Farhana Naaz¹, Preeti Lahiri¹*, Chanda Kumari¹, Hemant Kumar Dubey²

Spectroscopic, Magnetic and Morphological studies of MgFe₂O₄ Nanopowder

¹Department of Chemistry, Institute of Science, MMV, Banaras Hindu University, Varanasi-221005, India
²Department of Chemistry BRD PG College, Deoria-274001, India

Spinel type nano ferrite compound MgFe₂O₄ was synthesized through sol gel technique using metal nitrates as precursors. The phase composition, morphology and elemental analysis of magnesium ferrite (MgFe₂O₄) were performed by X-ray diffraction, fourier transform infrared, atomic force microscopy, energy dispersive x-ray and scanning electron microscopy, analyses.

The sample's X-ray diffraction pattern verifies the existence of single phase material, with the size of its crystallites estimated to be 39.9 nm. Fourier transform infrared examination supported metal-oxygen vibrations corresponding to tetrahedral and octahedral sites, respectively. From scanning electron microscopy image, grain size obtained about 97.7 nm. Raman spectra of the sample shows five Raman active modes (A₁g + E₄ + 3F₂g), which is compatible with the spinel structure. Magnetic measurement study at room temperature shows a hysteresis loop behaviour with a low saturation magnetization value, 27.192 emu g⁻¹ and a small coercivity value. The optical band gap determined using UV-visible transmittance spectra. Additionally, X-ray photoelectron spectroscopy are used to confirm oxidation states and explore the chemical composition of the sample.

Keywords: Spinel ferrite, Nanocrystalline, X-ray diffraction, Raman spectra, Magnetic properties.

Received 17 January 2023; Accepted 20 June 2023.

Introduction

Ferrites belong to ferromagnetic materials. Ferrite nanomaterials have shown applications in various ways, such as music recording heads, core materials for power transformer in electronics and telecommunications [1,2]. Importantly, magnetic oxide nano-based materials differently from bulk material in terms of their physical, chemical, magnetic and electrical properties [3]. Advantages of nanomaterials rest on their superior thermal stability, environmental and economic advantages to regular counterparts. Owing to their finite size effect, high surface to volume ratio, unique crystal structures, and magnetic characteristics, spinel ferrites nanoparticles have generated a lot of attention in recent years. The general formula for spinel ferrites is AFe₂O₄, where A and Fe are divalent and trivalent metal cations, respectively, with Fe as major constituent. Due to their interesting magnetic and electrical properties, they have drawn significant attention to the researchers. Structurally, the spinel ferrite is a cubic closed packed organization of oxygen atoms with 8 formula units per unit cell. Since the radius of oxygen ion is greater than that of metal ion, the oxygen touches each other and form a closed packed Face centered cubic (FCC) lattice. The spreading of cations between A or B site of the lattice depends on both parameters such as preparation routes and heat treatment [4-6].

MgFe₂O₄ is the most adaptable, vital and multipurpose spinel ferrites due to unique and reproducible characteristics. They include high saturation magnetization value, and electrical resistivity [7]. Through these magneto-electrical properties MgFe₂O₄ show a great utility in the area of catalysis, sensors, high-density recording media and other magnetic technologies [8]. Overall, it is a n-type semiconducting as well as soft
magnetic material. Both photocatalytic activity and catalytic effect of magnesium ferrite in a heterogeneous Fenton-like reaction have been studied [9, 10]. Magnesium ferrites typically have the structural formula \((\text{Mg}, \text{Fe}^{3+} \text{Fe}^{2+})_x \text{O}_4\), as stated as the percentage of \((\text{A})\) sites occupied by \text{Fe}^{3+} cations, where \text{A}, \text{B} stand for the cationic sites of tetrahedral and octahedral co-ordinations respectively, where \text{x} represents the degree of inversion. The structural and magnetic properties of MgFe₂O₄ obtained from transmission electron microscopy, XRD, Mossbauer spectroscopy, and magnetometry have been reported [11]. Numerous studies [12,13] on MgFe₂O₄ nanoparticles described their nanostructures and many properties, including IR, XRD, SEM, and VSM. N-Suresh kumar [14] synthesized nickel substituted magnesium ferrites by auto combustion method and also studied dielectric and magnetic properties. Magnetic and structural properties of MgFe₂O₄ nanoparticles synthesized by EDTA-based sol–gel reaction has been reported by Shaban I. Hussein [15]. The electronic structure of MgFe₂O₄ nanoparticles created chemically has also been examined by Singh et al [16]. Nanoparticles of MgFe₂O₄ have good photoelectrical properties [17]. The synthesis of spinel magnetic oxide involves different chemico-physical approaches that include co-precipitation [18], sol–gel method [19], wet chemical method [20], combustion method [21], hydrothermal method [22], microwave assisted [23], micro-emulsion [24], and ceramic technique [25]. Each method has its own merits and demerits in terms of its ease, efficiency, yield, and costs.

