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Magnetic properties of triethylene glycol coated CoFe_2O_4 and $\text{Mn}_{0.2}\text{Co}_{0.8}\text{Fe}_2\text{O}_4$ NP's synthesized by polyol method

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Abstract In this study, we reported on the structural and magnetic properties of TEG- CoFe_2O_4 and TEG- $\text{Mn}_{0.2}\text{Co}_{0.8}\text{Fe}_2\text{O}_4$ nanocomposites produced by the glycothermal reaction (polyol). X-ray diffraction (XRD), transmission electron microscopy (TEM) and vibration sample magnetometer (VSM) analysis have been carried out in order to understand the effect of Mn^{2+} into CoFe_2O_4 and it was observed that the addition of Mn^{2+} tends to reduce the crystallite size, increase the a_o (cell parameter) and increase the T_B . The presence of adsorbed polyol entities on the surface of the CoFe_2O_4 and $\text{Mn}_{0.2}\text{Co}_{0.8}\text{Fe}_2\text{O}_4$ NP's was also proven by TG measurements. FT-IR analysis suggested the presence of adsorbed TEG molecules on the surface of CoFe_2O_4 and $\text{Mn}_{0.2}\text{Co}_{0.8}\text{Fe}_2\text{O}_4$ NP's.

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1. Introduction

Both MnFe_2O_4 and CoFe_2O_4 ferrites are among the widely studied ferrites because of a number of possible applications

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such as high frequency magnetostrictive and electromagnetic applications (Zi et al., 2009; Msomi et al., 2011). In order to change the magnetic properties, substitution of other elements such as manganese into cobalt ferrites was proposed by many researchers (Paulsen et al., 2004; Caltun et al., 2007a,b). Zhou et al. (2002) reported that thin films and fine particles of the manganese doped cobalt ferrites were found to be suitable for magneto-optical applications. Mossbauer spectroscopy investigation of $\text{CoMn}_x\text{Fe}_{2-x}\text{O}_4$ was carried out by Kriebel et al. (2005). The superparamagnetic properties of ferrite materials can be controlled by particle sizes and concentration of non-magnetic atoms. Efforts are being made to produce fine articles and to find dopants that can improve the properties and find new applications. Recently, interest has also shifted to Mn doped CoFe_2O_4 ferrites, which appear to have high stress sensitivity and large magnetomechanical effects, making these

ferrites suitable for application as stress sensors (Msomi et al., 2011; Kambale et al., 2010; Shobana and Sankar, 2009; Shobana et al., 2009).

A number of synthetic polymers such as poly(ethylene glycol), poly(acrylamide), and poly(*N*-isopropylacrylamide), as well as naturally occurring polymers such as dextran and chitosan were used for modifying the surface properties of magnetic particles to achieve desirable surface functionalities (Takafuji et al., 2004).

Polyol process is a versatile chemical approach, which uses poly alcohols to reduce metal salts to metal particles, was successfully used to prepare a great variety of non-aggregated inorganic compounds. The polyols often serve as reaction medium with high boiling point solvent and reducing agent, as well as stabilizer to control the particle growth and prevent interparticle aggregation. The advantage of this method is the possibility to control experimental conditions and easy scale-up (Fievet et al., 1989; Feldman and Jungk, 2001; Cai and Wan, 2007; Kim et al., 2007; Chae et al., 2004; Gupta et al., 2007).

In this study, both TEG- CoFe_2O_4 and TEG- $\text{Mn}_{0.2}\text{Co}_{0.8}\text{Fe}_2\text{O}_4$ nanocomposites were synthesized by the glycothermal reaction (polyol) for the first time. Structural, morphological and magnetic characterization of both products was done by XRD, FT-IR, TGA, TEM and VSM methods.

2. Experimental

2.1. Instrumentation

X-ray powder diffraction (XRD) analysis was conducted on a Rigaku Smart Lab operated at 40 kV and 35 mA using Cu K α radiation ($\lambda = 1.54059 \text{ \AA}$).

Fourier transform infrared (FT-IR) spectra of the samples were recorded with a Perkin Elmer BX FT-IR infrared spectrometer in the range of 4000–400 cm^{-1} .

The thermal stability was determined by thermo gravimetric analysis (TGA, Perkin Elmer Instruments model, STA 6000). TGA thermo grams were recorded for 5 mg of powder sample at a heating rate of 10 $^\circ\text{C}/\text{min}$ in the temperature range of 30–800 $^\circ\text{C}$ under nitrogen atmosphere.

VSM measurements were performed by using a Vibrating sample magnetometer (LDJ Electronics Inc., Model 9600). The magnetization measurements were carried out in an external field up to 15 kOe at room temperature.

