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Influence of Sm^{3+} Substitution on the Structural, Magnetic, and Dielectric Properties of $\text{SrFe}_{12}\text{O}_{19}$ Hexaferrites

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Abstract: In this study, the sol-gel auto-combustion technique has been employed to synthesize M-type strontium hexagonal ferrite nanoparticles with the chemical composition $\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$, where $x = 0.0, 0.02, 0.04, 0.06, 0.08$ and 0.1 . The samples were sintered for 4 h at 1200°C for the reaction to occur and investigate the effects of Sm^{3+} substitution on the host material's structural, magnetic and dielectric properties. These properties were characterised by XRD, FESEM, FTIR, Magnetic, and impedance analysis. XRD patterns confirmed the formation of crystalline M-type hexagonal ferrites, identified as pure hexagonal with space group 167: R-3c and symmetry group P63/mmc. FESEM images revealed a nano-sized, speck-like morphology. FTIR analysis validated the synthesis of hexaferrite, showing bands in the 413 to $421 - 586$ to 593 cm^{-1} range attributed to the stretching vibrations of oxygen atoms and metal ions (M–O), specifically Fe–O bonds. The saturation magnetization decreases with increasing concentrations and reaches a low value at $x=0.10$. The concentration of Sm^{3+} increases the coercivity also increases. Dielectric analysis indicated frequency-dependent behaviour, with the sample exhibiting the highest dielectric constant among the synthesized samples and a low dielectric loss. The polarization and conduction mechanisms were interpreted using Koop's theory, the Maxwell-Wagner, and the Verwey hopping model.

Keywords: M-type hexaferrite; Auto-combustion technique; X-ray diffraction; Magnetic; Dielectric properties.

1. Introduction

The benefits of M-type hexagonal ferrite include its low cost, corrosion resistance, high saturation magnetization, high coercivity, and elevated resistivity [1-3]. This material is highly suitable for various applications, including high-frequency devices, magneto-optical systems, magnetic recording media, and microwave devices [4-6]. Due to its superior magnetic characteristics, strong stability, and affordability, M-type $\text{SrFe}_{12}\text{O}_{19}$ accounted for a significant share of ferrite production in 1963, and global annual production of M-type $\text{SrFe}_{12}\text{O}_{19}$ surpassed one million tons in 2020 [7]. Since ferrite permanent magnet materials are commonly used in motors, enhancing their performance has become a primary focus of research and development efforts [8, 9]. Strontium hexaferrite is frequently employed in various industrial applications. Furthermore, the economic viability of strontium hexaferrite, particularly its low cost per unit and widespread availability, adds to its appeal [10]. Various methods such as doping, heat treatment, ion substitution, and optimization of processing conditions have been utilized to improve strontium hexaferrite's electrical, dielectric, and magnetic properties [11]. These techniques provide excellent opportunities to carefully engineer site occupancy, thereby increasing the compound's net magnetization and fine-tuning the magnetic characteristics of the M-type hexaferrite compound [12]. Due to this, attempts are made to partially substitute Sr^{2+} or Fe^{3+} sites. For instance, replacing non-magnetic ions like Al^{3+} , Zn^{3+} , Ga^{3+} , and Cd^{3+} with magnetic ions like Co^{2+} and Cr^{3+} at Fe sites or partially replacing the Sr^{2+} site with RE^{3+} ions like La^{3+} , Nd^{3+} , Sm^{3+} , and Pr^{3+} , and replacing the $\text{Sr}^{2+}/\text{Fe}^{3+}$ pair with Pr-Zn, La-Cu, and La-Zn [13-18]. La-Co substituted M-type strontium ferrites were created by Long et al. using a low-temperature co-firing technique with a doping level of $x = 0.00\text{-}0.50$. Compared to conventional ferrites, this ferrite ($\text{SrFe}_{12}\text{O}_{19}$) has higher coercivity and remanence, making it highly preferred. These exceptional magnetic characteristics considerably aid the development of industry. To improve these magnetic characteristics, ion substitution is currently the focus of many studies. Fe^{3+} ions have five distinct lattice locations in the M-type ferrite crystal structure: 2a, 2b, 12k, 4f1, and 4f2. 2a, 2b, and 12k are the spin-up positions, and 4f1 and 4f2 are the spin-down positions [19]. Sr^{2+} and Fe^{3+} ions are routinely swapped out by researchers, either independently or in combination. Some researchers use La^{3+} , Eu^{3+} , and Pr^{3+} for Sr^{2+} ions [20-22]. Common options for the coupled $\text{Sr}^{2+}\text{-Fe}^{3+}$ ion substitution are $\text{La}^{3+}\text{-Co}^{2+}$, $\text{La}^{3+}\text{-Cu}^{2+}$, and $\text{Zn}^{2+}\text{-Ti}^{4+}$ [23]. There are other substitutes for Fe^{3+} ions, such as Ga^{3+} , Zn^{2+} , Al^{3+} , and Cu^{2+} [24]. The technique above has a remarkable impact, significantly enhancing the properties of ferrite, providing a platform for further research by other academics, and fostering global industrial progress [25]. By substituting ions, researchers have significantly increased M-type ferrite's saturation magnetization and coercivity [26]. Researchers have previously focused on

the magnetic characteristics of M-type ferrite doped with La^{3+} and Cu^{2+} ions [27]. Salma Aman et al., for instance, used the sol-gel technique to prepare samples $\text{Sr}_{1-x}\text{La}_x\text{Fe}_{12-x}\text{Cu}_x\text{O}_{19}$ [28]. The samples that they prepared have extremely low H_c . Instead of being concerned with the samples' decreased coercivity, they examine the dielectric constant and use it to lower the dielectric constant of samples in microwave devices. Our goal is to increase the sample's coercivity and remanence so that it can be used to produce sensors and permanent magnets. We hypothesize that the magnetic characteristics of the samples could be enhanced by rearranging some crystals within a specific concentration range [29]. Several techniques, including the sol-gel, the citrate precursor, the chemical coprecipitation, the hydrothermal, etc., are used to create M-type hexagonal ferrites [30-33]. The sol-gel auto-combustion process is used to synthesize hexagonal ferrites due to its simplicity, ease of use, and ability to adjust the grain size easily [34, 35].

