

FTIR, Optical, Electrical and Magnetic properties of Sm^{+3} doped Mg Nano Ferrites by Citrate-Gel Auto combustion method

Venkatesh Nakiraboina

Osmania University

Shyam sunder Goud

Osmania University

Hari Kumar Nama

Osmania University

D Ravinder (✉ ravindergupta28@rediffmail.com)

Osmania University <https://orcid.org/0000-0002-0626-3034>

Veera Somaiah P

Osmania University

Research Article

Keywords: Citrate-gel auto combustion method, X-ray diffraction, FTIR, TEP, VSM

Posted Date: February 24th, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-227009/v1>

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Abstract

Sm, doped Mg nano ferrites with composition $Mg_{1-x}Sm_xFe_2O_4$ (where $X = 0.000, 0.025, 0.050, 0.075, 0.1$) were synthesized by citrate-gel auto-combustion method. The Samples were analyzed through various experimental techniques. The structural confirmation was done with the basic characterization techniques such as XRD (X-ray Powder Diffraction) and Fourier Transform Infrared (FTIR) spectroscopic analysis. To study the optical absorption behavior of the prepared ferrites, UV-visible spectral analysis was carried out. XRD analysis established the formation of single-phase cubic spinel structure of the materials. FTIR spectra has shown two absorption peaks that are characteristic of spinel nano ferrites. The dc electrical resistivities of the Sm doped Mg nano ferrites were studied by using two probe method at the temperature range of 200–500 °C. A plot of $\log(\sigma T)$ vs inverse of temperature yields a curve which reveals a semiconducting nature of prepared nano ferrites. The Thermo Electric Power (TEP) studies of prepared samples carried out at high temperature (573K). The values of the Seebeck coefficient and curie temperature of Sm doped Mg nano ferrites were observed. The magnetization measurements were carried out by using Vibrating Sample Magnetometer (VSM).

1. Introduction

Material sciences have become significant in research because of their interesting properties and applications in various fields. Basically, material science contains variety of applications in various fields such as Physics, Chemistry, Biology, Medical, and Engineering etc. Material science origin is to observe the structure of the materials, and also their properties. The main aspect of the material science is characterization of materials. Materials properties and performance are associated with microstructure of the materials.

Magnetic nano materials are one kind of materials, which are having variety of applications such as storage of data, MRI, magnetic fluids and biotechnology etc. the magnetic nano materials preparation method is very significant due to its control over the size distribution, topography, shape, density of the particles on which its behaviors strongly depend. Mn nano spinel ferrite is soft ferrite, it contains high magnetic permeability and low losses. These materials have different kinds of applications such as magnetic recording media, transformer coil, microwave devices, computer memory chip etc.

Magnetic nano ferrite particles have received special attention over the last years. These nano particles are widely used in high density magnetic recording [1]. Their low cost, high saturation magnetization high curie temperature and hysteresis loop properties make them excellent candidates for high-density recording media, absorbents, and microwave devices [2] the nano magnetic particles have special properties as compared to the bulk because of the large volume fraction that atoms occupy at the grain boundary area, which in turn is responsible for several unusual properties like spin canting, surface anisotropy, super paramagnetic (sp), dislocations etc. This makes them quite flexible to tailor the material for specific applications [3]. Ferrites are extensively used in different types of magnetic devices such as transformers, inductors, magnetic heads, in resonance circuits for high frequency [4]. The interesting

physical and chemical properties of the nano ferrites arise from their distribute the cations among the tetrahedral (A) and octahedral (B) sites [5] Magnetic Resonance Imaging (MRI), Target drug delivery Hyperthermia for cancer treatment [6,7] high density storage devices, magnetic fluids [8,9] It was found that all rare-earth ions favor the occurrence of second phase, resulting in an increase of the electrical resistivity and bulk density. The electronic valence of the rare- earth metal ions is most important for compound formation. In general, rare-earth ions are most stable when they cations, where Ce and Tb are both trivalent and tetravalent while Sm, Dy are divalent and trivalent [10] owing to their large radius compared to that of Fe^{3+} ions, the lattice will be distorted, generating internal stress and increasing the lattice constant. For the composition with orthorhombic second phase, the lattice constant is slightly smaller than un substituted ferrite and will decrease with the increase of rare-earth ion radius which suggests the existence of solubility limit for rare-earth ions [11] Several methods are used for synthesizing nano sized spinel ferrites, such as co-precipitation, sol-gel, micro-emulsion, hydrothermal and reverse micelle [12-14]. Refluxing process [15], Ceramic Method [16], Hydro Thermal Method [17], Combustion Method [18], Spark Plasma Sintering [19] and ball milling method etc. In the present work we reported the results of synthesis and structural properties of Ni-Cd-Sm Ferrites by non-conventional citrate gel auto combustion method.

2. Experimental Method

2.1. Synthesis: The composition of Ferrite particles having chemical formula $Mg Sm_x Fe_{2-x} O_4$ (where $X = 0.000, 0.025, 0.050, 0.075, 0.1$), were synthesized by citrate-gel auto combustion method at low temperature. Magnesium nitrate, Samarium Nitrate, Ferric Nitrate, Citric acid and ammonia (All chemicals are 99% pure AR Grade SDFCL sd Fine chemical Ltd) are raw materials for the synthesis process. Calculated quantities of metal nitrates and citric acid were dissolved in minimum amount of distilled water to get clear solution. Here citric acid acts as a chelating agent and helps in the homogenous distribution of metal ions. The above mixture was stirred to get homogenous clear solution which is heated to $80^\circ C$ using a hot plate magnetic stirrer. Then the pH of the solution is adjusted at 7 by addition of ammonia. A sol is formed. The resulting solution was evaporated to dryness heating at about $180^\circ C$ on a hot plate with continuous stirring. The gel gave a fast flameless auto combustion reaction with the evolution of large number of gases which results a burned powder. The burned powder was grinding using Agate Mortar and pestle to get a fine ferrite powder. Finally, the grinded powder was calcinated in air at $500^\circ C$ for 4 hours and cooled to room temperature.

2.2 Characterization: The structural characterization of the synthesized samples was carried out by Philips X-ray diffractometer using $Cu K_\alpha$ radiation of wavelength 1.5405 \AA at room temperature by continuous scanning in the range of Bragg's angles 10° to 80° in steps of $4^\circ/\text{min}$ to investigate the phase and crystalline size).

