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Synthesis of $CoFe_2O_4$ nanoparticles embedded in a silica matrix by the citrate precursor technique

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Abstract

A metals–citrate–silica gel was prepared from metallic salts, citric acid and tetraethylorthosilicate by sol–gel method (citrate precursor technique) and it was further used to prepare magnetic nanocomposites. The gel was dried at 100 °C and then calcined at temperatures between 600 and 1000 °C to obtain powder samples. The nanocomposites were characterized by XRD, IR, VSM and TEM techniques. The diffraction patterns show the formation of a single magnetic phase identified as $CoFe_2O_4$. Magnetic nanoparticles with average size less than 50 nm were obtained which are well dispersed in the silica matrix. The combination of different metals concentrations and calcining temperatures allowed obtaining samples with magnetization ranging from 3.6 to 25.3 emu/g.

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Keywords: $CoFe_2O_4$ nanoparticles; Citrate precursor technique; Silica matrix; Magnetic nanocomposite; Magnetic properties

1. Introduction

Magnetic nanoparticles have been widely studied as they present an interest in both fundamental physics and potential applications such as catalysis and magnetic recording. Many techniques have been employed to prepare magnetic nanoparticles, i.e. mechanical alloying, coprecipitation, microemulsion [1–3]. The tendency of magnetic particles with nanometer dimensions to agglomerate makes the study of their behavior difficult. For this reason the dispersion of nanoparticles in silica matrix is an excellent method to reduce particle agglomeration and this technique allows to stabilize the particles and to study their formation reactions [4].

The citrate precursor technique is a way with a unique combination of the chemical sol–gel method. It has the advantages of using inexpensive precursors, a simple preparation process, and producing nanometric particles. This technique has been used to prepare different magnetic

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materials [5,6]. However, the synthesis of $CoFe_2O_4$ dispersed in silica matrix using citrate–sol–gel method has not been reported previously.

In this work, $CoFe_2O_4$ nanoparticles embedded in a silica matrix were prepared via citrate precursor technique. The samples were characterized with X-ray diffraction (XRD), infrared spectroscopy (IR), transmission electron microscopy (TEM). The magnetic properties of the powders were studied at room temperature by vibrating sample magnetometry (VSM).

2. Experimental

The starting chemicals used in this work were: TEOS, ethanol, iron nitrate, cobalt nitrate, citric acid and ethylene glycol all purchased from Aldrich. The weight ratio $CoFe_2O_4$:silica compositions chosen in this work were 40:60 and 50:50. The Fe:Co and TEOS:ethanol:water molar ratios were controlled at 2:1 and 1:4:11.67, respectively. In the preparation of samples, clear solutions were first prepared by mixing TEOS-ethanol solution and hydrolysis was then performed by adding water. After stirring for 1 h at room temperature, Fe and Co nitrates dissolved in citric acid and ethylene glycol were added into the solution drop by drop. The resultant solution was continuously stirred until it became transparent. After aging for 2 days at room temperature gel samples were obtained. The gels were dried at 100 °C for 24 h. The material obtained was ground to form a fine powder and then was calcined at temperatures between 600 and 1000 °C for 2 h in air atmosphere.

The magnetic phase was identified using an X-ray diffractometer Siemens D-5000 using Cu Kα radiation and operated at 25 mA and 35 kV. The infrared spectra were obtained with a FTIR spectrometer Thermo-Nicolet /Nexus 470, in the diffuse reflectance mode, mixing 0.05 g of powder sample with 0.45 g of KBr. The magnetic properties of the nanocomposites were measured using a vibrating sample magnetometer Lakeshore 7300 with a maximum field of 15 kOe. A transmission electron microscope JEOL JEM-1200EXII was employed to determine the particle size and morphology of $CoFe_2O_4$ nanoparticles inside the silica.

3. Results and discussion

Fig. 1 shows the XRD patterns of the $CoFe_2O_4$-silica samples with a weight ratio of 50:50 and calcined at different temperatures. It clearly indicates a single phase, identified as cobalt ferrite formed around 600 °C [7]. With the calcination temperature, the increase in sharpness of XRD lines indicates the growth of crystallite size. Similar results were obtained for the sample with a weight ratio of 40:60. The increase of the calcination temperature results in higher crystallization without changes in the obtained magnetic phase.

The infrared absorption spectra in the range of 400–1800 cm^{-1} obtained from the sample with weight ratio 50:50 calcined at 600, 800 and 1000 °C are shown in Fig. 2. The samples presented the characteristic bands of silica at 460,

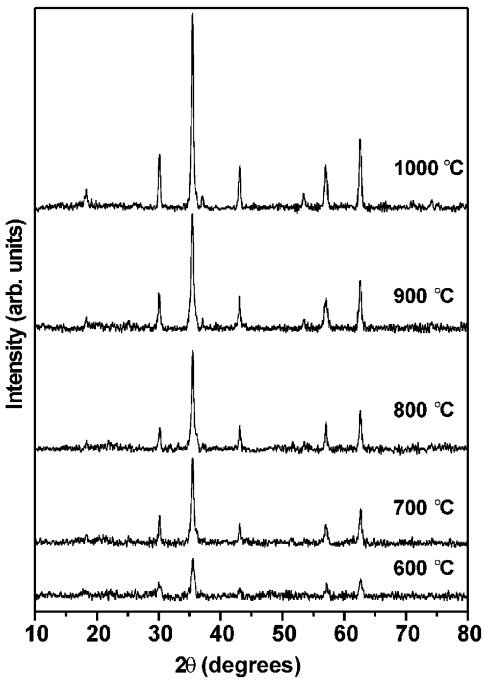

Fig. 1. XRD patterns of the $CoFe_2O_4$-silica samples with weight ratio of 50:50 obtained at different temperatures.