

Structural and magnetic properties of Gd^{3+} substituted Nanocrystalline Cobalt Ferrite

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Abstract

In this study, we have investigated the impact of Gd^{3+} substitution on the structural and magnetic properties of the spinel cobalt ferrite $CoFe_{2-x}Gd_xO_4$ ($x=0.05, 0.10, 0.15,$ and 0.20) which was synthesized by using the citrate precursor sol-gel technique and then control annealed at $750\text{ }^{\circ}C$. The gross-structural characteristics of the nanoparticles confirmed spinel crystal symmetry with the space group $Fd3m$. The Williamson-Hall (W-H) plot shows maximum value of lattice strain 2.71×10^{-3} for $CoFe_{1.85}Gd_{0.15}O_4$. The lattice parameter (a) shows a discernible increase as a result of induced strains. The lengths of the tetrahedral and octahedral M-O bonds are determined by the FTIR. The "Law of Approach to Saturation" quantifies that with an increase in Gd^{3+} concentration, saturation magnetization (M_s), Coercivity (H_c) and magneto-anisotropy (k_1) decreases from 73.16 to 49.89 emu/g, 1452 to 989 Gauss and 9.22 to 3.22×10^6 erg/cm³, respectively. We have investigated a relationship between induced lattice strain and magnetic response for $CoFe_{2-x}Gd_xO_4$ with $Gd=0.05$ to 0.20 . The very high coercivity of 1441 Gauss suggests employing such materials for the permanent magnet and high-density magnetic recording medium.

Keywords: Spinel ferrite; Coercivity, Lattice Strain, Anisotropy, Ferrimagnetic, Magnetization

Introduction

In the last few decades, Ferrimagnetic spinel ferrites have attracted significant research interest to the scientific community due to their use in high-density magnetic recording, resonance imaging, targeted drug delivery, biosensors, magneto-optical devices, radio frequency coils and ferrofluid technology [1–6]. Among spinel ferrites, cobalt ferrite (denoted as $CoFe_2O_4$) is the most studied magnetic oxide because of its high saturation magnetization, coercivity, and magneto anisotropy values (4×10^6 erg/cm³ [7]). The paramagnetic to ferrimagnetic transition temperature (Curie point) of $CoFe_2O_4$ is around 798 Kelvin [8]. The mixed spinel structure of cobalt ferrite crystallizes with the $Fd3m$ space group. $CoFe_2O_4$ has a mixed spinel structure denoted by the symbols $[Co_x^{2+}Fe_{1-x}^{3+}] [Co_{1-x}^{2+}Fe_{1+x}^{3+}]O_4$. Four crystallographic hard axes, designated $\langle 100 \rangle$ and $\langle 111 \rangle$, are aligned across the body diagonals of the $CoFe_2O_4$ lattice, whereas the six crystallographic easy axes are oriented toward the cube edges of the crystal [9]. The typical formula for spinel cobalt ferrite is AB_2O_4 , where A stands for a tetrahedral site and B for the octahedral site, both surrounded by 4 and 6 oxygen ions, respectively. A unit cell of AB_2O_4 has 32 octahedral sites and 64 tetrahedral sites, with oxygen ions occupying the remaining spots [10]. Metal cations occupy 8 tetrahedral and 16 octahedral sites, respectively. The substitution of Co^{2+} and Fe^{3+} sites in the $CoFe_2O_4$ lattice by any metal cation or the simultaneous substitution of Co^{2+} and Fe^{3+} sites has been the subject of numerous research studies [11]. Several researchers have highlighted the fact that the cationic distribution between the tetrahedral and octahedral sites of cobalt ferrite can fine-tune its structural and magnetic characteristics [12]. Large magneto-anisotropy energy is produced in the $CoFe_2O_4$ lattice by the presence of Co^{2+} ions on the B site of the spinel ferrite. Additionally, in such inverse spinel structures, octahedral sites are occupied by Co^{2+} and Fe^{3+} cations, with Co^{2+} ions having a stronger affinity for these sites [13]. Furthermore, a significant amount of magneto-crystalline anisotropic energy is contributed by strong L-S coupling in spinel crystal lattices due to remnant orbital magnetic moments [14]. For these reasons, the lattice strain generation in the $CoFe_2O_4$ lattice is the focus of interest for researchers. Efforts have also been undertaken to examine processes other than ionic substitution that cause lattice strains in samples. A group of researchers revealed in 2021 that the magnetic and magneto-elastic characteristics of $CoFe_2O_4$ can be successfully tuned through heat treatment. They improved saturation magnetization and decreased anisotropy by using thermal treatment to redistribute cations in the $CoFe_2O_4$ crystal lattice [15]. Few scholars have noticed that elements including the lattice parameter, crystallite size, bond angle, and bond lengths also affect the magnetic characteristics of $CoFe_2O_4$. According to them, saturation magnetization increased monotonically as crystallite

