

Synthesis of nickel ferrite nanoparticles via chemical co-precipitation method

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Abstract

In this article, an effort is made to synthesize the nickel ferrite nanoparticles via chemical co-precipitation method using the metal nitrates as precursors. The x-ray diffraction pattern of the as-synthesized powder sample indicates the formation of nickel ferrite with iron oxide and other impurity phases. The heat treatment of the as-synthesized powder at 600°C assists in the formation of inverse spinel phase of nickel ferrite accompanying with some fraction of iron oxide phase. The field emission scanning electron microscopy of the heat treated sample reveals the irregular particle size and shape with fine microstructures, while as-synthesized sample shows the lamina like particle morphology. The magnetization curve (M-H curve) of the heat treated sample shows the ferrimagnetic behavior with very small (negligible) values of remanent magnetization and coercive field. Such type of magnetic characteristic indicates the signature of superparamagnetism ($M_r \sim 0$, $H_c \sim 0$) in the heat treated nickel ferrite nanoparticles with significant value of the saturation magnetization (M_s). The superparamagnetic effect in the nickel ferrite particles has great potential in the field of biomedicine for certain applications such as the effective drug delivery, and for enhancing the contrast in case of magnetic resonance imaging. Copyright © 2016 VBRI Press.

Keywords: Co-precipitation, ferrite, ferrimagnetism, superparamagnetism, nanoparticle.

Introduction

The nanometer sized particles have attracted considerable attention in recent years because of their excellent magnetic, optical, chemical and electrical properties, and due to their usage for various applications [1, 4]. The magnetic properties of the nano sized samples depend on the size, shape, and purity of the corresponding materials [5]. The medical, electronic and recording industries have strong requirements of magnetic nano sized materials due to their outstanding physicochemical and magnetic properties [6, 7]. The ferrite nanoparticles have also recently been used in biomedical application for cancer treatment by hyperthermia [8]. The shape, size, surface chemistry and magnetic properties of ferrite nanoparticles depend on the preparation method and the starting materials used. The room temperature magnetic properties offer pure nickel as a material of biological interest. However, the high reactivity and toxicity restrict its application for biomedical purposes [7]. Therefore, the less toxic and chemically stable nickel ferrite nanoparticle appears to be viable choice for the replacement of pure nickel in biomedical application.

In the present work, the nickel ferrite nanoparticles are synthesized via chemical co-precipitation method using the metal nitrate precursors. The chemical co-

precipitation method is the easy way to synthesize nanoparticles of various sizes offering lots of flexibility.

The survey of related research papers shows that the preparation of nickel ferrite nanoparticles via co-precipitation method using the metal nitrate precursors has not yet been reported in the past. The structural properties and the confirmation of cubic inverse spinel phase of nickel ferrite nanoparticles are studied in the present work using the X-ray diffraction (XRD). The surface morphology of the nickel ferrite nanoparticles is observed using the field emission scanning electron microscopy (FESEM). The magnetic properties of the prepared nickel ferrite nanoparticles are studied from the magnetization measurement using vibrating sample magnetometer (VSM). The objective of this work is to synthesize the nickel ferrite nanoparticles by co-precipitation method using metal nitrates as precursor and study their structural, morphological and room temperature magnetic properties.

Experimental

For the synthesis of nickel ferrite nanoparticles, the nickel nitrate hexa hydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Loba Chemie,

99%), iron nitrate hexa hydrate ($\text{Fe}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, E. Merck, 98%) and NaOH (Loba Chemie, 99%) were purchased.

The nickel ferrite nanoparticles are prepared here using the chemical co-precipitation method [9]. In this method, the stoichiometric amounts of nickel nitrate hexa hydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and iron nitrate hexa hydrate ($\text{Fe}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) are added separately in 100 ml of de-ionized water in order to prepare their solutions. The de-ionized distilled water is taken as solvent for the minimization of the impurities in the final product. The prepared solutions are then mixed together and stirred continuously at 80°C . The 2M NaOH solution has been prepared separately which acts as the precipitating agent. The pH value of 12 has been maintained throughout the reaction by the continuous addition of NaOH solution slowly. After 2 hrs, the precipitated particles are filtered and then dried in electric oven at 100°C for 24 hrs. The obtained solid is crushed into powder using mortar and pestle in order to obtain as-synthesized nickel ferrite nanoparticles. The as-synthesized powder is heat treated at 600°C for 4 hrs in an electrical furnace, which is named as heat treated nickel ferrite nanoparticles.