In this study, the researchers synthesized Sm^{3+} substituted M-type hexagonal ferrites using the sol-gel auto-combustion approach. Analytic techniques such as XRD, FESEM, FTIR, magnetic and impedance analyzer were used to investigate the effects of various concentrations of the dopant ion ($\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$, where $x = 0.0, 0.02, 0.04, 0.06, 0.08$ and 0.1) on the crystal structure, microstructure, magnetic and dielectric characteristics of the host material under consideration. To the best of the researchers understanding, less work is available in doping the M-type hexagonal ferrites at such a concentration level and dopants involved. So this research adds scholarly knowledge, apple applications of such materials in the microwave, and many more aspects.

2. Experimental Methods

Analytically graded chemical raw materials were used to synthesize the intended materials using the sol-gel auto-combustion materials. Sm^{3+} -substituted M-type Sr nano-hexaferrite systems were prepared by weighing the nitrate precursor materials in a stoichiometric ratio. Nitrates of iron, samarium, and strontium—namely, $\text{Sr}(\text{NO}_3)_2$, $\text{Sm}(\text{NO}_3)_3$, and $\text{Fe}(\text{NO}_3)_3$ —were diluted with methanol. Citric acid was dissolved in 250 millilitres of double-distilled water, acting as a chelating agent. The precursor solution was continuously mixed on a computerized hotplate stirrer for 12 to 18 hours. The solution gradually formed a thick, dense gel during heating and stirring, eventually drying. The substance ignited and burned vigorously upon drying, leaving significant ash. This ash was ground using a mortar and pestle and then calcined at 900°C for 2 hours. Three-weight per cent of polyvinyl alcohol (PVA) was added as a binder to press the calcined powder into circular pellets, every 30

millimetres in diameter and 15 millimetres thick. The green pellets were then sintered in an open atmospheric furnace at 1200°C for 4 hours, with a heating rate of 5°C per minute.

In general, the structural and physical characteristics of the samples were studied using various analytical tools. For structural analysis, XRD data were collected over the 2θ range of 20° to 80° using a Rigaku Cu-K α diffractometer. Morphological and functional group analyses were conducted using a Carl Zeiss EVOMA 15, an Oxford FESEM, and a Shimadzu FT-IR-8900 FTIR spectrometer. Lakeshore-7407 vibrating sample magnetometer with the maximum available field of ± 17 kOe is used to measure the magnetic characteristics at room temperature. An impedance analyzer (Model E4990A) was employed for the dielectric study.

3. Results and discussions

3.1 XRD analysis

X-ray diffraction (XRD) analysis was utilized to investigate the structural and crystallographic characteristics of sintered $\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$, where $x = 0.0, 0.02, 0.04, 0.06, 0.08$ and 0.1 M-type hexaferrites. The XRD patterns of these synthesized samples are presented in Figure 1. The analysis was conducted on the prepared powder samples using Cu-K α radiation, a standard and reliable method for examining crystalline structures. This approach helps confirm the successful formation of the intended hexagonal ferrite phase [36].

The XRD results were meticulously compared with the reference peaks listed under the Joint Committee on Powder Diffraction (JCPD) card number 01-084-0757 [37]. This card serves as a standard reference for M-type hexaferrite structures, and the complete alignment of all observed peaks with the data in this card provides clear evidence that the synthesized hexaferrite possesses the desired single-phase hexagonal structure. Furthermore, the absence of additional or unexpected peaks in the diffraction patterns confirms that no secondary phases are present in the samples. This indicates that the synthesis process effectively produced pure M-type hexaferrite without contamination or structural disruption.

The X-ray diffraction data further verified that the basic crystallite arrangement of Sr-hexaferrite remains intact, even after the Sm^{3+} substitutions. The preserved crystallographic structure indicates that the doping does not disturb the intrinsic M-type hexagonal configuration. The structural stability of the hexaferrite phase is evidenced by the consistent arrangement of diffraction peaks, specifically the (107), (114), and (008) planes, which are characteristic of the P63/mmc space group with space group number 194 [38].

The XRD analysis conclusively demonstrates that the prepared $\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$, where $x = 0.0, 0.02, 0.04, 0.06, 0.08$ and 0.1 samples have a pure hexagonal crystal structure, exhibiting the M-type hexagonal shape from top to bottom. The patterns show no signs of

impurities, structural defects, or crystal deformations, confirming the high quality and purity of the synthesized hexaferrite materials [39].

