



Optimizing structural, and optical characteristic of Ni-doped Co-Zn nanoferrites: insights for biological evaluation on maize seed germination

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Received: 26 August 2025 / Accepted: 20 February 2026 / Published online: 1 April 2026
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Abstract

This study aimed to synthesize nickel-doped cobalt–zinc spinel ferrite ($\text{Co}_{0.5}\text{Zn}_{0.5-x}\text{Ni}_x\text{Fe}_2\text{O}_4$) nanoparticles and to investigate the influence of Ni^{2+} substitution on their structural, optical, and biological properties, with particular emphasis on their potential application in seed nano-priming. Spinel ferrites with different Ni concentrations ($x=0.0\text{--}0.5$) were synthesized *via* the citrate–nitrate auto-combustion method. X-ray diffraction and Rietveld refinement confirmed the formation of a single-phase cubic spinel structure with a prominent (311) reflection, indicating high crystallinity. Increasing Ni^{2+} substitution up to $x=0.3$ caused a slight increase in the lattice parameter from 8.397 to 8.398 Å, followed by a decrease to 8.390 Å at $x=0.5$, accompanied by variations in crystallite size (42.57–55.63 nm). X-ray density showed a marginal decrease with increasing Ni content, while porosity decreased up to $x=0.3$ and then slightly increased. Several structural parameters, including site radii, bond lengths, hopping lengths, and edge lengths, were derived from XRD data. HR-TEM analysis revealed mostly spherical, aggregated nanoparticles, consistent with their magnetic behavior. UV–Vis spectroscopy confirmed semiconducting characteristics, with optical band gaps ranging from 1.44 to 1.59 eV, placing all compositions within the visible-light region and indicating potential photocatalytic applicability. Germination assays on *Zea mays* seeds demonstrated that low concentrations of Ni-doped nanoferrite ($x \leq 0.3$) significantly enhanced germination percentage, seedling length, fresh and dry biomass, and vigor index, whereas higher concentrations exerted negligible effects. Notably, $\text{Co}_{0.5}\text{Zn}_{0.3}\text{Ni}_{0.2}\text{Fe}_2\text{O}_4$ produced the greatest stimulation, increasing seedling length, fresh weight, and dry weight by 45.5, 51.2 and 21.63%, respectively, compared with the control. These results highlight the potential of engineered spinel nanoferrites in agro-nanotechnology and emphasize the importance of optimizing concentrations for effective seed nano-priming and growth enhancement under controlled conditions.

Keywords Nanospinel ferrites · Ni substitution · Structural analysis · Optical evaluations · Germination assay, *zea mays*, vigor index, nano-priming

1 Introduction

Currently, spinel ferrites are garnering significant attention owing to their promising applications. Data storage, water purification, biomedical uses, color imaging, and magnetic materials are all on the horizon thanks to these developments

[1, 2]. Iron-based spinel ferrites are described by the formula MFe_2O_4 (with M representing Ni, Mn, Zn, Co, Cu, Mg, or Cd) and display a FCC structure that falls under the space group $\text{Fd}\bar{3}\text{m}$. M^{2+} and Fe^{3+} cations are found in A- and B-lattice positions, whereas the oxygen anions are distributed in a cubic close-packed array in the typical crystallographic description of the spinel structure. The cubic unit cell comprises 56 atoms, which include 32 oxygen anions organized in a cubic closely closed-pack configuration, along with 24 cations that fill 8 out of the 64 available tetrahedral (A) sites and 16 out of the 32 accessible octahedral (B) sites.

Typically, one can categorize spinel ferrite into three distinct varieties: normal, inverse, and intermediate spinel ferrite. These types are based on which A and B-sites cations

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prefer to occupy [3]. (i) In normal ferrite, the divalent ion is situated at the [A] site, whereas the trivalent ion is located at the [B] site. (ii) In the second type, known as inverse ferrites, the trivalent ions are distributed evenly between the tetrahedral and octahedral sites. (iii) In the third category, the intermediate ferrites, ions are distributed without a strict ordering rule across both sites without any specific order [4]. A tetrahedral site is one in which the metal ion is situated in the center of a tetrahedron with oxygen ions occupying the corners. Additionally, the corners of an octahedron are occupied by oxygen ions surrounding it in the octahedral site [5]. The preferential site selection for divalent ions is predominantly dictated by ionic size, which affects the material's magnetic and structural characteristics [6]. Ferrites nanomaterials present distinctive physical and chemical characteristics within the realm of engineering materials. The properties of ferrite are substantially impacted by the synthesis parameters, which include the doping of different cations, the temperature at which the sintering process occurs, the density of the sintered material, the grain size, and the dispersion [7]. The presence of dopants within the locations of the spinel structure meaningfully impacts the properties of these materials. Larger ionic radii can modify the unit cell parameters and shifted the oxygen ions diagonally, making the cation radius crucial for determining site habitation predilections. The distribution of cations across sublattices can profoundly affect the properties of ferrites, thereby impacting their performance in various applications [8].

