

RESEARCH ARTICLE

Structural and Morphological Enhancement of CrFe₂O₄/GO Nanocomposites via Sol–Gel Auto–Combustion Synthesis

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ABSTRACT: This study details the synthesis and characterization of a chromium ferrite-graphene oxide (CrFe₂O₄/GO) nanocomposite, prepared using a sol-gel auto-combustion method with 10 wt.% graphene oxide integrated into the ferrite matrix. The structural and morphological properties were systematically investigated through a suite of analytical techniques. X-ray diffraction confirmed the formation of a single-phase cubic spinel structure with a space group of Fd-3m (227) and a lattice constant of 8.50 Å, indicating high phase purity and crystallinity. Fourier-transform infrared and Raman spectroscopy provided evidence of successful GO incorporation and identified characteristic vibrational modes associated with both the spinel ferrite and the carbonaceous framework. Field emission scanning electron microscopy and high-resolution transmission electron microscopy revealed a homogeneous distribution of ferrite nanoparticles anchored onto exfoliated GO sheets, forming a dense, nano-phase microstructure with minimal agglomeration. Magnetic characterization using vibrating sample magnetometry demonstrated enhanced magnetic properties, including significant coercivity and saturation magnetization, which are attributed to effective interparticle coupling and domain wall interactions facilitated by the GO matrix. The uniform dispersion of GO within the ferrite host promoted conductive pathways and defect-induced polarization, contributing to improved functional behavior. These findings underscore the potential of the CrFe₂O₄/GO nanocomposite in advanced applications such as energy conversion, magnetic storage, and catalytic systems, owing to its structural robustness, magnetic responsiveness, and synergistic component interactions.

Keywords: CrFe₂O₄/GO nanocomposite, sol-gel auto-combustion, spinel ferrite, structural morphology, magnetic properties, Raman spectroscopy.

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1. INTRODUCTION

Spinel ferrites, with the general formula AB₂O₄, represent a critically important class of metal oxides that have sustained extensive academic and technological interest for decades, primarily due to their versatile and tunable magnetoelectric properties [1]. Their fundamental crystal structure is based on a face-centered cubic lattice of oxygen ions, within which the metallic cations (A and B) occupy specific interstitial sites, tetrahedral (A-site) and octahedral (B-site) positions [2]. This arrangement is not merely structural but foundational to their functionality; the deliberate selection

and distribution of different metal cations across these sites allow for precise engineering of the material's physical and chemical characteristics. This tunability renders spinel ferrites exceptionally suitable for a diverse array of applications, spanning from high-frequency electronics and magnetic recording media to biomedical technologies and heterogeneous catalysis [3-6]. In recent years, research impetus has further shifted toward leveraging these materials for pressing environmental and energy challenges. Their proven efficacy in processes such as photocatalytic water purification, electrochemical hydrogen evolution, catalytic carbon dioxide reduction, and air pollutant filtration underscores their potential as multifunctional platforms for sustainable technology [7-9].

Within this broad family, chromium-substituted iron oxides, particularly magnetic chromium ferrite (CrFe₂O₄), have garnered significant attention. These materials uniquely combine the robust magnetic character of ferrites with the catalytic and stability attributes imparted by chromium ions [10]. The resulting nanoparticles exhibit compelling magnetic properties, including adjustable coercivity and saturation magnetization, making them attractive candidates for advanced applications such as magnetic hyperthermia for cancer therapy, magnetic separation processes in environmental remediation, and targeted drug delivery systems [10]. Beyond their magnetic profile, CrFe₂O₄ nanoparticles demonstrate notable catalytic activity, establishing their importance in various chemical transformation processes and pollutant degradation [11-16]. However, a persistent challenge with standalone ferrite nanoparticles is their tendency to agglomerate due to strong magnetic dipole-dipole interactions and high surface energy, which can compromise their surface area, dispersibility, and overall functional performance in practical applications.

To overcome these limitations and unlock enhanced functionality, the strategic formation of nanocomposites by integrating ferrites with carbonaceous supports has emerged as a powerful strategy. Among these, graphene oxide (GO) has proven to be an exceptionally promising matrix. GO sheets offer a unique combination of a high surface area, excellent mechanical strength, and a rich surface chemistry populated with oxygen-containing functional groups (e.g., hydroxyl, epoxy, carboxyl). These groups not only facilitate good dispersibility in aqueous solutions but also provide abundant active sites for the anchoring of metal oxide nanoparticles. The modification of CrFe₂O₄ through incorporation with GO is therefore pursued to create a more efficacious hybrid material. The anticipated synergy between the two components is multifaceted: the GO sheets can act as a conductive scaffold to mitigate nanoparticle agglomeration, enhance electron transport, and provide additional active sites, while the ferrite nanoparticles impart magnetic responsiveness and catalytic activity. This synergistic effect is projected to significantly enhance the electronic and interfacial properties of the composite, leading to superior performance in applications ranging from dye adsorption and photocatalytic degradation to magnetoelectric sensing and energy conversion devices [6, 12].

