

Evolution of Structural, Dielectric and Magnetic Properties of Cobalt Ferrite Nanoparticles Produced by Sol-Gel Technique During Sintering Treatment

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Abstract

This study investigates the effect of sintering temperature on the structural, dielectric and magnetic properties of cobalt ferrite nanoparticles (CoFe₂O₄ NPs) synthesized by the sol-gel method. The samples were sintered at 400°C–1000°C for 2 h and characterized using XRD, FTIR, SEM, AFM, dielectric and VSM measurements. XRD confirmed the formation of a stable cubic spinel structure, with crystallite size increasing from 10.71 to 16.09 nm and porosity decreasing with increasing sintering temperature. SEM and AFM showed progressive grain growth and improved grain connectivity, with the sample at 800°C exhibiting the most uniform morphology before excessive grain growth occurred at 1000°C. The dielectric response showed decreasing real and imaginary permittivity with increasing frequency, while the sample at 800°C exhibited the lowest dielectric loss ($\tan \delta \approx 2$ at 1 kHz). Magnetic measurements revealed ferrimagnetic behavior, with saturation magnetization increasing from 27.5 emu/g at 400°C to 68 emu/g at 1000°C, whereas coercivity reached a maximum of 715 Oe at 800°C. Overall, sintering at 800°C provided the best balance between microstructural uniformity, low dielectric loss and magnetic performance, making it a strong candidate for further evaluation in high-frequency and multifunctional magnetic applications.

Keywords: Cobalt ferrite nanoparticles, sol-gel technique, sintering temperature, structural properties, dielectric properties, magnetic properties.

1. Introduction

Cobalt ferrite (CoFe₂O₄) is an inverse spinel ferrite that has attracted considerable interest because of its high chemical and thermal stability, moderate magnetocrystalline anisotropy, high coercivity, and useful electrical and magnetic properties [1–5]. These characteristics make CoFe₂O₄ nanoparticles (CoFe₂O₄ NPs) suitable for a wide range of applications, including magnetic and electromagnetic sensors, high-frequency electronic devices, electromagnetic interference (EMI) suppression, magnetic hyperthermia, drug delivery and other biomedical applications [3–5]. The functional properties of CoFe₂O₄ NPs are strongly influenced by their crystal structure, particle size, morphology, porosity, cation distribution and synthesis conditions.

Several techniques have been employed to synthesize CoFe₂O₄ NPs, including co-precipitation, hydrothermal processing, solid-state reactions and sol-gel methods [6–9]. Among these approaches, the sol-gel method provides good control over chemical composition and particle characteristics while enabling the preparation of relatively homogeneous and high-purity nanopowders at comparatively low processing temperatures [10–13]. In addition, it is relatively simple, cost-effective and suitable for producing nanostructured ferrites for magnetic, dielectric

and high-frequency applications [14–23]. These advantages make the sol–gel route appropriate for investigating the influence of subsequent thermal treatment on the functional properties of CoFe_2O_4 NPs.

The influence of heat treatment on the structural, dielectric and magnetic properties of cobalt ferrite has been investigated in several previous studies. Badr et al. [24] reported that annealing Ce-substituted cobalt ferrite between 600 and 1000°C increased both saturation and remanent magnetization, together with changes in grain size. Rahardjo et al. [6] observed an increase in crystallite size in Y-substituted cobalt ferrite with increasing treatment temperature, although the saturation magnetization decreased. Zhang et al. [25] found that increasing the sintering temperature of CoFe_2O_4 from 900 to 1300°C promoted grain growth and increased saturation magnetization, while coercivity decreased at higher temperatures. Silva et al. [26] also demonstrated a strong effect of heat-treatment temperature on the phase composition, morphology, and functional performance of sol–gel-derived CoFe_2O_4 . Collectively, these studies confirm that thermal treatment is a key parameter controlling the microstructure and functional properties of CoFe_2O_4 .

Although the general effects of sintering temperature on cobalt ferrite are well established, direct comparison of structural, dielectric and magnetic responses over a broad and identical set of processing conditions remains useful for identifying suitable conditions for multifunctional applications. Accordingly, the present study systematically evaluates sol–gel-prepared CoFe_2O_4 NPs sintered at 400°C, 600°C, 800°C and 1000°C for the same treatment duration. Structural and morphological evolution is correlated with dielectric and magnetic behavior using XRD, FTIR, SEM, AFM, dielectric measurements and vibrating sample magnetometry. Particular attention is given to identifying the sintering condition that achieves the best balance between crystallinity, microstructure, dielectric loss and magnetic performance rather than maximizing a single property. Therefore, this work is positioned as a systematic optimization study that extends previously reported trends and provides a comparative basis for selecting CoFe_2O_4 processing conditions for multifunctional and high-frequency applications.

2. Experimental Procedures

2.1. Synthesis of CoFe_2O_4 NPs

To produce cobalt ferrite nanoparticles (CoFe_2O_4 NPs), the sol-gel technique was used as a convenient procedure due to its precise control over the crystal structure and chemical composition. Initially, stoichiometric ratios of 0.5M cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) and 1M ferric chloride (FeCl_3), both 98% pure and from BDH, were dissolved in distilled water (D.W.), with a ferric-to-cobalt molar ratio of 2:1. After initial solution preparation, 2M sodium hydroxide (NaOH) solution from Fluka (98% purity) was gradually added to the mixture in droplets, under continuous stirring for 90 minutes (see Table 1). The pH of the mixture was adjusted to 13 to ensure the appropriate basic conditions for gel formation. The initial heating was carried out at a constant temperature of $100 \pm 2^\circ\text{C}$, with continuous stirring for several hours until a dark and wet gel was formed. The gel

was then dried and ground into a fine powder. The resulting powder was washed several times with distilled water and alcohol to remove impurities such as sodium chloride produced during the reaction. After purification, the powder was subjected to a sintering treatment process in a German Nabertherm furnace. The samples were sintered at different temperatures (400°C, 600°C, 800°C and 1000°C) for 2 hours. This wide temperature range was designed to determine the optimal sintering conditions that achieve a functional balance between structural, dielectric and magnetic properties of CoFe_2O_4 NPs in multifunctional applications. Fig. 1 shows the complete sequence of preparation steps using the sol-gel technique, from the preparation of chemical solutions to the sintering process of CoFe_2O_4 NPs.

Table 1. Chemicals used in the synthesis of CoFe_2O_4 NPs.

