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Impact of transition metal ion doping on electrical properties of lithium ferrite nanomaterials prepared by auto combustion method

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ABSTRACT

Nano crystalline cobalt substituted lithium ferrites having chemical formula $[Li_{0.5}Fe_{0.5}]_{1-x}$ Co_x Fe₂O₄ (where x=0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) have been prepared by the citrate gel auto combustion method at low temperature (180 °C). The prepared samples were sintered at 500 °C for 4 hours. Single phase of these samples were confirmed by x-ray powder diffraction technique and found that lattice parameter increases with increasing with the Cobalt substituted concentration, and the values of crystallite size, x-ray density, bulk density and porosity of the prepared samples were calculated from the XRD analysis. The dc electrical resistivities of the prepared Li-Co ferrites were studied by using the two probe method in the temperature range of 473-873 K (200-600 °C). A plot of log (σ T) vs inverse of temperature yields a almost straight line which reveals the semiconducting behavior of prepared ferrite samples. The dielectric properties namely dielectric constant (ϵ), dielectric loss tangent (tan δ) of these Li-Co nano crystalline ferrites have been studied using an LCR meter from the room temperature to 700 K at various selected frequencies up to 5 MHz. The dielectric constant (ϵ) of all prepared samples was increased with increasing in temperature. These results have been explained on the basis of Koop's theory, concentration of Fe⁺² and Fe⁺³ ions on the octahedral site and hoping frequency of charge carriers (electrons) between Fe⁺² and Fe⁺³ ions. Copyright © 2015 VBRI press.

Keywords: Citrate gel method; nano ferrite; XRD studies; dc resistivity; dielectric properties.



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field of science and Technology, UGC research award, Boyscast by

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DST (Department of science and technology), Government of India, JSPS fellowship Japan and Royal Society fellowship, UK. He also visited USA, UK, Sweden, Ireland, Singapore and Japan for Collaborative Research and invited talks.

Introduction

Magnetic spinel ferrites have captured the global market and grabbed the attention of many researchers due to their fascinating and exotic electromagnetic properties. The unique properties of nano-scaled magnetic ferrite particles have generated more interest in the science and engineering community because these nano-crystalline particles have a high surface to volume ratio which results novel properties contrary to those bulk particles. The excellent electrical and dielectric properties of the some of the nano ferrites make them suitable for the high frequency applications in the field of telecommunications. The properties of ferrites depend on several factors which include method of preparation, sintering temperature/time, pH value, amount

and type of substitution, and most important being the distribution of metal cations at tetrahedral and octahedral sites in the spinel lattice of the ferrites etc to obtain desired properties for specific applications [1]. These nano crystalline magnetic ferrites have been used as permanent magnets in various devices as these are regarded as better magnetic materials than pure metals because of their large value of resistivity and larger efficiency and reasonable cost. Lithium ferrites and substituted lithium ferrites have proved to be very good alternatives for garnet ferrites in microwave applications due to their low cost. Lithium ferrite belongs to the group of soft ferrite materials extensively used in many applications such as microwave devices computer memory chips, magnetic recording media, radio frequency coil fabrication, transformer cores rod antennas and many branches of telecommunication and electronic engineering [2-6]. Several researchers have studied transition metal substituted lithium ferrites to enhance their properties such as Li-Cr [7, 8], Li-Cd [9], Li-Zn [10], Li-Mg [11], Li-Co [12,13]. Several preparation methods are used for synthesizing nano sized spinel ferrites such as co-precipitation, sol-gel, micro emulsion, hydrothermal, citrate-gel, reverse micelle methods [14-16]. Among the all the preparation methods, citrate gel method is very adequate for the preparation of nano crystalline ferrites. This method has a special significances (i) in yielding ultrafine single phase nano crystalline ferrites (ii) no ball milling is required, (iii) better purity, (iv) gives homogeneous mixture of metal ions, (v) narrow grain size distribution, (vi) low temperature processing which reduces the lithium volatilization and (vii) relatively low cost process as compared with other wet chemical synthesis processes.