size grew. In a study, the change in the coercive field values on changing the crystallite size was observed [16]. Therefore, a thorough assessment of the literature reveals that research on improving the magnetic characteristics of CoFe_2O_4 has involved substituting its cations with elements like Cr, Mo, Zn, and Ti [17–20]. However, there are additional factors, such as the synthesis process used, the sintering temperature, etc., that can be useful in tuning lattice strain for improvement of CoFe_2O_4 physical properties. E. Hutamaningtyas *et al.*, 2016 concluded that the preparation method and annealing circumstances had an impact on the migration of cations between tetrahedral and octahedral sites in spinel ferrites [21]. B. R. Kumar *et al.*, (2002) have analyzed the thermo-electric properties of CoFe_2O_4 doped with Gd^{3+} [22]. K. K. Bharathia *et al.*, (2009) have explored the magneto-electric behavior of Gd^{3+} substituted CoFe_2O_4 [23]. A. Rana *et al.* (2011) studied the impact of Gd^{3+} doping on dielectric properties of CoFe_2O_4 [24]. These promising study findings inspired us to investigate the relationship between the lattice strains that emerged in cobalt ferrite lattices and the magnetic property refinements brought on by the Gd^{3+} doping.

We found limited literature works reported on the enhancement of the physical properties of cobalt ferrite with Gd dopants in its crystal lattice. Herein, we report the synthesis of pure cobalt ferrite by an economical sol-gel auto-combustion route and establish a relationship between induced lattice strain in the prepared samples, with improvement in structural and magnetic properties of CoFe_2O_4 to justify its utility for various technological applications, such as permanent magnet and high-density magnetic recording medium.

2. Experimental details

2.1. Materials and method

The chemical-based citrate precursor sol-gel method was used to prepare the pure phase cobalt ferrite, CoFe_2O_4 . High-purity chemicals, such as $\text{Co}(\text{NO}_3)_2$, $\text{Gd}(\text{NO}_3)_3$, and $\text{Fe}(\text{NO}_3)_3$ (Alfa Aesar, 99.9% pure), were employed in the synthesis process. Chelating agents such as citric acid were used during synthesis. The stoichiometric ratio of citric acid to metal cations was 1:3. First, individual chemical nitrates with a known molar ratio were weighed and dissolved into individual beakers with a small volume of deionized water. The citric acid solution-containing beaker was then filled with the aqueous solutions of $\text{Fe}(\text{NO}_3)_3$, $\text{Gd}(\text{NO}_3)_3$, and $\text{Co}(\text{NO}_3)_2$. The next step is to manually add NH_3 drop by drop to bring the pH of this final solution to neutral (7.0). Finally, a thick jelly-like product was produced by vigorously swirling this combined solution at a temperature of 120 °C for four to five hours. Then, after being held in a hot air oven chamber at 250 °C for two hours, this viscous jelly-like substance was allowed to grow in volume like a cake under the control of a self-propagating combustion reaction. After obtaining a substance that resembles burnt ash, it is further ground into fine powders using a mortar and pestle. The powdered samples were then placed in separate alumina crucibles and heated to 750 °C for two hours at a rate of 4 °C/min in a microprocessor-based annealing muffle furnace. These produced nanoparticles were then provided for various characterizations, which are mentioned below.

