Arabian Journal of Chemistry (2011) 4, 235–237

King Saud University

Arabian Journal of Chemistry

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ORIGINAL ARTICLE

New route for preparation and characterization of magnetite nanoparticles

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Received 29 June 2010; accepted 29 June 2010
Available online 3 July 2010

KEYWORDS

Magnetite;
Nanoparticles;
Co-preparation;
Characterization;
Iron(II) chloride;
Iron(III) chloride

Abstract We report here the synthesis of naked magnetic nanoparticles by using a facile method. Magnetic nanoparticles were prepared by mixing and stirring two equivalents of iron(II) chloride tetrahydrate with three equivalents of iron(III) chloride hexahydrate at room temperature. The mixture was treated by adding 100 ml of 28% ammonium hydroxide. Immediately, the color of the solution turned from orange to black. Magnetite nanoparticles precipitated and were washed three times with 5% NH_4OH solution using the magnetic decantation method.

$$2Fe(II) + 3Fe(III) \xrightarrow[NH_4OH]{pH=9} Fe_3O_4 \downarrow$$

The nanoparticles have been underwent full characterization. Their surface show a bunch of hydroxyl groups which they can be used for further complexion and removal of many hazardous compound.

1. Introduction

Nanoscale magnetite particles have drawn increasing interests in studying their application in environmental studies. There are different methods in the literature used for their synthesis with desired size, structure and other surface properties are described (Cornell and Schwertmann, 1996; Thuman, 1985; Trauth and Xanthopoulos, 1997; Solomons and Fryhle, 2004; Potter and Simmons, 1998; Readman et al., 2002; Anderson et al., 1980). These properties directly influence their chemical behavior of these nanoparticles and hence affect their application in different environmental application (Squillace et al., 1999; LaGrega et al., 2001; Rittman, 1987).

Nanoscale magnetite particles being a nanoscale material with typical size range of 1–100 nm. Recent articles in the literature have shown that many of these particles properties depend on their size which is in nanoscale size. Beside it shows that coercive force in magnetic material can be changed with the enhancement of their mechanical strength. It is also affect their surface chemistry (Rittman, 1987; Lagwaldt and Puhakka, 2000; Grady, 1986).

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Peer review under responsibility of King Saud University.
doi:10.1016/j.arabjc.2010.06.061

Production and hosting by Elsevier

There are vast environmental application of this nano-based material particularly in cleaning up contaminated soil and ground water. Because these particles have smaller size this nanoscale magnetic iron materials are much more reactive than the conventional iron powders. Moreover they can be suspended in slurry and pumped straight to the contaminated site. Elemental metal iron is known to be non-toxic, and when oxidized in present of organic contaminants, these organic can be broken down into simple carbon compounds that are less toxic. It also known that oxidizing iron can reduce heavy metals to an insoluble form that tends to stay locked in soil. In this paper the magnetic fluid containing Fe_3O_4 nanoparticles was prepared by the chemical co-precipitation of ferric and ferrous salts in alkaline medium using Reimer's procedure (Balba et al., 2002; Hou et al., 2003; Al-Khamis et al., 2009).

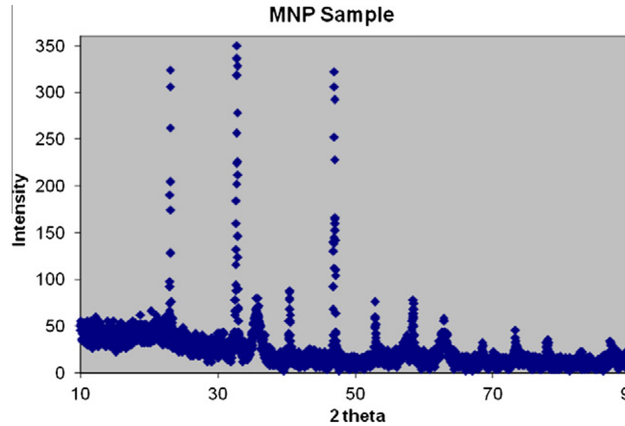

Figure 2 X-Ray Defraction.

2. Experimental

2.1. Chemicals and apparatus

All chemicals used in experiment were of analytical grade. Iron(II) chloride tetrahydrated (99%) and iron(III) chloride hexahydrated (97%), ammonium hydroxide (28%) NH_3 in double distilled water, are all from (BDH).

FT-IR spectra were recorded by prestige -21-FT-IR spectrophotometer (Shimadzo).

SEM spectra were recorded by JSM-6380LA scanning electron X-ray diffraction spectra were recorded by microscope (Jeol) Altima 4 X-ray diffraction (Rigaco).

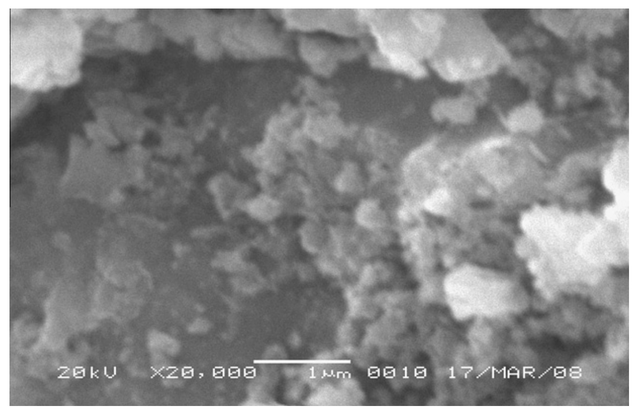

Figure 3 SEM of Fe_3O_4 nanoparticles.