The objective of present work is to obtain cobalt substituted lithium ferrite systems having compositional formula $[Li_{0.5}Fe_{0.5}]_{1-x}$ Co_x Fe₂O₄ (where x=0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) prepared by citrate gel auto combustion method at lower sintering temperature. Literature is accessible on structural properties of investigated system. To the best of our knowledge, investigations on detailed electrical and dielectric properties of Li-Co ferrite system prepared by citrate gel method have not been reported. The present study reports on detailed synthesis, structural, electrical and dielectric properties of Li-Co ferrite nano particles.

Table 1. Raw material information.

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5.NO	Chemical Name	Purity (%)	name	Placeof Manufacture
1	Lithium-nitrate (LiNO $_3$)	98.8	SIGMA-ALDRICH	Spruce street,St.Louis,USA.
2	Ferric-nitrate (Fe(NO ₃) ₂ 9H ₂ O)	99.0	CentralDrug House (P) Ltd	New Delhi, India
3	Cobalt-nitrate (Co(NO ₃) ₂ 6H ₂ O)	99.0	Sdfine-CHEM LIMITED	Mumbai, India.
4	Citric-acid (C ₆ H ₈ O ₇ .H ₂ O)	99.7	S d fine-CHEM LIMITED	Mumbai, India.
5	Ammonia solution (NH ₃).	99.7	Sdfine-CHEM LIMITED	Mumbai, India

*These chemicals were used in the material synthesis without further purification.

Experimental

Materials and methods

Nano crystalline cobalt substituted lithium ferrites having chemical formula $[Li_{0.5}Fe_{0.5}]_{1-x} Co_x Fe_2O_4$ (where x=0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) have been prepared by the citrate gel auto combustion method at low temperature (180°C). In this method of preparation, an exothermic reaction takes place between the oxidant and reductant in which NO³⁻ ions are acts like the oxidant and the carboxyl group is the reductant. The enthalpy of exothermic reaction is used to synthesize materials in nano form.

Raw materials

The starting materials information viz. their purity, manufacturer name and place of manufacturing are given in the **Table 1**.

Method of synthesis

Stoichiometric amounts of the metal nitrates along with citric acid were dissolved in minimum amount of distilled water. Molar ratio of metal nitrates and citric acid was taken as 1:1 [17]. The resulting clear solution was mixed and heated at 80°C with continuous stirring by using a magnetic stirrer with hot plate. The pH value of the solution was controlled at 7 by addition of ammonia drop by drop. A change in the viscosity and color of the solution was observed and converted to a gel after stirring for some hours. By increasing the temperature up to 180 °C, the gel ignited on drying due to self combustion with the evolution of gases giving rise to a dark gray ash as product. This resultant ash powder samples were sintered for 4h at a temperature of 773K at default heating rate of 4K/min using a temperature programmed box furnace. The powdered materials were characterized by various techniques.

Table 2. Structural parameters of the prepared Li-Co ferrite sample.

Composition	Mol.wt	Crystallite	Lattice Parameter(A%)	X-ray Density(d _x)	Experimental	Porosity
	(gin/mor)	3126(1111)		(ginee)	(gm/cc)	(1)
Li _{0.5} Fe _{2.5} O ₄	207.079	41.90	8.356	4.713	4.286	9.00
Li _{0.4} Co _{0.2} Fe _{2.4} O ₄	212.587	43.01	8.370	4.815	4.379	9.10
Li _{0.3} Co _{0.4} Fe _{2.3} O ₄	218.095	38.44	8.374	4.931	4.388	11.00
Li _{0.2} Co _{0.6} Fe _{2.2} O ₄	223.603	37.57	8.386	5.035	4.393	12.70
Li _{0.1} Co _{0.8} Fe _{2.1} O ₄	229.111	37.06	8.394	5.144	4.580	10.90
CoFe ₂ O ₄	234.619	36.90	8.404	5.250	4.685	10.70

Characteristic techniques

The X-ray diffraction analysis of the prepared samples were performed on a Philips X pert PRO diffractometer using Cu K α as radiation source to study the phase developed of the synthesized nano particles and to calculate the structural parameters. The crystallite size of the Li-Co nano ferrite samples was investigated based on the X-ray diffraction line broadening and calculated using the Debye-Scherer's formula **[18]**. Structural parameters like lattice parameter, X-ray density, Porosity have been calculated by using XRD data and given in the **Table 2**. For measuring the electrical properties, powders were added with a small amount 2 % PVA as a binder to press the prepared powders into circular pellets having 13 mm diameter and 2.5 mm thickness by applying a pressure of 4 tons. The prepared pellets were sintered at 500 °C for 4 hours in air in muffle furnace for the densification of the sample. For having a good ohmic contact silver paste was applied on both sides of the pellet. The dielectric properties of the sample were measured by using Agilent E4086 precession LCP meter from room

The dielectric properties of the sample were measured by using Agilent E4986 precession LCR meter from room temperature to 725 K at different frequencies up to 5 MHz. DC resistivity of the prepared samples was measured by using two probe methods in the temperature range 473-873 K.