Co-based ferrites have gained considerable attention due to their superior magnetic and catalytic properties, enabling applications in sensors, data storage, catalysis, and electromagnetic interference shielding [9, 10]. In particular, Zn-substituted cobalt ferrites (ZCF) exhibit enhanced antibacterial activity, improved electrical and electromagnetic performance, and excellent structural stability, making them promising materials for energy, electronic, environmental, biomedical, and spintronic applications with tunable band gaps [11–13]. Spinel ferrite nanocrystals, such as ZCF and mixed Ni–Co–Zn (NCZ) nano-ferrites, possess unique chemical, physical, and magnetic properties that make them highly attractive for a broad range of biomedical, technological, agricultural, and high-frequency industrial applications [14, 15]. ZCF nanocrystals are particularly notable for their environmentally safe nature and promising therapeutic potential, including drug delivery systems, antiviral sprays, antiviral surface coatings for household products, and biosensors for the early detection of viral strains such as COVID-19 when integrated with colossal magnetoresistance (CMR) sensors [13, 14]. In comparison with

Mn–Zn ferrites, Ni–Zn-based ferrites, including NCZ compositions, exhibit superior suitability for high-frequency applications due to the high thermal and chemical stability of Ni^{2+} and Zn^{2+} cations, which minimizes structural and compositional changes during thermal cycling [15]. Furthermore, strong exchange interactions among magnetic ions enhance ferrimagnetic behavior, resulting in negligible dielectric loss and dominant spin-rotation resonance, with magnetic loss being the main absorption mechanism [15]. Owing to their high permeability, high saturation magnetization, and low coercivity, NCZ spinel ferrites are regarded as ideal materials for various industrial and high-frequency applications [10].

In line with advances in nanotechnology, nanomaterials have gained increasing attention in agriculture for enhancing crop productivity and stress tolerance [16–18]. Engineered nanoparticles, particularly through seed nano-priming, improve germination, early growth, and resistance to biotic and abiotic stresses [18–20]. Within this context, spinel nanoferrites represent promising multifunctional materials, acting as efficient micronutrient carriers that enhance nutrient uptake, seed germination, and plant stress resilience, especially in nutrient-deficient soils, thereby contributing to improved crop yield and agricultural sustainability [21, 22]. Ferrite NPs have demonstrated potential as seed priming agents, where their interaction with seeds during imbibition can improve water uptake, activate metabolic enzymes, and promote early plant development [23–27]. Their nanoscale size enables them to penetrate seed coats and root tissues, where they may influence cellular signaling, nutrient absorption, and gene expression linked to germination and early growth. The beneficial effects of ferrite NPs are often concentration-dependent. At lower doses, they can act as micronutrient sources (Fe, Zn, Co, Ni) and biostimulants, while at higher concentrations they may induce oxidative stress and phytotoxicity [28, 29]. Recent studies have highlighted that doping ferrites with transition metals (e.g., Ni, Zn, Mn) modifies their structural and surface properties, thereby altering their biological interactions [30]. For instance, Ni-substituted spinel ferrites have exhibited increased catalytic and antioxidant activity, which could modulate the redox environment and influence energy metabolism [31]. Despite these promising findings, the precise mechanisms by which these nanoferrites affect seed germination and seedling development remain underexplored, especially in major cereal crops like *Zea mays*. Moreover, understanding how compositional changes (e.g., Ni substitution) in spinel ferrites influence their biological activity is crucial for developing safe and effective nanobiotechnology applications in agriculture.

This study focuses on the synthesis and investigation of the structural and optical characteristics of $\text{Co}_{0.5}\text{Zn}_{0.5-x}\text{Ni}_x\text{Fe}_2\text{O}_4$ nanoparticles (CZNF NPs) with nickel substitution values ($x=0.0, 0.1, 0.2, 0.3, 0.4$, and 0.5). In addition, the objectives of this research are to inspect the biological effects of nanoferrite on the germination of seeds and the early growth of *Zea mays* for the purpose of determining their possible applications in agriculture. In specifically, the nanoferrite is being investigated as a nano-promising agent that can improve germination dynamics and early seedling vigor.

