



The effect of cobalt substitution on magnetic hardening of magnetite



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ABSTRACT

In this work cobalt-substituted magnetite ($\text{Co}_x\text{Fe}_{1-x}\text{Fe}_2\text{O}_4$, $x=0, 0.25, 0.50$ and 0.75) nanoparticles were synthesized by coprecipitation method and their structural and magnetic properties were investigated. X-ray diffraction was carried out and the results show that all of the samples have single phase spinel structure. Microstructure of the samples was studied using a field emission scanning electron microscope and the results show that particle sizes of the prepared nanoparticles were uniform and in the 50–55 nm range. Room temperature magnetic properties of the nanoparticles were measured by an alternating gradient force magnetometer and the results revealed that substituting cobalt for iron in magnetite structure, changes the magnetite from a soft magnetic material to a hard one. So that coercivity changes from 0 (a superparamagnetic state) to 337 Oe (a hard magnetic material), which is a remarkable change. Curie temperatures of the samples were determined by recording their susceptibility-temperature ($\chi-T$) curves and the results show that by increasing cobalt content, Curie temperature of the samples also increases. Also $\chi-T$ curves of the samples were recorded from above Curie temperature to room temperature (first cooling), while the curves in the second heating and second cooling have the same behaviour as the first cooling curve. The results depict that all samples have different behaviour in the first cooling and in the first heating processes. This shows remarkable changes of the cation distribution in the course of first heating.

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1. Introduction

Magnetic nanoparticles have received much attention in recent years due to their underlying applications in targeted drug delivery, medical diagnostic, genetic screening [1–5], biosensors [6], ferrofluids [7], gas sensing [8], catalyses [9,10], high-density information storage devices [11–13], etc. Magnetic properties of the nanoparticles can be more complicated than those of their bulk counterparts. In fact, all relevant magnetic properties (coercivity, blocking temperature, saturation and remanent magnetizations) are functions of particles' size and shape and of surface chemistry [14,15]. For example as size reduces to a definite size, nanoparticles exhibit the so-called superparamagnetic regime, which is of great interest in macroscopic quantum tunnelling of spin states [16,17]. Among the various magnetic nanoparticles, spinel ferrites have been extensively studied because of their interesting magnetic properties. Specifically magnetite and cobalt ferrite, which both have spinel structure belonging to the space group ($Fd3m$), play a key role in magnetic applications.

Magnetite with the formula Fe_3O_4 is the most strongly magnetized material found in nature. At room temperature (RT) magnetite has a cubic inverse spinel structure which can be visualized as a face centred cubic arrangement of the oxygen anions [18]. Structurally, the cations in Fe_3O_4 are distributed between two sites, as $(\text{Fe}^{3+})_A[\text{Fe}^{3+}\text{Fe}^{2+}]_B\text{O}_4$, where A and B indicate tetrahedral and octahedral sites respectively [19]. In case of cobalt ferrite with formula $(\text{Co}_\delta\text{Fe}_{1-\delta})[\text{Co}_{1-\delta}\text{Fe}_{1+\delta}]\text{O}_4$, tetrahedral (A-site) and octahedral (B-site) sites are occupied randomly by $\text{Co}^{3+/2+}$ and $\text{Fe}^{3+/2+}$, respectively [20]. This type of cation distribution is called partially inverse spinel structure and δ as the degree of inversion which represents the ratio between divalent and trivalent ions on both A and B sites, depends mostly on the thermal history of the sample [21]. Also it has been reported that count upon synthesis conditions, cobalt ferrite can form both normal and inverse spinel structure [22]. Cobalt ferrite has been extensively investigated owing to its interesting properties such as high coercivity, moderate saturation magnetization, high chemical stability, wear resistant, and electrical insulation [23]. These beneficial characteristics make it as a suitable candidate for many applications mentioned above.

Each application of spinel ferrite nanoparticles requires a somewhat different set of magnetic properties in nanocrystals. Researching on a widespread range of several compositions and

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sizes, reveals an existence of a potential for tuning the magnetic properties and then vastly developing the range of applications. For example by preparation and characterisation of various spinel ferrites, MeFe_2O_4 (Me=Fe, Co, Mg, Mn, Zn, etc.) changing the amount of metals which exist in their composition, substituting one or more for other one, changing the synthesis method [11] or even changing the synthesis conditions, a desire characteristic can be achieved.

Both Fe_3O_4 and CoFe_2O_4 in bulk form have inverse spinel structures and their magnetocrystalline anisotropy constants (K) are 14 and 380 kJ/m^3 respectively. This huge difference in K values allows the possibility of controlling the magnetic properties of iron oxide nanoparticles by substituting different amounts of cobalt for iron [19]. Wide range applications of ferrites with small particles leads to great expansion of methods for their preparation, such as microwave plasma [24], Host template [25], microemulsion [26], hydrothermal synthesis [27], sol-gel [28], sonochemical reaction [29], forced hydrolysis [30], citrate precursor techniques [31], electrochemical synthesis [32], solid-state reaction [33], combustion methods [34], mechanical alloying [35], and coprecipitation [5,11,36–38]. As it is well known, chemical coprecipitation method is an economical [39] and the most frequent way to produce ultrafine powders. This method is quite simple, fast and cheap since it does not involve intermediate decomposition and does not need calcining steps. In this work, a series of $\text{Co}_x\text{Fe}_{1-x}\text{Fe}_2\text{O}_4$

($x=0.00, 0.25, 0.50$ and 0.75) nanoparticles was synthesized by coprecipitation method and the effect of Co substitution on the magnetic properties of magnetite was investigated.