3. Results And Discussion

3.1.XRD Analysis

For the characterization of crystalline powders, X-ray diffraction (XRD) analysis is used as a powerful tool. Thus, to confirm the phase formation and for micro structural studies, the x-ray Powder Diffraction analysis was carried out on the synthesized **Mg Sm_x Fe_{2-x} O₄ (where X = 0.000, 0.025, 0.050, 0.075,0.1)**. nano ferrite powder samples. The X-ray diffraction patterns of the samples were depicted in figure 1. It was indexed as (111), (220), (311), (222),(422), (511), (440). It indicates the formation of cubic spinel structure of the ferrites with single phase without any impurity pickup.

Scherrer Formula:

$$\text{Crystalline size of the sample } D = \frac{0.94\lambda}{\beta \cos\theta}$$

Where λ =wavelength of X-ray used

β = Full Width Half Maxima (FWHM) in radians.

θ = peak position.

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

d = inter planner spacing,

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

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.

Values of Crystallite size, lattice parameter, X-ray density and volume of all the samples were given in the table (1).

Table (1): Crystalline size, Lattice Parameter, X-ray density & volume.

| S.No | Sample | Mol. wt (gm/mol) | Crystallite size (nm) | Lattice constant (Å) | X-ray density (gm/cc) | Volume (Å) ³ |
|------|--|------------------|-----------------------|----------------------|-----------------------|-------------------------|
| .1 | MgFe ₂ O ₄ | 199.991 | 15.21 | 8.44 | 4.39 | 601.21 |
| 2 | MgSm _{0.025} Fe _{1.975} O ₄ | 202.353 | 16.08 | 8.45 | 4.45 | 603.35 |
| 3 | MgSm _{0.050} Fe _{1.95} O ₄ | 204.716 | 13.79 | 8.40 | 4.58 | 592.70 |
| 4 | MgSm _{0.075} Fe _{1.925} O ₄ | 207.079 | 20.36 | 8.37 | 4.69 | 585.37 |
| 5 | MgSm _{0.1} Fe _{1.9} O ₄ | 209.442 | 19.31 | 8.36 | 4.76 | 584.27 |

From the table we can observe that the crystallite size of the prepared samples were in the range of 15nm to 20nm. Value of lattice constant is increasing with samarium doping which shows the expansion of unit cell with rare earth doping.

Lattice parameter of Sm doped Mg nano ferrites:

The variation of lattice parameter with Sm⁺³ ion content shown in fig 2. The lattice parameter of Sm doped Mg nano ferrite increases with Sm content initially. Further decreases in small proportion. Such a change in the lattice parameter because of F⁺³ ions which are smaller radius (0.067 nm) then that of ionic radius of Sm⁺³ (0.0958).

X-ray density of Sm doped Mg nano ferrites:

The X-ray density of the prepared samples are directly proportional to the molecular weight. From the above fig 3. the variation of X-ray density with Sm⁺³ ion content, it is observed that the effect of 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.

3.2 Fourier Transform Infrared Spectroscopy Analysis (FTIR)

FTIR spectra acts as a powerful technique for the structural characterization. FTIR spectral analysis witnesses the formation of spinel structure of synthesized Sm, doped Mg nano ferrites. FTIR spectra of the prepared ferrites was recorded in the range of 200 to 3000cm⁻¹ at room temperature and was used to locate the band positions. The spectra is recorded in KBr pellet that is mixed in 1:20 ratio. The FTIR spectra of all the compositions were shown in the figure 6 where % Transmittance was plotted against wavenumber. From the figure, two clear

absorption bands ν_1 and ν_2 were observed at about 600cm^{-1} and 395cm^{-1} which signify the intrinsic stretching vibrations of tetrahedral (A) and octahedral (B) sites respectively. The first Absorption band identified in the range of $626 - 570\text{cm}^{-1}$ indicate the stretching vibration of tetrahedral M-O bond and the second band detected in the range of 496 to 400cm^{-1} results from stretching vibrations of metal-oxygen band at octahedral site.

Vibrational spectra of ferrites were studied by Waldron and Hafner and ascribed the high frequency band (ν_1) at about 600cm^{-1} to tetrahedral (A) site and low frequency band (ν_2) at about 400cm^{-1} to octahedral (B) site. The observed absorption bands within these specific range indicate the formation of single phased spinel structure with two sub-lattices namely tetrahedral (A) site and octahedral (B) site. The variance between ν_1 and ν_2 is because of the differences in distances of positions of Fe^{3+} and O^{2-} at tetrahedral an octahedral site.

3.3 Optical Studies

The optical studies of synthesized nano ferrites studied by UV-DRS (diffuse reflectance spectroscopy). The UV-DRS spectroscopy absorption and reflection in the UV region and, it was performed using reference which is barium sulphate (BaSO_4) with absorbance verses 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 higher or anti-bonding molecular orbit. Sm doped Mg ferrites wave length is 520 nm regions, and it is a visible region.

3.4 Electrical Properties:

DC resistivity of Sm doped Mg nano ferrites.

The Dc electrical resistivity is one the important property to study the conduction mechanism in nano ferrites. The conduction mechanism in nano ferrites was due to hoping charge carries (electrons) between the ions of same elements. The probability of hoping depends upon the separation between the involving ions and their activation energy [20]. The electrostatic interactions between the conduction of electrons and nearby ions result in polarization of surrounding region so that, 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 hoping mechanism [21]. In the prepared Sm doped Mg nano ferrite samples, the variation of resistivity have been observed that as the temperature increases resistivity decreases. It indicates normal behavior of semiconductor. And also, it gives the conductivity nature of a semi-conductor.

The dc electrical conductivity of the materials given by $\sigma = \sigma_0 \exp(-E_a / kT)$ where **Ea** is the thermal activation energy, **σ_0** is the pre-exponential factor which depends on the nature of the material composition and **k** is the Boltzmann constant. The electrical conductivity of the prepared samples were studied under the temp range 200°C to 500°C . The temperature dependance of electrical conductivity of prepared nano ferrites studied by plotting a graph between the $\text{Log}(\sigma T)$ vs $1000/T$. By plotting the graph,

we get a curve whose slope can be used to calculate the thermal activation energy of nano ferrite samples [22]

Table-2 Electrical properties of Ferrite Composition $MgSm_xFe_{2-x}O_4$ (where X = 0.000, 0.025, 0.050, 0.075, 0.1) Curie Temperature T_c (K) Para Region (E_p) eV Ferri Region (E_f) eV

| S.No | composition | Curie Temp ($^{\circ}C$) | Para Region (E_p) eV | Ferri Region (E_f) eV |
|------|-----------------------------|----------------------------|--------------------------|---------------------------|
| 1 | $MgFe_2O_4$ | 489 | 0.0571 | 0.03214 |
| 2 | $MgSm_{0.025}Fe_{1.975}O_4$ | 478 | 0.0403 | 0.0338 |
| 3 | $MgSm_{0.075}Fe_{1.925}O_4$ | 470 | 0.0554 | 0.03946 |

3.5 Thermo electric power studies of Sm doped Mg nano ferrites:

The thermo electric power studies of the prepared Sm, doped Mg nano ferrites are measured by the differential method at room temperature to $300^{\circ}C$. The thermo e m f of the prepared samples were measured during the temperature cooling cycle. This is because during the cooling cycle the sample having more thermal stability than heating cycle.

