

Synthesis, investigation, structural and elastic properties of $Mg_xZn_{1-x}Fe_2O_4$ nanoparticles

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ABSTRACT Magnesium-Zinc Ferrite nanoparticles of different compositions are synthesized by using the sol-gel auto-combustion method with citric acid as a fuel. Structural characteristics were studied using X-ray diffraction technique and it confirms the formation of cubic spinel structure. The ferrite nanoparticle size of synthesized powder ranges from 22 – 24 nm. The effect of change in Mg^{2+} content results in a change of the lattice parameter of ferrite nanoparticles. In the present paper, the structural parameters such as cation-cation and cation-anion distances, tetrahedral, octahedral bond lengths and bond angles, hopping lengths, shared, unshared tetrahedral, and octahedral edge are reported. FTIR spectra show two prominent peaks around $524 - 532\text{ cm}^{-1}$ (tetrahedral site) and $409 - 432\text{ cm}^{-1}$ (octahedral site) and the force constants of the octahedral and tetrahedral site of Mg-Zn ferrite were calculated. The elastic moduli and other factors such as longitudinal, transverse and mean velocity, Poisson ratio, and Debye temperature were determined.

KEYWORDS Magnesium-Zinc ferrite nanoparticles, sol-gel auto-combustion method, cubic spinel structure, bond lengths and bond angles, hopping lengths, elastic moduli

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1. Introduction

Spinel ferrites are an important magnetic materials for application in different branches of biomedical, ferrofluid, microwave and data storage devices, magnetocaloric refrigeration, gas sensors, etc. due to its electronic, magnetic, and catalytic properties [1]. The promising and extensively used Mg-Zn ferrite nanoparticles are suitable for applications not only in the field of electronics [2], heterogeneous catalysis, sensors, magnetic technologies, but also in medicine for cancer treatment by hyperthermia [3] and other biomedicines [4]. Chemically and thermally stable zinc ferrite material are used for photocatalysis, magnetic resonance imaging (MRI), drug delivery, and pigments [5,6]. Rahaman and Ichiyanagi [7,8] reported that in Mg-Zn ferrite, Mg ferrite has an inverse spinel structure with Mg^{2+} cations mainly on octahedral site while Zn ferrite has a normal spinel structure in which Zn^{2+} occupies the tetrahedral site [7,9]. The cations distribution is mainly dependent on the synthesis technique and ambient conditions [10–12].

To tailor the desired properties of ferrite nanoparticles, various physical and chemical routes are used for synthesizing ferrite materials such as ceramic, co-precipitation, auto-combustion, hydrothermal, citrate precursor, etc. [13]. The preparation of Mg-Zn ferrite nanoparticles [14–16] through the Sol-Gel auto-combustion route has its importance in the area of research and development. Better control over particle size can be possible by combustion route which helps one to improve properties of the materials. Keeping in mind the importance of this synthesis route, the different compositions of Mg-Zn ferrites has been synthesized in the present work and its structural and elastic properties as a function of composition are reported.

2. Experimental

The Magnesium Nitrate $Mg(NO_3)_2 \cdot 6H_2O$, Zinc Nitrate $Zn(NO_3)_2 \cdot 6H_2O$, Ferric Nitrate $Fe(NO_3)_3 \cdot 9H_2O$, and citric acid ($C_6H_8O_7$) are used for preparing the composition of $Mg_xZn_{1-x}Fe_2O_4$ ferrite for $x = 0.0, 0.2, 0.6, 0.8,$ and 1.0 . All the metal nitrates are dissolved in distilled water and are mixed with a 1:3 ratio of nitrate to citric acid. By keeping these solutions on the hot plate ($90\text{ }^\circ\text{C}$) on the magnetic stirrer, the mixed solutions become viscous and finally formed into a viscous gel. This viscous gel began frothing after evaporating all water molecules' contents and the gel automatically ignited and burned with glowing flints and fully loosed powdered ash remained in the container of the gel. This powdered ash was then sintered (at $1000\text{ }^\circ\text{C}$) for four hours in an automatic temperature-controlled muffle furnace.

