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SORPTION OF As^{5+} FROM AQUEOUS SOLUTION BY LEPIDOCROCIT (γ -FeOOH) NANOPARTICLE

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ABSTRACT

γ -FeOOH nanorods adsorbent for As^{5+} removal was prepared by a chemical co-precipitation method. The maximum adsorption capacities for As^{5+} was 63.75 mg/g at pH= 6.0, higher than those of Fe_2O_3 , Fe_3O_4 ... The adsorption data accorded with Freundlich isotherms. At study pH, for arsenic, the adsorption equilibrium was gained after 90 min. Kinetic data fitted well to the pseudo-second-order reaction model. Competitive ions hindered the adsorption according to the decreasing sequence sulfate, ammonium and chloride. The high adsorption capability and good performance on other aspects make the γ -FeOOH nanorods becomes a promising adsorbent for the removal of As (V) from groundwater.

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1 INTRODUCTION

Arsenic is highly toxic and carcinogenic and has been recorded by the World Health Organization as the first priority issue. Actual and chronic arsenic exposure via drinking water has been reported in many countries, especially Argentina, Bangladesh, Mexico, Taiwan, Chile and Vietnam, where a large proportion of groundwater is contaminated with arsenic at levels from 100 to 2000 μ g/L (Smedley and Kinniburgh, 2002). To address this problem, the WHO has set a maximum guideline concentration of 10 μ g/L for arsenic in drinking water.

In natural water, arsenic is primarily present in inorganic forms and exists in two predominant species, arsenate As^{5+} (H_3AsO_4 , $H_2AsO_4^-$, $HAsO_4^{2-}$) and arsenic As^{3+} (H_3AsO_3 , $H_2AsO_3^-$, $HAsO_3^{2-}$) (Ferguson and Gavis, 1972; Manning *et al.*, 2002). As^{3+} is much more toxic and mobile than As^{5+} . However, groundwater in nature, after exposure to air, the majority As^{3+} transferred to form As^{5+} (Cullenand Reimer, 1989).

Iron oxides indeed have been used for arsenic removal (Stalidis *et al.*, 1989; Lehmann *et al.*, 2005; Leupin *et al.*, 2005) as other chemicals e.g. alumina (Lin and Wu, 2001), zeolites (Elizalde-González *et al.*, 2001), titanium dioxide (Pena *et al.*, 2005), and hydrotalcite (Lazaridis *et al.*, 2002). In most cases, these low cost materials used as filters (Leupin *et al.*, 2005) while their modelling was attempted by the mechanism of surface complexation (Lehmann *et al.*, 2005).

Among the possible treatment processes, adsorption is considered to be less expensive than membrane filtration, easier and safer to handle as compared to the contaminated sludge produced by precipitation, and more versatile than ion exchange (Guo *et al.*, 2005). Adsorption process is considered to be one of the most promising technologies because the system can be simple to operate and cost effective (Jang *et al.*, 2006).

Among a variety of adsorbents for arsenic removal, iron (hydro)oxides including amorphous hydrous ferric oxide, poorly crystalline hydrous ferric oxide (ferrihydrite) (Jia and Demopoulos, 2005), goethite

(Fendorf *et al.*, 1997) and akaganeite (Deliyanni *et al.*, 2003) are well-known for their ability to remove inorganic arsenic from aqueous system and the low cost. In general, the As^{3+} adsorption is normally less effective than the As^{5+} adsorption by these adsorbents. FeOOH has high adsorption capacity on arsenic (Deliyanni *et al.*, 2003; Guo and Chen 2005), but none of them could effectively remove both As^{5+} and As^{3+} simultaneously.

In the present, a γ -FeOOH nanoparticle adsorbent was prepared by a chemical co-precipitation method, which was easy to operate and reduce cost. The adsorbent was characterized and evaluated for its adsorption behavior of arsenate. It exhibited high adsorption capacity for As^{5+} .

