

Characterization of $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ nanoparticles synthesized by co-precipitation method

G. Vaidyanathan^a, S. Sendhilnathan^{b,*}

^aDepartment of Physics, Pondicherry Engineering College, Pondicherry 605014, India

^bDepartment of Physics, Sri Manakula Vinayagar Engineering College, Pondicherry 605107, India

Received 1 February 2007; received in revised form 16 August 2007; accepted 29 August 2007

Abstract

Fine nanoparticles of $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ with stoichiometric proportion (x) varying from 0 to 1.0 were prepared by the chemical co-precipitation method. The samples were characterized utilizing X-ray diffraction (XRD), vibrating sample magnetometer (VSM), Fourier transform infrared spectroscopy (FTIR) and electron paramagnetic resonance (EPR) techniques. The specific saturation magnetization (M_s) of the particles was measured at room temperature. The precipitated particles were coated with oleic acid as the surfactant by suitable method for the preparation of ferrofluid.

© 2007 Elsevier B.V. All rights reserved.

PACS: 75.50.Mm; 75.75.+a; 75.75.Tt; 87.64.Pj

Keywords: Co-precipitation; Fine particles; Nanoferrites; Spinel

1. Introduction

Magnetic nanoparticles have attracted the attention of researchers of various fields due to their extensive applications in information storage system, medical diagnostics, ferrofluid technology, etc. [1–5]. This is mainly because the nanoparticles differ from those of the corresponding bulk material [6,7]. For the preparation of magnetic fluids, nanoparticles with a particle size of order of 10 nm and with a narrow size distribution are needed [8]. However, if the size of the magnetic particle is decreased below a critical length, domain formation is no longer energetically favored and the particles exist as single domain [9]. Zinc substituted mixed ferrites (Co–Zn) are chosen due to their high sensitivity of magnetization to temperature. Ferrofluids constituted by these ferrites may be good candidates for liquid carriers in heat exchange devices using magneto caloric energy conversion [10,11]. It is well known that the

magnetic property can be altered by the addition of the zinc. Addition of zinc also affects the lattice parameter (a_0). Various preparation techniques, such as reverse micelle technique [12–14], sol–gel pyrolysis method [15] hydrothermal technique [16] and mechanical alloying [17] are used to prepare ferrite nanoparticles. But co-precipitation method is considered to be economical means of producing fine particles [18,19] for the preparation of temperature sensitive ferrofluid. Hydrothermal method even though cheaper is much used in emulsion preparation for water based ferrofluid, for heat transfer studies it is required to prepare ferrofluid with higher boiling point and lower volatility. For this, chemical co-precipitation technique is much suitable for better surfactant adherence. Using co-precipitation method we can prepare nano-sized transformer oil based ferrofluids having high boiling point and viscosity, which could be used for the preparation of temperature sensitive magnetic fluid. The physical properties of nanoparticles are of current interest due to the size-dependent behavior observed in the nanometer length scale and high crystallinity. In the present work, we have synthesized $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ with x varying from 0 to 1.0. The influence of zinc substitution on the crystallite size and

*Corresponding author. Tel.: +91 413 2655281x647;
fax: +91 413 2655101.

E-mail addresses: gvn_pec@yahoo.com (G. Vaidyanathan),
sendhil29@yahoo.co.in (S. Sendhilnathan).

magnetic properties depends on the preparation conditions. Preparation and properties of Co–Zn ferrites have been reported only for particular value of x or limited values. Jeyadevan et al. [20] successfully prepared nearly monodispersed single-domain cobalt ferrite particles, which could be used for the high-density recording media. Morais et al. [21] have reported the possibility of controlling the size of the nanoparticles using different stirring speeds. Temperature sensitive magnetic fluid having $\text{Co}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$ particles was used for the study of thermal convection [10] and diester based ferrofluid having $\text{Co}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4$ nanoparticles have been reported [22]. Arulmurugan et al. [23] have reported variation of physical properties for $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ nanoparticles with x varying from 0.1 to 0.5. To the best of our knowledge, complete range of $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ nanoparticles with x varying from 0 to 1.0 have not yet been reported. Here we report the modified preparation condition for the production of fine particles of $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ with x varying from 0 to 1.0. The structural and magnetic properties, which depend on the effect of zinc substitution, are also studied. In the present paper, we report a comparative study of the Fourier transform infrared spectroscopy (FTIR) and electron paramagnetic resonance (EPR) spectra for $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ with x varying from 0 to 1.0 for the oleic acid uncoated (S1) and coated (S2) particles.

