

Original Article

Structural and Morphological Evaluation of Cobalt Zinc Ferrite synthesized by the sol-gel method

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Manuscript ID:
IJERSD-2026-020204

ISSN: 3067-2325

Volume 2

Issue 2

Pp. 19-23

April 2026

Submitted: 10 Mar. 2026

Revised: 18 Mar. 2026

Accepted: 10 Apr. 2026

Published: 30 Apr. 2026

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Quick Response Code:



Web: <https://rlgjaar.com>

DOI: 10.5281/zenodo.22872820

DOI Link:

<https://doi.org/10.5281/zenodo.22872820>



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Abstract

Nanocrystalline cobalt-zinc ferrites with the composition $\text{CoZn}_x\text{Fe}_{2-x}\text{O}_4$ ($x = 0.0, 0.2, \text{ and } 0.4$) were successfully synthesized via a facile sol-gel auto-combustion route and investigated for their structural and morphological properties. The prepared ferrite powders were characterized using X-ray diffraction (XRD), Williamson-Hall ($W-H$) analysis, high-resolution TEM (HRTEM), transmission electron microscopy (TEM), and selected-area electron diffraction (SAED). The single-phase cubic spinel phase formed in the ferrite structure without any detectable secondary phases was confirmed by XRD analysis. The lattice parameter decreased from 8.363 to 8.334 Å as the cobalt content increased, indicating lattice contraction due to cation redistribution and ionic size differences. The crystallite size increased from 13.52 to 22.20 nm. The observed structural evolution, lattice-strain modification, and particle growth induced by cobalt substitution demonstrate the potential of Co-Zn ferrites to tailor the physical properties required for magnetic and multifunctional nanomaterial applications.

Keywords: Cobalt-zinc ferrite, Sol-gel auto-combustion, Spinel ferrite, Lattice strain, Nanoparticles, TEM, XRD.

Introduction

Over the last few decades, research on novel magnetic nanomaterials capable of meeting the requirements of a variety of applications, from electromagnets to nanomedicines, has increased significantly [1-3]. The reduced size of magnetic materials is responsible for an increased response, driven by inter-particle interactions, the surface-to-volume ratio, surface interfaces, and superparamagnetism [4-6]. The modification in magnetic properties depends on careful control of nanoparticle dimensions. The magnetic anisotropy, magnetic moment, and coercivity depend on size and shape, as well as the structural disorder, of the synthesized ferrites. Novel chemical synthesis routes are being proposed in this direction to achieve control over the shape, size, and morphology of nanoparticles [7-9]. The complex structures of magnetic nanoparticles exhibit intriguing properties, demonstrating their usefulness in various technological applications, including microwave absorbers, MRI, drug delivery, hyperthermia, and gas sensors [10-12]. Spinel ferrites are extensively studied magnetic materials due to their variations in physical and magnetic properties. Spinel ferrite materials are useful for low- to high-frequency devices. The synthesis of spinel ferrite is performed by different methods, such as chemical co-precipitation, solvothermal, hydrothermal, spray-drying, and sol-gel auto-combustion [13-16].

Extensive research on ferrites has been conducted due to their applications across various fields. Among these ferrites, Co ferrite, Zn ferrite, and Cu ferrite have potential applications [17-19]. In the ferrite lattice structure, the positions of cations determine the magnetic and electrical properties. The tetrahedral and octahedral sites host super-exchange interactions involving magnetic and nonmagnetic cations, which determine the material's magnetic properties. So, Co plays a significant role in modifying the magnetic properties of ferrite. The magneto-crystalline anisotropy of the ferrite increased with the content of cobalt, which is due to L-S spin/orbit coupling in the lattice. The coercivity of the ferrite increases with the presence of cobalt in the ferrite. Co^{2+} commonly occupies octahedral sites in the ferrite lattice, leading to a change in magnetic behavior. The variation of Zn^{2+} in the ferrite lattice can also modify magnetic properties. The nonmagnetic Zn^{2+} ion, with a zero magnetic moment, exhibits soft magnetic properties. Co and Zn have opposite magnetic properties, but when they combine in the ferrite lattice, they enhance saturation magnetization and magnetic coercivity [20, 21].

