International Journal of Chemical Science
www.chemicaljournals.com
Online ISSN: 2523-2843, Print ISSN: 2523-6075
Received: 21-07-2025, Accepted: 20-08-2025, Published: 05-09-2025
Volume 9, Issue 2, 2025, Page No. 32-35

Synthesis and structural characterizations of mixed Mg-Co spinel ferrite via sol-gel auto-combustion method

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Abstract
The current investigation focuses on synthesis of $Mg_{0.5}Co_{0.5}Fe_2O_4$ (Mg-Co) ferrite nanoparticles via sol-gel auto combustion method starting from metal nitrates and citric acid ($C_6H_8O_7$) as a fuel and chelating agent. The prepared samples were calcinated at 600 ˚C for 4 h in air. X-ray diffraction (XRD) data used for estimation of structural parameters. The average crystallite size was calculated by using Debye Scherer's formula and obtained value is 24 nm. Tetrahedral bond length (d_{AX}), octahedral bond length (d_{BX}), tetra edge (d_{AXE}) and octa edge (d_{BXE}) were determine from XRD. FTIR in KBr pellets shows two main characteristic peaks for octahedral and tetrahedral metal-oxygen bonds which confirm the formation of single-phase Co-Mg ferrite.

Keywords: Mg-Co ferrite, auto-combustion, structural properties, XRD

Introduction

Spinel ferrites are a class of magnetic oxides with a cubic spinel structure with the chemical formula A B_2O_4 where, A and B are located between tetrahedral and octahedral sites in the spinel structure. Their physical properties strongly depend on cation distribution among A and B sub-lattices in the spinel structure, which will make them wieldy in technological applications [1]. These ferrite processes have excellent chemical stability, high electrical resistivity, and tunable magnetic properties. These features make them applicable in electronics, magnetics, and biomedical devices [2]. At the nanoscale, ferrites show enhanced magnetic behavior due to size and surface effects [3]. Wet chemical methods, especially sol-gel auto-combustion, offer controlled synthesis of nanosized, high-purity ferrites [4]. Cobalt ferrites have been comprehensively studied due to their high chemical stability, electrical resistivity and strong ferromagnetic nature with high Curie temperature. These properties make them highly suitable for advanced technological applications in electronic appliances, data storage and energy storage devices [5]. Magnesium-cobalt nano ferrite (Co-Mg) is a remarkable spinel ferrite which exhibits high coercivity, moderate saturation magnetization, good chemical stability and mechanical hardness properties at the nanoscale. The partial substitution of Mg^{2+} and Co^{2+} in the spinel lattice significantly influences the cation distribution, which can have an effect on magnetic and structural properties, making it suitable for magnetic storage, microwave devices, sensors, and electromagnetic interference (EMI) shielding applications [6, 7]. for the synthesis of nano-ferrites, the literature mentions two classes of synthesis. One class is a solid-state reaction [8]. This method requires a high temperature, a long time, and, unfortunately, it produces an uncontrolled grain size and another class represents wet chemical routes which include a sol–gel method, a co-precipitation method, and a hydrothermal method etc [9]. Among all wet chemical methods, sol-gel auto-combustion has emerged as a cost-effective and efficient route for synthesizing spinel ferrite nanoparticles due to its ability to offer better stoichiometric control, homogeneous mixing of precursors, and lower synthesis temperature. This technique involves the formation of a sol by dissolving metal nitrates in a chelating agent, which is then dried to a gel and ignited to undergo a self-sustained combustion reaction, producing ultrafine ferrite nanoparticles [10, 11].

The current study focused on the synthesis of $Mg_{0.5}Co_{0.5}Fe_2O_4$ ferrite nanoparticles via a sol-gel auto-combustion technique, and their structural properties were systematically investigated using X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR) are reported herein.

Experimental

Material and Methods

The Mg-Co spinel ferrite nanoparticles were synthesised using AR grade chemical were collected from Loba Chemie Pt. Ltd magnesium nitrate hexahydrate [Mg $(NO_3)_2{\cdot}6H_2O$], cobalt nitrate hexahydrate [Co $(NO_3)_2{\cdot}6H_2O$], ferric nitrate nonahydrate [Fe $(NO_3)_3{\cdot}9H_2O$], anhydrous citric acid ($C_6H_8O_7$), ammonia solution (25%) and distilled water as a solvent. (Produces in laboratory)

Preparation of Mg-Co ferrite nanoparticles

The $Mg_{0.5}Co_{0.5}Fe_2O_4$ spinel ferrite nanoparticles were prepared via sol gel auto-combustion route. The flow chart for synthesis of Mg-Co ferrite nanoparticles is depicted in figure 1. The stoichiometric amounts of magnesium nitrate, cobalt nitrate, and ferric nitrate are dissolved in distilled water separately and mixed thoroughly under constant stirring. Citric acid is then added as a fuel and chelating agent in a 1:1 molar ratio with respect to total metal nitrates. The pH of the solution is adjusted to around 7 using ammonia solution to facilitate gel formation. The solution was then heated on a hot plate at 80–90° C under constant stirring until a viscous gel formed, which upon further heating undergoes spontaneous auto-combustion, resulting in a loose, black powder. This powder is finely grinded, and then calcined at 600 °C for about 4 h in air to remove residual organics, obtain the desired phase and enhance crystallinity [12, 13]. the resulting product is grinding in to fine powder and used for characterization.

