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Research Article

Hexavalent Chromium Treatment by High Adsorption Magnetite (Fe_3O_4) Nanoparticle

Adeleh Aftabtalab[Ȧ*], Hamed Sadabadi[Ḃ], CH. Shilpa Chakra[Ḃ] and K.Venkateswara Rao[Ḃ]

[Ȧ]Centre of Environment, Institute of Science and Technology, Jawaharlal Nehru Technological university of Hyderabad, AP, India.
[Ḃ]Centre of Nano Science and Technology, Institute of Science and Technology, Jawaharlal Nehru Technological university of Hyderabad, AP, India

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Abstract

In this study Super paramagnetic Iron Oxid (Fe_3O_4) nanoparticles synthesized by sol- gel method. The synthesized nanoparticles are characterized by UV-Visible spectroscopy, TG-DTA, TEM, XRD, EDAX and SEM. Synthesized magnetite nanoparticles are used for removal of highly toxic and mobile hexavalet chromium in the environmental waste water. Magnetite nanoparticles (Fe_3O_4) are negatively charged and Cr (VI) is positively charged. These charges are determined by the Zeta potential. Cr (VI) and the interaction with magnetite nanoparticles was studied at concentrations (20, 40 ml) and pH (8.2) levels of chromium.

Keywords: *sol-gel, magnetite nanoparticles, hexavalent chromium*

1. Introduction

Super paramagnetic Iron oxide nanoparticles, have been extensively studied because of structural and functional elements and have various applications (Lucas W. Yeary *et al*, 2005). Magnetite (Fe_3O_4) nanoparticles have attracted much attention not only in the field of magnetic medical applications, including radiofrequency hyperthermia, photomagnetics, and magnetic resonance imaging (MRI), medical diagnostics and cancer therapy and microwave devices, magneto-optics devices, sensors, high frequency applications as well as waste water treatment (H.El Ghandoor *et al*, 2012; Q A Pankhurst *et al*, 2003; T.K. Indira *et al*, 2010). Different technologies have been investigated for removal of hexavalent chromium from wastewater which includes chemical precipitation, ion exchange, activated carbon adsorption, reverse osmosis and flotation. The techniques mentioned above in combination with reduction agents such as ferrous sulfate and sulfur dioxide applied for the reduction of Cr(VI) to Cr(III) but they are not completely remove chromium. These present techniques require high energy and produce large amount of sludge. On the other hand adsorption processes are one of the most important and reliable methods for metal removal from wastewater (J. Hu *et al*, 2004; A. Bampaiti *et al*). There are various chemistry- based methods to synthesize nanoscaled magnetite (Fe_3O_4) particles such as co-precipitation or precipitation, solution combustion, emulsion technique, hydrothermal preparation and sol-gel (Juliano Toniolo *et al*, 2007; QI Hong Zhang *et al*, 2011). Magnetite nanoparticles (Fe_3O_4) have great interest because it has good characteristic for example biocompatibility, superparamagnetic properties, non toxicity, high chemical stability, easy synthesis process, etc (Fauziatul Fajaroh *et al*,2011; Wang, P *et al*, 2009; Tomohiro Iwasaki *et al*).

In this studied we synthesized magnetite nanoparticles via sol- gel method with superparamagnetic property for Cr (VI) removal.

2. Experimental

2.1. Materials

The chemicals used in this experimental work were as follows: Ferric Nitrate ($Fe(NO_3)_3$ _ $9H_2O$), Ethylene Glycol ($C_2H_6O_2$), Potassium Chromate Salt (K_2CrO_4). The reagents were used without further treatment.

2.2. Preparation of magnetite nanoparticles

The procedure of magnetite nanoparticles is showed in the Fig. 1. 0.04 mol Ferric nitrite was dissolved in 25 ml ethylene glycol with the magnetic stirrer for 2 hours at 70℃ to obtained brown gel. Then gel kept in oven at 250 ℃ temperatures for drying. After drying, the xerogel was annealed at the temperature ranging from 300℃.

$$Fe(NO_3)_3 . 9H_2O + 46C_2H_6O_6 \longrightarrow 2 Fe_3O_4 + 92 CO_2 + 192 H_2O + 9N$$

2.3 Cr (VI) stock solution

*Corresponding author's Tel: +917799576311

Cr(VI) stock solution (1000 mg/L) was prepared by dissolving of analytical grade potassium chromate (K_2CrO_4) in distilled water. Standard base 0.1 mol l^{-1} NaOH and acid 0.1 mol l^{-1} HCL solutions were used for the pH adjustment.