The values of the See beck coefficient of the Sm doped Mg nano ferrites samples under study were calculated from the observed values, and their thermal e. m. f are given below in table.1.

From the table, that we can observe the See beck coefficient gradually increases with the increasing of Sm, concentration. This may be attributed that, by increasing the Sm, concentration they occupy the B – Sites of and transfer the Fe^{+3} ions to the A-site, resulting in the decreasing of Fe^{+3} ions in the B- site [23]. This indicates the decrease of Fe^{+2} ions in B- site. See beck coefficient for cubic system is given by

$$S = \frac{\text{Total number of } Fe^{+3} \text{ ions in B-site}}{\text{Total number of } Fe^{+2} \text{ ions in B- sites}}$$

With the increasing of Sm, concentration, the number of Fe^{+2} ions in B-sites decreases and See beck coefficient increases in the Mg nano ferrites which can be justified from the above expansion of See beck coefficient.

From the above explanation, the reason with the increasing of Sm, concentration, a large number of charge carriers are produced. So that more energy is required to transfer the charge carriers between the different cations, therefore, large e m f is produced which enhances the thermo electric power. The figure shows that the variation of See beck coefficient with the temperature for all the prepared nano ferrite samples. It can be seen from figure, that the See beck coefficient positive and low at low temperature, as the temperature increases the See beck coefficient value also increases for all the prepared nano ferrite samples under investigation. This indicates that all the prepared nano ferrite samples behave like as p-type semi – conductors.

Table:3 Seebeck Coefficient and Curie temperature of the prepared Mg Sm_x Fe_{2-x} O₄ (where X = 0.000, 0.025, 0.050, 0.075,0.1)

| Composition | Curie Temperature (K) | See back 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 |

By increasing the temperature, the conduction mechanism in these nano ferrites was due to the electrons, and they behave like p- type semiconductors. This type of conductivity is attributed to the hopping of electrons between the Fe⁺² and Fe⁺³ ions at octahedral sites. The p- type of conducting mechanism is



By observing the Seebeck coefficient values of prepared nano ferrite samples, they increased values with increasing of temperature indicates that more p-type charge carriers were released. Further increasing in temperature, the Seebeck coefficient suddenly decreases at particular temperature and again gradually increases, this sudden change in Seebeck value was named as Transition temperature (T_c).

From the above figure. 9, the transition temperature of all prepared nano ferrites was measured and are tabulated in table 1. The ferrites after reaching the transition temperature becomes paramagnetic. Hence there is no probability for hopping of electrons between the A-sites and B-sites behind the T_c. Thus, Seebeck coefficient becomes constant after transition temperature [24]. The transition temperature of pure Mg ferrite was around 294°C (568 K). In the present work the thermo electric power measurements carried out from room temperature to 300°C (300 -568 K) using the differential method. It is clear that the Sm, Gd doping Mg nano

ferrites thermo electric power study, exhibiting a well-defined transition at the curie temperature like the magnetic properties, susceptibility, permeability and spontaneous magnetization. The value of Seebeck coefficient shows minimum value at T_c, indicates that the magnetic ordering as a marked effect as the thermo electric power of these nano ferrite samples under investigation.

From the present study considering the semi conducting behavior of investigated nano ferrites the charge carriers are considered to be localized at ions or at vacant sites.

The variation of transition temperature with doping of Sm, concentration increases the transition temperature also increases, This may be due to the increase in the concentration of Sm, the Fe⁺³ ions decreases, which reduces the overall number of Fe ions in B – sites which in term results in increasing of A-B interaction Fe⁺³ – o – Fe⁺³ [25].

3.6 Magnetic properties of Sm, doped Mg Ferrites:

The room temperature magnetization measurements were carried out by using Vibration Sample Magnetometer (V S M). To know the magnetic measurements, the prepared nano ferrite samples were made in the form of pellets, and subjected to calcination at 500 °C temperature for four hours at a heating rate of 4 °C /min.

Hysteresis loops obtained from V S M for all the composition of Sm, doped Mg ferrite were shown in figure which shows the dependance of magnetization (M) on the applied magnetic field (H) and anisotropic constant (K).

| Composition | M.W | Magnetic moment (μB) | Anisotropic constant K (erg/Oe) | saturation magnetization M_S (emu/gm) | Coercivity H_C (Oe) | Remanence Magnetization M_R (emu/gm) | squareness ratio $S = M_R/M_S$ |
|---|---------|-----------------------------------|---------------------------------|---|-----------------------|--|--------------------------------|
| Mg Fe_2O_4 | 199.911 | 0.00268 | 6.953 | 0.075 | 89 | 0.023 | 0.306 |
| Mg $\text{Sm}_{0.025}\text{Fe}_{1.975}\text{O}_4$ | 202.353 | 0.000434 | 1.187 | 0.012 | 95 | 0.030 | 2.5 |
| Mg $\text{Sm}_{0.075}\text{Fe}_{1.925}\text{O}_4$ | 207.079 | 0.00233 | 7.743 | 0.063 | 118 | 0.018 | 0.285 |

Various magnetic parameters are measured by observing hysteresis loops such as saturation magnetization M_S (Maximum value of the magnetization), remanence magnetization M_r – (Magnetization at zero field), Coercivity $-H_C$ (Magnetic field require to reduce the magnetization of that material to zero after the magnetization of the sample has been driven to saturation).

The calculated magnetic parameters (M_S , M_r and H_C) for synthesized nano ferrite samples under the applied magnetic field (H) are mentioned in a tabular form.

The measured values show a clear hysteresis loop behavior, from the hysteresis loop fig.7 we can observe that the pure Mg ferrite ($X=0.000$) with high squareness ratio was the hard ferrite and maximum Sm, Gd doped ferrite ($X = 0.075$) with least squareness ratio was the soft ferrite. Hence by substituting Sm, Gd in the Mg ferrite the system changes from hard ferrite to soft ferrite.

The shape and width of the M – H loop is affected by several factors including chemical composition, fabrication method, sintering temperature by time and also grain size etc[26][27].

From the calculated values it can be observed that in Mg nano ferrites the saturation (M_S) and remanent magnetization (M_r) values were observed to be decreases with increasing of Sm doping concentration.

Coercivity is the magnetic field strength required for overcoming anisotropy to flip the magnetic moment which is influenced by the doping rare earth material ions[28]. The coercivity value were in the range of 89 – 145 Oe.