The literature survey motivates us to prepare cobalt-doped zinc ferrite, formulated as $\text{CoZn}_x\text{Fe}_{2-x}\text{O}_4$ (where $0.0 \leq x \leq 0.5$), via the sol-gel chemical route. The aim is to change the symmetry of the [B] site of cobalt zinc ferrite by the presence of Zn^{2+} . The changes in crystal structure, lattice strain, and morphology are explored by various methods. Effects of Zn^{2+} substitution on structure, morphology, and properties are explored.

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How to cite this article:

Gore, T. S. (2026). Structural and Morphological Evaluation of Cobalt Zinc Ferrite synthesized by the sol-gel method. *International Journal of Engineering Research for Sustainable Development*, 2(2), 19-23. <https://doi.org/10.5281/zenodo.22872820>

Experimental details

1. Preparation and characterization details

The cobalt-zinc ferrites were synthesized by the sol-gel route. The nitrate salt precursor used were $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$ of analytical grade (Sigma-Aldrich make). All metal nitrates were weighed in stoichiometric proportions, and clear solutions were prepared in separate beakers by adding sufficient de-ionized water. The clear solution of citric acid was also prepared. The proportion of citric acid to nitrates was kept at 3:1. The metal nitrates and citric acid solutions were mixed, and the pH was maintained with ammonium hydroxide. The pH-maintained solution was heated to 100°C with continuous stirring until it formed a thick gel. The gel turned into a powdery ash. The powders were collected, heated separately at 500°C in an open-air furnace for 5 h, and then used for further characterization.

Characterization detail

The sintered powder samples were inspected using a Philips X-ray diffractometer (model 3710) with $\text{Cu K}\alpha$ radiation ($\lambda = 1.54051 \text{ \AA}$). The obtained results confirmed the morphology and crystal structure of the synthesized samples using a high-resolution transmission electron microscope (HRTEM), model Philips CM 200.

Result and discussion

Phase identification and structural confirmation

The XRD pattern of cobalt zinc ferrite samples is shown in Fig. 1. The XRD data have been used for the determination of lattice constant (a), X-ray density (dx), bulk density (dB), crystallite size (DXRD, DW-H plot), and lattice strain (e). All reflection peaks of the XRD were exactly matched with the nanocrystalline phase of zinc ferrite and confirmed the formation of phase-pure inverse cubic spinel ferrite structure (JCPDS file no 82-1042) [22]. The reflection peak positions, slightly shifted towards higher angles, reveal a decrease in the lattice constant [23]. Table 1 presents structural data of the ZCC ferrite. The following equation was used to measure the lattice constant [24].

