Magnetite nanoparticles (Fe_3O_4) synthesis for removal of Chromium (VI) from waste water

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ABSTRACT

In this study adsorption behavior of Cr(VI) investigated by high adsorption iron oxide (Fe_3O_4) nanoparticle under experimental conditions of the initial concentration and pH of chromium hexavalent by Fe_3O_4 nanoparticles from waste water. Magnetite nanoparticles synthesized by sol-gel method and characterized by UV- Visible spectroscopy, XRD, EDAX, SEM and TEM. The adsorption efficiency was explained in terms of Freundlich equation. In this study we conclude that magnetite (Fe_3O_4) have potential in removal of chromium hexavalent from waste water.

Keywords: Adsorption, Hexavalent chromium, Magnetite nanoparticle, Sol-gel, Wastewater.

I. INTRODUCTION

Water pollution is becoming a most important problem that endangering and effect on all living being and becoming acute every day. The effect of water pollution has effects on gentle balance of nature. Heavy metals like mercury, chromium, nickel, lead, cadmium and copper present in the contaminated water, they present at low concentration in well balance water. However these are present at high concentration as terms of contaminated water. Among these heavy metals chromium hexavalent is considered to be of priority. Cr(VI) occur in the effluents produce during the leather tanning, dye fertilizer, electroplating, textile manufacturing, rings water, steel production factories, etc. The chromium hexavalent levels discharged may be ranging from tenth to hundreds of mg/l. are most important anthropogenic source of chromium (VI) [1,2,3].

A number of technologies for treatment of chromium compounds from wastewaters have been reported. Commonly used methods include ion exchange, evaporation, chemical precipitation, electrochemical reduction but some drawbacks like high energy requirement, large amount of sludge production and maintenance cost. Adsorption techniques are one of the great concerns to environment sciences and also adsorption processes are one of the most important methods for metal removal. In this study magnetic nanoparticles used for removal Cr(VI) from wastewaters. Magnetite nanoparticles (Fe_3O_4) have been widely studied because it has good characteristic for instance biocompatibility, super paramagnetic properties, non-toxicity, high chemical stability, easy synthesis process. Magnetite (Fe_3O_4) nanoparticles have attracted much attention in the field of magnetic recording media medical applications, including radiofrequency hyperthermia, magnetic resonance imaging (MRI), medical diagnostics and cancer therapy and microwave devices, and waste water treatment[4,5,6,7,8].There are various chemistry- based methods to synthesize nano scaled magnetite (Fe_3O_4) particles such as co-precipitation or precipitation, solution combustion, emulsion technique, hydrothermal preparation and sol-gel[8, 9].

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

II. MATERIALS AND METHODS

A. Materials and Methods

The chemicals used in this experimental work were higher grades and used without further treatment. The chemicals used were as follows potassium chromate salt (K_2CrO_4), Ferric nitrate ($Fe(NO_3)_3$ _ $9H_2O$) and ethylene glycol ($C_2H_6O_2$) of analytical grade. The reagents were used without further treatment.

B. Cr (VI) Stock solution

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.

C. Preparation of Magnetite nanoparticles

The procedure of magnetite nanoparticles is showed in the0.04 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 200-400℃.

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

D. Batch Tests

Batch tests adsorption studies were carried out by mixing0.01 mg magnetite nanopaticles with 20 mL K_2CrO_4 solution in different concentration of 20, 40, 60 and 80 mg/L at pH 8. All the adsorption studies carried out at room temperature and using shaker for 20 min with 250 rpm. The magnetite nanoparticles separated by external magnetic field. The concentration of hexavalent chromium was measured by double beam UV-Visible spectroscopy.

E. 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, JEOL JEM-2100) 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 [11]. Zeta potentials of magnetite nanoparticles were measured with particle size analyzer (Horiba SZ- 100).

III. RESULTS AND DISCUSSION

A. XRD patterns of the magnetite nanoparticles

The XRD patterns of magnetite nanoparticles obtained at various temperatures Fig. 1. The crystallite size calculated from FWHM (full width and half maximum) by using Scherrer formula, Eq. (1) was found. The diffraction peaks at 2θ= 30.12°, 35.48°, 43.12°, 62.62° and 65.85° can be assigned to (2 2 0), (3 1 1), (400), (4 4 0) and (5 3 1) planes of Fe_3O_4(PCPDF#750033). The average crystallite size of magnetite nanoparticles at 400° C is7.9 nm. 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.

