster et al. 2020; Matet et al. 2013). The two bands at 1150 and 896 cm$^{-1}$ can be associated with the stretching vibration of C–O–C. The bands occurring at 1058 and 1027 cm$^{-1}$ are referred to the C–OH stretching vibration (Medeiros Borsaglì et al. 2015). For Fe(III)-chitosan, the presence of additional new bands at 792 and 890 cm$^{-1}$ (overlapping with C–O–C stretching vibration at 896 cm$^{-1}$) can be observed, attributing to Fe–OH bending vibration (Adio et al. 2017). It was indicated that the –OH groups of chitosan were involved in the complexation with ferric ions. According to the literatures, the ferric ions in chitosan could be penta- or hexacoordinated, depending on the preparation conditions (Shen et al. 2013). For Fe(III)-chitosan-CTAB, the additional bands at 2920 cm$^{-1}$ (from antisymmetric stretching vibration of methylene in CTAB) and 1465 cm$^{-1}$ (due to the N–H bending of the primary aliphatic amine) are observed (Chatterjee et al. 2009). Furthermore, the appearance of vibrational peak at 600 cm$^{-1}$ in Fe(III)-chitosan and Fe(III)-chitosan-CTAB after As(V) adsorption ascribed the metal–oxygen interactions (Chatterjee et al. 2009; Harutyunyan et al. 2019).

**XPS spectra**

The chemical composition and surface oxidation state of Fe(III)-chitosan and Fe(III)-chitosan-CTAB before and after As(V) adsorption were investigated by XPS. As shown in Fig. 3, the XPS spectra revealed the presence of Fe, O, N, C, and As elements, which was consistent with the EDS results. The high-resolution XPS spectra of Fe 2p show four peaks at binding energies of 710.6, 712.6, 719.0, and 724.6 eV. The doublet at 710.6 and 712.6 eV is assigned to Fe(III) 2p$_{3/2}$, while the 724.6 eV is assigned to Fe(III) 2p$_{1/2}$ (Du et al. 2019; Yamashita and Hayes 2008). The presence of a Fe(III) satellite peak at 719.0 eV and absence of Fe(II) satellite peaks at 715.0 or 730.0 eV confirm that only ferric ions are present near the surface of Fe(III)-chitosan and Fe(III)-chitosan-CTAB. These results indicated that chitosan...

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**Fig. 3** XPS fully scanned spectra (a, b) and corresponding high-resolution XPS spectra of Fe 2p, N 1s, C 1s, and As 3d of Fe(III)-chitosan (c) and Fe(III)-chitosan-CTAB (d) before and after As(V) adsorption
was successfully complexed with Fe(III). N 1 s spectra were split into two peaks at 398.6 eV for C–N–Fe and 401.7 eV for –N+(CH3)3 (Rufato et al. 2021; Shen et al. 2013). The prominent N 1 s peak at 398.6 eV indicates the complexation of the amino group of chitosan with ferric ions. The other peak at 401.7 eV was observed only after Fe(III)-chitosan was impregnated with CTAB, indicating that the surface of synthesized Fe(III)-chitosan was successfully modified by cationic surfactant CTAB to increase surface functionality. C 1 s spectra were divided into three major components at 284.8 eV, 286.5 ± 0.1 eV, and 288.5 ± 0.2 eV, corresponding to C–C, C–N/C–O from overlapping of C–O–C and C–OH, and C=O, respectively (Rufato et al. 2021; Yu et al. 2021). An increase in atomic percent of C–C from 49.1% for Fe(III)-chitosan to 75.5% for Fe(III)-chitosan-CTAB was noted, which further confirms the formation of Fe(III)-chitosan-CTAB.