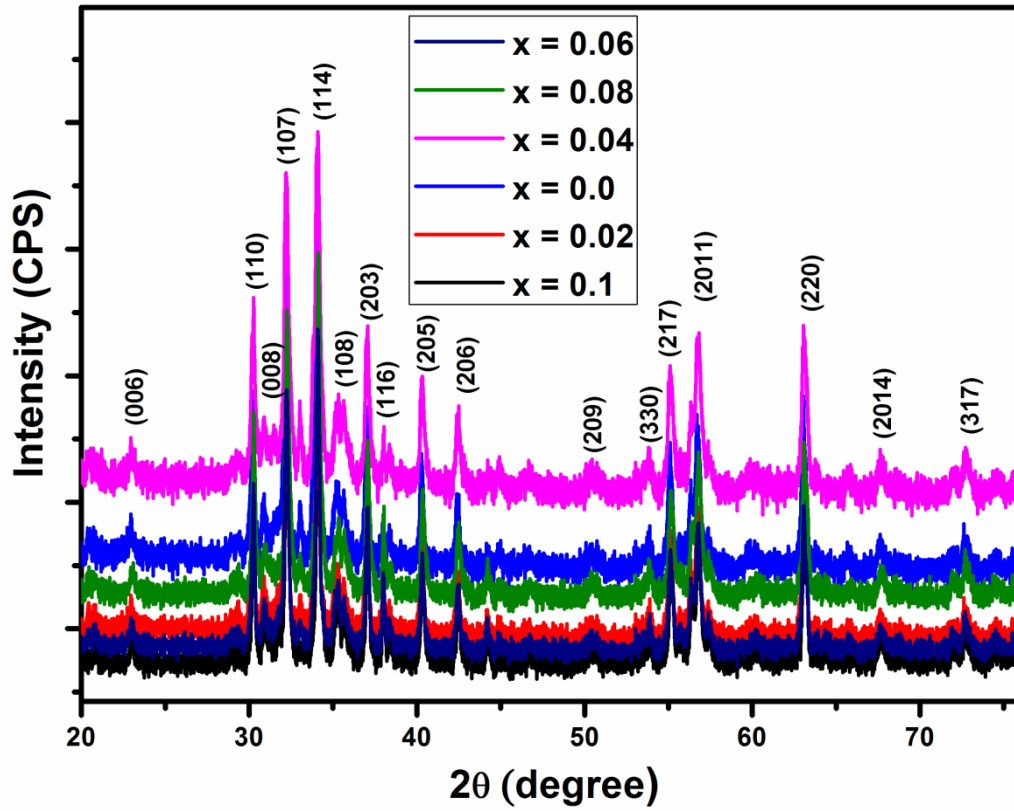


Figure 1: XRD for $\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$ nanoheaferrites

Table 1: Lattice parameters of $\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$ nanoheaferrites

Structural parameters	a (Å)	c (Å)	c/a	Crystallite size (nm)
0	5.867	23.13	3.94	41
0.02	5.845	23.05	3.95	37
0.04	5.813	22.95	3.95	34
0.06	5.786	22.86	3.95	31
0.08	5.745	22.79	3.97	28
0.1	5.716	22.71	3.97	26

The unicell calculator uses Bragg's equation to determine the lattice parameters a and c, which result in a slight variation [40]:

$$\frac{1}{d^2} = \frac{4}{3} \left(h^2 + l^2 + \frac{k^2}{a^2} \right) + \frac{l^2}{c^2} \quad (1)$$

Except for the Sm^{3+} dopant scenario, these characteristics decrease nearly as the dopants' concentrations rise, as supported by the c/a ratio. Consequently, these changes in the parameter also show that the co-substituted elements have been incorporated into the host strontium hexaferrite system. The Debye-Scherrer formula has been utilized to compute the sizes of the crystallites [41].

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (3)$$

Where λ is the wavelength of the X-rays, θ is the diffraction angle, and β is the full-width half maxima.

The samples' crystallite sizes fall in the 41 to 26 nm range. As Sm^{3+} concentration rises, crystallite size decreases. An XRD analysis confirms the decreasing tendency in crystallite size, which indicates that Sm^{3+} is forming a hexaferrite crystal structure. The small pragmatic crystalline size achieves an acceptable low signal-to-noise ratio [42]. The synthesized Sr-hexaferrite crystallite size is approved as suitable for recording media used in the current work.

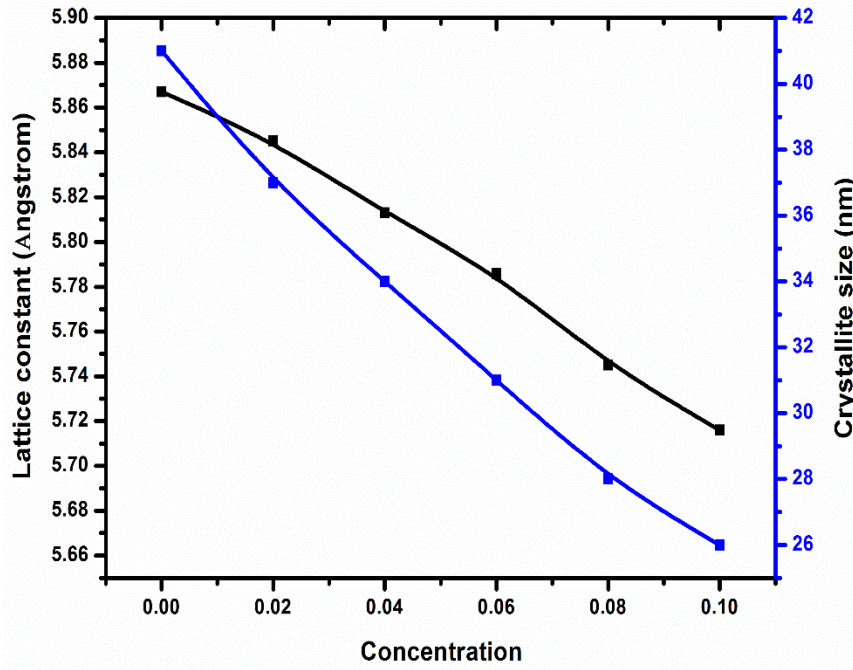


Figure 2: Variation of lattice parameters and crystallite size values of $\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$ nanohexaferrites

The synthesis ferrite lattice constants are shown in Figure 2. The average value of the length of the a -axis in a single M-type phase area is determined to be 5.867-5.716 Å, while the length of the c -axis steadily declines from 23.13 to 22.71 with increasing x . Unlike the previous study, this found that the length of the c -axis increased as the amount of Sm^{3+} substitution

increased, this study shows no such trend. Sm^{3+} (1.27 Å) and Sr^{2+} (1.32 Å) ionic radius differences can balance each other out nicely [43].