2.2. Characterizations

The X-ray diffraction data of $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ ($x=0.05, 0.10, 0.15$, and 0.20) were obtained at a wavelength ($\lambda=1.5406$ Å) using the Bruker D8 advance X-ray Diffractometer. The surface morphology of the $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ was examined with SEM (Carl Zeiss). The functional groups present in samples and bond lengths were found using Fourier Transform Infrared Spectroscopy (FTIR, Perkin Elmer). Especially for FTIR characterizations, samples were mixed with KBr and palletized using a hydraulic press. For XRD, SEM, and Hysteresis tests, samples were taken in a powdered state only. For SEM the powdered samples were gold coated with the sputtering technique before microscopy. Magnetic hysteresis (M-H) loops were recorded at 300 Kelvin using a Vibrating Sample Magnetometer (Lakeshore) capable of a magnetic field up to 3.2 Tesla.

3. Results and discussion

3.1. X-ray diffraction analysis

X-ray diffraction patterns depicted in Figure 1(a) shows pure phase synthesis of the samples having spinel crystal symmetry, classed into $Fd3m$ space group. The patterns of reflected X-ray intensity are consistent with other reports [11]. With the increasing Gd^{3+} content in the CoFe_2O_4 lattice, a steady increase in the broadening of the notable intensity peaks can be seen in Figure 1(a). It is evident that when Gd^{3+} ions increase, planes (220) and (311) monotonically get less intensified. The most intense planes (311) may be seen shifting to a lower Bragg's angle in Figure 1(a), which has been magnified. This could be a result of the lattice strain that was created in the samples with the Gd substitution at the Fe site. As shown in the diminishing intensity of the reflected planes (222), (422), (220), and (311) in the XRD patterns (Figure 1(a)), the crystallinity of samples improves monotonically with an increase in Gd^{3+} ions. Because thermal activations can break down the energy barrier between metastable states at relatively high temperatures, the percentage of crystallinity (spinel phase) increased with thermal activations [25]. Furthermore, the shifting of the prominent peak (311) in Figure 1(b) indicates that an increase in Gd^{3+} ions cause strain in the lattice parameter (a), which leads to a slight increase in the cell volume. However, the regularity in increment in lattice parameter is not observed. The FTIR spectra

analysis discussed below also supports this. In Table 1, these structural specifics are listed. Further, the affect on magnetic characteristics, due to the change in lattice parameters has been discussed as it alters the distance between magnetic ions, which disturbs the super exchange interactions between Fe^{3+} ions.

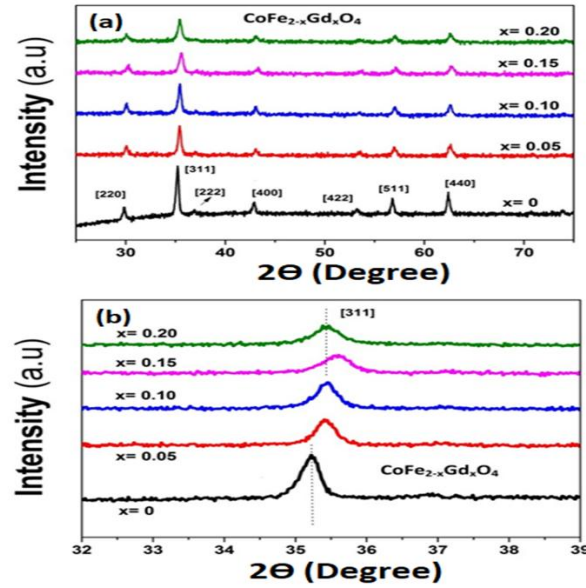


Figure 1. XRD pattern of (a) $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ ($x=0.05$ to 0.20) and (b) high-resolution XRD pattern in the range of $2\theta = 32$ to 39° (shift of highest intense plane (311)).

