

Artículo original

Sol-gel auto-combustion synthesis of CoFe_2O_4 spinel nanoparticles with ferrimagnetic and semiconducting properties

Síntesis mediante autocombustión asistida por sol-gel de nanopartículas de espinela CoFe_2O_4 con propiedades ferrimagnéticas y semiconductoras

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Abstract

Cobalt ferrite (CoFe_2O_4) is a multifunctional spinel oxide whose structural, optical, and magnetic properties are strongly influenced by the synthesis route and thermal treatment. Establishing the relationship between the synthesis conditions and the resulting structural, compositional, optical, and magnetic characteristics is essential for assessing the properties of the material obtained by sol-gel auto-combustion. Here, we synthesized CoFe_2O_4 powder by a urea-assisted sol-gel auto-combustion route and systematically characterized it to determine its crystal structure, chemical composition, optical response, and magnetic behavior. The precursors were dissolved in distilled water and heated progressively to 420 °C to promote spontaneous combustion; the resulting powder was subsequently calcined at 700 °C for 3 h. X-ray diffraction followed by Rietveld refinement confirmed the formation of a single-phase cubic inverse-spinel structure with space group Fd-3m and a lattice parameter of 8.3801 Å. SEM observations revealed fine, predominantly irregular to sub-spherical particles with moderate agglomeration; particle dimensions were estimated to be on the order of 100 nm, while EDS analysis showed a composition close to the nominal CoFe_2O_4 stoichiometry. Diffuse-reflectance measurements and Kubelka–Munk analysis yielded a direct optical band gap of 1.45 ± 0.05 eV. Room-temperature magnetization measurements up to 15 kOe revealed ferrimagnetic behavior, with a magnetization approaching 50 emu g^{-1} at the maximum applied field. These results establish the structural, compositional, optical, and magnetic characteristics of CoFe_2O_4 produced by the selected sol-gel auto-combustion route and provide a basis for further studies of its functional properties.

Principio del formulario

Keywords: Cobalt-ferrite spinel; fuel-grade urea; crystallographic features; optical properties; magnetic features.

Resumen

La ferrita de cobalto (CoFe_2O_4) es un óxido de tipo espinela multifuncional cuyas propiedades estructurales, ópticas y magnéticas dependen claramente de la ruta de síntesis y el tratamiento térmico que se empleen. En este contexto es fundamental establecer la relación entre las condiciones de síntesis y las características estructurales, composicionales, ópticas y magnéticas del material obtenido para evaluar sus propiedades. En este estudio, se sintetizó polvo de CoFe_2O_4 utilizando una ruta de autocombustión asistida por sol-gel y urea como combustible, y se caracterizaron de manera sistemática su estructura cristalina, composición química, respuesta óptica y comportamiento magnético. Los precursores se disolvieron en agua destilada, se calentaron progresivamente hasta los 420 °C para promover la autocombustión espontánea, y el polvo obtenido fue posteriormente calcinado a 700 °C durante 3 h. La difracción de rayos X complementada con el refinamiento

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de Rietveld confirmó la formación de una estructura de espinela inversa cúbica monofásica perteneciente al grupo espacial Fd-3m, con un parámetro de red de 8.3801 Å. Las observaciones con SEM revelaron partículas finas, predominantemente irregulares a subesféricas, con aglomeración moderada y dimensiones estimadas del orden de 100 nm, mientras que el análisis EDS mostró una composición próxima a la estequiometría nominal de CoFe_2O_4 . Las mediciones de reflectancia difusa y el análisis de Kubelka–Munk permitieron determinar una banda prohibida óptica directa de 1.45 ± 0.05 eV. Las mediciones de magnetización a temperatura ambiente, realizadas hasta 15 kOe, revelaron un comportamiento ferrimagnético, con una magnetización cercana a 50 emu g^{-1} en el campo máximo aplicado. Estos resultados establecen las características estructurales, composicionales, ópticas y magnéticas del CoFe_2O_4 obtenido mediante la ruta de autocombustión asistida por sol-gel y proporcionan una base para el estudio posterior de sus propiedades funcionales.

Palabras clave: espinela de ferrita de cobalto; combustible a base de urea; características cristalográficas; propiedades ópticas; naturaleza magnética.

Introduction

Spinel-type ferrimagnetic materials based on CoFe_2O_4 (cobalt ferrite) have a strategic position in functional materials science because they combine structural stability, robust magnetic response, and remarkable chemical versatility. From a crystallographic perspective, CoFe_2O_4 crystallizes in an inverse spinel structure where Co^{2+} and Fe^{3+} cations are distributed among tetrahedral and octahedral sites in a non-random manner. This cation distribution determines the Fe–O–Co superexchange coupling, which is responsible for its ferrimagnetic behavior, high magnetocrystalline anisotropy, and coercivity values significantly higher than those observed in other soft ferrites. These properties make CoFe_2O_4 an ideal model for investigating correlations among electronic structure, magnetic ordering, and crystalline defects in complex oxides. Furthermore, the chemical and thermal stability of this phase enables the preservation of these properties under demanding processing conditions, representing a significant advantage over other magnetic nanomaterials (Tran *et al.*, 2024). The scientific relevance of CoFe_2O_4 also stems from its classification within a family of rare-earth-free materials exhibiting competitive magnetostrictive properties. This has stimulated its study in applications where expensive or chemically less stable alloys were traditionally used. Cobalt ferrite can exhibit high magnetostriction and controllable magnetic losses, making it attractive for actuators, magnetoelectric transducers, strain sensors, and energy conversion devices. At the nanoscale, these properties become even more interesting, since surface anisotropy, reduced dimensionality, and the presence of disordered surface spins can modify the magnetic energy barrier and induce transitions between ferrimagnetic and superparamagnetic regimes. This controllable transition is one of the most intensively studied phenomena in the physics of magnetic nanoparticles (Parhizkar *et al.*, 2019).

