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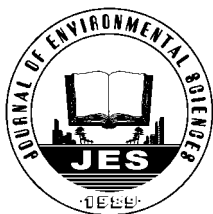
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Removal of perchlorate from aqueous solution by cross-linked Fe(III)-chitosan complex

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ABSTRACT

Cross-linked Fe(III)-chitosan composite (Fe-CB) was used as the adsorbent for removing perchlorate from the aqueous solution. The adsorption experiments were carried out by varying contact time, initial concentrations, temperatures, pH, and the presence of co-existing anions. The morphology of the adsorbent was discussed using FT-IR and SEM with X-EDS analysis. The pH ranging from 3.0–10.2 exhibited very little effect on the adsorption capability. The perchlorate uptake onto Fe-CB obeyed Langmuir isotherm model. The adsorption process was rapid and the kinetics data obeyed the pseudo second-order model well. The eluent of 2.5% (W/V) NaCl could regenerate the exhausted adsorbent efficiently. The adsorption mechanism was also discussed.

Introduction

Perchlorate salts are used as oxidizers in rocket and missile fuels, explosives, fireworks, vehicle safety flares, batteries and other industry products (Srinivasan and Sorial, 2009; Bardiya and Bae, 2011). Their use and the improper disposal of the wastes have led to the release of perchlorate into the environment (Dasgupta et al., 2006). By far, as an emerging trace contaminant, perchlorate has been widely detected in drinking water systems, surface water, groundwater, foodstuffs, and even in human saliva and breast milk (Kannan et al., 2009; Kirk et al., 2005). As the perchlorate ion is similar to iodide ion in size, excessive exposure of human bodies to perchlorate can interfere with the natural process of iodine uptake by the thyroid gland, inhibit thyroid hormone production and disturb normal metabolism, and subsequently lead to a series of diseases such as neurological damage and anemia (Attanasio et al.,

2011; Dasgupta et al., 2008). Because of its harm to human health, the US EPA has set an Interim Drinking Water Health Advisory level of 15 µg/L in 2008, and some states in the United States have established lower action levels (Hou et al., 2013).

Up to now, several treatment technologies have been developed to remove perchlorate from water viz., ion-exchange, adsorption, biological treatment, and chemical/catalytic reduction, etc. For light-polluted water of perchlorate, adsorption seems to be one of the most attractive methods, especially for drinking water (Kumar et al., 2010). Recently, several conventional and novel adsorption materials have been tested for perchlorate removal. As a common adsorbent in drinking water treatment, granular activated carbon has also caused great interests in perchlorate removal. To enhance the adsorption capacities of granular activated carbon for perchlorate, several modification methods were investigated, such as tailoring with cationic surfactants (Parette and Cannon, 2005), coating with the cetyltrimethyl ammonium chloride (Xu et al., 2011), anchoring with quaternary ammoni-

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um/epoxide (Hou et al., 2013), and oxidizing by nitric acid (Fang and Chen, 2012). Some other adsorbents were also investigated, including granular ferric hydroxide (Kumar et al., 2010), zero-valent aluminum and aluminum hydroxide (Lien et al., 2010), giant reed modified by quaternary amine (Baidas et al., 2011), magnetic permanently confined micelle arrays (Mag-PCMA) (Clark and Keller, 2012), Mg/(Al-Fe) hydrotalcite-like compounds and MgFe-CO₃ layered double hydroxides (Yang et al., 2012a), etc.

As an abundant natural organic matter, chitosan has attracted our attention because it has high content of amino (-NH₂) and hydroxy (-OH) functional groups, and exhibits high activity for modification and adsorption (Azlan et al., 2009). In our previous studies, raw chitosan solution and protonated cross-linked chitosan showed relatively high efficiency for perchlorate removal, but the applicable pH range was limited within 4.0 to 6.0 (Xie et al., 2010, 2011). Recently, chitosan impregnated with iron metal has been used to adsorb anions from water, which showed strong affinity with the ox-anions (Zimmermann et al., 2010).

In this work, chitosan impregnated with Fe(III) was prepared, characterized and used as an adsorbent for adsorbing perchlorate from water. The experimental data were fitted with different isotherms and kinetic models to understand the adsorption process.

1 Materials and methods

1.1 Chemicals

The chitosan powder was purchased from Golden-shell Biochemical Co. (Zhejiang, China), with the deacetylation degree of 95% and the viscosity of 150 mPa·sec. All reagents, such as NaClO₄·H₂O, NaNO₃, Na₂SO₄, HCl, NaOH, FeCl₃·6H₂O, acetate acid and glutaraldehyde were of analytical grade. Deionized water with a resistance of 18 MΩ·cm supplied by a Millipore filtering system was used in all the experiments.