2.3. Characterization

UV-Visible spectroscopy (UV- 2450, SHIMADZU) was used to measure adsorption of hexavalent chromium. The annealing process of Iron oxide carried out by used thermogravimetric- differential thermal analysis (TG-DTA) (XSTAR6000). X-ray powder diffraction carried out with (D8-Advanced XRD-Bruker) using Cu-Kα radiation (λ=0.15418 nm) for measuring the phase structure of magnetite nanopartictes. Transmission electron microscopy (TEM) is used to determine the size of magnetite nanoparticles. Scanning Electron Microscopy (SEM) equipped with an energy dispersive X-ray analyzer (EDX) (S- 3400 N) was used in this study. EDX analysis is an analytical technique used for the elemental analysis or chemical characterization of a sample. Zeta potentials of magnetite nanoparticles were measured with particle size analyzer (Horiba SZ- 100).

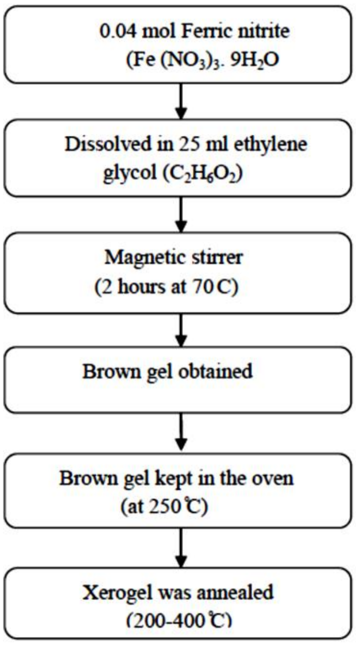

Fig.1. Magnetite nanoparticles synthesis procedure

3. Result and discussion

3.1. Characterization of magnetite nanoparticles

3.1.1. XRD patterns of the magnetite nanoparticles

The XRD patterns of magnetite nanoparticles obtained at 300° C annealing temperatures shown in Fig. 2. The diffraction peaks at 2θ= 30.12°, 35.48°, 53.50°, 57.03°, and 71.07° can be assigned to (2 2 0), (3 1 1), (4 2 2), (5 1 1) and (6 2 0) planes of Fe_3O_4 (PCPDF#750033) respectively and shows there is no peaks corresponding to ferric nitrite or other iron oxide such as, α-Fe_2O_3 and γ- Fe_3O_4. This result shows particles are pure Fe_3O_4. The crystallite size calculated from FWHM (full width and half maximum) by using Scherrer formula was found. The average crystallite size of Iron oxide synthesized at 300° C is 10.4 nm. The most intense peak (3 1 1) was used for calculating crystallite size of magnetite Fe_3O_4 nanoparticles.

$$K\lambda = D/\beta Cos\,\theta \quad (1)$$

Where K is constant and dimensionless which is approximately 0.9, λ=1.5418 Å and β is full width at half-maximum intensity (FWHM) and θ is the Bragg angle.

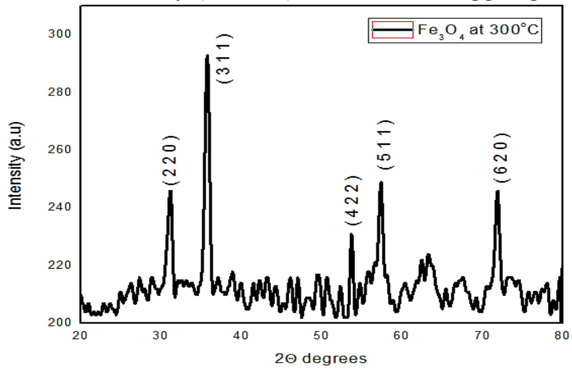

Fig. 2 X-ray diffraction of magnetite nanoparticles at 300℃

3.1.2. TG-DTA of magnetite nanoparticles

In Fig. 3 the TG curve shows three peaks that are related to weight loss and DTA curve shows two exothermic peaks. In the temperature 100-400℃ there are two weight losses due to water and organic precursor and from 400-600℃ is related to nitrite compound decomposition.

