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Structural and magnetic studies of magnesium substituted nickel ferrite nanoparticles by citrate precursor method

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Magnesium substituted nickel ferrite nanoparticles are prepared by using citrate precursor method. Synthesized nanoparticles are analyzed by X-ray diffraction (XRD), Scanning electron microscope (SEM), Energy Dispersive X-ray (EDX) Analysis, Fourier transform infrared (FTIR) and Vibrating sample magnetometer (VSM). The crystalline size of nanoparticles is estimated to be 17-70 nm by using Debye Scherrer formula, Modified Scherrer formula and Williamson-Hall method. Two main peaks near 570-590 cm⁻¹ and 400-470 cm⁻¹ confirm the spinel structure. Substitution of Mg²⁺ ion increases the saturation magnetization value up to x = 0.4 after that decreases. Yafet- Kittel angle value increases with increasing Mg²⁺ concentration.

Key words: Scherrer formula, lattice constant, bond length, Curie temperature, Yafet-Kittel angle, Tetrahedral.

Introduction

Nanocrystalline ferrites are, in general, most alluring on account of their fascinating physical, electrical, optical and magnetic properties. The spinel ferrites have numerous technical applications. To figure out a few, in ferro fluids [1], drug delivery [2], photoelectric devices [3], nano devices [4], catalysis [5], sensors [6], microwave devices [7, 8] and magnetic pigments [9].

It is of interest to note that nickel ferrite has inverse spinel structure with Ni²⁺ ions in octahedral site (B site) and Fe³⁺ ions are in tetrahedral and octahedral site (A and B site). Nickel ferrite is one of the most extensively studied magnetic materials used in numerous applications such as magnetic fluid, microwaves devices, fabrication of radio frequency coils, magnetic recording media etc., [10]. Bulk MgFe₂O₄ is an inverse spinel with Mg²⁺ ions occupying all the octahedral sites [11]. But according to Pradeep et al (2008) new cation distribution, Mg²⁺ ions prefer both tetrahedral and octahedral sites, i.e. nano-MgFe₂O₄ tends to exist as mixed spinel. This degree of inversion is thus attributed to the method of synthesis and is ultimately an impact of nano regime.

In literature very few studies of Mg substituted Ni ferrite are present. Manojit De et al. (2015) and Abdul Gafoor et al (2014) have prepared $Ni_{1-x}Mg_xFe_2O_4$ (x = 0.0 to 1.0) by auto combustion method and characterized through XRD, SEM, EDS, Raman and FTIR. To the best of our knowledge, the magnetic properties and the particle size evaluated by modified Scherrer formula and W-H method of Mg-Ni ferrites by sol-gel have not often been reported.

The present work focuses on the synthesis of Mg substituted nickel ferrite using sol-gel method and study on the effect of Mg^{2+} on the structural and magnetic properties using XRD, SEM, EDX, FTIR and VSM techniques.

Material and Methods

 $Ni_{1-x}Mg_xFe_2O_4$ ($0 \le x \ge 1.0$) ferrites are carried out in the presence of citrate and nitrate using a citrate precursor method. All high purity AR grade (99.99%) chemicals, such as nickel nitrate (Ni(NO₃)₂.6H₂O), magnesium nitrate (Mg(NO₃)₂.6H₂O), ferric nitrate (Fe(NO₃)₂.9H₂O) and citric acid (C₆H₈O₇.H₂O) are used as received without any purification.

All the reagents are weighed into stoichiometric portions. In the metal nitrate and citrate ratio is 1:3. Nickel nitrate, ferric nitrate and magnesium nitrate are dissolved in deionized water. Citric acid is combined with the metal nitrate solution. The pH of the resulting solution is 7 with an addition of ammonium hydroxide. The beaker is kept on a hot plate with continuous stirring at 100 °C. During evaporation, the solution changes to a sticky solution and at last to a thick viscous gel. Subject to the exclusion of all water molecules from the mixture, the gel automatically burns with glowing flints yielding ash. The decay reaction is continued until the whole citrate complex is consumed. The time required to entire the ignition is about one minute, yielding a black ash. The experiment is continued with different concentrations (x = 0.0 to)1.0) of Mg²⁺ doping on NiFe₂O₄. The as-prepared ferrites are heat treated separately at 500 °C for 4 hours

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so as to get the impurity-free desired products.