1.2 Preparation of cross-linked Fe(III) incorporated chitosan beads

The synthesis process was modified from the reported study by Viswanathan and Meenakshi (2008). In brief, chitosan solution was prepared by dissolving 2.5 g chitosan powder into 100 mL of 2% (V/V) acetate acid solution. After the chitosan powder was completely dissolved, 10 g FeCl₃·6H₂O was added to the solution and mixed with a stirrer at room temperature. Then, the mixture was dropped into 2.0 mol/L of NaOH, which thereby coagulated red brown spherical gel beads which were highly porous. The beads were gelled for 2 hr and then washed by distilled water. Finally, the washed beads were cross-linked for 8 hr with 7.5% (V/V) glutaraldehyde solution. The cross-linked

beads were then thoroughly washed by distilled water again, dried at 50°C for 36 hr, and sealed for storage at constant temperature in a humid box. The moisture content of the adsorbent used for sorption experiment was about 67%.

1.3 Batch experiments

Adsorption experiments were carried out using a batch equilibration method in duplicate. In a typical case, 0.5 g of cross-linked Fe(III)-chitosan composite (Fe-CB) was added to 50 mL of perchlorate solution with initial concentration of 10 mg/L. The contents were mixed thoroughly using a shaking incubator at a speed of 180 r/min and a constant temperature of 25 ± 1°C. After 4 hr of agitation, 10 mL samples were taken using a single injector, filtrated by 0.22 μm filter and analyzed by ion chromatography (IC) system. The effects of pH on adsorption were conducted by adjusting pH from 3.0 to 10.2 using HCl and NaOH. The effects of co-existing anion, including SO₄²⁻, Cl⁻, NO₃⁻ and HCO₃⁻, on perchlorate adsorption were investigated. The initial concentration of the co-existing anions ranged from 0 to 200 mg/L.

Kinetics studies were carried out at different initial concentrations, viz., 1.05, 4.05, 13.21 and 27.53 mg/L. Cross-linked Fe-CB of 5 g was added to 500 mL of the perchlorate solution. The solution was stirred for 12 hr at 25 ± 1°C, and samples were collected at different time intervals for the analysis of the perchlorate concentration. In the equilibrium experiments, 0.5 g of cross-linked Fe-CB was added to 50 mL perchlorate solution with the initial concentration varying from 1 to 200 mg/L. The equilibrium isotherms were determined for 4 hr at 20 ± 1°C, 35 ± 1°C and 50 ± 1°C, respectively. The amount of the adsorbed perchlorate at equilibrium (q_e , mg/g) was calculated as follows:

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (1)$$

where, C_0 (mg/L) is the initial concentration of perchlorate; C_e (mg/L) is the equilibrium concentration of perchlorate; V (L) is the solution volume; and W (g) is the dry mass of the adsorbent.

1.4 Analytic methods

Perchlorate concentration was determined using an IC system (Compac IC, Metrohm) which was equipped with a 250 μL sample loop, a set of 4 × 250 mm chromatogram column (Metroep A Supp 7) and guard column, a 4-mm suppressor, an electrical conductivity detector, and an auto-sampler. The mixture of 10 mmol/L Na₂CO₃ and 45% acetonitrile (V/V) was set into the eluent for ClO₄⁻ detection at 0.7 mL/min. The sample running time was 22 min. The detection limit was 15 μg/L for perchlorate.

Besides, FT-IR spectra of cross-linked Fe-CB before and after the sorption were recorded with Fourier trans-

formation infrared spectrometer (Nicolet 6700, Thermo Fisher). KBr pellets were prepared by mixing the samples with KBr. SEM images of chitosan and cross-linked Fe-CB were characterized by a cold field scanning electron microscope (Inspect F50, Holand). The SEM system was equipped with an Energy Dispersive X-ray Spectrometer (X-EDS) (INCA ENERGY 300, England), which could analyze element ranging from 4Be to 92U. The X-EDS spectra of chitosan and cross-linked Fe-CB before and after the sorption were recorded for investigating the element changes during the synthesis process and sorption process.

2 Results and discussion

2.1 Characterization of adsorbents

The FT-IR spectra were applied to identify the change of functional groups in chitosan molecule in the synthesis and adsorption processes. The FT-IR spectrum of the chitosan flake is presented in **Fig. 1, line a**. The strong broad band in the region of $3500\text{--}3200\text{ cm}^{-1}$ showed the characteristics of O–H and N–H stretching vibrations. The peaks at 2876 , 2200 , and 1085 cm^{-1} attributed to C–H, C–NH₂, and C–O stretching vibrations respectively (Singha et al., 2009; Ma et al., 2007). The main nitrogenous functions in chitosan appeared at 1600 cm^{-1} because of the free amine (–NH₂) forms of the glucosamine residues. The band at 1650 cm^{-1} corresponded to the C=O vibration conjugated with the N–H deformation (Guibal et al., 1999). While, C=O stretching band ($1900\text{--}1650\text{ cm}^{-1}$) was very weak due to the high deacetylation degree of chitosan flake used in this study. The FT-IR spectrum of cross-linked Fe-CB is shown in **Fig. 1, line b**. The main functional groups in chitosan were shifted to lower frequencies after being chelated with Fe(III) and cross-linked by glutaraldehyde.

