

RESEARCH ARTICLE



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SYNTHESIS AND CHARACTERISATION of $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ FERRITE NANOPARTICLES

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ABSTRACT

Nanoparticles Li-Mg-Cu ferrite synthesized by the co precipitation method. The powder samples were characterized by XRD, SEM, TEM and FTIR. X-ray diffraction (XRD) patterns of the sample indicate the formation of single-phase cubic spinel structure. X-ray analysis showed that the samples were cubic spinel. Using the Scherrer Equation the measured size of Li-Mg-Cu ferrites particles was in between 35-45 nm. From the hysteresis measurements the Li-Mg-Cu ferrite powders obtained in this investigation a ferromagnetic behavior. The Fourier transform infrared spectroscopy was used to confirm the presence of metal oxide bands.

Key words: $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ ferrite nanoparticles, Co precipitation method, Characterization, Magnetic properties

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Introduction

Nanosized ferrite magnetic materials are the subject of intense research work because of their numerous applications in various fields such as, magnetic disc drives, spintronics, ferrofluids, catalysts, medical diagnostics, drug delivery systems, pigments in paints and ceramics [1-3]. Lithium ferrite is an unusual and, in the same respect, a remarkable material. Where, several research programs have been undertaken to study its fundamental properties and to develop high-power microwave materials from it. Reddy (1988), studied the mixed ferrite $(\text{Li}_{0.5}\text{Fe}_{0.5})_{1-x}\text{Ni}_x\text{Fe}_2\text{O}_4$ and claimed to be the first study of this kind of ferrites. He measured the electrical conductivity and thermoelectric power as a function of the composition and temperature. Charge carriers mobility, dielectric constant, and loss tangent have

been studied at low frequencies[4]. Arana et al. (2012) studied the Li-substituted Mn-Zn ferrite structural and magnetic properties after different thermal treatments. Spinel ferrites which has the chemical formula $\text{Zn}_{0.6}\text{Mn}_{0.4}\text{Fe}_2\text{O}_4$ and $\text{Li}_{0.2}\text{Zn}_{0.2}\text{Mn}_{0.4}\text{Fe}_{2.5}\text{O}_4$ were prepared by the selfcombustion sol-gel method. Incorporating Li to the crystalline lattice increased the saturation magnetization and promoted a decrease in the secondary phase's segregation. This result was explained assuming that the Li incorporation produced a cationic redistribution in the spinel structure [5]. Till now, no other report has been found in the literature for Li-Mg-Cu ferrite nanoparticles by simplest co precipitation method. Lithium ferrites are low-cost materials which are attractive for magnetic and microwave device applications [6-7]. Hence, there has been a growing

interest in Li-substituted Cu-Mn ferrite for microwave applications and high permeability with low magnetic loss[6]. Therefore, this paper is devoted to study the structural, morphology and magnetic properties of $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ ferrites prepared by co precipitation technique.

Materials and Methods

Co-precipitation is an attractive method of producing ferrites because of increased homogeneity, purity, and reactivity over standard ceramic processing. It is a simple method to produce nanoparticle spinel materials. Coprecipitation is a phenomenon where a solute that would normally remain dissolved in a solution precipitate out on a carrier that forces it to bind together, rather than remaining dispersed. An important aspect of this process is the very exact control of final composition through the use of the pure metals as starting materials. The $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ ferrites were prepared by co precipitation technique. The analytical grade of $\text{Li}(\text{NO}_3)_2$, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, 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. The precipitate was further aged overnight, filtered and washed several times with double distilled water and dried in hot air oven at 110°C . The compounds obtained were ground finely in agate mortar and pestle.

The scanning electron microscopy was carried by analyze the microstructure of fractured surface of the pellets using the SEM JEOL-JSM 6360 MODEL, JAPAN. Infrared absorption spectra of powdered samples were record in the range of $4000\text{--}400\text{ cm}^{-1}$ using perkin-Elmer FT-IR spectrum one spectrometer by KBr pellet technique. The magnetic properties have been measured at room temperature by a Vibrating Sample Magnetometer (VSM) (155, PAR) up to a magnetic field of $\pm 10\text{ kOe}$.

The average crystallite diameter L was estimated by the Scherrer equation using the peak broadening (FWHM) of the most intense peak (3 1 1): $L = \frac{0.91\lambda}{\beta \cos \theta}$

where L is the crystalline size perpendicular to (hkl) plane, λ the wavelength of X-ray used ($\lambda = 1.5418\text{ \AA}$), β full width at half maximum of the diffraction peak (FWHM) and θ the peak angular position. The lattice parameter (a) was calculated by using the following relation:

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

The magnetic properties of the synthesized nanoparticles were measured using a VSM (Lakeshore) at room temperature and the hysteresis loop was drawn with the magnetic field, cycled between 16000 G to 16000 G.

