# Structural, properties of non Stoichiometric Lithium substituted Magnesium ferrite nanoparticles for multifunctional applications.

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### Structural analysis :XRD, SEM, FTIR and EDAX measurement

Fig 1.1 Illustrates the XRD pattern of the materials,  $(Mg_{0.5+x} Li_{1.2x}Fe_2O_4)$  with ( x=0.00, 0.15 and 0.35). The prepared materials were scanned in the range of 2 $\Theta$  from 20° to 70° and found seven intense characteristics peaks having hkl value as (220), (311), (222), (400), (422), (511) and (440) respectively. The elemental reflections from the planes of (311), (400), (511), (440) are prominent peaks. Reflections from the others plane (422), (220) was noticed with weak intensities. However, the presence of the other intermediate phases at around 33 degree belongs to  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. The strain value is increased and corresponding crystallite size also increases which is due to the presence of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> ,which might be the reason for such abnormal behavior of materials. This  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> may be removed by increasing duration hour of annealing or annealing on some other higher temperature. This is our future research work. The most prominent intense peak (311) was chosen for average crystalline size and other parameter, such as lattice constant, interplanner distance (d), unit cell volume which are shown in figure 1.3 table-1. The lattice parameter for cubic spinal phase is determined using following formula

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

And d-spacing was found using Bragg's law  $\lambda = 2d_{hkl}sin_{hkl}\Theta$ 

Williamson hall plot is shown in figure 1.2(a-c) and table 1.2 is used evaluate the crystalline size and strain broadening using the differential  $\Theta$  position.

$$\beta_{hkl} = \left[\frac{k\lambda}{D}\right] + 4\varepsilon \sin\Theta$$

and comparing it to standard straight line equation

#### y=mx+c

W-H plot shows that crystalline size are 24nm, 78nm and 50nm and lattice constant found were 8.400 Å, 8.392 Å and 8.420 Å respectively. Thus crystalline size increases (24nm-78nm) then decrease(50nm) as to molar ratio of  $Li^{+1}$  ion are 0.00, 0.15 and 0.35. But the lattice constant was found to decrease and again increases. This is may be due to bigger ionic radius and non-molar concentration of Li ion and Mg ion (Ionic radius of Li is 0.76 Å and Mg is 0.72 Å). Lattice strain was found to increase as the molar ratio of Li ion increases. The cell volume are function of crystalline size(table-1).

This non-stoichiometry, different ionic radii in this material causes the crystalline growth mechanism differently (table-1, fig-1), while all the materials are annealed at same temperature 550°C for same period of 2hr. Tetrahedral-octahedral sites (A-B) site interactions and properties of the materials also gets affected. Such strain is crystal defect may causes agglomerations at surfaces of materials obtained by SEM, shown in figure 1.4 (a-c). The another reason of agglomeration nanoparticles may be due to exchange interaction and high surface area. The grain size of different non-molar ratio of materials are in the different range of micrometer of nanocrystallites of different size. The EDS measurements shown in figure 1.5 (a-c).shows the quantitative chemical analysis of the materials. This measurements confirmed presence of Mg, Fe and O . No other impurity peak of other element was obtained and confirming the synthesis of non-stoichiometric magnesium substituted lithium ferrite. Since EDS detector cannot detect lighter element (Li) due to its smaller atomic nuclei. It has very low characteristic radiation which is very difficult to detect [28].The atomic percentage and weight percentage elements are determined, which are shown in Fig 1.5 (a-c).

# **FTIR measurement**

The FTIR spectrum shown in fig 1.6 using Perkin Elmer FTIR spectrometer in wavenumber (1000-400) cm<sup>-1</sup>range. The first high frequency absorption band which is denoted by  $v_1$  lies in the range (580–600) cm<sup>-1</sup> and the other low frequency denoted by  $v_2$  in the (400–436 cm<sup>-1</sup>) range. This is generally attributed to the vibration occurring in tetrahedral and octahedral sites in cubic spinel ferrites . An absorption peak occurred in the octahedral region around 435 cm–1. The peak intensity increase with increasing the lithium concentration and absorption slightly shifted as well which might be due to lithium ions distribution in the octahedral sites. The other band lying in

the region of 580 cm<sup>-1</sup> is due to the interaction of  $(Mg^{2+}-O^2)$  and  $(Fe^{3+}-O^2)$  range this is being resulted due to octahedral and tetrahedral sites of present sample which give rise to single phase cubic spinal structure having space group of  $Fd\overline{3}m$ .