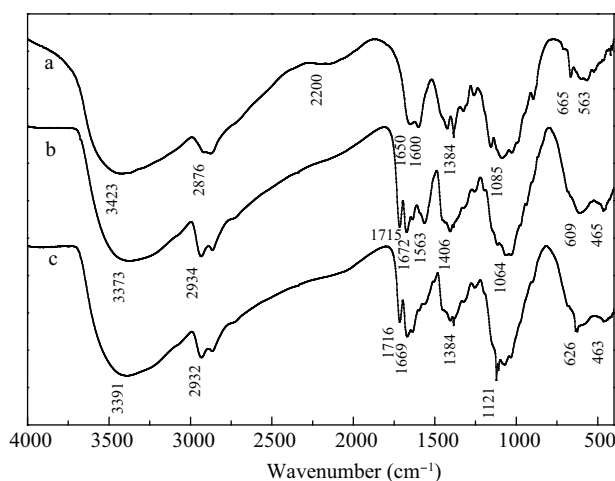


Fig. 1 FT-IR spectra of chitosan flake (line a), cross-linked Fe-CB (line b) and cross-linked Fe-CB loaded with ClO₄[−] (line c).

The band of free amine functions at 1600 cm^{-1} almost disappeared because of a high level of consumption during the chelation and cross-linking reaction. Both –NH₂ and –OH groups were bonded to the metal ions of Fe(III), and more than one chitosan chain was involved in the formation of such complexes (Bhatia and Ravi, 2000; Shen et al., 2013). An important peak appeared at 1672 cm^{-1} attributing to –C=N– (Schiff alkali) band, which made the adsorbent insoluble in aqueous solution (Guibal et al., 1999). The cross-linking reaction might take place between the amine groups in chitosan and the aldehyde groups of glutaraldehyde (Spinelli et al., 2004).

In addition, SEM was used to explain the morphology of the adsorbent. SEM images of chitosan flake and cross-linked Fe-CB are shown in **Fig. 2**. Chitosan flake had relatively tight surface structure with no porosity and poor surface area (Jagtap et al., 2011). After being incorporated with Fe(III) and cross-linked by glutaraldehyde, the adsorbent became loosened and many pores were observed on the surface. The irregularity surface was helpful for the enhancement of its adsorption ability. X-EDS analysis was employed to confirm the element change in the adsorbent during the synthesis process. **Figure 3** indicates that carbon and oxygen were the primary elements in chitosan flake. In the case of cross-linked Fe-CB, peaks of ferric iron emerged clearly, which further confirmed that ferric iron was chelated into chitosan molecule successfully.

2.2 Effect of pH

The pH of the medium was one of the important factors, which significantly affected the surface charge of the adsorbents (Le Leuch and Bandosz, 2007). The effect of pH on perchlorate adsorption by cross-linked Fe-CB was investigated in the pH range of 3.0–11.6 by keeping all other parameters constant and the results are shown in **Fig. 4**. The amount of perchlorate adsorbed at equilibrium varied slightly from 3.83 to 3.98 mg/g when the pH was ranged from 3.0 to 10.2. The results indicated that the adsorption capability of cross-linked Fe-CB for perchlorate was nearly independent of pH in the range of 3.0 to 10.2. When the pH was further increased, the adsorption capacity of the cross-linked Fe-CB was decreased significantly. At the pH of 11.6, the adsorption capacity was just 1.4 mg/g. However, compared with protonated cross-linked chitosan which has the applicable pH range of 4.0–6.0 (Xie et al., 2010), cross-linked Fe-CB exhibited a wider efficient pH region for adsorption anions from water.