Considering these criteria, we find the sol–gel method is effective and can work at low temperatures. Briefly, the sol–gel is a process for developing solid materials from small molecules. In this case, monomers are converted into colloidal solution (sol) that acts as a precursor for an integrated network (or gel) of discrete particles or network polymers. This approach is applicable for the production of metal oxides, like silicon and titanium. Metal alkoxides are common precursors. This chemical technique yields a “sol” which develops into a diphasic system with a liquid phase and solid phase that can have different morphologies, such as discrete particles or continuous polymer networks. Metal alkoxides are typical precursors. Several other names of the sol–gel method are low-temperature self-combustion, auto-ignition, self-propagation, and gel thermal decomposition methods.

We explain, in this paper, the characterization of single phasic cubic spinel ferrites using a sol gel technique by systematic examinations on the structural and physicochemical properties of magnesium ferrites nanoparticles based on several characterization parameter like XRD, SEM, FT-IR, EDX, and magnetic. AFM was utilized to determine the nanoparticles surface roughness and also to obtain 2D and 3D images. Moreover, Raman spectroscopic studies was added to understand the vibrational modes and structural studies of the materials, the optical behaviour of the nano-particles was investigated to determine the absorbance range 300-780 nm, which spans the visible region and is important for various photovoltaic and photocatalytic properties. Moreover, the chemical and elemental state of the nanoparticles was concluded by using XPS analysis.

Furthermore, we found that the sol gel technique is the most suitable for creating good molar ratio control with small particle size distribution due to molecular-level mixing, fast processing time, lower temperatures and inexpensive.

I. Experimental Procedure

MgFe₂O₄ was prepared via the auto-combustion aided sol–gel method. The energy required to produce the ferrite nano-particles is provided in this procedure by a redox process using a thermal precursor and citric acid as a fuel [26]. Metal nitrates were served as a source of soluble cations and the oxidant [27]. Citric acid (C₆H₆O₇) was employed as both a fuel and a chelating agent, forming interactions with metal ions and preventing the precipitation of hydroxylated compounds [28]. Analytical reagent grade, Merck India, used in this work were: iron nitrate \((\text{Fe(NO}_3)_2 \cdot 9\text{H}_2\text{O}, 99\%)\) magnesium nitrate \((\text{Mg(NO}_3)_2 \cdot 6\text{H}_2\text{O}, 98\%)\) and citric acid \((\text{C}_6\text{H}_6\text{O}_7\)). The desired composition was obtained by taking stoichiometric molar ratio \((2:1)\) of \((\text{Fe(NO}_3)_2 \cdot 9\text{H}_2\text{O}, 0.032 \text{ mol})\) and \((\text{Mg(NO}_3)_2 \cdot 6\text{H}_2\text{O}, 0.016 \text{ mol})\) dissolved in 20 ml of double distilled water. To promote homogeneity, an equimolar amount of citric acid \((0.046 \text{mol})\) was added to an aqueous mixture, which was magnetically agitated while the reaction temperature was kept at 60°C. The pH was optimize to 7.0 by adding ammonia solution drop by drop under steady stirring until the mixture was neutralised and turned into sol. The sol developed into a black gel after 8 hours of stirring, releasing a huge amount of gases \((\text{CO}_2, \text{H}_2\text{O}, \text{N}_2)\) in the process. The gel was dried for 12 hours at 100°C, with its volume swelling by nearly five times, yielding a fluffy, abundant powder. At end, the dried gel was grounded and annealed at 500°C for 3 h to get a dark brown ferrite nanoparticle as a final product. The Flow chart of the sol–gel synthesis process of MgFe₂O₄ nanoparticles is given below.