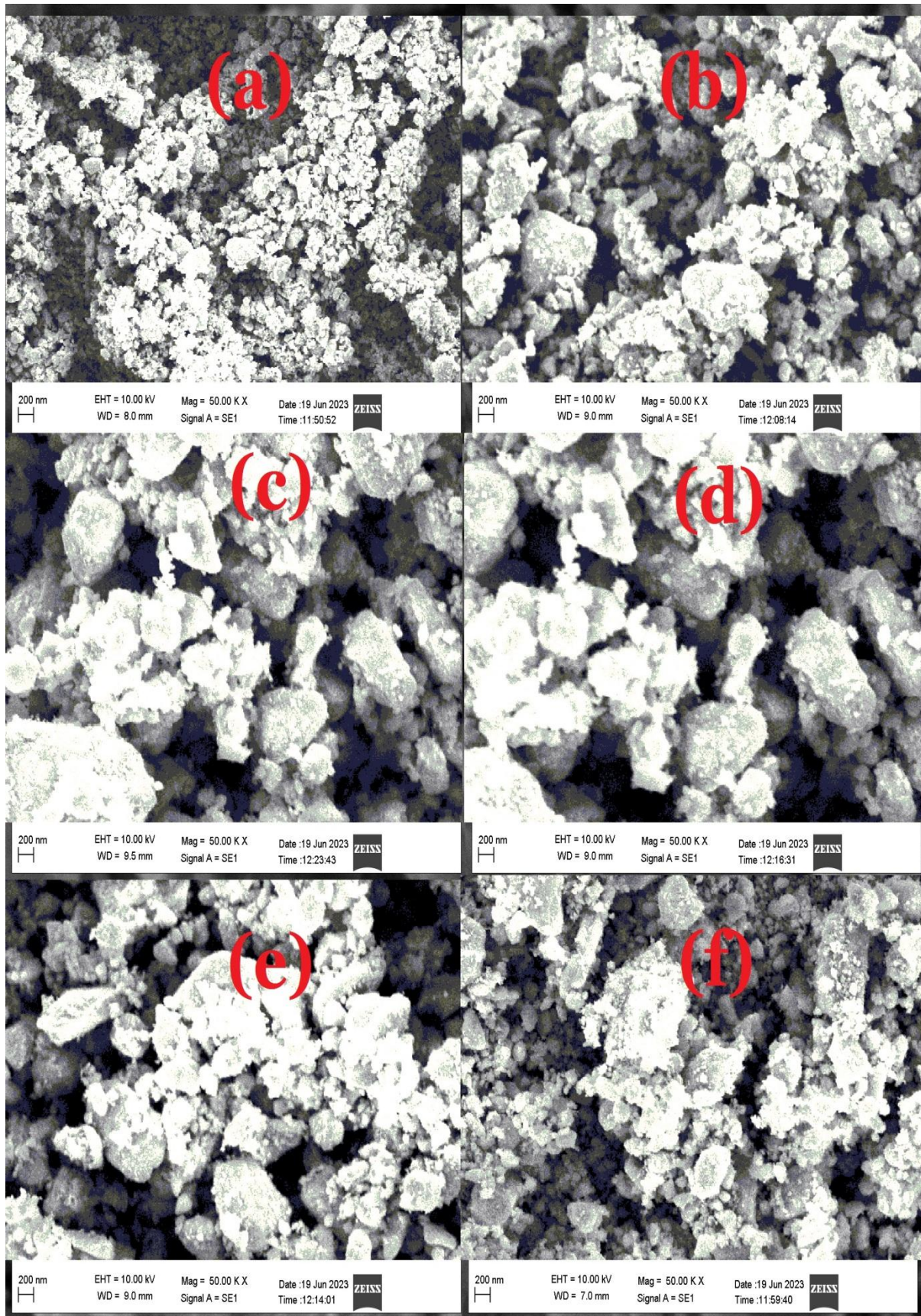


Figure 3: FE-SEM images for Sm^{3+} substituted $\text{SrFe}_{12}\text{O}_{19}$ nanohexaferrites

3.2 FESEM analysis

Figure 3 displays FESEM images of the hexagonal ferrites $\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$ nanohexaferrites. Interconnected particles form irregularly shaped aggregates, as shown in the SEM pictures. Ferrites with magnetic components typically have this dense, uniform structure [44]. This graphic illustrates how the FESEM images support the apparent morphology, which is the creation of nanostructured ferrite. As can be seen, samples have uniformly distributed morphologies and exhibit hexagonal platelet characteristics. And because magnetic particles are attracted to one another, this causes agglomeration and overlap between the particles. The average particle size of the samples, however, can be roughly calculated to be between 125 to 175 nm. It was discovered that there is a tendency for particle sizes to grow with an increase in the amount of dopants. The crystallite size estimated from the XRD study is smaller than the grain sizes obtained from FESEM. It exposes the clustering of crystallites into a single particle to create grains divided by internal crystallographic grain borders [45]. Another study team has noted a comparable disparity between the size of the crystallites (as determined by XRD analysis) and the size of the grains (as determined by FESEM analysis). It is a characteristic of oxide materials. It is an XRD technique constraint. However, because of their regular distribution and dense packing, the nanoparticles make it challenging to see the boundaries between neighbouring particles [46].