The structural analysis of the obtained powders is studied by X-ray diffraction technique (Rigaku MiniFlex 600 model). The surface morphology is analyzed by the field emission scanning electron microscopy (FESEM) using a model JSM-7100F; JEOL. A vibrating sample magnetometer (Princeton VSM model-150) is employed to record the room temperature M Vs H plots in the applied magnetic field range of -20K to 20K.

Results and discussion

Fig. 1 shows the x-ray diffraction patterns of nickel ferrite nanoparticles synthesized using chemical co-precipitation method. The X-ray diffraction patterns of as-synthesized and heat treated nickel ferrite nanoparticles are shown in **Fig. 1(a)** and **Fig. 1(b)**, respectively. In the XRD pattern of as-synthesized sample, the impurities of iron oxides and some unknown material are observed.

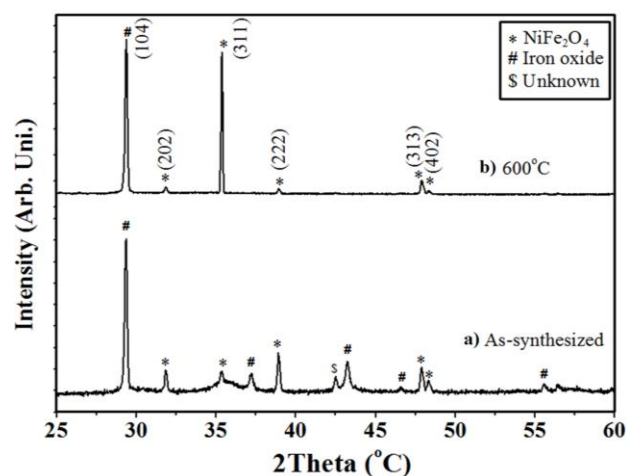


Fig. 1. X-ray diffraction patterns of synthesized NiFe_2O_4 samples, (a) As-synthesized (b) Heat treatment at 600°C .

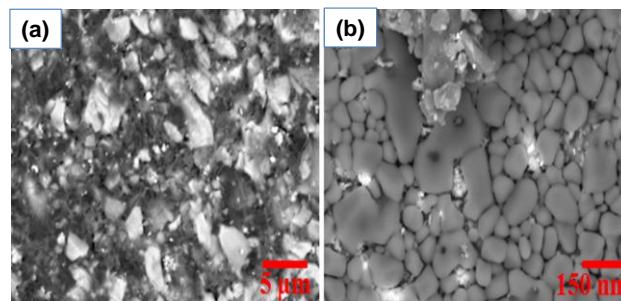


Fig. 2. Field emission scanning electron microscopy images of synthesized NiFe_2O_4 samples, (a) As-synthesized, (b) Heat treatment at 600°C .

The heat treatment of as-synthesized powder sample at 600°C for 4 hours assists to eliminate the most of the iron oxide and unknown material. Consequently, this heat treatment favors the formation of face centered cubic inverse spinel nickel ferrite nanoparticles accompanying with iron oxide phase. The XRD pattern of the heat treated nickel ferrite nanoparticles is matched with the ICSD card number: 01-074-1913. The average particle size of the nanocrystalline sample is calculated as 69 nm using well known Debye-Scherrer formula [10]. The field emission scanning electron microscopy images of synthesized NiFe_2O_4 samples are depicted in **Fig. 2**. The surface morphology of the as-synthesized sample is shown in **Fig. 2(a)**, which reveals the unclear, blurred and lamina like microstructures. This may be due to the nitrate residues present in the as-synthesized sample. When the sample is heat treated at 600°C for 4 hrs in air ambient, the surface morphology of the nickel ferrite nanoparticles looks very clear with irregular shapes. Some impurities are also observed in this sample, which may be the iron oxides as detected in the XRD results (**Fig. 1**). From the SEM image, the average particle size is measured using the sampling method, which is found to be 66.5 nm. The calculated (XRD) as well as measured (FESEM) values of average particle size are comparable.

