



# Structural and magnetic properties of calcium doped nickel ferrite nanoparticles by co-precipitation method



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## ABSTRACT

It is a truism that a sequence of calcium doped nickel ferrite (with  $x = 0.0, 0.2, 0.4, 0.6, 0.8$  and  $1.0$ ) nanoparticles are combined by co-precipitation technique. X-Ray Diffraction (XRD) and Fourier Transform Infrared (FTIR) scrutinize the formation of single-phase inverse spinel structure in all the compositions. The lattice framework increases with the increase in calcium concentration and it exhibits the development of unit cell. Crystallite size in the range of 22–34 nm is viewed and also augmented the level of calcium. The elemental composition of pure and calcium doped nickel ferrite has been procured from Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) and Energy Dispersive X-ray analysis (EDX). It is interesting to note that the substitution of calcium increasingly exerts influence on the magnetic characteristics. These observations paved the way for the room temperature of magnetization measurements. The saturation magnetization and the experimental value of magnetic moment are noticed to enlarge initially up to  $x = 0.2$ , and then decrease incessantly with increase in the Ca content  $x$ . The increase and the decrease of saturation magnetization have widely been expounded by Neel's collinear two-sublattice model and Yafet–Kittel (Y–K) three-sub lattice model.

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## 1. Introduction

Spinel ferrite nanoparticles are constituted by a general formula.  $A^{2+}Fe_2^{3+}O_4$ , where  $A^{2+}$  has been replaced by suitable divalent metal ions such as Co, Cu, Mg, Zn, Mn and Ni etc. In the recent past, the nanoparticles of spinel ferrite have been intensively studied on account of their electrical and magnetic properties and their large scale application in the fields of magnetic resonance imaging (MRI), magnetic high-density information storage [1], microwave absorption [2], telecommunication applications [3], high frequency transformer, memory core devices, and biomedical applications [4].

Nickel ferrite is one of the soft ferrite material due to its low conductivity [5], lower eddy current losses and high electrochemical stability [6] with inverse spinel structure. The nickel ferrite properties are, to a great extent, employed in technological applications, including telecommunication, memory devices, electronic devices, antenna and transformer cores [7].

The supreme structural, magnetic and other properties of nickel ferrite are determined by preparation methods and its occupancy at

tetrahedral and octahedral sites. In the literature, the properties of nickel ferrite are examined systematically by substituting  $Zn^{2+}$  [8],  $Cd^{2+}$  [9] in place of divalent metal ions and  $Al^{3+}$  [10],  $Bi^{3+}$  [11] in place of trivalent  $Fe^{3+}$  ions. A large number of methods have been employed to prepare nickel ferrite nanoparticle [12] such as solid-state reaction [13], hydro-thermal [14], decomposition [15], sol–gel [16] and co-precipitation [17]. Choosing an apt method is considered to be the indispensable facet to obtain ferrites of high quality. Therefore, chemical co-precipitation method is widely operated based on the ease and reproducibility, but it mainly leads to the precipitation of nano-crystals with a relatively broads.

It is of interest to note that investigation on the structural properties of  $Ni_{0.5}Ca_{0.5}Fe_2O_4$  nanoparticles have been reported earlier [18] but, the simultaneous detailed study of structural and magnetic properties of a series of Ni–Ca ferrite synthesized using co-precipitation method has not been reported. In the present paper, calcium substituted nickel ferrite is reported with a special view to studying the effect of substitution of non-magnetic Calcium ( $Ca^{2+}$ ) ion on the structural and magnetic behaviour. Magnetic measurements are also undertaken to probe into the variation of saturation magnetization with calcium concentration and gain a vital piece of information about the cation distribution.

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## 2. Experimental section

### 2.1. Chemicals used

Nickel chloride ( $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ), anhydrous ferric chloride ( $\text{FeCl}_3$ ), calcium chloride ( $\text{CaCl}_2$ ) and sodium hydroxide ( $\text{NaOH}$ ) were used as the initial materials. All the chemicals were purchased from Merck (AR Grade) with 99% purity.