The structural investigations (crystal structure and phase analysis) of the synthesized nanomaterial were performed by using XRD, Rigaku MiniFlex 600, Japan technique with Cu-Kα radiation \((λ=1.5406 \AA)\) within 20 range 20-80°. The FTIR spectrum was obtained in the range 400 cm⁻¹ to 4000 cm⁻¹ employing a FTIR spectrometer, Perkin Elmer. SEM, (ZEISS model EVO-18 research, Germany) and AFM, model NTEGRA PRIMA, NT-MDT, Russia was used to determine microstructure and surface roughness of nanomaterial respectively. The quantitative elemental analysis was carried out by means of EDAX analyser (EDX, Oxford instrument USA). The magnetic behaviour of the produced sample was investigated using a SQUID-based magnetometer (MPMS 3) in the magnetic field range of 20 kOe. Raman spectra was recorded to estimate the frequency mode of tetrahedral and octahedral sites of cubic spinel at frequency range of 100-800 cm⁻¹, using the model RENISHAW In-VIA Raman microscope. UV–VIS diffuse reflectance spectra were acquired by UV-VIS Spectrophotometer SHIMADZU in the wavelength range of 200–600 nm (UV-1700 Pharma Spec), chemical and elemental state analysis of the investigated surface were indicated by XPS, Thermo scientific K-ALFHA Surface Analysis.
II. Results and Discussion

2.1 FTIR analysis

Figure 1 illustrates room temperature IR spectrum of the magnesium ferrite sample determined in the range 400 to 4000 cm⁻¹. The calcined MgFe₂O₄ magnetic nanoparticle exhibits two main absorption bands at around 630 and 441 cm⁻¹. The higher frequency band (ν₁) and the lower frequency band (ν₂), specify the metal-oxygen intrinsic stretching vibration of the spinel unit cell in the tetrahedral (A) and octahedral (B) sites [29] respectively. The observed variance in band positions is due to the distance between the metal-oxygen ions connected to the octahedral and tetrahedral complexes. This indicates bond formation and the phase stability of synthesized material. The FTIR results confirmed that the sample has spinel structure of MgFe₂O₄, which was revealed by the XRD results. The strong signature band observed at 3420 cm⁻¹ is assigned to the symmetric stretching vibrations of O-H groups [30]. The symmetric stretching vibration of hydrogen bonded water molecules is responsible for the perturbations in the absorption band between 1030 and 1630 cm⁻¹, indicating the existence of absorbed or free water molecules in a sample. The force constant (k) was evaluated from the equation [31],

\[ k = \frac{4\pi^2 c^2 \nu^2 m}{1} \]  \hspace{1cm} (1)

where, c is the velocity of light (2.99×10⁸ m/sec), ν is the vibrational frequency of the A and B sites, m is the reduced mass for the metal-oxygen ions. Values obtained for force constant are shown in Table 1.

2.2 X-ray diffraction analysis

Powder X-ray diffraction studies are helpful in determining the structure and particle size of nanoparticles produced. The XRD of powdered sample of MgFe₂O₄ obtained by sol-gel method is illustrated in Fig. 2. The XRD pattern reveals formation of monophase face centered cubic (fcc) spinel structure. All Bragg reflections have been indexed as (111), (220), (311), (222), (400), (422), (511) and (440). All observed sharp diffraction peaks were compared with magnesium ferrite JCPDS card number 89-3084. The most intense peak at 2θ=35.55 degree corresponds to crystalline plane of...
magnesium ferrite which indicates a fine particle nature of the particles.

