Platinum Open Access Journal (ISSN: 2069-5837)

https://doi.org/10.33263/BRIAC116.1503715050

FTIR, Optical, Electrical and Magnetic Properties of Sm³⁺ Doped Mg Nano Ferrites

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Received: 1.03.2021; Revised: 1.04.2021; Accepted: 4.04.2021; Published: 9.04.2021

Abstract: Synthesized Sm, doped Mg nano ferrites with composition MgSm_xFe_{2-x}O₄ (x = 0,0.025, 0.050, 0.075, 0.100) using the technique of citrate-gel auto-combustion method were analyzed through various experimental techniques. XRD analysis confirmed single-phase cubic spinel structure, while FTIR spectroscopic analysis displayed two absorption peaks that are characteristic of spinel nano ferrites. UV-visible spectral analysis was carried out to study the optical absorption behavior of the prepared ferrites. Two probe method was used to study the dc electrical resistivities of Sm doped Mg nanoferrites between 200 °C–500 °C. A plot between log (σ T) vs. inverse temperature yields a curve that reveals prepared nano ferrites' semiconducting nature. A study of TEP (Thermo Electric Power) for prepared samples was carried out at a high temperature (573K). Seebeck coefficient and Curie Temperature of Sm doped Mg nanoferrites were observed. Measurements of magnetization were done by using VSM (Vibrating Sample Magnetometer).

Keywords: citrate-gel auto combustion method; X-ray diffraction; FTIR; TEP; VSM

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1. Introduction

Material science gained significance in research due to interesting applications and properties exhibited by materials in various fields. It deals with a variety of applications in chemistry, physics, biology, medicine, and engineering. The origin of material science is to observe the structure of materials and their properties. The main aspect of material science is the characterization of materials whose properties and performance are associated with the materials' microstructure. Magnetic nanomaterials exhibit a variety of applications such as storage of data, MRI, magnetic fluids and biotechnology, etc. [1-3]. The preparation method of these materials is very significant due to its control over the size distribution, topography, shape, density of the particles on which its behaviors strongly depend. Iron oxide nanoparticles have unique properties like ultrafine sizes and a large surface area to mass ratio and are the most used superparamagnetic nanoparticles for various biomedical applications [4]. Mn nano spinel ferrite is a soft ferrite with low loss and high magnetic permeability. Different kinds of applications include magnetic recording media, transformer coils, microwave devices, computer memory chips, etc. Magnetic nano-ferrite particles gained special attention over the last few years. These particles are widely used in high-density magnetic recording [5]. Low cost, high curie temperature, high saturation magnetization, and hysteresis loop properties

make them highly suitable for microwave devices, high-density recording media, and as absorbents [6]. They exhibit specific properties compared to bulk due to atoms' occupation in large volume at grain boundary area, which leads to unusual properties like dislocation, spin canting, superparamagnetic (sp) and surface anisotropy etc. This property makes these materials to be tailored flexibly for specific applications [7]. Ferrites are widely used in various magnetic devices like inductors, transformers, and magnetic heads in high-frequency resonance circuits [8]. The interesting physical and chemical properties of the nano ferrites arise from the distribution of cations among tetrahedral (A) and octahedral (B) sites [9]. They are also used in MRI, Target drug delivery, Hyperthermia for cancer treatment [10-13], high-density storage devices, and magnetic fluids [14-15]. Simi Debnath *et al.* [16] studied the effect of cobalt doping on nickel ferrite nanocrystals' structural and magnetic properties. It was observed that the crystallite size is decreasing with the substitution of magnesium (Mg²⁺) in ZnFe₂O₄ [17]. The dc-electrical measurements of magnesium doped nickel ferrite Ni_{0.6}Mg_{0.4}Fe₂O₄ show semiconducting behavior for which conductivity is governed by a small polaron hopping process [18].