Chemicals	Formula	Purity (%)	Company	Weights (g)	D.W. (ml)
Ferric chloride	FeCl_3	98	BDH	8.19	50
Cobalt chloride	$\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	98	BDH	6	50
Sodium hydroxide	NaOH	98	Fluka	8	100

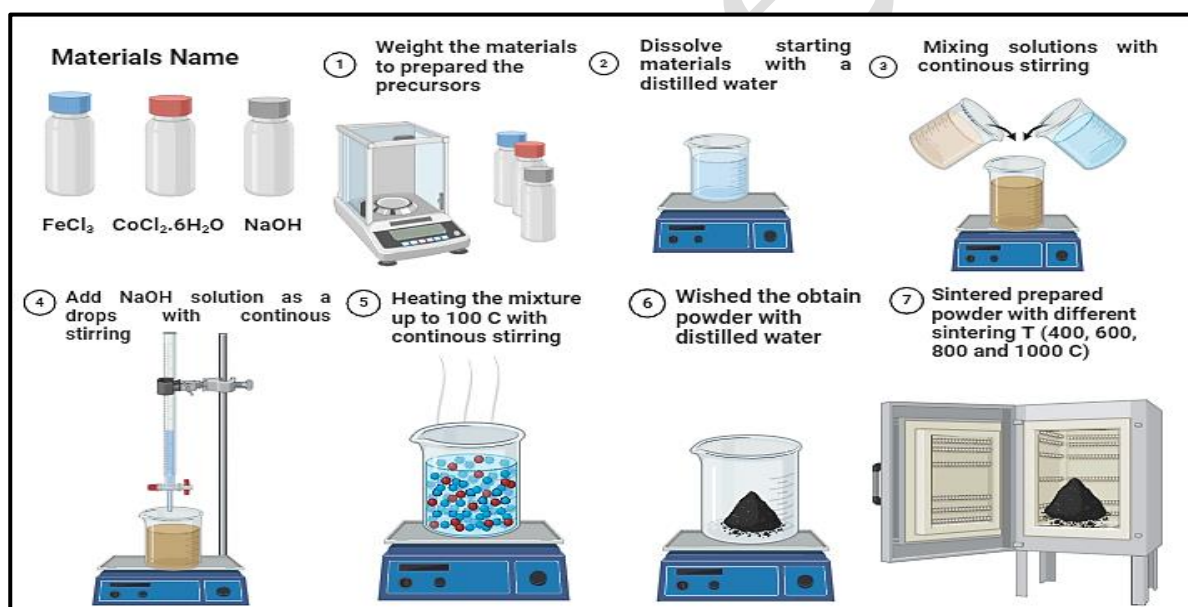


Fig. 1. Preparation steps of CoFe_2O_4 NPs at different sintering temperatures.

2.2. Characterization

Structural, morphological, dielectric and magnetic characterization was performed using X-ray diffraction (XRD) - (ADX-2500, $\text{Cu-K}\alpha$, $\lambda = 1.54060 \text{ \AA}$), scanning electron microscopy (SEM) - (FEI Inspect S50), atomic force microscopy (AFM) - (Angstrom Advanced Inc.), Fourier transform infrared (FTIR) spectroscopy (SHIMADZU-8400S; $400\text{--}4000 \text{ cm}^{-1}$), an LCR meter (MICROTEST) and vibrating-sample magnetometer (VSM) measurements at room temperature under an applied field of $\pm 15 \text{ kOe}$.

3. Results and Discussion

3.1. Structural and morphological analysis

The structural parameters of CoFe_2O_4 NPs were derived from the XRD data using standard relations. The crystallite size (D), lattice constant (a), X-ray density (ρ_x), estimated specific surface area (S), porosity (P) and hopping lengths at tetrahedral (L_A) and octahedral (L_B) sites were calculated as follows using Eqs. (1) – (6) [28–31]:

$$D = \frac{0.9 \lambda}{\beta \cos \theta} \quad (1)$$

$$a = d_{hkl} \sqrt{h^2 + k^2 + l^2} \quad (2)$$

$$\rho_x = \frac{8M}{N_A a^3} \quad (3)$$

Where: M - molecular weight of CoFe_2O_4 NPs; N - Avogadro number and a - lattice constant.

$$S = \frac{6000}{D \rho_x} \quad (4)$$

$$P (\%) = \left(1 - \frac{\rho_m}{\rho_x}\right) \times 100 \quad (5)$$

Where: P - porosity percentage; ρ_m - mass density and ρ_x - X-ray density.

$$L_A = 0.4325 (a); L_B = 0.3525 (a) \quad (6)$$

The XRD patterns in Fig. 2 confirm the formation of a single-phase cubic spinel CoFe_2O_4 structure with $\text{Fd}\bar{3}\text{m}$ symmetry, which corresponds well to JCPDS card No. 22-1086. The characteristic reflections corresponding to the (220), (311), (222), (400), (422), (511) and (440) planes were observed, with no detectable secondary phases. At higher sintering temperatures, particularly above 600°C, the (311) reflection showed a slight shift toward lower 2θ , accompanied by sharper diffraction peaks, indicating changes in lattice strain and improved crystallinity. As shown in Fig. 3a and Table 2, the crystallite size increased from 10.71 to 16.09 nm with increasing sintering temperature, reflecting thermally activated crystallite growth and coalescence. These values are comparable with those previously reported for CoFe_2O_4 prepared by related synthesis routes (Table 3) [32–36]. The lattice constant remained nearly unchanged between 400°C and 600°C and then subsequently increased, reaching 8.3753 Å at 1000°C, which may be associated with heat-induced lattice expansion and/or localized structural strain redistribution [37]. The hopping lengths L_A and L_B showed a similar variation because both depend directly on the lattice constant.

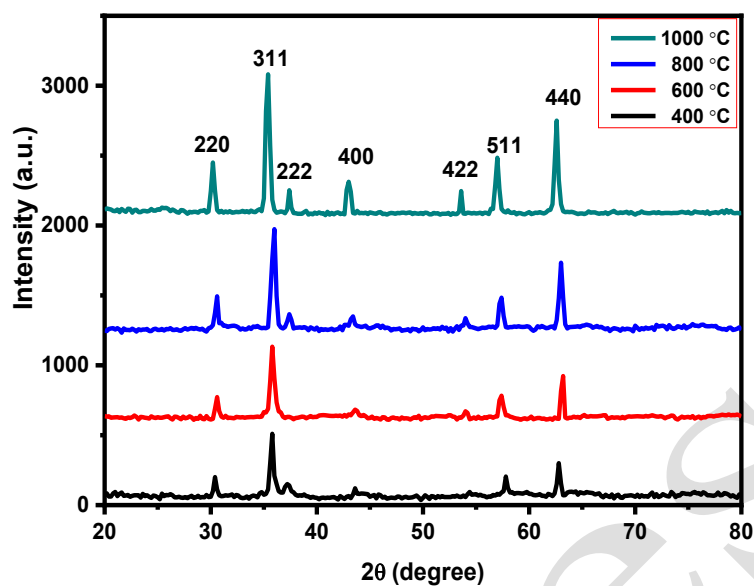


Fig. 2. XRD patterns of CoFe₂O₄ NPs at different sintering temperatures.