![Graph](image)

**Fig. 2.** X-ray diffraction patterns of Magnesium ferrites nanoparticles.

The average crystallite size (D) was estimated from the XRD peak broadening of the most intense peak (311) using the Scherrer’s relation [32],

$$D = \frac{k\lambda}{\beta \cos \theta}$$  \hspace{1cm} (2)

where, k is Scherrer’s constant (0.91), \(\lambda\) is wavelength (\(\lambda = 1.5406 \text{ Å}\)). The parameters D and \(\theta\) are taken as crystallite size and Bragg’s angle respectively. Here \(\beta\) is the full width at half maxima (FWHM) of XRD peaks. Table 1 shows the calculated values for crystallite size, x-ray density, lattice constant, and cell volume based on x-ray data. The average crystallite size (D) of the synthesized material was found to be 39.9 nm (Table I) for MgFe\(_2\)O\(_4\). The lower value (36.6 nm) was reported for the same sample synthesized by co-precipitation method [33].

The lattice constant ‘a’ was calculated for prominent (311) peak using the following relation,

$$a = \frac{d\sqrt{h^2+k^2+l^2}}{\sqrt{8}}$$  \hspace{1cm} (3)

where d is interplanar distance and h,k,l are the miller indices of the diffraction plane. The calculated lattice parameter ‘a’ (8.35 Å) for MgFe\(_2\)O\(_4\) agrees well with the reported value of 8.37 Å [34]. Following relation was used to calculate x-ray(\(\rho_x\)) density,

$$\rho_x = \frac{ZM}{N_Aa^3}$$  \hspace{1cm} (4)

where Z is the number of molecules per unit cell (Z = 8), M is the molecular mass of the sample, and N is the Avogadro's number (\(N_A = 6.026 \times 10^{23}\) atoms mol\(^{-1}\)). The sample of ferrite was determined to have a density of 4.554 g/cm\(^3\) (Table I).

Utilizing the following formula, the volume of the unit cell was computed,

$$V = a^3$$  \hspace{1cm} (5)

in(Å\(^3\)) units. Relationships were used to determine the distance between magnetic ions and the hopping length (L) in the A sites (tetrahedral) and B sites (octahedral) [35],

$$L_A = \frac{a\sqrt{2}}{4} \quad \text{and} \quad L_B = \frac{a\sqrt{2}}{4}$$  \hspace{1cm} (6)

It was observed that the hopping lengths for two sites were 3.615 Å\(^3\) and 2.951 Å\(^3\) respectively in Table 1.

### 2.3 SEM and EDX analysis

The surface morphologies and elemental composition of magnesium ferrite nano powder were examined by SEM, coupled with EDX analysis. In Fig. 3 (a) SEM image of calcined MgFe\(_2\)O\(_4\) sample is illustrated. Agglomerates and non-uniformity are evident in the sample. The grain size was estimated using AIL [36]. The number of grain boundaries intersected by a randomly chosen straight line of length L drawn on micrographs was counted, and the average grain diameter (G), which is around 98 nm, was computed, N is the total number of the complete sample, and M is the magnification,

$$G = 1.5L/MN$$  \hspace{1cm} (7)

The elemental analysis of the prepared MgFe\(_2\)O\(_4\) nano powder was obtained by EDX analysis and weight % and atomic % of elements were shown in the Table 2. The estimated ferrite composition is confirmed by EDX data, which is reinforced by the good agreement between experimental and theoretical Mg/Fe molar ratio (Table 2). The sample’s EDX pattern, which displays the presence of Mg, Fe, and O without precipitating cations, is demonstrated in Fig. 3(b).