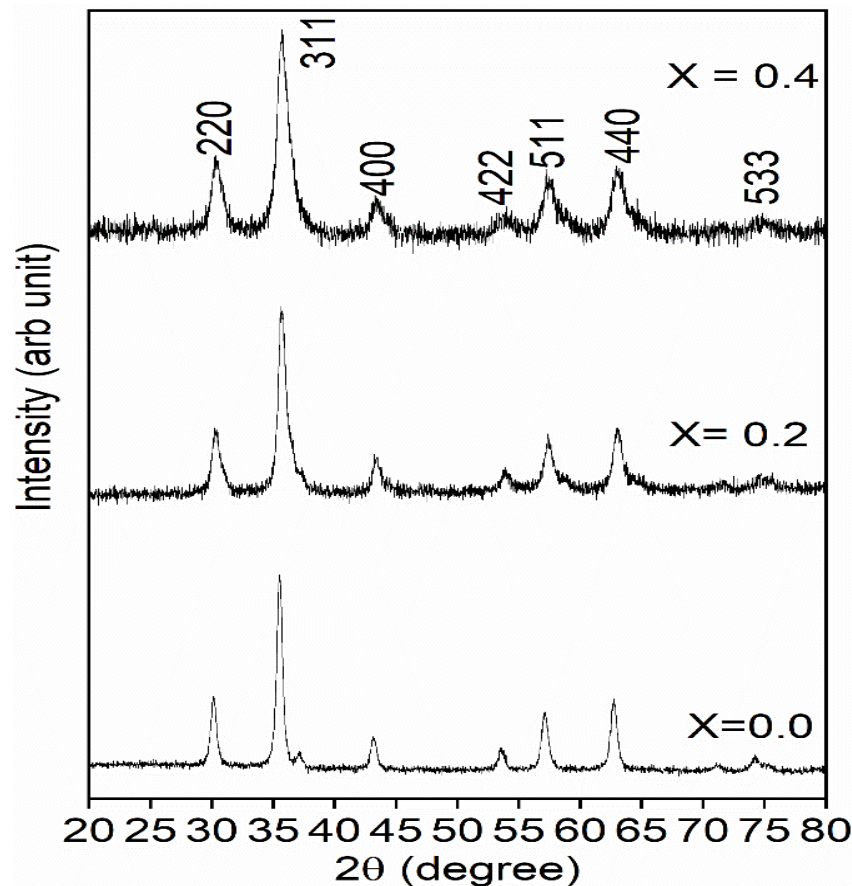


Fig. 1 The XRD patterns of cobalt zinc ferrites for various 'x' values.

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

The value of the lattice constant (a) decreases slightly with the increase in Zn^{2+} content. The decreasing nature of 'a' was due to the bigger Zn^{2+} ions ($0.83 \text{ \AA}$) being replaced by smaller Co^{2+} ions ($0.78 \text{ \AA}$), resulting in a shrinkage of the lattice cells of the cobalt zinc ferrite. The mixed spinel ferrite produces an inhomogeneous distribution of the cations due to more species of the cations [25]. The site preference of cations depends on their electronegativity. More electronegative ions prefer the octahedral site, while less electronegative ions prefer the tetrahedral site. The Co ions, which are more electronegative (1.9), prefer an octahedral site and try to replace cations from the octahedral to the tetrahedral site, causing lattice distortion [26]. This is one of the reasons cobalt zinc ferrite obeys Vigard's law and shows linear behavior in the lattice constant. The sample's X-ray density (dx) was calculated using the molecular weight (M) of the sample, lattice constant (a), and Avogadro's number (N) [27].

$$\rho_x = \frac{8M}{Na^3}$$

1

2

X-ray density (ρ_x) and bulk density (d_B) (Table 1) decrease with Zn²⁺ substitution. The x-ray density in ferrites is usually found to be higher than the bulk density. The higher value of ρ_x is due to the formation of grains and pores during preparation and sintering. The reason behind the decreasing densities in cobalt zinc ferrites can be explained with the help of the molar density of Co²⁺ and Zn²⁺ ions. The molar density of Co²⁺ ions is less than that of Zn²⁺ ions, which results in overall density reduction of the samples by replacing Zn ions with Co ions [28]. The powder sample crystallite size was calculated by using the Scherrer formula.

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

where λ is the wavelength of the x-ray, β is the corresponding reflection's full width at half maximum and θ is the corresponding Bragg angle.

The reduction in lattice strain was caused by the replacement of large-sized Zn²⁺ ions by small-sized Co²⁺ ions.

The crystallite size and lattice strains were calculated with the assistance of the Williamson–Hall (W-H) method. The strain in the lattice and crystallite size of the nanomaterials are directly related. The relation between size broadening (Scherrer equation) and strain broadening is given by the relation,

$$\beta_e + \beta_L = \beta_{hkl} \quad (4)$$

where, β_e is the lattice strain, β_L is the crystallite size, β_{hkl} is the Bragg's angle of peak at full width at half maxima. The equation can be written as,

$$4\varepsilon (\tan \theta_{hkl}) + k\lambda / (d \cos \theta_{hkl}) = \beta_{hkl} \quad (5)$$

where, ε is the lattice strain, $k = 0.98$ (constant), λ is the wavelength (1.54061 Å), and d is the crystallite size. The equation (2) can be extended to,

$$k\lambda / d + 4\varepsilon \sin \theta_{hkl} = \beta_{hkl} \cos \theta_{hkl} \quad (6)$$

The graph of “ $4\varepsilon \sin \theta$ ” against “ $\beta \cos \theta$ ” shown in fig.2 gives both lattice strain and crystallite size which can be obtained from of slope and Y-intercept of liner fit line respectively.