2 Experimental details

2.1 Samples preparation

In the citrate technique, nanoferrites with the following compositions were produced: $\text{Co}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ (CZNF0), $\text{Co}_{0.5}\text{Zn}_{0.4}\text{Ni}_{0.1}\text{Fe}_2\text{O}_4$ (CZNF1), $\text{Co}_{0.5}\text{Zn}_{0.3}\text{Ni}_{0.2}\text{Fe}_2\text{O}_4$ (CZNF2), $\text{Co}_{0.5}\text{Zn}_{0.2}\text{Ni}_{0.3}\text{Fe}_2\text{O}_4$ (CZNF3), $\text{Co}_{0.5}\text{Zn}_{0.1}\text{Ni}_{0.4}\text{Fe}_2\text{O}_4$ (CZNF4), and $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ (CZNF5). The precursor aqueous solutions were derived using high-purity $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, and citric acid each liquefied in 20 mL of distilled water with the following molarities: 0.05 M, $[(0.5-x)/10]\text{M}$, 0.1 xM, 0.2 M and 0.3 M, respectively. In these compositions, x fluctuates in steps of 0.1 from 0.0 to 0.5. Citric acid primarily functions as a fuel and a chelating agent, while used nitrates as oxidizers. The oxidizers to fuel ratio was established at 1:1 through propellant chemistry, as this specific ratio yields the highest energy output during the combustion process. The previously described blended solution was continuously stirred for 15 min to achieve a homogenous and clear mixture, employing a magnetic stirrer with a hot plate, while incrementally raising the temperature to 100 °C. Ammonia solution (33%) was incrementally introduced to this transparent clear solution to achieve a pH of 7, thereby facilitating and invigorating the process. After that, the solution was heated to 350 °C for approximately one hour on a hot plate. A progressive increase in viscosity was observed, accompanied by a distinct chromatic transition, culminating in the formation of a brown, porous dry gel with a puffy morphology. The dried gel exhibited a vigorous auto combustion reaction upon ignition, characterized by a significant exothermic release of gaseous byproducts. This combustion initiated at the beaker's hottest zone and propagated vertically in a volcanic-like eruption. The process yielded a dark-gray solid residue, displaying a distinct dendritic (branched) morphology. Subsequently, the resulting powder of the CZNF samples was pulverized in a mortar to produce fine, homogeneous CZNF

granules. Various methodologies were employed to ascertain the characteristics of the CZNF NPs.

The citrate approach was chosen for sample preparation due to its eco-friendliness, cost-effectiveness, and relative simplicity. It should be mentioned that the sol-gel auto-combustion process yields the following gases when metal nitrates, citric acid, and other reactants decompose during the production of CZNF: Nitrogen oxides (NO_x): mostly NO_2 and NO resulting from the breakdown of nitrate; The burning of citric acid produces carbon dioxide (CO_2). Vaporized water (H_2O): Produced by the hydrolysis of nitrates in metal, citric acid, and water; Oxygen gas (O_2): Byproducts of nitrate breakdown; the breakdown of ammonia in water produces ammonia (NH_3). Unburned citric acid leaves behind carbon monoxide (CO) as a byproduct. Consequently, adequate ventilation and safety measures are essential during the experiment due to the hazardous properties of NO_x and NH_3 [32].

2.2 Samples characterizations

The structural delineation of the CZNF ferrite specimens was conducted through X-ray diffraction (XRD) employing a Philips X'Pert diffractometer, utilizing a $\text{Cu-K}\alpha$ radiation source ($\lambda=1.54056 \text{ \AA}$) across a 2θ range of 10–80 degrees. Structural refinements were performed utilizing the Fullprof software, known for its high accuracy and precision in determining lattice and structural parameters. Using a HR-TEM, specifically a JEOL JEM2100 running at a 200 kV acceleration voltage, we were able to analyze the sensitized nanoferrite samples' shape and particle size. Samples were meticulously placed onto Cu TEM mesh grids covered with carbon through the process of drop-casting a dilute solution of the nanomaterials in ethyl alcohol. The analysis of particle size distribution, derived from TEM images, was performed utilizing ImageJ software. All produced samples' optical energy gap was evaluated using a JASCO model V-570 UV–vis spectrophotometer, which operates from 200 to 2500 nm.

2.3 Biological evaluation on maize seed germination

2.3.1 Preparation of nanoferrite suspensions

Different Ni-substituted Co-Zn spinel nanoferrites were suspended at a concentration of 100 $\mu\text{g/mL}$ in deionized and distilled water. The suspensions were sonicated for four hours in a bath sonicator (Branson's Model B200 ultrasonic) to make sure that these nanoferrites were dispersed and to avoid agglomeration and aggregation.

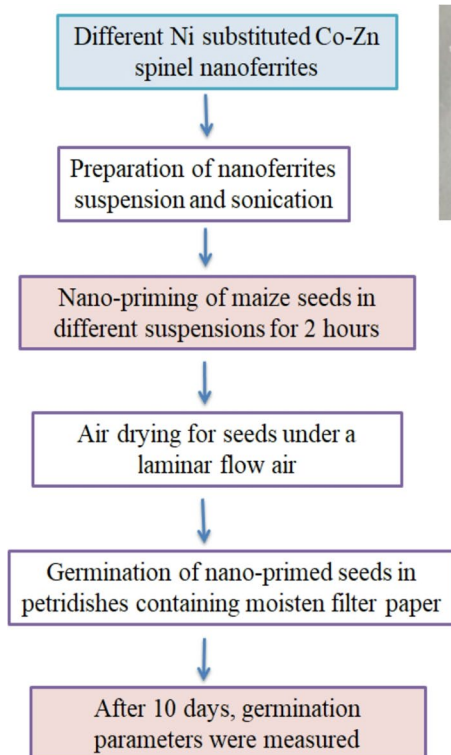
Table 1 Treatments used in the biological evaluation of Ni substituted Co-Zn spinel nanoferrites on maize seed germination

Treatments	Explanation
Control	Without nano-priming
T1	Nano-primed with $\text{Co}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ (CZNF0)
T2	Nano-primed with $\text{Co}_{0.5}\text{Zn}_{0.4}\text{Ni}_{0.1}\text{Fe}_2\text{O}_4$ (CZNF1)
T3	Nano-primed with $\text{Co}_{0.5}\text{Zn}_{0.3}\text{Ni}_{0.2}\text{Fe}_2\text{O}_4$ (CZNF2)
T4	Nano-primed with $\text{Co}_{0.5}\text{Zn}_{0.2}\text{Ni}_{0.3}\text{Fe}_2\text{O}_4$ (CZNF3)
T5	Nano-primed with $\text{Co}_{0.5}\text{Zn}_{0.1}\text{Ni}_{0.4}\text{Fe}_2\text{O}_4$ (CZNF4)
T6	Nano-primed with $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ (CZNF5)

2.3.2 Seed germination assay

• Seeds, nano-priming and experimental condition.