In nanoparticle form, CoFe_2O_4 exhibits a range of functionalities that extend beyond fundamental physics and project into multiple emerging technologies. In biomedicine, cobalt ferrite nanoparticles have shown strong potential in magnetic hyperthermia, where heat generated under alternating magnetic fields can be employed for localized tumor treatment. Their magnetic susceptibility also enables applications in targeted drug delivery, cell separation, and contrast enhancement in biomedical imaging. The possibility of surface functionalization with polymers, silica, bioactive molecules, or core-shell structures further expands their biocompatibility and selectivity. Recent reviews indicate that size control below 20 nm and surface engineering are decisive factors for minimizing aggregation and cytotoxicity. Current analyses of therapeutic applications confirm that CoFe_2O_4 nanoparticles are among the most promising platforms in magnetic nanomedicine (Arora *et al.*, 2025).

In the environmental field, nanoparticulate CoFe_2O_4 has demonstrated high potential in photocatalysis, pollutant adsorption, and Fenton-like processes. Its main advantage lies in the possibility of recovering the catalyst using an external magnetic field, thereby reducing separation costs and improving material reusability. Moreover, when these nanoparticles

are combined with semiconductor oxides, reduced graphene oxide, or carbon-based materials, heterostructures with improved charge separation and enhanced photocatalytic efficiency can be obtained. Applications in wastewater treatment include the removal of dyes, antibiotics, and heavy metals. Recent studies on green synthesis of CoFe₂O₄ have shown that morphology and surface chemistry directly influence catalytic performance (Tran *et al.*, 2024).

In the field of electromagnetic devices, nanoparticulate CoFe₂O₄ has also demonstrated applicability in microwave absorbers, electromagnetic shielding, and spintronic systems. Its high magnetic anisotropy allows the tuning of dielectric and magnetic losses in the GHz frequency range, especially when incorporated into polymer matrices or hybrid nanocomposites. Integration with soft magnetic materials enables the design of exchange-coupled nanoarchitectures with adjustable coercivity and magnetization, which are of particular interest for magnetic storage and magnetoelectronic applications (Nuñez *et al.*, 2022).

However, the functional performance of CoFe₂O₄ depends critically on the synthesis technique employed. Co-precipitation is one of the most commonly used routes due to its simplicity and industrial scalability, although it often produces broader particle size distributions and some degree of compositional heterogeneity. In contrast, hydrothermal methods provide better control over crystallinity and anisotropic growth, favoring particles with more controlled defect structures. Recent *in situ* studies on the hydrothermal nucleation of spinel ferrites have shown that CoFe₂O₄ can crystallize rapidly into domains of approximately 10–12 nm, partially limiting size control based solely on reaction time (Andersen *et al.*, 2024).

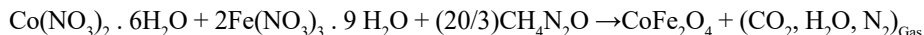
On the other hand, sol-gel synthesis offers important advantages in chemical homogeneity and stoichiometric control, making it particularly useful for fundamental studies of cation inversion and chemical substitution. For electronic and biomedical applications, thermal decomposition remains one of the preferred techniques, as it allows the production of highly monodisperse nanoparticles with excellent morphological control. Experimental studies have demonstrated the direct synthesis of CoFe₂O₄ with highly reproducible magnetic properties through controlled combustion methods (Cao & Zuo, 2020). In the sol-gel autocombustion route, the desired metallic ions are complexed with an organic agent (fuel), forming a solution and subsequently a gel that, when heated, undergoes self-sustained combustion. The quality of the resulting powder (phase purity, particle size, degree of agglomeration, and surface area) depends largely on parameters such as the type of fuel, the fuel-to-metal nitrate ratio, the pH of the solution, and the calcination temperature (Liu *et al.*, 2000). The amount of heat released during combustion and the temperature reached by the flame have a decisive influence on the final characteristics of the material, since different fuels exhibit distinct complexation capacities and redox properties, besides generating different volumes of gases. Therefore, the choice of fuel constitutes a key parameter for controlling the final product (Chauhan *et al.*, 2016).

Variables such as the pH, the calcination temperature, the nature of the complexing agent, the heating rate, and the aging time directly affect crystallite size, cation inversion, saturation magnetization, and coercivity. Consequently, two samples with the same chemical formula may exhibit radically different magnetic responses if they are prepared through different synthetic routes. This sensitivity to synthesis makes CoFe₂O₄ an ideal system for the rational engineering of properties and the design of multifunctional nanomaterials. Overall, the scientific importance of CoFe₂O₄ lies not only in its intrinsic properties as a spinel ferrite, but also in its extraordinary capacity to modulate its physical response through synthetic control at the nanoscale. This combination of fundamental physics, processability, and technological potential continues to position this material among the most relevant systems in contemporary research on nanomagnetism, advanced electronics, biomedicine, and environmental remediation (Duong *et al.*, 2021).

