Synthesis and Characterization of NiFe$_2$O$_4$ Nanoparticles by Chemical Co-precipitation Method

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ABSTRACT

Syntheses of spinal Nickel Ferrite nanoparticles were obtain from chemical co-precipitation technique in aqueous medium without any surfactants or organic capping agent. The synthesized nanoparticles have been characterized by using XRD, SEM and FT-IR measurements. From the XRD study, it is found that the prepared samples are in the monoclinic system. Scanning Electron Microscopy (SEM) pattern shows the spherical morphology of the prepared nanoparticles. The presence of dopant in the doped sample is found by using FTIR measurements.

Keywords: NiFe$_2$O$_4$, FT-IR, nanoparticles, co-precipitation.

1. INTRODUCTION

Magnetic nanoparticles and nanoassemblies with uniform size distribution are currently of emerging interest because of their extensive applications in memory storage devices, catalysis, sensors, MRI, magnetically controlled drug delivery, hyperthermia treatment of tumor cells$^{1-4}$. Among the materials explored so far, spinel ferrites MFe$_2$O$_4$ (M = Co, Mn, Ni) are emerging as promising materials especially for biomedical applications. Compared with iron oxides, spinel ferrites provide flexibility to control both crystal structures and magnetic properties by choosing different non-iron metals in spinel ferrite backbone and controlling their molar concentrations$^5$. Hence it is possible to obtain great tenability in chemical composition and bonding through the variation of chemistry at the atomic level. Therefore research on development of fabrication methods for ferrite nanoparticles with desirable magnetic property, acceptable chemical stability...
and surface chemistry that allow for straightforward fictionalization with different surface active moieties has continued to attract interest of several research groups. A number of chemical routes have been utilized for the controlled synthesis of monodispersed ferrite particles and to establish the correlation between magnetic properties and synthetic conditions. The properties of the synthesized NiFe₂O₄ are influenced by the composition, purity, and microstructure, which are sensitive to the preparation methodology used in their synthesis. In order to achieve materials of the desired physical and chemical properties, the preparation of nickel ferrite nanocrystals through different routes has become an essential part of research and development. Many fabrication methods for preparation of nickel ferrite nanocrystals have been employed, namely by co-precipitation, hydrothermal methods, sol-gel methods, reverse micelles, chemical method, and other methods. Although most of these methods have achieved particles of the required sizes and shapes, they require complicated procedures, expensive materials, high reaction temperatures, long reaction time, toxic reagents, by-products, and potential harm to the environment.

In this study, we report the synthesis of nano sized nickel ferrite particles by co-precipitation method. The employed this method can produce fine, high-purity, stoichiometric nickel ferrite particles in very short time.

2. EXPERIMENTAL METHODS

2.1 Preparation of Nickel Ferrite (NiFe₂O₄) nanoparticles

Nickel ferrite (NiFe₂O₄) nanoparticles were synthesized by the Co-precipitation method. All chemical reagents used as starting materials are of analytical grade and purchased without any further purification. In a typical synthesis process, 0.05M of ferrous Chloride (Fe₂Cl₃) was dissolved in 100 mL aqueous solution under vigorous stirring to form a clear solution, and then 0.25M of Nickel Chloride (NiCl₂·6H₂O) and 15ml of ammonia (NH₃) solution were added to the above solution. This mixture was then vigorously stirred at room temperature for 3 hours to form a homogeneous solution. Then the solutions were mixed suddenly green colour appears and continue to stirring the solution colour changed into black and the precipitate was immediately produced. After stirring for 3hr, the precipitate was filtered. The precipitate was washed with double distilled water several times to remove the excess amine molecules. The NiFe₂O₄ nanoparticles were finally collected as a black powder after drying at room temperature.

