Available online at www.sciencedirect.com
SCIENCE DIRECT®
ELSEVIER
Journal of Magnetism and Magnetic Materials 294 (2005) e33–e36
Journal of magnetism and magnetic materials
www.elsevier.com/locate/jmmm

Synthesis and magneto-structural study of $Co_xFe_{3-x}O_4$ nanoparticles

R. Betancourt-Galindo[a,b,*], O. Ayala-Valenzuela[b], L.A. García-Cerda[a], O. Rodríguez Fernández[a], J. Matutes-Aquino[b], G. Ramos[c], H. Yee-Madeira[d]

[a]*Centro de Investigación en Química Aplicada, Blvd. Enrique Reyna Hermosillo No. 140 CP 25000 Saltillo, Coahuila, México*
[b]*Centro de Investigación en Materiales Avanzados, Miguel de Cervantes Saavedra. No. 120. Complejo Industrial Chihuahua, Chihuahua, Chih. México*
[c]*Centro de Investigación en Ciencia Aplicada y Tecnología Avanzada, José Siurob No. 10. Col. Alameda, CP 76040 Querétaro, Qro. México*
[d]*Escuela Superior de Fisica y Matemáticas, IPN, Edif. 9, U. Prof. "ALM", 07738 Col. Lindavista, México D.F., México*

Available online 15 April 2005

Abstract

The Co^{2+} ion in an octahedral site of the cubic spinel structure has a highly anisotropic character. The electric crystal field produces a degenerate ground state with a orbital magnetic momentum fixed parallel to a $\langle 1\,1\,1 \rangle$ trigonal axis, and the spin–orbit interaction tends to align the spin magnetic moment parallel to this trigonal axis giving high anisotropy. Then, the use of $Co_xFe_{3-x}O_4$ system allows the tailoring of the magnetic properties by changing the cobalt content, which can be very useful in magnetic fluids, magnetic latex and free rotors applications. In this work $Co_xFe_{3-x}O_4$ nanoparticles over a compositional range $0.0 < x < 1.0$ were synthesized by chemical co-precipitation from iron and cobalt salts using ammonium hydroxide as precipitating agent. The powders were characterized by X-ray diffraction, transmission electronic microscopy and vibrating sample magnetometry. Nanoparticles with a size smaller than 15 nm with a narrow particle distribution size were obtained. X-ray diffraction confirmed the formation of a unique phase. The magnetic behavior of $Co_xFe_{3-x}O_4$ powders shows that an increase of the cobalt contain yields a steadily decrease in the maximum magnetization.

PACS: 75.75 + a; 76.80 + y

Keywords: Chemical co-precipitation; Nanoparticles; Superparamagnetism; Mössbauer effect

*Corresponding author. Centro de Investigación en Química Aplicada, Blvd. Enrique Reyna Hermosillo No. 140 CP 25000 Saltillo, Coahuila, México. Tel.: +52 844 4389830x1377; fax: +52 844 4389839.
E-mail address: rgalindo@ciqa.mx (R. Betancourt-Galindo).

0304-8853/$ - see front matter © 2005 Elsevier B.V. All rights reserved.
doi:10.1016/j.jmmm.2005.03.049

1. Introduction

Magnetic nanoparticles of the spinel-type receive much attention because of their interesting magnetic properties and potential applications [1]. It has been observed that they have superparamagnetic properties, due to its reduced size. This make them attractive candidates for information storage and many other applications including magnetic fluids, magnetic latex. They can be prepared using different methods like sol–gel [2], microemulsion [3], mechanical alloying [4] and chemical co-precipitation [5]. While techniques like microemulsion promise easy control over the particle size other techniques produce larger particles due to agglomeration during the different process steps.

In this work, we report the study on the size distribution and properties of $Co_xFe_{3-x}O_4$ nanoparticles prepared using chemical co-precipitation.

2. Experimental

As starting materials iron and cobalt chlorides ($FeCl_3 \cdot 6H_2O$, $FeCl_2 \cdot 4H_2O$, $CoCl_2 \cdot 6H_2O$) and NH_4OH reagent grade were used. The $Co_xFe_{3-x}O_4$ nanoparticle precipitates were obtained from two starting solutions, one with a mixture of metallic salts with the proper stoichiometric proportion, and the other containing the alkali hydroxide. The chemical reaction was carried out using a molar ratio of 1:2 of $Fe^{+2}Co^{+2}$:Fe^{+3}, at 70 °C under intense agitation. The alkali solution was added as fast as possible in order to keep a high pH level. No surfactants were used in our process. The precipitates were then thoroughly washed with distilled water to eliminate chloride ions and finally dried at room temperature for use. The precipitates were structurally characterized using a Siemens D5000 X-ray diffractometer. The magnetic characterization was carried out by means of magnetometry and Mössbauer spectrometry. The hysteresis curves were obtained using a Lakeshore 7300 vibrating sample magnetometer and a maximum field of 15000 Oe was used. The Mössbauer spectra were obtained in transmittance using a Wissel Scientific Instruments spectrometer with a 25 mCi Co57 (Rh) source in constant velocity mode at room temperature. The spectrometer was calibrated using in α-Fe standard prior to measurement. The morphology and particle size were characterized using a TEM JEOL 1200 EXII.

3. Results and discussion

Fig. 1 shows the X-ray diffraction patterns of the $Co_xFe_{3-x}O_4$ powders with $x = 0.0$, 0.2, 0.4, 0.8 and 1.0, where between six and seven broad diffraction peaks can be clearly observed in each pattern in good agreement with the diffraction peaks of $CoFe_2O_4$ small nanoparticles. Also a low angle amorphous halo that increases with x is observed.

Fig. 2 shows a micrograph of the $Co_xFe_{3-x}O_4$ nanometric sized particles with $x = 0.2$ obtained with TEM. The images for the other samples look

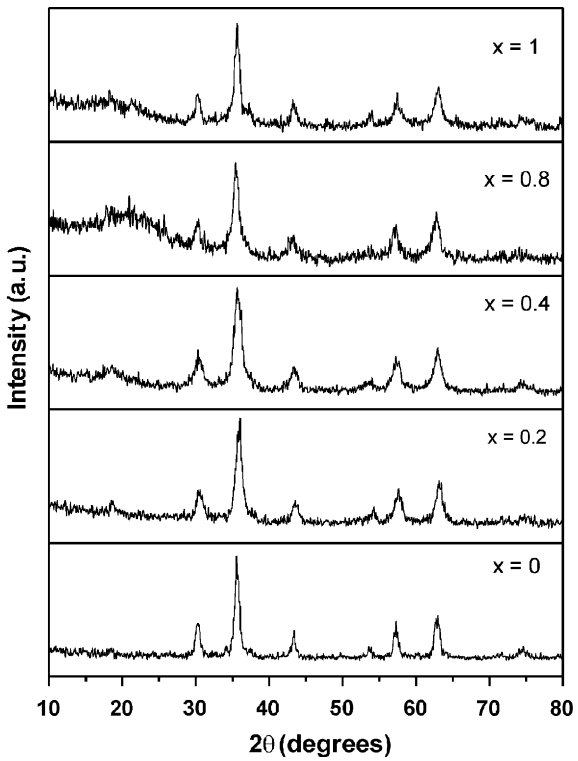

Fig. 1. X-ray diffraction patterns of $Co_xFe_{3-x}O_4$ samples.