The phase-purity and crystal structure of these ferrites are investigated through X-ray diffraction (XRD) spectroscopy, obtained on an Panalytical X'pert powder X'Celerator diffractometer with Cu-Ka radiation ($\lambda = 1.54$ Å) in the 2 θ range of 20 ° to 80 °. The morphology of the as-prepared ferrites is confirmed with the help of Carl Zeiss EVO18 scanning electron microscope (SEM). Identify the elements present in the prepared nanoparticles using Quantax 200 with X-Flash-Bruker energy dispersive X-ray spectrometer (EDS). Fourier transform infrared spectroscopy (FTIR: Schimadzu) transmittance spectra of wavenumber range of 400-4000 cm⁻¹ are obtained. At room temperature, vibrating sample magnetometer (VSM: Lake Shore, Model 7410) are used to measure the magnetic measurements of the Ni_{1-x}Mg_xFe₂O₄ ferrites.

Results and Discussion

XRD patterns of Ni_{1-x}Mg_xFe₂O₄ ferrites obtained for x = 0.0 to 1.0, are shown in Fig. 1. All compositions indexed the cubic spinel single phase structure. No traces of impurity peaks indicated the purity of the samples. The average crystallite size is calculated for all the samples using Scherrer formula [12],

$$D = (0.9\lambda)/(\beta \cos\theta) \tag{1}$$

Where D is particle size, λ is the x-ray wavelength, β_D is the full width at half maximum (FWHM) and θ is the Bragg angles for the actual peak.

The crystallite size is estimated using modified Scherrer formula [13]

$$\ln \beta = \ln 0.9 \lambda / (\text{D}\cos\theta) = \ln (0.9 \lambda / \text{D}) + \ln (1/\cos\theta) \quad (2)$$

Plot the results of $\ln \beta$ versus $\ln (1/\cos\theta)$, then a straight line with a slope of around one and an intercept of about $\ln (0.9\lambda/D)$ to be obtained. After getting the intercept, the exponential of the intercept has been obtained.

$$\exp(\ln(0.9\lambda/D)) = (0.9\lambda/D)$$
(3)

The value of D in nanometre can be calculated with $\lambda = 1.54$ Å. Fig. 2 indicates six plots of ln β versus ln(1/ cos θ) for different Mg²⁺ concentrations. At x = 0, the linear regression plot is obtained as y = 0.72x-5.781.







Fig. 2. Linear plots of modified Scherrer formula and obtained intercepts for Ni_{1-x}Mg_xFe₂O₄.

			Ŀ	D (nm)				
S.No	Composition	a (Å)	d_x (gm/cm ³)	Scherrer formula	Modified Scherrer formula	Williamson-Hall method		
1	NiFe ₂ O ₄	8.344	5.362	48	44	46		
2	$Ni_{0.8}Mg_{0.2}Fe_2O_4$	8.350	5.192	52	69	67		
3	$Ni_{0.6}Mg_{0.4}Fe_2O_4$	8.354	5.029	39	41	42		
4	$Ni_{0.4}Mg_{0.6}Fe_2O_4$	8.364	4.854	29	36	29		
5	$Ni_{0.2}Mg_{0.8}Fe_2O_4$	8.365	4.853	23	19	17		
6	$MgFe_2O_4$	8.377	4.521	30	43	34		
		y=0.003x+7E-5	and a second sec	y=0.00206x-4.94E-5	y=0.00334x-6.53E	-5		
		(a)		(b)	(c)			
	0.03- 0.00- 9.009- 0.09- 0.09-	y=0.0047x-7.27E-4	0004 0002 0000 0000 0000 0000	y=0.008x+0.0022	0005 y=0.0004x-70 0005 0005 0005	54. 		
	0000 10 12 14	4sin0	0004 1.4 1/5 1/6 1	(e)	0000 10 12 14 16 19 20 22 4sin0	• 24 20		

Table 1. Determination of lattice constant (a), x-ray density (d_x) and particle size (D).

Fig. 3. W-H analysis of Mg²⁺ substitute with NiFe₂O₄.

This is equivalent to $\ln \beta = 0.72 \ln 1/(\cos \theta) + (0.9\lambda/D)$. From this line, the intercept is -5.781 and and D = (0.9*0.154) / 3.085E-3 = 44 nm. Similarly all x values and the calculated particle size are listed in Table 1.