**Adsorption characteristics**

**Effect of pH**

The solution pH is a critical factor that may affect the surface charge of the adsorbents through protonation of the functional groups, as well as the chemical speciation of As (Escudero et al. 2009). The effect of pH on As(V) adsorption by Fe(III)-chitosan and Fe(III)-chitosan-CTAB is presented in Fig. 4a. For Fe(III)-chitosan, the removal efficiency of As(V) increased sharply as the solution pH increased from 2.0 to 3.0, and then gradually decreased with further increase in pH up to 10.0. For Fe(III)-chitosan-CTAB, the removal efficiency reached its maximal value (99.1 ± 0.1%) at pH 4.0, and then slightly decreased to 93.6 ± 0.2% with increase of pH from 4.0 to 7.0. The removal efficiency could remain 76.6 ± 2.0% at pH 10.0. It was attributed to the increased electrostatic attraction, because CTAB could enhance the electrostatic attraction by quaternary ammonium cations towards arsenate anions (Wang et al. 2016a). At pH 2.0, the removal efficiencies of As(V) by the two adsorbents were around 20.0%, and Fe(III) was detected in As(V) solutions treated with Fe(III)-chitosan and Fe(III)-chitosan-CTAB with means of 137.21 ± 1.54 and 119.96 ± 1.54 mg L−1, respectively (see Table S3). The leaching of Fe(III) into water at pH 2.0 may be one of the reasons for the low removal efficiencies of As(V) by the two adsorbents.

Figure 4b shows the zeta potentials of Fe(III)-chitosan and Fe(III)-chitosan-CTAB in As(V) solutions measured at different pH values. The zeta potential of Fe(III)-chitosan changed from positive (+10.3 mV) at pH 3.0 to negative (~8.7 mV) at pH 4.0, with the point of zero charge at around pH 3.4. By contrast, the zeta potential of Fe(III)-chitosan-CTAB was positive over the entire studied pH range (3.0–10.0). The absolute value of the zeta potential increased from pH 3 to 4, then decreased from 4.0 to 10.0. At pH 3.0–10.0, As(V) exists as a monovalent (H2AsO4−) or divalent (HAsO42−) anion. Higher removal of As(V) from an aqueous solution can be attributed to the adsorption of H2AsO4− and HAsO42− onto the positively charged surface of Fe(III)-chitosan at pH 3.0. As pH increased, the positively charged amino groups deprotonated, which may lead to a rapid decline in the removal efficiency of As(V) (Seyed Dorraj et al. 2014; Wang et al. 2016b). Fe(III)-chitosan-CTAB was maintained at a positive electric potential at pH 3.0–10.0, indicating that the adsorbent could effectively adsorb negative As(V). The reason why the As(V) removal decreased with an increase in pH might be attributed to the

![Fig. 4](image-url)

**Fig. 4** Effect of pH on As(V) adsorption (a) and the corresponding zeta potential of Fe(III)-chitosan and Fe(III)-chitosan-CTAB at different pH (b)
decrease of the positive surface potential of Fe(III)-chitosan-CTAB (Sun et al. 2019). Compared with Fe(III)-chitosan, Fe(III)-chitosan-CTAB was more conducive to As(V) removal from water. Therefore, pH 3.0 ± 0.1 and 4.0 ± 0.1 were selected as optimum solution pH in the subsequent experiments for Fe(III)-chitosan and Fe(III)-chitosan-CTAB, respectively.

Adsorption kinetics

The contact time is one of the significant parameters for the successful application of adsorbents. Figure 5a and b shows the effect of the contact time on the adsorption capacities of Fe(III)-chitosan and Fe(III)-chitosan-CTAB for As(V). It was found that the adsorption equilibrium of the adsorbents for As(V) adsorption was attained within 20 min. The adsorption capacities of Fe(III)-chitosan and Fe(III)-chitosan-CTAB were 12.94 and 12.88 mg g⁻¹, respectively. The rate of As(V) adsorption was fast, with more than 95% (for Fe(III)-chitosan) and 87% (for Fe(III)-chitosan-CTAB) of adsorption occurred during the first 20 min (see Table S4). The equilibrium time may depend on the characteristics of the synthetic chitosan derived materials. The adsorption equilibrium of the cross-linked Fe(III)-chitosan for As(V) adsorption could be reached within 2 h (Marques Neto et al. 2013), while 30 min was reported for the uncross-linked one (Lobo et al. 2020). Besides, a higher value of removal efficiency may correspond to a higher degree of protonation of amino groups at pH 3.0.

