# Impact on Structural and Magnetic properties of nano Lithium Cobalt Aluminates by the dopant Fe<sup>3+</sup> synthesized by coprecipitation method, for opto-magnetic applications and for Magnetic refrigeration system

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**Abstract:** Pure and Fe doped nano Lithium-Cobalt Aluminate were synthesized by Co-Precipitation method for the present study. Fe dopant in nano  $Li_{1.0}Co_{0.5}Fe_xAl_{2.x}O$  are in the concentration x=0, 0.5, 1.5 and 2.0. Precipitates are obtained at the pH of 10 and samples are sintered at 1173K before characterization. X-ray diffraction analysis is carried out with X-ray Diffractometer (XRD) to find structural parameters lattice constant, crystallite size, lattice strain, X-ray density and dislocation density. Crystallite size increase with increase in Fe<sup>3+</sup> ion and it lies between 23nm and 57 nm. Lattice constant also increases with increase in dopant and it ranges between 8.1987Å and 8.558Å.Morphology and composition of the prepared samples are observed with Scanning Electron Microscope (SEM) and Energy Dispersive Spectrometer (EDS) respectively. From SEM, morphology of the sample changes from agglomerated to crystalline form on increase in Fe dopant. Fourier Transform Infrared spectroscopy reveals that all the prepared samples shows octahedral. Vibration sample magnetometer (VSM) is used to investigate magnetic parameters Saturation Magnetization  $M_s$  Remanent Magnetization  $M_s$  Coercivity  $H_c$ , and Magneto crystalline Anisotropy K and magneton number. All magnetic parameters increases with increase in iron concentration in the sample. Maximum K value is observed for x=2 and the next value is for x=1.5 and corresponding values are 61561 erg/cm<sup>2</sup> and 17845 erg/cm<sup>2</sup>.

Keywords: Opto-Magnetic, Li-Co-Fe Aluminates, EDS, FTIR, SEM, VSM, XRD

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## I. Introduction

Spinel structure metal oxides are mostly chemically and thermally stable materials. Nano size of their bulk parts has wide range of application such as magnetic, optical, ceramics and catalysis [1]. The general formula of spinels is AB<sub>2</sub>O<sub>4</sub>. In the spinel structure, the anions are arranged in a cubic close packed array with the cations arranged in the holes of the array. It is constructed by 8 tetrahedral and 4 octahedral holes per molecule. Further spinles are classified into normal, inverse and mixed spinles according to the occupancy of cations. In normal spinels A<sup>2+</sup> ions occupy tetrahedral holes and B<sup>3+</sup> ions are present in the octahedral holes. In inverse spinels, one half of  $A^{2+}$  ions occupy tetrahedral holes and the remainder of  $A^{2+}$  ions and all  $B^{3+}$  ions in octahedral holes. In mixed spinel both cations present in tetrahedral and octahedral sites [2]. Among the spinel metal oxides nano spinel Aluminates are most striking material due its high thermal stability, high mechanical resistance, hydrophobicity, and low surface acidity [3]. Doping transition metal Cobalt with spinel aluminates tailors a new variety of normal spinel with aluminium in octahedral sites and cobalt in tetrahedral sites [4] and it has been used as a ceramic blue pigment called as Thenard's blue [5] for its impressive optical property and widely used in the ceramics, glass, paint industry and colour TV tubes as contrast-enhancing luminescent pigment, [6] as well as heterogenous catalyst [7]. Lithium aluminates are also most wanted candidate for counter parts of Lithium batteries and also used as tritium breeding material in fusion reactors [8], it is low cost substitution for garnets. Iron is most abundant metals in earth crust. Incorporating Iron ion with Lithium-Cobalt-Aluminates combination will tailor its structural and magnetic property. This novel composition has potential application in magnetic refrigeration, targeted drug delivery system, and for opto-magnetic application. Present investigation is to know the physio-chemical property of Iron doped Lithium-Cobalt Aluminates to tailor optically and magnetically potential material synthesized by co-precipitation.

#### II. Materials And Methods

# 2.1 Material for synthesis

 $Li_{1.0}Co_{0.5}Fe_xAl_{2-x}O_4$  nano Aluminates at various concentration of iron ion has been synthesized from Precursors LiCl, CoCl<sub>2</sub>, AlCl<sub>3</sub>, FeCl<sub>3</sub>.6H<sub>2</sub>O and NaOH. Analytical grade of these precursors are purchased from SIGMA ALDRICH, Germany with 98% purity.