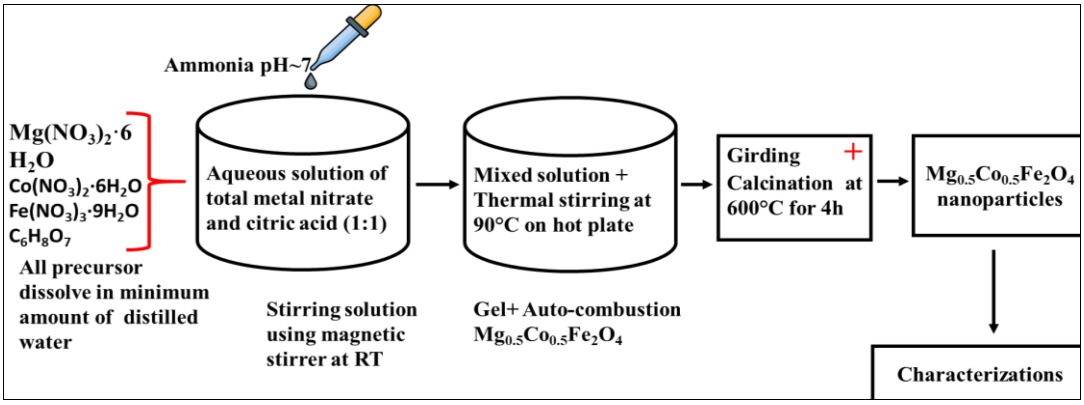

Fig 1: Flowchart of sol-gel auto synthesis of $MG_{0.5}CO_{0.5}O_4$ nanoparticles

Characterizations

The structural characteristics of Mg-Co ferrite nanoparticles were examined using a Tabletop X-ray diffractometer (REGAKU) through X-ray diffraction (XRD) analysis. The diffraction data were collected at room temperature within a 2θ range of 10° to 70°, take on Cu-K_α radiation with a wavelength of 1.54056 Å. Additionally, Fourier Transform Infrared (FTIR) spectra were recorded using a Shimadzu FTIR spectrophotometer (Japan) in the spectral range of 400 cm^{-1} to 4000 cm^{-1}, in a KBr pellet.

Result and Discussion

Structural analysis through XRD

An X-ray diffractogram of the sol-gel-derived Mg-Co spinel ferrite nanoparticles sintered at 600 °C for 4h in air with miller indices for each plane is shown in Fig2a. The xrd pattern consists of the most intense (311) peak, which confirms the formation of a single spinel phase without any additional impurity and crystallinity with all other peaks being indexed according JCPDS Card # 22-1086 belongs to Fd-3m space group. The structural parameters for synthesised Mg-Co ferrite nanoparticles were calculated using equation 1-7 and were depicted in table1.

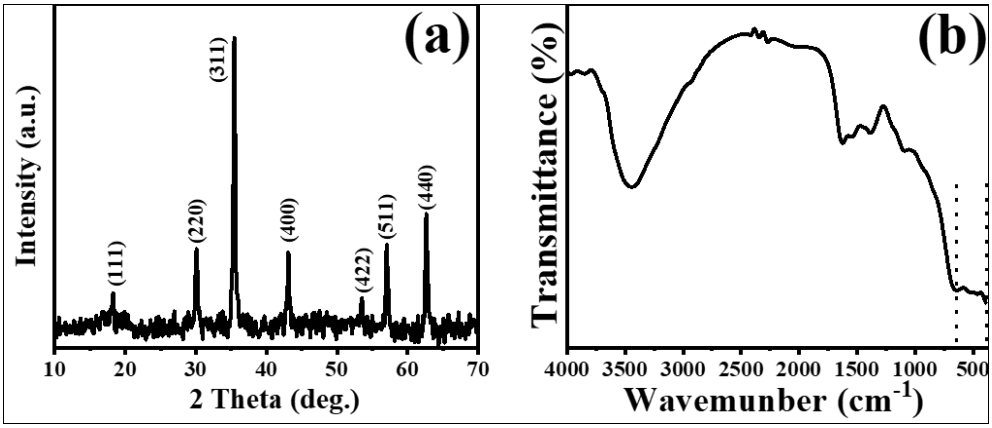

Fig 2: a XRD pattern b FTIR spectra of $MG_{0.5}CO_{0.5}FE_2O_4$

The average crystallite size (D) is calculated from Debye-Scherrer formula $D = \frac{0.9\lambda}{\beta Cos(\theta)}$ ……..(1)

Where, λ is the X-ray wavelength (1.5019 Å), β is the full width at the half maximum (FWHM) and θ is the Bragg angle for all diffraction peaks.