### Table 1.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Crystalline size (nm)</th>
<th>Lattice parameter (Å(^3))</th>
<th>X-ray density (g/cc)</th>
<th>Unit cell volume (Å(^3))</th>
<th>Force constant (k_0) (\times 10^5) (dyne/cm)</th>
<th>Force constant (k_3) (\times 10^5) (dyne/cm)</th>
<th>A site (d_A) (Å)</th>
<th>B Site (d_B) (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MgFe(_2)O(_4)</td>
<td>39.9</td>
<td>8.35</td>
<td>4.554</td>
<td>583.79</td>
<td>1.414</td>
<td>2.886</td>
<td>3.615</td>
<td>2.951</td>
</tr>
</tbody>
</table>

395
2.4 AFM analysis
Thin film topography of MgFe$_2$O$_4$ nanoparticles obtained by AFM (atomic force microscopy). This microscopic technique has several advantages for characterising nanoparticle. Images obtained via AFM display data in two and three dimensions, allowing for the quantitative generation of information on both individual particles and groups of particles. Fig. 4 display the 2D and 3D AFM images of MgFe$_2$O$_4$ respectively and its related histogram and size distribution. The average roughness (Rq) and root mean square roughness (RMS) values obtained about 5.982 nm and 7.541 nm respectively. Also, the maximum height (R$_p$) and a maximum depth (R$_v$) observed as 35.645 nm and 27.499 nm respectively.

2.5 Raman analysis
Raman spectroscopic technique is a powerful technique to identify the structural and vibrational properties of the material MgFe$_2$O$_4$ spinel. MgFe$_2$O$_4$ crystallises as an AB$_2$O$_4$ type spinel structure with eight formula units per unit cell in Fd$ar{3}$m space group. According to results from group theoretical calculations, AF$_e$O$_2$ type spinel structures consist of five raman active phonon modes, namely $A_{1g}$+$E_g$+$3F_{2g}$, which are made up of the motion of O ions and both A and B site ions [37,38]. Figure 5 displays the MgFe$_2$O$_4$ sample’s raman spectra in the frequency range of 100–800 cm$^{-1}$. In Table 3 and Fig. 5, several peaks have been identified. The $A_{1g}$ (1) mode is caused by the symmetric stretching of oxygen atoms along Fe–O (or M–O) tetrahedral bonds. The $E_g$ & $F_{2g}$ (3) modes are caused by the symmetric and asymmetric bending of oxygen with respect to Fe respectively. The $F_{2g}$ (2) mode is caused by the asymmetric stretching of the Fe–O bond, $F_{2g}$ (1) is due to translational movement of the whole tetrahedron (FeO$_4$) [39], while $F_{2g}$ (2) and $F_{2g}$ (3) related to the vibrations of octahedral group.

Table 2.
The observed and theoretical elemental composition value of MgFe$_2$O$_4$ obtained from EDX analysis

<table>
<thead>
<tr>
<th>Element</th>
<th>Experimental value</th>
<th>Theoretical value</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight %</td>
<td>Atomic %</td>
</tr>
<tr>
<td>O</td>
<td>21.51</td>
<td>44.41</td>
</tr>
<tr>
<td>Mg</td>
<td>11.95</td>
<td>16.23</td>
</tr>
<tr>
<td>Fe</td>
<td>66.54</td>
<td>39.35</td>
</tr>
<tr>
<td>TOTAL</td>
<td>100.0</td>
<td></td>
</tr>
</tbody>
</table>

Table 3.
Raman modes of spinel MgFe$_2$O$_4$ and their assignments

<table>
<thead>
<tr>
<th>Raman Modes (cm$^{-1}$)</th>
<th>Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>209</td>
<td>$F_{2g}$ (1)</td>
</tr>
<tr>
<td>330</td>
<td>$E_g$</td>
</tr>
<tr>
<td>479</td>
<td>$F_{2g}$ (2)</td>
</tr>
<tr>
<td>549</td>
<td>$F_{2g}$ (3)</td>
</tr>
<tr>
<td>707</td>
<td>$A_{1g}$</td>
</tr>
</tbody>
</table>

In our system, MgFe$_2$O$_4$ nanopowder contains five peaks at 209, 330, 479, 549 and 707 cm$^{-1}$ (Table 3). These bands are given the following designations: $F_{2g}$ (1), $E_g$, $F_{2g}$ (2), $F_{2g}$ (3) and $A_{1g}$ (1) correspondingly. These modes match the reported [40] values of 217, 333, 486, 554, and 715 cm$^{-1}$ for magnesium ferrites. It agrees well with the current strong band location for MgFe$_2$O$_4$ at 707 cm$^{-1}$, which J. Chandradass [41] attributed to the $A_{1g}$ mode in MgFe$_2$O$_4$.