To identify the adsorption mechanism, the pseudo-first- and pseudo-second-order kinetic models were applied to test the experimental data. The results showed that the As(V) adsorption process followed the pseudo-second-order kinetic model. The correlation coefficients for pseudo-second-order kinetic model were higher ($R^2 = 0.998$ for Fe(III)-chitosan and $R^2 = 0.992$ for Fe(III)-chitosan-CTAB) than pseudo-first-order kinetic model ($R^2 = 0.980$ for Fe(III)-chitosan and $R^2 = 0.942$ for Fe(III)-chitosan-CTAB) (Table 1). The

![Fig. 5](image_url)

Fig. 5 Pseudo-first- and pseudo-second-order kinetic models of Fe(III)-chitosan (a) and Fe(III)-chitosan-CTAB (b); adsorption isotherm curves for As(V) on Fe(III)-chitosan (c) and Fe(III)-chitosan-CTAB (d)
pseudo-second-order kinetic model has also been found more suitable for modeling the adsorption of As(V) and As(III) onto the other modified chitosan-based adsorbents, such as chitosan/clay/magnetite composite and chitosan/nano-iron oxide films (Cho et al. 2012; Kloster et al. 2020).

Adsorption isotherms

To examine the effect of initial concentration of As(V) on the adsorption capacity, adsorption isotherm experiments were conducted. The adsorption isotherm is very important to understand how the adsorbate interact with the adsorption sites of the adsorbent surface. The Freundlich and Langmuir isotherm models were applied to analyze the adsorption equilibrium data. The adsorption isotherms were shown in Fig. 5c and d, and the calculated adsorption parameters are summarized in Table 2. The experimental data were well fitted with the Freundlich isotherm model (correlation coefficient 0.963 for Fe(III)-chitosan and 0.987 for Fe(III)-chitosan-CTAB). The Freundlich isotherm model is valid for multilayer adsorption onto a heterogeneous surface (Jeyaseelan et al. 2018). Hence, the results demonstrated that the adsorption of As(V) onto Fe(III)-chitosan and Fe(III)-chitosan-CTAB occurred in multilayer possessing heterogeneous adsorption sites. The adsorption capacity for As(V) at equilibrium increased infinitely with increasing initial As(V) concentration, suggesting that the adsorption sites of adsorbents were not equivalent or independent (Nisticò et al. 2018). In addition, the values of $n$ (Freundlich isotherms) were 5.891 for Fe(III)-chitosan and 4.956 for Fe(III)-chitosan-CTAB, respectively, indicating that the adsorption between As(V) and adsorbents was favorable ($1 < n < 10$). That is because the value of $n$ above 1 is indicative of favorable chemisorption mechanism on heterogeneous surface (Foo and Hameed 2010). The larger $n$ value also denoted the stronger interaction between As(V) and Fe(III)-chitosan. Accordingly, the adsorption capacity of Fe(III)-chitosan for As(V) (33.85 mg g$^{-1}$) was a little more than that of Fe(III)-chitosan-CTAB (31.69 mg g$^{-1}$).

Although the adsorption capacities of Fe(III)-chitosan and Fe(III)-chitosan-CTAB were not the highest, they were superior to some adsorbents like granular adsorbents made from water treatment residuals materials and chitosan (Zeng et al. 2020), chitosan with magnetic nanoparticles (Kloster et al. 2020), and other chitosan-composite adsorbents as listed in Table S5. Moreover, compared with other chitosan-based adsorbents exhibiting high adsorption capacity for As(V), such as chitosan-magnetic graphene oxide grafted poly-aniline doped with cobalt oxide (Gabris et al. 2022), both Fe(III)-chitosan and Fe(III)-chitosan-CTAB were cheap and easy-to-synthesize materials with rapid adsorption rates for As(V) removal. These advantages will be helpful for their operational applications.