Materials and methods

Experimental procedures

The nanocrystalline cobalt ferrite with spinel structure (cobalt ferrite) was synthesized through the sol-gel auto-combustion method using urea as fuel. The synthesis procedure was designed to obtain approximately 1 g of cobalt ferrite starting from hydrated metal nitrates. The overall chemical balance for the process is given by the reaction used to calculate the required masses of the metal precursors and fuel:



During the first experimental session, all reagents were weighed using a high-precision analytical balance with an uncertainty of ± 0.0001 g. The amounts required to synthesize approximately 1 g of CoFe_2O_4 were calculated according to the stoichiometric reaction. The iron(III) nitrate, cobalt(II) nitrate, and urea were transferred into a precipitation beaker containing 40 mL of distilled water. A magnetic stirrer was introduced to ensure continuous mixing and complete dissolution of the reagents, producing a homogeneous solution. The beaker was then placed on a hot plate equipped with temperature and stirring-speed control. The mixture was maintained at 40 Hz and 90 °C for 30 min.

Subsequently, the temperature was increased to 120 °C and maintained for an additional 30 min without changing the stirring frequency. After this stage, the temperature was progressively raised to 170 °C for 15 min, followed by 200 °C for another 15 min. Finally, the temperature was carefully increased to 420 °C until spontaneous auto-combustion occurred, leading to the formation of the ferrite precursor. After completion of the combustion process, the beaker was removed from the heating plate and allowed to cool naturally to room temperature. The resulting solid was carefully collected and transferred to an agate mortar, where it was ground until a fine powder was obtained. The final recovered mass after synthesis was 0.9017 ± 0.0001 g. To eliminate residual impurities and improve crystallinity, the powder was subjected to a two-step calcination treatment in a tubular furnace at 700 °C for 3 h.

We analyzed the structural properties and phase constitution of the synthesized samples by X-ray diffraction (XRD). The diffraction patterns were recorded using a PANalytical X'Pert-Pro diffractometer operating in Bragg-Brentano configuration with $\text{CuK}\alpha$ radiation ($\lambda = 1.540598$ Å). The experimental data were collected with a 2θ step size of 0.01° and an acquisition time of 10 s per step. Subsequently, the patterns were processed through Rietveld refinement using the GSAS software (Toby & Von Dreele, 2013), allowing the determination of lattice parameters, identification of the predominant crystalline phase, and evaluation of the influence of iron incorporation on the crystal structure. The surface morphology of the samples was examined using a dual-beam focused ion beam/scanning electron microscope (SEM, Vega 3 TESCAN), employing both secondary electron and backscattered electron imaging modes. Elemental composition was assessed by energy-dispersive X-ray spectroscopy (EDS) through an X-ray detector integrated into the microscope. Additionally, the optical bandgap was experimentally determined using a VARIAN Cary 5000 spectrophotometer over the wavelength range from 200 to 2500 nm. **Figure 1** outlines the entire nanoparticle production process and the characterization techniques used. We carried out room-temperature magnetization curves as a function of applied field up to 15 kOe using a PPMS Quantum Design equipment in the Vibrating Sample Magnetometer (VSM) mode.

Results

Structural analysis

The refined X-ray diffraction pattern shown in **Figure 2** confirms the formation of a single crystalline phase corresponding to cobalt ferrite (CoFe_2O_4) with an inverse cubic spinel structure and Fd-3m space group. The excellent agreement between the experimental data



Figure 1. Graphical scheme of the synthesis process and the characterization of the CoFe₂O₄ nanoparticles

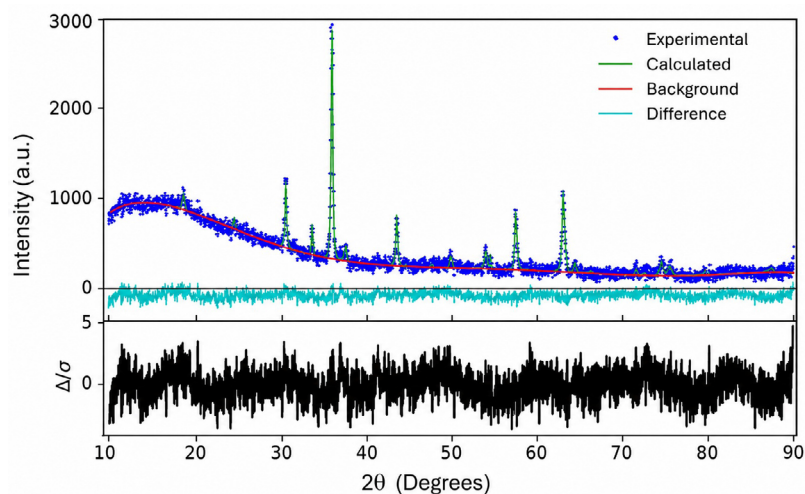


Figure 2. Refined diffraction pattern for the CoFe₂O₄ spinel

(blue dots) and the calculated profile (green line) demonstrates the successful convergence of the Rietveld refinement. Furthermore, the difference curve exhibits low-amplitude oscillations around zero across the entire angular range, indicating minimal discrepancy between the structural model and the experimental data. The absence of additional unindexed reflections suggests that the sample contains no detectable secondary phases within the sensitivity limit of the technique.

