



# Fabrication, characterization and magnetic behaviour of alumina-doped zinc ferrite nano-particles

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## ABSTRACT

Zinc ferrite nano-powders with a nominal composition of ZnFe<sub>2</sub>O<sub>4</sub> were prepared by combustion synthesis using mixture of urea and ammonium nitrate as fuel. The influence of alumina-doping on the structural, morphological and magnetic properties of ZnFe<sub>2</sub>O<sub>4</sub> nano-particles was investigated by means of X-ray powder diffraction (XRD), infrared (IR) spectroscopy, scanning and transmission electron microscopy (SEM and TEM) and vibrating sample magnetometer (VSM). XRD and IR analyses confirm the cubic spinel phase of ZnFe<sub>2</sub>O<sub>4</sub> nano-particles. The Zn ferrite presented a uniform microstructure with grain size in nano-scale. Alumina-doping brought about a change in the morphology of the as prepared ferrite from sphere-like to regular hexagon. Al<sub>2</sub>O<sub>3</sub>-treatment led to a decrease in the coercivity ( $H_c$ ), magnetization ( $M_s$ ) and magnetic moment ( $n_B$ ) of the investigated system. The maximum decrease in the values of  $H_c$ ,  $M_s$  and  $n_B$  due to the treatment with 1.5 wt% Al<sub>2</sub>O<sub>3</sub> attained 13.5, 17.4 and 13.5%, respectively. The observed results can be explained on the basis of particle size and the Fe<sup>3+</sup> concentration in the octahedral and tetrahedral sites involved in the cubic spinel structure.

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## 1. Introduction

Transition metal ferrites (MFe<sub>2</sub>O<sub>4</sub>) are magnetic materials with cubic spinel structure which have been extensively used in various technological applications such as electronic ignition systems, generators, vending machines, medical implants, wrist watches, inductor core, transformer circuits, magnetic sensors and recording equipment, telecommunications, magnetic fluids, microwave absorbers and other high frequency applications [1–5]. Distinct merit of ferrites over other magnetic materials such as iron and metallic alloys is their high electrical resistivity due to which ferrites perform much better at high frequencies. High electrical resistivity prevents induction of eddy currents and resultant loss of energy. High permeability and temperature stability are other advantages. In addition, ferrites are much cheaper than most of other magnetic materials, and their magnetic and mechanical properties can be “tailor made” per the requirement of application. Among different metal ferrites, the zinc ferrite is of special interest. It has a wide range of applications such as gas sensor, semiconductor photo-catalyst, photoelectron chemical cells and H<sub>2</sub> production, and also as heterogeneous catalyst for oxidation reactions [6–8].

It is a well-known fact that the properties of ferrite materials are strongly influenced by the materials' composition and microstructure, which are sensitive to the preparation methodology used in

their synthesis [9]. In addition, the sintering conditions employed and the impurity levels present in or added to these materials also change their properties [10]. The selection of an appropriate process is, therefore, the key to obtain good quality ferrites. These ferrites are usually prepared by the conventional ceramic method, in which the stoichiometric composition and final microstructure are extremely difficult to control. This method requires prolonged heating at high temperatures during preparation, which may cause some of the constituents to evaporate, thereby modifying the desired stoichiometry. Moreover, in zinc ferrites, the volatilization of zinc at high temperatures results in the formation of Fe<sup>2+</sup> ions, which increase electron hopping and reduce resistivity.