The magnetic characterization of the synthesized nickel ferrite nanoparticles has been carried out by vibrating sample magnetometer (VSM) at room temperature with maximum applied field of up to ± 20 kOe. **Fig. 3** shows the room temperature hysteresis curves of the synthesized nickel ferrite nanoparticles. The M-H curve of as-synthesized sample (before heat treated) is shown in **Fig. 3(a)**, which depicts a very weak ferrimagnetic behavior (almost straight line). The inset of the **Fig. 3(a)** shows the values of coercivity and remanent magnetization. When the as-synthesized powder is heat treated at 600°C for 4 hrs, the magnetization curve moves to ferrimagnetic region with a very little amount of remanent magnetization and coercivity that can be seen in **Fig. 3(b)**. The inset in **Fig. 3(b)** shows the clear visibility of remanent magnetization and coercivity. The insets in **Fig. 3** show the expanded field region around the origin in order to make the clear visibility of the coercivity and remanent magnetization in each case. The very little values (almost negligible) of remanent magnetization and coercivity give the sign of the superparamagnetic

characteristic in nickel ferrite nanoparticles [5]. The superparamagnetism in nickel ferrite nanoparticles is observed here with average particle size of 69 nm (<100 nm). The values of average particle size for superparamagnetic nickel ferrite nanoparticles are reported as 11-28 nm, 8 nm [5, 11], although the critical size for superparamagnetic nickel ferrite nanoparticles is 10 nm [11]. The difference in average particle size for superparamagnetic nickel ferrite nanoparticles may be attributed to the precursor materials that were used in the preparation of nickel ferrite nanoparticles. The signature of superparamagnetism in the synthesized nickel ferrite nanoparticles using metal nitrate precursors via chemical co-precipitation method is the unique and remarkable result. It is to be noted that in order to confirm the superparamagnetism in nickel ferrite nanoparticles, the low temperature magnetic measurement is needed for the blocking temperature (TB) determination which is presently being studied.

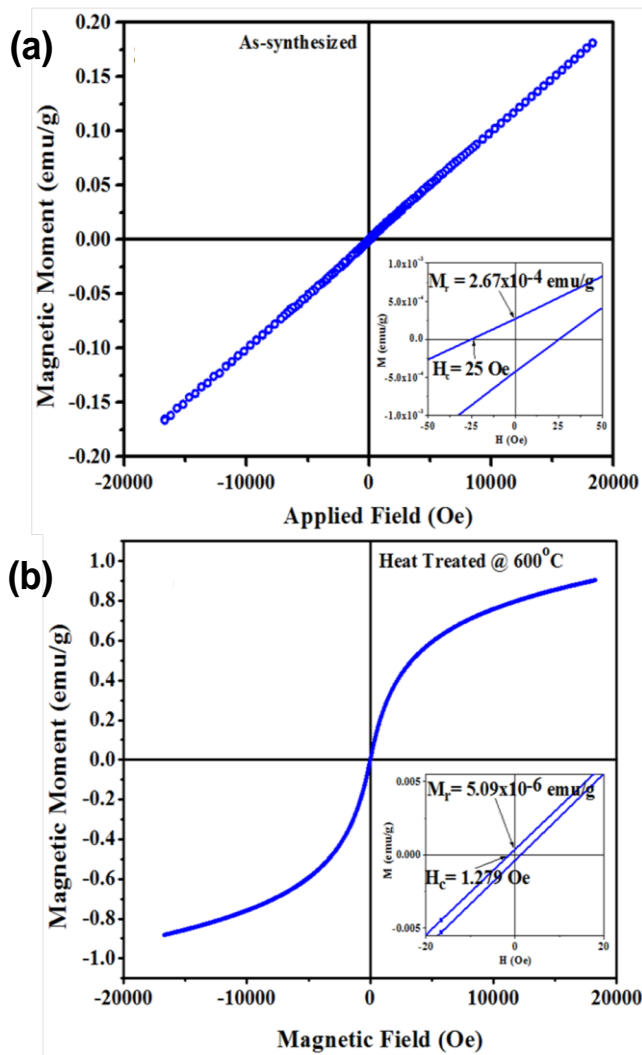


Fig. 3. The M-H curve for NiFe_2O_4 nanoparticles at room temperature, (a) As-synthesized, (b) Heat treatment at 600°C . The insets show the expanded field region around the origin for clear visibility of the coercivity and remanent magnetization.

Conclusion

The nickel ferrite nanoparticles have been synthesized by chemical co-precipitation method using the nitrate precursors. The X-ray diffraction analysis of the heat treated (600°C) sample confirms the formation of cubic inverse spinel nickel ferrite nanoparticles with average particle size of 69 nm (theoretical). The surface morphology of nickel ferrite nanoparticles has been studied using the field emission scanning electron microscopy, wherein the irregular particle size and shape have been observed. The average particle size is measured as 66.5 nm from SEM image using the sampling method. The magnetization curve of heat treated nickel ferrite nanoparticles shows the ferrimagnetic behavior with a very little amount of coercivity and remanent magnetization, which indicates the possible signature of superparamagnetism. The superparamagnetic nickel ferrite nanoparticle is the potential candidate for biomedical applications.

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