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ORIGINAL PAPER

Synthesis and characterization of nickel ferrite nanoparticles by chemical method

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Abstract: In this paper we synthesized nickel ferrite nanoparticles by using a chemical method. Synthesized nanoparticles were characterized by using X-ray diffraction, Fourier transform infrared spectroscopy, vibrating sample magnetometer, transmission electron microscopy, and energy-dispersive X-ray spectroscopy. Magnetic properties, size, purity, stoichiometry and morphology of the samples were determined. Some samples were calcined at different temperatures. We found that the size of nanoparticles increased with the increase in calcination temperature and the nanoparticles transformed from amorphous phase to crystalline phase. When the size of nanoparticles decreased to less than a critical size (10 nm) the nanomaterial underwent a transition from a ferromagnetic to a superparamagnetic.

Keywords: Nanoparticle; Nickel ferrite; Spinel; Superparamagnetic

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1. Introduction

In recent years much attention has been paid to the nanomaterials because of their interesting properties and applications compared to their bulk counterparts [1–8].

Among these nanomaterials, the magnetic nanomaterials are more interesting owing to their medical, electronic and recording applications. These applications depend on the size, shape, purity and magnetic stability of these materials [9–13].

In biomedical applications, nanomagnetic materials can be used as carriers of drug inside the body where the conventional drugs may not work. For this purpose the nanoparticles should be in the superparamagnetic phase with a low blocking temperature [10]. These properties can be obtained by reducing the size of nanoparticles because some of the fundamental properties of nanoparticles are affected by decreasing their size [10–13].

Nickel ferrite ($NiFe_2O_4$) has an inverse spinel structure [14]. In this structure Ni^{2+} ions occupy octahedron B sites and Fe^{3+} ions occupy both the tetrahedron A and the octahedron B sites. In a spinel structure there are 56 ions, 32 oxygen ions and 24 metal ions in a unit cell. In this structure eight molecules occupy one unit cell of the spinel. A general formula for a ferrite structure is $(M_{1-x}Fe_x)[M_xFe_{2-x}]O_4$, where M stands for cations which occupy tetrahedron sites and x is the degree of inversion [14]. The spinel nanoparticles are generally synthesized by using a chemical method. Nickel ferrite is one of the most important spinel ferrites [15]. It shows a proper ferromagnetism that originates from magnetic moment of antiparallel spins [16–18].

In this paper we have used co-precipitation method for synthesizing nickel ferrite nanoparticles. This is a proper technique for synthesizing monodisperse nanoparticles with small sizes, which are very important for applications.

2. Experimental details

Nickel ferrite nanoparticles ($NiFe_2O_4$) were prepared by using co-precipitation method, using analytical grade chemicals $FeCl_3{\cdot}6H_2O$ and $NiCl_2{\cdot}6H_2O$ (Merck Company) as starting materials for iron and nickel sources. Oleic acid was used as capping agent. The salts were dissolved in double distilled water separately. 0.2 M and 0.4 M solutions of nickel and iron chloride were prepared separately and then the solutions were added to each other. Sodium hydroxide solution (3 M) was added to the mixture drop by

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drop, till pH of the solution was close to 13. Finally 3 drops of oleic acid were added to the solution as a surfactant. Then the temperature of the solution was increased up to 80 °C for 40 min. The solutions were centrifuged and precipitation was washed several times with double distilled water and ethanol. The precipitation was dried in oven at 80 °C for several hours. Additional processes (calcination) were required in order to change these amorphous $NiFe_2O_4$ nanoparticles to crystalline phase of $NiFe_2O_4$.

X-ray diffraction patterns of the as synthesized sample and the samples calcined at different temperatures were taken in a diffractometer (Philips Xpert model). Source of X-ray was $Cu_{k\alpha}$ with a wavelength of 1.54 Å. The step of scanning was 0.02 degree with the speed of one step per second.

FT-IR transmission spectra were taken on JASCO 640 plus infrared spectrometer in the range of 4000–400 cm^{-1}. Samples were prepared by mixing samples powder with KBr, which were ground and pressed into a transparent pellet with a diameter of 1 cm.

Magnetization measurement was performed at room temperature using a vibrating sample magnetometer (VSM) device, in the Development Center of University of Kashan (Kashan, Iran).

