Research Article

Synthesis and Characterization of Lithium-Substituted Cu-Mn Ferrite Nanoparticles

M. A. Mohshin Quraishi¹ and M. H. R. Khan²

¹ Department of Physics, Manarat Dhaka International College, Dhaka 1212, Bangladesh

² Department of Arts and Sciences, Ahsanullah University of Science and Technology, Dhaka 1208, Bangladesh

Correspondence should be addressed to M. A. Mohshin Quraishi; m.phys@yahoo.com

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The effect of Li substitution on the structural and magnetic properties of $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ($x = 0.00, 0.10, 0.20, 0.30, 0.40$, and 0.44) ferrite nanoparticles prepared by combustion technique has been investigated. Structural and surface morphology have been studied by X-ray diffractometer (XRD) and high-resolution optical microscope, respectively. The observed particle size of various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ is found to be in the range of 9 nm to 30 nm. XRD result confirms single-phase spinel structure for each composition. The lattice constant increases with increasing Li content. The bulk density shows a decreasing trend with Li substitution. The real part of initial permeability (μ'_i) and the grain size (D) increase with increasing Li content. It has been observed that the higher the μ'_i is, the lower the resonance frequency in $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites is.

1. Introduction

Ferrite nanoparticles have attracted a growing interest due to their potential applications such as magnetic recording [1], storage [2], and biotechnology [3]. In the most recent years, the interest in the use of nanoparticles in biomedical applications has greatly increased [4, 5]. The size and composition of nanoparticles influence the bio-application of the magnetic nanoparticles [6]. It is well known that the physical and chemical properties of the nanosized magnetic materials are quite different from those of the bulk ones due to their surface effect and quantum confinement effects. These nanoparticles can be obtained through precipitation of metallic salts in different media as polymers [7], organic acid or alcohol [8], sugars [9], and so forth. In particular, sol-gel, autocombustion, thermal decomposition, hydrothermal, ball milling, reverse micelle synthesis, solid-phase reaction, thermally activated solid state reaction, and pulsed laser deposition have been developed to prepare the single-domain MnFe_2O_4 nanoparticles [10–23]. Manganese ferrite (MnFe_2O_4) nanoparticles have become very popular due to their wide range of magnetic applications, such as recording devices, drug delivery, ferrofluid, biosensors, and catalysis [10, 24–27]. Recently, Deraz and

Alarifi [28] have studied structural and magnetic properties of MnFe_2O_4 nanoparticles by combustion route. Till now, no other report has been found in the literature for Li-doped Cu-Mn ferrite. Lithium ferrites are low-cost materials which are attractive for microwave device applications. Hence, there has been a growing interest in Li-substituted Cu-Mn ferrite for microwave applications and high permeability with low magnetic loss. Therefore, this paper is devoted to study the effect of Li^+ substitution on the physical and magnetic properties of $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites prepared by combustion technique.

2. Experimental

2.1. Sample Preparation and Characterization. The $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites were prepared by autocombustion technique. The analytical grade of $\text{Li}(\text{NO}_3)_2$, $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$, $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, and $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ was taken as raw material and weighted according to the stoichiometric amount and then dissolved in ethanol. The mixture was placed in a magnetic heating stirrer at 80°C , followed by an ignition, the combustion takes place within a few seconds,

and fine nanosized powders were precipitated. These powders were crushed and ground thoroughly. The fine powders of the composition were then calcined at 900°C for 5 h for the final formation of $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites nanoparticles. Then, the fine powders were granulated using polyvinyl alcohol (PVA) as a binder and pressed uniaxially into disk-shaped (about 13 mm outer diameter, 1.5 mm–2.0 mm thickness) and toroid-shaped (about 13 mm outer diameter, about 6.5 mm inner diameter and 2 mm thickness) samples. The samples prepared from each composition were sintered at 1200°C for 1 hour in air. The temperature ranges for sintering was maintained at 5°C/min for heating and 10°C/min for cooling. All sintered samples were polished and thermal etching was performed. X-ray diffraction was carried out with an X-ray diffractometer (Model: D8 Advance, Bruker AXS) for each sample. For this purpose, monochromatic $\text{Cu-K}\alpha$ radiation was used. The lattice parameter for each peak of each sample was calculated by using the formula

$$a_0 = d \sqrt{h^2 + k^2 + l^2}, \quad (1)$$

where h , k , and l are the indices of the crystal planes. To determine the exact lattice parameter for each sample, Nelson-Riley method was used. The Nelson-Riley function $F(\theta)$ is given as

$$F(\theta) = \frac{1}{2} \left[\left(\frac{\cos^2 \theta}{\sin \theta} \right) + \left(\frac{\cos^2 \theta}{\theta} \right) \right]. \quad (2)$$

The values of lattice constant “ a ” of all the peaks for a sample are plotted against $F(\theta)$. Then, using a least-square fit method exact lattice parameter “ a_0 ” was determined. The point where the least-square fit straight line cuts the y -axis (i.e., at $F(\theta) = 0$ or $\theta = 90^\circ$) is the actual lattice parameter of the sample.