The Williamson-Hall (W-H) plot is analyzed to determine crystallite size and lattice strains in the CoFe_2O_4 lattice (Figure 2). The W-H equation is expressed as [26]

$$\beta \cos \theta = K\lambda/D + 4\epsilon \sin \theta \quad (1)$$

In the previous equation (1), θ stands for Bragg's diffraction angle and λ is the employed X-ray wavelength. D is the crystallite size, and FWHM (full-width half maximum) is the mid-width of the intensity of the diffraction. $4\sin \theta$ refers to the influence of strain, and the intercept ($K\lambda/D$) on the ordinate ($\cos \theta$) allows us to estimate the sizes of the crystallites in the produced samples. These values are computed using equation (1).

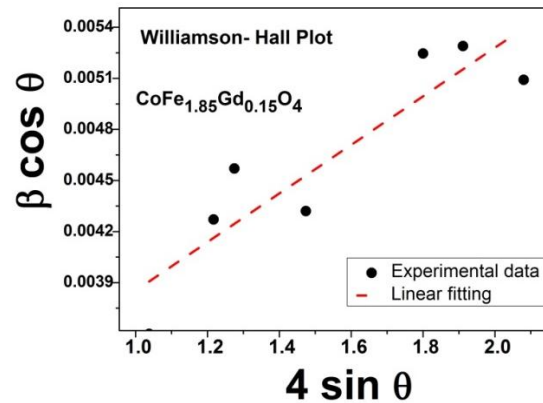


Figure 2. W-H plots to calculate crystallite size and lattice strain in $\text{CoFe}_{1.85}\text{Gd}_{0.15}\text{O}_4$.

Figure 2 displays the W-H plots for the $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ for ($x=0.15$). Data points from the XRD intensity patterns are fitted linearly. The value of crystallite size is determined by intercepts on the y-axis, and the quantity of lattice strain is determined by the angle of the straight line. Table 1 lists the dimensions of the crystallites as well as the lattice strains. To find out the lattice constants we use equation (2), in which significant planes [311], [220], [440], [400], and [511] were used.

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

Table 1. Structural details of $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ for ($x= 0.05$ to 0.20).

$\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$	<i>Crystallite size</i> (Williamson-Hall) <i>Nm</i> ($\pm$ Error)	<i>Strain</i> $\times 10^{-3}$ ($\pm$ Error)	<i>Crystal structure details</i>	
			<i>a=b=c(Å)</i>	<i>cell volume (Å³)</i>
X=0.0	57.8 (± 1)	1.58(± 0.00011)	8.361	584.486
X=0.05	49.3 (± 1)	1.87(± 0.00017)	8.366	585.535
X=0.10	44.8 (± 1)	2.16(± 0.00012)	8.372	586.796
X=0.15	36.4 (± 1)	2.71(± 0.00017)	8.379	588.269
X=0.20	42.7 (± 1)	2.05(± 0.00010)	8.377	587.848

The lattice strain rises monotonically as the Gd concentration up to $x=0.15$. For further increase in Gd composition, lattice strain starts decreasing, suggesting that optimized substitution at Fe site to be $x=0.15$. It's one of the reasons that can be attributed to the fact that the ionic radii of Gd ions are 0.938 Å and Fe ions have ionic radii of 0.645 Å only. All samples' XRD patterns were examined using the $Fd3m$ space group as a reference. Table 1 lists the improved structural parameters. It is evident that the lattice constant (a) and, therefore, the lattice volume have increased monotonically up to Gd=0.15 mole. These observations are in agreement with FTIR analysis. It was discovered that crystallite size decreased as Gd concentration rose. This might be a result of the agglomerations created by thermal activations, as shown by the SEM morphological analysis covered in section 3.2.

3.2 SEM analysis

In general, the crystallite size determined by the W-H plot and the particle size are different. SEM micrographs are therefore useful in supporting the crystallite size determined using W-H plots. As a result, Figure 3(a-d) depicts scanning electron micrographs of $\text{CoFe}_{1.85}\text{Gd}_{0.15}\text{O}_4$ samples at different magnifications. Nanoparticles are non-spherical in shape and get agglomerated with the Gd^{3+} substitution due to their strong surface energy and mutual magnetic interactions [27]. When compared to the XRD results, the particle size obtained by SEM is bigger. $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ for ($x= 0.05$ to 0.20) have average particle sizes of 97.04 nm. Particle size is determined using image-J software and analyzing concern histograms.