Transmission electron microscopy (TEM) analysis was performed using a FEI Tecnai G2 Sphera microscope. A drop of diluted sample in alcohol was dripped onto a TEM grid.

2.2. Chemicals

$\text{Co}(\text{acac})_2$, $\text{Mn}(\text{acac})_2$, $\text{Fe}(\text{acac})_3$, and triethylene glycol (TEG) were purchased from Merck and used as received without further purification.

2.3. Procedure

For the synthesis of TEG- CoFe_2O_4 nanocomposite, a stoichiometric amount of $\text{Co}(\text{acac})_2$ and $\text{Fe}(\text{acac})_3$ was dissolved in TEG (20 mL). And for the synthesis of TEG- $\text{Mn}_{0.2}\text{Co}_{0.8}\text{Fe}_2\text{O}_4$ nanocomposite, a stoichiometric amount of $\text{Co}(\text{acac})_2$, $\text{Mn}(\text{acac})_2$ and $\text{Fe}(\text{acac})_3$ was dissolved in TEG (20 mL). Then

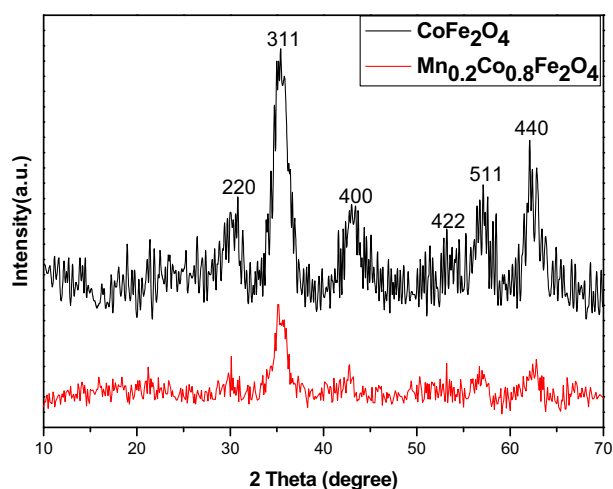


Figure 1 XRD powder patterns of as-synthesized: (a) CoFe_2O_4 and (b) $\text{Mn}_{0.2}\text{Co}_{0.8}\text{Fe}_2\text{O}_4$ NP's capped with TEG.

both solutions were continuously heated to 110 $^\circ\text{C}$ under vigorous magnetic stirring and nitrogen atmosphere separately. After heating for 1 h, the solutions were then heated to 210 $^\circ\text{C}$ and kept for 2 h at this temperature. The system was then refluxed at 295 $^\circ\text{C}$ for 1 h. Finally the black–brown mixture (for TEG- $\text{Mn}_{0.2}\text{Co}_{0.8}\text{Fe}_2\text{O}_4$ nanocomposite) and black mixture (for TEG- CoFe_2O_4 nanocomposite) were cooled down to room temperature by removing the heat source. Then, ethanol was added and the solutions were centrifuged at 8000 rpm for 15 min to remove the solvent. Then obtained precipitates were washed by ethanol for three times, which could be easily dispersed in water.

3. Results and discussion

3.1. XRD analysis

The X-ray diffraction patterns of the TEG- CoFe_2O_4 nanocomposite and TEG- $\text{Mn}_{0.2}\text{Co}_{0.8}\text{Fe}_2\text{O}_4$ nanocomposite (Fig. 1) show six reflection planes; (220), (311), (400), (422), (511) and (440), which indicate the presence of the spinel cubic structure

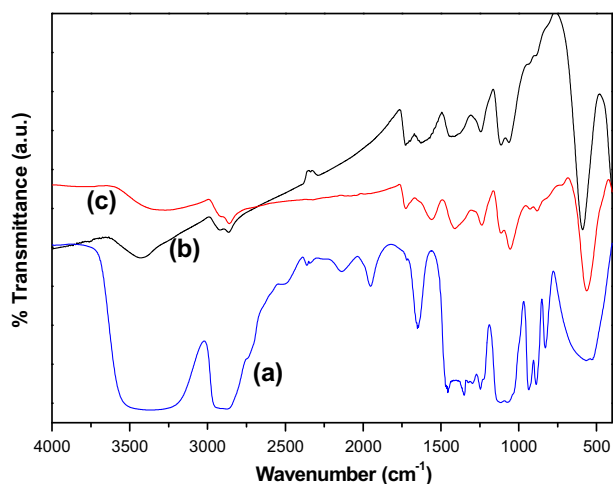


Figure 2 FT-IR spectrum of as-synthesized: (a) TEG, (b) CoFe_2O_4 and (c) $\text{Mn}_{0.2}\text{Co}_{0.8}\text{Fe}_2\text{O}_4$ NP's capped with TEG.

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