To assess the impact of Ni-substituted Co-Zn spinel nanoferrites on seed germination of one important crop, *Zea mays*, seeds were surface sterilized using 20% sodium hypochlorite solution for 15 min within a laminar flow cabinet that had a horizontal flow of sterile air. Seeds were nano-primed by soaking in a suspension of 100 $\mu\text{g/mL}$ from different Ni-substituted Co-Zn spinel nanoferrites (Table 1). Control treatment did not receive any priming suspension. For each type of seeds, 10 surface sterilized seeds were distributed in petridishes and allowed to germinate in vitro. Petridishes were kept in an incubator with a 16-h photoperiod and a controlled temperature of 27 °C. Figure 1 illustrates the scheme of the methodological steps of the germination bioassay. Germination percent was calculated after 3, 7 and 10 days. After 10 days, seedling growth parameters were measured.

Fig. 1 The scheme of the methodological steps of the germination bioassay

• Seed germination and early seedling growth.

Every day, the number of germinated seeds was observed. The germination percentage (GP) was calculated after 3, 7 and 10 days based on the formula below:

$$GP = (\text{number of seed germinated} / \text{total seed number}) \times 100$$

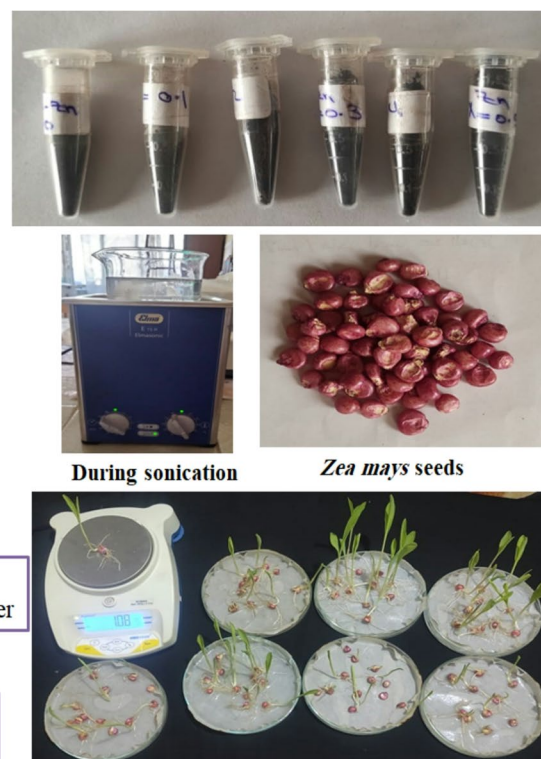
In each treatment, three normal seedlings were chosen randomly, and their root and shoot lengths were measured and reported in centimeters (cm). The length of the seedlings was also measured using the same three typical seedlings. The seedlings' mean fresh weight (fwt) was measured and their dry weight (dwt) was recorded in grams (g) after keeping for a whole day at 70 °C in a hot air oven. The seedling vigor indices were computed using the GP, seedling length, and seedling dwt data. Consistent with [33], the seedling length vigor index (SLVI) and seedling weight vigor index (SWVI) for each treatment were computed using the formulas below:

$$SLVI = [\text{seedling length (cm)} \times GP (\%)]$$

$$SWVI = [\text{seedling dwt (mg)} \times GP (\%)]$$

2.3.3 Statistical analysis

The mean value of observations recorded on different parameters of seed germination assay was subjected to statistical analysis. One-way ANOVA was used to analyze the



data, followed by Duncan's post-hoc test to detect significant differences between treatments ($p < 0.05$). All statistical analyses were achieved with SPSS software.

3 Results and discussion

3.1 Structural analysis

The structure of ferrite specimens must possess a single-phase cubic configuration in order to be used in magnetic and electrical transport applications. Consequently, examining the structural possessions of ferrite is crucial. The XRD technique was employed to analyze the $\text{Co}_{0.5}\text{Zn}_{0.5-x}\text{Ni}_x\text{Fe}_2\text{O}_4$ samples, which were characterized by six specific x values: 0.0, 0.1, 0.2, 0.3, 0.4, and 0.5. The CZNF material exhibits diffraction peaks at (111), (220), (311), (222), (400), (422), (511), and (440) within the specified angles. These observations correspond with the reference data found in (PDF card no. 01–085-5592), confirming the presence of the spinel phase, as indicated by the (311) diffraction peak illustrated in Fig. 2. Furthermore, CZNF samples crystallize in a single-phase cubic structure classified under the Fd-3 m space group, indicating a highly symmetrical crystalline arrangement owing to the lack of anticipated diffraction peaks. A slight deviation towards increased diffraction angles is observed for the majority of peaks. This is because dopant elements Ni^{2+} have smaller ions than the host element (Zn^{2+}). The displacement of the (311) peak signifies structural alteration of the synthesized materials.

