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Synthesis, Characterization and Dielectric properties of Nanoparticles of Lithium Doped Ferrite ($Li_x Fe_{1-x} Fe_2 O_4$)

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***Abstract**:*
Ferrites are a newly discovered class of material being utilized for many important applications. lithium ferrites are soft magnetic material with low coercivity. We successfully prepared lithium ferrites by using nanocrystalline powers by Sol gel method. In our study Crystalline nanoparticles of Lithium ferrites ($Li_x Fe_{1-x} Fe_2 O_4$) (where x= 0.2, 0.3, 0.4, 0.5) were synthesized by Sol Gel Method using ferric chloride and lithium nitrate with NaOH as a reactant. Structural characteristics of samples were determined by X-Ray diffraction and FESEM. Particle size found between 9.64 nm to 24.71 nm by using Debye Scherrer method. Dielectric properties were investigated using impedance analyzer. The relative dielectric constant of ferrites a function of frequency (1kHz-30MHz) was investigated at room temperature, parameter decreases as frequency increases.

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I. Introduction:

Nanotechnology is the understanding and control of matter at the nanoscale, at dimensions between approximately 1 -100nm. First ferrite compounds were synthesized by Yogoro kato and Takeshi Takei in 1930 at Tokyo institute of technology. Now a days their wide application not only in military, but also being used with the civil perspective, along with the development of science and technology, and now became a hot area of interest in the context of developing function materials [1,2,3]. Spinel ferrites are a complex oxide crystal which has face-centered cubic structure with formula MFe_2O4, where M is metallic cation. Spinel ferrite contains 16 octahedral positions and eight tetrahedral positions. Poly crystalline ferrites are very good dielectric materials and have many technological applications ranging from microwave to radio frequencies. Hence, it is important to study their dielectric behavior at different frequencies. The dielectric properties of ferrites are dependent on several factors, including the method of preparation [4,5]. A number of research reports are available concerning the preparation techniques thermal decomposition [6], ceramic [7], sol-gel [8], combustion methods [9], hydrothermal [10] and co-precipitation [11] etc. Among the various methods we used sol gel method [8]. Sol Gel method is very effective method. Bringing more into focus the electric properties of lithium ferrite, this is considered a very promising cathode material to be used in rechargeable Li-ion batteries, as it presents low dielectric losses [12-15].Characterization of lithium nanoparticles is done by XRD and FESEM. Particle size using XRD characterization was calculated by debye Scherrer method [16].

II. Methodology:

Synthesis- Magnetic nanoparticles were prepared by the sol-gel method. We used $FeCl_3.6H_2O$, $FeSO_4.7H_2O$, $Li(NO_3)_2.6H_2O$ and NaOH as reactant to make lithium doped ferrites. We made four types of lithium doped ferrites using reactants in different concentrations. We made solutions of stoichiometric amount of $FeCl_3.6H_2O$, $Li(NO_3)_2.6H_2O$ and $FeSO_4.7H_2O$ in 100ml distilled water and a solution by using 6.4g NaOH dissolved in 200 ml distilled water.

All solutions were constantly stirred using magnetic stirrer for 20minute separately. Then took 100ml solution of NaOH into a beaker of 500 ml capacity and add lithium nitrate, ferric chloride and ferrous sulphate solution into it with stirrer on. pH of this mixed solution was between 8-9 then NaOH was added drop wise in line to achieve pH between 11- 12 under continuous stirring for 15minute. Then shifted this mixed solution on hot plate and raised its temperature till 80^0C. After 10minute, oleic acid 5ml was added in mixed solution. Then kept Mixture at 80^0C for 20minute. Now switched off hot plate for one hour while Stirrer was in running (on) state. Hot plate was switched on after 1 hour and its temperature was raised till 90^0C. Switched off hot plate again and let the solution to reach at room temperature meanwhile stirrer was in running (on) state. Then added 6 to 7 drops of HNO_3 into it, precipitate and dirty water got separated. We removed dirty water and washed precipitate with distilled water and kept precipitate in distilled water for overnight. Next day we washed precipitate first with boiled water for 5-6 time then with acetone for 5-6 time. To make sample dry we kept precipitate on filter paper for some time then in petri dish in sunlight. and sample in powder form prepared.