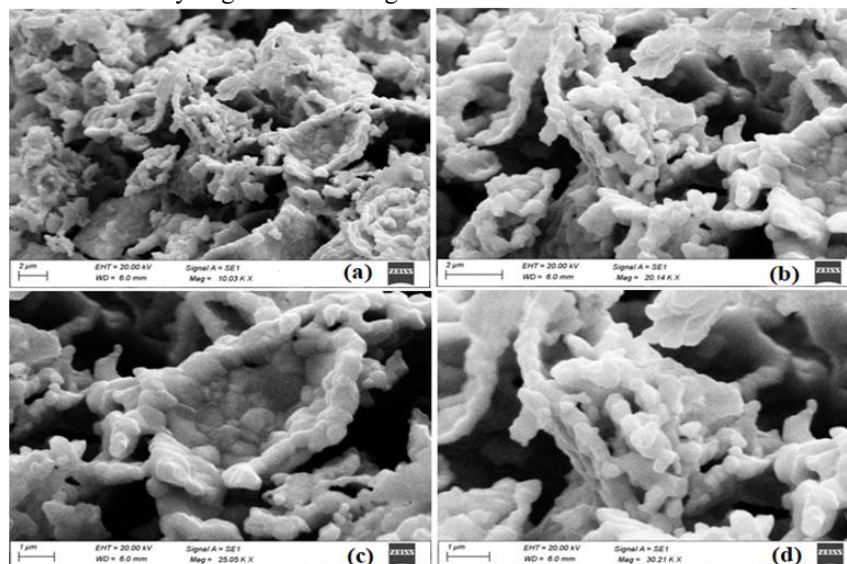


Figure 3(a-d). SEM micrograph of $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ for ($x= 0.15$) at different magnifications

3.3 FTIR analysis

FTIR absorption spectra for $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ ($x= 0.05$ to 0.20), captured in the wavenumber range (400 to 700 cm^{-1}), are shown in Figure 4. Two significant infrared peaks for spinel ferrites, such as CoFe_2O_4 , are observed at wavenumbers 439 cm^{-1} and 602 cm^{-1} , for bonds occurring at the octahedral sites and tetrahedral sites, respectively [28].

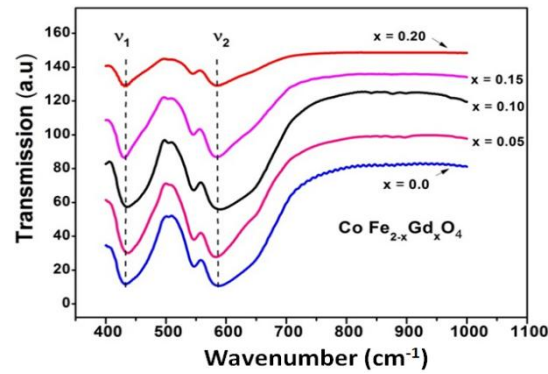


Figure 4. FTIR spectrum of $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ for $x = 0.05$ to 0.20 .

If our objective is to estimate octahedral bond length, the frequency range ($419 - 439 \text{ cm}^{-1}$) can be employed [29]. The length of the tetrahedral bonds between (Fe-O) and (Co-O) is determined using the frequency limit ($586\text{-}602 \text{ cm}^{-1}$) [30]. Tetrahedral sites have a higher vibrational frequency than octahedral sites because their bonds are longer relative to one another. The frequency (ν) of the vibration along with the force constant (K) is used to determine the lengths of the metal-oxygen (M-O) bonds. We initiate by computing the effective mass (μ), which is provided by [29].