The pH_{zpc} (pH at the surface has a net zero charge) of the complex was about 10.6, determined by potentiometric titration method (Fu et al., 2007; Mustafa et al., 2004). When the pH value of solution was less than the pH_{zpc} (10.6), the surface of the cross-linked Fe-CB was positively charged, which could adsorb anions by electrostatic attraction. Therefore, the adsorption ability of the cross-linked Fe-CB for perchlorate might be pH independence

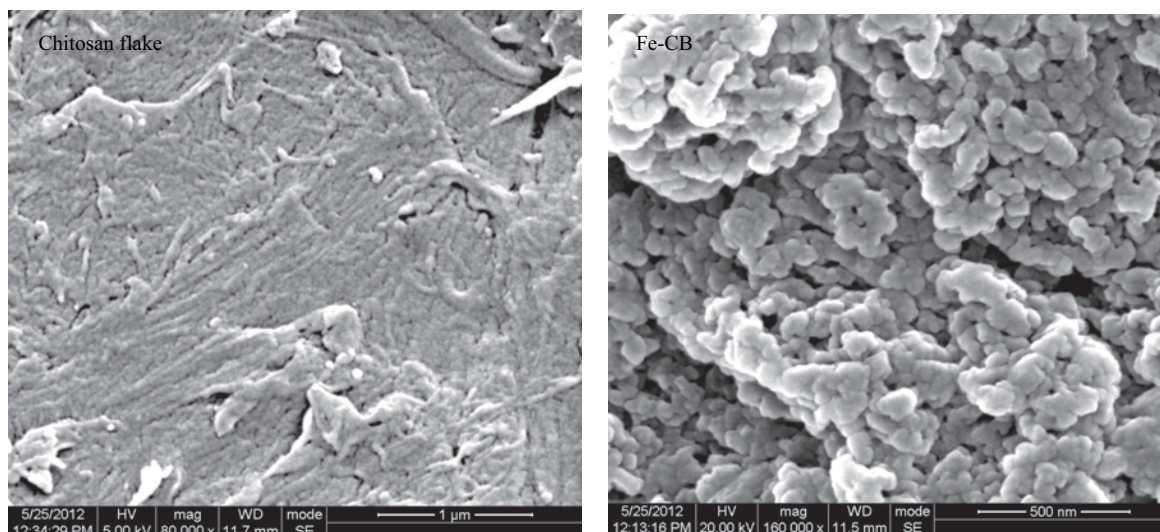


Fig. 2 SEM images of chitosan flake and cross-linked Fe-CB.

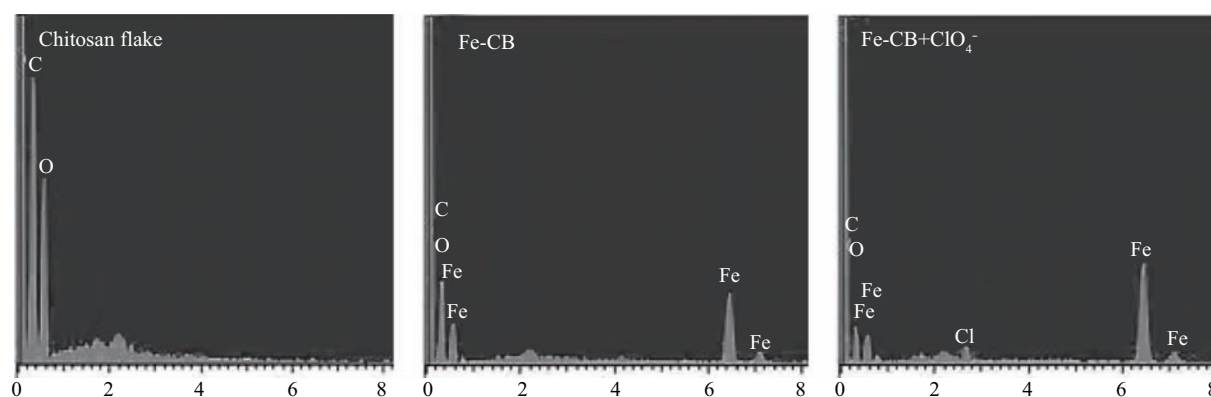


Fig. 3 X-EDS spectra of chitosan flake, cross-linked Fe-CB, and cross-linked Fe-CB loaded with ClO_4^- .

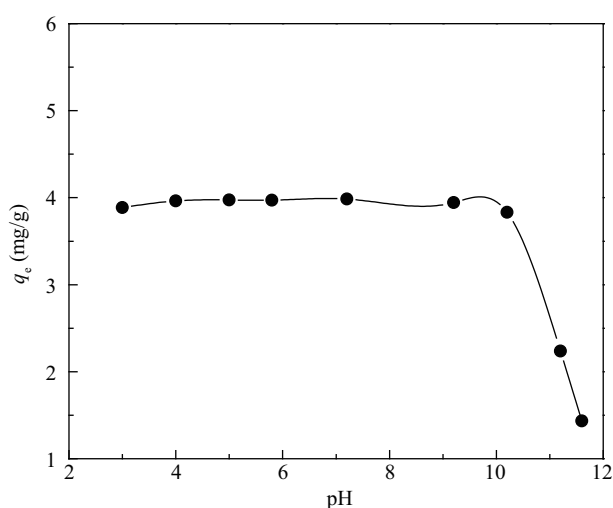


Fig. 4 Effect of pH on adsorption of ClO_4^- onto cross-linked Fe-CB. Experimental conditions: initial ClO_4^- concentration was 10 mg/L, dosage of cross-linked Fe-CB was 10 g/L, and temperature was $25 \pm 1^\circ\text{C}$.

when the pH was less than 10.6. With pH being further increased, the positive surface charge of the complex grad-

ually decreased and resulted in less adsorption capability for perchlorate. The results were consistent with the pH influence discussed above.