The structural characteristics of both of these sintered and non-sintered ferrite powder were studied by FTIR spectrometer (IRAffinity – 1S WL of Shimadzu Corporation, Japan) and powder X-ray diffractometer (BRUKER D8).

3. Results and discussion

3.1. X-ray diffraction(XRD)

Figures 1 and 2 show the X-ray diffraction patterns for $Mg_xZn_{1-x}Fe_2O_4$ ferrite compositions (both non-sintered and sintered) and confirm the formation of single-phase cubic spinel structure of ferrites. All the compositions also show the characteristics reflections of cubic spinel ferrites and confirm the formation of cubic spinel structure without any signs of the secondary phase. The peaks (222), (311), (222), (400), (422) were indexed using JCPDS. X-ray diffraction patterns clearly show the sintering temperature-dependent increase in peak intensity confirming the crystallinity increase with simultaneous decrease of full width at half maxima (FWHM).

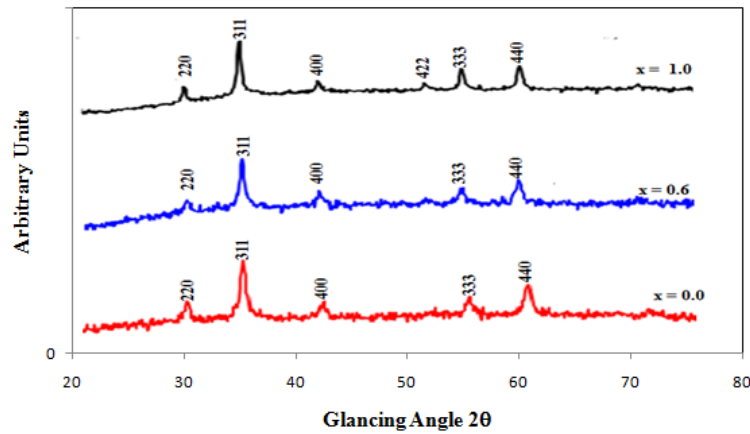


FIG. 1. XRD pattern of non-sintered (as synthesized) $Mg_xZn_{1-x}Fe_2O_4$ ferrite

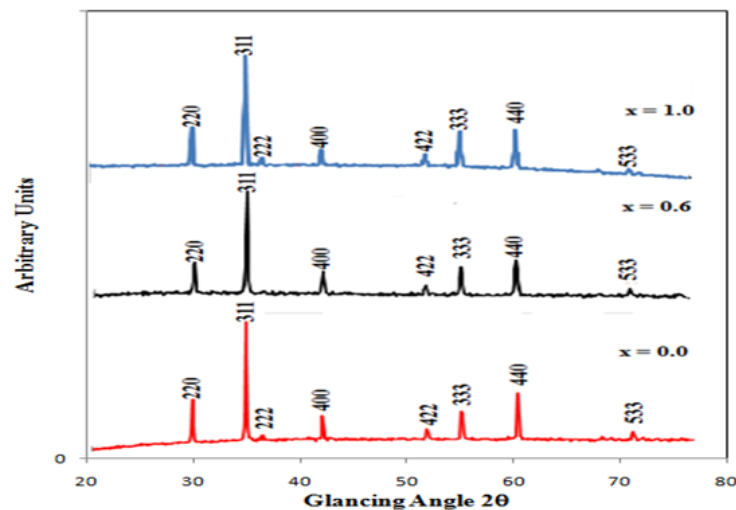


FIG. 2. XRD pattern of sintered $Mg_xZn_{1-x}Fe_2O_4$ ferrite

The broadening of the most prominent peaks (311) of synthesized powder of all compositions is used for the analysis of crystallite size. The Debye–Scherrer formula was used to calculate crystallite size of ferrite nanoparticles, $D_{311} = \frac{k\lambda}{\beta \cos \theta}$, where, $k = 0.9$ is a crystallite size constant, λ is the wavelength of radiation, θ is the diffraction angle and β is the full width at half maximum (FWHM) of intensity for (311) peak. Experimental lattice parameters were obtained using relation $a_{exp} = d_{hkl}[h^2 + k^2 + l^2]^{1/2}$ while the theoretical lattice parameter [17] was calculated by the following relation:

$$a_{th} = \frac{8}{3}\sqrt{3} \left[(r_A + R_0) + \sqrt{3}(r_B + R_0) \right]. \quad (1)$$

Here, $R_0 = 1.38 \text{ \AA}$ is the oxygen ion radius, ionic radii of the tetrahedral [A] and the octahedral [B] sites are r_A, r_B , respectively.