### 2.2. Synthesis

0.5M Nickel chloride and 1M Ferric chloride were dissolved separately in 100 ml distilled water. 0.4N Sodium hydroxide (used as a precipitating agent) was added in 250 ml distilled water. 50 ml of Nickel chloride and 50 ml of Ferric chloride solutions were mixed with constant stirring and kept at  $60^\circ\text{C}$  for 30 min. Sodium hydroxide solution was mixed with the above-mentioned solution, till the pH value reached 12. The final solution was maintained at  $85^\circ\text{C}$  for 1 h for the transformation of hydroxides into spinel ferrite resulting in a brown colour precipitate. It was filtered and washed several times using deionised water. Collected particle was then dried at  $100^\circ\text{C}$ . The product was then milled in an agate mortar and kept for calcinations at  $800^\circ\text{C}$  for 4 h, to form pure nickel ferrite nanoparticles.

For the synthesis of Ca doped  $\text{NiFe}_2\text{O}_4$ , the stoichiometry amount of Calcium chloride was added to the aqueous solution of Nickel chloride and Ferric chloride and then the same procedure was followed. A generalized flow sheet for the synthesis of calcium doped nickel ferrite nanoparticles is shown in Figs. 1 and 2.

### 2.3. Techniques

From XRD (Panalytical X'Pert Powder X'Celerator Diffractometer), the crystallinity and phase of the samples were observed by using  $\text{Cu K}\alpha$  radiations ( $\lambda = 1.5406 \text{ \AA}$ ) in  $2\theta$  ranging from  $20^\circ$  to  $80^\circ$ . The surface morphology of all the samples was gathered with the support of the FESEM (Carl Zeiss SUPRA-55). EDX (Quantax 200 with X-Flash – Bruker) clearly identified the elements present in the nanoparticles. Fourier transform infrared spectrums (FTIR-Schimidzu) of all the samples were recorded in the range  $400 \text{ cm}^{-1}$  –  $4000 \text{ cm}^{-1}$ . The coercivity and saturation magnetization were studied by using vibrating sample magnetometer (Lakeshore 7410) up to 1.5 T at room temperature.

## 3. Results and discussion

### 3.1. Structural study

Fig. 3, shows the typical XRD patterns of the  $\text{Ni}_{1-x}\text{Ca}_x\text{Fe}_2\text{O}_4$  ( $x = 0$  to 1) nanoparticles. It reveals the single cubic spinel phase with reflection planes (220), (311), (400), (422), (440), (511) and (533). The extra reflection peaks at  $32^\circ$  and  $50^\circ$  indicate that  $\text{Fe}_2\text{O}_3$  are present in the samples. Shobana [18] has also observed  $\text{Fe}_2\text{O}_3$  impurity phase in Ca-Ni ferrite powders synthesized by sol-gel method. Lattice constant, average grain size and x-ray density are calculated by using standard formulas [19] and summarized in Table 1. The average particle size and the lattice constant increase with increasing Ca content ( $x$  from 0 to 1) primarily because the calcium ionic radius ( $0.99 \text{ \AA}$ ) is relatively higher than that of the nickel ionic radius ( $0.69 \text{ \AA}$ ). But x-ray density decreases with increasing Calcium, because the density and atomic weight of  $\text{Ca}^{2+}$  ( $1.54$  and  $40.07 \text{ gm/cm}^3$ ), are lower than  $\text{Ni}^{2+}$  ( $8.90$  and  $58.69 \text{ gm/cm}^3$ ) and  $\text{Fe}^{3+}$  ( $7.86$  and  $55.85 \text{ gm/cm}^3$ ) and it's inversely proportional to  $a^3$  [20,21].