The synthesis pathway plays an equally crucial role in determining the final properties of such nanocomposites. The sol-gel auto-combustion method stands out as a particularly advantageous technique for fabricating nano-crystalline ferrites. This route offers exceptional control over stoichiometry, promotes homogeneous mixing of precursors at the molecular level, and employs a self-sustaining exothermic reaction that yields finely crystallized powders with high purity, all while being relatively low-cost and energy-efficient.

The novelty of the present work lies in the tailored application of this sol-gel auto-combustion synthesis to meticulously engineer a CrFe₂O₄/GO nanocomposite with a specific compositional ratio (10 wt.% GO), followed by a comprehensive multi-technique characterization paradigm to unravel the interconnected effects of GO incorporation on the structural integrity, morphological evolution, and magnetic behavior of the spinel system. While previous studies have explored ferrite-GO composites, detailed investigations correlating the sol-gel synthesis parameters with the resulting microstructural uniformity, crystallographic details, and the consequent enhancement in magnetic properties for the specific CrFe₂O₄/GO system remain less explored. This study aims to fill that gap by systematically delineating the synthesis, thorough characterization, and property evaluation of a CrFe₂O₄ nanocomposite integrated with graphene oxide, thereby providing foundational insights for its potential application in advanced technological fields.

2. EXPERIMENTAL DETAILS

2.1. Materials

All chemicals employed in this synthesis were of analytical reagent (AR) grade and were used without additional purification to ensure consistency and reproducibility. The metal precursors for the ferrite phase were chromium(III) nitrate nonahydrate [Cr(NO₃)₃·9H₂O, 99% purity, Sigma-Aldrich] and ferric nitrate nonahydrate [Fe(NO₃)₃·9H₂O, 99% purity, Merck]. Citric acid monohydrate (C₆H₈O₇·H₂O) served the dual role of a chelating agent and a fuel for the subsequent combustion reaction. Aqueous ammonia (NH₄OH, 25%) was used for pH adjustment. Graphene oxide (GO) powder, synthesized via a modified Hummers' method, was utilized as the carbonaceous support matrix. Deionized water (resistivity > 18 MΩ·cm) was used as the solvent throughout the synthesis to minimize ionic interference.

2.2. Synthesis of CrFe₂O₄/GO Nanocomposite via Sol-Gel Auto-Combustion

The CrFe₂O₄/GO nanocomposite was fabricated using a sol-gel auto-combustion technique, a method favored for its ability to yield homogeneous, nano-crystalline oxide powders at relatively low temperatures. The synthesis commenced with the preparation of a clear metal nitrate

solution. Stoichiometric quantities of chromium and ferric nitrates, corresponding to a molar ratio of $\text{Fe}:\text{Cr} = 2:1$, were dissolved in deionized water under continuous magnetic stirring at ambient temperature. To this solution, citric acid was introduced as a complexing agent. The molar ratio of total metal cations to citric acid was maintained at 1:3, providing an optimal fuel-to-oxidizer balance for the auto-ignition process [16]. The pH of the resulting mixture was then meticulously adjusted to approximately 7.0 using dropwise addition of dilute aqueous ammonia. This neutral pH is critical for promoting the complete complexation of metal ions with citrate ligands, leading to the formation of a homogeneous polymeric network and preventing premature precipitation of metal hydroxides.

Concurrently, a separate dispersion of graphene oxide was prepared by ultrasonication of 1.0 g of GO powder in 30 mL of deionized water for 45 minutes. This prolonged sonication ensured the effective exfoliation of GO sheets and yielded a stable, colloidal suspension. This GO suspension was then introduced dropwise into the vigorously stirring metal-citrate sol. The combined mixture was maintained at $80 \pm 5^\circ\text{C}$ with constant stirring for one hour. This step is crucial for facilitating the intimate interaction and adsorption of metal-citrate complexes onto the functionalized surfaces of the GO sheets via electrostatic and coordination interactions.