2.3 Effect of co-existing anions

In general, the perchlorate-contaminated water always contains several other common anions, viz., SO_4^{2-} , Cl^- , NO_3^- and HCO_3^- which can compete with perchlorate in the adsorption process (Gu et al., 2007). In order to illustrate the effect of interfering anions, adsorption studies were carried out by varying the initial concentrations of co-existing anions from 0 to 200 mg/L. The initial perchlorate concentration and chitosan dosage were kept at 10 mg/L and 10 g/L respectively. As is shown in Fig. 5, the co-existing anions exhibited negative effect on the removal of perchlorate. With the increase of the anion concentration, a reduction in perchlorate removal of cross-linked Fe-CB was observed. This may be due to the competition among the anions for the adsorption sites on the sorbent surface, which is decided by the concentration, charge and size of the anions (Viswanathana et al., 2009). Among these anions, Cl^- showed relatively weak effect on the

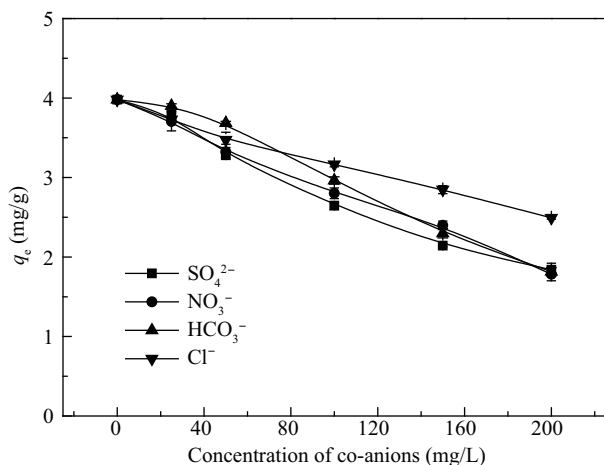


Fig. 5 Effect of co-existing anions on adsorption of ClO_4^- onto cross-linked Fe-CB. Experimental conditions: the initial ClO_4^- concentration was 10 mg/L, the dosage of cross-linked Fe-CB was 10 g/L, the pH was neutral, and the temperature was $25 \pm 1^\circ\text{C}$.

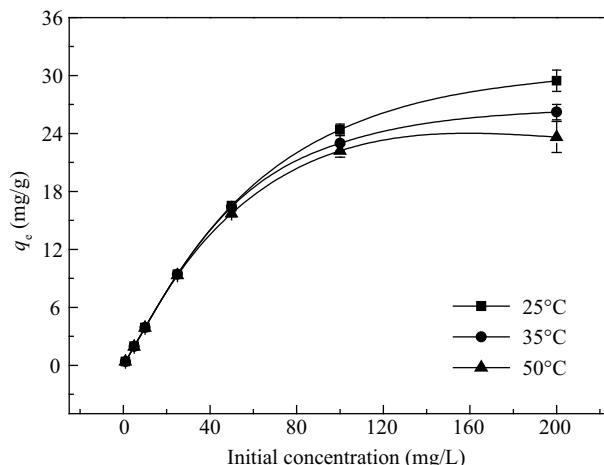


Fig. 6 Isotherm plots for the adsorption of ClO_4^- onto cross-linked Fe-CB at different temperatures. Experimental conditions: dosage of cross-linked Fe-CB was 10 g/L, pH was neutral, and initial ClO_4^- concentration was ranged from 1 to 200 mg/L.

adsorption process. With the concentration of Cl^- being increased from 0 to 200 mg/L, the removal efficiency of the perchlorate was decreased from 97% to 62%. While the anions of SO_4^{2-} , NO_3^- and HCO_3^- exhibited similar interference in the adsorption process. The removal efficiency of perchlorate was decreased to 45% when the co-existing anion concentrations were increased to 200 mg/L.