Since rare-earth ions favor second phase occurrence leading to enhanced electrical resistivity and bulk density, the electronic valence of rare-earth metal ions plays a vital role in compound formation. It is known that rare-earth ions are more stable when the cations Ce and Tb are both trivalent and tetravalent while Sm and Dy are divalent and trivalent [19] to their large radius compared to that of Fe³⁺ ions. For the composition with the orthorhombic second phase, the lattice constant is slightly smaller than unsubstituted ferrite and will decrease with the increase of rare-earth ion radius, which suggests a solubility limit for rare-earth ions [20]. Synthesis of nanosized spinel ferrites was done through sol-gel, co-precipitation, micro-emulsion, hydrothermal, reverse micelle [21-24], ceramic [25], solid-state reaction [26], combustion [27-28], and spark plasma sintering [29]. The present work reports the synthesis, optical, FTIR, electrical, and magnetic properties of Mg-Sm ferrites with the help of citrate gel auto combustion technique.

2. Materials and Methods

Ferrite particles of chemical formula Mg Sm x Fe_{2-x} O₄ (x = 0.0 to 0.1 with steps of 0.025) by using magnesium, samarium, ferric nitrates, citric acid, and ammonia of 99% purity as raw materials were synthesized by using the technique of citrate-gel auto combustion at low temperature. The required amount of metal nitrate and citric acid were dissolved in distilled water and was stirred to form a clear homogenous solution and heated up to 80 °C. Later the pH was set to 7 by adding ammonia. This liquid was evaporated at about 180 °C resulting in a burned powder that was ground with agate mortar and calcinated at 500 °C/4 hours and cooled to normal temperature [30]. Structural characterization related prepared samples were taken up with an X-ray diffractometer (Philips) using Cu K_a radiation (λ =1.5405 Å) within the Bragg's range 10° to 80° with a step size of 4°/min.

3. Results and Discussion

3.1. XRD analysis.

XRD was used as an efficient tool for the characterization of crystalline powders. Confirmation of phase formation and the microstructural study was done through XRD analysis of Mg Sm x Fe_{2-x} O₄ (x = 0.0 to 0.1) samples. The XRD patterns were depicted in Figure 1 and https://biointerfaceresearch.com/

indexed as (111), (220), (311), (222), (422), (511), (440). It indicated the cubic spinel structure of ferrites with a single-phase without any impurity pickup.



Figure 1. X-ray diffraction patterns of MgSm_xFe_{2-x}O₄.

The crystalline size of the sample is given by the following relation

$$\mathsf{D} = \frac{0.94\lambda}{\beta \cos\theta}$$

where λ = wavelength of X-ray used, β = Full-Width Half Maxima (FWHM) in radians and θ = peak position

Figure 2 indicates crystallite size of prepared samples ranging between 15 nm to 20 nm and lattice constant value increase with doping of samarium that confirms unit cell expansion with doping of rare-earth [31].



Figure 2. Average crystalline size variation for Sm doped Mg ferrites.

The lattice parameter (a) of the sample was calculated by the formula
$$a = d * (h^2 + k^2 + l^2)^{1/2}$$

where a = Lattice Constant, (hkl) are the Miller Indices and d = inter planner spacing.

Figure 3 shows the variation of lattice parameter with Sm^{+3} ion content. Sm doped Mg nano ferrite's lattice parameter increases with Sm content initially and further decreases in small proportion. Such variation in the lattice parameter is due to F^{+3} ions having a smaller ionic radius (0.067 nm) than that Sm^{+3} (0.0958) [32]. The volume of the unit cell was calculated using the formula:

The volume of a unit cell $V = a^3$ where V- the volume of the unit cell, a - lattice parameter.



Figure 3. Lattice parameter variation vs. Sm doped Mg ferrites.

Figure 4 depicts the volume of unit cell vs. composition of the sample. The figure shows that the volume of the unit cell is changing with the doping concentration of Sm.

The X-ray density
$$dx = \frac{nM}{a^3N} [g/cm^3]$$

where, M = molecular weight of the sample, n = number of molecules in a unit cell of spinel lattice, a = lattice parameter, and N is the Avogadro number.



Figure 4. Volume of unit cell variation for Sm doped Mg ferrites.