Results and Discussion

Figure 1 shows the XRD patterns of the $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ ferrite sample sintered at 950°C . The measurements were done in the 2θ range of $20^\circ\text{--}70^\circ$ for samples of Li-Mg-Cu ferrites obtained by the precipitation method. The calculated distance between the main crystal planes was 0.2 nm. Well defined diffraction peaks corresponding to the characteristic planes of (220), (311), (222), (400), (422), (511) and (440) planes that are indications of the presence of the cubic spinel structure. These diffraction lines provide clear evidence on the formation of $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$. All of the diffraction peaks match well with the reported values [11] and are indexed with the lattice parameter of $a = 8.339 \pm 1\text{ \AA}$. No secondary phase was detected in XRD, ensuring the phase purity of the final product. Using the Scherrer Equation the measured size of Li-Mg-Cu ferrites particles was in between 35-45 nm. The lattice parameters and crystallite size of the sintered ferrite specimen, evaluated by XRD analysis, inferred that the lattice constant is found to be 8.368 \AA , grain size is 38nm on average and ionic radii is 0.694 \AA .

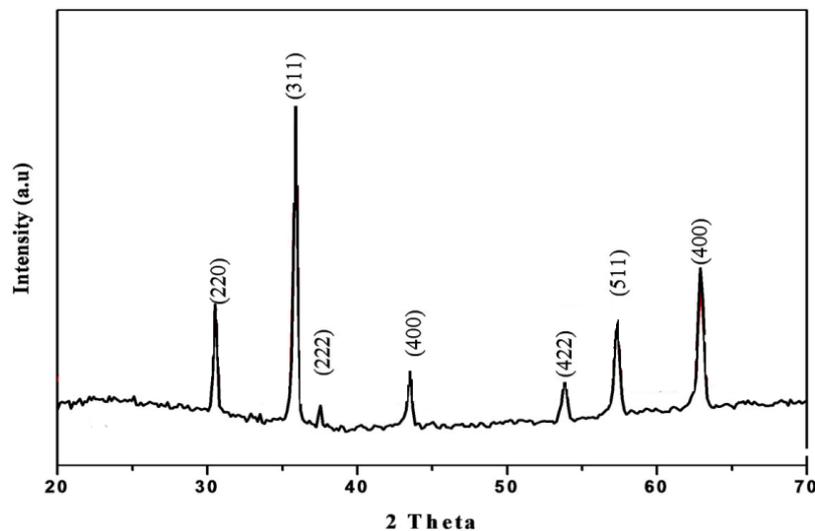


Figure 1: XRD pattern of $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ ferrite nanoparticles

Scanning Electron Microscopy (SEM) imaging was conducted to observe the shape and morphology of sample, by performing SEM technique, we analyzed the structure of $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ powder and show typical micrograph in Figures 2 synthesized by the coprecipitation method. It was observed from the image the surface has compacted shapes with round particles with smallest particles sizes typically less than 50nm. It is evident from the micrograph that the microstructure of the surface consists of detached, approximately closely-packed particles.

The TEM and FESEM micrographs of the synthesized nanoparticle along with the selected area electron diffraction [SAED] pattern for pure Li-

Mg-Cu ferrite nanoparticles are shown in Figures 3. The micrographs show largely agglomerated nanoparticles of the sample powder. An overview of the TEM image of nanoparticles shows that the particles have a size distribution of 34 to 46 (± 1) nm. The average size of the agglomerates is found to be 38 nm. Such aggregate formation and broader size distribution are characteristic of mechanically activated nanosized particles [10]. Very few large particles having a size at approximately 50 nm have also been observed. It is also cleared that the particle size obtained from FETEM measurements corroborates well with the crystallite size obtained from XRD analysis.

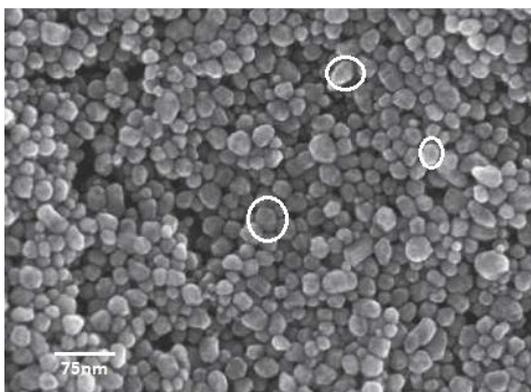


Figure 2: Scanning Electron Microscopy image of $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ at x12000 resolutions

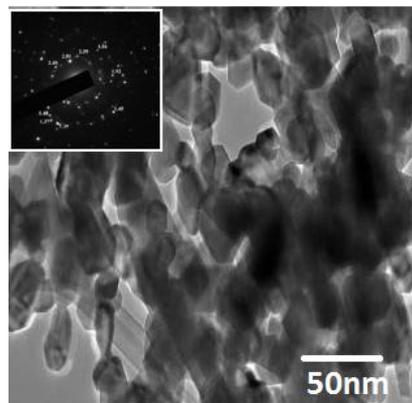


Figure 3: TEM image of $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ powder for sample (in inset SAED pattern)