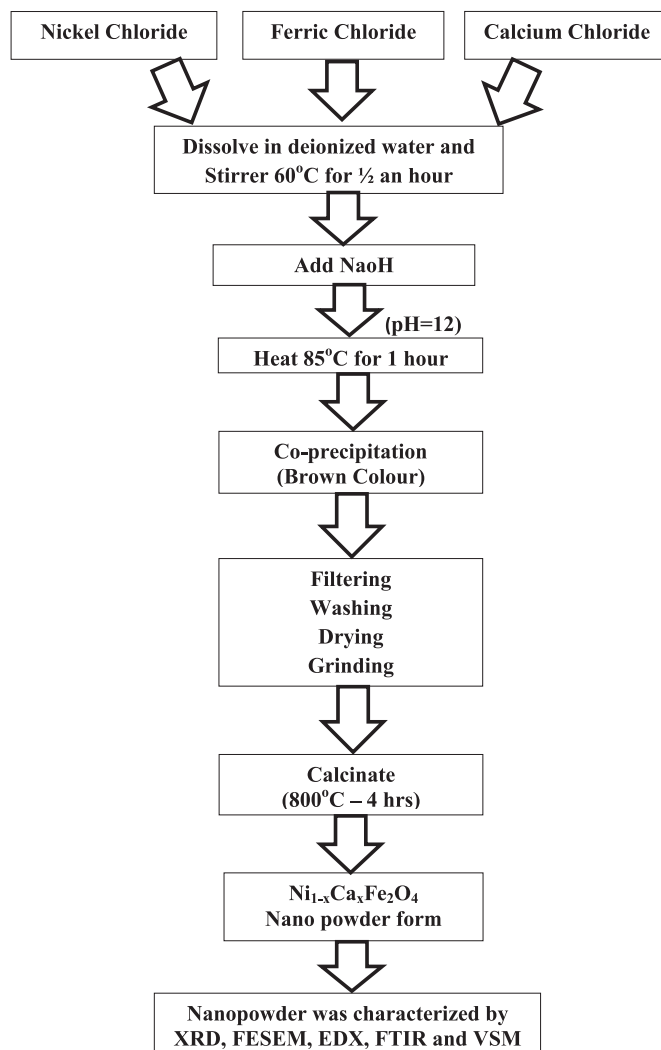
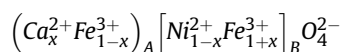


Fig. 1. Flowchart of the co-precipitation synthesis of  $\text{Ni}_{1-x}\text{Ca}_x\text{Fe}_2\text{O}_4$  nanoparticles.

The cation distribution in Ni-Ca ferrite can be represented by Ref. [22],



The presence of  $\text{Ni}^{2+}$  ion in the Ni-Ca ferrite enhances the exchange reaction  $\text{Ni}^{2+} + \text{Fe}^{3+} \leftrightarrow \text{Ni}^{3+} + \text{Fe}^{2+}$  in octahedral sites, while calcium is preferentially occupied by tetrahedral sites. This exchange interaction supports the electronic conduction mechanism in Ni-Ca ferrite; the octahedral sites get augmented when they are occupied by the  $\text{Fe}^{2+}$  ions instead of  $\text{Ni}^{2+}$  ions. The mean ionic radius of the tetrahedral A site  $r_A$  and octahedral B site  $r_B$  can be calculated by an accepted cation distribution using equation [22]. The calculated values of  $r_A$  and  $r_B$  are summarized in Table 2. The increase in  $r_A$  is due to the increasing amount of larger  $\text{Ca}^{2+}$  ions ( $0.99 \text{ \AA}$ ) to A-site which decreases the smaller  $\text{Fe}^{3+}$  ions ( $0.64 \text{ \AA}$ ) of A-site.

The oxygen positional parameter ( $u$ ) can be calculated using the value of lattice constant, the radius of oxygen ion ( $R_o = 1.32 \text{ \AA}$ ) and  $r_A$  [23]. The oxygen positional parameter ( $u$ ) increases from  $0.387 \text{ \AA}$  to  $0.408 \text{ \AA}$ . The oxygen positional parameter value rests upon the chemical composition and preparative conditions. In an ideal FCC structure,  $u = 3/8 = 0.375$  [24], the arrangements of oxygen ions

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