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

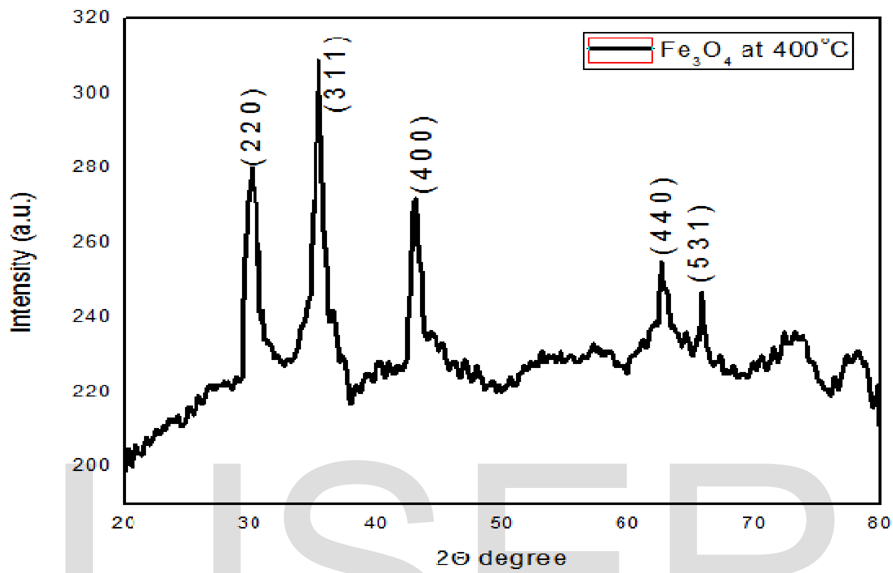

Fig.1. X-ray diffraction of magnetite nanoparticles at 400℃

B. SEM and EDAX image of the magnetite nanoparticles

SEM image of Fe_3O_4 sample showed different shape and sizes of nanoparticles (Fig.2.a).
EDAX image showed Fe and O elemental composition of magnetite nanoparticles.(Fig.2.b).
Results from table 1 confirm the percentage of existence elements in Fe_3O_4.

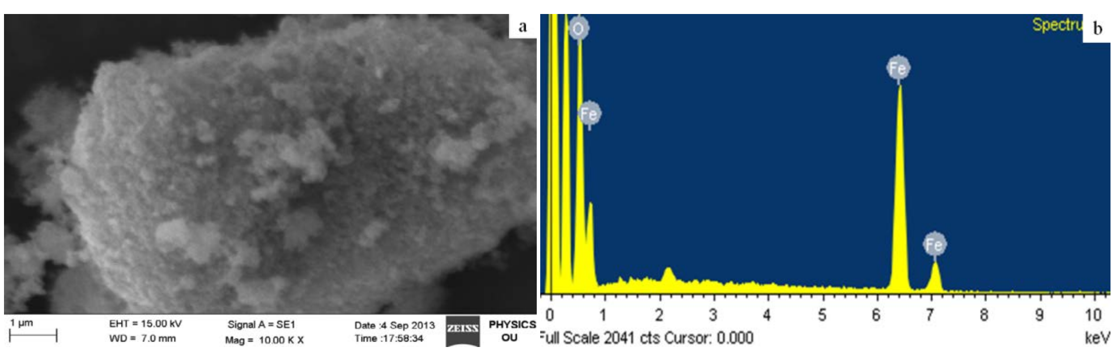

Fig.2. SEM image of magnetite nanoparticles (a) and EDAX image of magnetite nanoparticles(b).

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TABLE I

Percentage of elements in magnetite nanoparticles at 400℃

Element	Weight%	Atomic%
O K	28.42	58.09
Fe K	71.58	41.91
Totals	100.00	

C. TEM images of Fe_3O_4 nanoparticles annealed at 400℃

The Fig.3.shows presents TEM images of Fe-nanoparticles annealed at 400℃. the images shown, agglomeration of iron- nanoparticles the magnetite nanoparticles have tetrahedral and spherical shape with the average d-spacing 2.3 nm with the good homogeneity.