Both these experimental and theoretical lattice parameters for the present ferrite composition are listed in Table 1. The decrease in both the theoretical and experimental values of lattice constant (a) confirms the increase in Mg^{2+} content from 8.441 to 8.349 \AA , clearly obeying Vagard's Law [18]. The difference in the ionic radii of replaced and replacing ions causes this abatement, which depicts the linear change in the lattice constant with the substitution of different ions. The ionic radii difference of Mg^{2+} (0.65 \AA) and Zn^{2+} (0.83 \AA) causes the diminution of the lattice constant. Manikandan et al. [1] and Rahman [7] also reported similar results. The X-ray density was calculated by relation $\rho_x = \frac{ZM}{Na^3}$ and the bulk density or actual density was obtained by making the pellet of synthesized material, using relation $\rho_a = \frac{m}{\pi r^2 h}$. Here m is the mass, r is the radius and h is the thickness of the pellet.

TABLE 1. Lattice Parameter (a_{exp} and a_{th}), Crystallite size (D), X-ray density (ρ_x), actual density (ρ_a) [± 0.006]

x	a_{exp} (\AA)	a_{th} (\AA)	D (nm)	ρ_x (gm/cc)	ρ_a (gm/cc)
0.00	8.349	8.441	22.6	5.325	4.635
0.20	8.331	8.423	23.4	5.176	4.759
0.40	8.310	8.402	23.4	5.030	4.568
0.60	8.302	8.394	22.8	4.860	4.237
0.80	8.276	8.368	23.5	4.719	4.108
1.00	8.349	8.441	23.8	4.565	4.059

The bond length (d_{AX}, d_{BX}) site radii (r_A, r_B), tetrahedral edge (d_{XX}), octahedral edges (shared and unshared) (d'_{XX}, d''_{XX}), hopping length radii (L_A, L_B) are calculated by the following relations. The corresponding values are presented in Table 2.

$$\begin{aligned}
 d_{AX} &= a\sqrt{3}\left(u - \frac{1}{4}\right), & d_{BX} &= a\sqrt{3u^2 - \frac{11}{4}u + \frac{43}{64}}, & d_{XX} &= a\sqrt{2}\left(2u - \frac{1}{2}\right), \\
 d'_{XX} &= a\sqrt{2}(1 - 2u), & d''_{XX} &= a\sqrt{4u^2 - 3u + \frac{11}{16}}, & L_A &= a\frac{\sqrt{3}}{4}, & L_B &= a\frac{\sqrt{2}}{4}.
 \end{aligned}
 \tag{2}$$

TABLE 2. Hopping lengths (L_A, L_B), bond length (d_{AX}, d_{BX}), site radii (r_A, r_B), tetrahedral edge (d_{XX}), shared and unshared octahedral edges (d'_{XX}, d''_{XX}) for present ferrite composition [± 0.0004]

x	L_A	L_B	d_{AX}	d_{BX}	r_A	r_B	d_{XX}	d'_{XX}	d''_{XX}
0.00	3.6551	2.9843	1.8275	2.1103	0.4475	0.7302	2.9844	2.9843	2.9843
0.20	3.6473	2.978	1.8236	2.1058	0.4436	0.7257	2.9779	2.978	2.978
0.40	3.6382	2.9706	1.8190	2.1005	0.4391	0.7205	2.9705	2.9706	2.9706
0.60	3.6347	2.9677	1.8173	2.0985	0.4374	0.7185	2.9677	2.9677	2.9677
0.80	3.6235	2.9585	1.8117	2.0920	0.4317	0.7120	2.9585	2.9585	2.9585
1.00	3.6152	2.9518	1.8076	2.0873	0.4276	0.7072	2.9518	2.9518	2.9518

The values of all these parameters clearly show the effect of an increase in Mg^{2+} content in the composition which is related to larger values of Zn^{2+} ions as compared to Mg^{2+} ions. This substitution dependent bond length change for tetrahedral and octahedral sites was reported by Vara Prasad et al. [19]. The hopping length behavior may be attributed to the ionic radii difference and causes the decrease in hopping length [20].