2 MATERIALS AND METHODS

2.1 Materials

Stock solutions of As^{5+} 1000mg/L (Merck, Darmstadt, Germany) and $Na_2HAsO_4 \cdot 7H_2O$ (Merck, Darmstadt, Germany) were purchased and prepared. Arsenate working solutions were freshly prepared by diluting $Na_2HAsO_4 \cdot 7H_2O$ with double distilled water. HNO_3 (0.1–0.5 N) and NaOH (0.1–0.5 N) were used for adjusting the pH of the arsenate solution as necessary. The ammonium (NH_4^+) stock solution (500 mg NH_4^+ /L), the chloride (Cl^-) stock solution (500 mg Cl^- /L) and the sulfate (SO_4^{2-}) stock solution (500 mg SO_4^{2-} /L) were prepared separately from ammonium chloride (NH_4Cl) (Fisher, certified A.C.S., USA) and sodium sulfate (Na_2SO_4) (Fisher, certified A.C.S., USA). Both solutions were used as the competing ions in some arsenic adsorption experiments. Arsenic in solutions were analyzed by Atomic Absorption Spectrometer (AA 7000 – HVG1 Shimadzu, Japan). All sorption data was analysed by the Originlab 8.5.1 software.

2.2 Methods

2.2.1 Preparation of γ -FeOOH adsorbent

The γ -FeOOH adsorbent was prepared according to the following procedure described by Schwertmann and Fechter (1994). $FeCl_2 \cdot 4H_2O$ (12 g) was dissolved in 300 mL distilled water with vigorous stirring. The beaker was equipped with a glass electrode connected to a pH meter, a gas inlet connecting an air or oxygen cylinder and a dropping funnel containing 125 mL 1M NaOH. The pH of the system was adjusted to 6.5–6.8 by adding NaOH dropwise, then the gas cylinder was opened and aerated at rate of 2 L/min. The initial greenish black precipitate became orange after 20 min. Throughout the reaction, the pH of the suspension was maintained at 6.5–6.8, by adding NaOH from

the dropping funnel. The precipitate was then centrifuged, washed and dried. The dry material was stored in a desiccator.

2.2.2 γ -FeOOH nanoparticle

Powder X-ray diffraction (XRD) was recorded on a Scintag-XDS-2000 diffractometer with Cu $K\alpha$ radiation ($\lambda=1.54059$). Samples morphology was detected by scanning electron microscopy (SEM) on HitachiH-7500 (Japan).

2.2.3 Batch sorption tests

To determine the amount of adsorbed arsenate under the given conditions, approximately 0.1g of adsorbent was weighed and placed in a 250-mL Erlenmeyer flask. One hundred milliliters of arsenate solution were added into the flask. The concentration of the arsenate solution ranged from 40 to 1000 mg/L depending on the type of experiment. Ionic strength was not adjusted during adsorption. The flask was capped and shaken at 180 rpm on an orbital shaker for 24 h to ensure approximate equilibrium. All batch experiments were conducted at room temperature (20°C) unless stated otherwise. The pH was manually maintained at a designated value pH 6.0 in such a way as pH was initially adjusted to a defined value with 0.01 N HNO_3 and NaOH and then measured and adjusted at an interval of 2 h. After 24 h of the reaction period, all samples were centrifuged at 10000 rpm for 5 minutes and filtered through a 0.45- μ m membrane filter and the filtrate was analyzed for arsenate. This procedure was used in all adsorption experiments for evaluating isotherms, pH effects, and interferences of competing ions, except for kinetic experiments. The quantity of adsorbed arsenate was calculated by the difference of the initial and residual amounts of arsenate in the solution divided by the weight of the adsorbent. The amount of adsorbed metal was calculated from the following expression (Melichová and Hromada, 2013):

$$q = V (C_i - C_e) / B$$

Where q is the metal uptake or sorption capacity of adsorbent (in mg/g of adsorbent); C_i and C_e are the metal concentrations before and after adsorption, respectively, B is the mass of adsorbent used and V is the solution volume used.