The physical or bulk densities ρ_B of the samples were determined by Archimedes principle with water medium using the following expression:

$$\rho_B = \frac{W\rho}{W - W'} \text{ g/cm}^3, \quad (3)$$

where W is the weight of the sample in air, W' is the weight of the sample in the water, and ρ is the density of water in room temperature.

The theoretical density ρ_{th} was calculated using the following expression:

$$\rho_{th} = \frac{8M}{N_A a_0^3} \text{ g/cm}^3, \quad (4)$$

where N_A is Avogadro's number ($6.02 \times 10^{23} \text{ mol}^{-1}$) and M is the molecular weight.

The optical micrographs for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites have been taken by using high-resolution optical microscope (Model: NMM-800TRF). Average grain sizes of all samples were determined from optical micrographs by linear intercept technique [29]. The frequency-dependent initial permeability for each sample was measured by using a Wayne Kerr Impedance Analyzer (Model: 6500B).

The complex permeability measurement on toroid-shaped samples was carried out at room temperature in frequency range 10 KHz–100 MHz. Both the μ'_i and μ''_i of the complex permeability were calculated using the following relations:

$$\mu'_i = \frac{L_s}{L_0}, \quad (5)$$

$$\mu''_i = \mu'_i \tan \delta,$$

where L_s is the self-inductance of the sample core and $L_0 = \mu_0 N^2 S / \pi \bar{d}$ is derived geometrically. Here, L_0 is the inductance of the winding coil without the sample core, N is the number of turns of the coil ($N = 5$), and S is the area of cross-section of the toroidal sample as follows:

$$S = d \times h, \quad (6)$$

where $d = (d_2 - d_1)/2$, d_1 = inner diameter, d_2 = outer diameter, h = Height and $\bar{d}$ is the mean diameter of the toroidal sample as follows:

$$\bar{d} = \frac{d_1 + d_2}{2}. \quad (7)$$

The Loss factor, $\tan \delta$, was determined from the ratio ($= \mu''_i / \mu'_i$).

3. Results and Discussion

3.1. X-Ray Diffraction Analysis. The XRD analysis was performed to verify the formation of spinel structure of various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites, in which Mn^{2+} is replaced with Li^+ and Fe^{3+} . The XRD patterns of these Li^+ -substituted $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ (with $x = 0.00, 0.10, 0.20, 0.30, 0.40$, and 0.44) ferrites sintered at 1200°C in air for 1 h are shown in Figure 1. The patterns indicated that these materials have a well-defined single crystalline phase and formation of cubic spinel structure for each composition. Analyzing the XRD patterns, it is observed that the positions of the peaks comply with the reported value [30] and some traces of raw materials were found for $x = 0.00$, $x = 0.10$ and $x = 0.20$ and $x = 0.30$.

3.2. Lattice Constant. The values of lattice constant obtained from each plane are plotted against Nelson-Riley function [31]. The values of lattice constant were estimated from the extrapolation of these lines to $F(\theta) = 0$ or $\theta = 90^\circ$. It is noticed from Figure 2 that a_0 increases with the increase of Li^+ content in $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ (with $x = 0.00, 0.10, 0.20, 0.30, 0.40$, and 0.44) ferrites. Values of a_0 for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites are presented in Table 2. The increase in a_0 with Li content indicates that the present system obeys Vegard's law [32]. This increase of a_0 can be attributed to the ionic size. The ionic radius of Li^+ (0.76 Å) is greater than that of Mn^{2+} (0.67 Å) [29, 33]. When the larger Li^+ and Fe^{3+} ions enter the lattice, the unit cell expands while preserving the overall cubic symmetry.

FIGURE 1: The X-ray diffraction patterns for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ sintered at 1200°C .

3.3. Average Particle Size. The average particle size was estimated by using Debye-Scherrer [34] formula from the broadening of the highest intensity peaks (311) of XRD patterns:

$$D = \frac{0.9\lambda}{\beta \cos \theta}, \quad (8)$$

where D is the average particle size, λ is the wavelength of the radiation used as the primary beam of $\text{Cu-K}\alpha$ ($\lambda = 1.54178 \text{ \AA}$), θ is the angle of the incident beam in degree, and β is the full width at half maximum (FWHM) of the fundamental reflection (311) in radian of the FCC ferrites phase. Debye-Scherrer formula assumes approximation and gives the average particle size if the grain size distribution is narrow and strain-induced effects are quite negligible.

Figure 3 shows the XRD patterns of $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites sintered at 1200°C for 1 h, where (311) peak is shown in expanded form to understand the variation of FWHM of the Bragg peaks with the Li content. From Figure 3, it is seen that the value of FWHM decreases with the increase of lithium content. The particle size of the sample is inversely proportional to FWHM according to Debye-Scherrer formula. The observed particle size is in the range from 9 to 30 nm which has been listed in Table 1.

