Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2015.1135479

57 (2016) 23800–23807
October

Taylor & Francis
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Sulphate removal from aqueous solutions by granular ferric hydroxide

Mahmoud Shams[a,b], Mehdi Qasemi[b], Mojtaba Afsharnia[b], Amir Hossein Mahvi[a,c,d,*]

[a] *School of Public Health, Tehran University of Medical Sciences, Tehran, Iran, Tel. +98 21 88954914; Fax: +98 21 66462267; email: Shams1996m@yahoo.com (M. Shams), Tel. +98 21 88954914; Fax: +98 21 66462267; email: ahmahvi@yahoo.com (A. Hossein Mahvi)*

[b] *School of Public Health, Gonabad University of Medical Sciences, Gonabad, Iran, Tel. +98 51 57223028; Fax: +98 51 57220577; emails: Mehdi_544@yahoo.com (M. Qasemi), Mafsharnia2000@yahoo.com (M. Afsharnia)*

[c] *Center for Solid Waste Research, Institute for Environmental Research, Tehran University of Medical Sciences, Tehran, Iran*

[d] *National Institute of Health Research, Tehran University of Medical Sciences, Tehran, Iran*

Received 4 April 2015; Accepted 17 December 2015

ABSTRACT

Sulphate is an important ion occurring in both natural waters and industrial effluents. Granular ferric hydroxide (GFH), a poorly crystallized β-FeOOH, was used to remove sulphate in a series of batch experiments. Sulphate removal increased with increase in GFH dose and more than 50% of the adsorption happened within the first 15 min of agitation. In addition, sulphate adsorption was influenced by pH and the optimum pH range was 2–7. Adsorption also increased by increasing temperature from 15 to 55°C, indicated the endothermic nature of the process. Among the co-existing anions, phosphate and bicarbonate strongly inhibited the sulphate removal. The kinetics of the process follows the pseudo-second order with a high correlation coefficient value ($R^2 > 0.99$). Equilibrium tests also showed that the sulphate removal by GFH fitted well with the Freundlich model, which indicated the multilayer adsorption of sulphate and heterogeneous distribution of adsorption sites on GFH surface.

Keywords: Granular ferric hydroxide (GFH); Adsorption kinetic; Isotherm; Sulphate

1. Introduction

The adverse effects of high sulphate concentrations in treatment of water and industrial wastewaters such as molasses [1], paper mills [2,3], textile [4,5], fertilizer production industries [6], and mining [7,8] have been well documented. Sulphate also is an etiological factor of odor problem, water and sewer pipes corrosions [9–11], BOD consumption, and reducing methane production in anaerobic sludge digesters. Over the years, a wide range of techniques have been developed for the sulphate removal from water and wastewater streams, including ion exchange [12], crystallization, membrane technology, [13,14], and precipitation [15]. Each of these techniques has its own advantages and disadvantages. In anaerobic biological treatment of sulphate-rich wastewaters, microbial activities can be inhibited due to the accumulation of sulfides. Precipitation is an another technique which is not widely applied in practice, because of the non-selective nature of the process and producing high amount of sludge which is difficult to manage [16]. Crystallization, on the other hand, is only effective for streams containing high levels of sulphate [14]. Membrane processes also are not selective, thus their application is restricted by economic considerations [17].

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*Corresponding author.

Adsorption is endorsed by many scientists as a very promising technology due to its inherent simplicity of design and operation, together with reasonable cost, sociocultural acceptance, being environmental friendly, and the potential of adsorbent recovery. Over the past years, considerable efforts have been made to develop cheap, available, and environmental friendly adsorbents [18–21]. Volcanic ash soil [22,23], goethite [24,25], aluminum [26], powdered TiO_2 [27], γ-Al_2O_3 [28], poly(m-phenylenediamine)s [29], synthesized ion exchangers [30], magnetite [31], and modified rice straw [32] are among the adsorbents that have been studied for the sulphate removal.

Granular ferric hydroxide (GFH) has been extensively studied as an effective adsorbent for the removal of a wide variety of contaminants including fluoride, perchlorate [33–35], arsenic [36–38], natural organic matter (NOM) [39], and bromate [40]. However, to the best of our knowledge, this is the first report on the sulphate removal from aqueous solutions using GFH.

