



Synthesis and magnetic properties of $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrites using egg-white method

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ABSTRACT

A simple and low cost effective method involving egg white (ovalbumin) is used to synthesize the $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ nanoparticles. The structural and magnetic properties of the synthesized samples were studied as a function of Al content x . The formation of crystal phase was identified by the X-ray diffraction method. All the samples confirm the formation of single phase cubic spinel structure and lattice constant that were found to decrease with the increase in Al content x . The morphology and particle size were examined by scanning electron microscopy. We report the synthesis of nanoparticle with crystallite size ranging from 25 to 33 nm. A vibrating sample magnetometer was used to obtain the hysteresis parameters. The saturation magnetization and magneton number decreased with the substitution of Al ions. The Curie temperature was determined from a.c. susceptibility plots and it is found to decrease with Al substitution.

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1. Introduction

Magnetic nanoparticles are gaining much more importance and attention in the current era because of their distinct fundamental magnetic phenomena as compared to their bulk counterpart. In addition to the study of fundamental magnetic phenomena, nanomagnets may in the future form the basis of emerging technologies, such as in ultra-high density data storage media, ultra-strong permanent magnets, and biological and chemical sensing [1]. Particles with nanosize exhibit unique chemical and physical properties [2,3]. In particular, nanocomposite materials composed of nanometric metal and metal oxide particles embedded in vitreous matrices reveal a variety of interesting magnetic, electric and catalytic properties [4–6]. Ferrites are ferrimagnetic semiconductors that opened a new area in the physics of material science and the need for high resistivity ferrites led to the synthesis of the various ferrites. The electrical and magnetic properties of ferrites depend on the method of preparation [7,8], site preference [9] and valence distribution [10]. Magnetic properties of magnetic nanomaterials particularly in ferrites materials also depend on their chemical composition and method of synthesis [11]. Different synthesis methods have been suggested for the preparation of

nanophase materials namely; vapor deposition [12], ball milling technique [13], reversed micelles [14], Langmuir–Blodgett film [15], self-assembled monolayers [16] and sol–gel process [17,18]. In addition, the egg white method has also been used for the preparation of ferrite nanomaterials [19,20].

The substitution effect and the change of the preparation condition are allowed to improve the properties of ferrites. Generally, Cu–Zn ferrites were commercially used in radio frequency circuits, transformer cores, antennas and read/write heads for high-speed digital tape and in high quality filters [21,22]. A considerable amount of work has been carried out on Cu–Zn ferrite substituted with Al ions for different ratios and compositions of Cu and Zn ions [23,24].

In this work, we have systematically investigated the effect of Al^{3+} substitution on the structural and magnetic properties of $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4$. The egg white method is used to synthesize the nanoparticles of $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$. The structural and magnetic properties of the synthesized samples have been discussed in the content.

2. Experimental details

A ferrite series using a chemical formula $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ with $x=0.0$ –1.0 in a step of $x=0.2$ was synthesized by the egg white method. In this method $\text{Cu}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Al}(\text{NO}_3)_3 \cdot 7\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and freshly extracted egg white

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(ovalbumin) were used as starting materials. Initially, 60 ml of egg white was mixed with 40 ml distilled water and continuous stirring was carried out at room temperature until the solution becomes homogenous. The stoichiometry proportion of the metal nitrate was then added slowly one by one into the egg white solution. The entire solution was then stirred at room temperature for 2 h for homogenous mixing. The mixed solution was evaporated at 80 °C for 4 h until dried precursor is formed. The dried precursor was then finally calcined in the furnace at 600 °C for 4 h in air atmosphere. The samples were configured for X-ray investigation by a Philips X-ray diffractometer (Model PW 3710) using CuK α radiation. The surface morphology of sintered powder was characterized by scanning electron microscopy (SEM) (Model JOEL-JSM 840). A vibrating sample magnetometer (VSM) was used to obtain the hysteresis parameters. The a.c. susceptibility measurements were carried out at room temperature using a double coil setup. The temperature of the sample was measured by using a platinum–platinum–rhodium thermocouple. The powdered sample was gradually heated and signals corresponding to the magnetic moment were measured at different temperatures. The measurement was carried out from room temperature up to the Curie temperature (T_C).