Figure 3 illustrates the Rietveld refinement analysis pertaining to CZNF NPs at different level of doping. The XRD pattern of the ferrite underwent analysis through the Rietveld refinement technique, utilizing the Fullprof software. In the graph, the estimated data is signified by a red solid line (Y_{cal}), while the experimental intensities are represented by black hollow circles (Y_{obs}). The bottom line illustrates the disparity between the calculated and measured values ($Y_{\text{obs}} - Y_{\text{alc}}$). The experimental XRD patterns closely aligned with the refinement analysis, exhibiting negligible variation. The analysis of Rietveld refinement encompassed several parameters, including the lattice parameter (a_{exp}), expected R value (R_{exp}), profile factor (R_p), and weighted profile factor (R_{wp}) and (χ^2) the reliability factors, which are tabulated in Table 2. You can assess the fit quality using the dependability factor numbers. The fit is accurate, as seen by the small values of the minimal R -factor and χ^2 . Figure 4 clearly depicts the change in lattice constant (a) in relation to the Ni^{2+} concentration. The addition of nickel content up to $x = 0.3$ (that is, zinc content is greater than or equal to 0.2) results in a small rise in the lattice parameter. The presence of the Zn^{2+} ion in the tetrahedral location, along with a considerable amount of Co and Zn cations in the octahedral site, may account for this phenomenon. Fe^{3+} is thus compelled to move to the A- position. Due to the fact that Fe^{3+} ions possess a reduced ionic radius (0.645 Å), they tend to fill the A- positions rather than bigger divalent cations. As can be seen from the data, this causes the lattice constants to rise. A notable reduction in the lattice parameter is noted as the Ni concentration rises from $x = 0.4$ to 0.5 (where Zn content

Fig. 2 XRD patterns of $\text{Co}_{0.5}\text{Zn}_{0.5-x}\text{Ni}_x\text{Fe}_2\text{O}_4$ NPs with different compositions ($x = 0.0, 0.1, 0.2, 0.3, 0.4$ and 0.5)