2.2.4 Studies of Adsorption Isotherm effect

Experiments for studying arsenic adsorption isotherm were conducted at 20°C and pH 6 by following the batch adsorption procedure. A series of different initial concentrations of As^{5+} solutions (40–1000 mg/L) at pH 6 were used. For estimating the thermodynamic parameters of arsenic adsorp-

tion, the isotherm experiments were also conducted at 20°C.

a. Studies of Adsorption time effect

The effects of time on arsenate adsorption were examined in a series of batch sorption experiments that used the same initial As^{5+} concentration (100 mg/L) while maintaining time at different values from 0 to 180 min.

b. Adsorption Kinetics Studies

Arsenic adsorption kinetics was evaluated at 20°C and pH 6. The initial arsenate As^{5+} solution concentration was 100 mg As/L. The kinetic experiments were conducted in a 250mL flask. The flask was shaken at 180 rpm. In this experimental set up, the temperature of the solution inside the flask was well maintained at 20°C, pH was maintained at around pH 6 and experiment periods were from 0 to 180min.

c. Studies of Adsorption pH effect

The effects of pH on arsenic adsorption were examined in a series of batch sorption experiments that used the same initial As^{5+} concentration 100 mg/L while pH was maintained at different values between 4 and 8.

d. Arsenic Adsorption with competing other ions

The interference of ammonium (NH_4^+), chloride (Cl^-) and sulfate (SO_4^{2-}) on arsenate adsorption was evaluated in batch experiments. The experimental

method was similar to the batch adsorption method described previously. However, the arsenic working solutions for these competing adsorption experiments were prepared with the separate addition of ammonium, chloride and sulfate solutions into the arsenic solution. The initial addition of arsenic was 100 mg/g adsorbent using an arsenic solution of 100 mg As/L and the pH was maintained at pH 6 approximately. The concentrations of the competing anions used in the experiments were from 1 to 120 mg/L for ammonium, chloride and sulfate.

3 RESULTS AND DISCUSSION

3.1 Characterization of γ -FeOOH adsorbent

Lepidocrocite nanoparticles applied in this work consisted mainly of γ -FeOOH, characterized by the basic reflection appearing at 2θ of 44.9°, as shown in the XRD diagram in Figure 1a and 1b.

This is evident from the XRD diagram, where the oxide appears in the form of lepidocrocite (γ -FeOOH) and hematite (α - Fe_2O_3) (Fig. 1b). The α - Fe_2O_3 percentage is very low because it is a by-product of the synthesis process, and thus the corresponding peaks might be standing for γ -FeOOH (Fig. 1a).

From Figure 2, the research results indicated the γ -FeOOH was nanorods. A typical SEM image of the prepared sample was shown in Figure 2, which reveals that the Lepidocrocite nanorods was about 20nm in diameter and up to 100nm length.