2.2 Characterization

Prepared NiFe₂O₄ nano particles were characterized by various techniques. The structural characterization was performed using a Philip XPERT-PRO diffractometer system. CuKα line with wavelength of 1.5406 Å is generated with a setting of 30 mill amperes and 40 kV with the electrode is used for diffraction with CuKα radiation. The infrared spectra of prepared samples were recorded in the vibrational frequency ranging from 4000 to 400 cm⁻¹ using a JASCO 460 plus FTIR spectrometer. Size and shape of the particles were determined using Scanning electron microscopy (SEM JSM-5610) analysis.
3. RESULTS AND DISCUSSION

3.1. X-ray diffraction analysis

The XRD pattern of NiFe2O4 nanoparticles prepared at room temperature is shown in Fig.1. The XRD data clearly confirm the polycrystalline spinel structures of cobalt ferrite (NiFe2O4) diffraction peaks are consistent with the standard pattern for JCPDS Card No. 22-1086. For the prepared NiFe2O4 nanoparticles have the most intensive lines (311), (222), (400), (422), and (440) are observed the diffraction peak indicated at 2θ = 30.43°, 35.58°, 43.51°, 53.91° and 63.04°. These angles measured for the NiFe2O4 samples are in very good agreement with the reported values (JCPDS card No. 44-1485). The diffraction lines provide clear evidence for the formation of cubic phase of pure inverse spinel structure of nickel ferrite with Fd3m space group. The crystallite size of the NiFe2O4 product is determined by using Debye Scherrer’s formula

\[ D = \frac{k\lambda}{\beta \cos\theta} \]

Where, K=0.89 is the shape factor, \( \lambda \) is the X-ray wavelength of CuKα radiation, \( \beta \) is the full width at half maximum (FWHM) of the peaks and \( \theta \) is the glancing angle and the average crystallite size is 20.58 nm.

![X-ray diffraction pattern of NiFe2O4 nanoparticles synthesized by Co-precipitation method](image)

3.2. Fourier Transform Infrared (FT-IR) spectral Analysis

The vibrational frequencies of the various chemical bonds in the NiFe2O4 nanoparticles can assigned from FT-IR spectra of which recorded in the region 400 – 4000 cm\(^{-1}\) (Fig.3). FT-IR spectra of NiFe2O4 clearly shows an absorption bands around 3432.22 cm\(^{-1}\), which are characteristic stretching vibration of hydroxyl functional group (H-O-H) on the surface of nanoparticles or adsorbed water in the sample. The absorption band at 1721 cm\(^{-1}\) corresponds to stretching vibration of carbonyl group (C=O). The stretching vibration of the carboxylate group (C=O) is observed around 1400.73 cm\(^{-1}\). The bending vibration of H-O-H group also localized at 1632.59 cm\(^{-1}\). The absorption band around 1150.68 cm\(^{-1}\) is
assigned the stretching vibration of C-C-C group. The above observed FTIR spectra confirm the presence of organic impurities in the sample due to the preparation conditions. In addition, two absorption

![FTIR spectrum of NiFe2O4 nanoparticles](image)

Peaks at 584 cm\(^{-1}\) and 442.03 cm\(^{-1}\) are corresponding to the vibration of tetrahedral and octahedral complexes respectively, which are indicative of formation of spinel ferrite structure. As can be seen from the spectra, the normal mode of vibration of tetrahedral cluster (584.05 cm\(^{-1}\)) is higher than that of octahedral cluster (442.03 cm\(^{-1}\)).

3.3. Surface morphology Analysis

Fig. 3 shows the SEM images show the presence of voids and pores in the samples. The samples have spongy structure and the formation of multigrain agglomerations consisting of very fine crystallites. Micrographs clearly show the surface features, by which it highlight that nickel ferrite nanoparticle was successfully prepared, most of pores on the surface have smaller size, and the particles are more packed. It is cleared that the tested particles are spherical in shape with a narrow size distribution. Fig. 3 shows almost homogeneous and uniform distribution of these particles in the powder sample. The particles consisted of some regular and irregular polyhedrons with mean sizes of about 20 nm, which is in close agreement with the size obtained from XRD analysis.

![SEM image of NiFe2O4 nanoparticles](image)
4. CONCLUSION

NiFe$_2$O$_4$ nanoparticles with cubic structure with Fd3m space group have been successfully synthesized by co-precipitation method. From the morphological results, the prepared crystallites are nearly spherical in shape with a narrow size distribution and their particle sizes are more packed. The particles consisted of some regular and irregular polyhedrons with mean sizes of about 20 nm, which is in close agreement with the size obtained from XRD analysis. FT-IR study also confirms the presence of functional groups in NiFe$_2$O$_4$ nanoparticles. Finally, the cubic structure of NiFe$_2$O$_4$ nanoparticles and the average particle size of the nickel ferrite obtained from the XRD peak broadening was 19-21.57nm.

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