Effect of co-existing ions

Some anions and cations generally present in water could interfere in the removal efficiency of As(V) by competitive binding or complexation (Bhakat et al. 2006). So, the effects of co-existing anions (NO$_3^-$, SO$_4^{2-}$, HCO$_3^-$, and H$_2$PO$_4^-$), cations (Mg$^{2+}$ and Ca$^{2+}$), and organic matter (HA) on the As(V) adsorption were investigated. As seen in Fig. 6, the presence of Mg$^{2+}$, Ca$^{2+}$, NO$_3^-$, SO$_4^{2-}$, and HCO$_3^-$ had no significant impact on the adsorption of As(V), whereas H$_2$PO$_4^-$ significantly reduced As(V) removal. Decrease in As(V) adsorption onto Fe(III)-chitosan and Fe(III)-chitosan-CTAB in the presence of H$_2$PO$_4^-$ may be explained as a result of competition for the adsorption sites between H$_2$PO$_4^-$ and As(V). Another reason for this phenomenon could be that H$_2$PO$_4^-$ has chemical characteristics (such as ion size, structure, and $pK_a$) very similar to those of As(V) (Sun et al. 2019). Phosphate also had a strong tendency to form complexes with Fe(III) at low pH (Gupta et al. 2009). However, the phosphate concentration in most natural water is low enough (20–50 μg L$^{-1}$) (Katsoyiannis and Zouboulis

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Table 1 Kinetic parameters for As(V) adsorption on Fe(III)-chitosan and Fe(III)-chitosan-CTAB

| Adsorbents          | Pseudo-first-order model | Pseudo-second-order model |
|---------------------|--------------------------|----------------------------|
|                     | $k_1$ (min$^{-1}$) | $q_e$ (mg g$^{-1}$) | $R^2$ | $k_2$ (mg g$^{-1}$ min$^{-1}$) | $q_e$ (mg g$^{-1}$) | $R^2$ |
| Fe(III)-chitosan    | 0.487 | 12.593 | 0.980 | 0.065 | 13.114 | 0.998 |
| Fe(III)-chitosan-CTAB | 0.357 | 12.026 | 0.942 | 0.044 | 12.705 | 0.992 |

Table 2 Isothermal parameters for As(V) adsorption on Fe(III)-chitosan and Fe(III)-chitosan-CTAB

| Adsorbents          | Langmuir model | Freundlich model |
|---------------------|----------------|-----------------|
|                     | $q_m$ (mg g$^{-1}$) | $K_L$ (L mg$^{-1}$) | $R^2$ | $K_F$ (mg g$^{-1}$) (L mg$^{-1}$)$^{1/n}$ | $n$ | $R^2$ |
| Fe(III)-chitosan    | 25.076 | 5.896 | 0.761 | 14.606 | 5.891 | 0.963 |
| Fe(III)-chitosan-CTAB | 25.288 | 3.129 | 0.834 | 13.049 | 4.956 | 0.987 |
The competitive adsorption of phosphate on Fe(III)-chitosan and Fe(III)-chitosan-CTAB would be limited. Natural organic matter (NOM) is ubiquitous in underground and surface water. HA is a major constituent of NOM found in water systems (Chaukura et al. 2018). In this study, HA had no observed inhibitory effect on the removal of As(V) by Fe(III)-chitosan, while the removal of As(V) by Fe(III)-chitosan-CTAB was declined from 98.2 to 85.6%. This decrease may be related to the amphiphilic nature of both CTAB and HA, which causes their mutual attraction especially when HA and surfactant are oppositely charged (Ishiguro et al. 2007).