The FTIR spectra for Fe_3O_4 and for $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ is shown in figure 4. The broad feature between $3441.43 - 3219.90 \text{ cm}^{-1}$ is due to O-H stretch which corresponds to the hydroxyl groups attached by the hydrogen bonds to the iron oxide surface and the water molecules chemically adsorbed to the ferrite particle surface (associated water content). From these results, it appears that the hydroxyl groups are retained in the samples during the preparation of the uncoated $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ spinel ferrites prepared by coprecipitation

method. S.Dey [8] have reported that the presence of some hydroxyl ions are completely removed when the sample is sintered at temperatures $\geq 973\text{K}$. The O-H in-plane and out-of-plane bonds appear at $1537.80 - 1500.31 \text{ cm}^{-1}$ and $960.03 - 874.235 \text{ cm}^{-1}$, respectively. In the spectrum a strong band from 635.57 to 573.51 cm^{-1} due to Fe_3O_4 ²⁹. The transmittance waveband from 635.57 to 573.51 cm^{-1} , which corresponds to the metal-oxygen bonds may be due to LiO , MgO , CuO and Fe_3O_4 .

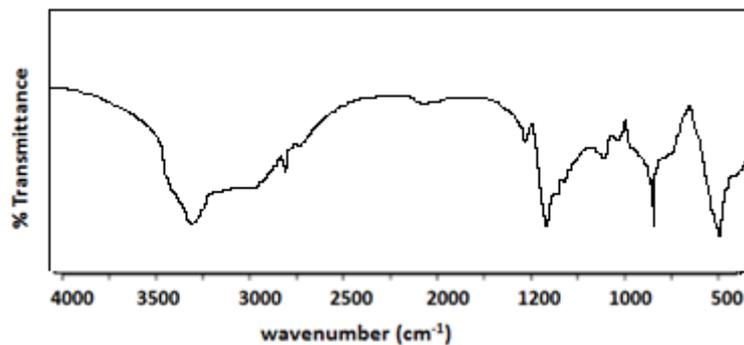


Figure4: FTIR spectra of $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ ferrite nanoparticles

Magnetic Hysteresis Measurements

The magnetic behavior of the Li-Mg-Cu ferrite obtained by coprecipitation measuring the magnetization as a function of an applied 50 Hz, AC magnetic field amplitude up to 160 kA/m [9]. In Figure 5 the M-H curve of the ferrite sample obtained by co precipitation method given. In the case of the sample obtained by coprecipitation a

superparamagnetic-like behavior was observed, with a maximum magnetization of 198 A/m at 160 kA/m. From the hysteresis measurements the Li-M-Cu ferrite powders obtained in this investigation a ferromagnetic behavior with stronger hysteresis-which means that the sample can be stronger magnetized.

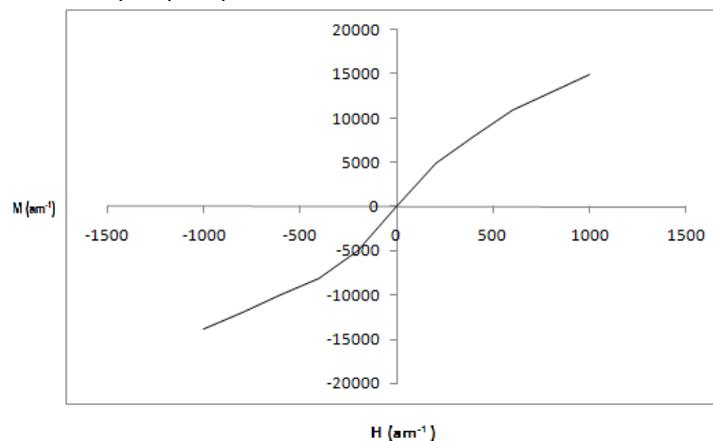


Figure 5. Hysteresis curves of the $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ powder for sample

Conclusions

To summarize the main ideas obtained, the few conclusions can be drawn from this work. Mixed $\text{Li}_{0.4}\text{Mg}_{0.2}\text{Cu}_{0.4}\text{Fe}_2\text{O}_4$ ferrites were successfully prepared by co precipitation technique. The formation of single phase crystallite structure with size in the range 35-45 nm was confirmed by X-ray diffraction. It is also confirmed by the SEM and TEM micrographs. Structural studies proved the nanocrystalline nature of the samples. The microstructure of ferrite material prepared by this technique consists of crystallite with clearly seen spinel cubic forms. The synthesized nanoferrites are expected to be useful in several technological applications such as soft magnets and magnetic fluids for hyperthermia. The structural and properties of spinel ferrites

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