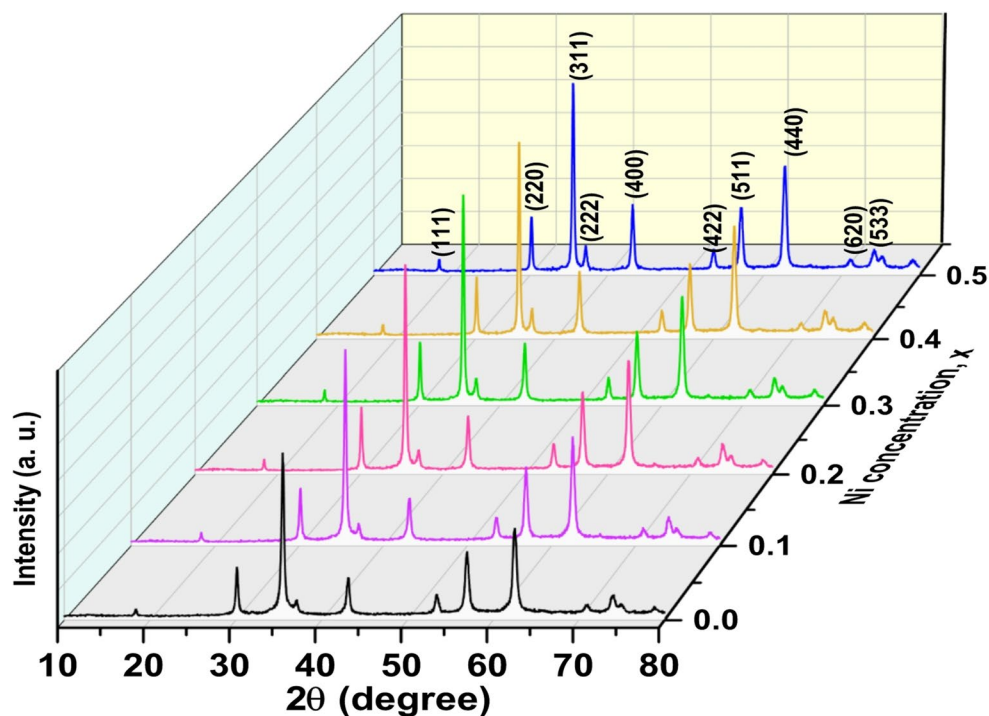
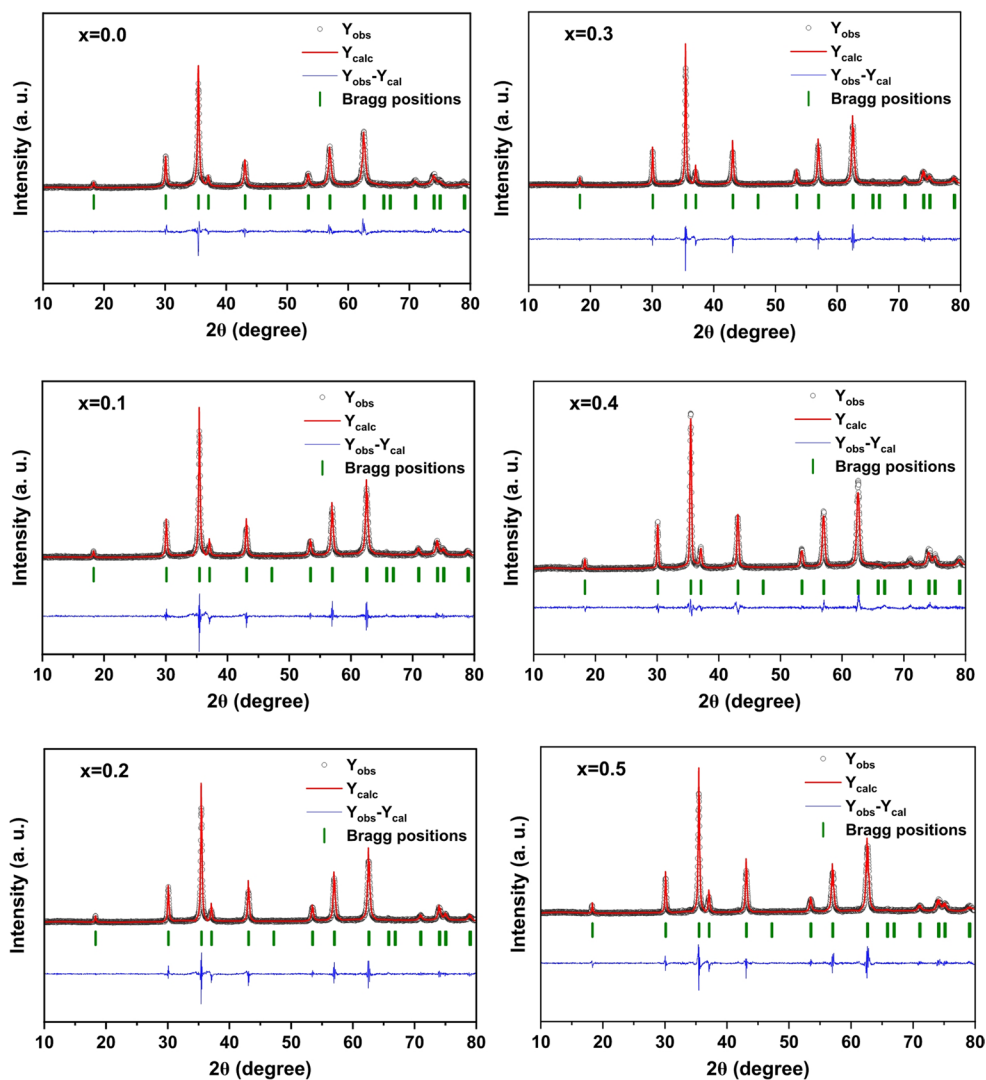


Fig. 3 Rietveld refinement patterns of CZNF NPs**Table 2** The reliability factors of XRD data from Rietveld refinement of Ni doped Co-Zn ferrites with $x = 0, 0.1, 0.2, 0.3, 0.4$ and 0.5

Parameters	$x=0.0$	$x=0.1$	$x=0.2$	$x=0.3$	$x=0.4$	$x=0.5$
Rexp	8.3	8.13	8.15	8.2	7.95	7.91
Rp	9.4	9.53	9.62	9.55	8.85	9.99
Rwp	11.9	12.3	12.5	12.2	11.6	12.5
DW-Stat	0.834	1.12	1.09	1.17	0.97	1.03
χ^2 (goodness of fit)	2.04	2.28	2.37	2.20	2.12	2.5

stays at or below 0.1 and Zn^{2+} ions are contained in tetrahedral sites). Also, the reduction in ‘a’ is associated with the gradual inclusion of nickel ions, which are lesser with an ionic radius of 0.72 Å, substituting the superior Zn^{2+} ions (0.80 Å) in the Ni^{2+} doped Co-Zn ferrite system. According to Vegard’s law, the lattice constant is reduced when Ni^{2+} is present because its smaller ionic radius causes a modest shrinkage of the unit cell [34]. Furthermore, upon the complete substitution of zinc ions with nickel ($x=0.5$), a reduction in the lattice parameters is anticipated. This reduction could be linked to the spinel structure’s configurational

entropy decreasing as a result of fewer ion kinds [35]. An analogous finding documented by other workers [36]. These variations in lattice parameters are key evidence of modifications to the material’s structural characteristics.