There are no more significant absorbance peaks available in the range. The position of wave number and absorption bands of prepared materials may be responsible to changes in exchange interaction forces between cations and oxygens as well as crystalline size of the sample. As a results magnetic properties, optical properties and Curie temperature was found to change



**Fig- 1.1** XRD result for Mg<sub>0.5+x</sub>Li<sub>1-2x</sub>Fe<sub>2</sub>O<sub>4</sub>different concentration having  $0 \le x \le 0.35$ .



(b) W-H plot of Mg<sub>0.85</sub>Li<sub>0.7</sub>Fe<sub>2</sub>O<sub>4</sub>



**W-H plot of Mg**<sub>0.65</sub>Li<sub>0.3</sub>Fe<sub>2</sub>O<sub>4</sub> Figure 1.2(a-c)- W-H plots of Mg<sub>0.5+x</sub>Li<sub>1-2x</sub>Fe<sub>2</sub>O<sub>4</sub>different concentration having  $0 \le x \le 0.35$ .



Fig. 1.3 Shift of highest intensity peak brag's angle (311) in the XRD pattern which clearly indicates the change in lattice parameter with the decrement of  $Li^{+1}$  substitution.



Fig: 1.4 (a) SEM images of Mg<sub>0.5</sub>LiFe<sub>2</sub>O<sub>4</sub>



Fig: 1.4(c) SEM images of Mg<sub>0.85</sub>Li<sub>0.3</sub>Fe<sub>2</sub>O<sub>4</sub>



Element	App Conc.	Intensity	% Weightage	% Atomic
O K	33.52	1.842	27.29	53.53
Mg K	2.90	0.5656	7.68	9.92
Fe K	39.74	0.9166	65.03	36.55

Fig 1.5(a) EDX image of Mg 0.5LiFe2O4



Fig: 1.5(b) EDX image o Mg 0.65Li 0.7 Fe 2O4



Element	App Conc.	Intensity	% Weightage	% Atomic
O K	37.77	1.8153	24.30	49.53
MgK	3.99	0.5625	8.27	11.10
Fe K	53.28	0.9227	67.43	39.37
тск	33.20	0.7221	07.43	57.51

Fig:1.5© EDX image o Mg 0.80 Li 0.3 Fe2O4



Fig- 1.6 FTIR Of  $Mg_{0.5+x}Li_{1-2x}Fe_2O_4$ different concentration having  $0 \le x \le 0.35$ 

Parameter	At x=0	At x=0.15	At x=0.35
d-spacing (Å)	2.54002	2.52905	2.53655
2 <del>O</del> (degree)	35.36811	35.46599	35.35772
Lattice Constant	8.400 Å	8.3921 Å	8.4207 Å
Crystalline size	24 nm	78 nm	50 nm
Cell Volume (a=b=c) Å <sup>3</sup>	592.704	591.033	597.096

**Table 1-** Structural parameter for  $Mg_{0.5+x}Li_{1-2x}Fe_2O_4$  different concentration having  $0 \le x \le 0.35$ . Using Xpert Highscore software.

# Table 1.2 W-H plot parameter

Sample Name	Crystallite Size (nm)	Lattice Strain
Mg <sub>0.5</sub> LiFe <sub>2</sub> O <sub>4</sub>	25 (±1 nm)	0.00129
$Mg_{0.65}Li_{0.7}Fe_2O_4$	77 (±1 nm)	0.00239
$Mg_{0.85}Li_{0.3}Fe_2O_4$	51 (±1 nm)	0.00104