2.4 Adsorption isotherms

Adsorption equilibrium isotherm studies were carried out at 25°C , 35°C and 50°C (Fig. 6). Langmuir, Freundlich, and Tempkin isotherm models were used to analyze the adsorption process and their linear forms were given as follows, respectively (Kavitha and Namasivayam, 2007; Chatterjee and Woo, 2009):

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0} \quad (2)$$

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (3)$$

$$q_e = B \ln A + B \ln C_e \quad (4)$$

where, q_e (mg/g) is the amount of perchlorate adsorbed per unit mass of the adsorbent at equilibrium; Q_0 (mg/g) is the amount of adsorbate at complete coverage, which gives the maximum adsorption capacity; and b (L/mg) is the Langmuir constant reflecting the energy of adsorption; K_f is the Freundlich constant that relates to adsorption capability of the adsorbent, and $1/n$ is the adsorption intensity; A (L/g) and B are the Tempkin constants which refer to the maximum binding energy and the heat change of the adsorption, respectively. All of the constants can be

calculated from the slopes and intercepts of their respective linear equations. The fitting constant values of the three models along with the regression coefficients (R^2) are listed in Table 1. Compared with the Freundlich and Tempkin isotherms, the Langmuir isotherm fitted to the equilibrium data best with R^2 being higher than 0.99 at different temperatures. The results indicated that perchlorate was adsorbed mainly in the form of monolayer coverage on the surface of the adsorbent. With the rise of temperature, the values of Q_0 decreased, which indicated lower temperature was relatively favorable for the adsorption reaction.

The essential characteristics of the Langmuir isotherm could be expressed through a dimensionless constant named equilibrium parameter R_L (Tan et al., 2007), which was calculated by the following equation:

$$R_L = \frac{1}{(1 + bC_0)} \quad (5)$$

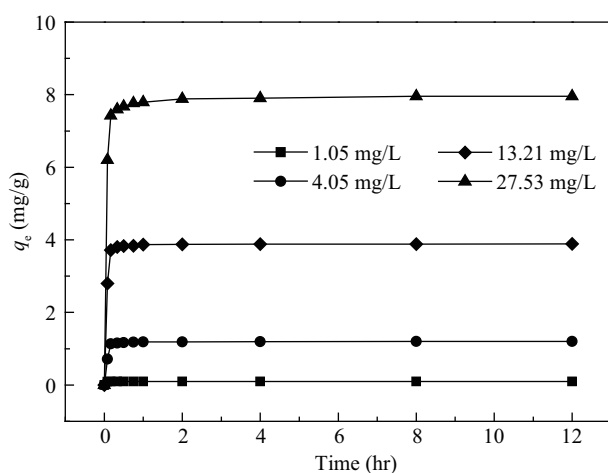
The R_L values reflect whether the adsorption is irreversible ($R_L = 0$), favorable ($0 < R_L < 1$), linear ($R_L = 1$), or unfavorable ($R_L > 1$). The R_L values (data were not shown) of the sorbent valued between 0 and 1, which indicated that the adsorption of perchlorate on the adsorbent was favorable at all the temperatures studied.

2.5 Adsorption kinetics

Adsorption kinetics was conducted at different initial concentrations, respectively 1.05, 4.05, 13.21 and 27.53 mg/L, and the data are plotted in Fig. 7. It is observed that the uptake rate of perchlorate by cross-linked Fe-CB was very fast in the first 10 min. Then the adsorption rate slowed down, and the reaction almost approached equilibrium within 30 min. This characteristic will facilitate its application in practice, such as shortening reaction time, minimizing apparatus volume, and saving energy, etc. It is worth noticing that this adsorbent was efficient

Table 1 Langmuir, Freundlich and Tempkin isotherm model constants and correlation coefficients for adsorption of perchlorate onto cross-linked Fe-CB

<i>T</i> (K)	Langmuir isotherm				Freundlich isotherm			Tempkin isotherm		
	<i>Q</i> ₀ (mg/g)	<i>b</i> (L/mg)	<i>R</i> ²	<i>R</i> _L	<i>K</i> _f (mg ^(1-1/<i>n</i>) ·L ^{1/<i>n</i>} /g)	1/ <i>n</i>	<i>R</i> ²	<i>A</i> (L/g)	<i>B</i>	<i>R</i> ²
298	29.851	0.296	0.995	0.771–0.017	5.306	0.4398	0.919	29.587	3.296	0.957
308	26.525	0.347	0.998	0.742–0.014	4.792	0.4455	0.909	24.710	3.129	0.972
323	25.773	0.351	0.999	0.740–0.014	4.313	0.4481	0.896	20.641	2.980	0.973

**Fig. 7** Kinetic plots for the adsorption of ClO₄[−] onto cross-linked Fe-CB at different initial ClO₄[−] concentrations. Experimental conditions: dosage of cross-linked Fe-CB was 10 g/L, pH was neutral, and temperature was 25 ± 1 °C.

for perchlorate removal at a low concentration. When the initial perchlorate concentration was 1.05 mg/L, the adsorption reaction approached the equilibrium within 20 min. Meanwhile, the residual content of the perchlorate was about 0.015 mg/L which was close to the Interim Drinking Water Health Advisory level of 15 µg/L set by the US EPA in 2008.