2.6 VSM analysis
To investigate the magnetic behaviour of spinel MgFe$_2$O$_4$, SQUID based magnetometer has been employed under magnetic field upto 20 kOe. By applying a magnetic field to a material that becomes magnetized by the magnetic field, the M-H loop or hysteresis curve is
Spectroscopic, Magnetic and Morphological studies of MgFe₂O₄ Nanopowder

formed. Remanence magnetization is the value of magnetization at which field is zero. The point on the curve where the magnetization is zero and the field is negative is referred to as the coercive field, and the saturation magnetization is the point at which the magnetising force stops increasing the magnetic induction in a magnetic material. The room temperature magnetization curve of the calcined MgFe₂O₄ nanoparticle obtained from M-H loop is depicted in Fig. 6. This curve denotes hysteresis ferromagnetism and is typical for soft magnetic materials.

Magnetic parameters such as saturation magnetization \( (M_s) \), coercivity \( (H_c) \), retaintivity \( (M_r) \) and remanant ratio \( (R) \) values; 27.192 emu g⁻¹, 36.0 Oe, 0.4646 emu g⁻¹, 0.0170 respectively of ferrite nanoparticles are listed in Table 4. It was observed that the saturation value of

\[
\begin{array}{|c|c|c|c|c|}
\hline
\text{Sample} & \text{\( M_s \) (emu/g)} & \text{\( H_c \) (Oe)} & \text{\( M_r \) (emu/g)} & \text{\( \frac{M_r}{M_s} \)} \mu_B \\
\hline
\text{MgFe}_2\text{O}_4 & 27.192 & 36.00 & 0.4646 & 0.0170 \\
\hline
\end{array}
\]

Table 4.

The coercivity (\( H_c \)), saturation magnetization (\( M_s \)), retaintivity (\( M_r \)), remanant ratio (\( R \)), and magnetic moment (\( \mu_B \)) obtained at a magnetic field at 20 kOe at room temperature.
27.192 emu/g obtained in the sample calcined at 500°C (crystallite size of 39.9 nm) is close to the values of 33.4 emu g⁻¹ for bulk MgFe₂O₄ [42] and 30.6 emu g⁻¹ for sol-gel/combustion synthesized MgFe₂O₄ (crystallite size of 78 nm) [43], while it is higher than the values of 14.09 emu g⁻¹ for co-precipitation synthesized MgFe₂O₄ nanoparticles (diameters of 34.4 nm) [44]. Remanent magnetization (Mᵣ) value for the sample was obtained from the Fig. 6. The MgFe₂O₄ nanopowder's Mr/Ms ratio, which measures remanent magnetization to bulk saturation magnetization, was calculated to be 0.0170. A significant portion of superparamagnetic particles are indicated by the low Mr/Ms value. The coercivity (Hᵥ) was found to be 36 Oe (Table 4) for the MgFe₂O₄ nanoparticle. The value is less than the value of 165 Oe for MgFe₂O₄ produced by sol-gel/combustion but is comparable to the value of 48.86 Oe for nanoparticles made by coprecipitation.

Sub-lattices A and B are magnetised in opposing directions by the spins of the A and B site ions, producing a magnetic moment that is equal to the difference between the magnetic moments of the A and B site ions [45]. The magnetic moment per formula unit is given by the equation,

\[ M = Mₐ - Mₐ \]  

where \( Mₐ \) and \( Mₐ \) stand for the B- and A- sublattice magnetic moments in \( B₂ \), respectively. This formula is based on Neel's two sub-lattice model of ferrimagnetism [46]. The Ms value is typically determined by the net magnetic moment. According to the literatures [47,48], the octahedral positions are preferentially occupied by \( Mg^{2+} \) cations in the bulk magnesium ferrite, which has an inverted spinel structure. The cationic distribution on tetrahedral and octahedral lattice sites has a significant impact on the Ms of spinel ferrite nanoparticles [49].