Various preparation techniques have been developed to produce ZnFe<sub>2</sub>O<sub>4</sub> nano-particles, such as co-precipitation [11–13], micro-emulsion [14], sol-gel processing [15], hydrothermal synthesis [16,17], thermal plasma synthesis [18] and high energy ball milling [19]. In the recent past, combustion route is also emerging as a potential alternative for the synthesis of ferrites, in specific, of nano-dimensions. The combustion technique, which is employed for propellants and explosives, involves a mixture of oxidizing reagents (such as nitrates) and a fuel (such as mixture of urea and ammonium nitrate), which acts as a reducing reagent. The method is actually self-sustainable after the reaction has initiated and, owing to the exothermic characteristic of the reaction, high temperatures ensure the crystallization and formation of oxides in a short period of time. These conditions normally prevent the instantaneous formation of particle agglomeration [20]. In addition, the advantages of this unconventional process are high purity, chem-

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ical homogeneity on an atomic scale, small uniform particle sizes and controlled particle shapes.

The present work is focused on the effects of alumina-doping on the structural, morphological and magnetic properties of zinc ferrite obtained via combustion route. The X-ray powder diffraction patterns, the microstructure and the magnetic properties are discussed as a function of the dopant content.

## 2. Experimental

### 2.1. Materials

Various alumina-doped ZnFe<sub>2</sub>O<sub>4</sub> samples were prepared by mixing calculated proportions of zinc and iron nitrates, mixture of urea and ammonium nitrate and different amounts of aluminium nitrate. The mixed precursors were concentrated in a porcelain crucible on a hot plate at 250 °C for 5 min. The crystal water was gradually vaporized during heating and when a crucible temperature was reached, a great deal of foams produced and spark appeared at one corner which spread through the mass, yielding a voluminous and fluffy product in the container. In our experiments, the ratio of the mixture urea and ammonium nitrate: zinc and ferric nitrates was 1.33. The concentrations of aluminium expressed as wt% Al<sub>2</sub>O<sub>3</sub> were 0.75 and 1.5. The chemicals employed in the present work were of analytical grade supplied by Fluka Company.

### 2.2. Techniques

An X-ray measurement of various mixed solids was carried out using a BRUKER D8 advance diffractometer. The patterns were run with Cu K $\alpha$  radiation at 40 kV and 40 mA with scanning speed in 2 $\theta$  of 2° min<sup>-1</sup>.

The crystallite size of Zn-ferrite present in the investigated solids was based on X-ray diffraction line broadening and calculated by using Scherrer equation [21].

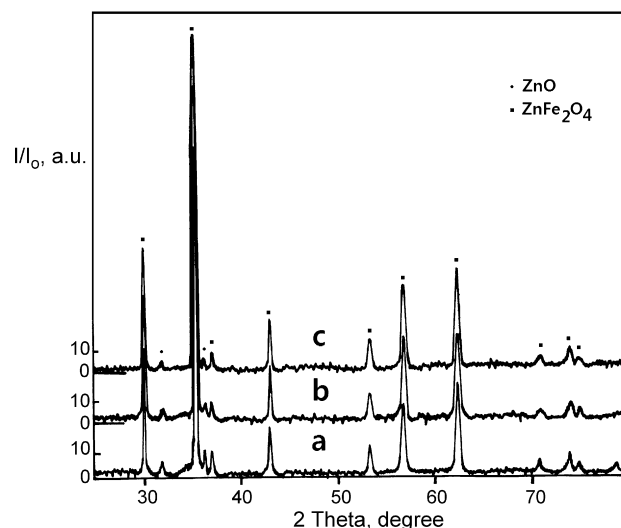
$$d = \frac{B\lambda}{\beta \cos \theta} \quad (1)$$

where  $d$  is the average crystallite size of the phase under investigation,  $B$  is the Scherrer constant (0.89),  $\lambda$  is the wave length of X-ray beam used,  $\beta$  is the full-width half maximum (FWHM) of diffraction and  $\theta$  is the Bragg's angle.

An infrared transmission spectrum of various solids was determined using Perkin-Elmer Spectrophotometer (type 1430). The IR spectra were determined from 900 to 300 cm<sup>-1</sup>. Two milligrams of each solid sample were mixed with 200 mg of vacuum-dried IR-grade KBr. The mixture was dispersed by grinding for 3 min in a vibratory ball mill and placed in a steel die 13 mm in diameter and subjected to a pressure of 12 tonnes. The sample disks were placed in the holder of the double grating IR spectrometer.