The typical size of crystallites is calculated by applying Debye-Scherrer’s equation, relying on the broadening noted in the X-ray line, especially at the prominent (311) diffraction peak [37];

$$t_{Sch} = \frac{K\lambda}{\beta_{hkl}\cos\theta_{hkl}} \quad (1)$$

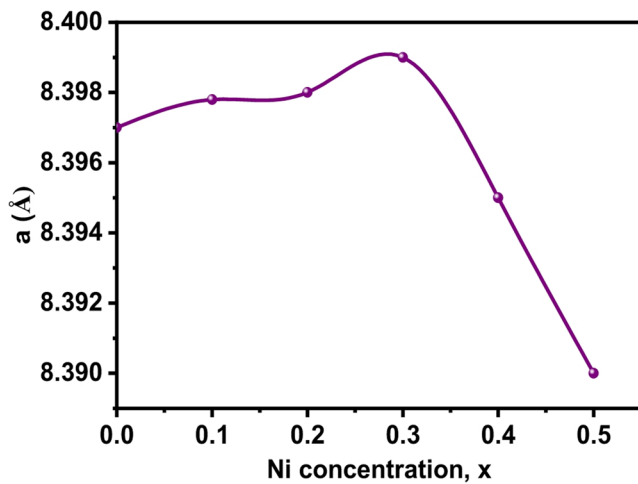


Fig. 4 Variation of experimental lattice parameter with nickel substitution

In this context, t_{sch} denotes the crystallite size, K indicates the Scherrer constant valued at 0.90, λ signifies the wavelength of X-ray radiation, hkl indicates the Miller indices, which serve as a perpendicular measurement to the planes, β refers to the full width at half maximum (FWHM) of the diffraction peak, where θ is the angle of diffraction. The average crystallite size was ascertained through the analysis of peak broadening at (311) and the application of the Debye-Scherrer equation. Table 3 also encompasses the values pertaining to the crystallite size (t_{sch}). The t_{sch} values were measured within the range of 42.57–55.63 nm, indicating their nanoscale characteristics. Figure 5 shows that when the dopant is added, the crystallite size first gets bigger, but as the concentration of the dopant goes up, the crystallite size gets smaller. The findings align with those observed for the lattice parameters, indicating that an intensification in the lattice parameter could result in an elevation of the total crystallite sizes, and conversely. The observed augmentation in crystallite size with the elevation of Ni content

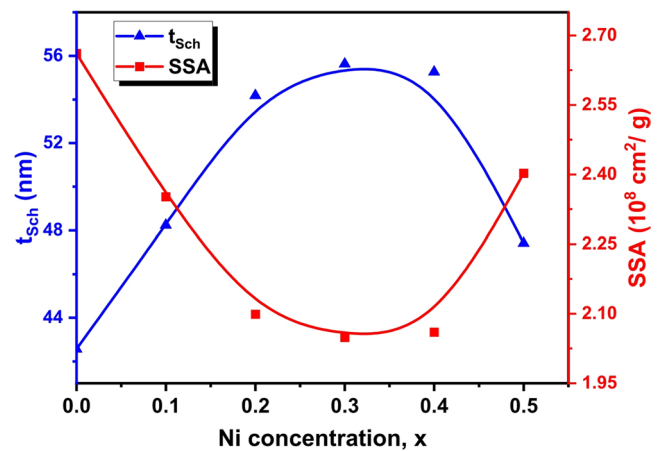


Fig. 5 Crystallite size (t_{sch}), and specific surface area (SSA) versus Ni content in Co-Zn nanoferrites

($x \leq 0.3$) can be elucidated through the electronic configuration of Ni^{2+} ($3d^8$) and propensity to engage with ligands and oxygen anions, in contrast to Zn^{2+} ($3d^{10}$), which possesses a fully filled electronic configuration. Moreover, it is noteworthy that Co^{2+} and Ni^{2+} ions in the spinel structure exhibit a pronounced affinity for octahedral sites, whereas Zn^{2+} ions demonstrate a comparable high preference for tetrahedral sites. In the interim, Ni^{2+} ions (≤ 0.3) and Zn^{2+} are located among both A- and B- positions. Consequently, the development of Ni-ferrite is more advantageous, resulting in a modest increase in crystallite size with rising Ni concentration due to site preferences. On the other hand, the reduction in crystallite size for $x > 0.3$ could be associated with the widening of the XRD lines. The observed broadening contributes to the increase in the full width at half maximum (FWHM) of the strongest peak, while the crystallite size decreases in accordance with Scherrer's formula. Moreover, the rationale for the observed trend of diminishing crystallite size can be ascribed to the decreased ionic radius of Ni^{2+} ions in contrast to Zn^{2+} .