Characterization: Different techniques for the characterization of nanomaterials was done. A complex analytical system is necessary which is capable to determine the composition and the properties of the substances. We used X-Ray Diffraction (XRD) and Field Emission Scanning Electron Microscopy (FESEM) to study Structural morphology. Dielectric behavior is one of the most important property of ferrites. Impedance Analyzer was used to study dielectric properties.

III. Result And Discussion:

X Ray diffraction analysis

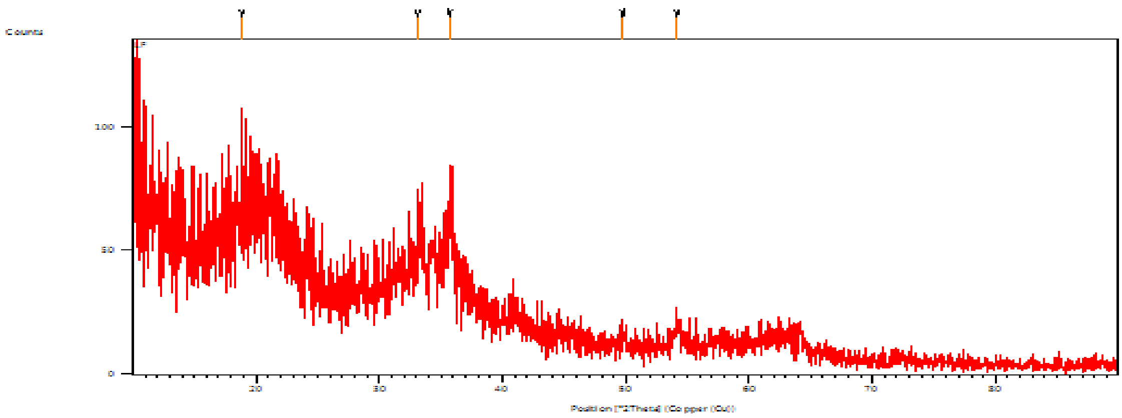
Its composition, phase structure and morphology are characterized by X-ray diffraction (Cu target, Wavelength 1.54184 Å). XRD patterns of different lithium doped ferrites are shown in figures (1.a -1.d). In these patterns one peak (h k l) value (3 1 1) is presented intensively. Crystalline size of every sample was calculated by debye scherrer formula [16] –

$$t=k\lambda/\beta\cos\theta,$$

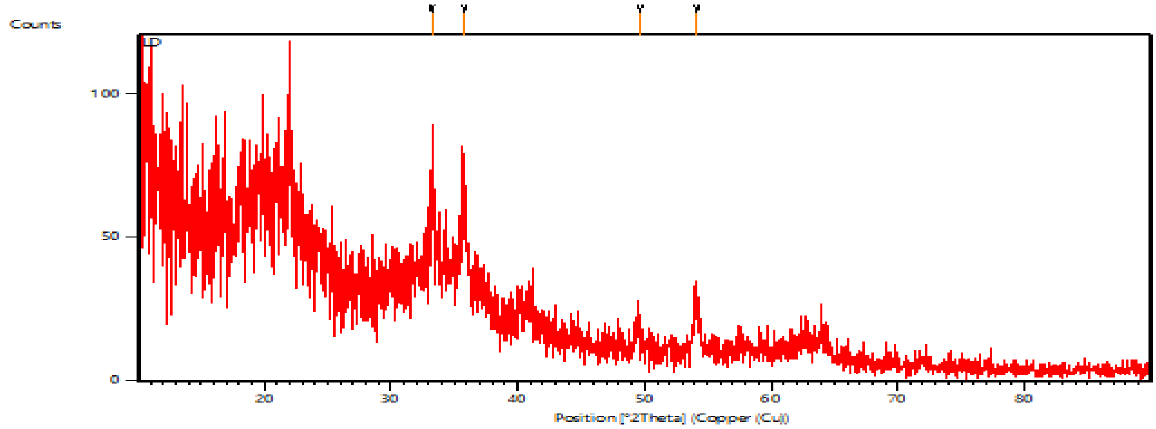
Where k is shape factor. In this the value of k is 0.9. λ is wavelength of X Ray used, θ is Bragg's angle. β is the full width at half maximum (FWHM) (in radian). From the above formula it was found that values of particle size 't' between 9.64nm-24.71nm. One can obtain lattice constant by Bragg's equation. Particle size, D spacing and lattice constant of different lithium ferrites are shown in Table 1.