The temperature was then gradually raised to between $80\text{--}90^\circ\text{C}$ to drive off excess water, leading to the formation of a viscous, resin-like gel. Upon further heating, the gel underwent a self-propagating exothermic combustion reaction, ignited spontaneously due to the redox reaction

between the nitrate oxidizers and the citrate fuel. This rapid auto-combustion event yielded a voluminous, fluffy precursor powder. This as-combusted powder was lightly ground using an agate mortar and pestle and subsequently subjected to calcination. The calcination was performed in a static air atmosphere inside a muffle furnace at 650°C for 3 hours. This high-temperature treatment served two essential purposes: first, to completely eliminate any residual organic components from the combustion; and second, to induce the crystallization of the desired cubic spinel CrFe_2O_4 phase and ensure its firm anchoring onto the thermally reduced GO framework. The final product was a fine black powder, identified as the $\text{CrFe}_2\text{O}_4/\text{GO}$ nanocomposite, which was stored in airtight glass vials to prevent moisture adsorption and oxidation. Figure 1 shows the synthesis flowchart for the prepared $\text{CrFe}_2\text{O}_4/\text{GO}$ nanocomposite.

2.3. Characterization Techniques

The structural and phase purity of the synthesized nanocomposite was investigated using X-ray diffraction (XRD). Patterns were recorded at room temperature on a Rigaku Micromax-007HF diffractometer operated with $\text{Cu K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). Data were collected in the 2θ range of 20° to 80° with a continuous scan speed of 3° per minute and a step size of 0.02° . Chemical bonding and functional group analysis were conducted using Fourier-transform infrared (FTIR) spectroscopy in the attenuated total reflectance (ATR) mode across a wavenumber range of $400\text{--}4000 \text{ cm}^{-1}$.

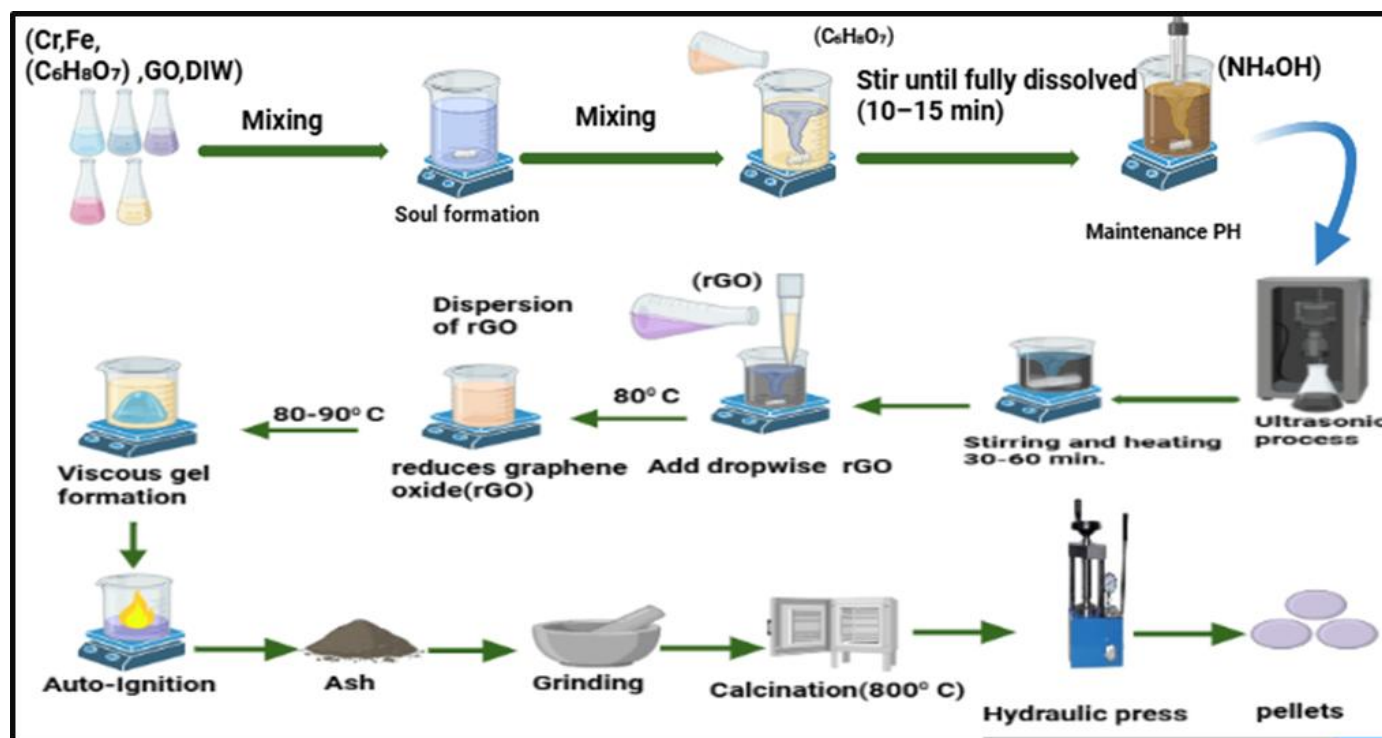


Fig. 1. Schematic representation of synthesis flowchart of $\text{Cr}_x\text{Fe}_2\text{O}_4/10\% \text{GO}$.