3.3 FTIR Study

The FT-IR spectra (Figure 4) of $\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$ nanohexaferrites were recorded after pelletizing the samples with KBr. Monitoring the ferrite formation process is feasible because the bond type, the symmetry of the lattice sites, and the elements in the crystal lattice determine the vibrational modes in FT-IR spectroscopy [47]. Such data reveals the different functional groups and molecular interactions. The samples' adsorption bands are located at similar positions, but their relative intensities differ. In the $\text{SrFe}_{12}\text{O}_{19}$ sample, M-type hexagonal strontium ferrites exhibit three distinctive absorption bands in the $400\text{--}800\text{ cm}^{-1}$ range. The band at 435 cm^{-1} is attributed to the Fe-O bending vibrations. The stretching vibrations of Fe-O and Sr-O occur at 545 cm^{-1} and 596 cm^{-1} , respectively. The substitution of Sm^{3+} ions for Sr^{2+} ions weakens the absorption bands of the synthesized hexaferrites in the $400\text{--}800\text{ cm}^{-1}$ range. The band at 596 cm^{-1} shifts to a higher wavenumber, while the bands at 435 cm^{-1} and 545 cm^{-1} shift to lower wavenumbers. This can be explained by the fact that the radius of the Sm^{3+} ion

(0.099 nm) is smaller than that of the Sr^{2+} ion (0.144 nm) [48]. In other words, the $\text{La}^{3+}\text{-O}^{2-}$ bond exhibits higher bond energy than the $\text{Sr}^{2+}\text{-O}^{2-}$ bond.

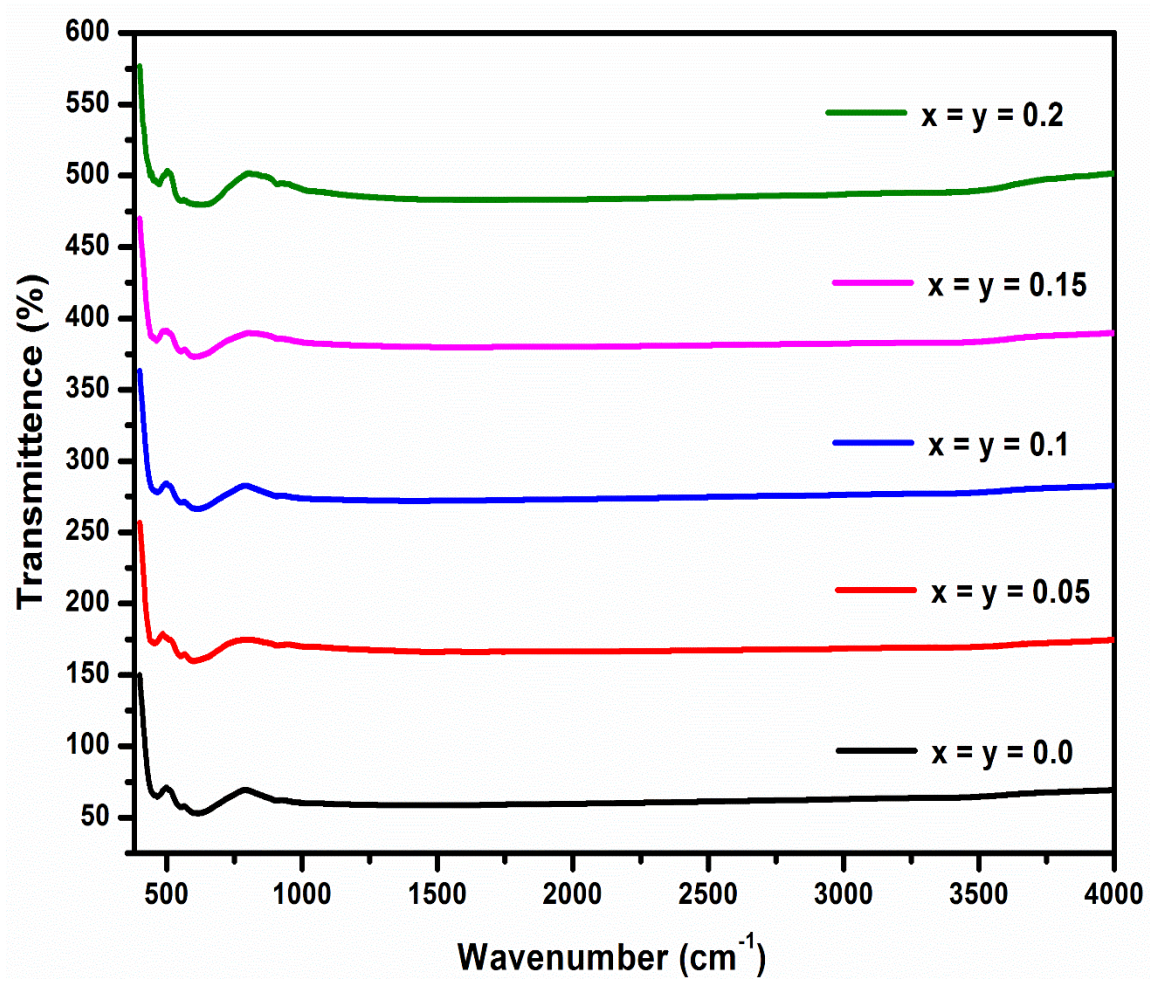


Figure 4: Infrared spectra of Sm^{3+} substituted $\text{SrFe}_{12}\text{O}_{19}$ nanoheaferrites