$Li_xFe_{1-x}Fe_2O_4$	x =0.5	x =0.4	x =0.3	x =0.2
Particle size	24.70nm	24.71nm	24.70nm	9.64nm
D spacing	2.505 Å	2.507 Å	2.510 Å	2.513 Å
Lattice constant	8.310 Å	8.316 Å	8.325 Å	8.337 Å

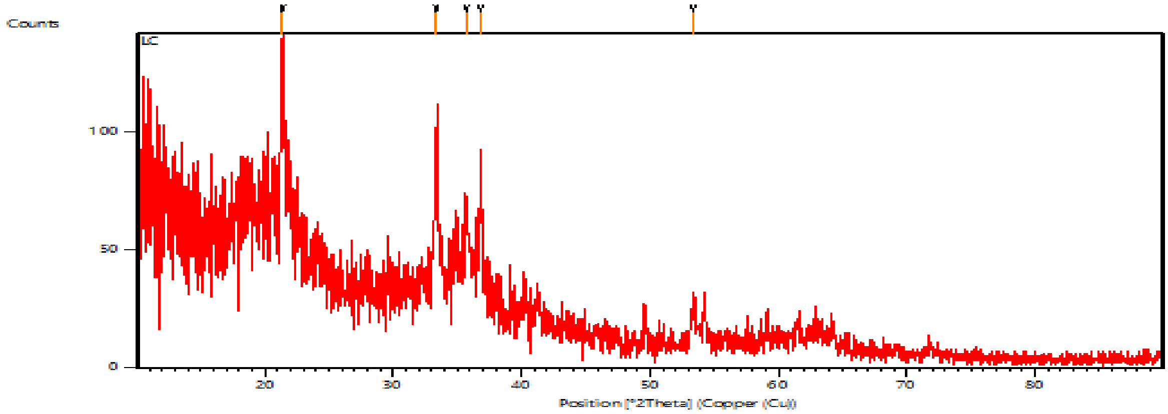
Table 1



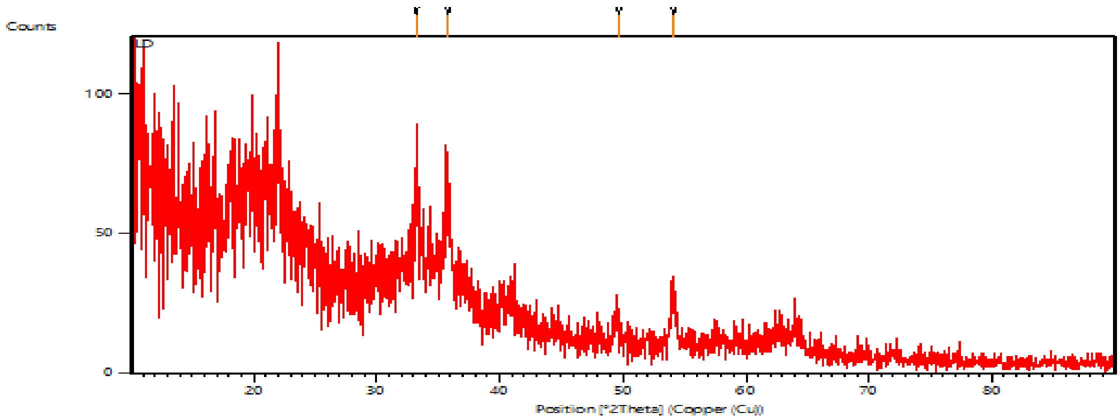
1.a: XRD pattern of Li0.5Fe2.5O4



1.b: XRD pattern ofLi0.4Fe2.6O4



1.c: XRD pattern of Li0.3Fe2.7O4



1.d: XRD pattern of Li0.2Fe2.8O4

SEM Analysis: FE-SEM analysis of one cobalt nano crystalline ferrite was done. SEM images of cobalt ferrite is shown in figure 2. The particle size of this sample is not uniform and a little bit large from we analysis by XRD. Particle size is found approximate 40 nm by FESEM analysis.