The size of the crystallite is also evaluated from Williamson-Hall equation [14]

$$\beta \cos \theta = \frac{k\lambda}{d} + 4\varepsilon \sin \theta \tag{4}$$

Plots (4sin θ) versus ($\beta cos \theta$) for the different concentration of Mg²⁺ doped with nickel ferrites are shown in Fig. 3. From the slope and y-intercept of the fitted line, the crystallite sizes were estimated. It is to be noted that all the crystallite values are summarized in Table 1. From the table, it is observed that the crystallite size increases and also it gradually decreases with further increase in Mg concentration. This can be explained by inhomogeneous driving force produced by sintering process [14].

The lattice constant is calculated using d - spacing and (hkl) parameters [15].

$$a = d\sqrt{h^2 + k^2 + l^2}$$
(5)

Increase in lattice constant is due to the replacement of Ni^{2+} (0.69 Å) cations by Mg^{2+} (0.72 Å) cations. These are presented in Table 1. The lattice constant increases with Mg concentration. It indicates that all the samples obey Vegard's law.

X-ray density (d_x) for different compositions are calculated using the formula,

$$\mathbf{d}_{\mathbf{x}} = (\mathbf{8M} / \mathbf{NV}) \tag{6}$$

Where N is the Avogadro's number, M is the molecular weight of the sample, V is the volume of the unit cell. The calculated x-ray density values of different compositions are tabulated in Table 1. The X-ray density is expected to decrease with the magnesium content primarily because the lattice parameter increase with magnesium content. Similar result has been observed by literature [16, 17]. The decrease in X-ray density can be ascribed to the density and atomic weight of Mg^{2+} (1.74 and 24.31 gm/cm³), which are lower than those of Ni²⁺ (8.90 and 58.69 gm/cm³) and Fe³⁺ (7.86 and 55.85 gm/cm³).

Using the lattice constant, oxygen position parameter (u = 0.381 Å) and radius of oxygen ion (R_o) as 1.32 Å,

Table 2. Calculated values of tetrahedral and octahedral bond length (d_{Ax} and d_{Bx}), tetrahedral edge, shared and unshared octahedral edge (d_{AxE} , d_{BxE} and d_{BxEU}), distance between the magnetic ions at A-site and B-site (L_A and L_B), ionic radius of A-site and B- site (r_A and r_B) and oxygen position parameter (u).

Magnesium Content	d _{Ax} (Å)	d _{Bx} (Å)	d _{AxE} (Å)	d _{BxE} (Å)	d _{BxEu} (Å)	L _A (Å)	L _B (Å)	r _A (Å)	r _B (Å)
0.0	1.8932	2.0372	3.0912	2.8080	2.9517	3.6130	2.9500	0.5732	0.7159
0.2	1.8945	2.0386	3.0934	2.8100	2.9539	3.6156	2.9522	0.5745	0.7174
0.4	1.8955	2.0396	3.0949	2.8114	2.9553	3.6173	2.9536	0.5755	0.7184
0.6	1.8977	2.0420	3.0986	2.8148	2.9588	3.6216	2.9571	0.5777	0.7208
0.8	1.8980	2.0423	3.0990	2.8151	2.9592	3.6220	2.9575	0.5780	0.7211
1.0	1.9007	2.0452	3.1034	2.8191	2.9634	3.6272	2.9617	0.5807	0.7240

tetrahedral and octahedral bond length (d_{Ax} and d_{Bx}), shared and unshared octahedral edge (d_{BxE} and d_{BxEU}), tetrahedral edge (d_{AxE}), distance between the magnetic ions at A-site and B-site (L_A and L_B), and ionic radius of A-site and B-site (r_A and r_B) are calculated the following equations [18],

$$d_{AX} = a\sqrt{3}\left(u - \frac{1}{4}\right) \tag{7}$$

$$d_{Bx} = a \sqrt{3u^2 - \frac{11}{4}u + \frac{43}{64}}$$
(8)

$$d_{AXE} = a\sqrt{2} \left(2u - \frac{1}{2} \right) \tag{9}$$

$$d_{BXE} = a\sqrt{2}(1-2u) \tag{10}$$

$$d_{BxEu} = a \sqrt{4u^2 - 3u + \frac{11}{16}}$$
(11)

$$L_{A} = a \frac{\sqrt{3}}{4}$$
(12)
$$L_{B} = a \frac{\sqrt{3}}{4}$$
(13)



Fig. 4. Morphological image of (a) $Ni_{0.8}Mg_{0.2}Fe_2O_4$ and (b) $Ni_{0.2}Mg_{0.8}Fe_2O_4$ nanoparticles.