3.4 Magnetic properties

The magnetization curve at room temperature as a function of the applied magnetic field of 17 kOe is displayed in Figure 6. The hysteresis loops showed a constricted characteristic for an applied field close to zero. This result suggests that, in comparison to a typical ferromagnetic, the synthesized $\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$ samples with $x = 0.0, 0.02, 0.04, 0.06, 0.08$, and 0.1 showed a more steep relaxation of the magnetization value when the applied field direction was reversed. Figure 7 shows the saturation magnetization (M_s) and coercivity (H_c) values as a function of Sm^{3+} doping content, which were taken from the hysteresis curves. All samples have a low H_c value, which indicates soft magnetic materials [1] and is closely correlated with low magnetic anisotropy [38]. This value rises as Sm^{3+} doping increases. This rise is explained by a decrease in particle size, which is typically seen when iron ions are replaced with rare-earth ions [46], [47], and an increase in magnetocrystalline anisotropy [21],

[45] with Fe^{2+} anisotropic ions situated in the 2a sites. Additionally, the M_s value drops from 79.43 emu/g at $x=0.0$ to 64.57 emu/g at $x=0.10$ as the Sm^{3+} doping increases. As Sm^{3+} doping increased up to $x=0.10$, the M_s value rapidly decreased, as reported for other strontium M-type hexaferrite doped with rare earths [11], [16].

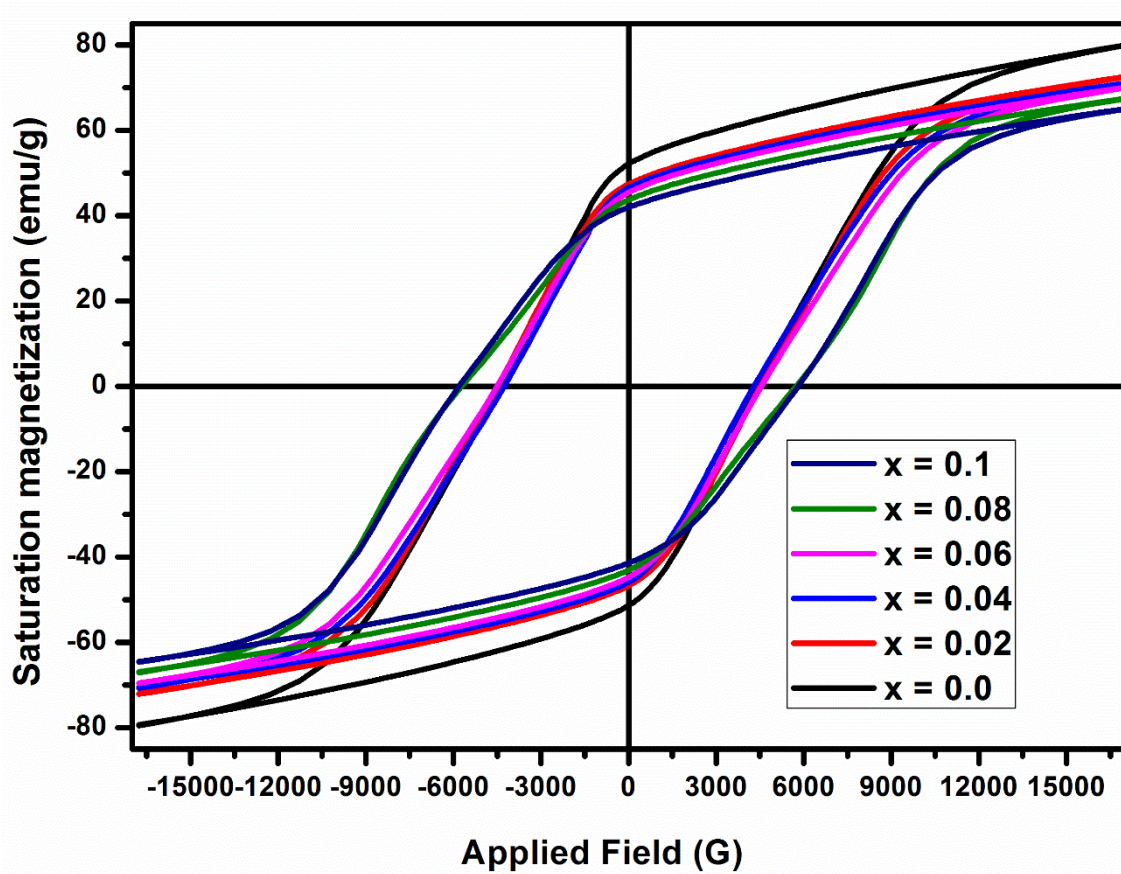


Figure 5: Hysteresis curve of Sm^{3+} substituted $\text{SrFe}_{12}\text{O}_{19}$ nano-hexaferrites

This is linked to changes in the valence state of iron from Fe^{3+} to Fe^{2+} , which lowers the concentration of Fe^{3+} ions in the 12k and 2a sites. Strong magnetic dipole coupling caused by substituting Sm^{3+} ions for Sr^{2+} ions prepared an extra demagnetizing force, which caused the M_s value for the Sm^{3+} -doped samples to drop sharply. However, due to the induction of distortion in the crystal lattice, the substitution of the rare-earth ion in the ferrite system also causes a reduction in the M_s . When $x > 0.10$, the M_s value may also rise due to cationic vacancies and disruption of the collinear ferromagnetic order [48], which is likely caused by an increase in the spurious phase. The presence of Fe_2O_3 partly maintains the strontium hexaferrite's electric neutrality. However, a greater magnetization in the lattice is caused by the superexchange interactions in $\text{Fe}^{3+}\text{-O-Fe}^{3+}$, which increases the hyperfine fields in the 12k and 2b sites [48].