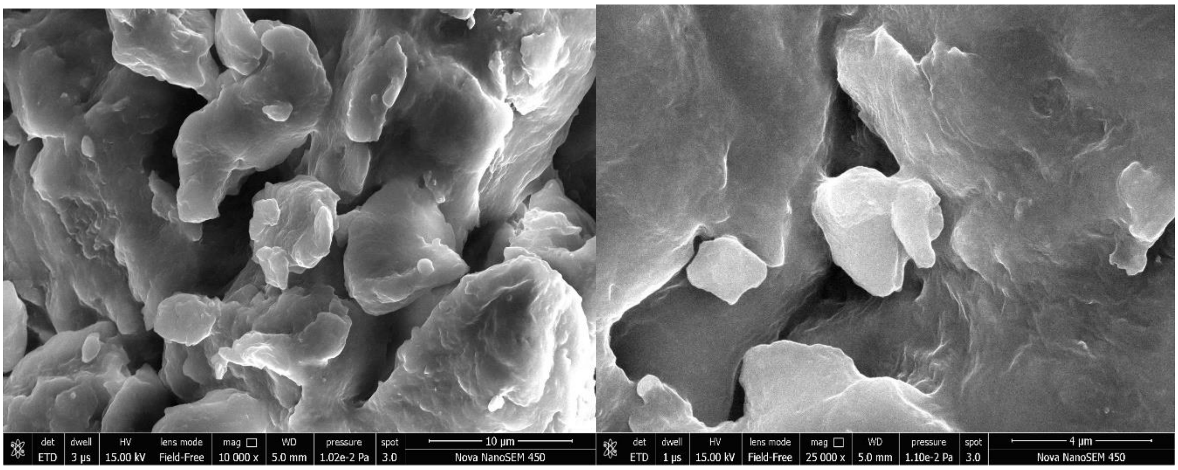


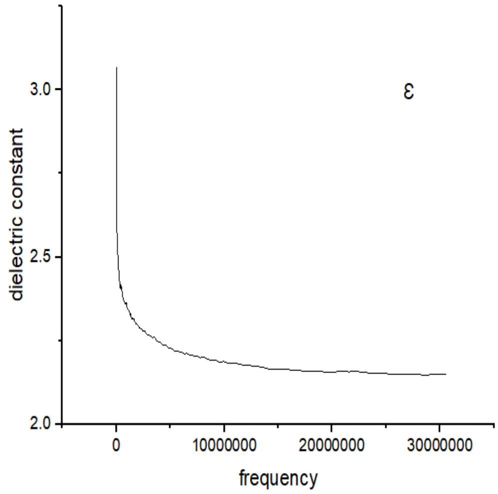
Fig 2: FESEM pattern of Li0.4Fe2.6O4

Dielectric constant: Dielectric measurement was performed by using 6500 series of precision Impedance Analyzer. For dielectric measurements, the sample powder was dried and compressed into disc shaped pellet of diameter 13 mm and thickness about 2 mm by applying the pressure of 5,6 tons. After that silver electrodes were deposited on both sides of the samples. Dielectric constant was calculated using the following formula-

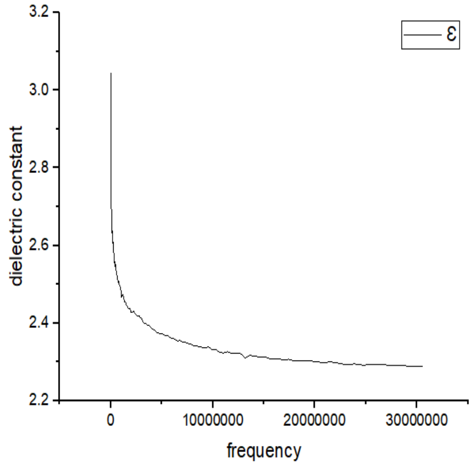
$\varepsilon = cd/\varepsilon_0 A$

where C- capacitance of the pellets, d - thickness of the pellets; A- cross-sectional area of the flat surface of the pellets (m^2) and ε_0 – permittivity of free space. The dielectric constants measurement was done in the range 1kHz to 30MHz using Impedance Analyzer. The graphs of dielectric constant against frequency are depicted in Fig. 3 (a-d). Dielectric constant is highest at the frequency of 1kHz and it decreases with the increase of frequency in all the samples. This dependence upon frequency is because of the interfacial polarization as predicted by Maxwell-Wagner [17]. In their model, they assumed that the dielectric structure of a ferrite material is made up of two layers; the first layer which is a conductive layer consists of large ferrite grains and the second layer being the grain boundaries are poor conductor. Rabinkin and Novikova [18] showed a mechanism similar to the conduction process that result in the polarization in ferrites. Due to the electron exchange between Fe^{+2} and Fe^{+3} the local displacement of the electrons in the direction of the applied field occurs and these electrons determine the polarization.