The lattice and oxygen parameter variation in terms of cation-cation [Me–Me] and cation-anion [Me–O] bond length and bond angles [21, 22] are obtained using relations. Particularly, cation-cation [Me–Me] bond lengths are as follows :

$$b = \sqrt{2}\frac{a}{4}, \quad c = \sqrt{11}\frac{a}{8}, \quad d = \sqrt{3}\frac{a}{4}, \quad e = \sqrt{3}\frac{3a}{8}, \quad f = \sqrt{6}\frac{a}{4},
 \tag{3}$$

cation-anion [Me–O] bond lengths:

$$p = a \left(\frac{5}{8} - u \right), \quad q = a \left(u - \frac{\sqrt{3}}{4} \right), \quad r = a \left(u - \frac{\sqrt{11}}{4} \right), \quad s = a \left(\frac{u}{3} + \frac{1}{8} \right) \sqrt{3}. \quad (4)$$

Here $u = \frac{5\sqrt{3}l_A^T + 6l_A^O}{8[\sqrt{3}l_A^T + 3l_A^O]}$ for normal spinel and $u = \frac{5}{8} - \frac{9l_A^O}{8[\sqrt{3}l_B^T + 3\frac{Z_A l_A^O + Z_B l_B^O}{Z_A + Z_B}]}$ for inverse spinel structure [21].

The values of all these parameters in Table 3 show that both these [Me–Me] and [Me–O] bond lengths decreases due to an increase in ions of Mg^{2+} in the composition. The variation in the ionic radii of Mg^{2+} and Zn^{2+} ions and the lattice constant affects the changes in the bond length of all the compositions.

TABLE 3. Cation-cation [Me–Me] and cation-anion [Me–O] bond length for present ferrite composition [± 0.0004]

x	cation-cation					cation-anion			
	b	c	d	e	f	p	Q	r	s
0.00	2.9843	3.4995	3.6551	5.4825	5.1690	2.1102	1.8275	3.4994	3.6551
0.20	2.9779	3.4920	3.6473	5.4709	5.1580	2.1057	1.8236	3.4919	3.6473
0.40	2.9705	3.4833	3.6382	5.4572	5.1451	2.1005	1.8190	3.4832	3.6382
0.60	2.9677	3.4800	3.6347	5.4520	5.1402	2.0985	1.8173	3.4799	3.6347
0.80	2.9585	3.4692	3.6235	5.4352	5.1243	2.0920	1.8117	3.4691	3.6235
1.00	2.9518	3.4613	3.6152	5.4228	5.1127	2.0872	1.8076	3.4613	3.6152

The concentration-dependent bond angles ($\theta_1, \theta_2, \theta_3, \theta_4, \theta_5$) shown in Fig. 3 were calculated by trigonometric relations [21] and were found to be 125.25 close to ideal and reported values [23] for all compositions of Ni–Zn ferrite.

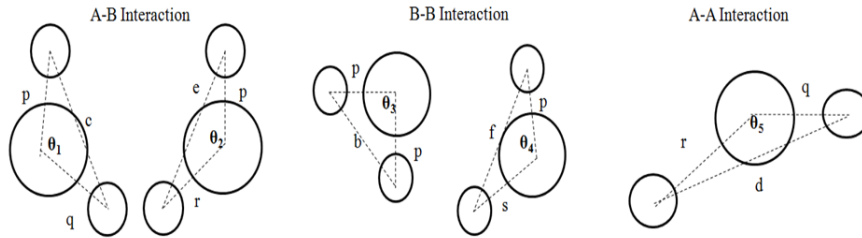


FIG. 3. Spinel structure with bond length and bond angles

$$\theta_1 = \cos^{-1} \left[\frac{(p^2 + q^2 - c^2)}{2pq} \right], \quad \theta_2 = \cos^{-1} \left[\frac{(p^2 + r^2 - c^2)}{2pr} \right], \quad \theta_3 = \cos^{-1} \left[\frac{(2p^2 - b^2)}{2p^2} \right], \quad (5)$$