#### 2.2 Synthesis Methodology

A productive synthesizing methodology depends on starting material, and its composition. Environment of reaction carried out also plays an important role. Wet chemical methods like co-precipitation, sol-gel, hydrothermal and colloid emulsion technique, pH plays an important role. For the present investigation eco friendly NaOH is used to maintain pH. The physio-chemical properties of nanoparticles are greatly influenced by particle size, morphology, purity and chemical composition. Using chemical methods, have been conformed to efficiently control the morphology and chemical composition of prepared nano powder. Among wet chemical techniques sol-gel, hydro thermal and colloid emulsions are time consuming and involve highly unstable alkoxides and difficult to maintain reaction conditions. Co-precipitation is one of the more successful techniques for synthesizing ultrafine nanoparticles having narrow particle size distribution [9]. These advantages on co-precipitation method motivated authors to synthesize  $\text{Li}_{10}\text{Co}_{0.5}\text{Fe}_x\text{Al}_{2x}\text{O}_4$  (x = 0, 0.5, 1.5, 2.0) nano aluminates by co-precipitation method. The precursors for Fe ion is taken as 2 M and 1M for other Metals chlorides. They are mixed in stochiometric ratio and added one by one on the basis of their electro-negativity value, combination of Aqueous solution is stirred rigorously at 338K for 30 minutes, mean while NaOH is added to the brain solution by drop by drop using a burette till solution reaches pH value 10. The required composition of nano ferrites are formed from conversion of metal salt into hydroxide and then transformed into ferrites. The precipitates obtained were thoroughly washed more than three times with double distilled water and acetone. For the present work as synthezied sample and samples sintered at 1173 K are considered for study.

#### 2.3 Physical measurements

Crystal structure of all the samples were examined by powder X-Ray diffraction XRD patterns at room temperature PANalytical-X'Pert PRO powder diffractometer using Cu-K $_{\alpha 1}$  radiation. Scanning Electron Microscopy (SEM) study was performed by VEGA 3 TESCAN Scanning Electron Microscope, operated at 120 KV. Elemental analysis has been done with BRUKER EDS. Fourier Transform Infrared (FT-IR) spectra were recorded on SHIMADZU FT-IR spectrophotometer using KBr pellets in the range 4000-400 cm $^{-1}$ . The magnetic properties were measured at room temperature by LAKESHORE vibrating sample magnetometer (VSM).

# **III.** Result And Discussion

# 3.1 X - Ray Diffraction analysis

X-ray diffraction pattern of synthesized samples of Fe doped Lithium-Cobalt nano spinel Aluminates for the concentration x = 0, 0.5, 1.5 and 2.0 sintered at 1173K are shown in the Fig.1. Major peaks (220), (311), (400), (511), (440) are present in XRD pattern of all the samples which reveals these are having the disordered spinel ferrite formation of space group Fd3m, with Fe3+ in tetrahedral (8c) and octahedral (12d) sites whereas , Li<sup>+</sup> and Co<sup>2+</sup> ions occupy the octahedral position [10]. This matches with the (CoAl<sub>2</sub>O<sub>4</sub> ) JCPDS file No.44-0160, Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> JCPDS file.No.88-0671, CoFe<sub>2</sub>O<sub>4</sub> JCPDS file No. 01-1121, Co<sub>3</sub>O<sub>4</sub> JCPDS file No. 74-2120 [11, 12]. For x= 1.5 minor peak 422 and for other samples 422 is not present this is due to amorphization samples [13]. The extra peak (321) is observed only in x = 1.5 which represents presence of  $\alpha$ -Lithia in the sample. Generally intensity of XRD peaks corresponds to the crystalline nature of the samples. Addition of Iron with Lithium-Cobalt Aluminates increase the crystalline nature of samples it results in increases intensity of XRD peaks. Peaks of XRD pattern shift towards lower diffraction angle, this is due to replacement of lower ionic radius  $A1^{3+}$  ion  $(r_{A1} = 0.51 \text{ Å})$  by higher ionic radius  $Fe^{3+}$  ion  $(r_{Fe} = 0.64 \text{ Å})$  in Li-Co nano Aluminates. The intensities of (220) and (440) planes are more sensitive to cations in tetrahedral and octahedral sites respectively [14, 15]. From TABLE 1 it is clear that intensity of (440) decrease with increasing Fe<sup>3+</sup> and this expressing decreasing Al<sup>3+</sup> ions in the octahedral sites. Intensity found to reverse for the concentration x = 2 this may be due to absence of  $Al^{3+}$  ions in this ratio. Non-linearity in intensity of (220) peak maybe due to replacement of Fe<sup>2+</sup>, Fe<sup>3+</sup> instead of Al<sup>3+</sup> and migration of Co<sup>3+</sup>, Li<sup>+</sup> ions from octahedral site to tetrahedral site vice versa. Average crystallite size 'D' and lattice constant has been estimated from X-ray reflections indexed (220), (311), (400), (422), (511) and (440) using Scherer's equation  $D = 0.9 \lambda / \beta \cos \theta$ , where D is the average crystallite size,  $\beta$  is the full width half maxima,  $\lambda$  is the X-Ray wavelength and  $\theta$  is the Bragg's angle [16]. Lattice constant has been calculated from equation  $a = d (h^2 + k^2 + l^2)^{1/2}$  Where 'a' is lattice constant, d be the inter planar distance, hkl is miller indices. Lattice strain of Li<sub>1.0</sub>Co<sub>0.5</sub>Fe<sub>x</sub>Al<sub>2-x</sub>O<sub>4</sub> (x = 0, 0.5, 1.5, 2.0) nano aluminates were determined using the Williamson-Hall formula  $\varepsilon = \beta / 4 \tan \theta$ , Where  $\varepsilon$  is the

