



# Microwave combustion synthesis of zinc substituted nanocrystalline spinel cobalt ferrite: Structural and magnetic studies



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## ABSTRACT

Zinc doped cobalt ferrite spinel nanoparticles were prepared by the microwave combustion method. All the samples were characterized by using X-ray diffraction technique (XRD), Scanning Electron Microscopy, energy dispersive X-ray analysis, UV–visible diffuse reflectance spectroscopy, Fourier transformed infrared (FT-IR) spectroscopy and vibrating sample magnetometry. The XRD patterns confirmed the formation of single phase  $\text{CoFe}_2\text{O}_4$  inverse spinel structure without impurities. The lattice parameter increased from 8.380 to 8.396 Å with increasing  $\text{Zn}^{2+}$  fraction. The average crystallite sizes obtained by a Scherrer method varied between 46.22 nm and 30.79 nm. The estimated band gap energy values increases with an increasing zinc fraction (1.88–2.10 eV). The elemental composition of Zn, Co, and Fe was qualitatively obtained from energy dispersive X-ray (EDX) analysis.

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## 1. Introduction

Nanostructured materials that are finding wider applications are prepared using bottom-up approach in the past few years. The spinel ferrite structure  $\text{MFe}_2\text{O}_4$ , where M refers to the metal, can be described as a cubic close-packed arrangement of oxygen atoms, with  $\text{M}^{2+}$  and  $\text{Fe}^{3+}$  at two different crystallographic sites. These sites have tetrahedral and octahedral oxygen coordination termed as A and B-sites, respectively [1,2]. The spinel crystal structure has  $\text{A}^{2+}$  cations at Wyckoff positions 8a (1/8, 1/8, 1/8),  $\text{B}^{3+}$  cations at

16d (1/2, 1/2, 1/2), and oxygen at 32e (u, u, u), where the positional parameter of oxygen is conventionally called as u-parameter. There are three ideal structures namely normal, inverse and mixed spinel structures. Normal spinel structure, where all  $\text{M}^{2+}$  ions occupy A-sites (tetrahedral site) and  $\text{Fe}^{3+}$  ions occupy B-sites (octahedral site); structural formula of such ferrites is  $\text{M}^{2+}[\text{Fe}_2^{3+}]_4\text{O}_4^{2-}$ . Inverse spinel structure, where all  $\text{M}^{2+}$  ions are in B-positions and  $\text{Fe}^{3+}$  ions are equally distributed between A and B-sites, structural formula of these ferrites are  $\text{Fe}^{3+}[\text{M}^{2+}\text{Fe}^{3+}]_4\text{O}_4^{2-}$ . Mixed spinel structure, when cations  $\text{M}^{2+}$  and  $\text{Fe}^{3+}$  occupy both A and B-positions; structural formula of this ferrite is  $\text{M}_{1-\delta}^{2+}\text{Fe}_\delta^{3+}[\text{M}_\delta^{2+}\text{Fe}_{2-\delta}^{3+}]\text{O}_4^{2-}$ , where  $\delta$  is the degree of inversion [3,4].

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Among the ferrites, cobalt ferrite ( $\text{CoFe}_2\text{O}_4$ ) is a well known inverse spinel structure, because of its high coercivity, moderate saturation magnetization, large magnetocrystalline anisotropy, large magnetostrictive coefficient at room temperature [5]. The mechanical hardness, chemical stability, flexible magnetic properties makes these materials a promising one for magneto-optical recording, electronic devices, high density magnetic recording devices [6,7], supercapacitors, photo-magnetic devices, photocatalysis and hydrogen production [8–11]. Various preparation methods like microemulsions [12], solvo-thermal, hydrothermal approach [13], sol-gel method [14], combustion, laser deposition, thermal decomposition method [15], chemical co-precipitation technique etc., [16] are reported for the synthesis of cobalt ferrites. Among the various methods reported, microwave combustion technique is probably opted for homogeneity, high purity and improved characteristics. The microwave energy is an internal means of heat energy generation and conversion. The microwave energy is transformed into heat energy by strong intermolecular friction and rises the temperature of the precursor materials suddenly [17]. Fuels like urea, glycine, citric acid, L-alanine or carbonylhydrazide in an appropriate stoichiometric ratio controls the combustion process in accordance with the propellant chemistry principles [22]. As a result, the crystallite size, morphology, textural, surface area, and other physico-chemical properties are largely altered. Also, the use of microwave energy as heating source, speeds up the chemical reaction and kinetics, improve the economical viability, and reduces the energy loss [18].