The X-ray density of the prepared samples is directly proportional to the molecular weight. FromFigure 5,the variation of X-ray density with Sm^{+3} ion content, it is observed that the effect of an increase in the molecular weight of the sample due to the doping of Sm^{+3} ion in place of lighter Fe⁺³ ions. X-ray density increases with an increase in Sm^{+3} ion doping in Mg ferrite. It is inferred that the change in X-ray density depends on the mass of the doped ions in place of Fe⁺³ ions [33].The crystallite size, lattice parameters, x-ray density, and volume of the unit cell for compositions of MgSm _xFe_{2-x}O₄ (x = 0.00 - 0.1) are given in Table 1.



Figure 5. X-ray density variation for Sm doped Mg ferrites.

Table 1.Crystallite size, lattice parameters, x-ray density and volume of the unit cell for various compositions of MgSm Fee. O: (x=0.00, 0.1)

| S. No | Name of the composition | Avr.cry. size (nm) | Lattice constant (A ^o) | X-ray density (gm/cc) | Vol. of the unit cell (A ^o) |
|-------|--|-----------------------|---------------------------------------|--------------------------|--|
| 1 | MgFe ₂ O ₄ | 15.21 | 8.44 | 4.39 | 601.21 |
| 2 | MgSm0.025Fe1.975O4 | 16.08 | 8.45 | 4.45 | 603.35 |
| 3 | MgSm0.050Fe1.95O4 | 13.79 | 8.40 | 4.58 | 592.70 |
| 4 | MgSm _{0.075} Fe _{1.925} O ₄ | 20.36 | 8.37 | 4.69 | 585.37 |
| 5 | MgSm _{0.1} Fe _{1.9} O ₄ | 19.31 | 8.36 | 4.76 | 584.27 |

3.2. FTIR spectroscopic analysis.

FTIR spectra act as a powerful technique for structural characterization. FTIR spectral analysis witnesses the formation of spinel structure of synthesized Sm, doped Mg nano ferrites. FTIR spectra of the prepared ferrites were recorded between 200 to 3000 cm⁻¹at normal temperature and were used to locate the band positions. The spectra are recorded in a KBr pellet that is mixed in a 1:20 ratio. The FTIR spectra of all the compositions were shown in Figure 6, where % Transmittance was plotted against wavenumber. Two clear absorption bands v1 and v2 were observed from the figure at about 600 cm⁻¹ and 395 cm⁻¹, signifying the intrinsic stretching vibrations of tetrahedral (A) and octahedral (B) sites, respectively. The first Absorption band identified in the range of 626–570 cm⁻¹ indicates the stretching vibration of tetrahedral M-O bond, and the second band detected in the range of 496 to 400 cm⁻¹ results from stretching vibrations of the metal-oxygen band at the octahedral site [34-35].

Vibrational spectra of ferrites were studied by Waldron and Hafner and ascribed v_1 at about 600 cm⁻¹ (high-frequency band) to A site and v_2 at about 400 cm⁻¹ (low-frequency band) to B site. The observed absorption bands within this range indicate single phased spinel structure formation with two sub-lattices, namely tetrahedral and octahedral sites A & B. The variance between v_1 and v_2 is because of the differences in distances of positions of Fe³⁺and O²⁻at tetrahedral and octahedral site.



Figure 6. FTIR spectra of Mg-Sm series (a) $MgFe_2O_4$ (b) $MgSm_{0.025}Fe_{1.975}O_4$ (c) $MgSm_{0.050}Fe_{1.960}O_4$ (d) $MgSm_{0.075}Fe_{1.925}O_4$ (e) $MgSm_{0.1}Fe_{1.9}O_4$.

3.3. Optical studies.

The optical studies of synthesized nano ferrites were studied by UV–DRS (diffuse reflectance spectroscopy). The UV-DRS spectroscopy absorption and reflection in the UV region were performed using reference, which is barium sulfate (BaSO4)with absorbance versus wavelength. In the absorption, molecules of electron or non –bonding (n-electron) can absorb the energy in the form of ultraviolet or visible light to excite this electron to a higher or anti-bonding molecular orbit. Sm doped Mg ferrites wavelength is 520 nm regions shown in Figure 7, and it is a visible region.