lattice strain of the structure [17, 18]. X-ray Density can be calculated by  $\rho_x = ZM/Na^3$ , Where Z is number of molecules per unit cell, here it is 8. M is Molecular weight of the sample N is Avagadro's Number, 'a' lattice constant. Dislocation density has been found by using the relation  $\delta = 15~\epsilon$  / a D, here  $\delta$  be the dislocation density. All these structural parameters are calculated and tabulated in TABLE 2. The average crystallite size 'D' and lattice constant gets increases with increasing higher ionic radius Fe<sup>3+</sup>ion ( $r_{Fe} = 0.64~\text{Å}$ ) instead of lower ionic radius Al<sup>3+</sup> ion ( $r_{Al} = 0.51~\text{Å}$ ). Increase in Iron in Li-Co nano spinel aluminates increase in average crystallite size linearly and it lies in between 22 nm and 57 nm. Lattice constant also increases with incorporation of Fe ion instead of Al ion and their values increase from 8.1987 Å to 8.5580 Å. These linear variation in 'D' and 'a' reveals that replacement of Al ion by Fe obeys the Vegard's law. Molecular weight of the Li<sub>1</sub>Co<sub>0.5</sub>Al<sub>x</sub>Fe<sub>2-x</sub>O<sub>4</sub> composition increases with this is supported by increase in X-Ray density values obtained from XRD pattern, this is also due to replacement of higher atomic mass Fe<sup>3+</sup> (55.84 gm) by lower atomic mass Al<sup>3+</sup> (26.98 gm). Dislocation density value decreases with increase iron ion.

# 3.2 Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopic (EDS) analysis

External morphology of synthesized samples of  $Li_{1.0}Co_{0.5}Fe_xAl_{2-x}O_4$  (x=0, 0.5, 1.5, 2.0) nano aluminates sintered at 1173 K have been investigated with the help of VEGA 3 TESCAN for all concentration of the samples. Fig.2 shows morphology of  $Li_{1.0}Co_{0.5}Fe_xAl_{2-x}O_4$  nano aluminates samples for the increase in concentration of Fe (x=0, 0.5, 1.5, 2.0). The micrographs show the agglomerated grain structure with clusters of fine particle clinging together due to presence of Lithium and aluminium in the composition for x=0, 0.5, 1.5. The morphology is almost uniform and regular having cubic shaped particles at x=2.0 only. Replacement of Aluminium by Iron manipulated the morphology [19]. Thus results of SEM in agreement with X-Ray diffraction pattern. Especially for x=0 to 1.5 agglomeration is more, this matches with disappearance of reflection planes (111), (222) and (620) due to amorphization and x=0, 0.5, 1.5 morphology shows fine particle nature. The surface of the aluminates samples has a number of fine pores or voids that are attributed to the large amount of Oxygen and chlorine gas liberated during the sintering process. Presence of vacancies results in contraction of Lattice even higher ionic radius dopant is added to the sample. This observation are obtained for x=0, 0.5, 1.5 except x=2.0. [20].

