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Full Length Research Paper

Synthesis of nickel ferrite nanoparticles by co-precipitation chemical method

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In this research work, we have prepared nickel ferrite nanoparticles by using chemical route. Nanoparticle materials are characterized using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), vibrating sample magnetometer (VSM), transmission electron microscopes (TEM) and energy dispersive X-ray (EDX) systems. We have determined magnetic properties, size, purity, stoichiometry and morphology of samples. The samples are calcinated at different temperatures, then we found that size of particles increase with heating and powder transfer from amorphous to crystalline phase of nickel ferrite. When the size of nanoparticles decreased to less than a critical grain size (10 nm), the nanomaterials transfer from ferromagnetic to super paramagnetic materials.

Key words: Nanoparticle, nickel ferrite spinel, superparamagnetic.

INTRODUCTION

In the recent years, so much attention has been paid to the nanomagnetic materials that show very interesting magnetic properties. In this material, different properties and applications are appeared as compared to their bulk counterparts. The magnetic properties of nanomaterials are used in medical, electronic, and recording industries that depend on the size, shape, purity and magnetic stability of these materials (Maaz et al., 2009; Sellmyer and Skomski, 2006; Cullity and Graham, 2009).

In biomedical application, one can use nanomagnetic materials as drug carriers inside body where the conventional drug may not work. For this purpose, the nanosize particles should be in the superparamagnetic form with a low blocking temperature (Sellmyer et al., 2006). Ferrite nanomaterials are object of intense research because of their proper magnetic properties. It has been reported that when the size of particles reduced to small size or in range of nanomaterials, some of their fundamental properties are affected (Sellmyer and Skomski, 2006; Cullity and Graham, 2009; Billas et al., 1994). Nickel ferrite $NiFe_2O_4$ is a cubic structure and has an inverse spinel structure. At this structure, Ni^{2+} ions occupy octahedron B site and Fe^{3+} ions occupy both tetrahedron A and octahedron B-sites. The spinel nano-particles generally are prepared by using chemical route which is a proper method. Nickel ferrite is one of the most important spinel ferrites. It shows a proper ferromagnetism that originates from magnetic moment of anti-parallel spins (Martinez et al., 1998; Misra et al., 2004; Nathani et al., 2005).

In a spinel structure, there are 56 ions, 32 oxygen and 24 metal ions in a unit cell. At this structure eight molecules occupy a unit cell of spinel that they are 32 inions and 24 cations. A general formula of ferrite structure is shown as $(M_{1-x}Fe_x)[M_xFe_{2-x}]\ O_4$, in which M shows cations that occupy tetrahedron sites and x is degree of inversion (Abdullah et al., 2008).

In this research, work we have used co-precipitation method for making nickel ferrite nanoparticles. It is a proper technique for making small size and

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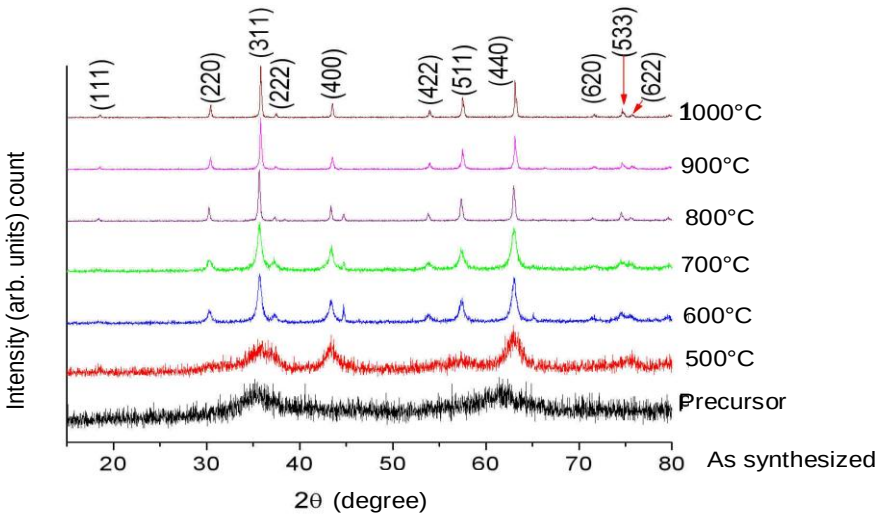

Figure 1. XRD Patterns of $NiFe_2O_4$ nanoparticles for different temperatures.

mono-dispirsity nanoparticles. Those are characterizing which are very important in application.