 (\mathbf{b})

$$r_A = a\sqrt{3}\left(u - \frac{1}{4}\right) - R_a \tag{14}$$

$$r_B = a \left(\frac{5}{8} - u\right) - R_0 \tag{15}$$

All these values are calculated and summarized in Table 2. All the calculated values are increased with increasing magnesium content. This could be associated to the larger radius of Mg^{2+} ion as compared with Ni^{2+} ions. In Table 2, r_A values increase with increasing magnesium content. The increase in tetrahedral and octahedral radius is due to the replacement of Nickel ions by nonmagnetic ions Mg^{2+} . In all the samples the tetrahedral radius is smaller than octahedral radius.

Fig. 4 shows the scanning electron micrographs of



Fig. 5. EDX spectrum of Ni_{0.6}Mg_{0.4}Fe₂O₄.

Flomont	$Ni_{0.6}Mg_{0.4}Fe_2O_4$				
Element	Expected	Observed			
Ni	8.578	8.29			
Mg	5.722	3.16			
Fe	28.57	22.16			
0	57.13	57.78			
		1			

Table 3. EDX result of Ni_{0.6}Mg_{0.4}Fe₂O₄.



Fig. 6. FTIR spectra of $Ni_{1-x}Mg_xFe_2O_4$.



Fig. 7. Hysteresis loop of Ni_{1-x}Mg_xFe₂O_{4.}

 $Ni_{1-x}Mg_xFe_2O_4$ (x = 0.2, and 0.8). The grains are randomly distributed and agglomerated. Agglomeration of the particles indicated the magnetic attraction and the highly interfacial surface tension produced by the high surface to volume ratio [19].

The purity and the chemical composition of sample are checked using the EDS analysis, shown in Fig. 5, and their values are summarized in Table 3. The elements of Ni, Mg, Fe and O are detected. Additionally carbon element is also present due to the coating applied on the samples prior to the SEM analysis [20]. The molar proportions of the present elements are in good



Fig. 8. Variation of saturation magnetization values of Ni-Mg Ferrite.

agreement with that of excepted values which shows that there is no chemical reaction or any loss of ingredients.

FTIR spectrums of Ni_{1-x}Mg_xFe₂O₄ are recorded in the range of 400-4000 cm⁻¹ as shown in Fig. 6. The vibrations of ions in the crystal lattice are assigned by the IR bands of solids. In ferrites, two main broad metal-oxygen bands are appeared in FTIR. The spinel absorption band at 570-590 cm⁻¹ and 400-470 cm⁻¹ attribute to the stretching vibrations due to interaction of oxygen and the cations in A-site and B-site. The frequency difference between γ_1 and γ_2 due to bond length changes in Fe³⁺-O²⁻ at A site and B site. The band at ~1385 cm⁻¹ [21] associates with the symmetric and asymmetric N-O vibrations from nitrate groups. The peak at ~1600 cm⁻¹ [21] indicates OH stretching vibration interacting through H bonds.

Fig. 7 shows the magnetic hysteresis loops of all the samples recorded at room temperature in the range of 15000 G. The VSM studies the saturation magnetization (M_s), coercivity (H_c), remanence (M_r), squareness ratio (M_r/M_s), magnetic moment and Yafet-Kittel angle (θ_{yk}) are estimated and are listed in Table 3. The M-H curves confirm soft ferrimagnetic nature of the samples. The saturation magnetization value of pure nickel ferrite nanoparticle is lower than bulk value (55 emu/g) [22]. The possible reasons are spin canting effect and non-collinearity of spin with magnetic core on the surface [23, 24]. From the results, it is observed that with increase of magnesium concentration, the specific saturation magnetization increases upto x = 0.4 and then decreases (Fig. 8).

According to Neel theory, three kinds of exchange interaction exist in ferrites: interaction between magnetic ions in A and B site (AB interaction), interaction between the magnetic ions in B sites (BB interaction) and interaction between the magnetic ions in A site (AA interaction). But AB interaction is very strong compared with AA and BB. The net magnetization is the difference

angle (v_{yk}) , ansonopic constant (K) and cure temperature (r_c) .									
S.No	Mg ²⁺ Content	M _s (emu/g)	M _r (emu/g)	H _c (G)	R	n _B	θ_{yk}	K (erg/g)	T _c (°C)
1	0.0	20.36	4.38	167.01	0.22	0.85	33 °18"	3542	329
2	0.2	11.73	2.16	137.81	0.18	0.48	57 °47"	1684	196
3	0.4	19.88	2.93	111.43	0.15	0.78	67 °16"	2308	344
4	0.6	17.27	1.12	74.204	0.06	0.66	79 °40"	1335	310
5	0.8	13.33	2.90	113.83	0.22	0.49	84 °44"	1581	249
6	1.0	9.29	1.27	103.12	0.14	0.33	88 °38"	997	180