Table 1. Average Crystallite size and physical parameters obtained from XRD data.

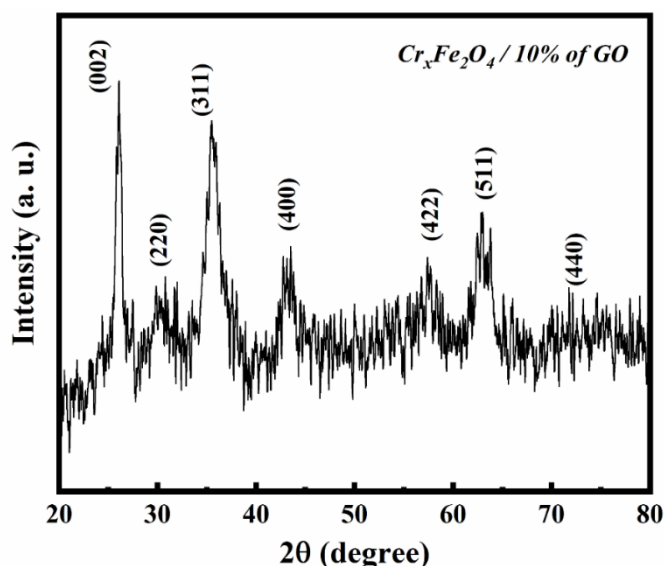
Content (x)	Lattice constant, 'a' (Å)	Average crystallite Size 'D' (nm)	X-ray Density 'dx' (g/cm ³)	Cell volume 'a ³ ' (Å) ³	d-spacing (nm)	Hopping Length	
						J _{Tet}	J _{Oct}
CrFe ₂ O ₄	8.50	31.61	4.90	614.12	2.52	3.73	2.87

The morphological features, surface topography, and elemental distribution were examined using field emission scanning electron microscopy (FESEM) on a Zeiss Gemini 300 instrument. For higher-resolution microstructural analysis, including lattice fringes and precise particle size measurement, high-resolution transmission electron microscopy (HRTEM) was employed. Raman spectroscopic studies were carried out using a Horiba Jobin Yvon LabRam HR800 spectrometer equipped with a 633 nm He-Ne laser excitation source to probe the graphitic structure of GO and its interaction with the ferrite. The magnetic properties at room temperature were quantified using a Lakeshore 7400 series vibrating sample magnetometer (VSM) with an applied field sweep of ± 15 kOe.

3. RESULTS AND DISCUSSION

3.1. X-ray Diffraction

XRD spectra of the as-prepared ferrites and composites are depicted in Figure 2. The diffraction planes (220), (311), (400), (422), (511) and (440) indicate the spinel matrix with the addition of the GO plane (002) at 23° [3, 13]. The appearance of a large peak in the diffraction plane (311) towards a greater diffraction angle in the Cobalt ferrite sample, due to the substitution of Cr³⁺ into the cobalt ferrite sample [14].

**Fig. 2.** XRD pattern of Cr_xFe₂O₄/GO nanocomposites.

The lattice parameter is given in Table 1. The lattice constant (a), interplanar distance (d) and unit volume (V) were determined. Interplanar spacing values were reduced by substituting the Cr²⁺ ion into the cobalt ferrite/GO matrix. Moreover, with the doping of Cr²⁺ and NPs in the cobalt ferrite spinel lattice, the interplanar spacing value is 2.52 nm [2].

The unit cell volume and lattice constant have a similar trend to interplanar spacing. For phase identification and structural parameter calculations, powder X-ray diffraction was performed over the 2θ range of 20° to 80°. The patterns belong to corresponding peaks without any impurity. The peaks are sharp and narrower, implying the moderate crystalline nature of the synthesized materials. The peaks shift towards the higher value of 2θ as the concentration of Cr³⁺ rises, hence corroborating a decreasing lattice parameter. The lattice parameter has been calculated using Bragg's law, which is found in the range of 8.50 Å.

The crystallite sizes are also calculated using the maximum peak from the XRD pattern (311) and by employing a formula [15].

$$D = \frac{0.94 \lambda}{\beta \cos \theta} \quad (1)$$

Where λ is the X-ray wavelength of Cu K α = 0.154 nm, θ is the Bragg's angle, β is the full width of the diffraction line at half maximum, and D is the crystallite size (nm). The average crystallite sizes of the synthesized materials were found to be 31.61 nm.