Application to real water samples

To assess the effect of real water matrices, Fe(III)-chitosan-CTAB was used to real water naturally contaminated with As(V) and collected from the water pipe, lake, and river. The pH values of these samples ranged from 7.91 to 8.01, and the removal efficiencies of As(V) by Fe(III)-chitosan-CTAB were 80.0%, 95.5%, and 88.5%, respectively (see Table S2). Considering low concentration of As(V) in real water (2.85, 17.69, and 12.08 μg L⁻¹, respectively), three different water matrices were spiked with various concentrations of As(V) (0.1, 0.2, 0.5, 1.0, 5.0, and 10.0 mg L⁻¹). It was showed that the residual concentration of As(V) reduced to less than 0.01 mg L⁻¹ (WHO permissible limit for drinking water) with more than 99.0% removal efficiency, when the initial As(V) concentration raised from 0.1 to 1.0 mg L⁻¹ (Fig. 7). However, 97.8%, 93.5%, and 93.9% removal of As(V) were achieved at initial As(V) concentration of 5 mg L⁻¹ for tap water, lake water, and river water, respectively. Just as the initial As(V) concentration rose from 5 to 10 mg L⁻¹, the As(V) removal efficiency declined to 90.7%, 76.8%, and 78.2%. It showed a more decrease tendency in lake water and river water, which may be caused by more complex matrix components (such as suspended solids and macromolecules). Generally, the concentrations of As in natural waters are often below 5 mg L⁻¹ (Mandal and Suzuki 2002). Accordingly, Fe(III)-chitosan-CTAB could effectively be used to remove As(V) from natural waters.

Reusability of the adsorbents

Reusability is a critical property of adsorbents due to the economic applicability. From the desorption studies, it was found that the structure of both adsorbents got destroyed
within few minutes in strong acid (HCl) aqueous solution. However, use of NaOH solution was found to be effective. Figure 8a shows the desorption efficiency of Fe(III)-chitosan and Fe(III)-chitosan-CTAB by various concentrations of NaOH solution (1, 2, 5, and 10% w/v). As shown in Fig. 8a, the desorption of As(V) from the two adsorbents reached 96.3% and 96.1%, respectively, when using 1% w/v NaOH solution. The adsorption–desorption cycle was repeated five times by using the same adsorbents. It can be seen from Fig. 8b that the adsorption capacity of Fe(III)-chitosan remained almost unchanged after five cycles. Fe(III)-chitosan-CTAB only decreased slightly from 7.16 to 6.29 mg g\(^{-1}\) with an increase in the reuse cycles. The slight decrease in adsorption of As(V) on Fe(III)-chitosan-CTAB is possibly explained by the loss of active sites during the desorption process. Moreover, the surface structure of Fe(III)-chitosan-CTAB may be partially destroyed after stirring and washing. According to the obtained results, the two adsorbents had good reusability and chemical stability. It was indicated that both Fe(III)-chitosan and Fe(III)-chitosan-CTAB were of excellent potential for application in adsorptive removal of As(V) from water.

**Possible mechanism of As(V) adsorption on adsorbents**

The adsorption mechanism of As(V) onto Fe(III)-chitosan and Fe(III)-chitosan-CTAB can be supposed to both electrostatic attraction, as well as complexation (Fig. 9). From the FT-IR spectra data, it was observed that three new peaks appeared at 890, 792, and 600 cm\(^{-1}\) after the adsorption of As(V) (Fig. 2). The peak at 792 cm\(^{-1}\) is attributed to Fe–O–As bending vibration (Adio et al. 2017). The peaks at 890 and 600 cm\(^{-1}\) are assigned to the asymmetrical stretching of the As–O bond in the H\(_2\)AsO\(_4\)\(^–\) group (Kloster et al. 2020; Kumar and Jiang 2016). These results revealed the possible mechanism of the complexation of As(V) with Fe–OH of Fe(III)-chitosan and Fe(III)-chitosan-CTAB via the formation of monodentate and bidentate inner-sphere complexes (Li et al. 2010). In addition, the decreases of the relative intensities of the peaks at 3450, 3280, 1058, and 1027 cm\(^{-1}\) after As(V) adsorption are mainly associated with the interaction of As(V) with –NH\(_2\) and –OH of the adsorbents. The nitrogen and oxygen atoms of –NH\(_2\) and –OH could provide lone pair of electrons for bonding with As(V), which suggests that the adsorption mechanism of Fe(III)-chitosan and Fe(III)-chitosan-CTAB for As(V) in water includes electrostatic attraction.

