Journal of Petroleum & Environmental Biotechnology

Aftabtalab and Sadabadi, J Pet Environ Biotechnol 2015, 6:1
DOI: 10.4172/2157-7463.1000200

Research Article Open Access

Application of Magnetite (Fe_3O_4) Nanoparticles in Hexavalent Chromium Adsorption from Aquatic Solutions

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Abstract

Environmental pollution such as hexavalent chromium has been extremely released into the environment and significantly increased global concern. Hexavalent chromium has carcinogen and mutagen effect on organisms. In this study magnetite (Fe_3O_4) nanoparticles were successfully synthesized by sol-gel method with the purpose of removing Cr(VI) from waste water. The phase structures, morphologies, particle sizes, chemical composition, and magnetic properties of magnetite nanoparticles have been characterized by X-ray diffraction, scanning electron microscopy, energy –dispersive analysis by X-ray spectrometer and vibrating sample magnetometer. Synthesized magnetite nanoparticles demonstrated high capacity of hexavalent chromium adsorption. The maximum adsorption of Cr(VI) by Fe_3O_4 nanoparticles occurred at pH 8.2. The adsorption efficiency of Cr(VI) was explained in terms of Freundlich and langmuire equations.

Keywords: Freundlich; Hexavalent chromium; Langmuire; Magnetite nanoparticles; Sol-gel

Introduction

The large portions of different chemical compounds are reasons of adverse effects on the environment. Heavy metals are non-biodegradable and accumulated in living organisms through the food chain. One of these heavy metals which have the toxic effect on mankind is hexavalent chromium. The main sources of Cr(VI) contamination are leather tannery, alloy and steel manufacturing, pain production and metal cleaning industries. The intake of Cr(VI) by humans leads to gastrointestinal irritation, central nervous system irritation and lung carcinoma. Different technologies have been used for removal of chromium compound from an aquatic system which includes chemical precipitation, ion exchange, activated carbon adsorption, use of membrane technology, evaporation recovery and reverse osmosis. However, the conventional treatment technologies require expensive equipment, high energy and generate enormous quantity of sludge. Adsorption processes are one of the most important methods for metals' removal and attract much attention because of inexpensive, efficient and simple methods [1,2].

The use of magnetic nanoparticles for separation and treatment of waste water is new methodology that is faster and simpler. Magnetite nanoparticles have been widely studied because of structural and functional elements have various novel applications [3]. Magnetite (Fe_3O_4) nanoparticles received considerable attention not only in the fields of medical applications, including radiofrequency hyperthermia, photo magnetics, magnetic resonance imaging (MRI), medical diagnostics, cancer therapy, but also in the field of waste water treatment [4-7]. There are various chemistry- based methods to synthesize nanoscaled magnetite (Fe_3O_4) particles such as co-precipitation or precipitation, solution combustion, emulsion technique, hydrolysis and thermolysis of precursors, flow injection synthesis, sonochemical reaction and sol-gel. Sole-gel method has advantages in comparison with other techniques for examples: it can produce high purity nanoparticles because the organo-metallic precursor can be highly controllable, by sol-gel can provide a simple, economic, effective method to produce high quality nanoparticles and also ecofriendly procedure [8-11]. In this studied we synthesized magnetite nanoparticles via sol-gel method with super paramagnetic property for Cr (VI) removal from waste water.

Experimental Details

Preparation of magnetite nanoparticles

Ferric nitrate (Fe $(NO_3)_3$. $9H_2O$) extra pure purchased from Finar Chemicals Limited (india) and ethylene glycol ($C_2H_6O_2$) extra pure purchased from Finar Chemicals Limited (India) was used for the preparation of magnetic nanoparticles in this present study. Exactly 0.04 M of ferric nitrite was dissolved in 25 ml ethylene glycol and placed in magnetic stirrer for 2 hours at 70°C to obtained brown gel. Then the gel kept in oven at 250°C temperatures for drying. After drying, the xerogel was annealed at 250°C.

Characterization

X-ray powder diffraction carried out with (D8-Advanced XRD-Bruker) using Cu-Ka radiation (λ=0.15418 nm) for measuring the phase structure of magnetite nanoparticles. Scanning Electron Microscopy (SEM) equipped with an energy dispersive X-ray analyzer (EDS) (S-3400 N) was used to determine the morphology of the synthesized magnetite nanoparticles. EDX analysis is an analytical technique used for the elemental analysis or chemical characterization of a sample [9]. Magnetization measurements were performed at room temperature by using (Lake Shore's VSM) (7410).