EDS spectrum for the  $\text{Li}_{1.0}\text{Co}_{0.5}\text{Fe}_x\text{Al}_{2\text{-x}}\text{O}_4$  (x = 0, 0.5, 1.5, 2.0) nano aluminates are recorded with BRUKER EDS and illustrated in Fig.3. The result shows each peak corresponds to the element added in the prepared nanoferrite which confirmed the presence of elements in respective concentration. Aluminium and iron are the major constituents in the composition. Oxygen, Lithium and Cobalt are the next major constituent in the sample. Peak for Lithium is not obtained in EDS spectra because Silicon detector used in Energy Dispersive X-Ray spectroscope for present study is not having sensing ability to detect small amount of energy emitted by very small Lithium ion which is having K shell for auger electron. It is interesting to note that the preparation condition completely favours the formation of mixed ferrite and allow us to study the effect of increasing the Fe content on the properties of the Fe doped Li-Co nano Aluminates. The peak values variation is due to it stochiometry, for all the concentration. The values of Aluminium vary with the increase in Iron concentration.

# 3.3. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR spectrums of the investigated sample are shown in Fig.4. Infrared spectroscopy study supported the formation of Fe doped Li Co Al spinel nano Aluminates with informative two strong absorption bands around 400 cm<sup>-1</sup> and 600 cm<sup>-1</sup> that are common features of all spinel structure [21]. The spinel structure are attributed to the stretching vibrations of the unit cell of the spinel in the tetrahedral (A) Site and the metaloxygen vibration in the octahedral (B) site. These absorption bands are highly sensitive to changes in interaction between oxygen and cations, as well as to the size of the obtained nano-particles [22]. The broadening of the spectral band depends on the statistical distribution of cations over A and B sites. The vibration frequency depends on the cation mass, cation-oxygen distance and bending force [23]. From Fig 4 and Table 3, Intrinsic stretching vibration frequency of metal- oxygen at tetrahedral site observed in a range 667 cm<sup>-1</sup> - 592 cm<sup>-1</sup> and its value shifting linearly toward lower frequency with increase in  $Fe^{3+}$  concentration except x=1.5. And replacement of Al<sup>3+</sup> ions by bigger Fe<sup>3+</sup> in octahedral sites also results in a decrease in metal oxygen bond length and consequently decrease the wave number of octahedral and tetrahedral sites by increasing substitution content [24, 25]. Additional subsidiary peak observed at 523 at x= 0.5 concentration. Intensity of the peaks corresponds to octahedral and tetrahedral bonds increases upto x= 0.5 then decreases for 1.5 and 2.0 concentration with the addition of Fe<sup>3+</sup>content. It is well known that the intensity ratio is function of change of dipole moment with the internuclear distance. This value represents the contribution of ionic bond Fe-O in the lattice. So the observed increase and decrease in the absorption band intensity with increase in Fe content, is due to perturbation occurring in Fe-O bonds. The electronic distribution of Fe-O bonds greatly affected by the dopant Fe<sup>3+</sup> which is having comparatively bigger radius and high atomic weight Al (26.98amu) and Fe (55.84 amu).

# 3.4 Vibrational Sample Magnetometer (VSM) analysis

Fe doped  $Li_{1.0}Co_{0.5}Fe_xAl_{2-x}O_4$  nanoaluminates (x = 0, 0.5, 1.5, 2.0). Hysteresis loops for the samples are recorded with LAKESHORE Vibrational sample magnetometer at 300 K with applied field as 20 KOe are shown in Fig.5. The value of anisotropy constant was calculated from Stoner-Wohlfarth relation as follows  $H_c = K/M_s$  [26] ,Where  $H_c$  is the coercivity,  $M_s$  saturation magnetization and K magnetic anisotropy constant .Calculation of magnetic moment in bohr magneton was carried out using the following relation,  $n_B = (Molecular Weight \times M_S)/5585$  [27]. Magnetic parameters saturation magnetization  $(M_s)$ , Remanence Magnetization (M<sub>r</sub>), Coercivity (H<sub>c</sub>), Squareness ratio, Magnetic anisotropy constant (K) and magneton number are calculated from Hysteresis loop and tabulated in Table 4.Generally magnetic properties in the prepared sample arise from coupling between spin and orbital angular momentum (L-S coupling) and electron spin (S-S coupling) [28]. In the case of spinel nano magnetic ferrite material magnetic parameters are influenced by cation distribution, collinearity and non collinearity (canting) of spins on their surface, Crystallite size and dopant. In present study undoped nano Li<sub>1.0</sub>Co<sub>0.5</sub>Al<sub>2</sub>O<sub>4</sub> have the hysteresis loop of Paramagnetic nature of the sample. Small amount of the dopant Fe in Li-Co aluminates shows 'S' shaped superparamagentic behaviour. Low amount of Al in x = 1.5 resembles behavior of ferri nature. For x = 0 and 0.5 samples magnetization values increases upto infinity. The magnetization increases with increase in the field up to 1000 Oe and then increase slowly and saturates at nearly 2500 Oe of the applied magnetic field for x = 1.5 and 2.0. From the Fig.5 mixed spinel having highest Saturation magnetization value i.e 135 emu/g and 51 emu/g for the concentration x = 2.0and 1.5 respectively due to most disordered spinel structure, which will in agreement with XRD and SEM results. Coercivity varies linearly due to the influence of Fe<sup>3+</sup> ion in the sample and variation in crystallite size. High value of coercivity is observed for higher concentration of iron ion for all other concentrations. Coercivity and saturation magnetization value is increases with increase in Fe in the Li-Co nano aluminates. This is due to more number of incorporation of iron ion on the sublattices. The appearance of super-paramagnetism indicates that the magneto crystalline anisotropy, which is important to hold magnetic ions in certain direction, has been overcome by thermal energy [29]. The magnetic anisotropy constant is maximum for the concentration 2.0 and 1.5 with more crystallinity. From these results magneto crystalline anisotropy is more for high crystallinity sample. And high crystallinity is possible in disordered spinel structure. Magneton number also maximum for those concentration x = 2.0 and 1.5. Ordered spinel structure have low value of magnetic anisotropy constant. No sample in the present study have ordered structure. Thus VSM result matches with XRD, SEM and FTIR findings.