2. Materials and methods

All chemicals used in our study, except for GFH, were of reagent grade. GFH was purchased from GEH Wasserchemie (gmbh & Co. KG, Osnabrück, Germany). GFH is a poorly crystallized β-FeOOH, mainly composed of the mineral akaganeite.

At first, GFH was placed in an oven at 103–105˚C for 30 min and stored in a desiccator until further use.

Sulphate removal efficiency as a result of addition of GFH dose, contact time, pH, and the presence of interfering anions was studied in a series of batch experiments. Hundred milliliters of Erlenmeyer flasks containing 50 ml sulphate solution were agitated at 250 rpm at determined times. Then, the samples were filtered through Whatman no. 42 filter papers and analyzed for residual sulphate. The concentration of sulphate determined using UNICO UV-2100 UV–vis spectrophotometer by turbidimetric method [41]. All the experiments were done in duplicate and at room temperature (23 ± 1˚C) without controlling pH. In order to better understand the sorption phenomena, the equilibrium and kinetic modeling of the process were also included in the study.

3. Results and discussion

3.1. Effect of GFH dose

Effect of GFH dose on sulphate adsorption efficiency, performed by mixing different weight of GFH in 50 ml solution containing 500 mg L^{-1} sulphate. As expected, Fig. 1 shows a higher sulphate removal with increasing GFH dose. Increasing the sulphate removal is due to higher adsorption sites being available in the solution. Fast sulphate removal was observed specially in higher adsorbent doses. More than 50% of sulphate adsorbed within the first 15 min of contact time when GFH dose increased beyond 15 g L^{-1}.

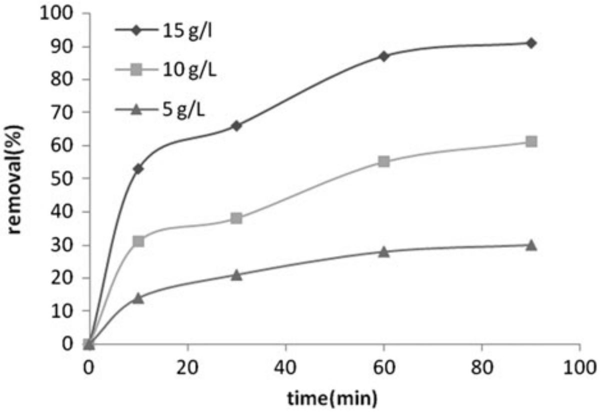

Fig. 1. Effect of GFH dose on sulphate removal—sulphate: 500 mg L^{-1}.

Other researchers also reported a higher pollutant removal when they increased the adsorbent doses [18,21,42].

3.2. Effect of initial sulphate concentration and contact time

Sulphate solutions with different initial concentrations in the range of 300–800 mg L^{-1} were agitated with fixed 15 g L^{-1} of GFH. The sulphate removal efficiency was monitored as a function of contact time.

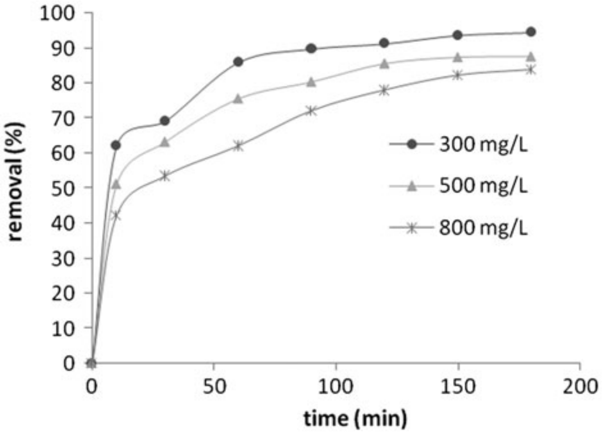

Fig. 2. Effect of time and initial concentration on sulphate removal—GFH: 15 g L^{-1}.