The diffraction maxima observed at approximately $2\theta \approx 30.1^\circ$, 35.5° , 43.2° , 53.5° , 57.0° , and 62.6° corresponded to the characteristic (220), (311), (400), (422), (511), and (440) reflections, respectively, typical of CoFe_2O_4 with a spinel structure. The high relative intensity of the (311) plane, which constitutes the dominant reflection in the pattern, agrees well with values reported in the literature for highly crystalline cobalt ferrites. The narrow peak widths and the low contribution of instrumental broadening suggest the presence of well-developed crystalline domains and a reduced level of internal microstrain. From the position of the (311) reflection and considering $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$), a lattice parameter of $8.3801 \text{ \AA}$ was calculated from the refinement, a value that is consistent with those reported for stoichiometric CoFe_2O_4 ($8.37\text{--}8.40 \text{ \AA}$) (Arora *et al.*, 2025). A structural quantification derived from the Rietveld analysis is shown in **Table 1**.

Table 1 presents the structural and geometrical parameters obtained for CoFe_2O_4 spinel through Rietveld refinement. The Wyckoff positions assigned to Co^{2+} (8a), Fe^{3+} (16d), and O^{2-} (32e), with coordinates (0.125, 0.125, 0.125), (0.500, 0.500, 0.500), and (0.256, 0.256, 0.256), respectively, are consistent with the crystallographic description of a cubic inverse spinel. In particular, the 32e oxygen position determines the local geometry of the cationic sites and influences the interatomic distances and bond angles that govern interactions between magnetic cations. The Co–Co ($3.6289 \text{ \AA}$), Co–Fe ($3.4744 \text{ \AA}$), Fe–Fe(1) ($2.9630 \text{ \AA}$), and Fe–Fe(2) ($5.1320 \text{ \AA}$) distances indicate different cation–cation separations within the crystal lattice. Likewise, the Co–O–Co (109.5°), Co–O–Fe (125.0° and 153.6°), and Fe–O–Fe (90.3° and 125.3°) angles reveal the diversity of oxygen-mediated interaction pathways. These geometries are particularly relevant to the superexchange mechanisms between tetrahedral and octahedral sites, which contribute to the characteristic ferrimagnetic ordering of CoFe_2O_4 . Overall, the parameters listed in **Table 1** provide a detailed description of the structural connectivity of the spinel and establish a basis for relating its crystallographic configuration to its magnetic response.

These results confirm that the incorporation of Co^{2+} and Fe^{3+} into the crystal lattice occurs without significant modifications to the cubic symmetry or the generation of macroscopic structural distortions. The expected structure is shown in **Figure 3**.

The three-dimensional structural representation shows the characteristic arrangement of an inverse spinel, where oxygen anions form a cubic close-packed framework, and the cations occupy tetrahedral (A) and octahedral (B) interstitial sites. In an ideal inverse spinel, the cation distribution can be expressed as $(\text{Fe}^{3+})_A[\text{Co}^{2+}\text{Fe}^{3+}]\text{BO}_4$, where the brackets indicate octahedral occupancy. This distribution generates two antiparallel magnetic

Table 1. Structural parameters obtained for the CoFe_2O_4 spinel using Rietveld refinement

Atom	Wyckoff	x	y	z
Co^{2+}	8a	0.125	0.125	0.125
Fe^{3+}	16d	0.500	0.500	0.500
O^{2-}	32e	0.256	0.256	0.256
Bond	Distance ($\text{\AA}$)	Bond	Angle ($^\circ$)	
Co–O	1.9064	Co–O–Co	109.5	
Fe–O	3.3909	Co–O–Fe (1)	125.0	
Co–Co	3.6289	Co–O–Fe (2)	153.6	
Co–Fe	3.4744	Fe–O–Fe (1)	90.3	
Fe–Fe (1)	2.9630	Fe–O–Fe (2)	125.3	
Fe–Fe (2)	5.1320			

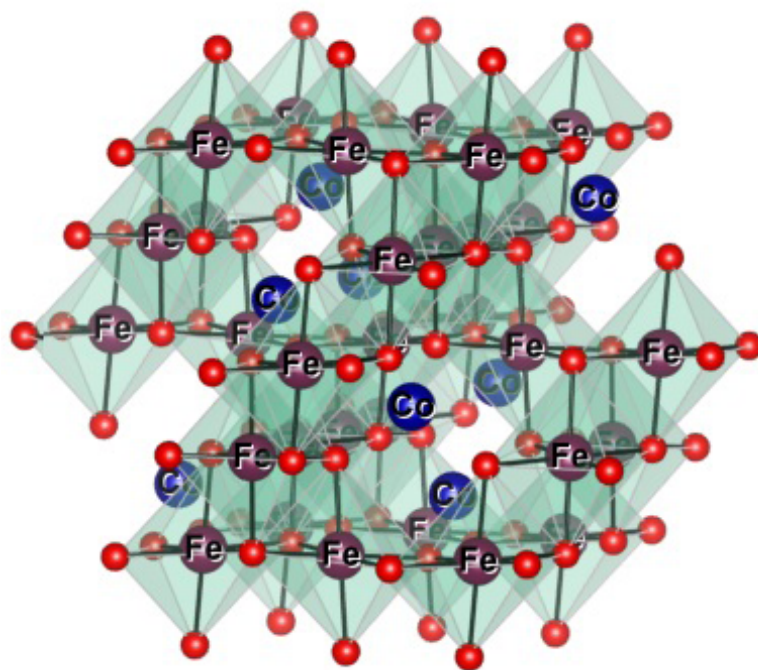


Figure 3. Structural scheme constructed by Vesta for the CoFe₂O₄ spinel from the Rietveld refinement of the XRD experimental data