## IV. Conclusion

 $Li_{1.0}Co_{0.5}Fe_xAl_{2.x}O_4$  (x = 0, 0.5, 1.5, 2.0) nano aluminates were effectively synthesized by trouble-free and uncomplicated co-precipitation method with average crystallite size between 22 nm and 57 nm. Physiochemical properties are studied for samples sintered at 1173K. Addition of Fe ion in Li-Co Aluminate influenced the structural properties such as Average crystallite size, lattice constant, and X-Ray density .Their values increases with increase in Fe content in the sample. Lattice strain and dislocation density of the synthesized samples decrease increase in Fe. SEM shows morphology manipulated by Fe<sup>3+</sup> ion in the sample and FTIR observation have fine match with results of XRD and SEM. EDS reports shows the presence of percentage of elements present in composition. Pauli Paramagnetic aluminium in nano Li-Co aluminates replaced by iron ion converts the composition into superparamagnetic samples which could be revealed from the VSM results which will be a great candidate for opto magnetic applications and for magnetic refrigeration. At higher concentration Iron induced most disordered structure which is found from the magnetic anisotropy constant value. Important finding in this work is 1.5 and 2.0 concentration of Iron in Li-Co nano ferrialuminates has its maximum magnetic anisotropy constant value, which will release more thermal energy in alternating magnetic field, and should be act as best candidate for magnetic fluid hyperthermia and in targeted drug delivery system.

#### References

- [1] Duan.X, Pan.M, Yu.F, Yuan.D, J.Alloys Compd. 509 (2011) 1079-1083.
- [2] Sepleak.V, Bergmann.I, Indris.S, Feldhoff.A, Hahn.H, Becker.K.D, J.Mater. Chem 21 (2011) 8332
- [3] Dhak.D, Pramanik.P, J.Am.Ceram.Soc. 89 (2006) 1014 -1021.
- [4] Zayat.M, Levy.D, Chem.Mater. 12 (2000) 2763
- [5] Llusar.M, Fores.A, Badenes.J.A, Calbo.J, Tena.M.A, Guillermo.M, J.Eur.Ceram.Soc 21 (2000) 2763
- [6] Busca.G, Lorenzelli.V, Escribano.V.S, Guidetti.R, J.Catal 131 (1991) 167.
- [7] Renoult.O, Boilot.J.P, Korb.J.P, Petit.D, Boncoeur, J. Nucl.Mater. 219 (1995) 233.
- [8] B.K.K.uanr, G.P.Srivastava, J.Appl. Phys. 75 (1994) 6115
- [9] Marinca.T.F, Chicinas.I, Isnard.O, Pop.V, Pop.F, J.Alloy., and compounds, 509 (2011) 7931-7936.
- [10] Dhage.S.R, Khollam.Y, Deshpande.S.B, Ravi.V, Mater.Res.Bulls. 378 (2003) 1601-1605.
- [11] Carta.G, Casarin.M, Habra.E.L, Electrochemica Acta 50 (2005) 4592-4599.
- [12] Bahgat.M, Farghaly.F.E, Abdel Basir, Fouad.A.O, J. Mate.Process. Tech. 183 (2007) 117-121
- [13] Ammankutty.C.G, Sugunna. S, J.App.Catal.A Gen 218 (2001) 39