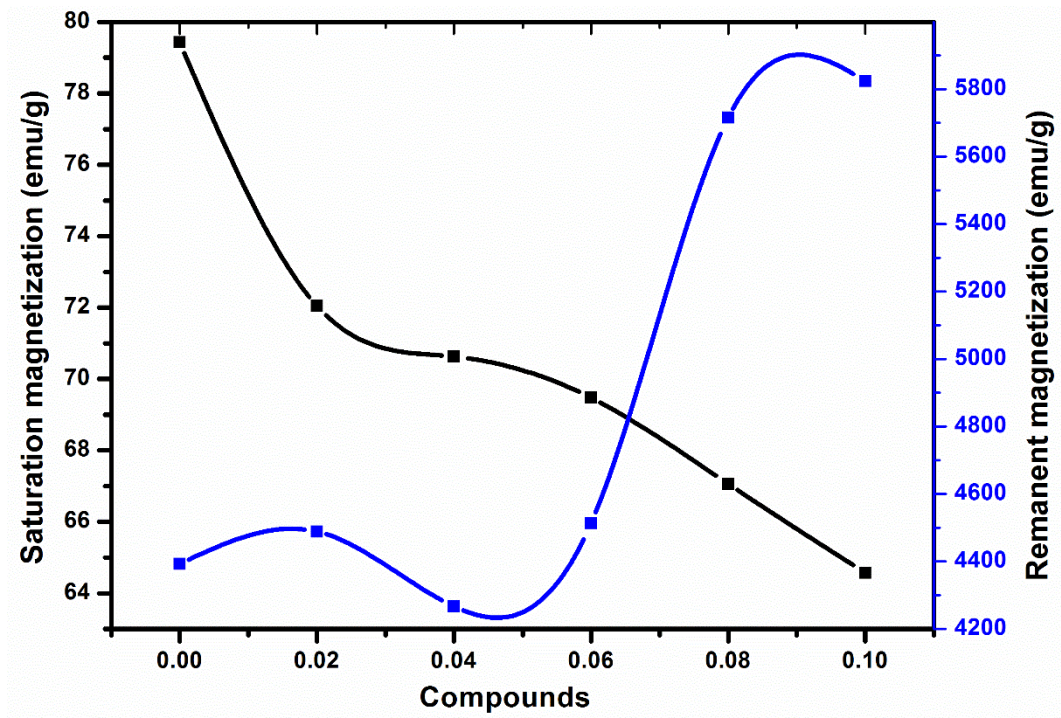


Figure 6: Coercivity (H_c) graph of Sm^{3+} substituted $\text{SrFe}_{12}\text{O}_{19}$ nanohexaferrites

Table 2: Saturation magnetization (M_s), remanent magnetization (M_r), and Coercivity (H_c) data of Sm^{3+} substituted $\text{SrFe}_{12}\text{O}_{19}$ nanohexaferrites

Magnetisation parameter	Compounds ($x = y$)					
	0.0	0.02	0.04	0.06	0.08	0.10
M_s (emu/g)	79.43	72.05	70.63	69.47	67.06	64.57
M_r (emu/g)	45.26	40.25	38.12	35.25	33.10	30.14
H_c (Oe)	4393	4489	4267	4514	5716	5824
M_r/M_s	0.56	0.55	0.53	0.51	0.49	0.47

3.4 Dielectric properties

Figure 5 shows the dielectric constant curves for $\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$ ($x = 0.0, 0.02, 0.04, 0.06, 0.08, \text{ and } 0.1$) nanohexaferrites. The dielectric constant decreases sharply and rapidly, and the curve exhibits a typical step-shoulder decline. The slope transitions into an exponential monotonic decline as the substitution level increases. A similar effect was observed in experiments with Ni-substituted Sr ferrites [49]. At low frequencies, the dielectric constant consistently decreases. Grain size analysis, corroborated by FESEM images, also shows a gradual decrease. The reduction in particle size may increase grains and grain boundaries, contributing to the decrease in the dielectric constant [50]. As the substitution level continues to rise, the dielectric constant of the sample gradually increases. The dielectric constant

decreases for all samples [52]. A high and low-resistance grain boundary is on one layer. While the ferrite exhibits the nature of grains at high frequencies, grain boundaries are visible in the material at low frequencies [53]. Once the free charge carriers have gathered at the grain boundaries, they move on to the grain boundaries and proceed through space charge polarization [54]. At low frequencies, the dielectric constant is more significant because the jump length of free charge carriers is less than half that of the applied alternating electric field's period of change [55]. High frequencies cause a change in the free charge carriers' direction of motion [56].