$$\mu = \frac{(M_o \times M_{Fe})}{(M_o + M_{Fe})} \quad (3)$$

Where M_o = Mass of Oxygen atoms, and M_{Fe} = Mass of Fe atoms.
Next, the frequency of the harmonic oscillation is related to,

$$\nu = \frac{\sqrt{k/\mu}}{2\pi C} \quad (4)$$

Where C denotes the velocity of light. Moreover, force constant (K) is related to bond length (r) as

$$K = (17/Ar^3) \quad (5)$$

Table 2 provides a summary of all the relevant parameters that were determined using equations (3), (4), and (5). As the Gd concentration rises, we can see in Figure 4 that the absorption peaks gradually move towards a larger wavenumber value. This change may have a likely cause, which could be the stretching of the M-O bonds brought on by induced lattice strain in samples due to Gd substitution in cobalt lattice. As such bond lengths at the octahedral sites (12k, 2a, and 4f2) are observed to lengthen (Table 2).

Table 2. Force constants and bond length at octahedral and tetrahedral sites for CoFe_2O_4 .

Site	$\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$	Wavenumber (cm^{-1})	K (N/m)	Bond-length ($\text{\AA}$)
Octahedral	X=0.0	432.7	173.22	2.141
	X=0.05	433.3	170.11	2.154
	X=0.10	435.06	164.54	2.178
	X=0.15	437.4	159.44	2.201
	X=0.20	436.7	162.07	2.189
Tetrahedral	X=0.0	590.3	289.11	1.805
	X=0.05	592.4	282.45	1.819
	X=0.10	596.9	276.03	1.833
	X=0.15	598.5	259.58	1.871
	X=0.20	596.3	265.04	1.858

3.5 Magnetic Properties

Figure 5 shows the magnetic hysteresis (M-H) loops of $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ for $x = 0.05$ to 0.20 . The ferrimagnetic behavior and moderate coercivity values are displayed by hysteresis curves. Hysteresis curves seem to achieve saturation in the ± 1.5 Tesla applied magnetic field. Table 3 lists the computed values for coercivity (H_c), magnetization (M_s), retentivity (M_r), and anisotropy (K_1).

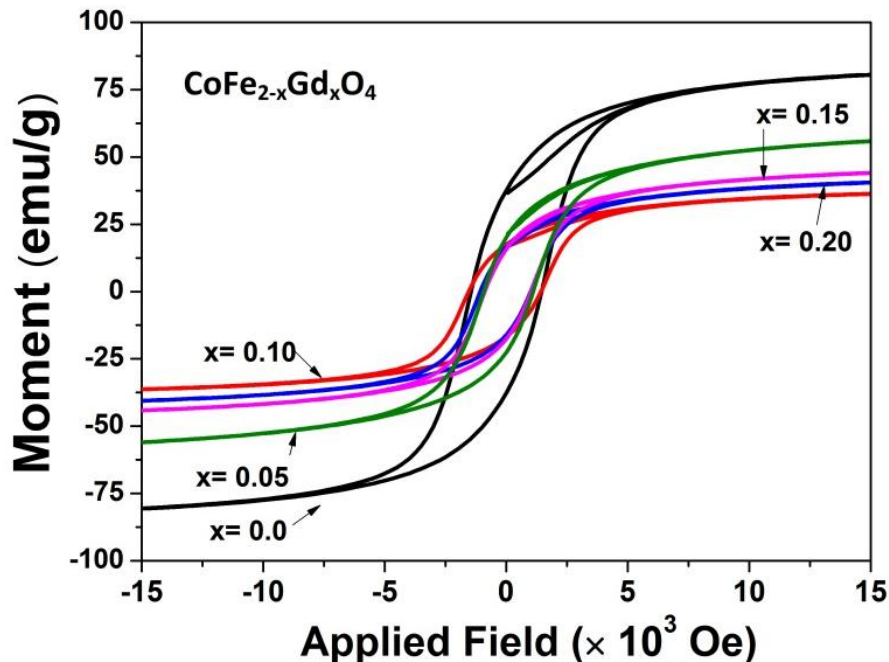


Figure 5. M-H loops at room temperature for $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ ($x = 0.05$ to 0.20).