All the structural parameters are calculated and summarized in Table 1. To determine the atoms in each sample, the X-ray density (dx) of the unit cell complex was computed using the following formula [16].

$$dx = \frac{Z \times M}{N_A \times V} \quad (2)$$

where Z represents the number of atoms present in the cubic system i. e. 8, and for a tetragonal system, which is 4, M is the molar mass, N_A is Avagadro's number $6.022 \times 10^{23} \text{ mol}^{-1}$, and V is the volume of the unit cell.

In composites of cobalt ferrite and graphene-derived materials, the fundamental cubic spinel structure of CoFe₂O₄ is generally preserved, and the ferrite nanoparticles are observed to be anchored on the carbonaceous sheets rather than incorporated into the spinel lattice [17]. Structural investigations reported for CoFe₂O₄/graphene oxide systems indicate that the characteristic spinel diffraction features remain intact upon graphene oxide addition, suggesting that

the carbonaceous component does not participate in the crystallographic framework of the ferrite [18].

3.2. FTIR Spectroscopy

Figure 3 shows the FTIR spectrum of CrFe₂O₄. These spectra provide information about the vibration states in these compounds. The FTIR spectrum of the CrFe₂O₄ spinel contains distinctive bands corresponding to the chemical bonds in this compound. In spinel ferrite nanoparticles, the stretching vibrations of metal oxygen bonds typically exhibit strong absorption bands in the range of 400 to 1000 cm⁻¹ [11]. The vibrations in the 3400 cm⁻¹ region would indicate the presence of O–H stretching from water molecules or hydroxyl groups in the sample. Additionally, bands around 1600 cm⁻¹ may be associated with bending vibrations of the metal-oxygen metal bonds. The precise position and intensity of the absorption bands depend on factors such as sample purity, crystalline structure, and synthesis conditions [19]. FTIR analysis of GO typically shows several key features. A broad peak around 3300–3600 cm⁻¹ indicates the presence of hydroxyl groups, while peaks near 1700 cm⁻¹ are associated with carbonyl (C=O) groups. Additionally, peaks around 1000–1300 cm⁻¹ relate to epoxy groups [20].

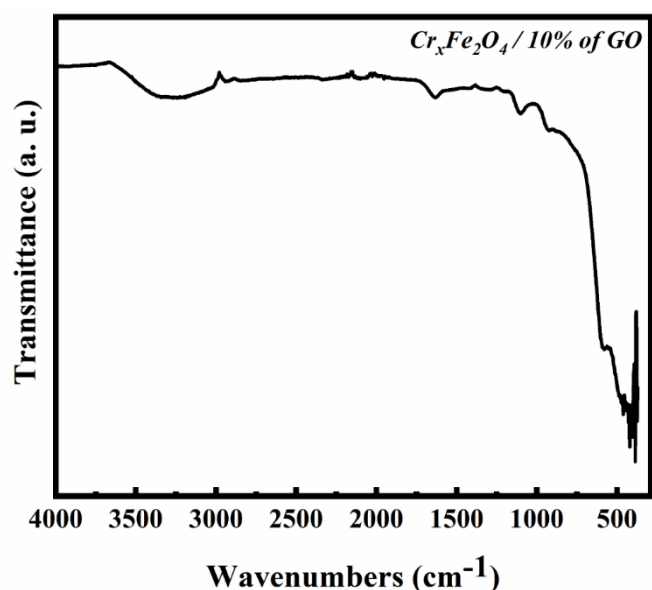


Fig. 3. FT-IR spectra of Cr_xFe₂O₄/10% graphene.

Figure 3 shows the FT-IR spectrum of CrFe₂O₄; this spectrum indicates the presence of bands that confirm the existence of the CrFe₂O₄ spinel and GO. The band around 2900 cm⁻¹ can be assigned to C–H stretching, and the successful incorporation of graphene oxide into CrFe₂O₄ composite material. The FT-IR spectrum of the CrFe₂O₄/GO nanocomposite can provide information about the chemical composition and interactions within the composite. In addition to the functional groups mentioned earlier, the vibrations related to the incorporated GO are also observed.

Table 2. The band positions in the infrared absorption spectra.

<i>x</i> content	ν_{1s1} (cm ⁻¹)	ν_{1s2} (cm ⁻¹)	ν_2 (cm ⁻¹)	ν_{2s1} (cm ⁻¹)
<i>x</i> = 1	580.71	432.15	430.81	398.52