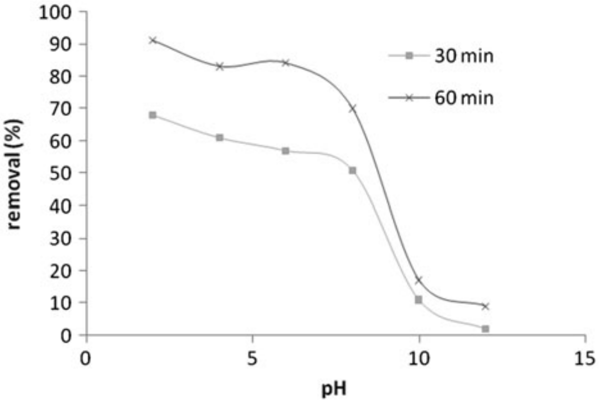

Fig. 3. Effect of pH on sulphate removal—GFH: 15 g L^{-1}, sulphate: 500 mg L^{-1}.

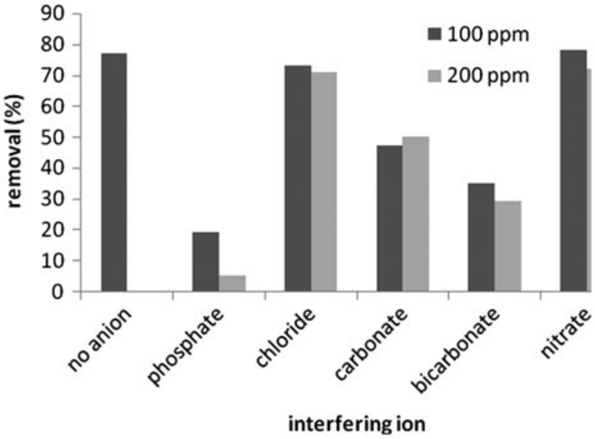

Fig. 4. Effect of interfering ions on sulphate removal—Sulphate: 500 mg L^{-1}, mixing time: 30 min, GFH: 15 g L^{-1}.

As seen in Fig. 2, after a rapid sulphate adsorption in the initial mixing time, the removal was continued with time and remained fairly constant after 150 min higher adsorption rate at the beginning of agitation time which was also observed by other researchers. The study of Eva Kumar showed that about 95% of *F* adsorbed onto GFH in 10 min and the equilibrium was achieved in 60 min of agitation [35]. In another study, 75% of bromate adsorbed by GFH when the solution was mixed for 5 min and the equilibrium was achieved in 20 min [40]. Uptake of perchlorate by GFH was also rapid in the first 30 min of mixing time and the equilibrium was achieved in 60 min [34].

3.3. Effect of pH

Sulphate adsorption efficiency, as a function of pH, was studied by adjusting the pH of sulphate solutions to 2, 4, 6, 8, 10, and 12. A specified amount of adsorbent was then added and the flasks were shacked for 60 min. As shown in Fig. 3, the sulphate removal decreased with increasing pH from 2 to 8. A sharp decrease in the sulphate removal observed when pH of the solutions was increased beyond 8. Optimum pH for the adsorption of sulphate by GFH was found to be in the range of 2–7.

It was found that the fluoride removal by GFH was optimum when pH of the solutions was adjusted between 4 and 8. [35]. An initial pH in the range of 3–7 was also favorable for the adsorption of perchlorate onto GFH [34].

3.4. Effect of interfering anions

To evaluate the possible interference effect of competing anions on sulphate adsorption onto GFH, experiments were carried out in the presence of 200 and 400 mg L^{-1} of CO_3^{2-}, HCO_3^-, NO_3^-, Cl^-, and PO_4^{3-}. As seen in Fig. 4, phosphate and bicarbonate have the greatest inhibitory effects on sulphate adsorption and the degree of anion interference decreased in the