Result and Discussion

Characterization of magnetite nanoparticles

XRD patterns of the magnetite nanoparticles: The XRD patterns

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Received September 27, 2014; **Accepted** November 28, 2014; **Published** February 10, 2015

Citation: Aftabtalab A, Sadabadi H (2015) Application of Magnetite (Fe_3O_4) Nanoparticles in Hexavalent Chromium Adsorption from Aquatic Solutions. J Pet Environ Biotechnol 6: 200. doi:10.4172/2157-7463.1000200

of magnetite nanoparticles obtained at 250°C. The synthesized nanoparticles were matched with the PDF card No. #750033, where the diffraction peaks at 2θ= 35.48°, 37.11°, 43.12°, 57.03°, and 62.62° can be assigned to (311), (222), (400), (511) and (440) planes of Fe_3O_4 respectively and shows there is no peaks corresponding to ferric nitrite or other iron oxide Figure 1. The average crystallite size was calculated from FWHM (full width and half maximum) by using Deby- Scherrer equation (1). The average crystalline size (D) for synthesized magnetite nanoparticles at 250°C is12.46 nm and d-spacing is calculated to be 2.02 nm.

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

The inter planner spacing obtain by the Bragg's equation (2).

$$n\lambda = 2dx\sin\theta \quad (2)$$

SEM and EDS image of the magnetite nanoparticles: SEM image of Fe_3O_4 sample indicated different shape and sizes of nanoparticles. SEM image showed nanoparticles are agglomerated. EDS shows elements that are present in magnetite nanoparticles Figure 2.

Results from Table 1 confirm the percentage of existence elements in Fe_3O_4.

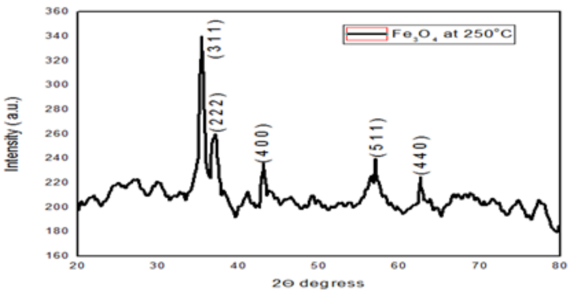

Figure 1: X-ray diffraction of magnetite nanoparticles at 250°C.

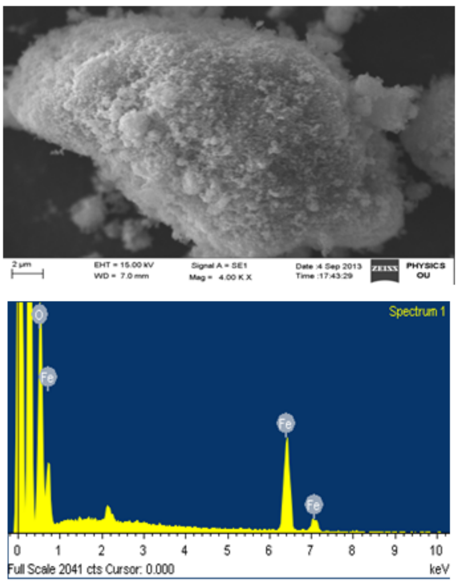

Figure 2: SEM and EDS images of magnetite nanoparticles at 250°C.

Element	Weight%	Atomic%
O K	43.08	72.54
Fe K	56.92	27.46
Total	100	

Table 1: Percentage of elements in magnetite nanoparticles at 250°C.

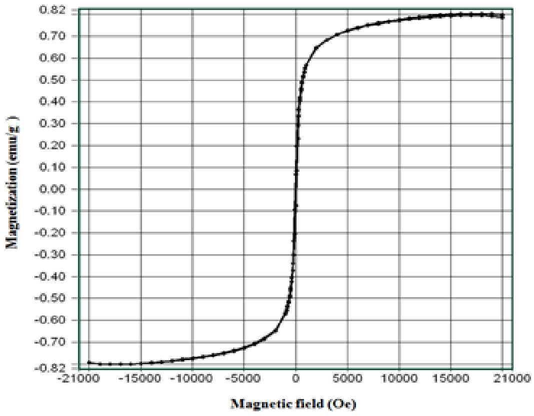

Figure 3: Magnetization Curves of magnetite nanoparticles synthesized at 250°C.

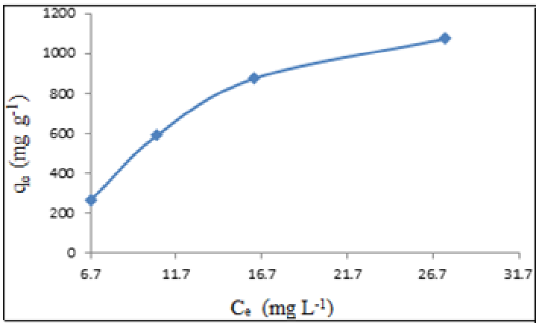

Figure 4: Plot of q_e vs. C_e for adsorption of Cr(VI) on to Fe_3O_4 nanoparticles at pH 8.2, 0.01 g Fe_3O_4.