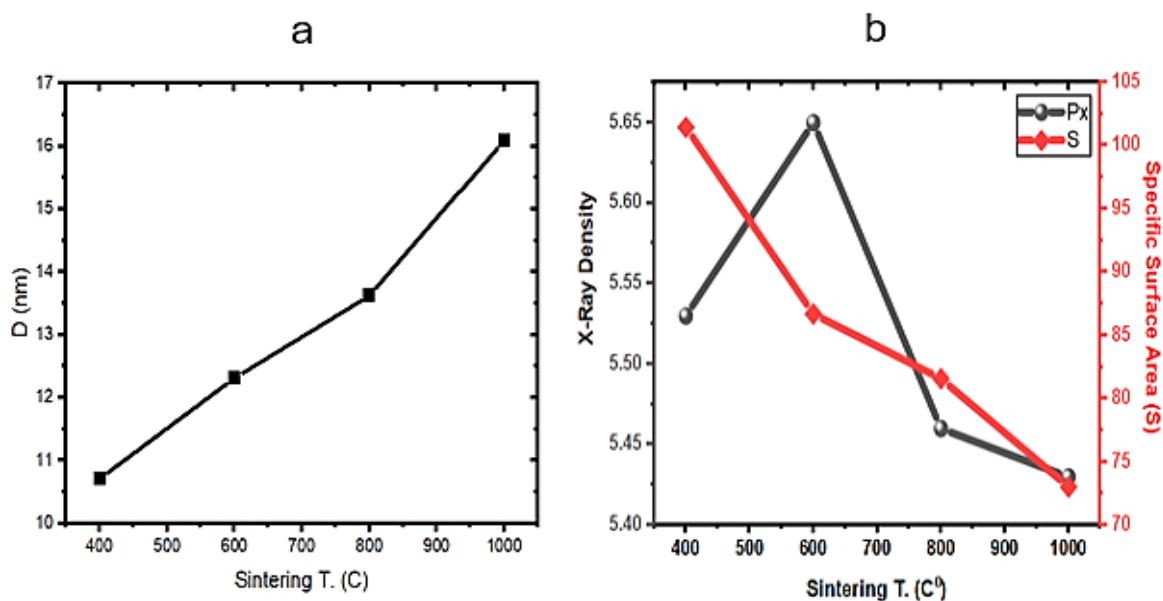


Fig. 3. Variation of crystallite size (a) and X-ray density and specific surface area as a function of sintering temperature (b).

The dislocation density (δ) and microstrain (ϵ) of the CoFe₂O₄ NP samples were calculated using Eqs. (7) and (8) [38]:

$$\delta = \frac{1}{D^2} \quad (7)$$

$$\epsilon = \frac{\beta \cos \theta}{4} \quad (8)$$

Similar XRD-based analyses of structural evolution in nanostructured ferrite and oxide systems have also been reported [39, 40]. With increasing sintering temperature, crystallite size increased while both microstrain and dislocation density decreased, indicating improved crystallinity and reduced structural defects. The specific surface area also decreased, along with a drop in porosity from 65% to 34%, which is consistent with progressive densification. The X-ray density showed only a slight non-monotonic variation, as shown in Table 2.

Table 2. XRD parameters of CoFe₂O₄ NPs at different sintering temperatures.

Sintering Temperatures (°C)	D (nm)	a (Å°)	d ₃₁₁ (Å°)	ρ _x (g/cm ³)	S (m ² /g)	P (%)	L _A	L _B	Dislocation density (δ) x 10 ¹⁵ (m ⁻²)	ε	Volume of unit cell a = b = c (Å ³)
400	10.71	8.1874	2.4686	5.679	101.5	65	3.545	2.895	8.7181	0.167	548.83
600	12.31	8.1838	2.4675	5.686	86.8	62	3.544	2.893	6.5991	0.122	548.11
800	13.62	8.2986	2.5021	5.454	81.9	49	3.593	2.934	5.3907	0.111	571.50
1000	16.09	8.3753	2.5252	5.305	73.2	34	3.627	2.961	3.8627	0.082	587.49

Table 3. Comparison of XRD parameters of CoFe₂O₄ NPs with other works.

Synthesis method	Heat treatment temperature (°C)	D (nm)	a (Å°)	Ref.
PEG-assisted sol-gel method	300 – 800	7.6 – 12.8	~ 8.39	[32]
Co-precipitation method	600 – 750	15.96 – 19.11	8.2940 – 8.3313	[34]
Green synthesis of the sol-gel method	200 – 750	3.7 – 9.4	8.0688 – 8.3779	[37]
Hydrothermal method	As prepared, 700 – 800	43.49 – 53.85	8.391 – 8.400	[41]
Sol-gel auto-combustion method	200 – 1000	26.38 – 56.53	8.3801 – 8.3937	[42]
Sol-gel precipitation method	400 – 1000	10.71 – 16.09	8.1838 – 8.3753	Present work

Fig. 4 shows SEM images that reveal the gradual evolution of the microstructure with increasing sintering temperature. At 400°C (Fig. 4a), the grains are relatively small (28 ± 4 nm), irregularly distributed and separated by visible voids, indicating incomplete densification. At 600°C (Fig. 4b), partial coalescence occurs and the average grain size increases to 39 ± 6 nm. At 800°C (Fig. 4c), larger and more interconnected grains (53 ± 5 nm) are observed, indicating advanced sintering and improved grain connectivity. Further heating to 1000°C (Fig. 4d) produces pronounced coalescence and excessive grain growth, with an average grain size of 65 ± 5 nm. Overall, increasing temperature promotes diffusion and grain-boundary migration, leading to grain growth and reduced porosity, in agreement with the XRD results. Fig. 5 shows 3D AFM surface morphology of CoFe₂O₄ NPs at different sintering temperatures. The surface becomes

progressively more homogeneous from 400°C to 800°C (Figs. 5a to 5c), indicating improved recrystallization and grain connectivity. The sample at 800°C exhibits the most uniform surface morphology, whereas further heating to 1000°C (Fig. 5d) leads to marked grain growth and increased surface roughness due to particle coalescence. These observations are consistent with the SEM and XRD results.

FTIR spectroscopy was used to confirm the characteristic metal–oxygen vibrations of the spinel CoFe_2O_4 structure and to assess the effect of sintering temperature on the local bonding environment. As shown in Fig. 6, the higher-wavenumber bands at 516.94–592.17 cm^{-1} are attributed to M–O vibrations at the tetrahedral A-site, whereas the lower-wavenumber bands at 408.92–499.58 cm^{-1} correspond to M–O vibrations at the octahedral B-site. The presence of these bands in all samples confirms the preservation of the spinel structure throughout the investigated temperature range. Their shifts and splitting with increasing sintering temperature indicate changes in the local M–O environment, which are likely associated with lattice distortion, defect evolution and possible cation redistribution. However, FTIR band splitting alone is insufficient to quantitatively determine the degree of cation inversion. Additional bands at higher wavenumbers are attributed mainly to surface hydroxyl groups, adsorbed moisture and carbon-containing residual species. The main vibrational assignments are summarized in Table 4.