SYNTHESIS PROCEEDING

Nickel ferrite nanoparticles ($NiFe_2O_4$) has been prepared by using co-precipitation method. Chloride salts ($FeCl_3$ and $NiCl_2$) was used as starting materials for iron and nickel sources, respectively. All chemicals were analytical grade from Merck Company. The oleic acid also is used as capping agent. Each of salts dissolved in double distilled water separately. We have used 0.2 and 0.4 M solutions from nickel and iron chloride, respectively. Then the previous solution was added to each other.

Sodium hydroxide solution (3 M) was added to mixture solution drop wise till PH received close to 13. Finally, 3 drop of oleic acid is added as a surfactant to the previous solution. Then the temperature is increased up to 80°C for 40 min. We have centrifuged and washed precipitation with double distilled water and ethanol several times. The precipitation was dried in oven at 80°C for several hours. Now we have got amorphous $NiFe_2O_4$ nanoparticles and also additional process is used for getting crystalline powder of nickel ferrite nanoparticles.

RESULTS AND DISCUSSION

X-ray diffraction (XRD) analysis

We have taken XRD patterns from as synthesized and heated samples at different temperatures. The XRD system which we have used is Xpert Philips model, made in Holand. Source of X-ray was Cu $_{k\alpha}$ with wavelength 1.54 A°. The step of scanning is 0.02° with speed of a step per second.

Figure 1 shows XRD patterns of seven samples that the calcinations temperatures are between 500 to 1000°C. It is found that width of peaks decreasing when calcinations temperature increased. This indicates that particle size increasing when temperature increasing. The grain size of particle for sample that calcinated at 500 and 1000°C obtained 7 and 82 nm by using Sherer's formula, respectively. All crystalline size calculation have been obtained using "Xpert HighScore plus" software.

The critical grain size of $NiFe_2O_4$ is 10 nm for transition from ferromagnetism to superparamagnetic materials (John and Abdul, 2010). We have found that two samples are (heated at 400 and 500°C) obtained grain size less than 10 nm by calculations of Scherer's formula. One can see very nice agreement in size of particles. Size of particles for 600°C calcination temperature is obtained around 23 nm from shere's formula and Transmission electron microscopes (TEM) image.

Fourier transform infrared spectroscopy (FT-IR) analysis

Two peaks were shown at 3448.10 and 1638.23 cm^{-1} in spectrum (Figure 5) related to O-H as reported at literature (Santi et al., 2007; de Paiva et al., 2009). Presence of 3752.00 to 3650.59 stretching modes corresponding to CO_3^{2-} and No_3^- bonds in which have very low intensity. The stretching modes at position of 574.00 and 422 cm^{-1} are showing Fe-O and Ni-O stretching modes, which indicate formation of $NiFe_2O_4$ nanoparticles (Figure 2).

Vibrating sample magnetometer (VSM) analysis

For particles with large sizes multi-domain are there and becoming more bulk-like with increasing size. When particle size reduces, magnetic domains from multi transfer to a single domain. Thus, below a critical particle size domain walls will no longer form due to energy considerations and single domain particles are stable.