To investigate the adsorption mechanism of cross-linked Fe-CB, pseudo first-order and pseudo second-order models have been applied, and their linear forms are expressed as follows, respectively (Özer and Dursun, 2007):

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (6)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (7)$$

where, *q_t* (mg/g) are the amount of perchlorate adsorbed per unit mass of the adsorbent at any time (*t*); *k₁* and *k₂* are the rate constants of the pseudo first and second-order models, respectively. The values of *k₁* and *k₂* can be calculated from the slopes of the linear equations. The rate constants of the kinetic models along with the regression coefficients (*R*²) are listed in **Table 2**.

Compared with the pseudo first-order model, the pseudo second-order model fitted the experimental data better, and the *R*² were closed to 1.0 at different initial concentrations. The pseudo second-order model was based on two assumptions: (1) the adsorption followed the Langmuir isotherm, and (2) the rate-limiting process should be chemical adsorption (Ho and McKay, 2000). The results of the adsorption isotherms proved that this adsorption process followed the Langmuir isotherm indeed (see in the Section 2.4), therefore it could speculate that the uptake of perchlorate onto cross-linked Fe-CB might be a chemical adsorption.

Secondly, intra-particle diffusion model was adopted to study the diffusion mechanism as follows (Ho and McKay, 2003):

$$q_t = k_{p,i} t^{1/2} + C_i \quad (8)$$

where, *k_{p,i}* (mg/(g·hr^{1/2})) (*i* = 1–3) is the intra-particle diffusion rate constant, i.e., *k_{p,1}* is the constant in the first stage called the external surface adsorption; *k_{p,2}* is the constant in the second stage as the gradual adsorption; and *k_{p,3}* is the constant in the third stage as the final equilibrium

Table 2 Kinetic model constants and correlation coefficients for adsorption of perchlorate onto cross-linked Fe-CB

<i>C</i> ₀ (mg/L)	Pseudo first-order		Pseudo second-order		Intra-particle diffusion					
	<i>k</i> ₁ (hr ^{−1})	<i>R</i> ²	<i>k</i> ₂ (g/(mg·hr))	<i>R</i> ²	<i>k</i> _{p,1} (mg/(g·hr ^{1/2}))	<i>R</i> ²	<i>k</i> _{p,2} (mg/(g·hr ^{1/2}))	<i>R</i> ²	<i>k</i> _{p,3} (mg/(g·hr ^{1/2}))	<i>R</i> ²
1.05	0.247	0.282	1759.14	1.000	0.097	0.782	–	–	–	–
4.05	0.646	0.845	630.644	1.000	2.725	0.992	0.083	0.956	0.005	0.831
13.21	0.538	0.695	34.851	0.999	9.220	0.996	0.135	0.921	0.006	0.798
27.53	0.432	0.717	6.309	1.000	18.794	0.982	0.475	0.959	0.038	0.929

(Wu et al., 2005). The rate constants are also listed in **Table 2**. The external surface adsorption stage was completed within 10 min. With the rise of the initial concentration, the $k_{p,1}$ increased significantly because of the enhancement of the concentration gradient force (Özer and Dursun, 2007). The linear portions of the second and the third stages did not pass through the origin, which indicated that intra-particle diffusion was not the rate limiting step for the adsorption process (Chatterjee and Woo, 2009; Tan et al., 2007). Moreover, $k_{p,1}$ was much greater than $k_{p,2}$ and $k_{p,3}$ (**Table 2**), which indicated that the external surface adsorption was the main adsorption mechanism.

2.6 Regeneration ability

Desorption studies were carried out to investigate the reusability of the adsorbent. The exhausted adsorbent was eluted by NaCl with different concentrations, and the results are represented in **Fig. 8**. When the NaCl concentrations were 0.5% and 1%, the desorption efficiencies of the adsorbent were just 39.91% and 43.40%, respectively. However, desorption efficiency achieved nearly 100% at equilibrium when the concentration of NaCl solution was increased to 2.5%. And the desorption reaction mainly occurred within 1 hr with the desorption efficiency being up to 95%. The reusable ability of the adsorbent was also investigated. Besides, the 2.5% NaCl solution was chosen as the eluent for adsorbent regeneration. Three cycles of adsorption–regeneration were repeated under the same condition. Compared with the fresh adsorbent, the adsorption capability of the regenerated adsorbent was not significantly decreased, and the removal efficiency of perchlorate was close to 97%. In addition, there was no obvious change in physical properties of the adsorbent after it was regenerated. The results indicated that the eluent of NaCl solution with relatively high concentration could regenerate the exhausted adsorbent efficiently, and

the cross-linked Fe-CB exhibited a good property for repeated use.