Table 3. Magnetic parameters details of nanocrystalline CoFe_2O_4 samples.

$\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$	Magnetization (M_s) (emu/g)	Retentivity (M_r) (emu/g)	Squareness ratio	Coercivity H_c (Oe)	Anisotropic constant $K_1 \times 10^6$ (erg/cm ³)
$x = 0.0$	73.16	39.66	0.53	1452	9.22
$x = 0.05$	59.34	27.01	0.45	1441	8.06
$x = 0.10$	57.50	23.52	0.40	1124	5.63
$x = 0.15$	55.11	21.15	0.38	989	3.22
$x = 0.20$	49.89	22.08	0.44	992	4.61

With an increase in Gd^{3+} concentration, the remanence, and saturation magnetization, M_r and M_s both decrease monotonically. As magnetization and the hyperfine field have a linear connection, M_s decreases as the hyperfine field does [31]. The varying super-exchange interaction ($\text{Fe}^{3+}\text{-O}^{2-}\text{-Fe}^{3+}$) network is what has led to a decrease in the hyperfine field. Additionally, we are aware that most of the magnetic moments of the Fe^{2+} ions in CoFe_2O_4 are along the c-axis, which causes them to pair via super-exchange interactions with the O^{2-} ions [13]. The substituted CoFe_2O_4 material with a Gd concentration of $x=0.05$ has the highest coercivity values. It's interesting to observe that coercivity values decrease monotonically as Gd content rises to $x=0.15$. One reason for this observation is that, in the applied field of ± 1.5 Tesla, the samples achieved saturation with a maximum Gd content of 0.15 moles at the Fe^{3+} site. The second reason is the samples increased anisotropy because of the redistribution of cations brought on by Gd^{3+} doping, causing the coercive field to decrease monotonically [32]. Furthermore, coercivity, an extrinsic attribute, is also influenced by changes in the microstructure and particle size in nanomaterials.

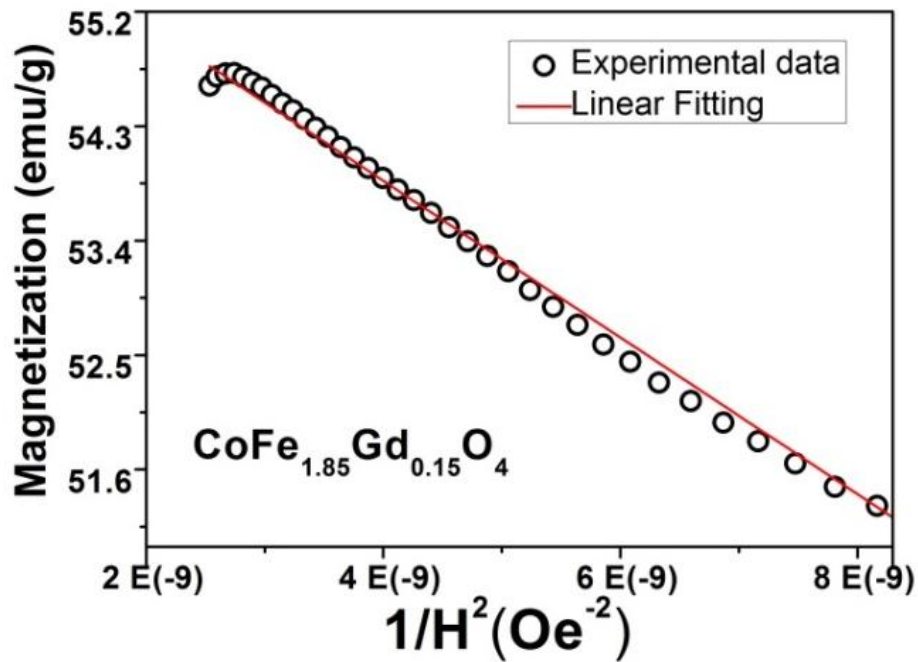


Figure 6. M-H loops at room temperature for CoFe_{2-x}Gd_xO₄ (x=0.15).