Results and discussion

XRD analysis

The phase identification and determination of structural parameters of the prepared samples were measured by the X-ray diffraction analysis. The X-ray diffraction pattern of the prepared cobalt substituted lithium ferrite samples were shown in below **Fig. 1**. The strong diffraction from the (220), (311), (400), (422), (511), and (440) planes confirms the pure spinel phase of the prepared samples. The XRD pattern perfectly matches with the standard pattern with JCPDS reference code 00-013-0207.

It can be seen that the location of the peaks in XRD pattern of all samples are coincident, which indicate that there were no distinct differences in phase structure of Li-Co nano crystalline ferrites with different cobalt composition. It was observed that cobalt ions in the lattice were partly replaced by iron and lithium ions and then increased the lattice constant. The XRD analysis of the prepared Li-Co nano crystalline samples provides the estimation of the crystallographic lattice constant (a), crystallite size (D), X-ray density (d_x) and experimental density (d_e) etc as given in **Table 2**.



Fig. 1. XRD pattern of the $[Li_{0.5}Fe_{0.5}]_{1-x}$ Co_x Fe₂O₄ nano ferrites.

The values of the crystallite size which are calculated from the Debye-Scherer's formula were in the range of 36-43 nm which confirm the nano crystalline form. The lattice parameter of the samples increases with cobalt composition which obeys the Vegard's law [19]. From the **Table 2** we observed that lattice parameter of the prepared samples increases with increase in the cobalt composition because ionic radii of the six-fold coordinated Co^{+2} (0.82A°) being larger than the six-fold coordinated Fe^{+3} (0.64A°) and $Li^{+1}(0.76A^{\circ})$ similarly X-ray density of the prepared samples increases with cobalt composition because molar masses of the substituted cobalt ions were larger than that of iron ions. The experimental density of the samples were observed to be less than the X-ray density which reveal that the some porous nature of the prepared samples. The variation of lattice parameter and experimental density of the prepared samples with cobalt composition was shown in below **Fig. 2**.



Fig. 2. Variation of (a) Lattice parameter and (b) Experimental density with Co composition of $[Li_{0.5}Fe_{0.5}]_{1-x}$ Co_x Fe₂O₄ ferrites.

DC resistivity studies

The dc electrical resistivity was the important property to study the conduction mechanism in ferrites. The conduction mechanism in ferrites was due to the hopping of charge carriers (electrons), between the ions of same element, present in more than more valence state, distributed randomly over equivalent crystallographic sites in lattice. The probability of hopping depends on the separation between the involving ions and activation energy [33]. The electrostatic interaction between the conduction electron and nearby ions may result in the polarization of the surrounding region so the electron present at the center of the polarization well. This electron is transferred to the neighboring site by thermal activation. This mechanism in conduction is called hopping mechanism [20].

The cation distribution of lithium ferrite and cobalt substituted lithium ferrite having the inverse spinel structure were given by $(Fe_{1.0}^{+3})[Li_{0.5}^{+}Fe_{1.5}^{+3}]O_4^{-2}$ and $(Fe^{+3}_{1.0-x+y}Co^{+2}_{x-y})[Li_{0.5-0.5x}Fe^{+3}_{1.5+0.5x-y}Co_y^{+2}]O_4^{-2}$ for x=0

and x>0 respectively, where the parenthesis denote the Asites and square brackets denote the B-sites **[34]**. For lithium ferrite, the conduction mechanism taking place between the Fe⁺² and Fe⁺³ ions present in the equivalent crystallographic sites in the structure of the ferrite.

$$Fe^{+2} \leftrightarrow Fe^{+3} + e^{-3}$$

In ferrites the electrons transfer between the adjacent Bsites in the spinel structure. Local displacement of electrons in the direction of applied electric field can be obtained that occur due to the displacement in determining the polarization effect in ferrite [21].