Scanning electron micrographs (SEM) and transmission electron micrographs (TEM) were recorded on JEOL JAX-840A and JEOL JEM-1230 electron micro-analyzers, respectively. The samples were dispersed in ethanol and then treated ultrasonically in order to disperse individual particles over a gold grid.

The magnetic properties of the investigated solids were measured at room temperature using a vibrating sample magnetometer (VSM; 9600-1 LDJ, USA) in a maximum applied field of 15 kOe. From the obtained hysteresis loops, the saturation magnetization ( $M_s$ ), remanence magnetization ( $M_r$ ) and coercivity ( $H_c$ ) were determined.



**Fig. 1.** X-ray diffraction patterns of ZnFe<sub>2</sub>O<sub>4</sub> sample treated with different amounts of aluminium oxide; (a) 0 wt% Al<sub>2</sub>O<sub>3</sub>, (b) 0.75 wt% Al<sub>2</sub>O<sub>3</sub> and (c) 1.5 wt% Al<sub>2</sub>O<sub>3</sub>.

**Table 1**

The effect of alumina-doping on the height of some lines at 'd' spacing of 2.54 Å (100% ZnFe<sub>2</sub>O<sub>4</sub>), 2.49 Å (100% ZnO) and 1.48 Å (49% ZnO + 60% ZnFe<sub>2</sub>O<sub>4</sub>) involved in XRD pattern of the investigated system.

Concentration of Al <sub>2</sub> O <sub>3</sub> (wt%)	Peak height (a.u.)			Ratio between b/a (R)
	2.54 Å	2.49 Å (a)	1.48 Å (b)	
0.00	129	10	36	3.60
0.75	151	6	38	6.33
1.50	160	4	45	11.25

## 3. Results and discussion

### 3.1. Structural analysis

The X-ray diffractograms of pure and alumina specimens of ZnFe<sub>2</sub>O<sub>4</sub> nano-particles fabricated by the combustion technique are depicted in Fig. 1. The characteristic peaks of ZnFe<sub>2</sub>O<sub>4</sub> (Franklinite) are determined in the investigated diffractograms. It was found that for various samples, the phase spinel structure is formed. Obvious trace amounts of zinc oxide as a second phase can be detected in the pure and doped sample. However, the increase in the amount of aluminium oxide led to an increase in the crystallinity of the zinc ferrite powders. This observation is assured by decreasing in both the broadening of the peaks in the diffraction patterns with subsequent an increase in the peak height of zinc ferrite phase as shown in Fig. 1 and Table 1. These findings indicate that the zinc ferrite formation has increased with increase of Al<sub>2</sub>O<sub>3</sub> added. In other words, Al<sub>2</sub>O<sub>3</sub> doping enhances larger motion force for the formation of the ferrite phase via the conversion of most un-reacted oxides with subsequent increase the number of Fe<sup>3+</sup> ions involved in the spinel structure via converting Fe<sup>2+</sup> ions that may be present in Fe<sub>2</sub>O<sub>3</sub> crystals [22].

The enhancement effects of the treatment with different amounts of Al<sub>2</sub>O<sub>3</sub> on zinc ferrite formation are better investigated by measuring the height of certain diffraction lines characteristic for ZnO (2.47 Å, 100%) and relative to one the common lines of both ZnO (1.48 Å, 49%) and ZnFe<sub>2</sub>O<sub>4</sub> (1.48 Å, 60%). This was done and the results obtained are given in Table 1 depending upon all diffraction lines of zinc ferrite phase are sharp and only their relative intensity ( $I/I_0$ ) alter by changing the extent of Al<sub>2</sub>O<sub>3</sub> added to the investigated solids [23]. Inspection of Table 1 revealed that: (i) the ratio,  $R$ , between the peak height of the lines at "d" spacing of 1.48 and

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