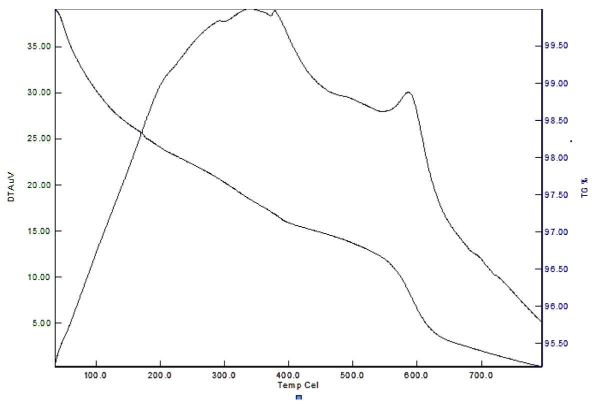

Fig. 3 TG-DTA graph of Fe_3O_4

3.1.3 TEM micrographs of Fe_3O_4 nanoparticles annealed at 300°C

Fig. 4 Shows TEM micrographs of magnetite nanoparticles annealed at 300℃ for 4 hours, the magnetite nanoparticles have tetrahedral shape with the average d spacing 1.69 nm with the good homogeneity

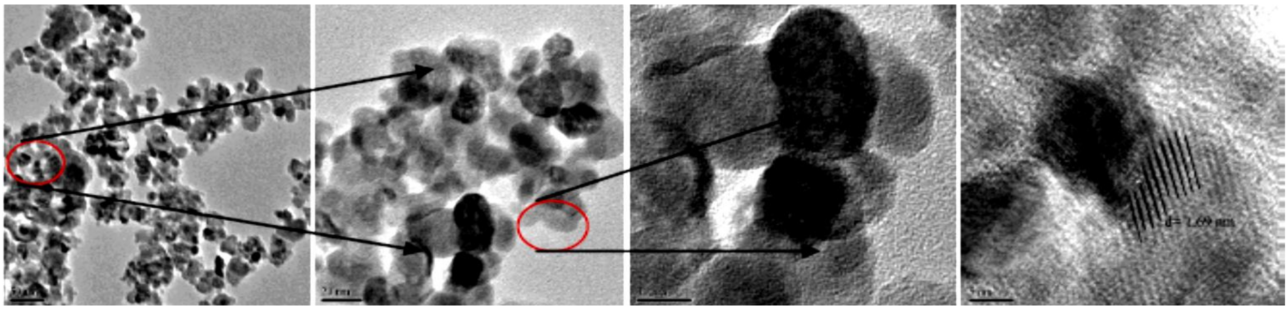

Fig. 4 TEM images of Fe_3O_4 at 300℃

3.1.3. SEM and EDAX image of the magnetite nanoparticles

SEM image of Fe_3O_4 nanoparticles at 300̊ C showed different shape and sizes of nanoparticles (Fig.5.a). EDAX image showed Fe and O elemental composition of magnetite nanoparticles. (Fig.5.b).