sublattices responsible for the characteristic ferrimagnetic behavior of cobalt ferrite. From a geometrical point of view, the refined structure allows the inference of important local parameters. Considering a lattice parameter of approximately 8.38 Å and the typical oxygen positional parameter for spinels ($u \approx 0.255$ – 0.260), the average interatomic distances are expected to be on the order of 1.90–1.95 Å for Fe–O bonds in tetrahedral coordination and between 2.00 and 2.10 Å for Co–O and Fe–O bonds in octahedral coordination. These distances are consistent with the ionic radii of Co²⁺ and Fe³⁺ and reflect the stability of the coordination polyhedra present in the lattice.

Bond angles constitute another relevant aspect for understanding the physical properties of the material. In an ideal spinel structure, the tetrahedra exhibit O–Fe–O angles close to 109.5°, characteristic of a regular tetrahedral coordination. On the other hand, the octahedra display O–M–O angles close to 90° and 180° (M = Co or Fe), although small deviations may arise due to local oxygen displacements. These slight distortions modify the Fe³⁺–O²⁻–Fe³⁺ and Fe³⁺–O²⁻–Co²⁺ superexchange interactions, directly influencing the magnetocrystalline anisotropy and coercivity of the material (Barrera *et al.*, 2018).

A particularly useful parameter for evaluating the chemical consistency of the refinement is the bond valence sum (BVS). This method allows the estimation of the effective oxidation state of each cation from the refined bond distances through the expression (Deluque *et al.*, 2023):

$$V_i = \sum_j \exp\left(\frac{R_0 - R_{ij}}{b}\right), \quad (1)$$

where R_{ij} represents the cation–oxygen distance, while R_0 and b are empirical parameters specific to each ionic pair. For a properly refined CoFe₂O₄ structure, BVS values close to +2 for Co and +3 for Fe are expected. Values approaching these numbers indicate that the obtained bond distances are chemically reasonable and that no anomalous occupancies or significant structural disorder are present. Likewise, systematic deviations could reveal cation defects, oxygen vacancies, or partial redistributions between tetrahedral and octahedral sites.

Overall, the Rietveld refinement results and the structural representation demonstrate that the sample corresponds to a single-phase cobalt ferrite with a well-crystallized inverse spinel structure. The obtained lattice parameter, the expected interatomic distances, the geometry of the coordination polyhedra, and the BVS values compatible with the nominal oxidation states support the structural stability of the material. These characteristics are essential for explaining the outstanding magnetic properties of CoFe_2O_4 , such as its high magnetocrystalline anisotropy, elevated coercivity, and chemical stability, which make it a material of great interest for applications in magnetoelectric devices, information storage, sensors, and advanced multifunctional systems.

Surface morphology and composition

The SEM micrographs obtained using secondary electron (SE, left) and backscattered electron (BSE, right) detectors are shown in **Figure 4**. The images provide complementary information regarding the surface morphology and compositional homogeneity of the sample. In the SE image, the surface appears to be composed of fine particle aggregates distributed relatively uniformly throughout the analyzed area, whereas the BSE image exhibits a largely homogeneous contrast across the field of view. The absence of regions with markedly different brightness levels in the BSE mode suggests a uniform compositional distribution and rules out, at least on the micrometer scale, the presence of secondary phases with significantly different average atomic numbers.

Morphologically, the material exhibits a highly granular structure of particles with predominantly irregular to sub-spherical shapes. The primary grains appear strongly agglomerated, forming larger aggregates with poorly defined grain boundaries due to the high degree of coalescence. Based on the 5 μm scale bar, the size of the individual particles can be estimated at approximately 100 nm. However, the agglomerates observed reached dimensions on the order of 1–5 μm .

The microstructure displays a moderate level of open porosity, evidenced by irregular voids distributed among the particle conglomerates. These pores appear to originate mainly from incomplete packing of the aggregates during synthesis and subsequent thermal processing. No large macropores or extended cracks that could compromise the structural integrity of the material are observed. Another noteworthy feature is the pronounced tendency toward cluster or agglomerate formations, a characteristic commonly observed in ferrites with submicrometer particle sizes (Soufi *et al.*, 2021). This behavior can be attributed to the high surface energy of the fine particles and, in the

