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 NiFe₂O₄ is a cubic structure and has an inverse spinel structure. At this structure, Ni²⁺ ions occupy octahedron B site and Fe³⁺ ions occupy both tetrahedron A and octahedron B-sites. The spinel nanoparticles 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 (MₓFe₁₋ₓ)[MₓFe₂₋ₓ] O₄, 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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RESULTS AND DISCUSSION

SYNTHESIS PROCEEDING

Nickel ferrite nanoparticles (NiFe$_2$O$_4$) 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 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$_2$O$_4$ nanoparticles and also additional process is used for getting crystalline powder of nickel ferrite nanoparticles.

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$_2$O$_4$ 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.
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 decreing, 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.

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

**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\(_2\)O\(_4\) nanoparticle and also the samples which heated...
Figure 3. Hysteresis lops for different sizes of NiFe₂O₄ nanoparticles.

Figure 4. a. TEM image for sample which heated at 600°C. b. SEM image of NiFe₂O₄ heated at 600°C.
at 400 and 500°C show a superparamagnetic property. FT-IR spectrum also shows that NiFe₂O₄ nanoparticle has been prepared properly.

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REFERENCES

Abdullah CB, Sadan Ozcanb, Nic C, Ismat Shah S (2008). Solid state reaction synthesis of NiFe₂O₄ nanoparticles. J. Magn. Magn. Mater. 320:857-863.

Billas IML, Chatelain A, de Heer WA (1994). Magnetism from the Atom to the Bulk in Iron, Cobalt and Nickel Clusters. Science, 265: 1682.

Cullity BD, Graham CD (2008). Introduction to Magnetic Materials, Second Edition, John Wiley & Sons, Inc., Hoboken, NJ, USA.

de Paiva JAC, Grac MPF, Monteiro J, Macedo MA, Valente MA (2009). Spectroscopy studies of NiFe₂O₄ nanosized powders obtained using coconut water. J. Alloys Compd. 485:637-641.

John J, Abdul KM (2010). Investigation of mixed spinel structure of nanostructured nickel ferrite. J. Appl. Phys. 107:114310

Maaz K, Karim K, Muntaz A, Hasanain SK, Liu J, Duan JL (2009). Synthesis and magnetic characterization of nickel ferrite nanoparticles prepared by co-precipitation route. J. Magn. Magn. Mater. 321:1838-1842.

Martinez B, Obradors X, Balcells LI, Rouanet A, Monty C (1998). Low Temperature Surface Spin-Glass Transition in γ-Fe₂O₃ Nanoparticles. Phys. Rev. Lett. 80:181.

Misra RDK, Gubbala S, Kale A, Egelhoff Jr. WFA (2004). comparison of the magnetic characteristics of nanocrystalline nickel, zinc, and manganese ferrites synthesized by reverse micelle technique. Mater. Sci. Eng. B 111:164-174.

Nathani H, Gubbala S, Misra RDK (2005). Soft magnetic material (NiFe₂O₄) particles synthesized by solvent co-precipitation method. Sci. Eng. B 121:126.

Santi M, Chivalrat M, Banjong B, Supapan S (2007). A simple route to synthesize nickel ferrite (NiFe₂O₄) nanoparticles using egg White. Scripta Mater. 56:797-800.

Sellmyer DJ, Skomski R (2006). Introduction to Advanced Magnetic Nanostructures" (2006). Faculty Publications: Materials Research Science and Engineering Center. Paper 28.