3.3. Morphological study by using FESEM and HRTEM

Field Emission Scanning Electron Microscopy (FESEM) was employed to investigate the microstructural features, including surface morphology and particle size distribution of the synthesized Cr_xFe₂O₄/10% graphene. The FESEM micrographs reveal the soft agglomeration of well-defined, crystalline nanoparticles with a predominantly spherical morphology. The images, captured at different magnifications, clearly demonstrate that the cobalt/chromium ferrite nanoparticles are unevenly distributed and tend to form round-shaped particles [9, 13, 21]. Furthermore, the successful fabrication of the ternary nanocomposite was confirmed by FESEM, which showed that the nanoparticles are efficiently anchored to the graphene oxide (GO) sheets [22, 23]. This intimate interfacial contact between ferrite nanoparticles and GO nanosheets enhances the structural durability and stability of the synthesised nanocomposite [24]. Figure 4 presents FE-SEM micrographs of the synthesized material recorded at different magnifications. Figure 4(a), acquired at higher magnification (100k×, 200 nm scale bar), reveals fine nanoscale features with densely packed particulates and pronounced surface roughness. Figure 4(b), recorded at lower magnification (40k×, 1 μm scale bar), provides an overall view of the surface morphology, showing relatively uniform coverage and agglomerated regions distributed across the matrix.

3.4. High-resolution transmission electron microscopy (HRTEM) analysis

HRTEM analysis is a very powerful technique for examining the structural and morphological characteristics of materials at the nanoscale. The resulting images of CrFe₂O₄/GO facilitate visualization of individual nanoparticles, providing valuable insights into their shape and arrangement. CrFe₂O₄ nanoparticles appear as dark spots in the TEM images due to their high electron density, while graphene oxide sheets are represented as thin, transparent layers. The average grain size of the TEM images evaluated from ImageJ software by measuring their diameter, and the average grain size of the nanocomposite was found the 11.14 nm range. Where every suitable for magnetic devices, where they can easily be magnetised by giving an external magnetic field to them [25, 26]. TEM micrograph of the synthesized material is presented as Figure 5 (a). The TEM image was instrumental in providing insight into its internal microstructure and particle morphology at the nanoscale. The image revealed well-defined nanosized particles with relatively uniform contrast, confirming the formation of a nanocrystalline

structure. The particles appear closely packed with partial agglomeration, which can be attributed to high surface energy and interparticle interactions typical of nanoscale systems. Clear particle boundaries and consistent size distribution observed in the micrographs further indicate good structural integrity of the synthesized phase. Figure 5(b) illustrates the particle size distribution, revealing a relatively narrow distribution centered in the nanometer range, which indicates good control over particle growth during synthesis. The uniformity in particle distribution and limited agglomeration further suggest homogeneous nucleation and stable nanostructure formation.

3.5. Raman Spectroscopy

The Raman spectrum of the $\text{CrFe}_2\text{O}_4/\text{GO}$ nanocomposite

scan provides valuable insights into the vibration states and structural properties of the material. Raman spectroscopy is a powerful technique that measures the inelastic scattering of light, allowing for the identification of molecular and crystalline structures. Figure 6 shows the Raman spectra of the $\text{CrFe}_2\text{O}_4/\text{GO}$ nanocomposite. Graphene Oxide has a distinct Raman signature due to its unique structural characteristics [27]. The D band, observed in the range of 1300 to 1350 cm^{-1} , is attributed to defects and disorders in the graphene network, associated with the breathing modes of sp^3 carbon atoms [28, 29]. The G band, appearing in the range of 1580–1600 cm^{-1} , represents the in-plane stretching of sp^2 carbon atoms on the graphene network, a distinct feature of GO materials [30]. The 2D band, observed in the range of 2600–2800 cm^{-1} , provides information about the density of graphene layers [31].