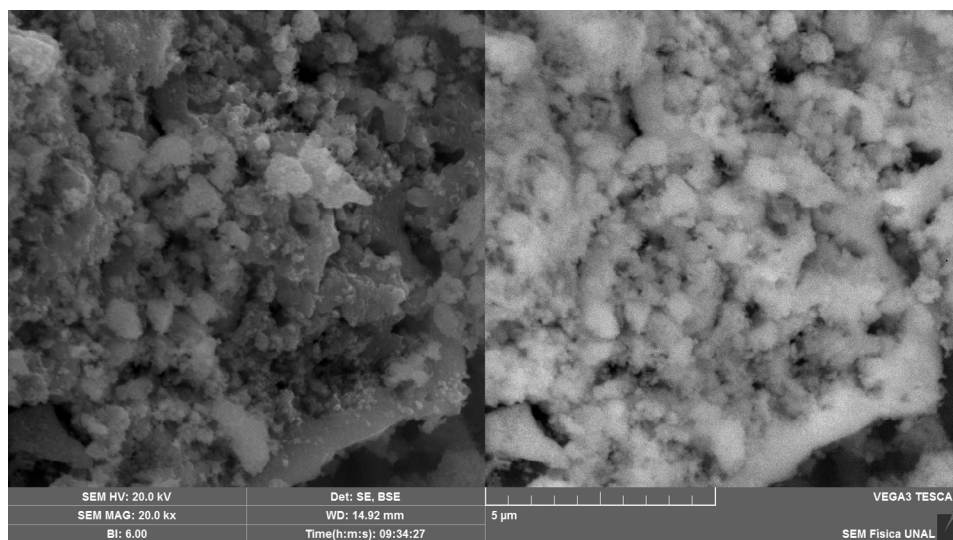


Figure 4. SEM images of the CoFe_2O_4 spinel from the SE and BSE detectors

specific case of CoFe₂O₄, to magnetostatic interactions between neighboring magnetic domains. Overall, the SEM images revealed a compositionally homogeneous sample of fine particles grouped into micrometer-sized aggregates, with moderate intergranular porosity and a relatively uniform morphological distribution. These characteristics are consistent with ferrite materials synthesized through chemical routes followed by thermal treatment.

The Energy-Dispersive X-ray Spectroscopy (EDS) spectrum in **Figure 5** confirms the presence of the chemical elements expected for cobalt ferrite (CoFe₂O₄), namely Fe, Co, and O, without evidence of significant impurities. The characteristic peaks observed at low energies correspond to the O-K α , Co-L α , and Fe-L α transitions, while the intense peaks located between approximately 6 and 8 keV are associated with the Fe-K α , Co-K α , Fe-K β , and Co-K β emission lines. The predominance of the Fe-related peaks compared to those of Co is consistent with the nominal stoichiometry of CoFe₂O₄, which contains twice as much iron as cobalt. The absence of additional peaks attributable to foreign elements suggests that the synthesis process yielded a chemically pure material within the detection limits of the EDS technique.

Furthermore, the relatively low background intensity and the sharp, well-defined characteristic peaks indicate good signal quality and adequate counting statistics. The simultaneous detection of Fe, Co, and O, together with the XRD results showing a single spinel phase, provides strong evidence for the successful formation of cobalt ferrite. Although EDS is not sufficiently accurate for precise oxygen quantification due to the low atomic number of oxygen, the spectrum qualitatively supports the expected elemental composition and confirms the compositional homogeneity of the sample at the microscale.

The quantitative EDS analysis shows an excellent agreement between the experimental composition and the theoretical stoichiometry expected for CoFe₂O₄. Based on the chemical formula, the theoretical weight percentages are 25.11 wt.% Co, 47.60 wt.% Fe, and 27.29 wt.% O, whereas the experimental values obtained from EDS are 25.19 wt.% Co, 48.02 wt.% Fe, and 26.79 wt.% O. The deviations are very small, amounting to only +0.08 wt.% for Co, +0.42 wt.% for Fe, and -0.50 wt.% for O, which confirms the successful synthesis of cobalt ferrite with a composition very close to the nominal stoichiometry. The slight decrease observed in the oxygen content can be attributed

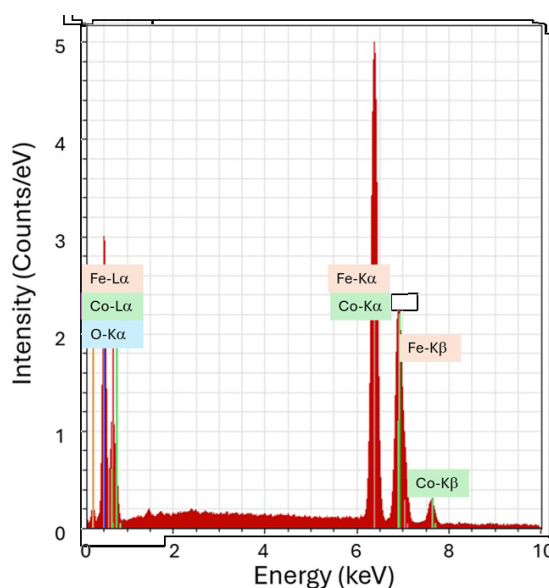


Figure 5. EDS spectrum with the characteristic L α , K α , and K β transitions for the CoFe₂O₄ spinel

to several factors. First, EDS has inherent limitations in the quantification of light elements such as oxygen due to their low fluorescence yield and the strong absorption of low-energy X-rays within the sample and detector system. Second, minor oxygen deficiencies may arise during thermal treatment, where localized oxygen vacancies can form because of high-temperature processing. Additionally, surface roughness, particle agglomeration, and matrix effects can introduce small uncertainties in the measured oxygen concentration. Therefore, the observed oxygen deficit is within the expected experimental error of the technique and does not indicate a significant departure from the stoichiometric CoFe_2O_4 composition.