In this article we discussed the electrical conductivity of the prepared samples in the temperature range 200-600 °C, which reveal that the electrical conductivity of the prepared samples increases with increasing temperature, which indicated the semiconducting nature of the prepared samples. The dc electrical conductivity of the of materials has a general form $\sigma = \sigma_0 \exp(-E_a/kT)$ where E_a is the thermal activation energy, σ_0 is the pre exponential factor depending on material nature and k is the Boltzmann constant. The temperature dependence of the electrical conductivity of the prepared samples were studied by plot a graph between the Log (σ T) vs 1000/T which shown in **Fig. 3**, which yields a straight line whose slope can be used to calculate thermal activation energies of the ferrite samples.



Fig. 3. Arrhenius plots for electrical conductivities of nano crystalline $[Li_{0.5}Fe_{0.5}]_{1-x}$ Co_x Fe₂O₄ ferrites.

According to Fig. 3 except the pure lithium ferrites, Arrhenius plots for electrical conductivities of cobalt substituted lithium ferrites shows a discontinuity (kink occurs) in the straight line which shows two different regions with a variation in the activation energies. The temperature where the discontinuous kink occur was called transition temperature (T^{*}). V.R.K. Murthy et al. [22] reported that discontinuity in the Arrhenius plot may be due to the change in the conduction mechanism. For $T < T^*$ region the conduction mechanism may be due to the electron hopping between the Fe^{+2} and Fe^{+3} ions in the octahedral sites of the crystal lattice. For $T > T^*$ region the conduction mechanism may be probably due to the ionic conduction caused by the lithium and cobalt ions in the Bsites [23]. But according to Raghasudha et al. [24] proposes that a discontinuity in the Arrhenius plot may be due to the ferri to para magnetic transition from low to high temperature. This transition temperature (T^*) represents the Curie temperature (T_c) . Therefore it suggests that change in slope due to the magnetic transition. Similar transitions near the Curie temperature have also been observed by several investigators for different ferrites [25, 26].

The variation of dc resistivity with cobalt concentration was shown in **Fig. 4** which reveals that dc resistivity of the prepared Li-Co samples decreases with Co composition. This may be due to the hoping of electrons between the $Fe^{+2} \leftrightarrow Fe^{+3}$ and $Co^{+2} \leftrightarrow Co^{+3}$ increases in the octahedral sites of the prepared samples with increase in the Co composition [**27**].



Fig. 4. Variation of dc resistivity with cobalt composition.

Table 3. Curie temperature and activation energies of Li-Co nano ferrites.

S.N0	Composition	Curie Temp	Activation Energy(eV)	
		(°C)	Paramagnetic	Ferrimagnetic
1	Li _{0.5} Fe _{2.5} O ₄	>600		1.340
2	Li _{0.4} Co _{0.2} Fe _{2.4} O ₄	572	0.945	0.875
3	Li _{0.3} Co _{0.4} Fe _{2.3} O ₄	560	0.798	0.703
4	Li _{0.2} Co _{0.6} Fe _{2.2} O ₄	553	0.659	0.526
5	Li _{0.1} Co _{0.8} Fe _{2.1} O ₄	540	0.621	0.480
6	CoFe ₂ O ₄	521	0.712	0.575

The **Table 3** summarizes the activation energies in ferri magnetic and paramagnetic regions and Curie temperatures

of the Li-Co ferrites. From **Table 3** one can observed that activation energy of the paramagnetic region is larger than the ferri magnetic region [28]. This is because ferri magnetic state was an ordered state one while the para magnetic state is disordered one hence charge carriers require more energy for conduction in paramagnetic state compared to ferri magnetic state.



Fig. 5 Variation of Curie temperature with Co composition.



Fig. 6. Variation of Dielectric constant with temperature of $[Li_{0.5}Fe_{0.5}]_{1-x}$ Co_x Fe₂O₄ ferrites.