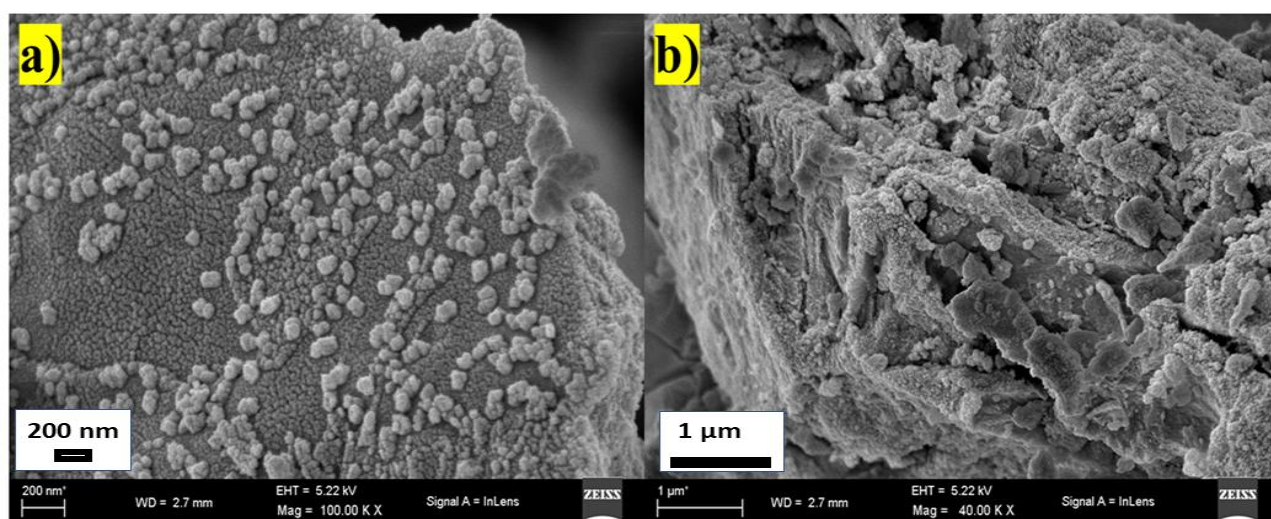


Fig. 4. FESEM micrographs of $\text{Cr}_x\text{Fe}_2\text{O}_4/10\%$ graphene.

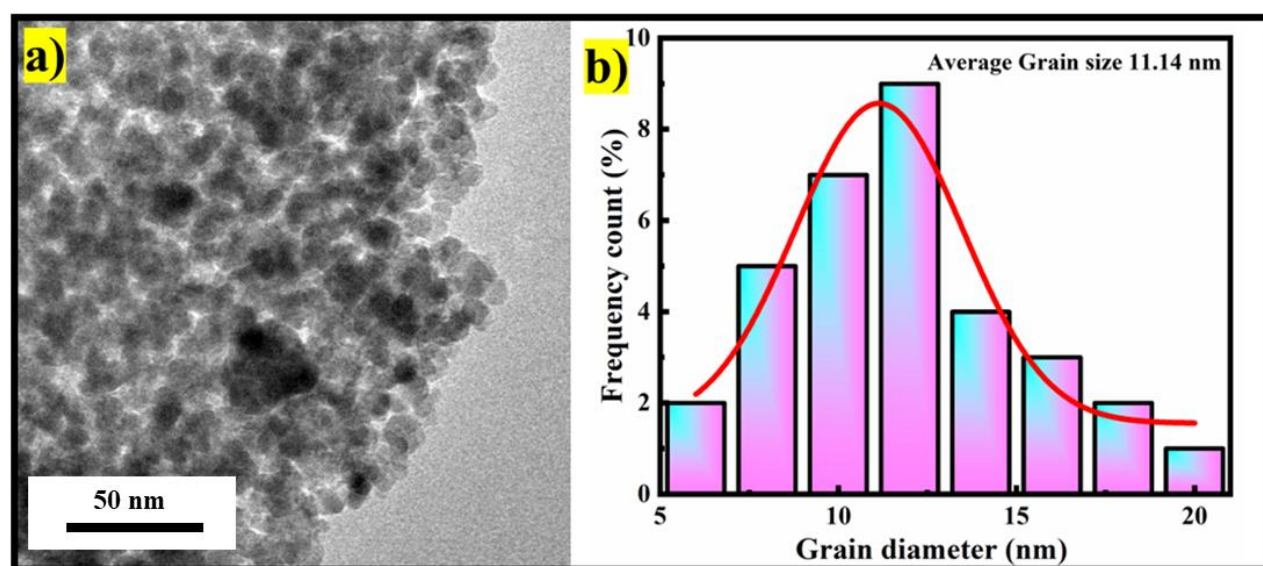
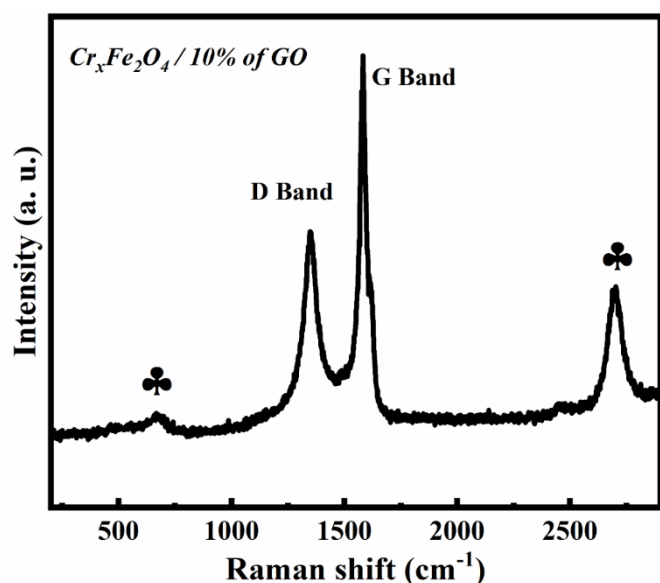
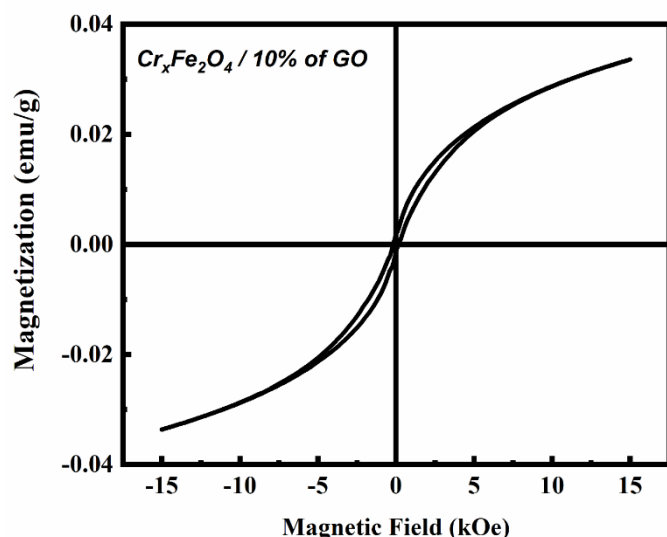