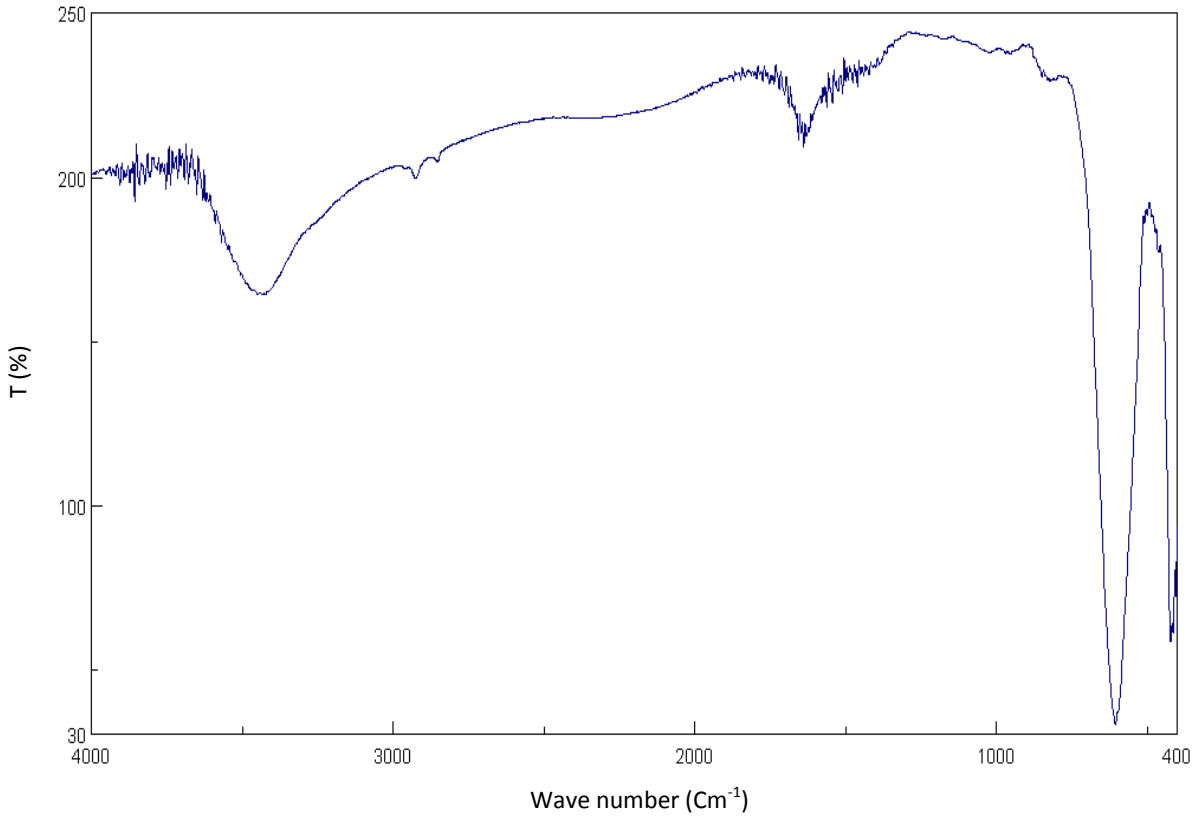

Figure 2. FT-IR spectrum for sample that heated at 800°C.

This critical size corresponds to the peak in the coercivity. The particles are then superparamagnetic. The superparamagnetic size strongly depends on the magnetocrystalline anisotropy of the material. In ferromagnetic and ferrimagnetic materials when size of particles decreeing, the particle transfer from multi domain to single domain and transfer to superparamagnetic (Sellmyer and Skomski, 2006; Cullity and Graham, 2009).

Some of samples are calcined at different temperatures (400, 500, 800 and 1000°C), conditions for all the samples were same except calcinations temperatures. The hysteresis loops show (Figure 3) a good magnetization. Hysteresis loops according 400 and 500°C with particle size less than 8 nm that is less than critical grain size, show superparamagnetic properties that are meaning magnetic remanence (M_r) and coercive force (H_c) are zero.

Transmission electron microscopes (TEM) and energy dispersive X-ray (EDX) and scanning electron microscopy (SEM) analysis

The TEM system, which we have been used for morphology and size determination was JEOL JEM-2100 FTEM model. The SEM system that has been used for morphology of sample was CAMSCAN MV2300 model with 15 KV applied voltage.

Figure 4 shows the morphology of particles. Photograph has been taken from the samples which were calcinated at 600°C. Particle size is obtained around 23.0 nm with monodisperesed nanoparticles as one can see from the photograph. In comparison to grain size of particles from XRD results, the sizes are matching well. The voltage range which we have used was between 160 to 200 KV. The EDX pattern also is taken from the same sample (600°C). The model of this system is CAMSCANMV 2300 and 15 KV was applied. Figure 5 shows that sample is very pure and there is no impurity in the sample.

Conclusions

In this research work, pure nickel ferrite nanoparticles in the ranges of 7 to 82 nm is obtained, calcinations samples show that size of particles increase when calcinations temperature increase. Crystallinity of samples also increases with high temperature calcinations. Calculation of size from Sherer's formula and TEM image show a good agreement.