- [14] Ladgaonkar.B.P, Vaigainkar.A.S, Materials chemisty and physics 56 (1998) 280-283.
- [15] Narasimhan.C.S, Swamy.C.S, Physica status dolidi 59 (1980) 817
- [16] Culity B.D, Elements of X-ray diffraction Chapter 14 (London, Addison-Wesley publishing company Inc., 1976) (Chapter 14).
- [17] Wolska. E, Piszora. P, Nowicki.W, Darul. J, Int.J.Inorganic Mater., 3 (2001), 503-507.
- [18] Williamson.G.K., Hall.W.H, Acta Metall.1(1953) 22-31.
- [19] Rahman.S, Nadeem.K, Rehman.M.A, Mumtaz.M, Naeem.S, I.L.Ipst, Ceram.Int. 39 (2013) 5235-5239
- [20] Prabahar. S, Dhanam. M, J.Cryst.Growth, 41 285 (2005) 1-2
- [21] Hashim.M, Alimuddin, Shirsath.S.E, Kumar.S, Kumar.R, Roy.A.S, Shah.J, and Kotnala.R.K, Journal of alloys and compounds, 549 (2012) 348-357.
- [22] Farid.M, Ahmad.I, Aman.S, Kanwal.M, Murtaza.G, Alia.I, Ishfaq.M, Journal of Ovonic research 11 (2015) 1
- [23] Wahba.A.M, Mohammad.M.B, Ceramic International, 40 (2014) 6127-6135
- [24] Boshale.A.G, Chougule.B.K, Material chemisty and physics 97 (2006) 273
- [25] Smit.J, And H.P.J ijn.Ferrite (1959)
- [26] Singh.N, Agrawal.A, Sanghi.S, Singh.P, Physica B, 406 (2011) 687
- [27] Singhal.S, Chandra.K, J.Solid state Chem. 180 (2007) 296
- [28] Gabbal.M.A, Angari.Y.M.A, Material chem. Physics 118 (2009) 153
- [29] Verma.A, Goel.T.C, Meadiratta.R.G, Kishan.P, Journal of Magnetism and magnetic material 208 (2000) 13-19.

**TABLE 1:** Comparison of X-Ray Intensity

| Tibel 1. Comparison of 11 Tay intensity |                                                                                        |           |       |  |  |
|-----------------------------------------|----------------------------------------------------------------------------------------|-----------|-------|--|--|
| Fe                                      | Composition                                                                            | $I_{440}$ |       |  |  |
| content 'x'                             |                                                                                        |           |       |  |  |
| 0                                       | Li <sub>1.0</sub> Co <sub>0.5</sub> Al <sub>2</sub> O <sub>4</sub>                     | 49.42     | 49.39 |  |  |
| 0.5                                     | Li <sub>1.0</sub> Co <sub>0.5</sub> Al <sub>1.5</sub> Fe <sub>0.5</sub> O <sub>4</sub> | 37.66     | 48.09 |  |  |
| 1.5                                     | Li <sub>1.0</sub> Co <sub>0.5</sub> Al <sub>0.5</sub> Fe <sub>1.5</sub> O <sub>4</sub> | 49.71     | 35.25 |  |  |
| 2                                       | Li <sub>1.0</sub> Co <sub>0.5</sub> Fe <sub>2</sub> O <sub>4</sub>                     | 17.79     | 46.89 |  |  |

**TABLE 2:** Structural parameters of Li<sub>1.0</sub>Co<sub>0.5</sub>Fe<sub>x</sub>Al<sub>2-x</sub>O<sub>4</sub> (x=0.0, 0.5, 1.5, 2.0) sintered at 1173K

| Fe content | Composition                                                                            | Crystallite | Lattice  | Molecular | X-ray             | Lattice          | Dislocation |
|------------|----------------------------------------------------------------------------------------|-------------|----------|-----------|-------------------|------------------|-------------|
| 'x'        |                                                                                        | Size        | Constant | Weight    | Density           | Strain           | Density     |
|            |                                                                                        | D (nm)      | a (Å)    | g/mole    | g/cm <sup>3</sup> | 10 <sup>-3</sup> | $10^{15}$   |
| 0          | Li <sub>1.0</sub> Co <sub>0.5</sub> Al <sub>2</sub> O <sub>4</sub>                     | 22.73       | 8.1987   | 154.32    | 3.719945          | 4.614            | 4.0805      |
| 0.5        | Li <sub>1.0</sub> Co <sub>0.5</sub> Al <sub>1.5</sub> Fe <sub>0.5</sub> O <sub>4</sub> | 25.27       | 8.2409   | 168.76    | 4.005851          | 5.138            | 5.8532      |
| 1.5        | Li <sub>1.0</sub> Co <sub>0.5</sub> Al <sub>0.5</sub> Fe <sub>1.5</sub> O <sub>4</sub> | 42.45       | 8.4233   | 197.62    | 4.392718          | 3.206            | 2.9417      |
| 2          | Li <sub>1.0</sub> Co <sub>0.5</sub> Fe <sub>2</sub> O <sub>4</sub>                     | 56.8        | 8.558    | 212.05    | 4.494389          | 2.189            | 0.9119      |