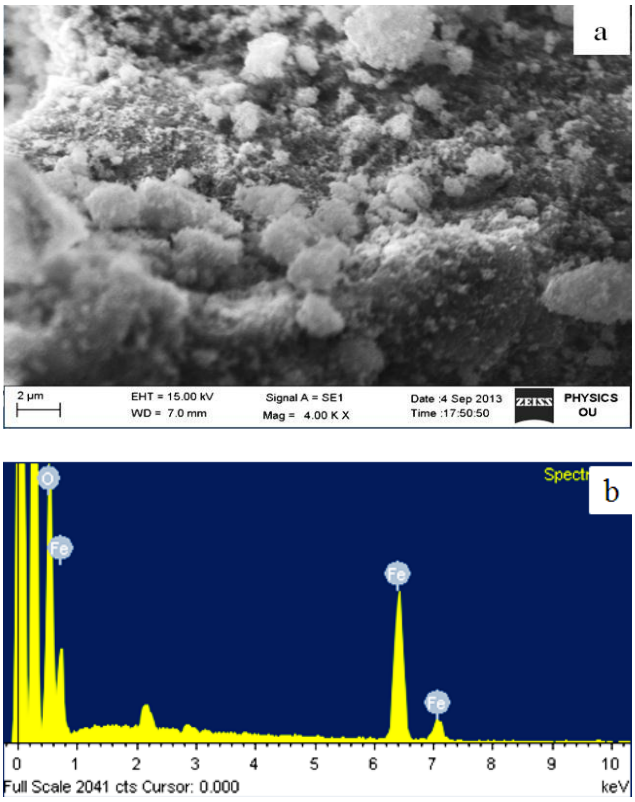

Fig. 5 SEM image of magnetite nanoparticles (a) and EDAX image of magnetite nanoparticles (b)

3.1.4. Zeta Potential of magnetite nanoparticles and hexavalent chromium

Zeta potential result of magnetite nanoparticles and hexavalent chromium showed that Fe_3O_4 nano particles is negatively charged and hexavalent chromium is positively charge. Charge of magnetite nanoparticle is -17.3 mV which helps Cr(VI) removal from wastewater, where the Cr(VI) charge is 0.1 mV which is shown in (Fig.6).

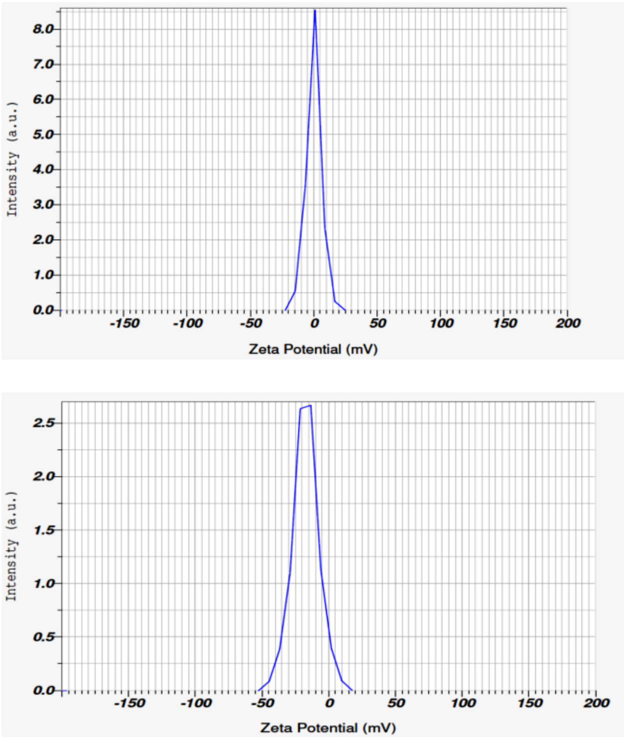

Fig. 6 Zeta potential of nanoparticles (a) and chromium hexavalent (b).

3.2. UV spectroscopy of hexavalent chromium

(Fig.7) shows UV spectroscopy pattern of initial concentrations (20 ml and 40 ml) of Cr(VI) at pH 8.2 and amount of chromium treated with magnetite nanoparticles.

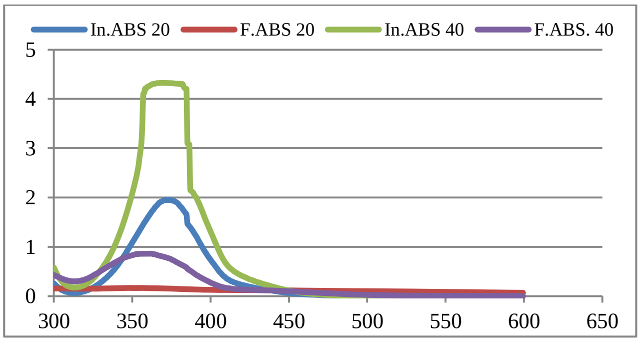

Fig. 7 Spectrum graph of hexavalent chromium at pH 8.2

4. Conclusion

In summary, Fe_3O_4 nanoparticles have been prepared by sol- gel method with annealing temperature 300℃. Result revealed synthetic process is cost efficiency and eco-friendly.

Magnetite nanoparticles have been synthesized by sol-gel method demonstrated high surface area to volume ratio and high Cr(VI) removal from wastewater in pH 8.2 and concentration 20.

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