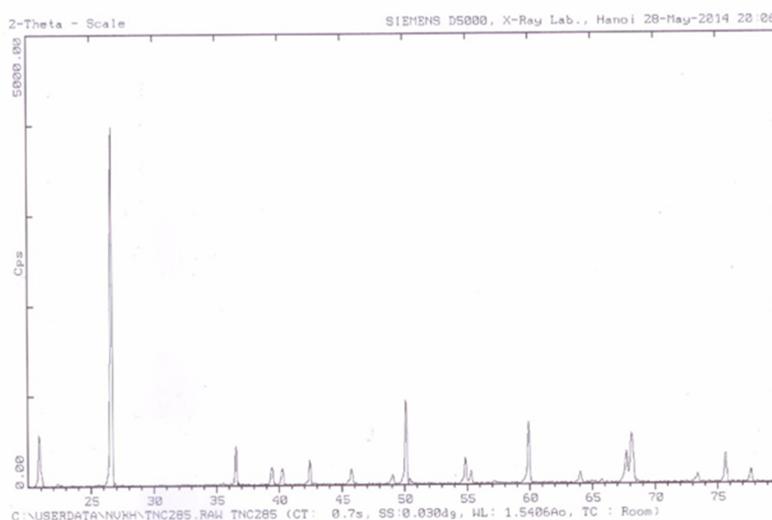


Fig. 1a: XRD patterns of γ -FeOOH samples was synthesized

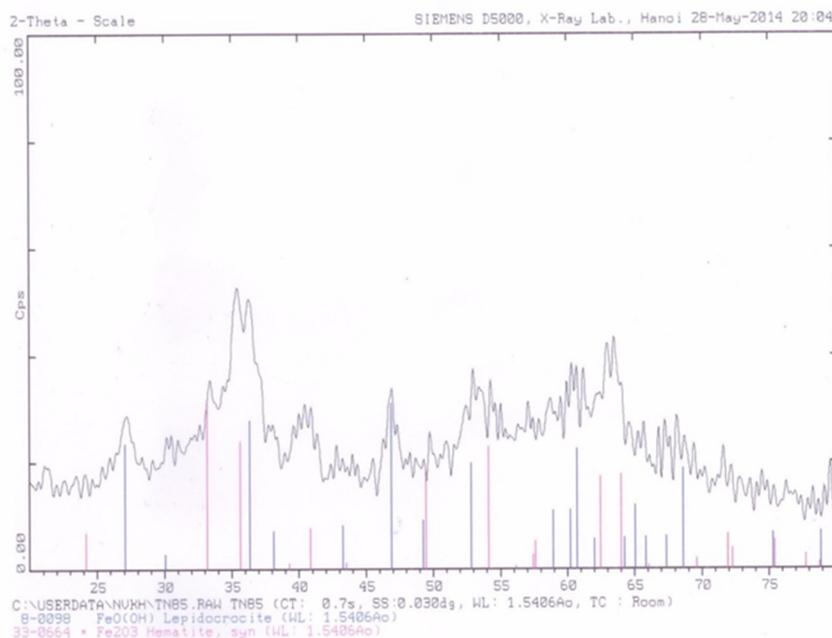


Fig. 1b: XRD patterns of samples was synthesized

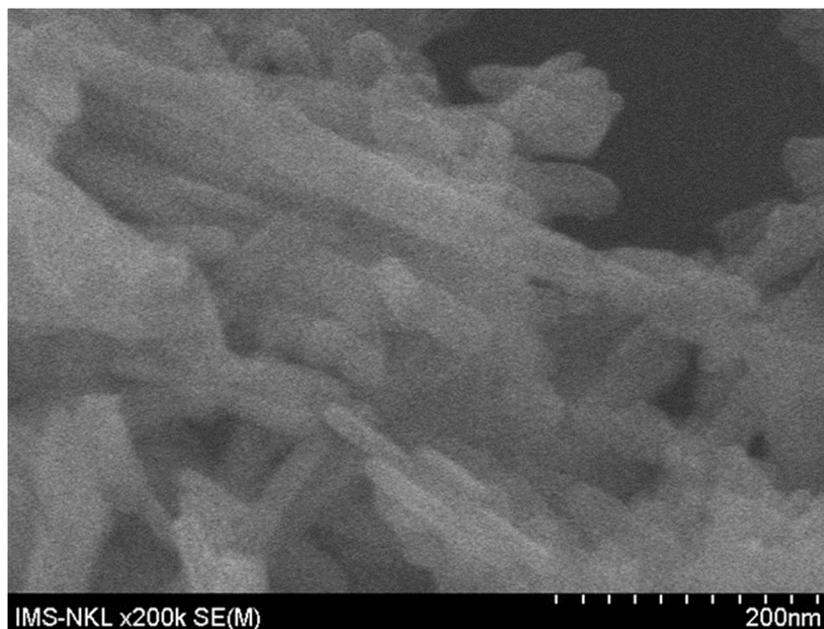


Fig. 2: The SEM of γ -FeOOH samples

3.2 Batch sorption tests

3.2.1 Adsorption isotherm of γ -FeOOH adsorbent

The adsorbents were tested for adsorption of As (V), as shown in Figure 3. The sorption capacity of As (V) by γ -FeOOH was high when it was compared to goethite (Matis *et al.*, 1999).

The adsorption isotherm of γ -FeOOH for As⁵⁺ was presented in Figure 3. In this study, both Langmuir

and Freundlich isotherms were employed to describe the adsorption isotherms obtained in the figure. The adsorption constants obtained from the isotherms are listed in Table 1. The correlation coefficient (R^2) values of the Langmuir isotherms for As⁵⁺ was 0.92, while 0.93 was for Freundlich isotherms. High regression coefficients suggested that both Freundlich and Langmuir models were suitable for describing the adsorption behavior of As⁵⁺ by γ -FeOOH.