Optical response

The optical band gap of the CoFe_2O_4 sample was determined from diffuse reflectance spectroscopy using the Kubelka–Munk approach proposed by **Kumar et al.** (1999) for optically thick samples, in which the reflectance data are transformed into a quantity proportional to the absorption coefficient. The resulting plot (**Figure 6**) exhibits a well-defined linear region whose extrapolation to the energy axis yields an optical direct band gap value of $E_g = 1.45 \pm 0.05$ eV. This result indicates that cobalt ferrite behaves as a narrow-band-gap semiconductor, capable of absorbing a significant fraction of visible and near-infrared radiation. The obtained value is consistent with previous reports for CoFe_2O_4 , where optical band gaps typically range from 1.3 to 2.0 eV depending on the synthesis route, the particle size, the cation distribution, the crystallinity, and the defect concentration. The relatively low band gap observed here may be associated with localized electronic states generated by cation inversion, oxygen vacancies, and $\text{Fe}^{3+}/\text{Co}^{2+}$ crystal-field interactions, which introduce intermediate energy levels within the forbidden band and facilitate optical transitions.

A comparison with classical semiconductors highlights the intermediate electronic character of CoFe_2O_4 . Crystalline silicon, the most widely used semiconductor in microelectronics and photovoltaics, possesses an indirect band gap of approximately 1.12 eV (**Battaglia et al.**, 2016), while germanium exhibits an even smaller band gap of about 0.66 eV. In contrast, direct-gap semiconductors such as gallium arsenide (GaAs) have a band gap close to 1.42 eV (**Ahn et al.**, 1971), remarkably similar to the value obtained for

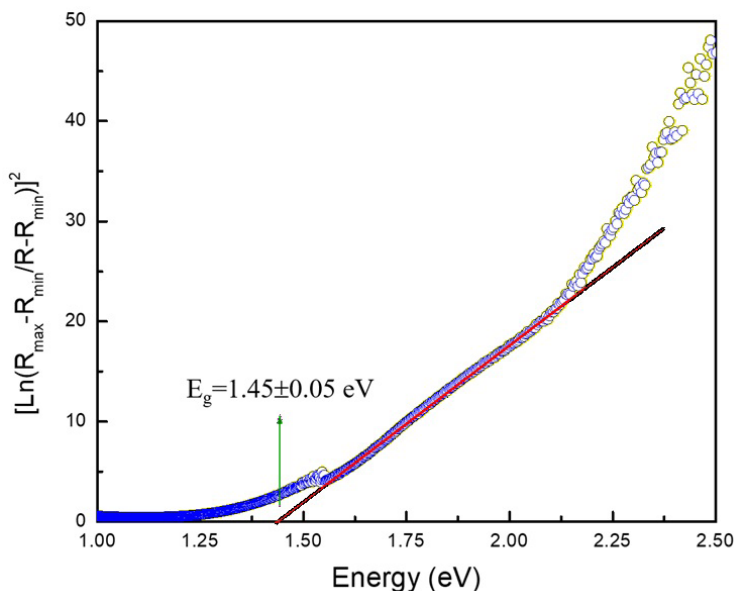


Figure 6. Kubelka-Munk analysis to determine the band gap value for CoFe_2O_4 following the technique for thick samples

the present CoFe₂O₄ sample. Wide-band-gap semiconductors (~3.37 eV) (Holonyak *et al.*, 1972) absorb predominantly in the ultraviolet region and, therefore, require substantially higher photon energies for electronic excitation.

The measured band gap of 1.45 eV is particularly attractive from an application standpoint because it lies close to the optimum range predicted for efficient solar-energy conversion, enabling absorption over a broad portion of the solar spectrum. Furthermore, the coexistence of semiconducting and ferrimagnetic properties in CoFe₂O₄ makes this material especially promising for multifunctional devices, including photocatalysts, photoelectrochemical systems, magneto-optical components, spintronic devices, and magnetically recoverable photocatalysts. The diffuse reflectance analysis confirmed not only the semiconducting nature of the synthesized cobalt ferrite but also its potential for applications requiring efficient visible-light absorption combined with magnetic functionality.

Magnetic features

The magnetization versus the applied magnetic field (M–H) curve (**Figure 7**), measured at room temperature, exhibits the characteristic sigmoidal behavior of a ferrimagnetic material and is fully consistent with the magnetic response expected for cobalt ferrite (CoFe₂O₄).

The hysteresis loop is symmetric with respect to the origin, indicating the absence of exchange-bias effects or significant magnetic anisotropy induced by secondary phases. The continuous increase in magnetization with the applied field and the subsequent tendency toward saturation confirmed the establishment of long-range magnetic ordering within the sample. The maximum magnetization achieved at the highest applied field (~15 kOe) was approximately 50 emu/g, suggesting a saturation magnetization (M_s) close to this value. This result falls within the range commonly reported for polycrystalline CoFe₂O₄ synthesized by chemical methods, where M_s values typically vary from 40 to 80 emu g⁻¹ depending on the particle size, the crystallinity, the cation distribution, and the synthesis conditions. The fact that complete saturation is not fully reached even at 15 kOe reflects the intrinsically high magnetocrystalline anisotropy of cobalt ferrite, which requires relatively strong magnetic fields to align all magnetic moments along the field direction. The hysteresis loop presents a finite width around the origin, indicating the existence of remanent magnetization and coercivity. Although the exact values cannot be accurately extracted from **Figure 7** alone, the coercive field appears to be relatively small