3. Results and discussion

Fig. 1 shows the X-ray diffraction (XRD) patterns of the typical samples of a $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ferrite system. XRD patterns show well developed diffraction line assigned to the pure spinel phase. All major XRD peaks of $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ match well with the standard pattern of spinel ferrite. All the peaks of the XRD patterns were indexed using Bragg's law. The lattice parameter (a) was calculated using the following relation:

$$a = \frac{\lambda(h^2 + k^2 + l^2)^{1/2}}{2 \sin \theta} \quad (1)$$

where (hkl) is the Miller indices, λ is the wavelength of X-ray radiation and θ is the Bragg angle. The values of lattice parameter obtained from the X-ray diffraction data are given in Table 1. The lattice parameter is very sensitive to the substitution of Al^{3+} ions. It is observed from Table 1 that the lattice parameter decreased with the increase in Al^{3+} substitution. The decrease in lattice parameter is attributed to the difference in the ionic radii between Al^{3+} and Fe^{3+} ions. In the present ferrite series $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$, larger Fe^{3+} ions (0.67 Å) are replaced by smaller Al^{3+} ions (0.51 Å) and hence it is observed that lattice parameter decreased

Table 1

Lattice parameter (a), X-ray density (d_x) and crystallite diameter (D_{311}) for $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$.

Comp. 'x'	a (Å)	d_x (g/cm ³)	D_{311} (nm)
0.0	8.362	5.438	25
0.2	8.343	5.344	28
0.4	8.325	5.246	29
0.6	8.314	5.132	30
0.8	8.304	5.017	32
1.0	8.295	4.898	33

with the increase in Al^{3+} substitution. Similar variation is also observed for Al^{3+} substituted nickel ferrite [25]. The X-ray density (d_x) of all the samples of the $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ series was determined by the following relation:

$$d_x = \frac{ZM}{NV} \quad (2)$$

where Z is the number of molecules per unit cell, M is the molecular weight of sample, N is the Avogadro's number and V is the volume of the unit cell. The values of X-ray density are presented in Table 1. It can be seen from Table 1 that like lattice parameter, X-ray density also decreased with the increase in Al^{3+} substitution. The decrease in X-ray density is attributed to the decrease in mass that overtakes the decrease in volume of the unit cell.

The average crystallite diameter (D_{311}) of the powder samples was determined from the (311) XRD peak and by using the following relation:

$$D_{311} = \frac{C\lambda}{\beta_{1/2} \cos \theta} \quad (3)$$

where $\beta_{1/2}$ is the full width at half maximum in (2θ), θ is the corresponding Bragg angle and $C=0.9$. The values of the crystallite diameter are presented in Table 1. The values of the crystallite diameter are ranged between 25 and 33 nm. The microstructure of the calcined samples was examined by SEM. Typical SEM images for $x=0.2$ and $x=0.4$ samples are shown in Fig. 2. Surface morphology of the samples indicating that the fine crystals show the tendency of agglomeration, at the same time relatively dense microstructure with well-developed grains along with few pores, is also observed.

In spinel ferrite cations are distributed among the tetrahedral (A) and octahedral [B] sites. The distribution of cations depends on the various parameters such as the method of preparation, sintering temperature, sintering atmosphere and amount of dopant. The cation distribution can be estimated from the X-ray diffraction, Mössbauer analysis and magnetic moment [26]. In the present work, cation distribution of $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ was estimated by using the X-ray diffraction method. Bertaut [27] and Furuhashi et al. [28] obtained cation distribution in spinel ferrite based on the comparison between the diffraction intensities observed experimentally and those calculated for a large number of hypothetical crystal structures. The best information on cation distribution is achieved by comparing experimental and calculated intensity ratios for reflections whose intensities are

- nearly independent of the oxygen parameter,
- vary with the cation distribution in the opposite way, and
- does not differ significantly.

The most suitable reflections for estimating the cation distributions are (220), (400) and (440). The basic equation used to calculate the intensity of the reflection due to the plane (hkl) is

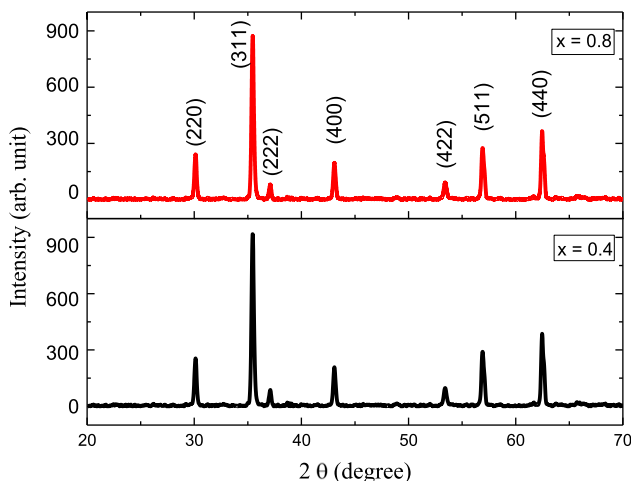


Fig. 1. X-ray diffraction patterns of the typical samples, $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_{0.4}\text{Fe}_{1.6}\text{O}_4$ ($x=0.4$) and $\text{Cu}_{0.7}\text{Zn}_{0.3}\text{Al}_{0.8}\text{Fe}_{1.2}\text{O}_4$ ($x=0.8$).

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