The magnetic moment M and anisotropic constant K of the pure sample to Sm doping ferrite increases, and Gd doping sample it decreases. It was observed that the magnetic momentum μ_B and squareness ratio (S) values decreases with increasing Sm concentration in Mg nano ferrites.

From these results it is observed that by increasing the Sm concentration in the prepared Mg nano ferrites magnetic property of the samples are converted from hard magnetic to soft magnetic. Such magnetic materials can be used for the fabrication of soft magnets, used in transformers cores, motors, and electro magnets etc.

4. Declarations

Acknowledgements:

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 and J.S.Harinakshi, Vice Principal , Dr.B.R.Ambedkar College,,Baghlingampally, Hyderabad for their encouragement in the present Research work.

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Figures

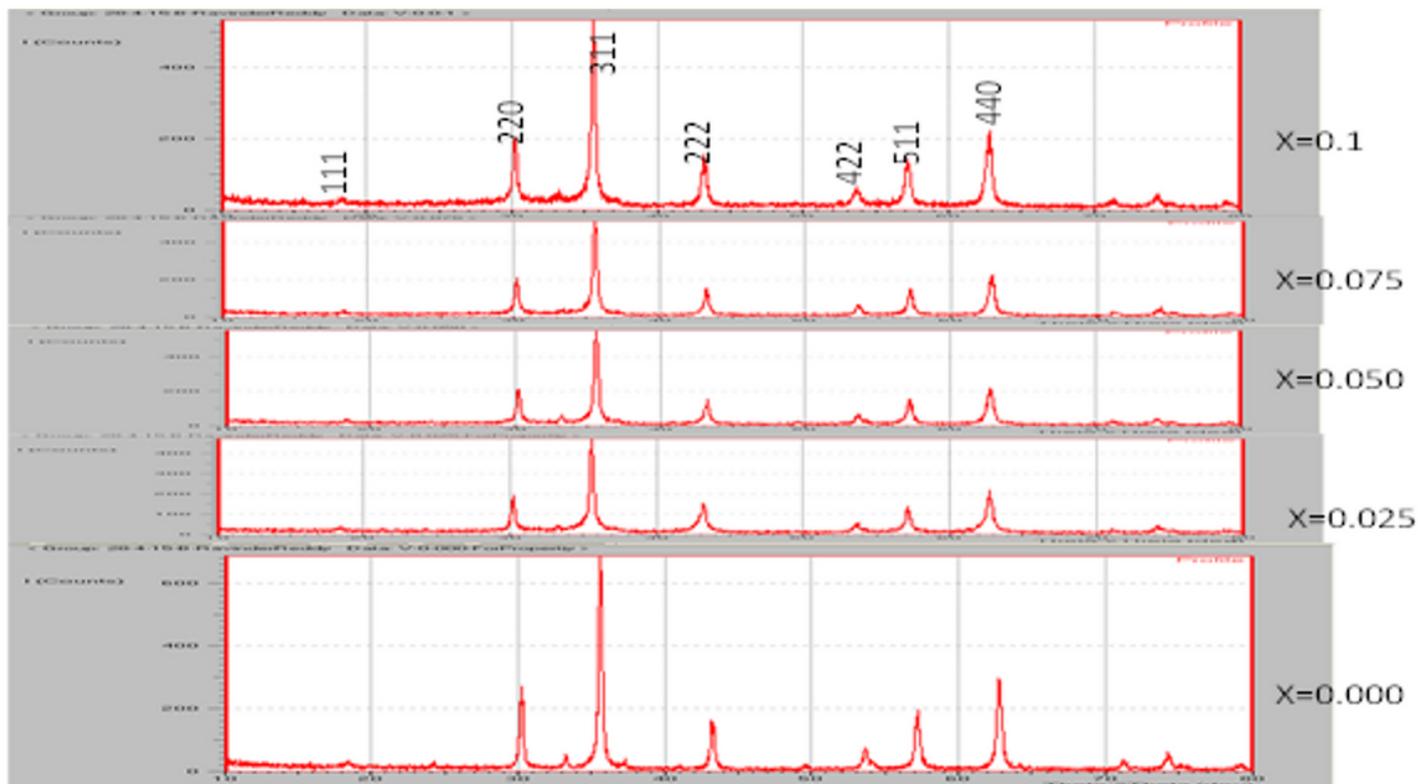


Figure 1

XRD pattern of Sm substituted Mg Nano Ferrite

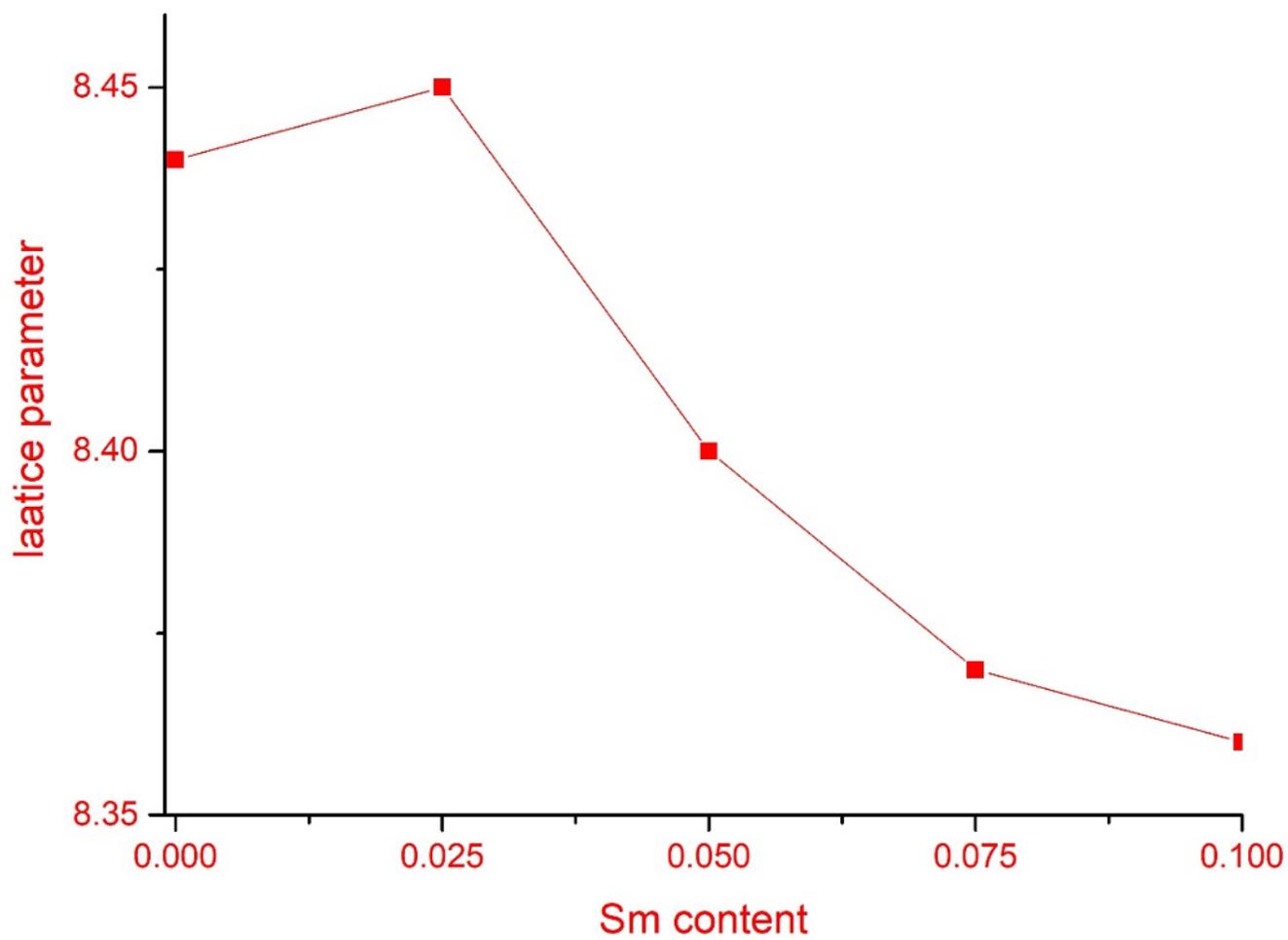


Figure 2

Lattice parameter variation vs Sm doped Mg ferrites.

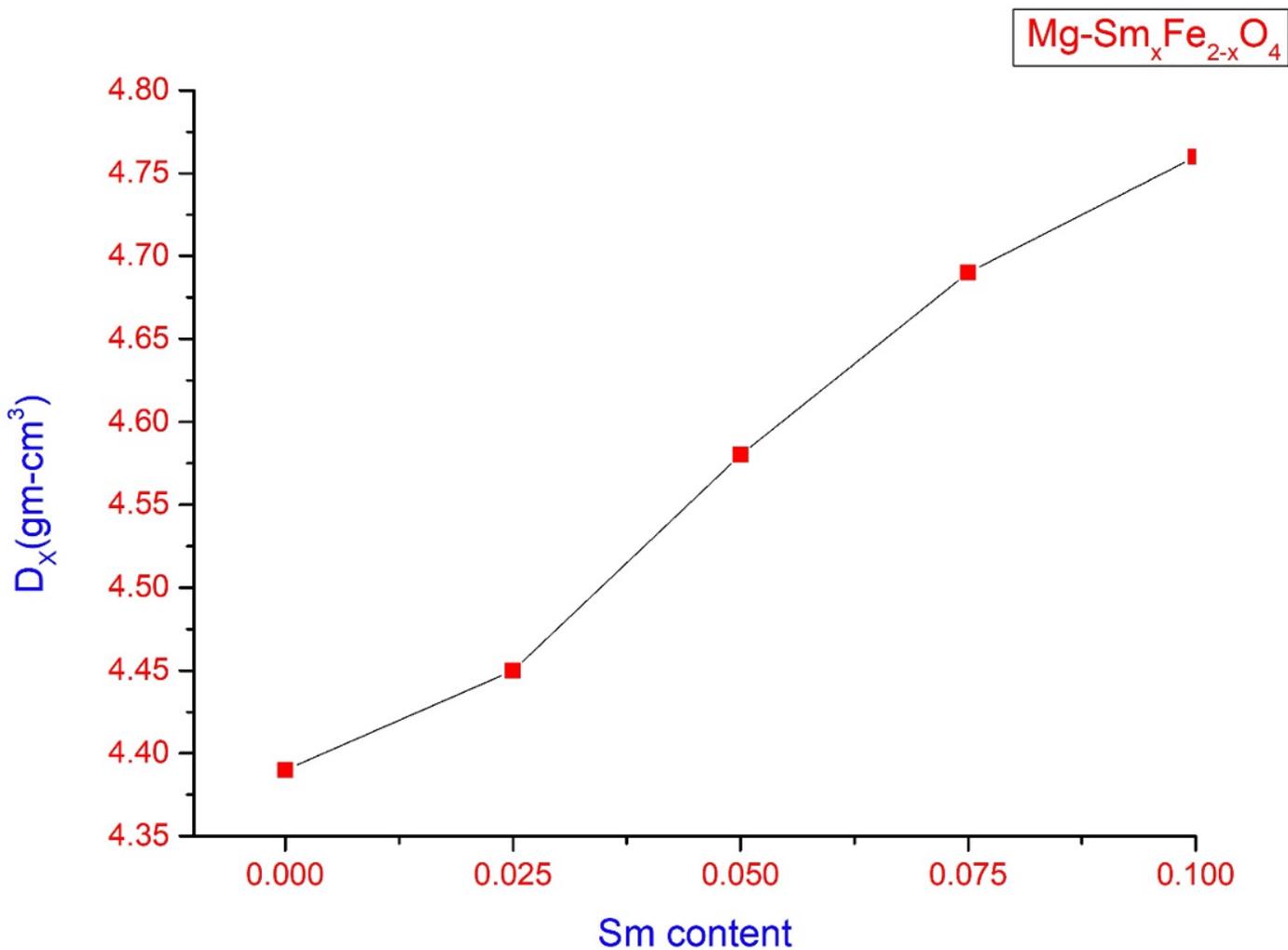


Figure 3

X-ray density variation for Sm doped Mg ferrites.

