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A study of the effect of annealing temperature on structural and magnetic properties of $LiFe_5O_8$ (Lithium Ferrite) nanomaterials and synthesized by citrate precursor method

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Abstract: *Lithium ferrite ($LiFe_5O_8$) nanoparticles were synthesized using chemical based Citrate precursor method. The citrate precursor was annealed at temperatures 700°C and 800°C. The annealed powder were characterized using X-ray diffraction and Vibrating sample magnetometer. The average particle size was determined using Scherrer equation and its crystalline size were found to be 56nm and 86nm at annealing temperatures 700°C and 800°C respectively. The magnetic parameters were found different for these two samples. The magnetization, coercivity and retentivity were found 23 emu/g, 135G, 9emu/g at annealing temperature 700°C and 59 emu/g, 55G, 6 emu/g at annealing temperature 800°C respectively.*

Keywords : Ferrite, Nanoparticles, Annealing temperature, Magnetic behaviour.

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Introduction:

Ferrites are mixed oxides of iron having the general formula MFe_2O_4, $MFe_{12}O_{19}$ and MFe_5O_8 where M stands for Zinc, Strontium, Barium, Cobalt, Nickel, Lithium etc (Ishino and Narumiya 1987; West 2007). In last two decades Ferrites have been receiving growing attention because of their various commercial and technological applications in Electronics, Medical sciences, Material technologies etc (Ishino and Naruiya 1987; Smit and Wijn 1959). The spinel structure of ferrites was first proposed by Hilpert (1909) and has been investigated since then in quite detail. Ferrites were prepared in nanocrystalline state for the first time in late eighties (Hilpert1909; Hilpert and Wille 1932). In the nanocrystalline phase, ferrites exhibit properties that are notably different from their bulk phase properties and are strongly dependent on the conditions, size and method of preparation.

Lithium ferrite ($LiFe_5O_8$) has been of great technological interest in many electromagnetic devices for a long time. High electrical resistivity, low eddy current losses, low magnetic losses, and very good thermal and chemical stability make ithium ferrite a material of great importance for microwave applications (Jovic G et al, 2009; Jozef et al 2010). This soft ferrite material with high Curie temperature, square loop properties, and high saturation magnetization has also been used in ferrite core memory system.Thermal stability and safety of lithium ferrite make this material suitable for application in Li-ion batteries.

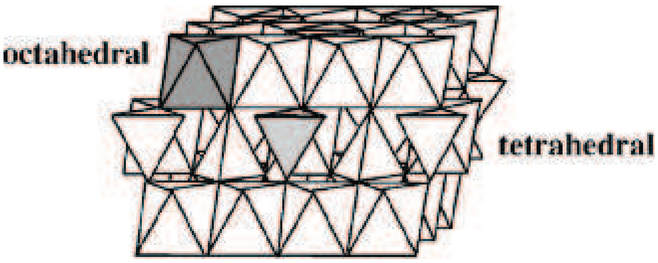

Fig.1: Crystal structure of lithium ferrite

$LiFe_5O_8$ is an inverse spinel with the Li^+ and three-fifth of the Fe^{3+} ions occupying the octahedral B sites of the cubic spinel structure of the general formula AB_2O_4. Because of the peculiar 1:3 ratio of the Li^+ and Fe^{3+} ions in the octahedral sites, there exits two different crystalline forms of lithium ferrite. The á- $LiFe_5O_8$ with space group P4332 [212] has an ordered primitive cubic structure where Li^+ and Fe^{3+} ions are in the ratio 1:3 in the octahedral sites of the cubic spinel structure whereas the â-ferrite phase with space group Fd3m (227) has a disordered face centered cubic structure, in which Fe^{3+} and Li^+ ions are randomly distributed in the octahedral interstices. The ordered phase is obtained by a slow cooling process from above 755ºC, where as the disordered phase can be obtained by quenching from high temperatures. An order-disorder phase transition takes place in the temperature range735-755ºC. Disordered phase also possess several attractive properties which are not seen in bulk lithium ferrite (Nartasa et al 2009; Jozef et al 2010; Ito et al 1998). Important applications of Li-ferrite are in following fields:

Gas sensors - Gas sensors are important in environmental monitoring, home safety and chemical controlling process. Many different semiconducting oxides in bulk ceramic, thick film and thin film form have been studied as a potential sensor element for gas sensing. Spinel type oxide semiconductors (ferrites) are an alternative material for inexpensive and robust detection systems because of good chemical and thermal stability under operating conditions. The sensing mechanism operates through in the change of electrical resistivity resulting from chemical reaction between gas molecules and the metal oxide surface. The surface morphology has an essential role on the sensitivity of solid-state sensors. The nanograined materials offer new opportunities for enhancing the performance of gas sensors because of their high surface to volume ratio and quantum confinement. Li ferrite behaves as an n- type semiconductor, based on the inverse spinel structure, being characterized by high electrical resistivity (10^5-10^9Wcm) and high Curie temperature (640-680^{0}C).

Ferrofluids- ferrofluids are stable colloidal solutions of ferro or ferromagnetic nanoparticles of ~10-nm size dispersed in a liquid carrier. These fluids have diverse technological use in seals, dampers, speakers, lubricants, etc. The most important parameter which controls the magnetic properties of dispersed fine magnetic particles controlling the properties of fluid is magnetization of particles of 10nm diameter in a magnetic fluid. Lithium ferrite nanoparticles in the size range 1nm-15 nm show superparamagnetic behaviour and have adequate properties for applications in low magnetization ferrofluids.

Microwave devices- Lithium ferrite

nanoparticles have significantly lower values of permittivity and permeability. This produces reduction in looses, which makes these ferrites suitable for higher frequency application.

Nanoscale Ferrite Particles: In the last two decades, it is the most important advance metalic material. Ferrimagnetic materials such as ferrites exhibit a substantial spontaneous Magnetization at room temperature just like a ferromagnetic material. Apart from technology and experimental research, developments of theoretical description of magnetic properties of these materials have many advantages. Ferities crystallize in three different crystal types namely, spinal, garnet and magnetoplumbite (Smit and Wijn1959). In crystalline phase, ferrities have exhibited properties that are notably different from their bulk phase properties and are strongly dependent on the conditions and the method of preparation. Ferrities in this phase exhale a substantial spontaneous magnetization at room temperature just like ferromagnetic materials. Thus, new approaches are phenomenon with which superparamagnetism, collective magnetic excitation, random anisotropy, spin caning etc. can be realized, which are not relevant to bulk magnetic materials (Kumar et al 2008). The physical properties of Nanomaterials also differ a lot in many respect when compared to the corresponding bulk material.

Materials and Methods:

Out of several methods that have been used for preparing nanocrystalline samples, we have chosen a citrate precursor method because this method can yield very small particles and also this method is relatively unsophisticated, so that sample preparation using this method is manageable within our own lab. Advantages of precursor technique are -Maximum condition of reactivity, Short time, Low temperature preparation, Homogeneous distribution of ions, Low cost, 100% efficiency, Rigorous control over particle size (Singh et al 2011; Mohamed et al 2010). Stoichiometry detail for synthesizing lithium ferrite nanoparticles is, Mass of lithium nitrate taken=3.497g, Mass of ferric nitrate taken=67.33g, Mass of citric acid taken=57.037g. Aqueous solutions of lithium nitrate, ferric nitrate and citric acid were mixed together and stirred at 60ºC to 80ºC temperature for two hours. Brown slurry so formed is known as precursor. Then the precursor was dried in an oven at a temperature of 80 ºC in an oven till it gets dried. This dried material is the citrate precursor. The half part of the citrate precursor was annealed at 700ºC in a temperature controlled muffle furnace and the other half part was annealed at 800ºC. Annealing, in metallurgy and materials science, is a heat treatment wherein a material is altered, causing changes in its properties such as strength and hardness. It is a process that produces conditions by heating to above the recrystallization temperature, maintaining a suitable temperature, and then slow cooling. Annealing is used to induce ductility, soften material, relieve internal stresses, refine the structure by making it homogeneous, and improve cold working properties. In this work, we have annealed the synthesized material at temperatures $700^{\circ}C$ and $800^{\circ}C$.