The "Law of Approach to Saturation" is used to calculate key magnetic properties including M_s , the anisotropy constant (K_1), and anisotropy fields (B). The approach law is provided by [33]

$$M = M_s \left[1 - \left(\frac{A}{H} \right) - \left(\frac{B}{H^2} \right) \right] + \kappa H \quad (6)$$

Here (M_s) represents magnetization, A is a material constant, and B is the magneto-crystalline anisotropy field. Ignoring insignificant terms equation (5) reduces as

$$M = M_s \left[1 - \left(\frac{B}{H^2} \right) \right] \quad (7)$$

The anisotropy is expressed as [34]

$$B = (H_a^2)/15 \quad (8)$$

The magneto-anisotropy is determined using the relation

$$H_a = (2K_1)/M_s \quad (9)$$

The value of magnetization (M_s) is determined by the intercept on the y-axis of the Moment versus the ($1/H^2$) plot of the experimental data that were linearly fitted (Figure 6). The magneto-anisotropy field's value (B_1) is immediately quantified by the slope of this linearly fitted curve. Equations (8) and (9) are then solved to determine the magneto-crystalline anisotropy (K_1) value after obtaining the M_s and B_1 values. Table 3 lists all the relevant magnetic parameters. In conclusion, substituted CoFe₂O₄ with Gd=0.05 has the highest saturation magnetization and magneto-crystalline anisotropy constant. This observation is explained by the perturbed Fe-O-Fe super-exchange interaction in all relevant crystal directions due to Gd³⁺ substitution [30]. The octahedral sites (12k, 2a, 4f2) bond length's increment is discussed in FTIR results, earlier in this article. The decrease in saturation magnetization is due to the decreased magnetic moment since the magnetic moment bears a linear relationship with the magnetization [35]. Further, M_s ranges from 49.89 to 73.16 emu/g, whereas K_1 is ranging from 3.22×10^6 to 9.22×10^6 erg/cm³. The observed decrease in anisotropy constants may be due to increased lattice strain. The coercive field is higher for CoFe₂O₄ with Gd=0.05, however, it is contradictory with the increase in anisotropic constant. The coercivity of these nanomaterials, which is of the order of 1441 Gauss, suggests that they might be useful in applications involving perpendicular recording medium [36]. Except for sample Gd=0.15, all samples had the Squareness ratio, which is calculated as (M_r / M_s) $\approx$ 0.5 (Table 3). This implies that CoFe₂O₄ would likewise be chosen for use as a permanent magnet and high-density magnetic recording medium.

4. Conclusions

In summary, a low-cost citrate precursor sol-gel method was effectively used to synthesize the cobalt ferrite CoFe_{2-x}Gd_xO₄ for x= 0.05 to 0.20. It was then controlled and annealed at 750 °C for two hours at a pace of 4 °C/min. The XRD patterns reveal the pure-phase, spinel crystal symmetry with the space group Fd3m of all the samples. The Gd doping effect causes an increase in the lattice parameters (a) and lattice volume. With a rise in

Gd concentration in CoFe_2O_4 nanomaterials, crystallite size decreased but lattice strain increased. Stretch in the metal-oxygen bonding caused by induced lattice strain is justified by the FTIR spectra. The substituted $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ has the maximum saturation magnetization at $x=0.05$, with an order of 59.34 emu/g. Also, the substituted $\text{CoFe}_{2-x}\text{Gd}_x\text{O}_4$ has a maximum coercivity value of 1441 Gauss for $x=0.05$. It has been noted that this sample has the highest anisotropy field values. One of the causes suggested is enhanced exchange interactions between Fe ions with thermal activations. The improved magneto-anisotropy values of CoFe_2O_4 point to its potential multifunctional uses. The current work provides a window into how lattice strain brought on by regulated Gd doping might improve the structural and magnetic properties of nanocrystalline cobalt ferrite.

Data availability statement

All data that support the findings of this study are included within the article.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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