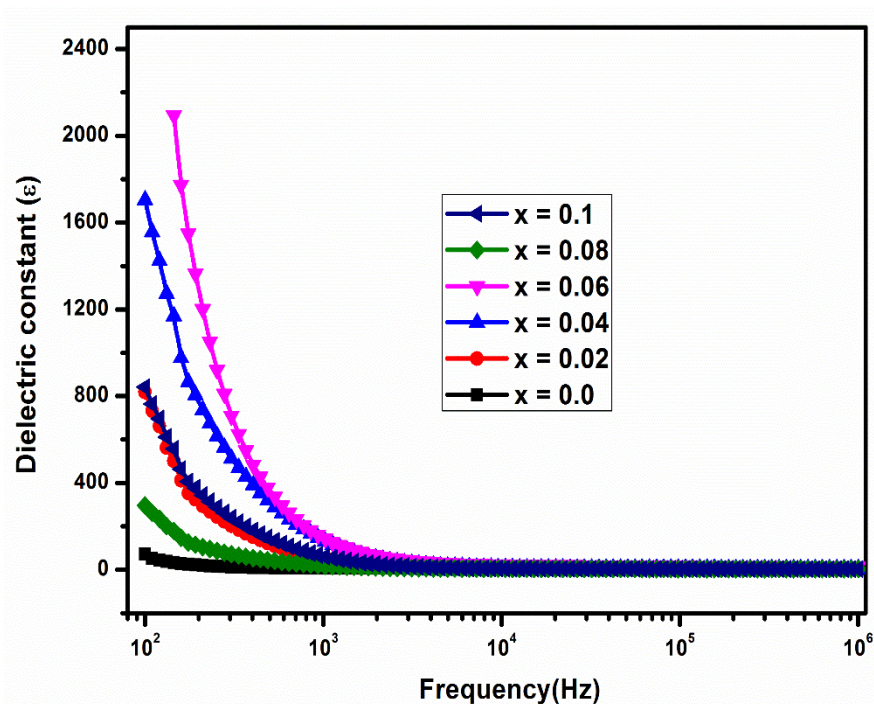


Figure 5: Dielectric constant vs frequency curve Sm^{3+} substituted $\text{SrFe}_{12}\text{O}_{19}$ nanohexaferrites

Electrical energy can be lost by dielectric relaxation, dielectric resonance, electrical conduction, non-linear loss in the dielectric, and tangent loss of material. Extrinsic loss and intrinsic loss are two different categories of dielectric loss that can be distinguished. Many factors, including crystal impurities, disordered crystals, and random orientation in crystalline materials, may cause extrinsic loss. On the other hand, the crystals' symmetry, the AC field's frequency, and the experiment's temperature might be to blame for the inherent loss [57]. The tangent losses follow the same trends as the dielectric constant, as seen in Figure 6. More precisely, its value falls with increasing frequency and is higher in the low-frequency area. Numerous variables, including the inhibition of domain wall movement, space charge polarization, and magnetization, may be responsible for decreased dielectric and tangent loss

with frequency. In high-frequency zones, these modifications reduce tangent and dielectric loss [58]. The measured loss of the doped material and its doped material as a function of the Sm^{3+} dopant concentration differ slightly. The low resistance of the electrode, hopping conduction, decreased oxygen vacancies, grain borders, and grain interface may all be responsible for the drop in tangent loss and dielectric that occurs with increased dopant content. Relatively less energy was needed to transition from Fe^{3+} to Fe^{2+} because the co-doped material has a lower resistance. In other words, co-doped samples with reduced resistivity have lower dielectric and tangent loss values [59].

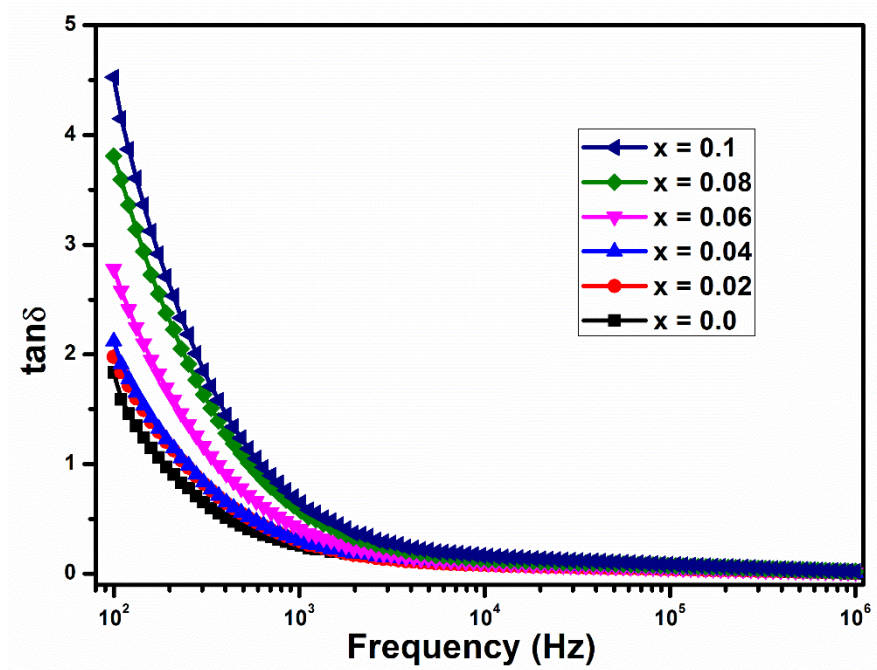


Figure 6: Variation of $\tan \delta$ as a function of the frequency of Sm^{3+} substituted $\text{SrFe}_{12}\text{O}_{19}$ nanohexaferrites

4. Conclusions

The composition $\text{Sr}_{1-x}\text{Sm}_x\text{Fe}_{12}\text{O}_{19}$ ($x = 0.0, 0.02, 0.04, 0.06, 0.08, \text{ and } 0.1$) nanohexaferrites was successfully synthesized as single-phase M-type hexaferrites using the sol-gel auto-combustion method. After annealing at 1200°C for 4 hours, M-type hexaferrite nanomaterials were obtained. The XRD patterns of all synthesized samples showed clear evidence of a unique hexagonal phase structure, which perfectly agreed with the JCPDS 01–078–0133 standard. The average crystallite size was 41 to 26 nm, which is small enough to provide a good signal-to-noise ratio for recording media. M–H data analysis revealed that as Sm doping increased, the saturation magnetization decreased, and the coercive field significantly decreased. The rise in magnetocrystalline anisotropy, the transformation of the

valence state from Fe^{3+} to Fe^{2+} , and the distortion of the lattice and cationic vacancies were all studied for this behaviour. As the applied field frequency and dopant concentration increased, the Maxwell, Wagner, and Koops models predicted that the dielectric constant, dielectric loss, and tangent loss would decrease while the AC conductivity would increase. Due to their improved dielectric properties, these synthesized materials are promising candidates for modern energy storage systems.

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Declarations

Conflict of interest

The authors declare they have no competing interests.

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