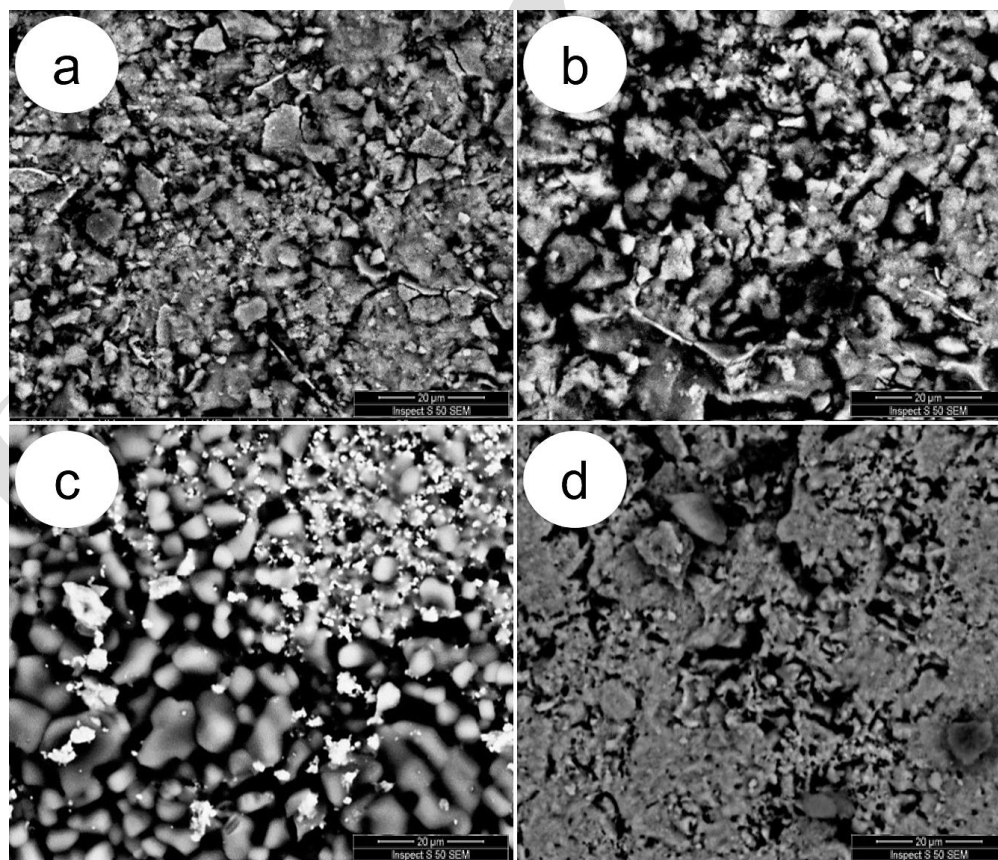


Fig. 4. SEM images of CoFe_2O_4 NPs at different sintering temperatures: 400°C (a), 600°C (b), 800°C (c) and 1000°C (d).

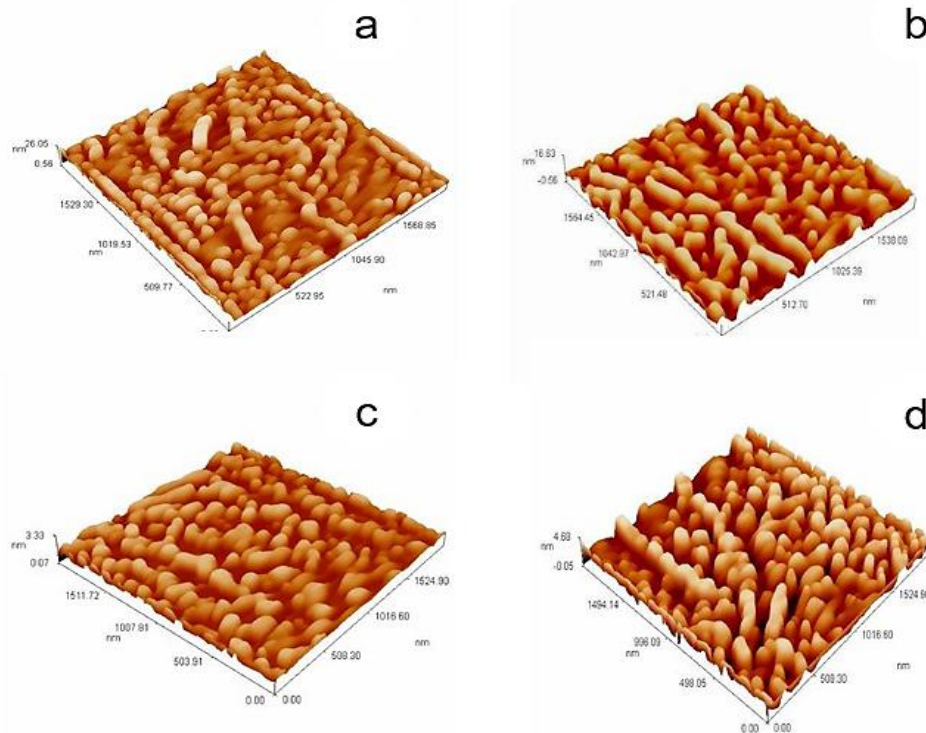


Fig. 5. 3D AFM images of CoFe₂O₄ NPs at different sintering temperatures: 400°C (a), 600°C (b), 800°C (c) and 1000°C (d).