Fig. 5. (a) TEM micrographs and (b) particle size histogram.

Table 3. Magnetic Parameters calculated from VSM.

Sample Name	M _s (emu/g)	M _r (emu/g)	H _c (Oe)	K x 10 ³ (erg/cm ³)	n _B	R = M _r /M _s
Cr _x Fe ₂ O ₄ /10%	14.67	1.19	310.10	4.87	0.67	0.081

**Fig. 6.** RAMAN spectra of Cr_xFe₂O₄/10% graphene.**Fig. 7.** VSM spectra of Cr_xFe₂O₄/10% graphene.

3.6. Vibrating Sample Magnetometry

Spinel nano ferrites made up of cobalt are a well-known composition with promising magnetic properties shown in Figure 7. The level of magnetic response can be revealed by

the distribution in the binary interstice and the nature of the cations. Strong magnetic characteristics are caused due to the tetrahedral occupancy of Fe³⁺ cations with ferro and antiferromagnetic ordering, and a double exchange between Fe³⁺ and Co²⁺, respectively [26, 32–34]. To investigate the room temperature magnetic behaviours of the synthesized materials, the vibrating sample magnetometry has been utilized in the range of magnetic field 15 kOe. The magnetic parameters, such as the saturation magnetization, remanent magnetization, coercivity, anisotropy constant, Bohr magneton and squareness ratio were calculated and tabulated in Table 3. The hysteresis loop is shown in Figure 7. The calculated values of the saturation magnetization show that it decreases with increases in Cr²⁺ content, but such is not the case for the other parameters.

4. CONCLUSION

In this work, a chromium ferrite-graphene oxide nanocomposite was successfully synthesized via a sol-gel auto-combustion route and subjected to comprehensive structural, morphological, and magnetic characterization. The integration of graphene oxide into the CrFe₂O₄ matrix significantly improved the material's structural coherence and morphological uniformity, as validated by X-ray diffraction, electron microscopy, and spectroscopic analyses. The formation of a well-defined cubic spinel phase, coupled with the homogeneous distribution of ferrite nanoparticles on GO sheets, facilitated enhanced interfacial interactions and charge transport pathways. Magnetic measurements revealed notable magnetic parameters, including substantial saturation magnetization and coercivity, which are indicative of strong dipole coupling and effective domain wall pinning within the nanocomposite. These magnetic enhancements are directly linked to the tailored nano-architecture and the synergistic effect between the conductive GO network and the ferrimagnetic CrFe₂O₄ particles. The methodological approach adopted in this study not only ensures reproducible nanocomposite fabrication but also highlights the critical role of hybrid design in optimizing functional properties. The resulting CrFe₂O₄/GO system exhibits considerable promise for applications in energy conversion technologies, magnetic recording media, and electromagnetic shielding, where materials with tunable magnetic responses and structural stability are essential. Future work may focus on further optimizing the GO loading, investigating thermal and electrical properties, and exploring photocatalytic or sensing

functionalities to broaden the application spectrum of this versatile nanocomposite.

DECLARATIONS

Ethical Approval

We affirm that this manuscript is an original work, has not been previously published, and is not currently under consideration for publication in any other journal or conference proceedings. All authors have reviewed and approved the manuscript, and the order of authorship has been mutually agreed upon.

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Availability of data and material

All of the data obtained or analyzed during this study is included in this paper.

Conflicts of Interest

The authors declare that they have no financial or personal interests that could have influenced the research and findings presented in this paper. The authors alone are responsible for the content and writing of this article.

Author's contribution statement

Authors contributed equally to this work.

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