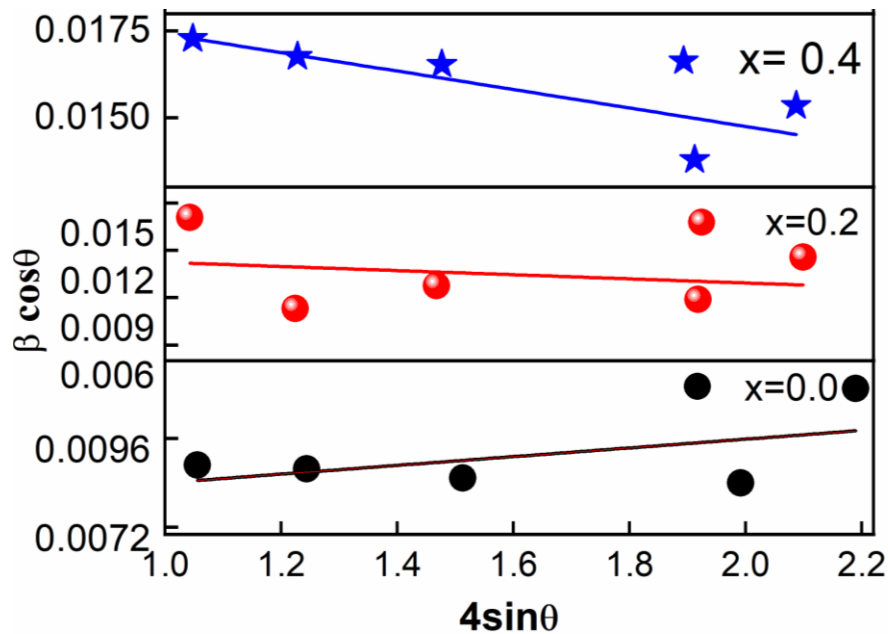


Fig.2. The lattice strain curves of cobalt zinc ferrites for various x values.

The W-H plots of cobalt zinc ferrites for various ‘x’ values are given in Fig. 2. For $x = 0.0$, the slope is positive, indicating a tensile lattice strain as shown in Table 1. The slope decreases with the increase of Co²⁺ content; $x = 0.2$ and $x = 0.4$. The negative slope value indicates compressive strain because Zn²⁺ ions are replaced by Co²⁺ ions. When Co²⁺ ions occupy the octahedral B-site, which already has Fe³⁺ ions, Fe³⁺ ions are replaced by tetrahedral sites. The ionic radii of the overall octahedral sites increase, creating compression in the lattice. [29].

Table 1: The lattice constant ‘a’, X-ray density ‘ ρ_x ’, Bulk density ‘ d_B ’, Crystallite size ‘ D_{XRD} ’, crystallite size from M-H plot ‘ D_{M-H} ’ and lattice strain ‘ ε ’.

Parameters (error values)	x=0.0	x= 0.2	x=0.4
a (Å) (± 0.002)	8.363	8.337	8.334
ρ_x (g/cm ³) (± 0.002)	5.429	5.425	5.418
d_B (g/cm ³) (± 0.002)	3.320	2.624	2.617
D_{XRD} (nm) (± 1)	13.52	14.39	22.2
$D_{M-Hplot}$ (nm) (± 1)	7.2	12.54	20.011.6
ε (± 0.00001)	0.00118	-0.0013	-0.00266