The transmission electron microscopy (TEM) was taken in the JEOL JEM-2100 FTEM model which operates in voltage range 160–200 kV.

The EDX pattern was taken using CAMSCANMV 2300 and the working voltage was 15 kV.

3. Results and discussions

Figure 1 shows XRD patterns of seven samples calcined at temperatures between 500 and 1000 °C. It is apparent that the width of peaks decreases with increasing calcination temperature. This indicates that particles size increases with the increase of calcination temperature. By using Scherer's formula we have obtained 7 and 82 nm for the grain sizes of the nanoparticle samples calcined at 500 and 1000 °C respectively.

The critical grain size of $NiFe_2O_4$ nanoparticles for transition from ferromagnetic phase to superparamagnetic phase is 10 nm [19]. By using Scherer's formula we found that the grain sizes of the nanoparticle samples calcined at 400 and 500 °C were less than 10 nm.

TEM image of the nanoparticles calcined at 600 °C show that the nanoparticles size is 23 nm which is in good agreement with value obtained by using Scherer's formula.

Figure 2 shows the FT-IR spectrums the sample calcined at 600 °C. Two peaks at 3448.10 and 1638.23 cm^{-1} on the FT-IR spectrum are related to OH bond as reported in literature [20, 21].

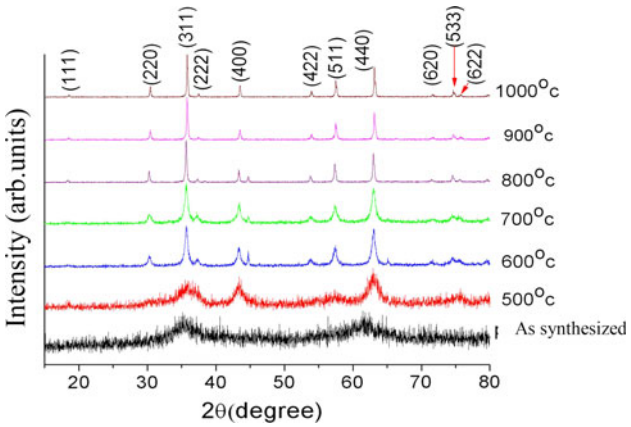

Fig. 1 XRD patterns of NiFe2O4 nanoparticles calcined at different temperatures

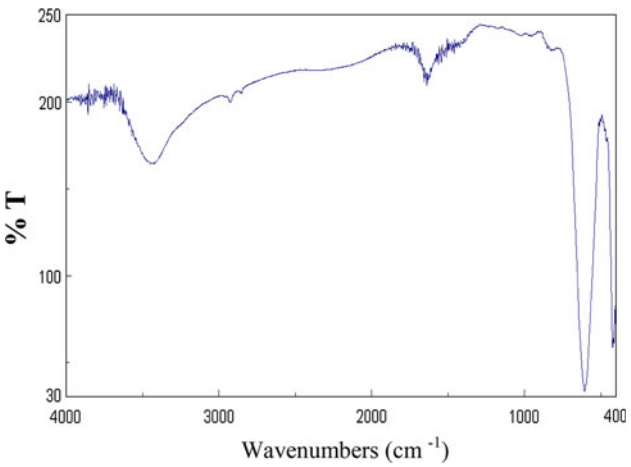

Fig. 2 FT-IR spectrum for sample calcined at 800 °C

The presence of 3752.00 till 3650.59 cm^{-1} stretching modes corresponds to CO_3^{2-} and NO^{3-} bonds, which have very low intensity. The stretching modes at 574.00 and 422.00 cm^{-1} positions show Fe–O and Ni–O stretching modes, which indicate formation of $NiFe_2O_4$ nanoparticles.

For particles with large sizes multi magnetic domains are formed and the nanoparticles behave more bulk-like with increasing size. When the particle size reduces magnetic domains change from multi domains to a single domain. Thus below a critical particle size domain walls will no longer form due to energy considerations and single domain particles will be stable. This critical size corresponds to the peak in the coercivity [10, 11]. The superparamagnetic size strongly depends on the magnetocrystalline anisotropy of the material. In ferromagnetic and ferrimagnetic materials when the size of nanoparticles decreases the particles change from multi domain to single domain and transform to superparamagnetic [10, 11].