$$\theta_4 = \cos^{-1} \left[\frac{(p^2 + s^2 - f^2)}{2ps} \right], \quad \theta_5 = \cos^{-1} \left[\frac{(r^2 + q^2 - d^2)}{2rq} \right].$$

3.2. FTIR study

The Fourier transforms IR spectroscopy is useful to determine the crystalline symmetry. It is used for the analysis of spinel structure formation in ferrites. Fig. 4 shows FTIR spectra of sintered ferrite nanoparticles of $Mg_xZn_{1-x}Fe_2O_4$ for $x = 0.0, 0.6, 1.0$ composition.

As shown in the Fig. 4, FTIR spectra for these compositions depict the spinel structure formation of ferrite by absorption dip corresponding to tetrahedral and octahedral vibrational complexes at around $528 - 465$ and $409 - 432 \text{ cm}^{-1}$ respectively. The increase in Mg^{2+} concentration results in the shifting of the wavenumber of band: ν_1 shifts towards higher values and ν_2 shifts towards lower values. The tetrahedral site is occupied by Zn^{2+} ions with a larger ionic radius which affects the band position. The stretching of tetrahedral ions and oxygen bonding causes absorption band ν_1 while the transverse vibrations of oxygen with tetrahedral sites cause band ν_2 . Absorption band ν_1 with the intrinsic vibration

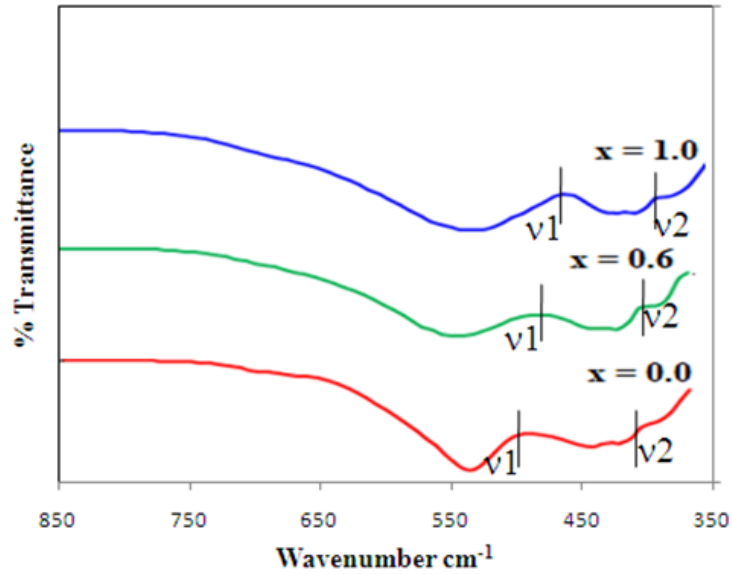


FIG. 4. FTIR spectra for sintered $Mg_xZn_{1-x}Fe_2O_4$ ferrite composition for $x = 0.0, 0.6, 1.0$

of tetrahedral groups corresponds to the restoring force and band ν_2 of octahedral groups corresponds to bond bending vibrations [24].

The difference in $Fe^{3+}-O^{2-}$ distance of A and B site also reflects the dissent between the positions of both ν_1, ν_2 bands in FTIR spectra. In the present work, the absorption bands for the present ferrite composition (Mg–Zn) were found close to the reported values. Table 4 shows force constants for the tetrahedral and octahedral sites calculated in [25] using the following formulas, $K_t = 7.62 \times M_1 \times \nu_1^2 \times 10^{-7}$ N/m and $K_0 = 10.62 \times \frac{M_2}{2} \times \nu_2^2 \times 10^{-7}$ N/m, where, K_t, K_0 are the force constants, M_1, M_2 are the molecular weights and ν_1, ν_2 are the vibrational bands corresponding to tetrahedral and octahedral sites, respectively.