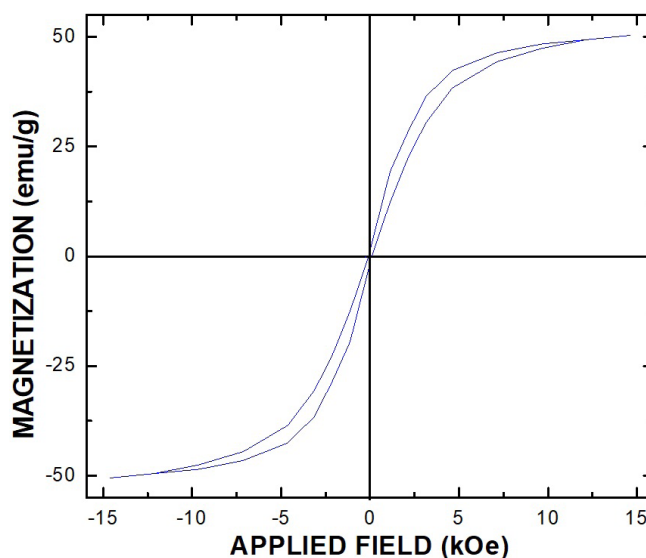


Figure 7. Hysteresis magnetic curve for the CoFe₂O₄ material produced using the combustion technique

compared with the maximum applied field, suggesting that the sample exhibits moderate magnetic hardness. Such behavior is frequently observed in ferrite powders composed of submicrometric grains, where the magnetic response results from the combined effects of magnetocrystalline anisotropy, domain-wall motion, and interparticle interactions.

The observed ferrimagnetism originates from superexchange interactions between Fe^{3+} ions occupying tetrahedral (A) sites and $\text{Fe}^{3+}/\text{Co}^{2+}$ ions located at octahedral (B) sites within the inverse spinel structure. Because the magnetic moments of the A and B sublattices are antiparallel but unequal, a net spontaneous magnetization is generated. The measured magnetization value, together with the phase purity evidenced by XRD and the compositional homogeneity revealed by SEM-EDS, confirms the successful formation of cobalt ferrite with well-developed ferrimagnetic ordering.

An additional feature of the curve is the absence of constrictions, steps, or secondary switching events, which indicates that the magnetic response is dominated by a single ferrimagnetic phase. This observation is in excellent agreement with the structural characterization results, which showed no evidence of secondary iron oxide or cobalt oxide phases. Consequently, the magnetic measurements corroborate the formation of a structurally homogeneous CoFe_2O_4 phase exhibiting robust ferrimagnetic behavior at room temperature, making it suitable for applications in magnetic storage, sensors, magnetoelectric devices, and multifunctional magnetic-semiconducting systems.

The combined optical and magnetic results demonstrate that CoFe_2O_4 exhibits a remarkable coexistence of semiconducting and ferrimagnetic properties at room temperature. The optical band gap of approximately 1.45 eV confirms its ability to absorb visible light and generate electronic excitations, while the well-defined hysteresis loop and saturation magnetization close to 50 emu g^{-1} reveal robust long-range magnetic ordering. This dual functionality places cobalt ferrite within the class of magnetic semiconductors, a family of materials that has attracted considerable attention for next-generation multifunctional technologies. The simultaneous presence of charge and spin degrees of freedom enables the manipulation of electronic transport through magnetic fields, opening opportunities for applications beyond conventional electronics. In particular, CoFe_2O_4 is a promising candidate for spintronic devices, where information is carried not only by the electron charge but also by its spin polarization. Furthermore, its combination of visible-light absorption, chemical stability, and ferrimagnetism makes it attractive for multifunctional devices such as magneto-optical sensors, spin filters, magnetic memories, photocatalysts, photoelectrochemical systems, and magnetoelectric heterostructures. Therefore, the coexistence of semiconducting and magnetic behaviors significantly expands the technological potential of CoFe_2O_4 compared with traditional semiconductors or purely magnetic materials, making it a valuable platform for integrated electronic, photonic, and spin-based applications (Khana & Hong, 2024).

Conclusions

Based on our results, we can conclude that the urea-assisted sol-gel auto-combustion technique constitutes a highly efficient route for the synthesis of cobalt ferrite (CoFe_2O_4) with a single-phase spinel structure, high chemical purity, and well-defined multifunctional properties. The Rietveld refinement confirmed the exclusive formation of the inverse cubic Fd-3m phase, while the SEM and EDS analyses revealed a homogeneous distribution of the constituent elements and a composition very close to the theoretical stoichiometry. Furthermore, the measured band gap of 1.45 eV and the saturation magnetization of approximately 50 emu/g demonstrate the coexistence of semiconducting and ferrimagnetic behavior at room temperature. Compared with other synthesis methods, such as co-precipitation, solid-state reaction, or hydrothermal processing, the sol-gel auto-combustion route offers significant advantages in terms of chemical homogeneity, stoichiometric control, reduced processing time, and the ability to obtain nanocrystalline materials with reproducible properties. The combination of

structural purity, robust magnetic response, and efficient visible-light absorption makes this technique particularly attractive for producing ferrites intended for applications in photocatalysis, magnetoelectric devices, sensors, magnetic storage systems, and advanced spintronic technologies.

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Author contributions

ASMB, BJAL, and EAVR synthesized the samples, took measurements, and processed the data. DALT coordinated the structural and morphological analyses. JRR proposed and directed the project, coordinated the work team, interpreted and correlated the experimental and theoretical results, analyzed the interaction mechanisms, and wrote the paper.

Conflicts of interest

The authors declare that there is no conflict of interests of any kind regarding the publication of the results of our research work.

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