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Figure 4

X=0.000

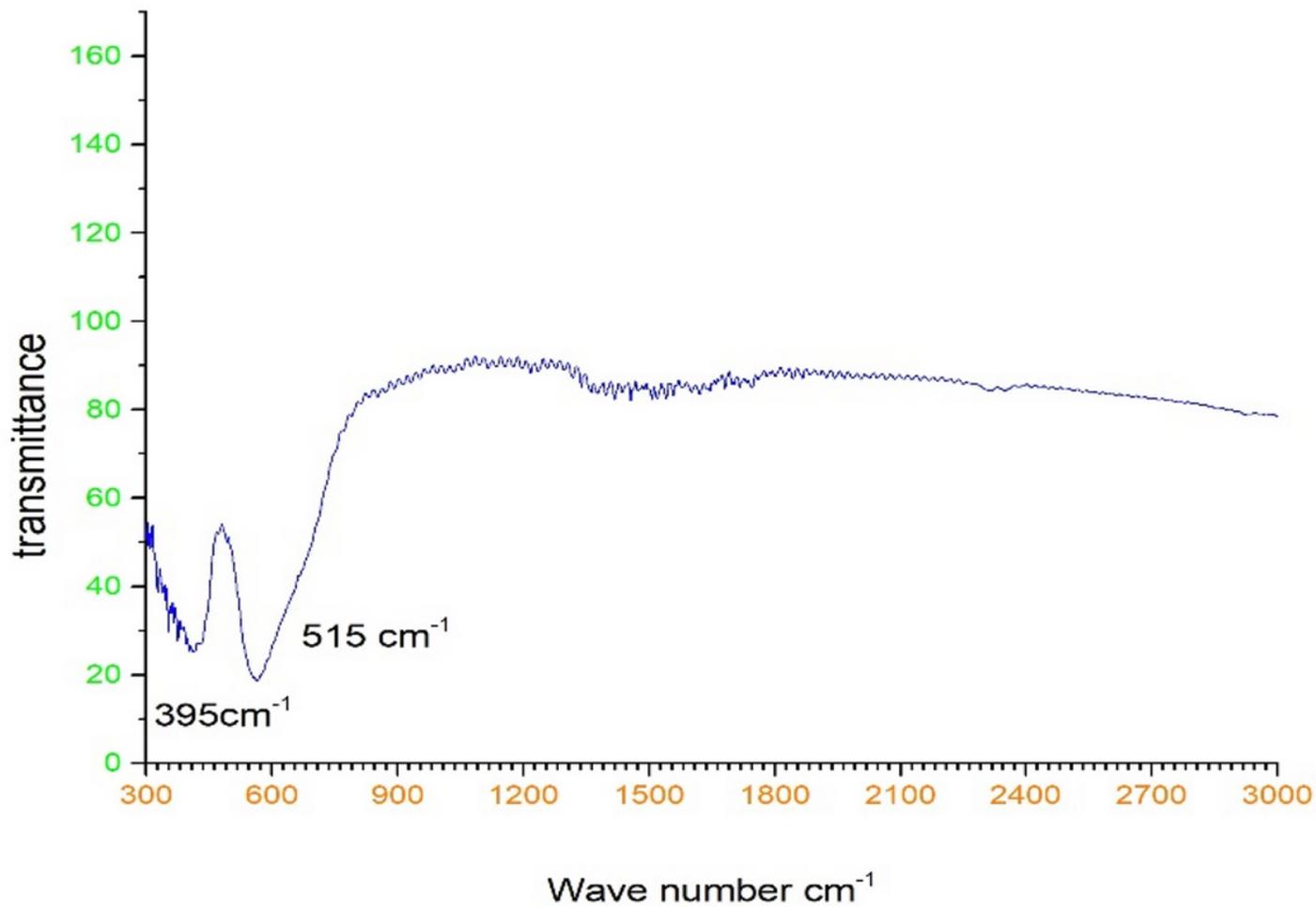


Figure 5

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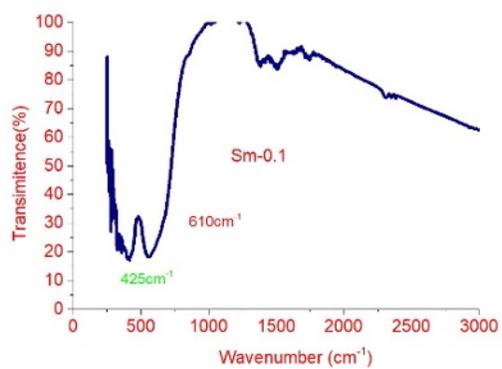
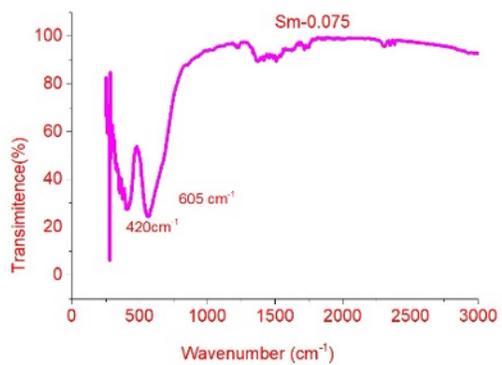
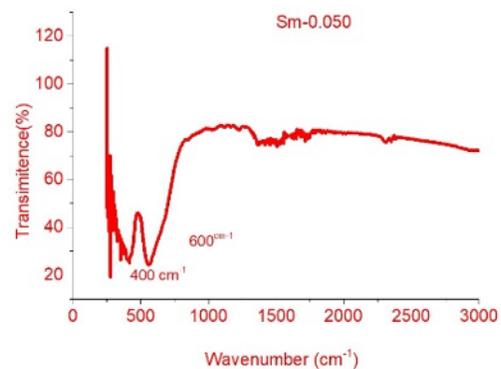
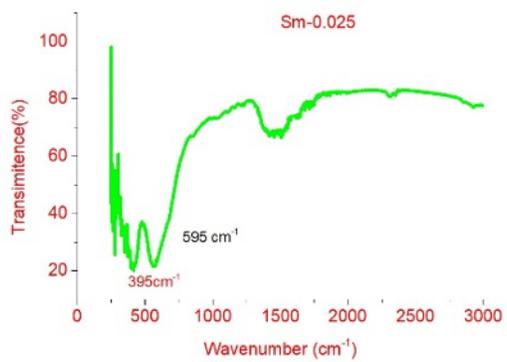