**TABLE 3:** Vibrational frequency of tetrahedral and octahedral sites

| Fe content 'x' | Composition                                                                            | $v_{1 \text{ tetra}}$ | V <sub>1 tetra</sub> | V <sub>2 octa</sub> |  |
|----------------|----------------------------------------------------------------------------------------|-----------------------|----------------------|---------------------|--|
|                |                                                                                        | cm <sup>-1</sup>      | cm <sup>-1</sup>     | cm <sup>-1</sup>    |  |
| 0              | Li <sub>1.0</sub> Co <sub>0.5</sub> Al <sub>2</sub> O <sub>4</sub>                     | 666.96                |                      | 541.68              |  |
| 0.5            | Li <sub>1.0</sub> Co <sub>0.5</sub> Al <sub>1.5</sub> Fe <sub>0.5</sub> O <sub>4</sub> | 654.31                | 523.16               | 419.3               |  |
| 1.5            | Li <sub>1.0</sub> Co <sub>0.5</sub> Al <sub>0.5</sub> Fe <sub>1.5</sub> O <sub>4</sub> | 592.11                |                      | 418.54              |  |
| 2              | Li <sub>1.0</sub> Co <sub>.0.5</sub> Fe <sub>2</sub> O <sub>.4</sub>                   | 598.44                |                      | 467.03              |  |

**TABLE 4:** Magnetic parameters of  $Li_{1.0}Co_{0.5}Fe_xAl_{2-x}O_4$  (x = 0.0, 0.5, 1.5, 2.0) sintered at 1173K

| Fe content | Composition                                           | Saturation   | Remanent      | Coercivity | Mr/Ms   | Magneto     | Magneton    |
|------------|-------------------------------------------------------|--------------|---------------|------------|---------|-------------|-------------|
| 'x'        |                                                       | Magnetizatio | Magnetization | $H_c$      |         | Crystalline | Number      |
|            |                                                       | n            | $M_{\rm r}$   | (Oe)       |         | Anisotropy  | $n_{\rm B}$ |
|            |                                                       | $M_s$        | (emu/g)       |            |         | K (erg)     |             |
|            |                                                       | (emu/g)      |               |            |         |             |             |
| 0          | Li <sub>1.0</sub> Co                                  | 0.6369       | 0.0219        | 3.497      |         |             |             |
|            | $_{0.5}\mathrm{Al}_{2}\mathrm{O}_{4}$                 |              |               |            | 0.03439 | 2.227239    | 0.017598    |
| 0.5        | Li <sub>1.0</sub> Co <sub>0.5</sub> Al <sub>1.5</sub> | 0.5916       | 0.1025        | 322.08     |         |             |             |
|            | $Fe_{0.5}O_4$                                         |              |               |            | 0.17326 | 190.5425    | 0.017876    |
| 1.5        | Li <sub>1.0</sub> Co <sub>0.5</sub> Al <sub>0.5</sub> | 51.832       | 28.69         | 344.29     |         |             |             |
|            | $Fe_{1.5}O_4$                                         |              |               |            | 0.55352 | 17845.24    | 1.834027    |
| 2          | Li <sub>1.0</sub> Co                                  | 135.196      | 59.48         | 455.35     |         |             |             |
|            | 0.5Fe <sub>2</sub> O <sub>4</sub>                     |              |               |            | 0.43995 | 61561.5     | 5.133091    |