Table 4. Estimated values of Magnetization (M_s), Remanence (M_r), Coercivity (H_c), Squareness ratio (R), Bohr magneton (n_B), Yafet-Kittel angle (θ_{yk}), anisotropic constant (K) and Curie temperature (T_c).

between the magnetic moments of individual sublattices $M\,{=}\,M_{B}{\text{-}}M_{A}.$

In Ni-Mg ferrite, Ni²⁺ ions have a strong preference towards octahedral (B) sites; Mg²⁺ ions in the spinel structure would be randomly distributed into both the A- and the B-site [25] while the Fe³⁺ ions are distributed equally in A and B sites. Substitution of non-magnetic ions (Mg²⁺) in A site displaced some of Fe³⁺ ion to B site. This Fe³⁺ ion strengthens A - B interaction and increases the saturation magnetization up to x = 0.4. Further the increase of Mg²⁺ ion decreases the saturation magnetization. This may be related to the migration of Mg²⁺ ions in B site. At that time number of Fe³⁺ ions will decrease in B site and increase in A site weakening the whole lattice. This suggests that canting angle is established and it can be explained by Y –K three sublattice model.

Our observations also support the earlier occupation of Mg ions in A site at low concentration and B site in Higher concentration. Our results support the earlier works [11, 26, 27] Where substitution of Mg at higher concentration decrease the saturation magnetization due to strengthening of B-B interaction compared with A-B interaction.

The magnetic moments (n_B) of the samples are calculated using relation [28],

$$n_{\rm B} = M.M_{\rm s}/5585$$
 (16)

Where M is the molecular weight and M_s is saturation magnetization. The values of magnetic moments are presented in Table 3.

Yafet-Kittel angle was calculated from the following formula [29],

$$\cos\theta_{\rm YK} = [n_{\rm B} + 5(1-x)] / [7(1+x)]$$
(17)

Where n_B is the magnetic moment expressed in the units of Bohr magneton and θ_{YK} is Y-K angle are presented in Table 3. It is evident that the calculated value of the Y-K angles gradually increase with the increase of Mg concentration and finally extrapolates 88 °38" for magnesium ferrite.

The magnetic anisotropy constant can be calculated as [30],

$$K = H_c. M_s / 0.96$$
 (19)

The Curie temperature can be obtained by upadhyay model [31],

$$T_{c}(x) = [M^{*}(x = 0) n(x) T_{c}(x = 0)] / [M^{*}(x) n(x = 0)]$$

Since [32], M^{*}(x) = 2 μ_{Fe} + (1-x) μ_{Ni} + x μ_{Mg} (20)

Where $T_c~(x=0)$ is Curie temperature at $x=0.~\mu_{Ni,}~\mu_{Mg}$ and μ_{Fe} are the magnetic moment of Ni^{2+} ion $(2~\mu_B)$, Mg^{2+} ion $(0~\mu_B)$ and Fe^{3+} ion $(5~\mu_B)$, x is the Mg^{2+} concentration and n(x) is the saturation magnetization. The anisotropic constant and Curie temperature values are summarized in Table 3. Substitution of Mg changes the cation distribution between A and B sites decrease the Curie temperature.

Conclusions

The mixed Ni-Mg ferrite samples are prepared successfully by sol gel citrate precursor method. The lattice parameter increases along with the increase of magnesium content. The XRD confirms single phase cubic spinel structure. The crystalline sizes are calculated from Scherrer formula, modified Scherrer formula and W-H plot method in the range of 17-70 nm. Tetrahedral bond length, tetrahedral edge, octahedral bond length, shared octahedral edge, unshared octahedral edge, oxygen parameter, magnetic ions of A-site and B-site, ionic radius of A-site and B-site values were calculated. SEM image clearly shows the particles are irregular and agglomerated. In FTIR, high and low absorption bands are in the range of 590 cm⁻¹ and 470 cm⁻¹ respectively. VSM analysis exhibits the ferromagnetic nature exceedingly. Initially saturation magnetization increases with small concentration (x < 0.4) and then decreases with higher concentration. Our results support the occupation of Mg ions for A sites at low concentration and B site for higher concentration.

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