These samples were crushed in a crucible and the powdered samples were stored. After that, these samples were characterized through X-Ray diffraction for its crystalline size and purity of Phase. The magnetic Properties of the samples were characterized using Vibrating sample magnetometer (VSM).

Results and Discussion :

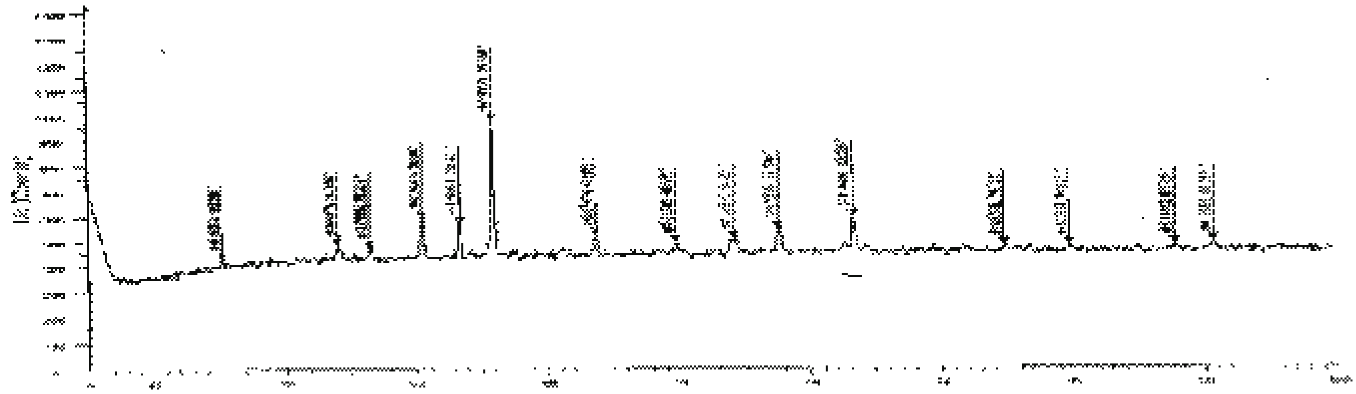

Fig 1. XRD pattern for $LiFe_5O_8$ annealed at 700ºC

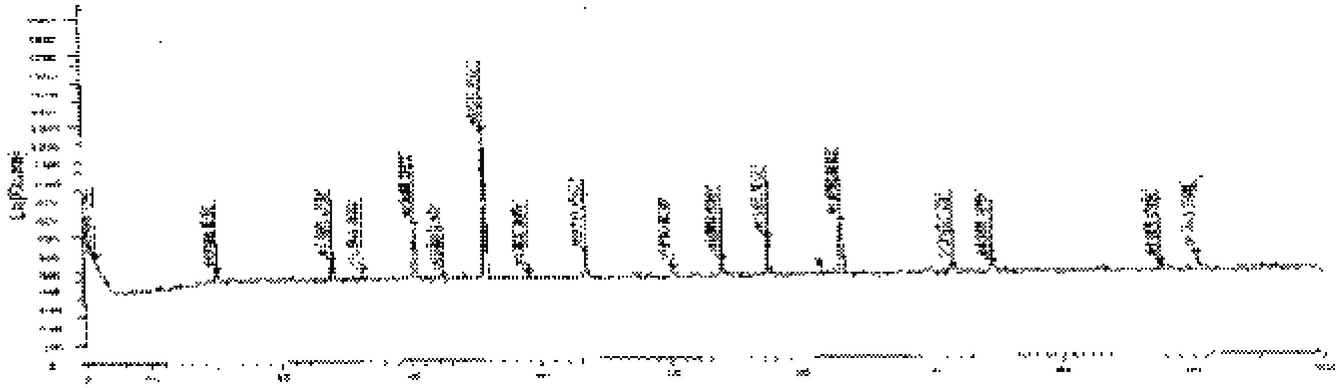

Fig 2. XRD pattern for $LiFe_5O_8$ annealed at 800ºC

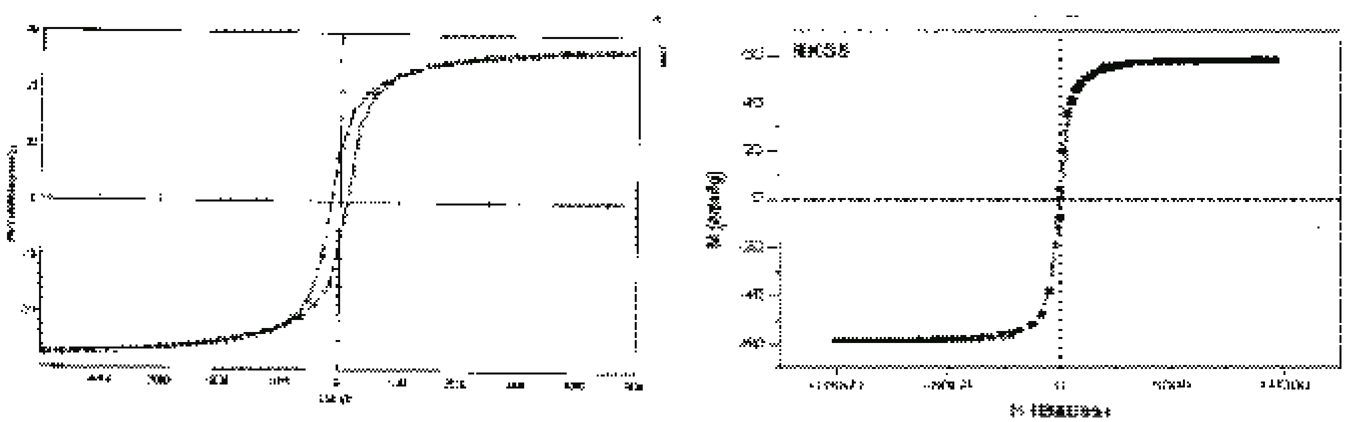

Fig 3. Magnetization curve for Li ferrite Nanomaterial annealed at 700ºC & 800ºC

Table-1. Observed data of XRD and VSM Nanomaterials annealed 700ºC & 800 ºC.

Annealing temp.	Particle size(nm)	Magnetization (emu/g)	Coercivity (G)	Retentivity(emu/g)
700ºC	56	23	135	9
800 ºC	86	59	55	6

The XRD pattern for Lithium ferrite particles annealed at temperatures 700ºC & 800ºC are shown in Fig 1 and 2 respectively .Also, the magnetization curve for these samples are shown in Fig 3 and 4. The X-ray diffraction pattern annealed at 800ºC have better quality as compared to sample annealed at 700ºC. We observed increase in particle size with increase in annealing temperature. Many researchers have observed the similar phenomenon i.e. structural and magnetic behaviour (Tung et al 2002; Singh et al 2009 and Singh et al 2011). The average particle size was calculated using Scherrer's formula (Chatterjee, 2010)and was found 56nm and 86nm (Table 1). The XRD pattern belongs to Lithium ferrite nanomaterial as compared to JCPDS database (JCPDS 1977). The magnetic properties were measured using vibrating sample magnetometer. We have observed that the magnetic properties depends on particle size. Only magnetization increases with increase in the annealing temperature while retentivity and coercivity decrease. $LiFe_5O_8$ behaves as an- type semiconductor based on the inverse spinel structure being characterized by high electrical resistivity (10^5-10^6 Wcm), high Curie temperature (640-680°C) and magnetization (25 emu/g) but in the present work we have observed maximum magnetization 59 emu/g. This is the maximum value of magnetization at this temperature (800°C), not observed earlier using citrate precursor method to the best of my knowledge.

Conclusion :

The synthesized Li- Ferrite samples are in Nanometer range i.e 56nm and 86 nm. Particle size increases with increase in the annealing temperature. Coercivity and retentivity decrease with increase in the particle size. Magnetization is greater for particles having larger size i.e 59 emu/g at annealing temperature 800°C. We observed that as the annealing temperature is increased from 700ºC to 800ºC the particle size increased from 56nm to 86nm which is close to bulk sizes. Thus for synthesizing lithium ferrite nanoparticles using citrate precursor method annealing temperature should be done below 800ºC.

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