3.4. Theoretical and Bulk Density. The values of ρ_{th} and ρ_B for the various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites (with $x = 0.00, 0.10, 0.20, 0.30, 0.40$, and 0.44) are tabulated in Table 2. It is noticed from Figure 4 that both ρ_{th} and ρ_B decrease with the increase of Li substitution in $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites for constant sintering temperature. This phenomenon could be explained in terms of the atomic weight. The atomic weight of Mn (54.94 amu) is greater than that of combined atomic weight of the Li (6.941 amu) and Fe (55.845 amu) [33].

3.5. Microstructure. The optical micrographs of $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites (where $x = 0.00, x = 0.10, x = 0.20, x = 0.30, x = 0.40$, and $x = 0.44$) are shown in Figure 5 sintered

FIGURE 2: Variation of lattice constant and r -variant with Li content for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ sintered at 1200°C .

FIGURE 3: XRD patterns of (311) peak with Li content, for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ nanoparticles.

FIGURE 4: The variation of theoretical density and bulk density with Li content for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$.

FIGURE 5: The optical micrographs of $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ sintered at 1200°C .

at 1200°C . The grain size (D) is significantly dependent on Li substitution. The D increases with increasing Li substitution for fixed sintering temperature which is shown in Figure 5. This is probably due to the lower melting temperature of Li (180°C) compared to Mn (1245°C). The values of D for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites are presented in Table 2.

3.6. Complex Initial Permeability. The compositional variations of complex initial permeability spectra for the various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ samples sintered at 1200°C are shown in Figure 6. It is observed that the μ'_i remains fairly constant in the frequency range up to some critical frequency which is called resonance frequency, f_r . A sharp decrease in μ'_i and increase in μ''_i are observed above the f_r . The μ'_i increases with the increase of Li^+ content for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$. On the other hand, f_r was found to decrease with Li substitution. It was observed that

μ'_i of $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites sintered at 1200°C increases from 18 to 55. Figure 7 shows both μ'_i and D as a function of Li content for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites. According to Globus and Duplex model [35], the μ'_i can be explained as $\mu'_i = M_s^2 D / \sqrt{K}$, where M_s is the saturation magnetization and K is the magnetocrystalline anisotropy constant. This increase in permeability is expected, because grain size of all samples increases with Li content. It is known that the mobility of domain walls is greatly affected by the microstructure of ferrites. Therefore, in the present case, variation of the initial permeability may be influenced by its grain size.

The variation of loss factor, $\tan \delta (= \mu''_i / \mu'_i)$ with frequency for all samples, has been studied. The variation of initial loss with frequency for the various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ samples sintered at 1200°C is shown in Figure 8. At lower frequencies magnetic loss is observed and remains constant up to, a certain frequency,

(a)

(b)

FIGURE 6: The μ'_i and μ''_i for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites sintered at 1200°C as function of frequency.

TABLE 1: Particle size and FWHM of $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ nanoparticles.

x	FWHM (in degree)	Particle size (nm)
0.00	0.015379	9.4
0.10	0.00733	19
0.20	0.006109	23
0.30	0.004712	30
0.40	0.004887	30
0.44	0.005411	27

9 MHz; this frequency limit depends upon the sintering temperatures. The lag of domain wall motion with respect

FIGURE 7: The μ'_i and grain size with Li content for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ sintered at 1200°C .

FIGURE 8: Loss factor as a function of frequency for various $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ sintered at 1200°C .

TABLE 2: The lattice parameter, density, average grain size, and initial permeability of $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ sintered at 1200°C .

x	a_o (Å)	ρ_{th} (g/cm ³)	ρ_B (g/cm ³)	D (μm)	μ'_i (at 10 KHz)
0.00	8.3370	5.32	4.82	17.92	18
0.10	8.3615	5.15	4.62	19	22
0.20	8.3746	5.02	4.58	39	33
0.30	8.3866	4.90	4.44	92	39
0.40	8.4024	4.76	4.41	145.45	50
0.44	8.4091	4.56	4.30	295.45	55

to the applied magnetic field is responsible for magnetic loss and this is accredited to lattice imperfections [36]. At higher frequencies, a rapid increase in loss factor is observed. A resonance loss peak is shown in this rapid increase of magnetic loss. At the resonance, maximum energy transfer occurs from the applied field to the lattice which results in the rapid increases in loss factor.

4. Conclusion

The $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ($x = 0.00$ to $x = 0.44$) nanoparticles have been successfully synthesized by the combustion technique. The observed particle size is in the range from 9 nm to 30 nm. The XRD patterns confirm that the compositions are single phase and form cubic spinel structure. The lattice parameter increases linearly with increasing Li content and obeys Vegard's law. The study of microstructure shows that grain size increases with increasing Li content. The bulk density decreases with increasing Li substitution in $\text{Li}_x\text{Cu}_{0.12}\text{Mn}_{0.88-2x}\text{Fe}_{2+x}\text{O}_4$ ferrites. The real part of initial permeability increases with increase of Li content for a fixed sintering temperature. This result may be explained with the help of average grain size. The highest μ'_i was found 55 for $x = 0.44$ which is three times greater than that of parent composition. It was also observed that the resonance frequency, f_r , and real part of initial permeability, μ'_i , are inversely proportional which confirms Snoek's relation, $f_r\mu'_i = \text{constant}$.

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