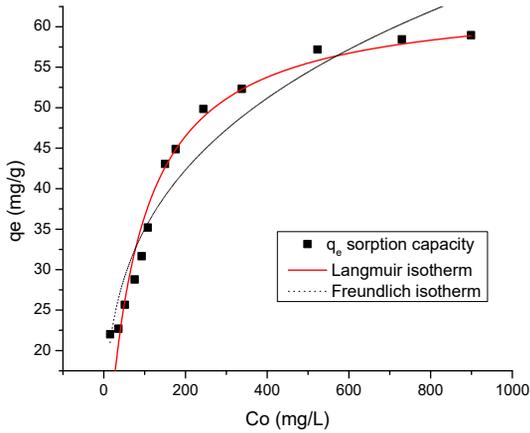


Fig. 3: Langmuir and Freundlich isotherm

The Langmuir expression was described as

$$q = \frac{q_m b C_e}{1 + b C_e}$$

Where q is the amount of As⁵⁺ adsorbed, mg/g; q_m is the maximum As⁵⁺ uptake value corresponding to sites saturation, mg/g; C_e is the equilibrium As⁵⁺ concentration in solution, mg/L; and b is the ratio of adsorption/desorption rates, the sorption of As⁵⁺ by γ-FeOOH is shown in the Table 1.

Table 1: Langmuir and Freundlich isotherm parameters for As⁵⁺ adsorption on γ-FeOOH adsorbent at pH 6

Langmuir model			
As species	q _m (mg/g)	b (L/mg)	R ²
As ⁵⁺	63.75	0.90	0.92
Freundlich model			
As species	K _F (L/mg)	n	R ²
As ⁵⁺	9.88	3.64	0.93

The Freundlich expression was described as

$$q_e = K C_e^{1/n}$$

Where K is equilibrium constant indicative of adsorption capacity, n is adsorption equilibrium constant

3.2.2 Adsorption kinetics of As⁵⁺ by γ-FeOOH adsorbent

The kinetic of adsorption is one of the important characteristics that define the adsorption efficiency. Hence, in the present study, the kinetic of arsenic

adsorption was analyzed to understand the adsorption behavior of γ-FeOOH. Figure 4 shows the adsorption data of As⁵⁺ by γ-FeOOH at different time intervals. As seen in Figure 4, the adsorption of As⁵⁺ to γ-FeOOH was found to be time dependent. The adsorption of As⁵⁺ was rapid for the first 45 min when the removal rate reached 87%. At pH 6, for As⁵⁺, the removal rate was 97 % after 90 min and the adsorption equilibrium was approached.

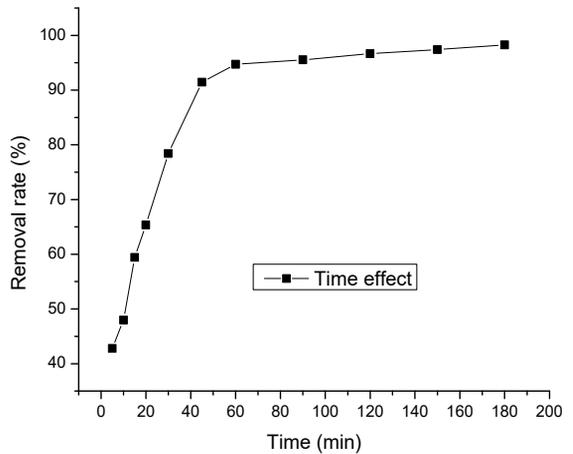


Fig. 4: Time effect sorption of As (V) by γ-FeOOH

The pseudo-first-order adsorption and pseudo-second-order adsorption were used to test the adsorption kinetic data. The pseudo-first-order rate expression of Lagergren is given following Chiou and Li (2003).

$$\log (q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (1)$$

or

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

Where q_e and q_t are the amount of arsenic adsorbed on adsorbent (mg/g) at equilibrium and time, and k₁ is the rate constant of pseudo-first-order adsorption. The pseudo-second-order rate model is expressed following Ho and McKay (1999).

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

Where k₂ is the constant of pseudo-second-order rate (g/mg·min). The experimental data of q_e, q_t and k₂ can be determined from the slope and the intercept of the plot of t/q_t against t.