2.7 Adsorption mechanism

Compared with the cross-linked Fe-CB (**Fig. 1, line b**), the FT-IR spectrum of the cross-linked Fe-CB loaded with perchlorate is also presented in **Fig. 1, line c**, which showed no significant changes. However, two new acuti-peaks appeared at 626 and 1121 cm^{-1} , which were due to the vibration of perchlorate anions (Xie et al., 2001). **Figure 3** shows the X-EDS spectrum of the cross-linked Fe-CB loaded with perchlorate. The peaks of the elemental chlorine emerged clearly, which further proved that perchlorate was adsorbed by the cross-linked Fe-CB. Adsorption kinetics and isotherm analysis indicated that the uptake of perchlorate by the cross-linked Fe-CB was a chemical adsorption.

According to the literature, the Fe(III) iron was coordinated to three chitosan molecules, chelated through the $-\text{NH}_2$ and $-\text{OH}$ groups of chitosan polymer (Klepka et al., 2008; Shen et al., 2013). And the pH_{zpc} of the complex was about 10.6 which was higher than the protonated cross-linked chitosan whose pH_{zpc} is 6.3 (Boddu et al., 2008). When the pH value of solution was less than the pH_{zpc} , the surface of the adsorption was positively charged, which resulted in anion adsorption through electrostatic attraction. Thus, the cross-linked Fe-CB exhibited a wider efficient pH region (from 3 to 10.6) for perchlorate adsorption, especially for the pH value of natural water. In addition, the cross-linked Fe-CB still exhibited adsorption ability when the pH was higher than the pH_{zpc} . Therefore, besides the electrostatic attraction, the chelation of the Fe(III) center in the complex might be another driving force for the perchlorate uptake by cross-linked Fe-CB (Zimmermann et al., 2010; Shen et al., 2013). The possible mechanism of perchlorate removal by cross-linked Fe-CB is proposed in **Fig. 9**.

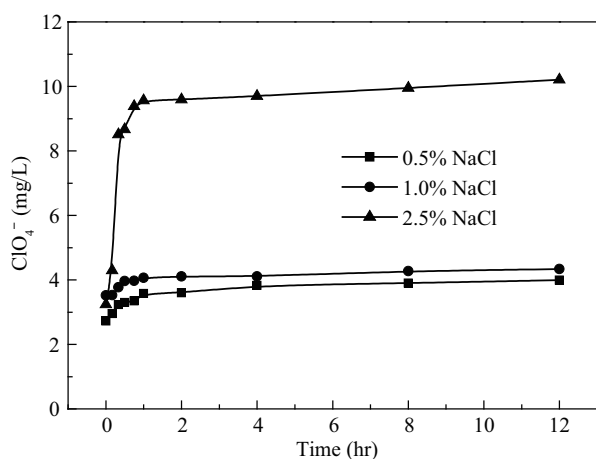


Fig. 8 Regeneration of cross-linked Fe-CB exhausted with ClO_4^- using NaCl. Experimental conditions: dosage of cross-linked Fe-CB was 10 g/L, pH was neutral, and temperature was $25 \pm 1^\circ\text{C}$.

3 Conclusions

Cross-linked Fe-CB composite was used for the removal of perchlorate from water, wherein the adsorption process was almost independent of pH in the range from 3.0 to 10.2. The co-existing anions showed negative effects on the uptake process. The equilibrium data were best described by the Langmuir isotherm. Further, the adsorption kinetics of perchlorate onto Fe-CB showed that the uptake process was rapid and the data obeyed the pseudo second-order model well. The eluent of NaCl solution with relatively high concentration could regenerate the exhausted adsorbent efficiently. Electrostatic attraction and chelation of the Fe(III) center in the complex were the main driving forces for perchlorate adsorption.

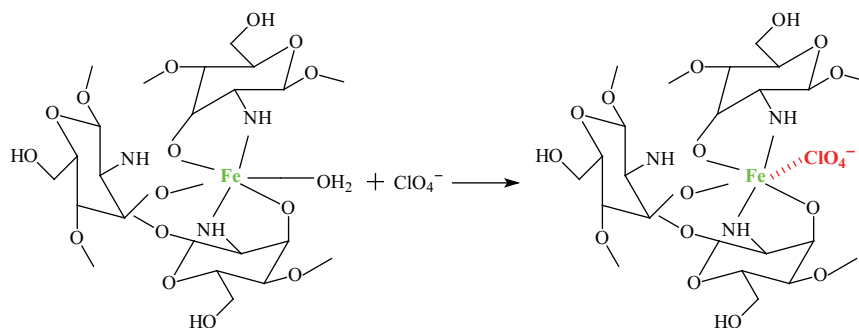


Fig. 9 Mechanism of ClO_4^- sorption by cross-linked Fe-CB.

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