Figure 6

Optical studies of Mg-Sm nano-ferrites

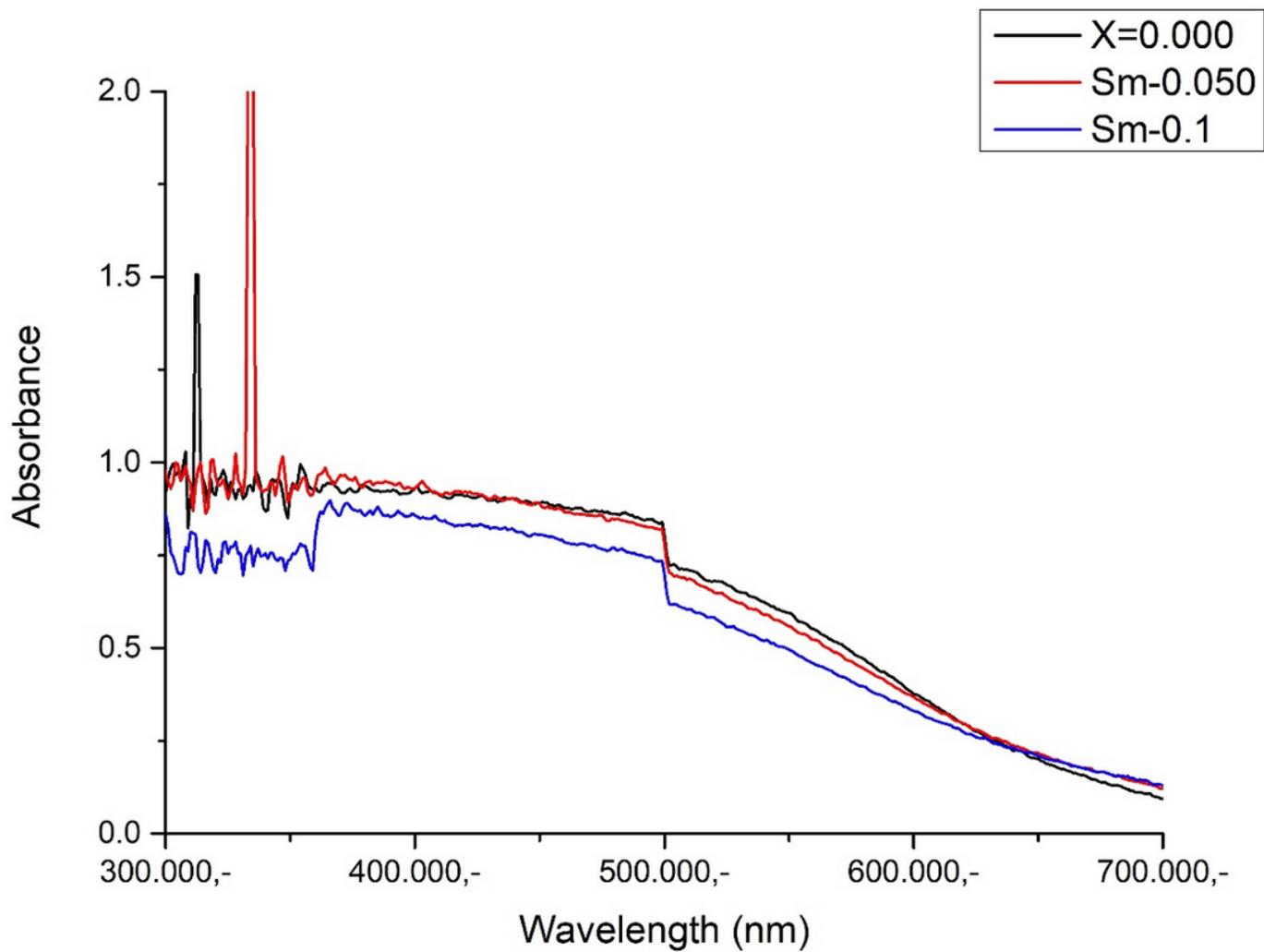


Figure 7

A caption was not provided with this version

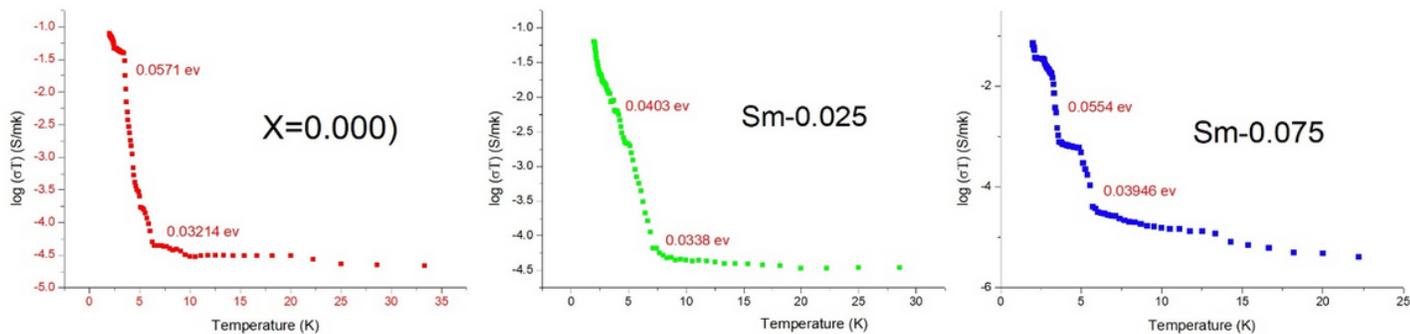


Figure 8

Electrical resistivity of Mg Sm x Fe_{2-x}O₄ nano ferrites.

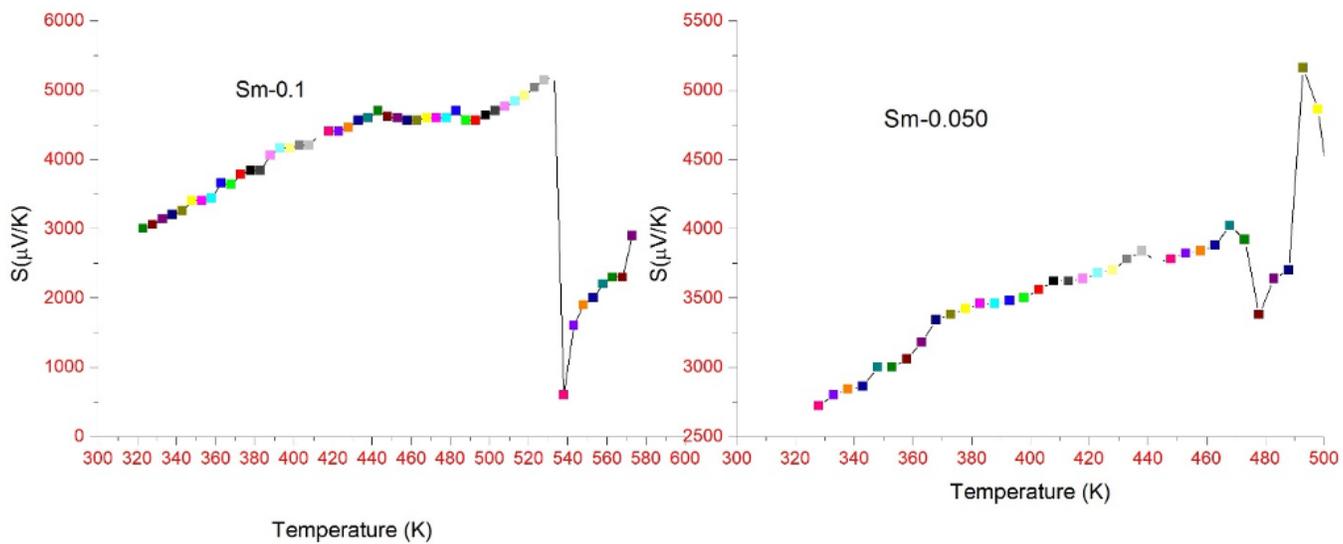
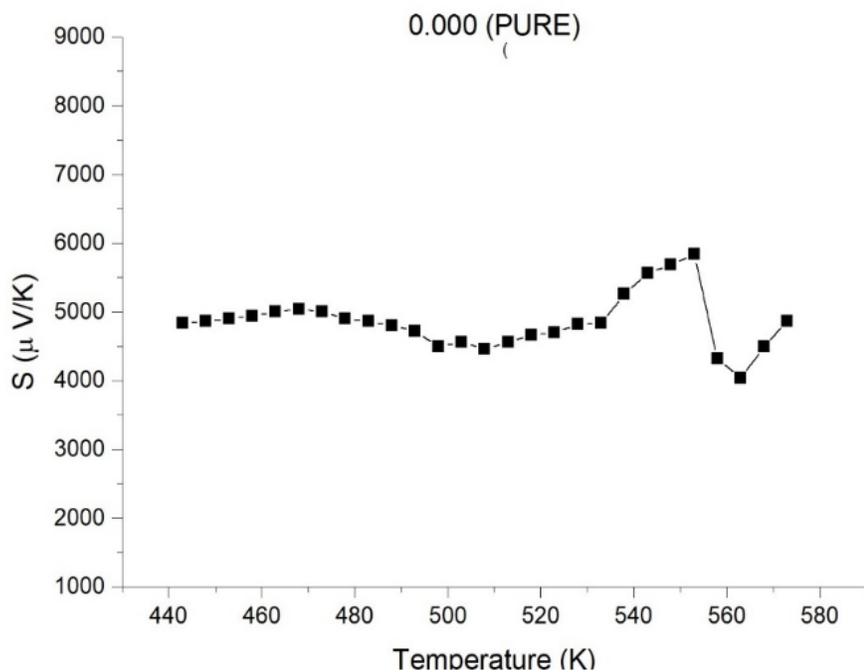