2.2. Procedure

To prepare Fe_3O_4 particles add two equivalent of $FeCl_2{\cdot}4H_2O$ to three equivalent of $FeCl_3{\cdot}6H_2O$ in 500 ml beaker and add 100 ml of double distilled water and stir the solution

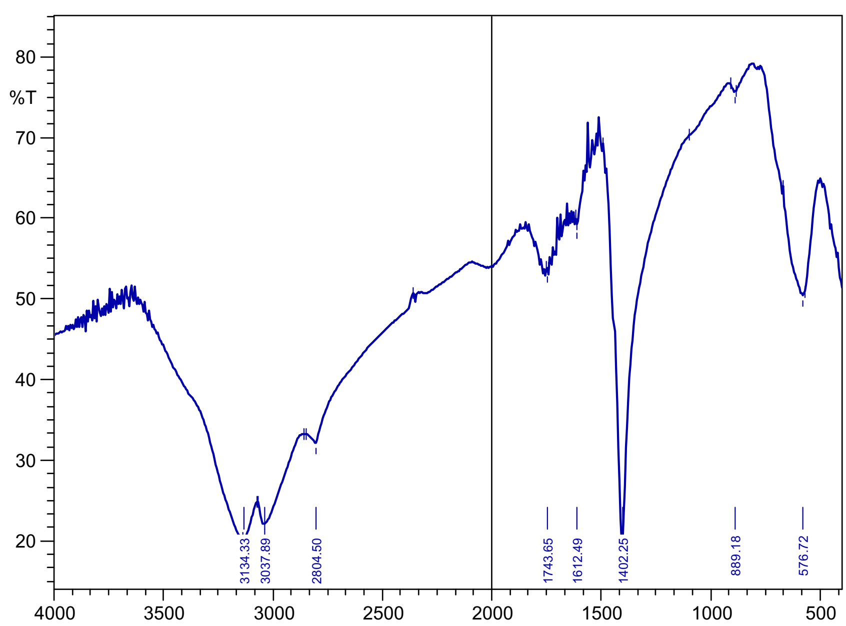

Figure 1 FTIR Spectra.

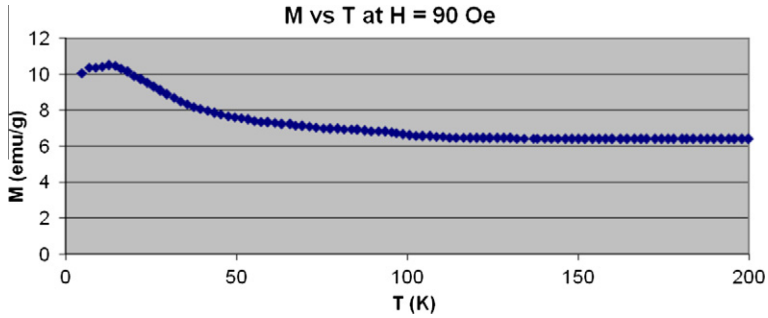

Figure 4 Magnetic properties of Fe_3O_4 nanoparticles.

continuously at room temperature till complete dissolution is achieved. Then add 200 ml of ammonium hydroxide and allow the reaction to occur for around 15 min. The resulting particles were then washed three times with 5% ammonium solution.

The precipitates were then filtered and allowed to dry in air. The dried particles were then grounded in a mortar and then were examined with FT-IR, scanning electron microscopy, X-ray diffraction and PPMS (quantum design) measurements to investigate the crystal structure of the particles and magnetization of the sample material.

3. Results and discussion

Nanoscale magnetic particles were prepared by the chemical co-precipitation of iron(III) and iron(II) chloride salt in alkaline medium using Reimer's procedure with slight modification. Spectral characterizations have proven the formation of super magnetite nanocrystals of Fe_3O_4.

In Fig. 1 the peak at 576.72 cm^{-1} is attributed to the vibration of Fe–O band of Fe_3O_4, the peak at 1402 cm^{-1} is assigned the Fe–O stretch of the Td. entity and the peak at 3134.33 cm^{-1} is attributed to the stretching vibration of –OH which is assigned to OH^- adsorbed by Fe_3O_4 nanoparticles.

Fig. 2 shows X-ray diffraction pattern of the Fe_3O_4 nanoparticles. The three main-peaks indicates that the nanoscale magnetite particles can be identified as Fe_3O_4. The X-ray spectra doses not show any other crystalline phases. The use of the X-ray also gave an indication of the crystallite size of Fe_3O_4 nanoparticles to be approximately 10 nm.

Fig. 3 illustrates the SEM micrograph of the nanoscales magnetite particles. Morphology of the particles was uniform and each particle was approximately ranging between 10 and 70 nm in diameter.

Fig. 4 presents the magnetization of Fe_3O_4 nanoparticles in a field of 90 Oe on a super conducting quantum interference device magnetometer. The absent of a well-defined maximum in the ZFC curve indicates that Fe_3O_4 nanoparticles exhibit blocking temperature above room temperature. It is known that the maximum of the ZFC curve for a collection of super paramagnetic non-interaction single-domain nanoparticles is dependent on the size of nanocrystals and their degree of clustering, as well as on the mutual dipolar interaction between them.

We conclude from that the efficiency and simplicity of the chemical co-precipitation method for the preparation of nanoscale magnetite with a super para-magnetism from the solution of iron(II)/iron(III)mixed salt-solution in aqueous ammonium hydroxide solution. The results show that Fe_3O_4 nanoparticles can be prepared in the sizes range from 10 to 20 nm. The use of these nanoscale particles in purification of water from organic contaminant is under taken and is going to be reported later. As mentioned previously Fe_3O_4 nanoparticles which were prepared by the previous method can of course be promising as potentiality good magnetic material that can have good magnetic quality.

Acknowledgement

The authors would like to thank the Research Center, College of Science for financial support (Project No. (Chem/2009/56).

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