From the spectra, absorption bonds wavelength is a low region with dopant concentration due to the variation of ionic radii of Sm^{3+} and Fe^{3+} ions. Table 2 indicates

obtained band gap energies for Sm doped Mg nano ferrites. The bandgap energy increases with increasing Sm^{3+} content, which is 2.50 to 2.61 eV [36-37].



Figure 7. UV-Vis spectra of Sm doped Mg nano ferrites.

| Name of the composition | Cut off wavelength (nm) | Bandgap energy (eV) | |
|--|-------------------------|---------------------|--|
| MgFe ₂ O ₄ | 495 | 2.50 | |
| MgSm _{0.025} Fe _{1.975} O ₄ | 493 | 2.51 | |
| $MgSm_{0.050}Fe_{1.95}O_{4}$ | 490 | 2.53 | |
| MgSm _{0.075} Fe _{1.925} O ₄ | 485 | 2.55 | |
| MgSm _{0.1} Fe _{1.9} O ₄ | 475 | 2.61 | |

| Table 2. Bai | nd gap energy | of Sm-Mg nano | ferrite series. |
|--------------|---------------|---------------|-----------------|
| | | | |

3.4. Electrical properties.

3.4.1. DC electrical resistivity of Sm doped Mg nanoferrites

The DC electrical resistivity is an important property to study the conduction mechanism in nano ferrites, which was due to the hopping of electrons between the ions of the same elements. The probability of hoping depends upon the separation between the involving ions and their activation energy. The electrostatic interactions between the conduction of electrons and nearby ions result in polarization of the surrounding region. The electron is present at the center of polarization. This electron is transferred to the neighboring site by the thermal activation energy. This kind of mechanism in conduction is called the hoping mechanism [38]. In the prepared Sm doped Mg nano ferrite samples, the variation of resistivity has been observed that as the temperature increases, resistivity decreases. It indicates the normal behavior of semiconductors. And also, it gives the conductivity nature of a semiconductor.

The dc electrical conductivity of the materials given by $\sigma = \sigma_0 \exp(-E_a/kT)$ where E_a , σ_0 , k are thermal activation energy, a pre-exponential factor that depends on materials composition and Boltzmann constant. The prepared samples' electrical conductivity was studied in the temperature range between 200 °C and 500 °C. The temperature dependence of prepared nano ferrites' electrical conductivity was studied by plotting a graph between the log (σ_T) vs. 1000/T is shown in Figure 8. By plotting the graph, we get a curve whose slope can be used to calculate the thermal activation energy of nano ferrite samples tabulated in Table 3 [39].



Figure 8. DC electrical resistivity of Mg-Sm nano ferrites. (a) $MgFe_2O_4$ (b) $MgSm_{0.075}Fe_{1.925}O_4$ (c) $MgSm_{0.025}Fe_{1.975}O_4$.

Table3. Electrical properties of ferrite composition $Sm_xFe_{2-x}O_4$ (x = 0 to 0.1 in steps of 0.025), Curietemperature (T_c), activation energy in para region (E_P) eV and in ferri region (E_F) eV.

| Composition | Curie Temp (°C) | Para Region (E _P) eV | Ferri Region (E _F) eV |
|--|-----------------|----------------------------------|-----------------------------------|
| MgFe ₂ O _{4.} | 489 | 0.0571 | 0.03214 |
| MgSm _{0.025} Fe _{1.975} O ₄ . | 478 | 0.0403 | 0.0338 |
| MgSm _{0.075} Fe _{1.925} O ₄ . | 470 | 0.0554 | 0.03946 |

3.4.2. Thermoelectric power studies of Sm doped Mg nanoferrites.

Studies on thermoelectric power related to prepared samples were taken up by differential method from room temperature to 300 °C. Given the thermal stability, thermo emf was measured during the cooling cycle temperature than the heating cycle. The Seebeck coefficient of the current samples was calculated from observed values whose thermal emf values are enlisted in Table.4. It indicates a gradual increase in the Seebeck coefficient with an increase in the concentration of Sm. This might be attributed to the transfer of Fe⁺³ to A-site resulting in decreasing of Fe⁺³ ions in B-site [40]. For a cubic system, the Seebeck coefficient is given by

S =Total number of Fe $^{+3}$ ions in B-site / Total number of Fe $^{+2}$ ions in B- sites.