To further evaluate the effect of sintering temperature on the local metal–oxygen bonding, the force constants associated with the tetrahedral and octahedral sites were calculated from the corresponding FTIR wavenumbers using [43]:

$$F = 4\pi^2 c^2 \mu v^2 \quad (9)$$

Where: F - force constant; c - speed of light; μ - reduced mass of the vibrating metal–oxygen pair and v - corresponding FTIR wavenumber. The average force constant (F_{av}), when both tetrahedral (F_T) and octahedral (F_O) contributions are available as a corresponding pair, was estimated from [43]:

$$F_{av} = \frac{F_T + F_O}{2} \quad (10)$$

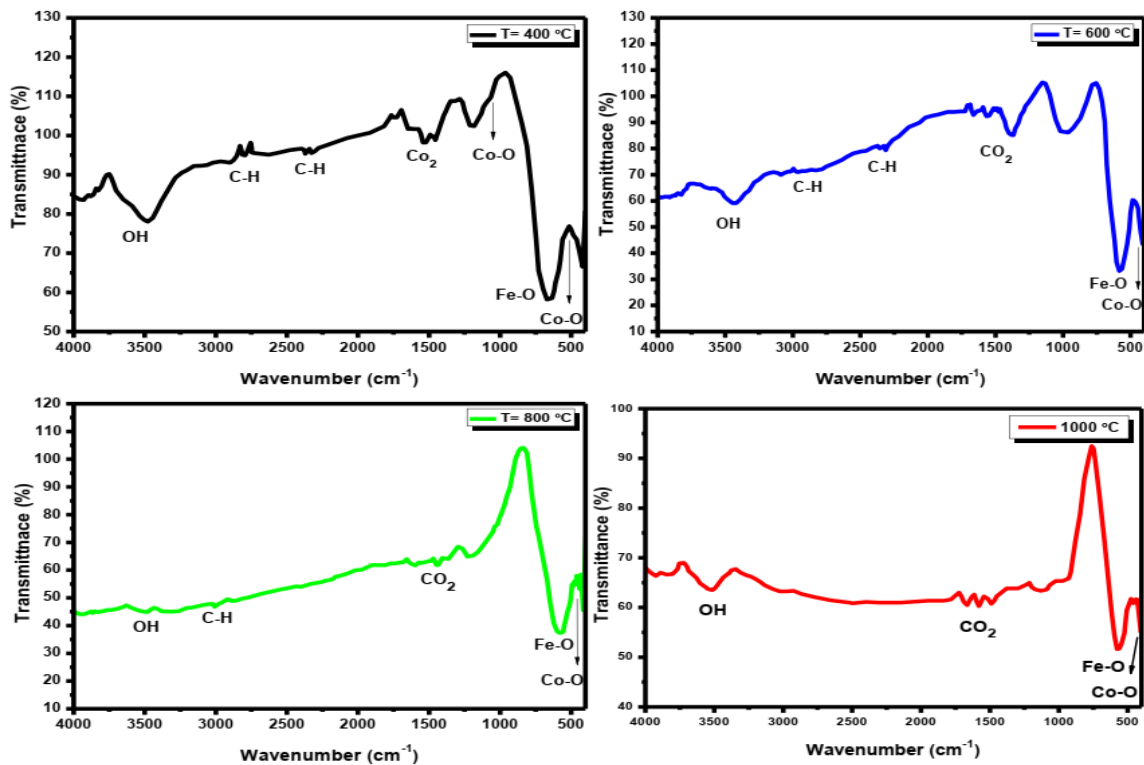


Fig. 6. FTIR pattern of CoFe₂O₄ NPs at different sintering temperatures.

Table 4. Functional groups identified in CoFe₂O₄ NPs synthesized at different sintering temperatures.

Sintering temperatures	M _{octa} -O	M _{tetra} -O	Co-sub. spinel ferrites Group	Sym. & assym. stretching vibration of CO ₂	Aliphatic & aromatic C-H bond	C-H stretching bands	OH-group
400°C	499.58	592.17	1065.03	1561.10 1560.45 1632.89 1700.34	2226.58 2362.88 2362.88	2744.49 2878.01 3030.30	3305.33 3459.81
600°C	416.64 457.14	572.88	—	1440.90 1470.74 1580.29 1650.13	2330.44 2588.31	2785.30 3005.20	3443.76
800°C	443.64 484.15	585.52 592.17	1077.53	1470.16 1519.51 1527.34 1688.88	3103.14	—	3430.70 3476.65
1000°C	408.92	516.94 574.81 588.31	1078.24	1536.12 1578.13 1601.28 1633.51	2362.88	3130.27	3448.84

Similar FTIR-based evaluations of tetrahedral and octahedral force constants and their average have also been reported for related spinel ferrite systems [44–46]. The force constants associated with the tetrahedral and octahedral sites were calculated separately for each resolved M–O vibrational component using Eq. (9). The higher-wavenumber components (ν_1) were assigned to tetrahedral A-site vibrations and were therefore used to calculate F_T , whereas the lower-wavenumber components (ν_2) were assigned to octahedral B-site vibrations and were used to calculate F_O . Accordingly, multiple F_T or F_O values at a given sintering temperature arise from resolved/split vibrational components within the same sample and do not represent different samples. The average force constant (F_{av}) was calculated only when a corresponding tetrahedral–octahedral vibrational pair was available; otherwise, it was not determined. The resulting force constants are summarized in Table 5.

Table 5. Vibrational properties of CoFe_2O_4 NPs at different sintering temperatures.

Sintering temperatures (°C)	$\nu_1 \text{ cm}^{-1}$	$\nu_2 \text{ cm}^{-1}$	$F_T \times 10^5$ (dyne/cm)	$F_O \times 10^5$ (dyne/cm)	$F_{av} \times 10^5$ (dyne/cm)	Ref.
400	592.17	499.58	2.57	1.83	2.19	Present work
600	572.88	416.64	2.41	1.27	1.83	
	—	457.14	—	1.53	—	
800	585.52	443.64	2.51	1.44	1.97	
	592.17	484.15	2.57	1.72	2.14	
1000	516.94	408.92	1.96	1.23	1.59	
	574.81	—	2.42	—	—	
	588.31	—	2.54	—	—	
400	584.46	382.89	2.53	1.07	1.80	[6]
800	599.67	355.24	2.63	0.92	1.77	[45]
500	570	364	2.37	0.96	1.67	[47]

3.2. Dielectric properties

The dielectric properties of spinel ferrites, such as CoFe_2O_4 NPs, are generally appropriate for use in high-frequency devices, and new possible uses are continuously being investigated. The synthesis process, material composition, sintering temperature and time are important factors in regulating the characteristics of these dielectric materials [41].