The following relation [50] was used to compute the magnetic moment per formula unit in Bohr magneton (B) of the sample,

\[ \mu₀ = Mₛ × Mₛ / 5585 \]  

where \( Mₛ \) is the saturation magnetization in the electromagnetic unit and \( Mₛ \) is sample's molecular weight. Table 4 displayed the magnetic moment's value.

### 2.7 Optical studies

The UV-vis spectra of MgFe₂O₄ were conducted using dil HCl as a solvent in order to examine the optical characteristics of the prepared nanoparticles. The absorbance extends around 300-600 nm shows visible region which is important for numerous application in photo-catalytic and photo voltaic activity. Fig. 7(a) displays the UV-visible absorption spectra of 500°C-calcined magnesium ferrite nanopowder as a function of wavelength (nm). Typical band throughout 300-500 nm is proclaimed in optical absorption spectra of MgFe₂O₄ nanoparticles. These bands results from octahedral coordination of \( Fe^{3+} \) ions [51]. The absorption cutoff wavelength is 427.5 nm. The optical band gap energy (Eg) of magnetic nanoparticles sample was calculated according to the Tauc relationship as given below [52], where \( (α) \) absorption coefficient, \( (Eg) \) the energy gap, \( (A) \) is constant for different transition, \( (h) \) is Plank constant \( (6.62 × 10^{-34} \text{ J s}\text{ photon}^{-1}) \), \( (h\nu) \) are the energy of photon and the value of \( n \) is either \( 1/2 \) for indirect band gap or 2 for direct band gap. The intercept on the (\( h\nu \)) axis can be found by projecting the plot to the point where \( (h\nu)^2 = 0 \) as shown in Fig. 7(b). The intercept of the straight line at \( α=0 \) was used to calculate the band gap energy, which was found to be \( 2.91 \text{ eV} \), somewhat higher than the stated value range of 2.0-2.2 [53,54].

\[ Αh\nu = A(h\nu−Eg)^n \]  

### 2.8 XPS analysis

XPS is used to study the chemical and elemental state analysis at the depth of about 5-10 nm of the investigated surface of the sample. XPS is that technique which not only shows elements are present but also what other elements they are bounded to. Also it examined the energy of the component.

XPS spectrum displayed in Fig. 8, the spectrum reveals chemical composition of the sample MgFe₂O₄ nanoparticles attributed to element Mg, Fe, O and unintentional element like carbon. As shown in Fig. 8 (a) two characteristic peaks at 724.88 eV and 711.28 eV indicated as binding energy which corresponds to \( Fe^{3+}2p_{3/2} \) and \( Fe^{2+} 2p_{3/2} \) orbital which is similar to the...
Spectroscopic, Magnetic and Morphological studies of MgFe₂O₄ Nanopowder

reported earlier by Hengli Xiang [55]. The single peak located at 1303.28 eV would be attributed to Mg 1s which reveal the oxidation state Mg²⁺ in the sample which is shown in Fig. 8 (b) that is in fine agreement with some earlier results [56]. The peak located at 530.18 eV is attributed to O 1s region which appears due to metal-oxygen bonding and the peak at 532.00 eV as shown in Fig. 8 (c) represents oxygen vacancies or metal hydroxyl bonds at the surface, full spectrum of the sample are shown in Fig. 8d. Table 5 shows the elements and its peak position of Fe 2p, O 1s, Mg 2p of the sample.

Table 5. Peak position in (eV) of Fe2p, O1s, Mg2p, spectra of MgFe₂O₄ nanoparticles.