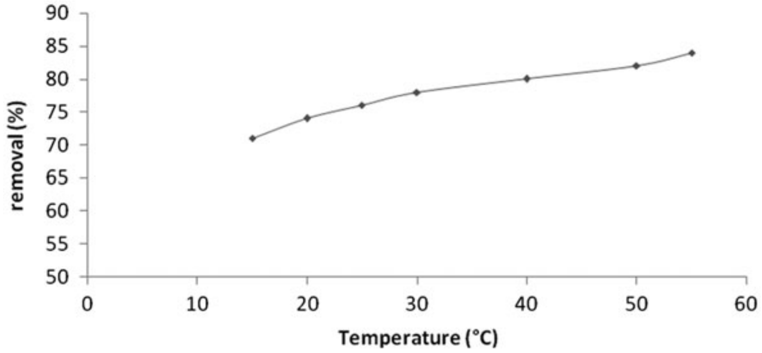

Fig. 5. Effect of solution temperature on sulphate removal—sulphate: 500 mg L^{-1}, mixing time: 30 min, GFH: 15 g L^{-1}.

following order: carbonate, chloride, and nitrate. Fig. 4 also shows a negligible decrease in sulphate adsorption when the competing ions, other than phosphate, increased from 200 to 400 mg L^{-1}. The sulphate removal decreased from 19 to 5% when phosphate concentration increased from 200 to 400 mg L^{-1}.

Phosphate also had the highest inhibitory effect on the fluoride removal by GFH and the level of interference decreased in the presence of carbonate and sulphate, respectively [35]. A negligible interference was observed due to the presence of competing ions on bromate adsorption by GFH [40].

3.5. Effect of adsorption temperature

To determine the exothermic or endothermic nature of the process, the sulphate removal efficiency was studied as a function of temperature range from 15 to 55°C. In contrast to exothermic processes, in endothermic processes, the adsorption increased with increase in temperature. As presented in Fig. 5, sulphate adsorption onto GFH increased with increase in temperature, indicating the endothermic nature of the adsorption.

Other studies showed that the fluoride and arsenic removal by GFH are also endothermic [35,43].

3.6. Kinetic study

The kinetic study is important in understanding the rates and the mechanisms of adsorption processes and gives key information about designing the full-scale treatment facilities [44]. The Lagergren's pseudo-first-order and pseudo-second-order rate equations along with Morris intraparticle diffusion model, applied to the experimental data in order to find the best-fit model.

The Lagergren's first-order rate equation is a widely used model to describe the adsorption process occurred in a liquid phase. The linear form of Lagergren's first order is expressed by Eq. (1) as below:

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

where q_e and q_t are the milligrams of sulphate adsorbed per gram of GFH, at equilibrium and at time t, respectively. K_1 is the rate constant of first-order adsorption (min^{-1}). As presented in Fig. 6, from the slope and intercept of a plot of $\log(q_e - q_t)$ vs. t, the values of k_1 and q_e can be calculated, respectively.

The linear form of pseudo-second-order is expressed by Eq. (2) as below:

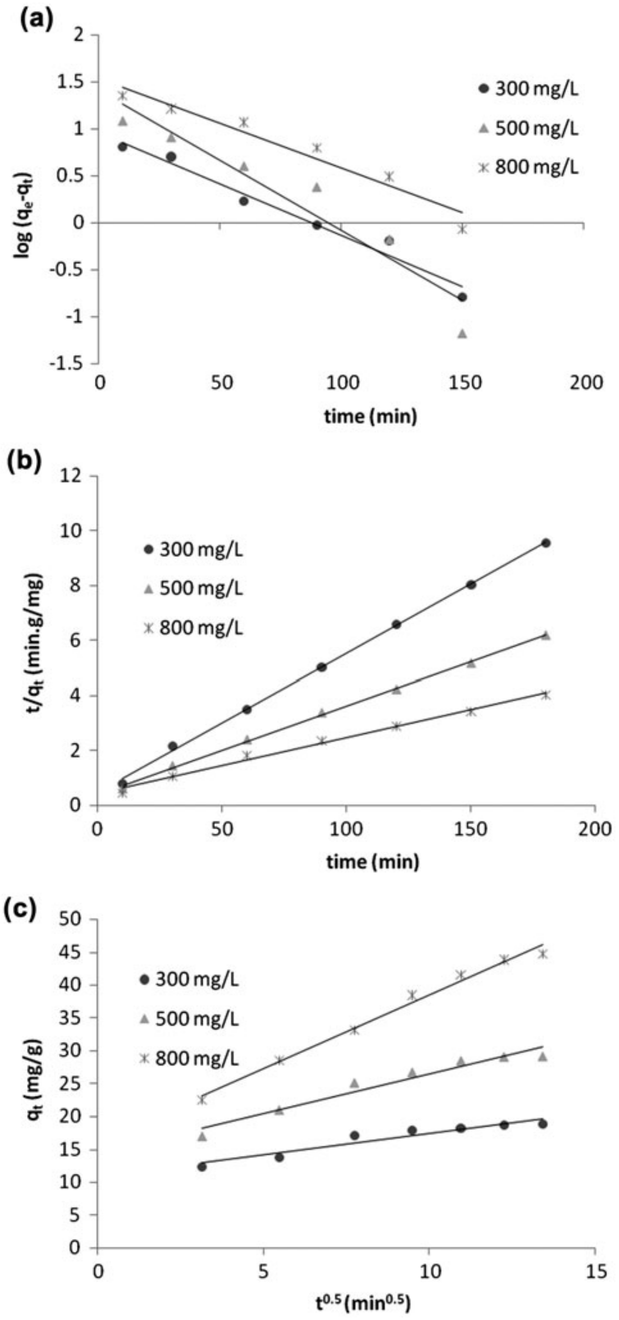

Fig. 6. Fitting of (a) pseudo-first order, (b) pseudo-second order, and (c) intraparticle diffusion models for different initial sulphate concentrations—GFH dose: 15 g L^{-1}.

$$\frac{T}{Q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \cdot t \tag{2}$$

where k_2 is the equilibrium rate constant of second-order adsorption ($\text{g mg}^{-1}\text{ min}^{-1}$) and can be

Table 1
Constants obtained from kinetic models for sulphate adsorption onto GFH

		Pseudo-first-order			Pseudo-second-order			Intraparticle diffusion	
C_0 (mg/L)	$q_{e,exp}$ (mg/g)	$q_{e,cal}$ (mg/g)	K_1 (min^{-1})	R^2	$q_{e,cal}$ (mg/g)	K_2 (min^{-1})	R^2	K_p (mg/g min$^{-0.5}$)	R^2
300	18.86	9.24	0.025	0.971	19.71	0.005	0.998	0.736	0.927
500	29.1	25.81	0.034	0.913	15.55	0.002	0.998	1.211	0.945
800	44.64	34.54	0.022	0.946	48	0.001	0.992	2.245	0.988

determined from the slope of the plot of T/Q_t vs. t. The value of q_e can also be obtained from the intercept of the plot.

The intraparticle diffusion equation can be written as Eq. (3) as follows:

$$Q_t = k_p \cdot t^{0.5} + C \tag{3}$$

where k_p and C are the intraparticle diffusion rate constant (mg g^{-1} min$^{-0.5}$) and intercept, respectively. Plot of the q_t versus the square root of time ($t^{0.5}$) results in a straight line if the intraparticle diffusion is controlled in the adsorption process.

Kinetic studies were performed by mixing GFH with solutions containing 300–800 mg L^{-1} of sulphate. The experimental data were then analyzed to find out the best-fit kinetic model.

As seen in Table 1, the values of R^2 for pseudo-second-order reaction are higher than the pseudo-first order, and the intraparticle diffusion model indicates that the rate-limiting step in sulphate adsorption onto GFH is probably a chemical adsorption. In other words, pseudo-second-order reaction showed a better conformity of $q_{e,cal}$ and $q_{e,exp}$. As presented in Table 1, $q_{e,cal}$ and $q_{e,exp}$ are increased with increase in sulphate concentrations.

Other studies showed that the pseudo-second-order kinetic model favorably explains the sorption mechanism of perchlorate and bromate onto GFH [34,40]. Fluoride and arsenic adsorption by GFH, on the other hand, followed the pseudo-first-order kinetic model [35,43].