XPS analyses were applied to further explore the adsorption mechanism of Fe(III)-chitosan and Fe(III)-chitosan-CTAB for As(V). As seen from Fig. 3c and d, the typical new peaks of As 3p\(_{3/2}\) appeared at 44.79 and 44.64 eV. These results indicated the presence of As(V) on the surface of Fe(III)-chitosan and Fe(III)-chitosan-CTAB after adsorption, which are similar to previous studies like Du et al. 2019. For the C 1 s spectra, the peak area ratio at 286.5 eV (C–OH) decreased from 26.5 to 23.6% for Fe(III)-chitosan and from 17.11 to 13.93% for Fe(III)-chitosan-CTAB. These decreases may be due to the substitution of H\(_2\)AsO\(_4\)\(^–\) for part of hydroxyl groups during adsorption (Sun et al. 2019). For the N 1 s spectra, the intensity of the peak at 398.6 eV decreased, which may be related to –NH\(_2\)…Fe located in the inner sphere of the complex. A new peak appeared at a higher binding energy of 401.0 eV showing the formation of

![Fig. 8](image-url)  Reusability of Fe(III)-chitosan and Fe(III)-chitosan-CTAB for As(V) adsorption. (a) Desorption efficiency of As(V) from adsorbents in NaOH solution with different concentrations. (b) Adsorption capacities of the regenerated adsorbents in five consecutive cycles

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The protonated amino group (–NH₃⁺) on Fe(III)-chitosan after As(V) adsorption (Rufato et al. 2021). Theoretically, the –NH₂ group on Fe(III)-chitosan is easily protonated to form positively charged –NH₃⁺ group. H₂AsO₄⁻, a negatively charged ion, could be attracted to the –NH₃⁺ group, leading to the enrichment of H₂AsO₄⁻ on the surface of Fe(III)-chitosan. With regard to Fe(III)-chitosan-CTAB, the peak intensity at 401.8 eV decreased, suggesting that the positively charged –NH₃⁺ and –N(CH₃)₃ groups attracted the H₂AsO₄⁻ ions after As(V) adsorption (Rufato et al. 2021; Shen et al. 2013). The binding energy of Fe 2p was slightly shifted by 0.1–0.4 eV after As(V) adsorption. As previously noted, it can be inferred that the functional groups of Fe–OH, C–OH, –NH₃⁺, and –N(CH₃)₃⁻ were the main active sites engaged in the As(V) adsorption.

**Conclusions**

Fe(III)-chitosan and Fe(III)-chitosan-CTAB were successfully synthesized via a ionotropic gelation method for efficient As(V) removal from water. The two adsorbents exhibited irregular shapes and rough surfaces with a diameter of 10–150 μm. The best removal efficiency of As(V) was observed at pH = 3.0 for Fe(III)-chitosan, while high removal efficiency was achieved in a wide pH range (3.0–8.0) for Fe(III)-chitosan-CTAB. The kinetics data described well with pseudo-second-order kinetic model, suggesting that the adsorption was affected by chemical interactions. Equilibrium adsorption values were satisfied with the Freundlich isotherm model, and supported the occurrence of multilayer adsorption process. The presence of co-existing ions, with the exception H₂PO₄⁻, had no negative effect on As (V) removal efficiency by the two adsorbents. They had stable adsorption capability after five cycles and when utilized in real water matrices. Complexation and electrostatic attraction were the dominant mechanisms involved in the adsorption of As(V) on Fe(III)-chitosan and Fe(III)-chitosan-CTAB. In summary, both Fe(III)-chitosan and Fe(III)-chitosan-CTAB provide an efficient method for removing As(V) from water and they have the potential for practical application.
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Data availability The data used in this manuscript is included in the text.

Declarations

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