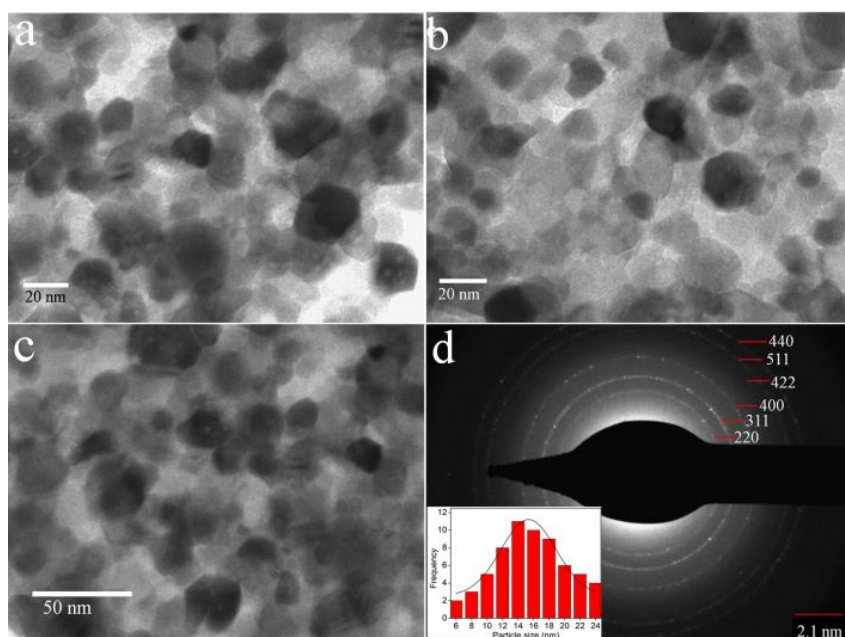


Fig. 3 (a- c) TEM images, (d) SEAD pattern and histogram at the inset for a typical cobalt zinc ferrite sample with $x = 0.0$.

1. Morphological evolution and particle size confirmation

The evolution of morphology and confirmation of particle size in cobalt zinc ferrites was performed by analysis of TEM images. The nanoparticles synthesized by the sol-gel method show variation in their sizes and shapes with variation in the values of Zn substitution ' x ' in cobalt zinc ferrites. For $x = 0.0$ (Fig. 3 a-c), cobalt zinc ferrite exhibits nanoparticles of rounded, cubic, and hexagonal shapes whose dimensions vary from 10 to 30 nm with an average particle size of 18 nm. The spotted circles for (222), (311), (400), (422), (511), and (440) planes are observed in the selected area electron diffraction (SEAD) image of the ferrite. The d-spacings obtained from the SEAD pattern rings are well matched to those obtained from XRD data, suggesting the polycrystalline nature of the cobalt zinc ferrite.

Conclusions

The cobalt zinc ferrites were successfully synthesized by the facile sol-gel auto-combustion method. The single-phase cubic spinel structure of the cobalt zinc ferrite is revealed from the XRD analysis. The lattice strain decreases with Zn^{2+} content, thereby increasing crystallite size and tuning the magnetic and electrical parameters. The TEM micrographs of cobalt zinc ferrites display nanoparticles of rounded, cubic, and hexagonal shapes, with sizes ranging from 10-38 nm, with average sizes of 18 and 26 nm for the ferrites with $x = 0.0$ and 0.5, respectively. The HRTEM and SEAD images confirm the polycrystalline nature of the cobalt zinc ferrites.

'Declaration of Generative AI and AI-assisted technologies in the writing process.'

During the preparation of this manuscript, the authors used Grammarly and AI-assisted technologies for language editing, grammar correction, and improving readability.

Acknowledgment

The authors express their sincere gratitude to all those who contributed to the successful completion of this research work entitled "Structural and Morphological Evaluation of Cobalt Zinc Ferrite Synthesized by the Sol-Gel Method." We are thankful to our institution and the Department for providing the necessary laboratory facilities, technical support, and a conducive research environment. We also acknowledge the valuable guidance and encouragement provided by our mentors and colleagues throughout the course of this study.

Financial support and sponsorship

Nil.

Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper

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