From the **Fig. 5** we can observe that Curie temperature of the prepared samples was observed to be decrease with the increase in cobalt composition. This can be explained on the basis of the number of magnetic ions present in the two sub-lattices and their mutual interaction. An increase in the cobalt composition at the B-site has replaced the Fe^{+3} ions at this site, which leads to reducing the AB interaction of the type $^{+3}(A)$ -O⁻²- Fe⁺³(A). Since T_c depends on the strength of AB exchange interaction, so the weakening the interaction results in a decrease in Curie temperature (T_c).



Fig. 7. Variation of Dielectric loss (tan δ) with temperature of $[Li_{0.5}Fe_{0.5}]_{1-x}$ Co_x Fe₂O₄ ferrites.

Dielectric studies

The dielectric parameters of the prepared samples were depends on the temperature and frequency. The temperature dependence of the dielectric constant of the prepared Li-Co ferrites was shown in Fig. 6. From this Fig. 6 one can be concluded that except the pure lithium ferrite, dielectric constant of the cobalt substituted lithium ferrite increases with increasing the temperature. In case of pure lithium ferrites dielectric constant was increases up to certain temperature beyond that temperature, dielectric constant going to decrease [29]. Similar behavior can be observed by the various researchers for different compositions [30]. The hopping of electrons between the Fe^{+2} and Fe^{+3} ions present in the octahedral sites is thermally activated by increasing temperature which causes local displacements in the direction of external field, which enhances their contribution to the space charge polarization thereby leading to increase in the dielectric constant [31]. The variations of dielectric loss with temperature of the prepared samples were shown in Fig. 7 this behavior is similar to that of variation of dielectric constant with temperature shown in **Fig. 6**.

The frequency dependent dielectric constant (ε ') and dielectric loss tangent (tan δ) of the prepared Li-Co ferrite samples for x=0.2 were shown in **Fig. 8** remaining samples show similar behavior (not shown). From the **Fig. 8** one can be concluded that dielectric parameters like dielectric constant and loss tangent were observed to be decrease with increase in frequency which can be understood by

Maxwell-Wagner two layer models in agreement with the Koop's theory [32]. According to this theory, ferrite structure was made up of well conducting grains separated by poor conducting grain boundaries. The high dielectric constant comes from the space charge polarization produced at the grain boundary.

During the hopping mechanism, the electrons have to pass through the grains and grain boundaries of the dielectric medium. Due to the high resistance to the grain boundary, the electrons were accumulating at the grain boundary and produce space charge polarization. It was well known that the grain boundaries were more effective at low frequency and grains were more effective at the high frequency. Therefore due to the grain boundary effect the dielectric constant decreases rapidly in the low frequency region and at high frequency the grains come into action and also exchange of electrons cannot follow the applied ac field hence dielectric constant decreases and frequency independent.



Fig. 8. Variation of dielectric parameters with frequency of $[Li_{0.5}Fe_{0.5}]_{1-x}$ Cox Fe_2O4 ferrites at x=0.2.



Fig. 9. Variation of dielectric constant with Co composition.

The variation of dielectric constant with cobalt concentration of the prepared samples was shown in **Fig. 9**. From this we concluded that the dielectric constant of the prepared samples increases with cobalt composition in lithium ferrites. This is because in Li-Co ferrites Li^{+1} ions occupy B-site and Co^{+2} ions have prefer to occupy both A and B sites. With increase of Co^{+2} compositions, some of the Co ions occupy A-site and displace the Fe⁺³ ions from the A-site to B-site. Thus more Fe⁺³- Fe⁺² ion pair are available at B-site for hopping of electrons resulting in the

increase of dielectric polarisation which results a increase of dielectric constant.

Conclusion

Citrate gel auto combustion method was a very suitable technique for the preparation of nano sized (36-43 nm) cobalt substituted lithium ferrite at low sintering temperature. The lattice parameter of the prepared samples observed to increases with increasing the cobalt composition which obeys the Viguard's law. The dc electrical resistivity of the prepared samples was decreases with increasing the temperature which shows the semiconducting nature of the samples. A plot of Log (σ T) vs 1000/T shows straight line and the discontinuity in the straight line shows a Curie temperature at which ferri magnetic state of the material shift to paramagnetic state. The dielectric parameters such as dielectric constant and dielectric loss tangent were observed to be increased with increasing temperature and decreased with increasing frequency. Finally by observing the results of these measurements one can say that dc resistivity is inversely proportional to the dielectric constant of the prepared samples.

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