3.7. Isotherm study

3.7.1. Langmuir isotherm

The theoretical Langmuir isotherm assumed that the sorption occurred at specific homogeneous sites within the adsorbent and in monolayer. The linear form of Langmuir isotherm expressed by Eq. (4) as below:

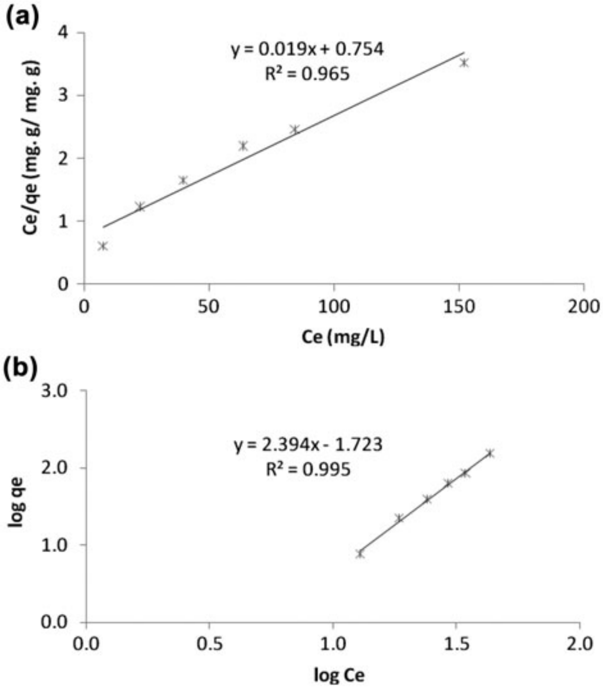

Fig. 7. Fitting the (a) Langmuir and (b) Freundlich models for sulphate sorption by GFH—mixing time: 8 h, GFH: 15 g L^{-1}.

$$\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{q_m b} \tag{4}$$

Hence, as shown in Fig. 7, by plotting C_e/q_e vs. C_e, q_m, and b can be obtained from the slope and the intercept, respectively.

3.7.2. Freundlich isotherm

This empirical equation is the earliest known model describing the non-ideal and reversible adsorption [45]. The Freundlich expression is an exponential equation, and therefore, assumes that the concentration of adsorbate on adsorbent surface increased with concentration to form a multilayer adsorption [46].

Log q_e against log C_e plotted according to the linear form of the Freundlich Eq. (5), presented below:

$$\log q_e = \log k_f + \left(\frac{1}{n}\right) \log C_e \tag{5}$$

GFH was mixed with solutions containing 200–800 mg L^{-1} of sulphate for 8 h and the data were then analyzed for isothermal study. According to Fig. 7(b), Freundlich isotherm is the best-fit model for sulphate adsorption by GFH, which shows that the adsorption of sulphate onto GFH occurred in multilayer and the active sites on the GFH surface distributed heterogeneously.

In a study on the fluoride removal by GFH, the q_{emax} was obtained as 7.0 mg g^{-1} and the Langmuir model was consistent with the experimental data [35]. Langmuir model also described the bromate adsorption by GFH [40]. In another study on perchlorate adsorption onto GFH, q_{emax} was obtained to be 20 mg g^{-1} [34].

Langmuir model descried the sulphate sorption of modified rice straw as a lignocellulosic agricultural residue [32]. Sulphate adsorption by Goethite also described well by the Langmuir equation in another study [47]. The adsorption capacity of sugarcane bagasse cellulose amended with zirconium oxychloride was found to be 0.4 mol g^{-1} [48].

4. Conclusion

The present study confirms the applicability of GFH in the sulphate removal from aqueous solutions. The following are the highlights of the current study:

The sulphate removal increased with increase in GFH dose due to the more available adsorption sites in higher doses.

Adsorption occurred rapidly and most of the adsorption happened in the first minutes of agitation time.

The sulphate removal by GFH was highly influenced by pH and optimum pH was found to be in the range of 2–7.

Anionic impurities inhibited the sulphate adsorption in the below order:

phosphate > bicarbonate > carbonate > chloride > nitrate.

The sulphate removal by GFH was endothermic and followed the pseudo-second-order reaction.

The multilayer sorption and heterogeneous distribution of active sites on the GFH predicted the behavior of sulphate adsorption onto GFH.

Acknowledgments

The authors gratefully acknowledge the financial support from the Research Office, Gonabad University of Medical Science.

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