The dielectric loss factor ($\tan \delta$), the real part of the dielectric constant (ϵ') and the imaginary part of the dielectric constant (ϵ'') were determined using the following equations [46]:

$$\epsilon' = \frac{C d}{\epsilon_0 A} \quad (11)$$

$$\epsilon'' = \epsilon' \tan \delta \quad (12)$$

Fig. 7 shows the frequency-dependent dielectric properties at room temperature of CoFe_2O_4 NPs sintered at different temperatures. Both ϵ' and ϵ'' decrease with increasing frequency over the

investigated sintering-temperature range (Figs. 7a and 7b). This dispersion is consistent with the Maxwell–Wagner interfacial polarization mechanism and Koops’ model, in which charge accumulation at resistive grain boundaries enhances polarization at low frequencies, whereas charge carriers are unable to follow the rapidly varying electric field at high frequencies [47–50]. Fig. 7c shows the frequency dependence of the dielectric loss factor ($\tan \delta$) at different sintering temperatures. At 1 kHz, $\tan \delta$ decreases from 4.03 at 400°C to a minimum value of approximately 2 at 800°C, before increasing to 3.1 at 1000°C. The sample sintered at 800°C exhibited the lowest dielectric loss at 1 kHz among the investigated samples. The dielectric response of CoFe_2O_4 is mainly associated with electron hopping between $\text{Fe}^{+2}/\text{Fe}^{+3}$ ions and hole hopping involving $\text{Co}^{+2}/\text{Co}^{+3}$ ions [41]. As shown in Fig. 7d, both ϵ' and ϵ'' at 1 kHz decrease with increasing sintering temperature, which can be attributed to grain growth and the consequent reduction in grain-boundary and interfacial polarization contributions [51]. Table 6 shows the dielectric properties of CoFe_2O_4 NPs, including the real part of the dielectric constant (ϵ'), the imaginary part (ϵ'') and the dielectric loss factor ($\tan \delta$), which were measured at different frequencies and sintering temperatures. The observed values are consistent with the frequency- and temperature-dependent dielectric behavior discussed above.

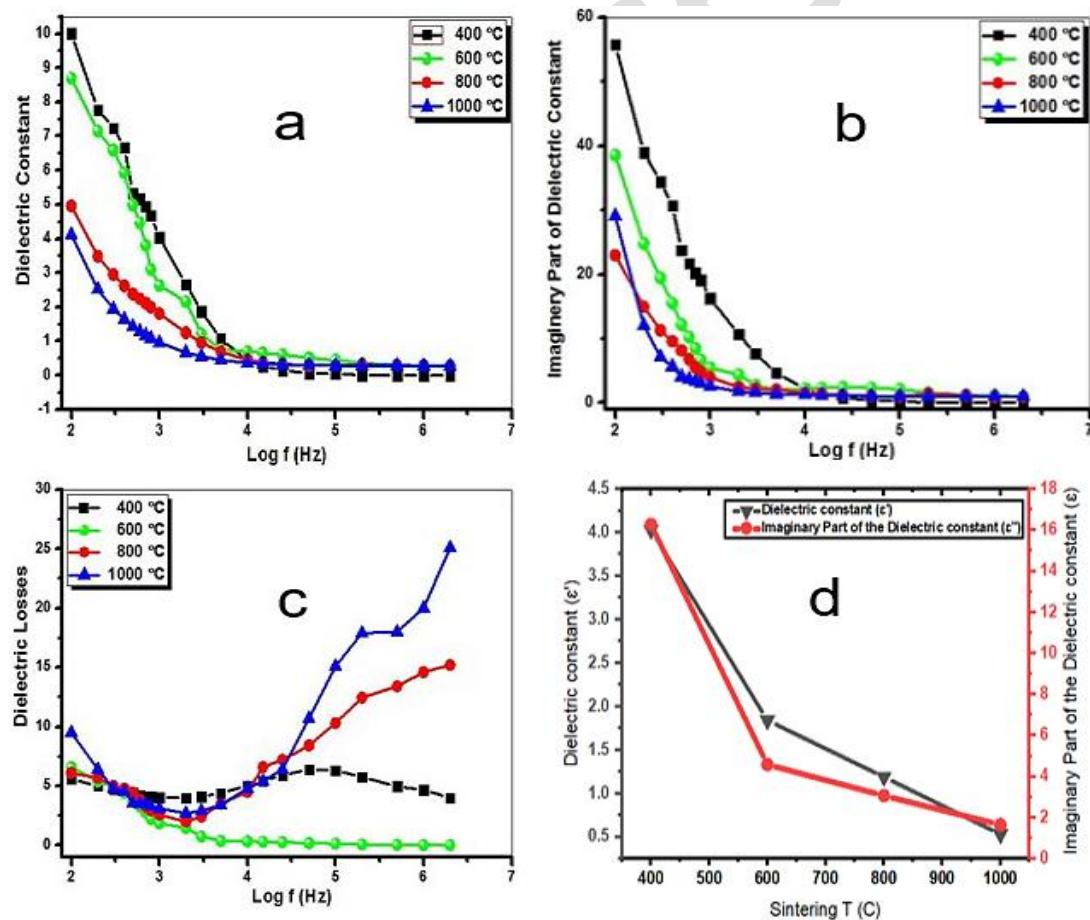


Fig. 7. Schematic representations of variation of dielectric constant (a), imaginary part of dielectric constant (b), dielectric losses factor with frequency (c) and value of dielectric constant and imaginary part of dielectric constant as a function of sintering temperatures at 1 kHz (d).

Table 6. Dielectric properties of CoFe₂O₄ NPs at different frequencies as a function of sintering temperatures.

Sintering temperature (°C)	Dielectric constant (ϵ')			Imaginary part of dielectric constant (ϵ'')			Dielectric losses factor ($\tan \delta$)		
	100 Hz	1 kHz	1 MHz	100 Hz	1 kHz	1 MHz	100 Hz	1 kHz	1 MHz
400	10.01	4.03	10 ⁻³	55.76	16.27	4x10 ⁻³	5.57	4.03	4.67
600	6.58	1.84	0.01	38.21	4.58	0.21	5.8	2.38	11.6
800	3.66	1.19	6x10 ⁻⁴	22.37	3.07	5x10 ⁻³	6.1	2	15.2
1000	3	0.53	9x10 ⁻⁴	28.55	1.65	0.02	9.5	3.1	25.1

The AC conductivity (σ) was calculated using the following relationships (13 and 14) [41]:

$$\sigma = \epsilon' \epsilon_0 \omega \tan \delta \quad (13)$$

Where: ω - angular frequency

$$\omega = 2\pi f \quad (14)$$

Where: f - frequency.

Fig. 8 shows the frequency-dependent AC conductivity (σ) of CoFe₂O₄ NPs sintered at 400–1000°C at room temperature. In general, AC conductivity increases with frequency, consistent with the Maxwell–Wagner model and electron hopping between Fe⁺²/Fe⁺³ ions at octahedral B-sites. At low frequencies, charge accumulation at resistive grain boundaries limits conduction, whereas at higher frequencies, enhanced charge-carrier hopping within the grains leads to increased AC conductivity [52]. Regarding the effect of sintering temperature, the sample at 600°C exhibited the highest conductivity, whereas the samples at 800°C and 1000°C maintained comparatively lower values over the measured frequency range. The sample at 400°C showed a distinct non-monotonic response, with conductivity reaching a maximum near 1 kHz, followed by a decrease, and then a slight recovery at higher frequencies. Its small crystallite size (≈ 10.71 nm), high defect density and larger contribution of resistive grain boundaries may influence this response. However, these factors alone cannot fully explain the observed maximum value. The relaxation-related processes or electrode/contact effects may also contribute. Therefore, without additional impedance or electrode-control measurements, the exact origin of this feature cannot be conclusively determined and requires further investigation [53].