In the present work, we have synthesized zinc substituted cobalt ferrite nanoparticles by the microwave combustion method employing L-arginine as a fuel. L-arginine is found to facilitate the homogeneous mixing of cations in solution and it undergoes decomposition and promotes combustion. The literature on the preparation of zinc ferrite using L-arginine is found to be scarce. We have investigated the effect of this fuel to evaluate the characteristics like surface morphology, size distribution, and magnetic properties of pure and zinc substituted cobalt ferrite nanoparticles. The ferrites thus prepared were characterized by X-ray diffraction (XRD) for the structural analysis; scanning electron microscopy (HR-SEM) for surface morphology; energy dispersive X-ray (EDX) analysis for chemical composition; diffuse reflectance spectroscopy (DRS) for energy band gap estimation; and Fourier transform infrared (FT-IR) spectral studies for vibrational stretching frequencies. The magnetic behavior of the samples was studied using vibrating sample magnetometry (VSM).

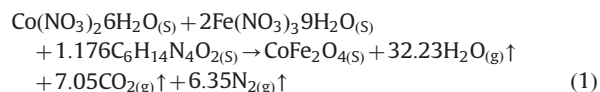
## 2. Experimental

### 2.1. Materials

All the chemicals used in the present study, such as, zinc nitrate, cobalt nitrate, ferric nitrate and L-arginine were of analytical grade, purchased from Merck, India. The chemicals were used as received without further purification. Double distilled water was used during the sample preparation.

### 2.2. Preparation of Zn doped $\text{CoFe}_2\text{O}_4$ spinels

The mole percentage of  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  with  $x$  fraction varying as 0, 0.1, 0.2, 0.3, 0.4, and 0.5 was prepared by dissolving stoichiometric quantities of precursors like zinc nitrate, cobalt nitrate, ferric nitrate and L-arginine in double distilled water. The precursor solution in a beaker was stirred for about 1 h at room temperature to obtain a clear homogeneous solution. L-arginine served as a fuel, while the nitrates in the precursors served as the oxidizers. The fuel to oxidizer ratio (F/O) was taken to be 1 as per the concept of propellant chemistry. The homogeneous reaction solution mixture after rigorous stirring were transferred into silica crucible and placed inside a microwave oven (SAMSUNG, India Limited) for irradiation. Microwave was irradiated over the precursor solution for 10 min at 900 W output power at 2.54 GHz frequency. Primarily, the solution boiled and underwent rigorous decomposition with gas evolution. When the solution reached the point of spontaneous combustion, ignition took place resulting in a rapid flame and yielding a solid fluffy final products of  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ( $x=0-0.5$ ). The samples  $\text{CoFe}_2\text{O}_4$ ,  $\text{Co}_{0.9}\text{Zn}_{0.1}\text{Fe}_2\text{O}_4$ ,  $\text{Co}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$ ,  $\text{Co}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4$ ,  $\text{Co}_{0.6}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$ ,  $\text{Co}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ , were labeled as CoF1, CoZF2, CoZF3, CoZF4, CoZF5, and CoZF6 respectively. The chemical reaction involved in the formation of cobalt ferrite during the combustion process employing L-arginine is given in Eq. (1)



### 2.3. Characterization techniques

The crystalline structure of the resultant nanoparticles was performed using Rigaku Ultima IV high resolution X-ray powder diffractometer for  $2\theta$  values ranging from  $10^\circ$  to  $80^\circ$  using  $\text{CuK}\alpha$  radiation at  $\lambda=1.5418 \text{ \AA}$ . The surface morphology of the samples were analyzed at a desired magnification with a Joel JSM 6360 high resolution scanning electron microscope (HR-SEM) equipped with energy dispersive X-ray (EDX) analyzer for elemental composition analysis. The diffuse reflectance spectrum (DRS) in the UV-visible range was recorded using Thermo scientific evolution 300 UV-visible spectrophotometer to estimate the optical band gap. The FT-IR spectrum was recorded on a Thermo scientific Nicolet iS10 infrared spectrophotometer. The vibrating sample magnetometry measurements were carried out using Lakeshore VSM 7410 model equipped with 3 T magnets at 300 K.

## 3. Results and discussion

### 3.1. XRD analysis

The crystal structure and phase purity of pure and Zn doped  $\text{CoFe}_2\text{O}_4$  samples were confirmed from the X-ray diffraction patterns as shown in Fig. 1(a)–(f). The diffraction peaks at  $2\theta$  values of  $18.27^\circ$ ,  $30.05^\circ$ ,  $35.43^\circ$ ,  $37.01^\circ$ ,  $43.02^\circ$ ,  $56.95^\circ$  and  $62.48^\circ$  are indexed to (111), (220), (311),

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