2. Experimental

As mentioned before Co substituted magnetite nanoparticles with nominal formula of $\text{Co}_x\text{Fe}_{1-x}\text{Fe}_2\text{O}_4$ ($x=0.00, 0.25, 0.50$ and 0.75) were prepared by the coprecipitation method. The starting materials were corresponding stoichiometric quantities of high purity FeCl_3 , $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$, CoCl_2 and NaOH (all of analytical grades) from Merck Company, Germany. Stoichiometric ratios (1:2) ($(\text{Fe}^{2+} + \text{Co}^{2+}):\text{Fe}^{3+}$) of FeCl_3 , $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ and CoCl_2 were dissolved in deionized double distilled water and a 5 M sodium hydroxide (NaOH) solution, with a ratio (1:5) (metal chlorides: sodium hydroxide) was added as fast as possible. Black precipitates were obtained after the rapid addition of NaOH solution. The precipitates were washed several times with deionized double distilled water and were then dried at room temperature over days.

To study the effect of annealing on magnetic properties of the samples, sample with $x=0.50$ ($\text{Co}_{0.5}\text{Fe}_{0.5}\text{Fe}_2\text{O}_4$) has been chosen as a typical Co substituted magnetite and heated from room temperature to 800°C for 2 h by a rate of $8^\circ\text{C}/\text{min}$ and then

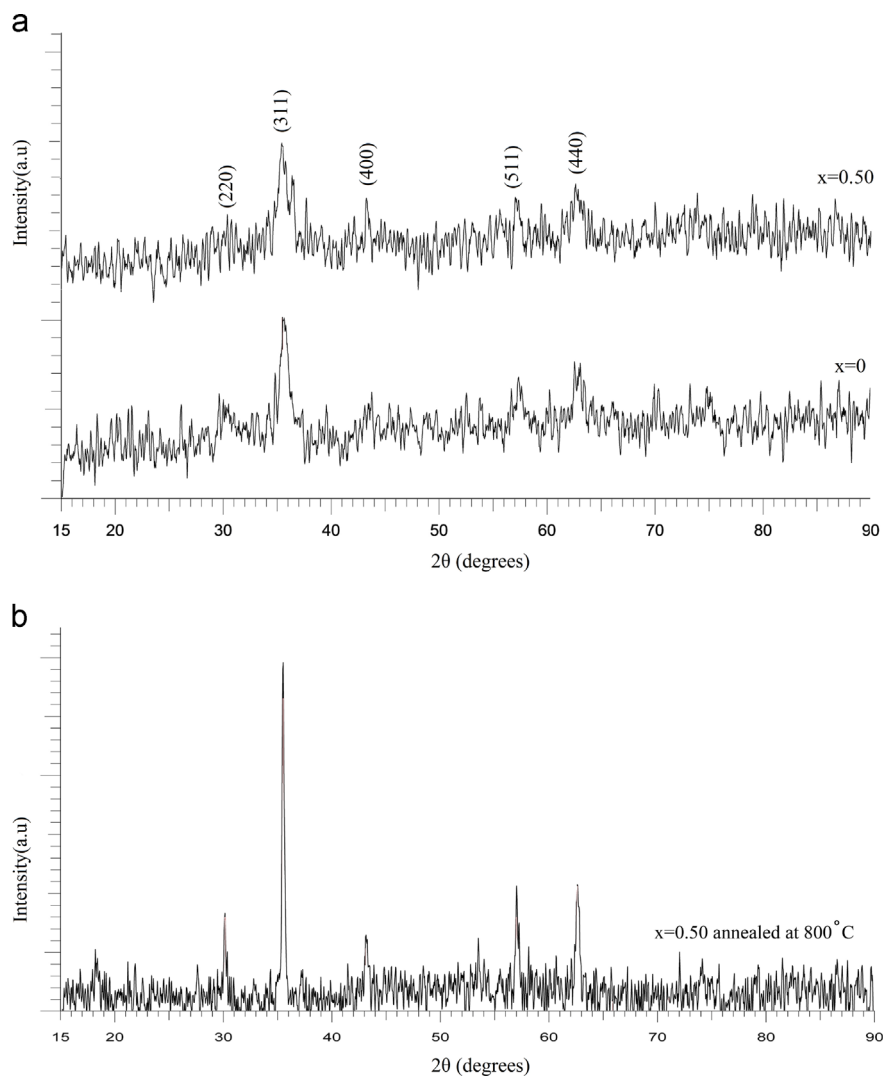


Fig. 1. (a) XRD patterns of the as-precipitated samples with $x=0.00$ and 0.50 . (b) XRD pattern of the sample with $x=0.50$ annealed at 800°C for 2 h in air.

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