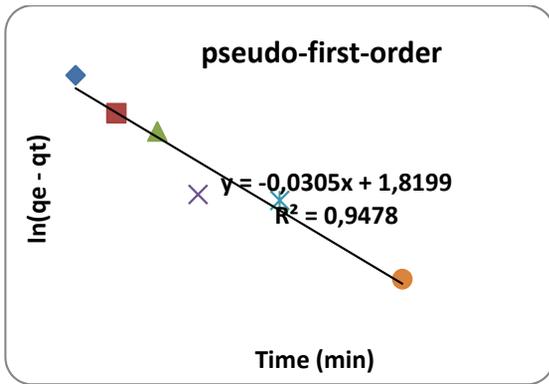


Fig. 5: Pseudo-second-order

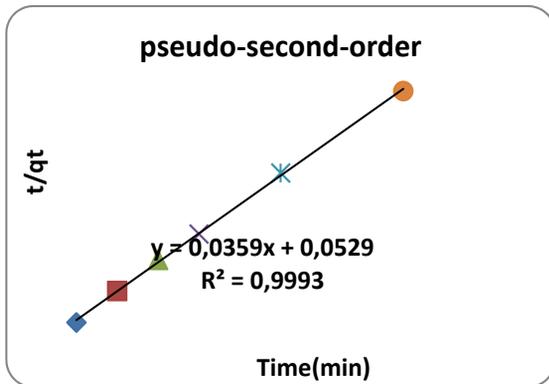


Fig. 6: Pseudo-second-orders

Table 2 presents lists of the results of rate constant studies for As^{5+} by pseudo-first-order and pseudo-second-order. The value of correlation coefficient R^2 for the pseudo-first-order adsorption model are 0.947 for As^{5+} , (Fig. 5) and the adsorption capacities calculated by the model are different to those determined by experiments. The values of R^2 for the pseudo-second-order are extremely high (>0.99) (Fig.6), for As^{5+} , the experimental data fitted to the pseudo-second-order model better than the pseudo-first-order model. Therefore, it can be concluded that the pseudo-second-order model is more suitable to describe the adsorption kinetic of arsenic on γ -FeOOH. The rate of adsorption depends on the driving force, concentration gradient. In case of pseudo-first-order, it is proportional to the concentration (ΔC) and in pseudo-second-order, rate is proportional to the square of concentration gradient (ΔC^2) which refers to both external as well as internal mass transfer (Jain *et al.*, 2003). This evidence shows that both the external and internal mass transfer is taken place. The As^{5+} ions existed as negative ions (Kim *et al.*, 2004) in the experimental conditions. Therefore, As^{5+} ions may be easy to diffuse into the external and internal adsorption sites of adsorbent. So there was a fast removal rate of As^{5+} before 45 min. This was not contradicting with the adsorption isotherms.

Table 2: composition of pseudo-first-and pseudo-second-order adsorption rate constants

Pseudo-first-order model				
As species	$q_{e, exp}$ (mg/g)	k_1 (min^{-1})	$q_{e, cal}$ (mg/g)	R^2
As^{5+}	61.79	0.03	58.17	0.947
Pseudo-second-order model				
As species	$q_{e, exp}$ (mg/g)	k_2 ($g/mg \cdot min$)	$q_{e, cal}$ (mg/g)	R^2
As^{5+}	61.79	0.02	63.75	0.999

3.3 Effect of pH on arsenate removal

As^{5+} adsorption onto γ -FeOOH as a function of pH at initial concentration of 100 mg/L was studied, as shown in Figure 7. Obviously, the As^{5+} removal was evidently dependent on pH with the greatest adsorption occurring under acidic conditions and decreased with the increase of pH solution. The removal rate of As (V) decreased from 100 to 31.2 % with increasing pH from 4 to 8. During this experiment we do all the tests at pH 6 because the pH of the groundwater (Lam Dong province) in the range of 5.8-6.3