The above explanation infers that an increase in Sm concentration produces many charge carriers that require more energy for transferring charge carriers between various cations. Hence, large emf is released, enhancing thermoelectric power. Figure 9 shows the change in Seebeck coefficient with temperature for all the prepared samples, indicates a low positive Seebeck coefficient at low temperature. As the temperature increases, the Seebeck

coefficient also increases for all the prepared samples under investigation. This indicates that all the prepared samples behave like p-type semiconductors.

Table4. Curie temperature and Seebeck coefficient of prepared $MgSm_xFe_{2-x}O_4$ (x = 0.000, 0.025, 0.050, 0.075,

| 0.1). | | | | |
|---|-------------------------------------|---------------------|--|--|
| Composition | Curie Temperature (T _c) | Seeback coefficient | | |
| Mg Fe ₂ O ₄ | 563 | 4000 | | |
| Mg Sm _{0.05} Fe _{1.95} O ₄ | 478 | 3250 | | |
| Mg Sm _{0.1} Fe _{1.9} O ₄ | 539 | 4300 | | |

With the increase in temperature, conductivity in these ferrites was due to electrons which was attributed to the hoping of electrons between Fe^{+2} and Fe^{+3} ions at B-sites given by:



Figure 9. Variation of Seebeck coefficient (S) with temperature of Mg-Sm nano ferrite (a) MgFe₂O₄; (b) MgSm_{0.050}Fe_{1.950}O₄; (c) MgSm_{0.1}Fe_{1.9}O₄.

The Seebeck coefficient's observed values indicate an increase with increasing temperature due to the release of more p-type charge carriers. Further increase in temperature leads to the sudden decrease in Seebeck coefficient at a particular temperature and gradually increases once again. This sudden change in the Seebeck coefficient value's value was named Transition temperature (Tc).

The transition temperatures for all prepared nano ferrites were measured from Figure 9 and are presented in Table 4. They become paramagnetic once the transition temperature is reached. Hence probability for hoping of electrons between the A-sites and B-sites beyond Tc is zero. Thus, the Seebeck coefficient becomes constant after transition temperature [41]. The transition temperature of homogenous Mg ferrite was around 294 °C (568 K). This work reports

 $Fe^{+2} \rightarrow Fe^{+3} + 1e^{-1}$

thermoelectric power measurements carried between room temperature to $300 \text{ }^{\circ}\text{C}$ (300K-568 K) using the differential method.

It is clear that thermoelectric power study of Sm doped Mg nano ferrites exhibit a welldefined transition at curie temperature similar to susceptibility, permeability, and spontaneous magnetization. The Seebeck coefficient is minimum at Tc, indicating the magnetic ordering as a marked effect similar to thermoelectric power of samples under investigation. The present work semiconducting behavior of current samples indicates that charge carriers are supposed to be localized at ions or vacant sites. The transition temperature increases with increased Sm concentration doping, reducing Fe⁺³ ions, which decreases the Fe ion number in B–sites, leading to increased A-B interaction Fe⁺³ – o – Fe⁺³ [42].

3.5. Magnetic properties of Sm doped Mg ferrites.

Measurements of magnetization at room temperature were done with Vibration Sample Magnetometer (VSM). This was done by forming the prepared nano ferrite samples into pellets and calcinated at 500 °C for four hours with 4 °C /min. Hysteresis loops from VSM for Sm doped Mg ferrites shown in Figure 10 indicated magnetization (M) dependence on applied magnetic field (H).