Magnetic properties of magnetite nanoparticles obtained at 250°C: The paramagnetic properties of the magnetic particles were verified with the magnetization curve measured by vibrating sample magnetometer (VSM). The hysteresis loops of the Fe_3O_4 nanoparticles measured at room temperature are illustrated in Figure 3. The saturated magnetization value Ms of Fe_3O_4 nanoparticles obtained at 250°C is found to 0.82 emu/g.

Adsorption isotherm

The equilibrium adsorption isotherm model is the number of mg adsorbed per gram of adsorbent (q_e) versus (C_e) which is the equilibrium concentration of the chromium in the solution (mg L^{-1}), the equilibrium concentration of adsorbate describing as an interactive behavior between adsorbate and adsorbent. The Figure 4 showed equilibrium adsorption isotherms were fulfilled with different initial concentrations of hexavalent chromium from 20-80 mgL^{-1} and pH 8.2. Freundlich and Langmuir models used to analyze the equilibrium adsorption data.

Freunlich: The Freundlich experimental model is, Eq. (3).

$$q_e = KC_e^{1/n} \quad (3)$$

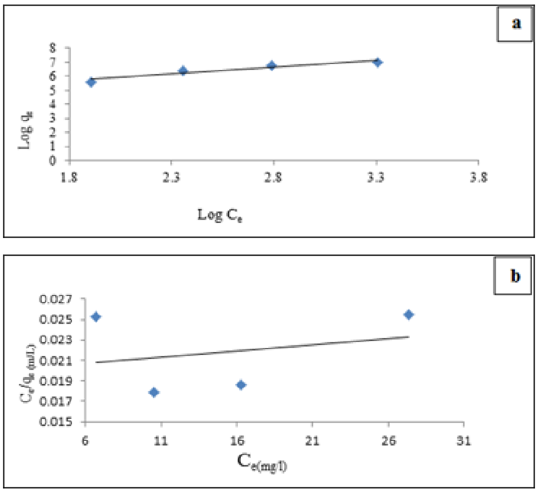

Figure 5: Freundlich (a) and Langmuir (b) isotherms of Cr(VI) onto Fe_3O_4 at pH 8.2.

Where q_e is the amount of chromium adsorbed at equilibrium in mg/g, K and n are Freundlich constants related to adsorption capacity and intensity, C_e is the solute equilibrium concentration in mg/L. Adsorption isotherms were obtained at pH 8.2 by varying the initial concentration of Cr(VI) from 20 to 80 mg/L and obtained R square value was 0.98. Figure 5 showed the adsorption of hexavalent chromium by Fe_3O_4 nanoparticles.

The K and n for different concentrations were calculated using Eq. (4) for given data of initial concentration. The finding 1/n= 0.96 (1/n<1.0) shows that the adsorption of Cr(VI) by Fe- nanoparticle is favorable.

$$Logq_e = \log K + 1/n \log C_e \tag{4}$$

Langmuir: The amount of hexavalent chromium was calculated from Langmuir equation (5):

$$\frac{q_e a_L}{K_L} = \frac{K_L C_e}{(1+K_L C_e)} \tag{5}$$

where C_e is the equilibrium concentration of the Cr (VI) in the solution (mg L^{-1}), q_e is the amount of hexavalent chromium adsorbed per unit mass of adsorbent (mg g^{-1}), at equilibrium concentration, C_e, aL (L mg^{-1}) and KL (L g^{-1}) are the Langmuir constants with aL related to the adsorption energy and qm [=KL/aL] signifies the maximum adsorption capacity (mg g^{-1}). The linearization form of Langmuir isotherm after is (6):

$$\frac{C_e}{q_e} = \left(\frac{a_L}{K_L}\right) + \left(\frac{1}{K_L}\right) \tag{6}$$

The aL and K_L values calculated from the slope and intercept of the plotted C_e/q_e vs. C_e. In the Figure 4b shown the adsorption of Cr(VI) by magnetite nanoparticles. The R square value calculated from data was 0.58

Conclusion

The synthesized magnetite nanoparticles were confirmed with different characterization techniques. The EDS results revealed synthetic magnetite nanoparticles have purity. The synthetic process is cost-effective and eco-friendly because it is inexpensive and less toxic iron salt. Magnetite nanoparticles have been synthesized by sol-gel method demonstrated high surface area to volume ratio which is associated to their ability for surface chemical modification. The results obtain from adsorption isotherms confirm magnetite nanoparticles have efficiency and high capacity for Cr(VI) and other pollution treatment from wastewater. The results obtain from adsorption isotherms shown magnetite nanoparticle have efficiency and high capacity for removal of hexavalent chromium at pH 8.2.

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