The VSM graphs show a good magnetization for $NiFe_2O_4$ nanoparticle and also the samples which heated

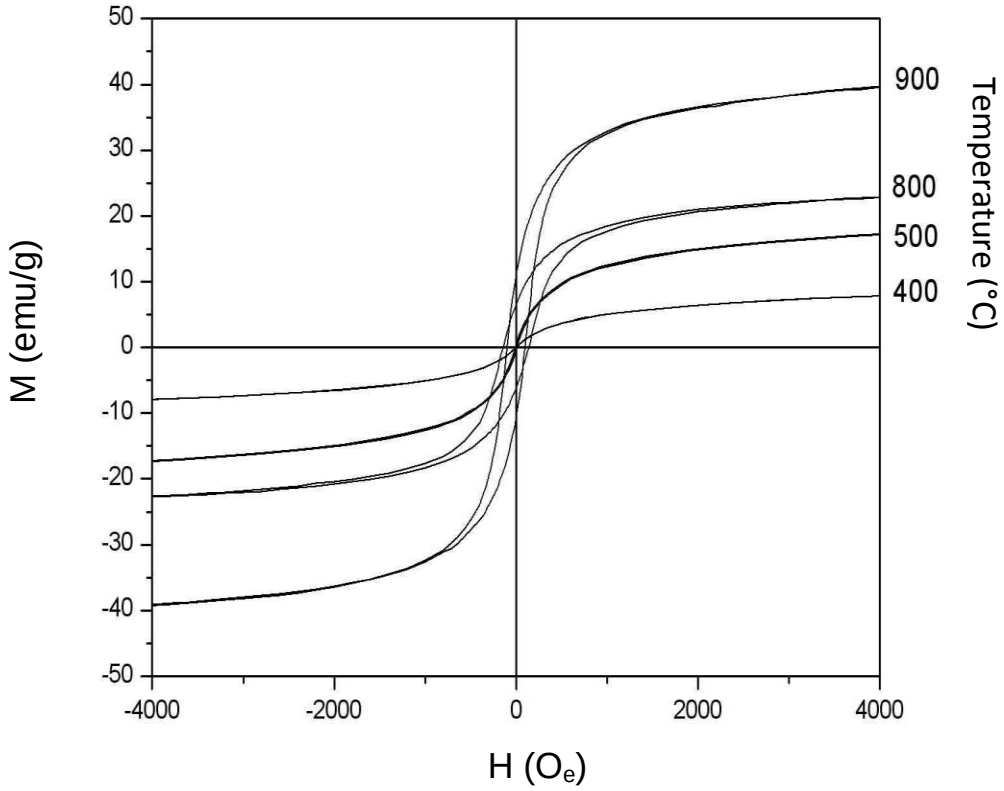

Figure 3. Hysteresis lops for different sizes of $NiFe_2O_4$ nanoparticles.