Lattice strain was calculated using

$$\varepsilon = \frac{\beta}{4*Tan(\theta)} \quad \ldots\ldots\ldots \quad (2)$$

The experimental lattice constant estimated from

$$a_{exp} = d\sqrt{h^2 + k^2 + l^2} \quad \ldots\ldots\ldots \quad (3)$$

Where d is inter planner spacing and (hkl) are miller indices for diffraction planes

The theoretical lattice constant calculated using

$$a_{th} = 1.5396[(r_A + R_0) + 1.732(r_B + R_0)] \quad \ldots\ldots \quad (4)$$

Where r_A is average ionic radius of the cations in the tetrahedral (A) sites, r_B is average ionic radius of the cations in the octahedral (B) sites and R_0 is the ionic radius of the oxygen anion (1.38Å)

The X-ray density find using relation

$$d_x = \frac{Z*M}{VN_A} \quad \ldots\ldots \quad (5)$$

Where, Z number of molecules per unit, M is molecular weight, V is volume of unit cell and N_A is Avogadro's number.

The bulk density of disc shaped pellets of Mg-Co ferrite was calculated using $d_b = \frac{m}{\pi r^2 h}$ ……(6) where m is mass, r is radius and h is width of the pellet.

Porosity (%) of was calculated from [14]

$$P = \left(1 - \frac{d_b}{d_x}\right) * 100\% \ \ldots\ldots\ldots\ (7)$$

Tabel 1: Average particle size, lattice strain, Lattice parameter (a_{exp} and a_{th}), X-ray density (d_x), Bulk density (d_b) and Porosity (P %) for Mg-Co ferrite.

sample	D (nm)	$\varepsilon * 10^4$	a_{exp}(Å)	a_{exp}(Å)	d_x (gm/cm^3)	d_b (gm/cm^3)	P (%)
Mg-Co ferrite	24	6.2140	8.3930	8.3929	4.8099	3.25	32.43

The T. Dabbebi *et al* [15] reported the cation distribution for sol- gel synthesised $Mg_{0.5}Co_{0.5}Fe_2O_4$ as

$$(Fe_1^{3+})_A(Mg_{0.5}^{2+}Co_{0.5}^{2+}Fe_1^{3+})_BO_4^{2-},$$

the same cation distribution is used for calculating distance between metal ions at tetrahedral and octahedral sites called as hopping length denoted as L_a=$0.25a * \sqrt{3}$ & L_b=$0.25a * \sqrt{2}$. Using the experimental values of lattice constant 'a' and oxygen positional parameter 'u' (u = 0.381 Å) The inter atomic structural parameters such as tetrahedral bond length (d_{AX}), octahedral bond length (d_{BX}), tetra edge (d_{AXE}) and octa edge (d_{BXE}) [14] were calculated and listed in table2.

Table 2: Hopping length (L_a, L_b) tetrahedral and octahedral sites, bond lengths (d_{AX} and d_{BX}), tetra edges (d_{AXE}), shared (d_{BXE}) and unshared (d_{BXEU}) octa edges for Mg-Co ferrite.

L_a (Å)	L_b (Å)	d_{AX} (Å)	d_{BX} (Å)	d_{AXE} (Å)	d_{BXE} (Å)	d_{BXEU} (Å)
3.6343	2.9674	1.9044	2.0496	3.1098	2.8249	2.9091

FTIR

Figure1b represents the Fourier Transform Infrared (FTIR) spectrum of Mg-Co ferrite recorded in the range of 4000–400 cm^{-1}. The spectrum exhibits characteristic absorption bands associated with metal-oxygen stretching vibrations, confirming the formation of the spinel ferrite structure. A prominent absorption band observed around 638 cm^{-1} is attributed to the stretching vibrations of metal-oxygen bonds at tetrahedral sites, while a weaker band at 400 cm^{-1} corresponds to vibrations at octahedral sites [16]. Additional absorption features in the higher wavenumber region, particularly between 1400–1600 cm^{-1} and around 3400 cm^{-1}, may be linked to adsorbed water molecules and hydroxyl groups, indicating surface-bound species or minor impurities. Overall, the FTIR analysis supports the successful synthesis of the spinel ferrite phase with characteristic vibrational modes of the constituent metal-oxygen bonds. Force constant values for Mg-Co ferrite were calculated from $k = 4\pi^2c^2\mu v^2$ depicted below.

sample	v_1	v_2	F_A* 10^4 (dyne/cm^2)	F_B* 10^5 (dyne/cm^2)
Mg-Co ferrite	400	638	11.3135021	23.17053358

Conclusion

Mg-Co ferrite nanoparticles were successfully synthesized by sol-gel auto-combustion technique using AR-grade metal nitrates and citric acid as a fuel. The X-ray diffraction results of Mg-Co ferrite showed the formation of a single-phase cubic spinel structure. The average crystallite size of the synthesized ferrite, calculated using the Debye Scherrer formula, was 24 nm. FTIR in KBr pellets shows two main characteristic peaks for octahedral and tetrahedral metal-oxygen bonds which confirm the formation of pure Co-Mg ferrite. These ferrites have a potential application in the photocatalysis, magnetic data storage and biomedical applications.

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