| Table 5. Various magnetic parameters of sin doped wig nano retries. | | | | | | |
|---|--------|------------|---------------------|-------------------------|-----------------|--|
| Composition | M.W | Ms(emu/gm) | H _C (Oe) | M _R (emu/gm) | $S = M_R / M_S$ | |
| Mg Fe ₂ O _{4.} | 199.91 | 37.63 | 76.45 | 8.21 | 0.21 | |
| Mg Sm0.025 Fe1.975O4 | 202.35 | 7.34 | 67.10 | 1.01 | 0.13 | |
| Mg Sm0.075 Fe1.925 O4 | 207.07 | 36.51 | 88.00 | 7.14 | 0.19 | |

Table 5. Various magnetic parameters of Sm doped Mg nano ferrites

Different magnetic parameters such as saturation magnetization Ms, Remanence magnetization Mr, Coercivity H_C were measured and tabulated using the hysteresis loops. Hysteresis loop (Figure 10) indicated the pure Mg ferrite with high squareness ratio was the hard ferrite, and maximum Sm, doped ferrite (x = 0.025) with least squareness ratio was the soft ferrite. Hence substitution of Sm in the Mg ferrite changes the system from hard to soft ferrite. It is reported that the shape and width of M - H loop depends on various factors, including fabrication technique, chemical composition, sintering temperature by time and also grain size, etc. [43-44]. The calculated values indicate that saturation and remanent magnetization values in Mg nano ferrites decrease with increasing doping concentration of Sm shown in Table 5. The saturation magnetization ($M_s = 36.51 \text{ emu/gm}$) value of the MgSm_{0.075}Fe_{1.925}O₄ sample showed that the samples have applications like microwave and recording devices [45]. Coercivity is influenced by the doping of rare earth material ions [46]. The coercivity value range between 76–88 Oe. The synthesized Sm doped Mg nano ferrites squareness ratio decreased with substituted concentration. From these results, it is observed that by increasing Sm concentration in the prepared Mg nano ferrites, the samples' magnetic property converts from hard magnetic to soft magnetic. Such magnetic materials can be used to fabricate soft magnets, used in transformers cores, motors, electromagnets, etc.



Figure 10. Hysteresis (B-H loop) curves of Sm doped Mg ferrites (a) $Sm^{3+} = 0.000$ (b) $Sm^{3+} = 0.025$ (c) $Sm^{3+} = 0.075$ (d) $Sm^{3+} = 0.000$, 0.025, 0.075 (combined graph).

4. Conclusions

A series of samarium doped Mg nano ferrites $MgSm_xFe_2-xO_4$ with (x = 0.000,0.025, 0.050, 0.075, 0.1) were synthesized by citrate-gel auto combustion method. XRD analysis confirmed that the formation of a single-phase spinel structure without any impurity in all the compositions. The substitution of samarium in Mg ferrite has increased lattice parameter and average crystallite size. The decrease in lattice parameter for the composition x = 0.050, 0.075indicates a possible cationic distribution. The absorption bands in FTIR spectra of all the prepared samples are found in an expected range that is 400-600 cm⁻¹. The UV-Visible spectroscopy gave the optical absorption range of 450–500 nm and observed that by samarium concentration increases, absorbance decreases. From the thermoelectric study of prepared samples, the Seebeck coefficient of Sm doped Mg nano ferrite was low and positive at low temperature that indicates p-type semiconducting behavior, and as the temperature increases, the p-type semiconducting nature also increases. The saturation magnetization decreased with an increase in samarium concentration. This confirms that the change is hard ferrite material into soft ferrite by substitution of samarium. The change in coercivity with samarium concentration is explained based on the variation of nanoparticles' coercivity in the multidomain region.

Funding

This research received no external funding.

Acknowledgments

The authors are very grateful to Prof.K Bhaskar, Head, Department of Chemistry, University College of Science, Osmania University, Hyderabad. The authors are also very grateful to Dr. Shekar Matta, Principal, Dr. B. R. Ambedkar College, Baghlingampally, Hyderabad J. S. Harinakshi, Vice Principal, Dr. B. R. Ambedkar College, Baghlingampally, Hyderabad and S. Narendar Faculty, Department of Physics, for their encouragement in the present Research work.

Conflicts of Interest

The authors declare no conflict of interest.

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