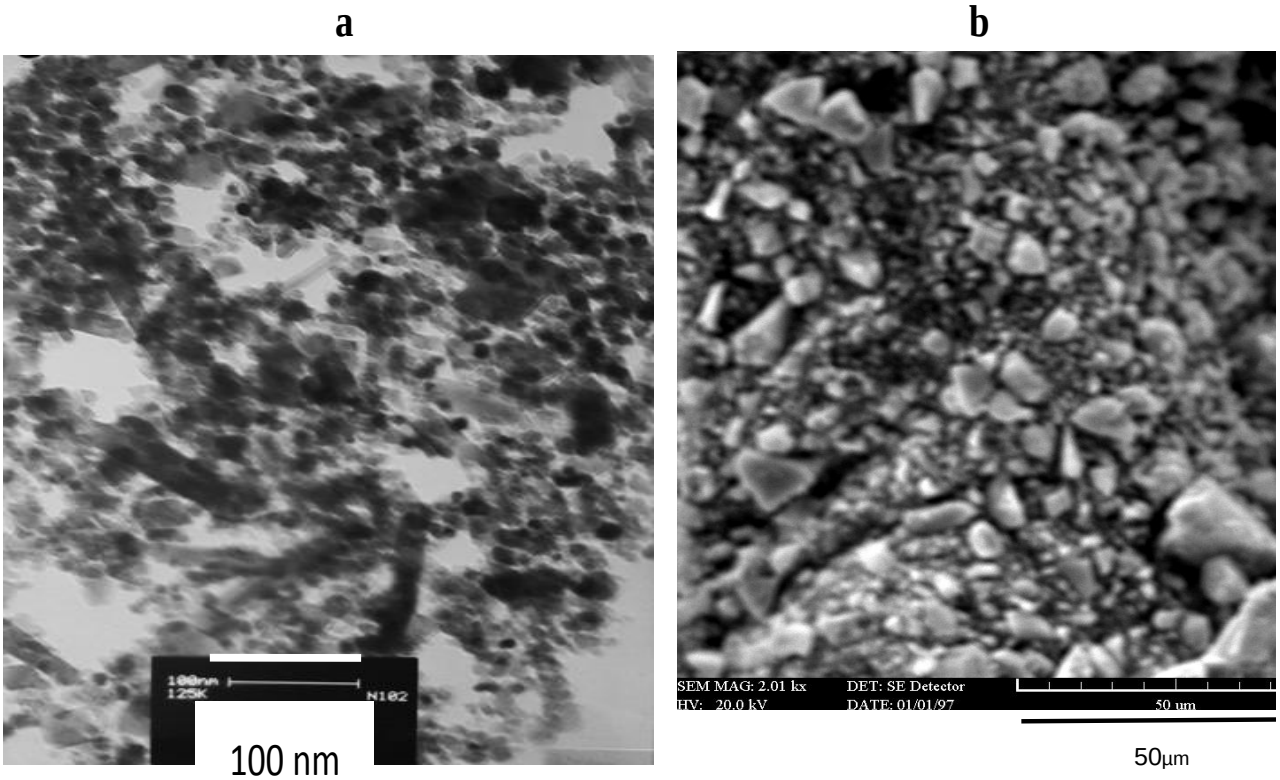

Figure 4. a. TEM image for sample which heated at 600°C. b. SEM image of $NiFe_2O_4$ heated at 600°C.

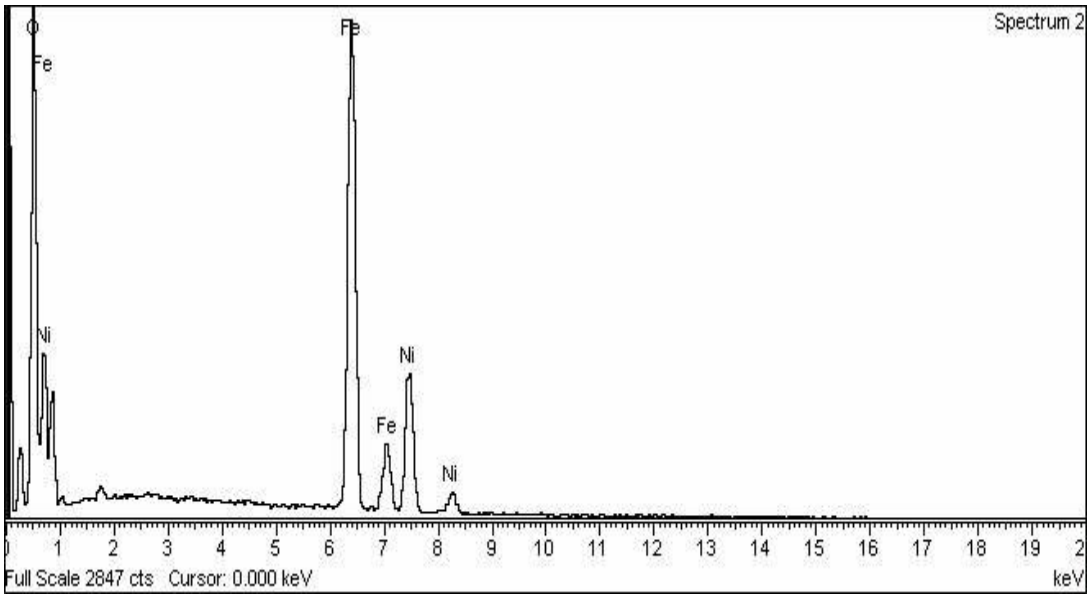

Figure 5. EDS of sample which heated at 600°C.

at 400 and 500°C show a superparamagnetic property. FT-IR spectrum also shows that $NiFe_2O_4$ nanoparticle has been prepared properly.

ACKNOWLEDGEMENT

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