2. Experimental

2.1. Synthesis of $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ uncoated nanoparticles (S1)

$\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ fine particle with x varying from 0 to 1.0 were prepared by chemical co-precipitation method. Initial molar proportion of salts $\text{Me}^{2+}/\text{Fe}^{3+}$ was 0.5, where $\text{Me}^{2+} = (\text{Co}^{2+} + \text{Zn}^{2+})$. Aqueous solutions of CoCl_2 , ZnCl_2 and FeCl_3 in their respective stoichiometry (100 ml of 0.5 M CoCl_2 , 100 ml of 0.5 M ZnCl_2 and 100 ml of 2 M FeCl_3 in the case of $\text{Co}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ and similarly for the other values of x) were mixed and kept at 60 °C. This mixture was added to the boiling solution of NaOH (0.63 M dissolved in 1200 ml of distilled water) after adjusting the pH to be around 12, within 10 s under constant stirring. The solutions were maintained at 85 °C for 1 h. This duration was sufficient for the transformation of hydroxides into spinel ferrite (dehydration and atomic rearrangement involved in the conversion of intermediate hydroxide phase into ferrite) [24]. Sufficient amount of fine particles were collected at this stage by using magnetic separation. These particles were washed several times with distilled water followed by acetone and dried at room temperature (RT). We shall denote this precipitate as S1.

2.2. Synthesis of $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ coated nanoparticles (S2)

The pH of the solution was reduced to ≈ 10.5 as coating of surfactant takes place only at pH in between 10 and 11.

Oleic acid ($\text{C}_{18}\text{H}_{34}\text{O}_2$) was used as the surfactant after heating it with NaOH solution at a pH of 10, for the conversion of oleic acid to sodium oleate. The sodium oleate solution was transferred to the reaction vessel and stirred continuously for nearly 3 h. Coating of surfactant was carried out at a temperature of about 80 °C and maintained at that temperature for 30 min. To coagulate the oleic acid coated particles, dilute HCl was added. After decantation, the product was washed a number of times with distilled water to remove soluble impurities. After removing the excess water by washing it with acetone, the coated particles were collected. We shall denote this precipitate as S2.

2.3. Particle characterization

The X-ray diffraction (XRD) patterns of the samples were recorded on a Philips PANALYTICAL X'PERT PRO X-ray powder diffractometer using Cu K_α ($\lambda = 1.54060 \text{ \AA}$) radiation. Slow scans of the selected diffraction peaks were carried out in step mode (step size 0.05° , measurement time 5 s, measurement temperature 25 °C, standard: Si powder). The crystallite size of the nanocrystalline samples was measured from the X-ray line broadening analyses using Debye–Scherrer formula after accounting for the instrumental broadening:

$$D_{\text{XRD}} = \frac{0.89\lambda}{\beta \cos \theta},$$

where λ is the wavelength of X-ray used in $\text{\AA}$, β the FWHM in radians in the 2θ scale, θ the Bragg angle, D_{XRD} the crystallite size in nm [25]. The lattice constant (a_0) was determined as a function of zinc content. Cobalt zinc ferrite has a spinel structure [23].

2.4. Magnetic measurements

RT magnetic measurements with a maximum magnetic field of 1194.15 kA/m were carried out using a Lakeshore vibrating sample magnetometer (VSM) (model 7404) and parameters like specific saturation magnetization (M_s), coercive force (H_c) and remanence (M_r) were evaluated for $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ uncoated nanoparticles (S1).

2.5. FTIR measurements

FTIR spectra were recorded for dry samples (uncoated (S1) and coated (S2) with oleic acid) of $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ with x varying from 0 to 1.0 with an ABB BOMEM 104 FTIR (range $400\text{--}4000 \text{ cm}^{-1}$) spectrometer. The dry samples were in KBr matrix, and spectra were measured according to transmittance method. The spectra were resolved with a resolution of 4 cm^{-1} . For spinel structure, it is allowed to identify some Me–O vibrations as well as the presence of the water adsorbed on the particle surface. To understand the adsorption mechanism of the oleic acid on the surface of the cobalt nanoparticles FTIR

Download English Version:

<https://daneshyari.com/en/article/1813062>

Download Persian Version:

<https://daneshyari.com/article/1813062>

[Daneshyari.com](https://daneshyari.com)