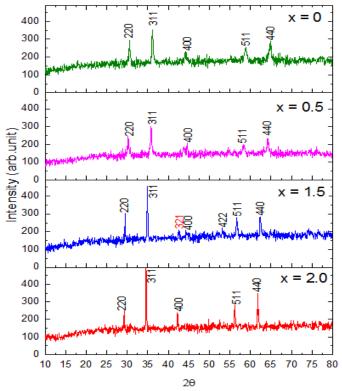
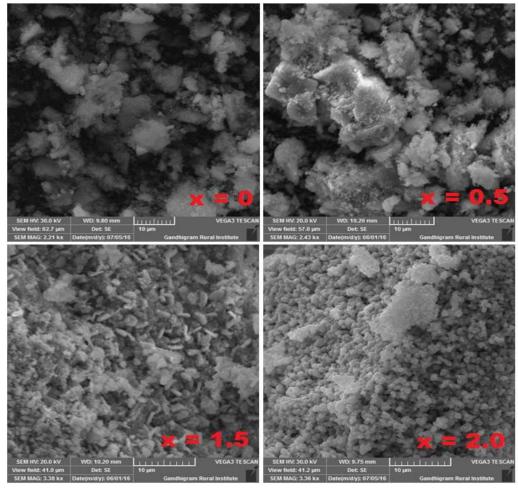
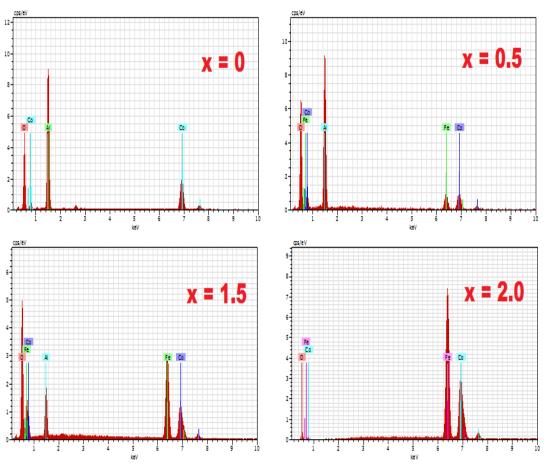


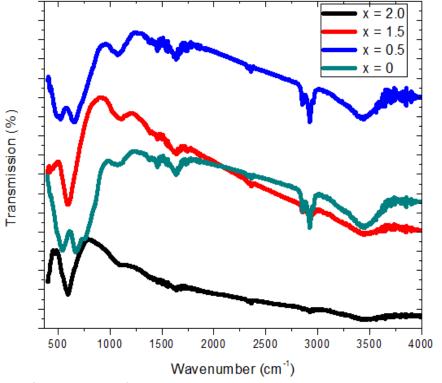
Figure 1: X-Ray Diffraction pattern of Li<sub>1.0</sub>Co<sub>0.5</sub>Fe<sub>x</sub>Al<sub>2-x</sub>O<sub>4</sub> (x=0.0, 0.5, 1.5, 2.0) sintered at 1173K



**Figure 2.** SEM micrograph of  $Li_{1.0}Co_{0.5}Fe_xAl_{2-x}O_4$  (x=0.0, 0.5, 1.5, 2.0) sintered at 1173K



**Figure 3:** EDS of  $Li_{1.0}Co_{0.5}Fe_xAl_{2-x}O_4$  (x=0.0, 0.5, 1.5, 2.0) sintered at 1173K



**Figure 4:** FTIR spectra of  $Li_{1.0}Co_{0.5}Fe_xAl_{2-x}O_4$  (x=0.0, 0.5, 1.5, 2.0) sintered at 1173K

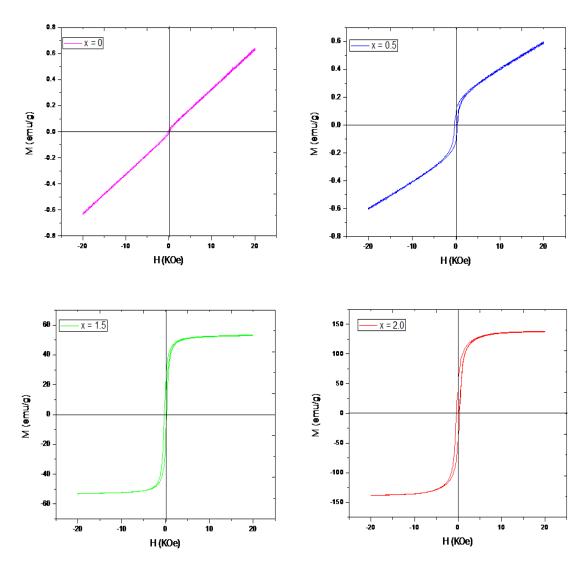


Figure 5: Magnetic hysteresis curves of  $Li_{1.0}Co_{0.5}Fe_xAl_{2-x}O_4$  (x=0.0, 0.5, 1.5, 2.0) sintered at 1173K

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