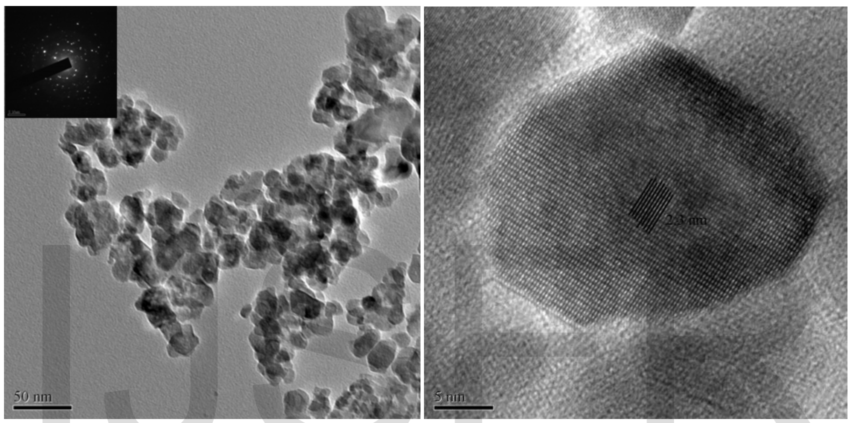

Fig.3. TEM images of magnetite nanoparticles

D. UV spectroscopy of hexavalent chromium

Fig.4. Shows UV spectroscopy pattern of Cr(VI) before and after treatment with magnetite nanoparticle at pH 8. From spectrum graph can investigated removal of chromium hexavalent chromium from waste water.

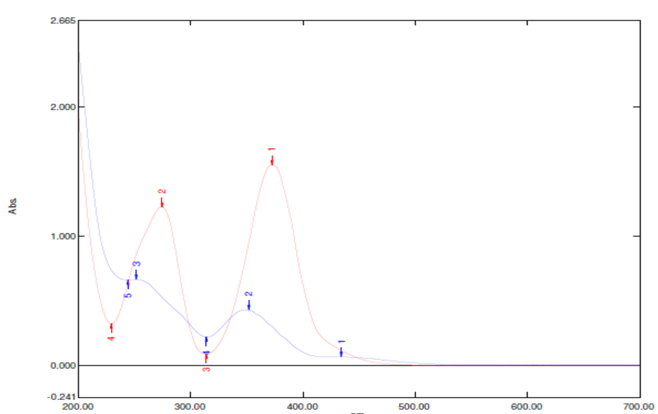

Fig.4. Spectrum graph of hexavalent chromium at pH 8.

E. Adsorption equation

The amount of hexavalent chromium adsorption was calculated from Freundlich adsorption isotherm, Eq. (2).

$$q_e = KC_e^{1/n} \qquad (2)$$

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 8by varying the initial concentration of Cr(VI) from 20 to 80 mg/L. Fig 5 shows the adsorption of hexavalent chromium by Fe_3O_4 nanoparticles.

The K and n for different concentrations were calculated using Eq. (3) for given data of initial concentration. The finding 1/n= 0.79 (1/n<1.0) shows that the adsorption of Cr(VI) by Fe- nanoparticle is favorable.

$Log\ q_e = logK + 1/n\ logC_e$(3)

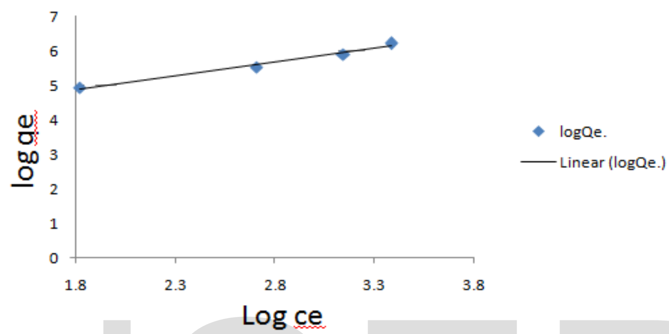

Fig.5. Freundlich isotherms of Cr(VI) onto Fe_3O_4

IV. **CONCLISION**

Fe_3O_4 nanoparticles have been prepared by sol- gel method with annealing temperatures at 400℃.

The Iron oxide nanoparticles were applied for removal of Cr(VI) from wastewater shown high efficiency. Magnetite nanoparticles demonstrated high surface area to volume ratio that increased removal efficiency of chromium. The contaminants and Fe_3O_4 can be removed from wastewater by a simple magnetic field. However should effort to better the adsorption capacity of Iron- nanoparticles and apply this method to the removal of heavy metals in large-scale.

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