Figure 3 shows the hysteresis loops of some samples calcined at different temperatures (400, 500, 800 and

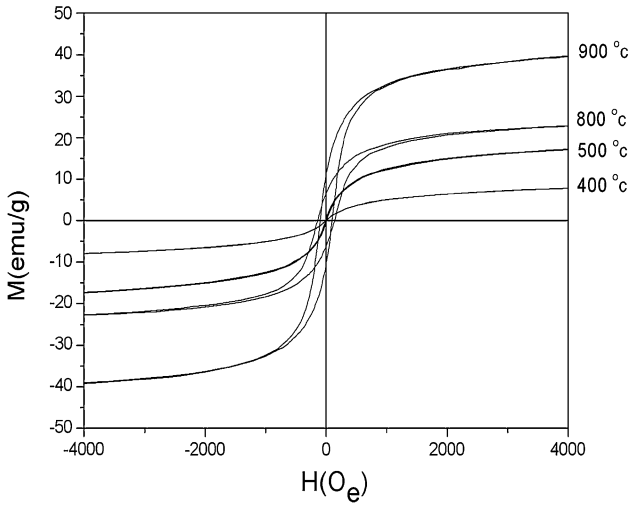

Fig. 3 Hysteresis lops for different sizes of $NiFe_2O_4$ nanoparticles

1000 °C) while other conditions for the samples were the same. The hysteresis loops show a good magnetization. Hysteresis loops corresponding to nanoparticles calcinated at 400 and 500 °C with particle sizes less than 8 nm show superparamagnetic properties which means magnetic remanence (M_r) and coercive force (H_c) are zero.

Figure 4 shows TEM image of the sample calcined at 600 °C, which shows the morphology of particles. The particles size is around 23.0 nm with monodisperesed nanoparticles as one can see on the figure. This size is in good agreement with the grain size of the nanoparticles obtained from XRD.

Fig. 4 TEM image for sample calcined at 600 °C

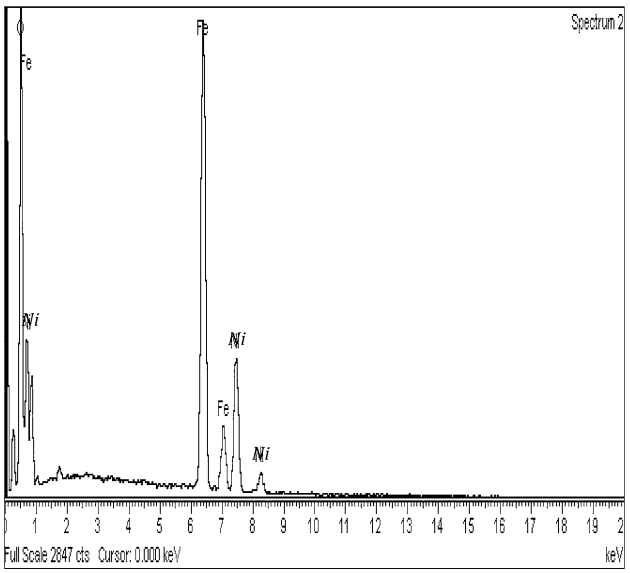

Fig. 5 EDS of sample calcined at 600 °C

Figure 5 shows the EDX of the sample calcined at 600 °C. It indicates that the sample is very pure and there is not any impurity in the sample.

4. Conclusions

In this paper pure nickel ferrite nanoparticles with size in the rage of 7–82 nm were synthesized. Results from calcinated samples showed that the size of nanoparticles increased with the increase of calcination temperature. Crystallinity of the samples also increased with the increase of calcination temperature. Grain size of the nanoparticles obtained by using Scherer's formula, and the grain size of the nanoparticles obtained from TEM image were in good agreement.

The VSM graphs showed a good magnetization for the synthesized $NiFe_2O_4$ nanoparticle. In addition, the samples which were heated at 400 and 500 °C showed superparamagnetic property. FT-IR spectrum of $NiFe_2O_4$ nanoparticle showed that the nanoparticles were synthesized properly.

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