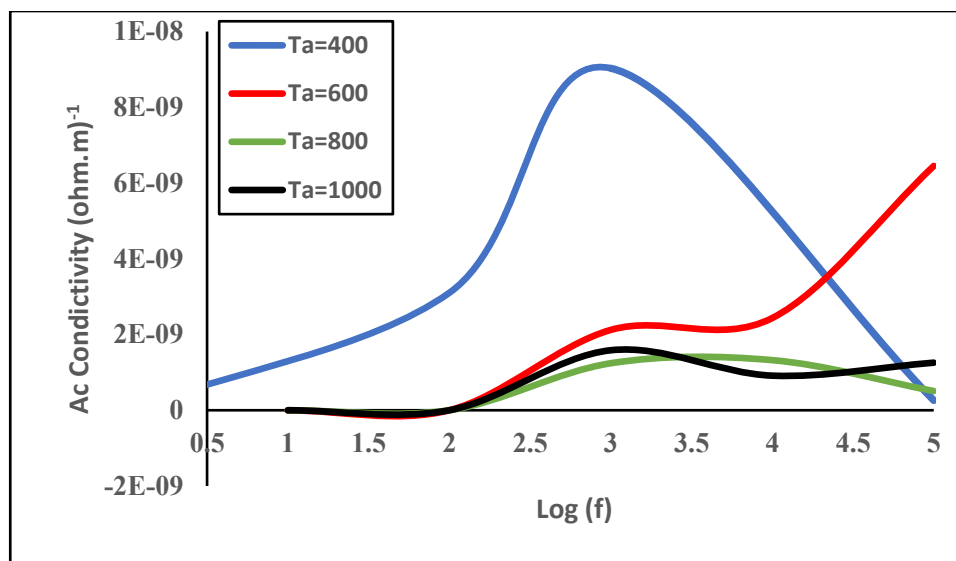


Fig. 8. AC conductivity of CoFe₂O₄ NPs at different frequencies as a function of sintering temperatures.

3.3. Magnetic properties

Fig. 9 shows the room-temperature M–H hysteresis loops of CoFe₂O₄ NPs sintered at 400, 600, 800 and 1000°C. All samples exhibit ferrimagnetic hysteresis behavior, with systematic changes in saturation magnetization (M_s), remanent magnetization (M_r) and coercivity (H_c) as a function of sintering temperature. The corresponding magnetic parameters calculated from the hysteresis loops are summarized in Table 7. The magnetic moment per formula unit can be found using hysteresis loops at room temperature from the following equation [28]:

$$\mu_B = \frac{M \times M_s}{5585} \quad (15)$$

The saturation and remanent magnetizations increased with sintering temperature, as shown in Fig. 10a, with M_s increasing from 27.5 emu/g at 400°C to 68 emu/g at 1000°C. This behavior is mainly attributed to grain growth, improved crystallinity, stronger exchange interactions and reduced surface-spin disorder, in agreement with previous studies [6, 27, 48, 54, 55]. However, the M_s values remained below the bulk value of CoFe₂O₄ (80.8 emu/g), which can be explained by finite-size effects and spin disorder at the nanoparticle surface [48, 49]. The maximum M_s value obtained in the present study is also comparable to values reported for CoFe₂O₄ NPs in previous studies [56, 57]. Table 7 shows relatively low remanence ratios ($M_r/M_s = 0.17$ – 0.36). According to the Stoner–Wohlfarth model, these values are lower than the expected ideal value for non-interacting single-domain particles with randomly oriented easy axes, indicating a large contribution from uniaxial anisotropy. The coercivity increased from 209 Oe at 400°C to a maximum of 715 Oe at 800°C, and then decreased to 211 Oe at 1000°C, as shown in Fig. 10b. The initial increase is associated with enhanced magnetic anisotropy and grain growth, whereas the subsequent decrease at 1000°C is attributed to the transition toward a multidomain structure and easier domain-wall motion [58]. Thus, the sample at 800°C exhibits the highest resistance to magnetization reversal. The magnetic

moment of ideal inverse-spinel CoFe_2O_4 is $3.0 \mu\text{B}$ per formula unit at 0 K, according to the collinear Néel model. In the present samples, μB increased from 1.15 to $2.88 \mu\text{B}$ with increasing sintering temperature. The lower experimental μB values may be associated with surface spin disorder, spin canting, finite-size effects, structural defects and possible changes in cation distribution [59]. Therefore, the increase in μB with sintering temperature is mainly attributed to improved magnetic ordering and reduced surface-spin disorder. However, the degree of cation inversion cannot be quantitatively determined from the present room-temperature magnetic measurements alone. Among the investigated samples, the sample at 800°C showed the best overall balance between microstructural uniformity, low dielectric loss and magnetic performance ($M_s = 51 \text{ emu/g}$ and $H_c = 715 \text{ Oe}$), and was therefore selected as the optimum sintering condition for multifunctional applications. Table 8 compares the magnetic parameters obtained in the present work with values reported in recent studies. Considerable variations in M_s and H_c are observed in the literature, reflecting differences in synthesis routes, crystallite/grain sizes, cation distributions and heat-treatment conditions. The present sample at 800°C ($M_s = 51 \text{ emu/g}$ and $H_c = 715 \text{ Oe}$) is within the reported range and shows a balanced magnetic response, whereas the sample at 1000°C shows a higher M_s but markedly lower H_c .

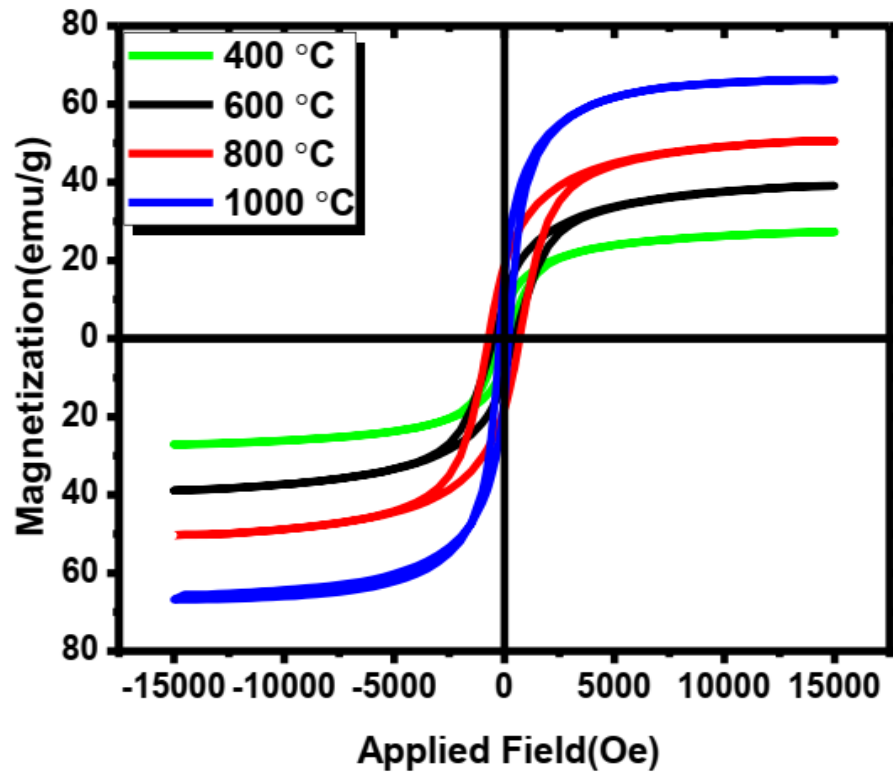


Fig. 9. VSM of CoFe_2O_4 NPs at different sintering temperatures.