TABLE 4. Vibrational bands, force constants, longitudinal modulus (L), shear modulus (G), bulk modulus (B), Young modulus (E)

x	ν_1 (cm $^{-1}$)	ν_2 (cm $^{-1}$)	K_t (N/m)	K_0 (N/m)	L (GPa)	G (GPa)	B (GPa)	E (GPa)
0.00	490	415	136.79	110.68	146.59	48.86	81.44	122.16
0.20	526	426	133.82	101.55	139.70	46.57	77.62	116.42
0.40	526	428	129.8	96.37	134.58	44.86	74.77	112.15
0.60	492	402	128.66	82.4	125.71	41.90	69.84	104.76
0.80	528	412	122.68	77.93	119.85	39.95	66.59	99.88
1.00	465	392	120.44	71.19	114.76	38.25	63.76	95.63

The force constants values from Table 4 show decrement in force constant K_0 and K_t as a function of increasing Mg^{2+} content in all compositions, such that $K_t > K_0$.

The elastic parameter and Debye temperature [24, 26] are calculated here using frequency band relation $\theta_1 = 1.438\nu_{av}$, where $\nu_{av} = \left(\frac{\nu_1 + \nu_2}{2}\right)$. The Debye temperature variation from 676 to 687 K regulates the heat conduction mechanism in the ferrites. Based on specific heat theory and the heat conduction mechanism, a decrease in Debye temperature with an increase in Mn content in Li–Mn ferrites also was reported in [27].

Debye temperature (θ_D) was calculated using the density of the sample, the molecular weight and the mean wave velocity in accordance with the following relation:

$$\theta_D = \frac{h}{K_B} \left(\frac{3\rho q N_A}{4\pi M} \right)^{1/3} V_m, \quad (6)$$

where h is Planck's constant, K_B is Boltzmann's constant, N_A is Avogadro's number, M is the molecular weight, q is the number of atoms in unit formula, ρ is the density of the sample [28].

The product of the stiffness constant ($C_{11} = L$; Longitudinal modulus) and the lattice constant (a) was used to obtain elastic moduli [29] estimation by the force constant ($K = (K_t + K_0)/2$). The pore fraction ($p = 1 - \rho_a/\rho_x$) for each composition was obtained using the corresponding values of the X-ray density (ρ_x) and the bulk density (ρ_a).

The longitudinal wave velocity (V_L) and the transverse wave velocity (V_T) [30] were determined by, $V_L = \left(\frac{C_{11}}{\rho_x}\right)^{1/2}$ and $V_T = \frac{V_L}{\sqrt{3}}$. Similarly, the shear modulus (G), the bulk modulus (B), the Young modulus (E), Poisson's ratio (σ), the mean wave velocity (V_m) [31] are determined using the following relations,

$$G = \rho \times (V_T)^2, \quad B = L - \left(\frac{4}{3}\right) G, \quad E = (1 + \sigma) 2G, \quad \sigma = \frac{3B - 2G}{6B + 2G}, \quad V_m = \left[\frac{1}{3} \left(\frac{1}{V_L^3} + \frac{2}{V_T^3}\right)\right]^{-1/3}. \quad (7)$$

The elastic moduli and the wave velocity values are listed in Table 5. This elastic moduli value depicts the dependence on the Mg^{2+} content due to weaken interatomic bonding. The effect of changes in the interatomic bonding strength on the elastic parameters with Zn^{2+} content in Zn-Co ferrites was reported in works [32, 33].