Figure 9

A caption was not provided with this version

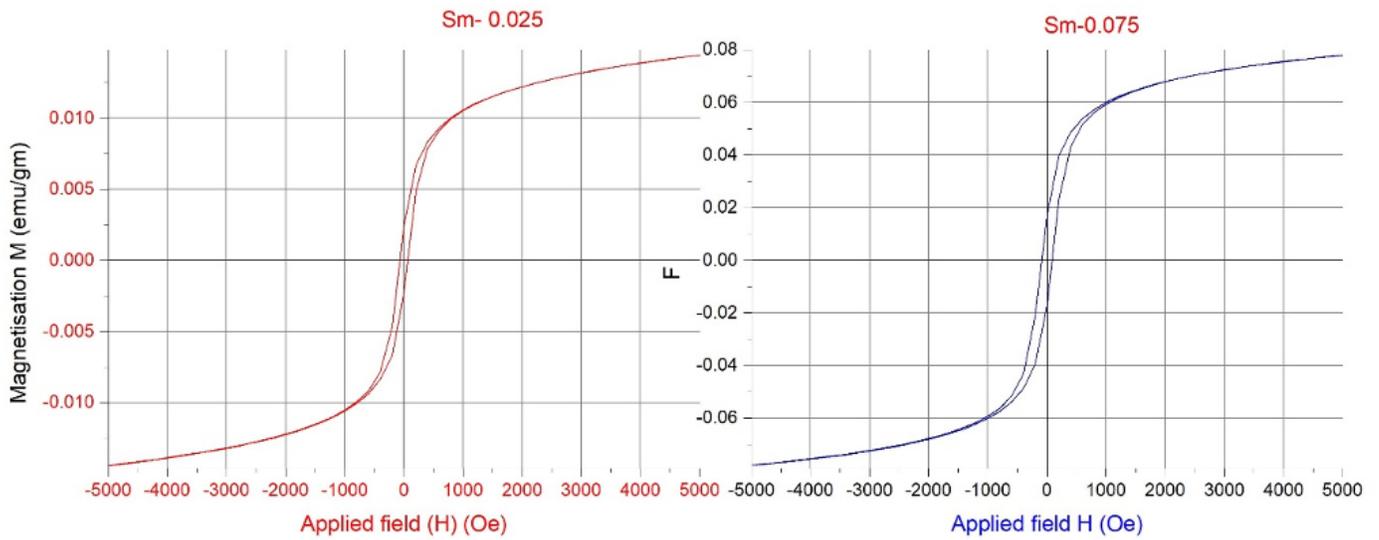
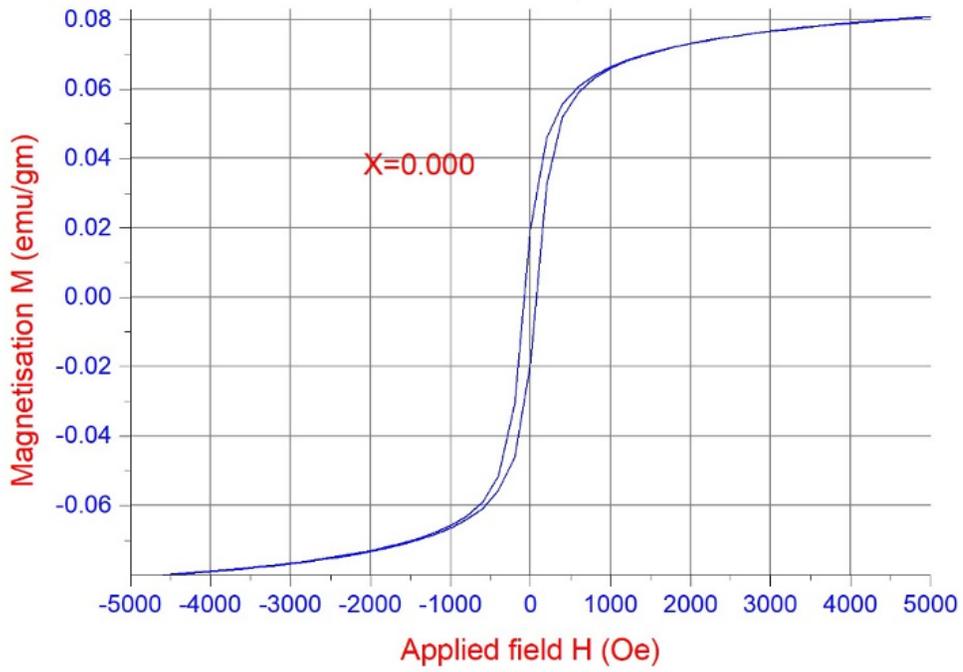


Figure 10

A caption was not provided with this version