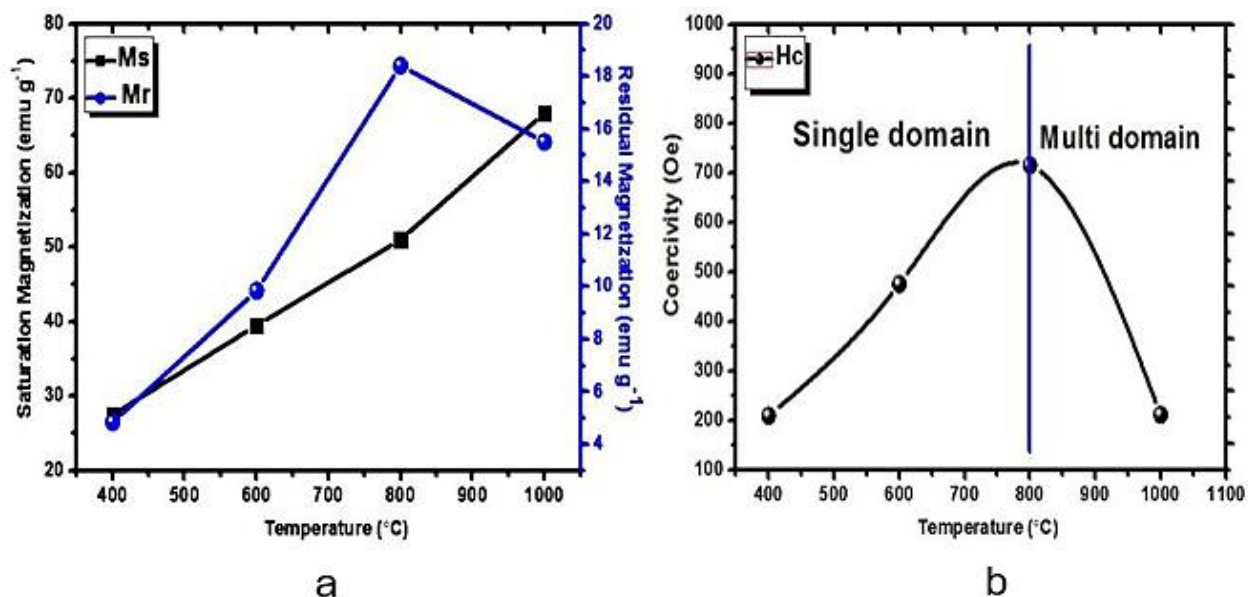


Fig. 10. Effect of sintering temperature on (a) saturation magnetization (M_s) and remanent magnetization (M_r) and (b) coercivity (H_c) of CoFe₂O₄ NPs.

Table 7. Magnetic parameters of CoFe₂O₄ NPs at different sintering temperatures.

Sintering Temperature (°C)	(M_s) (emu g ⁻¹)	(M_r) (emu g ⁻¹)	(H_c) (Oe)	(M_r/M_s) ratio	μB (B.M.)
400	27.5	4.84	209	0.17	1.15
600	39.5	9.84	475	0.24	1.66
800	51	18.4	715	0.36	2.14
1000	68	15.5	211	0.22	2.88

Table 8. Comparison of magnetic parameters of CoFe₂O₄ NPs with other works.

Ref.	Heat treatment temperature (°C)	D (nm)	M_s (emu g ⁻¹)	H_c (Oe)	M_r/M_s ratio	μB (B.M.)
[6]	400	16.05	30.79	1588	0.39	1.29
[25]	1300	830	87.32	1368.33	0.19	—
[32]	800	12.80	85.5	1920	0.36	—
[39]	600	56.27	73	1958	0.52	—
[41]	800	53.85	46.20	467.73	0.47	1.94
[42]	1000	56.53	77.94	679	0.27	3.27
[56]	600	13.98	68.03	1439.59	0.48	—
[60]	700	21	41	580	0.30	1.72
[61]	900	40	84.97	720	0.30	3.57
[62]	600	25.32	50	1100	0.42	—

[63]	800	55.60	72.58	1005.33	0.47	3.05
[64]	1200	—	80.03	189.1	0.12	—
[65]	500	24.11	50.60	1077	0.56	2.26
Present work	800	13.62	51	715	0.36	2.14
	1000	16.09	68	211	0.22	2.88

4. Conclusions

CoFe₂O₄ NPs were successfully synthesized by the sol–gel method and sintered at 400, 600, 800 and 1000°C to evaluate the influence of sintering temperature on their structural, dielectric, and magnetic properties. XRD analysis confirmed the formation of a stable cubic spinel structure, while increasing the sintering temperature enhanced crystallinity and grain connectivity and reduced structural defects and porosity, accompanied by increases in crystallite and grain sizes. The dielectric response showed a general decrease in the real and imaginary parts of the permittivity with increasing frequency. In contrast, the sample sintered at 800°C exhibited the lowest dielectric loss ($\tan \delta \approx 2$ at 1 kHz). Magnetic measurements showed an increase in saturation magnetization with sintering temperature, reaching 68 emu/g at 1000°C, while coercivity reached its maximum value of 715 Oe at 800°C. Despite the higher saturation magnetization obtained at 1000°C, the associated excessive grain growth and deterioration in dielectric performance indicate a trade-off between magnetic and dielectric properties. Therefore, the sample sintered at 800°C provides the most favorable overall balance, with a crystallite size of 13.62 nm, $M_s = 51$ emu/g, $H_c = 715$ Oe and relatively low dielectric loss, making it a strong candidate for further evaluation in high-frequency and multifunctional magnetic applications. Future work should directly evaluate the electromagnetic interference shielding effectiveness of the 800°C sample in the X-band (8–12 GHz) and investigate the stability of its magnetic properties under repeated thermal cycling.

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