TABLE 5. The longitudinal wave velocity and the transverse wave velocity, the mean wave velocity (V_m), Poisson's ratio ($\sigma \pm 0.06$), the elastic moduli corrected to zero porosity ($E_0, G_0, B_0, L_0, \sigma_0$)

x	V_L (m/s)	V_T (m/s)	V_M (m/s)	E_0 (GPa)	G_0 (GPa)	B_0 (GPa)	L_0 (GPa)	σ	σ_0
0.00	5623.8	3246.9	3604.66	165.054	60.08	95.57	186.346	0.25	0.21
0.20	5418.2	3128.2	3472.89	138.84	55.28	94.79	168.49	0.25	0.25
0.40	5427.9	3133.8	3479.11	137.51	54.70	94.27	167.21	0.25	0.26
0.60	5447	3144.8	3491.35	141.01	55.93	98.15	172.73	0.25	0.26
0.80	5401.5	3118.6	3462.21	134.91	53.51	93.96	165.31	0.25	0.26
1.00	5317.3	3069.9	3408.2	122.98	48.85	84.95	150.09	0.25	0.26

The transverse propagation of energy keeps the particles in vibration mode that results in the collisions. Hence, more energy is needed during the transverse wave propagation than for the longitudinal wave [34].

The Debye temperature is used to determine the mode of vibrations and the rigidity of the ferrite material. From Table 6, it is observed that the Debye temperature obtained from the Waldron equation is greater than that obtained from the Anderson equation. Similar behavior was observed by Mazen and Elsaad [35] for Li-Mn ferrite. Both Debye temperatures show decreasing trend with increasing Mg^{2+} content. According to the theory of specific heat, part heat absorbed by electrons causes a decrease in the Debye temperature (θ_D) [35]. It is suggested that the conduction is due to electron in the synthesized ferrite material.

TABLE 6. Debye temperatures (θ_D, θ_1), B_0/G_0 ratio [± 0.08]

x	θ_D (K)	θ_1 (K)	B_0/G_0
0.00	460.84	687.36	1.70
0.20	456.79	684.48	1.71
0.40	456.84	685.92	1.72
0.60	452.68	676.57	1.75
0.80	450.07	675.86	1.75
1.00	447.28	676.58	1.87

These two temperature relations (θ_D, θ_1) reflect the dependence of Mg^{2+} content. The elastic moduli do not show any significant dependence on porous polycrystalline ferrites by nature [36]. The magnitudes of the elastic moduli corrected to zero porosity are higher than non-corrected to zero porosity.

The Pugh criteria [37], the ratio of the Bulk modulus and the Rigidity modulus B_0/G_0 , is used to decide ductility and brittleness of synthesized material. The ratio greater than the critical value of 1.75 indicates the ductile nature and the ratio lower than 1.75 indicates the brittle nature. In the present case, the ratio B_0/G_0 for all the compositions presented in Table 6 varies between 1.70 and 1.87. It leads to the conclusion that the compositions with $x = 0.60$ and 0.80 are ductile and the remaining are brittle.

4. Conclusions

Compositions of $\text{Mg}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ with $x = 0.0, 0.2, 0.4, 0.6, 0.8,$ and 1.0 were successfully synthesized using sol-gel auto-combustion process. Both the X-ray diffraction and FTIR spectra of non-sintered (as-synthesized) and sintered compositions confirm the formation of cubic spinel structure. The crystallite size lies within the $22 - 24$ nm for all compositions. An increase in Mg^{2+} content shows a decrease in the lattice constant from 8.441 to 8.349 Å and verifies Vegard's law. There is a decrease in the structural parameters such as tetrahedral, octahedral bond lengths, cation-cation, cation-anion distances, bond angles, hopping length with the increase in Mg^{2+} content. Two absorption bands of FTIR spectra depict tetrahedral site $524 - 532$ cm^{-1} and octahedral site at $409 - 432$ cm^{-1} . Tetrahedral absorption band ν_1 shifts towards higher values and octahedral absorption band ν_2 shifts towards lower values when there is an increase of Mg^{2+} concentration. The force constants for tetrahedral and octahedral sites show Mg^{2+} content dependence. FTIR data also depict that the increase in Mg^{2+} content affects the changing of the wave velocity, the elastic constants, and the Debye temperature. All the elastic moduli are corrected to zero porosity. The bulk modulus to the rigidity modulus ratio found to vary between 1.70 and 1.87 which leads to the conclusion that the compositions with $x = 0.60$ and 0.80 are more ductile than their counterparts (brittleness).

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