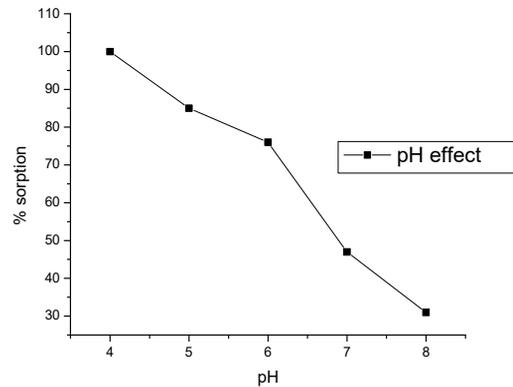


Fig. 7: pH effect sorption of As^{5+} by γ -FeOOH

The speciation of arsenic in solution and the surface charge of the solid particles were significantly affected by pH. The As (V) species and the corresponding pH values for their stability were H_3AsO_4 (pH < 2), $H_2AsO_4^-$ (pH 2-7), $HAsO_4^{2-}$ (pH 7-11), and AsO_4^{3-} (pH > 12) (Guo and Chen 2005; Maity *et al.*, 2005).

3.4 Effect of other constituents on arsenic removal

In natural groundwater or waste water, several components might exist, which could compete with arsenate for the available adsorption sites or interact with arsenic itself. In this study, we selected some ions (Cl^- , SO_4^{2-} , and NH_4^+) to test the effect

of co-existing constituents. Figure 8 shows the result of the effect of co-existing ions. Cl^- had little or no effect on the arsenic removal performance of the adsorbent. This is probably explained by the ions do not compete with the arsenic ions. The oxyanions SO_4^{2-} were selected to assess the effects of co-existing anions on arsenate removal. At fixed

pH of 6, the effects of this oxyanions increased concentration levels were illustrated in Figure 8, the NH_4^+ also had little effect on the arsenic removal performance of the adsorbent. This result is in agreement with previous studies (Gu *et al.*, 2005; Ghosh *et al.*, 2006; Zhang *et al.*, 2007).

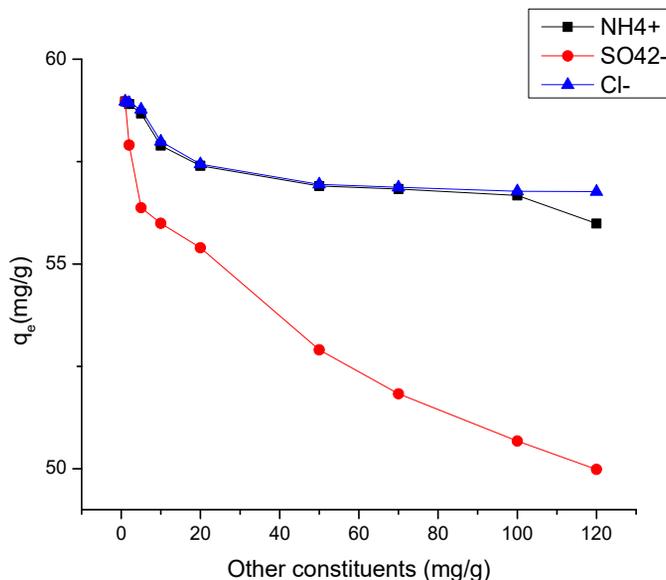


Fig. 8: Other constituents effect sorption of As^{5+} by $\gamma\text{-FeOOH}$

4 CONCLUSIONS

A novel $\gamma\text{-FeOOH}$ nanorods adsorbent for effective As^{5+} removal has been prepared by a chemical coprecipitation method. The maximal adsorption capacities for As^{5+} was 63.75 mg/g at pH 6.0. At this pH, for As^{5+} , the removal rate reached 95% after 90 min, in order to reveal useful information for the sorption mechanism, typical adsorption isotherms (Langmuir and Freundlich) were determined and X-ray photoelectron spectroscopy analysis was used. The mechanism of removal seemed rather to be chemisorption, based also on the kinetic sorption.

The adsorption of As^{5+} by $\gamma\text{-FeOOH}$ was found to be effective following pH and time exposure. The sulfate was a competitor with arsenate for adsorptive sites on the adsorbent. These results indicated that the $\gamma\text{-FeOOH}$ nanorod was an attractive adsorbent for the removal of arsenic from aqueous solution.

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