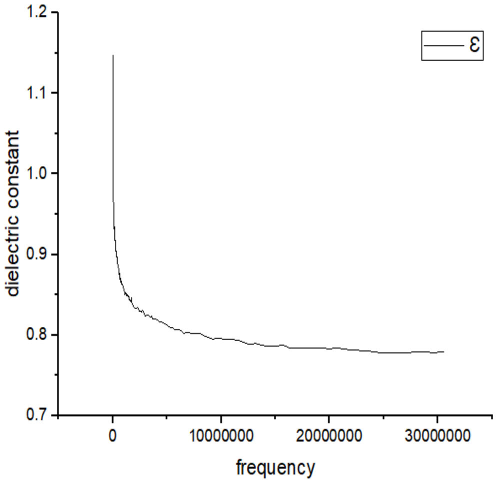
The decrease in dielectric constant with frequency is explained that any effect which contributes to polarization is found to show lagging behind the applied field at higher frequencies. Increasein frequency beyond a certain limit, the electron hopping cannot follow the electric field fluctuations result in a decrease in the dielectric constant. Koop [19] argued that the dielectric constant at low frequency comes from the grain boundaries which due to high resistivity at the grain boundary region have a high dielectric constant. The dielectric constant at high frequency comes from the grains which due to low resistivity have a small value of dielectric constant. It may be concluded that the electrons exchange between Fe^{+2} and Fe^{+3} ions result in the direction of an electric field, that is responsible for electrical polarization of ferrites. At lower frequencies higher value of dielectric constant may be due to dislocations, void sand defects etc.



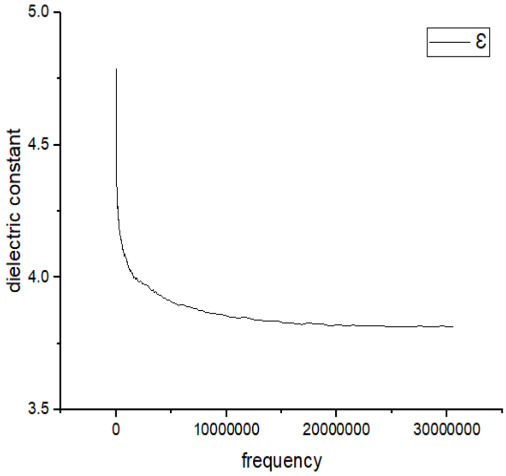
3a $Li_{0.5}Fe_{2.5}O_4$



3b $Li_{0.4}Fe_{2.6}O_4$



3c $Li_{0.3}Fe_{2.7}O_4$



3d $Li_{0.2}Fe_{2.8}O_4$

IV. Conclusion:

This paper presents the preparation of Lithium ferrite nanoparticles sized with the range between 9-25nm. The size of the nanoparticles was measured using XRD technique, the size distribution of prepared nanoparticles was in lower range. The collected data indicates that sol gel method is easy and effective method for the preparation of cobalt ferrite nanoparticle. The result show that Particle size varies from 9.64 nm to 24.71 nm. To find the particle size from X-Ray diffraction pattern, we used Debye Scherrer method. When the values of 'x' are 0.2, 0.3, 0.4 and 0.5 then particle sizes are 9.64, 24.70, 24.71 and 24.70nm respectively. X-Ray diffraction pattern study concluded that D spacing and lattice parameter increase as the value of 'x' decreases. D spacing changes from 2.505 Å to 2.513 Å with the value of 'x' decreases from 0.5 to 0.2. Similarly lattice parameter changes from 8.310 Å to 8.337 Å with the value of 'x' decreases from 0.5 to 0.2. The dielectric constant decrease rapidly with increase in frequency initially and later reaches to a constant value.

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