<table>
<thead>
<tr>
<th>Peak</th>
<th>Peak position (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe 2p</td>
<td>724.88</td>
</tr>
<tr>
<td></td>
<td>711.28</td>
</tr>
<tr>
<td>O 1s</td>
<td>530.18</td>
</tr>
<tr>
<td></td>
<td>532.00</td>
</tr>
<tr>
<td>Mg 2p</td>
<td>1303.28</td>
</tr>
</tbody>
</table>

Conclusions

In this study, we present evidence that sol-gel-produced magnesium ferrites nanoparticles can form a single-phase cubic spinel structure. Surface morphology image illustrated with some agglomerated size particles. The synthesized composition's Mg, Fe, and O content are confirmed by the EDX analysis spectrum. Five Raman active modes E₂g, 3F₂g, A₁g in the range of 209-707 cm⁻¹, which are anticipated in the spinel structure, were validated by a Raman investigation. Raman study confirmed the existence of five Raman active modes E₂g, 3F₂g, A₁g in the range of 209-707 cm⁻¹ which are predictable in the spinel structure. Magnetic measurement shows soft magnetic behaviour of the sample with the Mₛ value 27.129 emu/g. AFM is utilized to estimate the height, grain size, and average roughness of the particles. The band gap energy at 2.91 eV are obtained by UV-VIS absorbance spectra while chemical composition and elemental states are determined by X-ray photoelectron spectroscopy (XPS).

Acknowledgement

For financial support, authors are sincerely grateful to CSIR [No.09/013(0823)/2018-EMR-1] New Delhi, India. We appreciate Prof. Ranjan Kumar Singh from the Department of Physics at the BHU Institute of Science in Varanasi for providing the Raman spectral facility, and we also appreciate CIF from the IIT for giving technical assistance with XRD, SEM, EDX, and XPS.

Conflicts of Interest

There are no conflicts of interest, according to the authors.

Farhana Naaz – M.Sc, Research Scholar;
Preeti Lahiri – Ph.D., Professor;
Chanda Kumari – Ph.D., Senior Research Scholar;
Hemant Kumar Dubey – Ph.D., Assistant Professor.


52. C. Kumari, H.K. Dubey, F. Naaz, P. Lahiri, Structural and optical properties of nanosized Co substituted Ni ferrites by coprecipitation method, Phase Transition, 93, 1 (2020); https://doi.org/10.1080/01411594.2019.1709120.


Ф. Нааз¹, П. Лахірі¹*, Ч. Кумарі¹, Х. Кумар Дубей²

Спектроскопічні, магнітні та морфологічні дослідження нанопорошку MgFe$_2$O$_4$

¹Кафедра хімії, Інститут наук, ММВ, Університет Банараса Хіндуд, Ванарас-221005, Індія, plahiri16@yahoo.com
²Кафедра хімії, коледж BRDPG, Деорія-274001, Індія

Нано феритову сполуку типу шпінелі MgFe$_2$O$_4$ синтезовано золь-гель методом із використанням нітратів металів як прекурсорів. Фазовий склад, морфологію та елементний аналіз фериту магнію (MgFe$_2$O$_4$) здійснено методами дифракції Х-променів, перетворення Фур'є в інфрачервоному діапазоні, атомно-силової мікроскопії, енергетичної Х-променевої та скануючої електронної мікроскопії.

Х-променева дифракційна картина зразка підтверджує існування однофазного матеріалу, для якого оцінка розмірів кристалітів склала 39,9 нм. Дослідження в інфрачервоному діапазоні із використанням перетворенням Фур’є підтвердило наявність коливань метал-кисень, що відповідають тетраедричним і октаедричним вузлам, відповідно. Із результатів скануючої електронної мікроскопії отримано розмір зерен, який склав приблизно 97,7 нм. Спектри комбінаційного розсіювання зразка демонструють п’ять активних мод розсіювання (A$_{1g}$ + E$_{g}$ + 3F$_{2g}$), що сумісно зі структурою шпінелі. Дослідження магнітних характеристик при кімнатній температурі показує поведінку петлі гістерезису з низьким значенням намагніченості насичення, 27,192 emug$^{-1}$ і незначним значенням коерцитивної сили. Ширину забороненої зони визначено за допомогою спектрів пропускання в УФ-діапазоні. Крім того, для підтвердження ступенів окислення та дослідження хімічного складу зразка використано методи рентгенівської фотоелектронної спектроскопії.

Ключові слова: феритова шпінель, нанокристали, дифракція X-променів, спектри комбінаційного розсіювання, магнітні властивості.