Table 3 Structural, optical parameters obtained from XRD and UV-DRS results of the synthesized CZNF NPs

Parameters	x=0.0	x=0.1	x=0.2	x=0.3	x=0.4	x=0.5
t_{sch} (nm)	42.57	48.24	54.18	55.63	55.26	47.22
t_{WH} (nm)	60.81	82.53	97.64	98.33	91.22	90.03
ε (10^{-4})	8.19	8.60	10.40	8.60	10.40	12.00
SSA (10^8 cm ² /g)	2.66	2.35	2.10	2.05	2.06	2.40
d_x (g. cm ⁻³)	5.34	5.32	5.30	5.29	5.28	5.27
d_b (g. cm ⁻³)	1.86	2.07	2.32	2.56	2.52	2.43
P (%)	64.83	60.85	56.03	51.30	52.20	53.83
r_A (Å)	0.5853	0.5854	0.5855	0.5857	0.5848	0.5837
r_B (Å)	0.7288	0.7291	0.7291	0.7294	0.7284	0.7272
L_{AE} (Å)	3.1113	3.1116	3.1117	3.1120	3.1106	3.1087
L_{BE} (Å)	2.8263	2.8266	2.8266	2.8269	2.8256	2.8239
L_{BUE} (Å)	2.9705	2.9708	2.9709	2.9712	2.9698	2.9680
d_A (Å)	3.6360	3.6364	3.6364	3.6369	3.6351	3.6329
d_B (Å)	2.9688	2.9691	2.9691	2.9695	2.9681	2.9663

The Scherrer method serves as a valuable tool for ascertaining crystallite size; however, it falls short in assessing the contributions of instrumental and micro-strain broadening. The Williamson-Hall (W-H) approach offers enhanced precision in estimating crystallite size and micro-strain broadening. Micro strain incorporates the consequences of structural flaws such as intergrowths, twin boundaries, stacking faults, and dislocations. Equation (2) was employed to ascertain the comprehensive broadening of reflection attributed to size and micro strain in a precise XRD peak characterized by the (hkl) value [38]:

$$\beta_{hkl}\cos\theta_{hkl} = 4\epsilon\sin\theta_{hkl} + \frac{K\lambda}{t_{WH}} \quad (2)$$

In this context, t_{WH} represents the particle size measured in nanometres, while ϵ denotes the lattice strain. As shown in Fig. 6, a linear fit can be used to find the size of the crystallite and the lattice strain. The slope represents the average strain, whereas the y-axis intersection denotes the dimensions of the nanocrystals. Table 3 provides a summary of the crystallite size and microstrain values. The W-H graphs in Fig. 6 exhibit positive slopes across wholly ratios, indicating the existence of tensile strain within the lattice. This tensile strain results in the elongation of particles, suggesting that these materials experience strain [39]. A discrepancy is noted between the values of t_{sch} and t_{WH} , which is because the estimate of effective crystallite size via the W-H plot is deemed more suitable, given that strain components are

associated with contact stress, grain boundaries, and coherency stress.

The specific surface area, or SSA, which quantifies a substance's total surface area relative to its mass, was determined using the following formula [40]:

$$SSA = \frac{6000}{d_x t_{sch}} \quad (3)$$

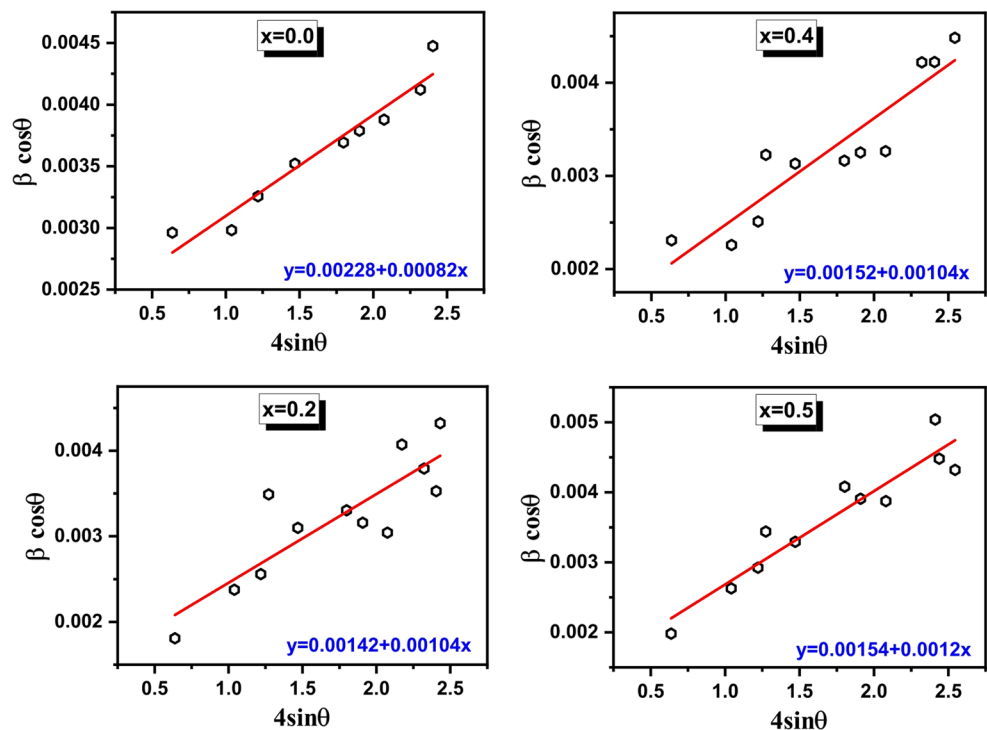
As shown in Fig. 5, the value shows a varied trend with respect to crystallite size due to their inverse connection; it initially drops with adding nickel up to $x=0.3$, and then upsurges with successive increments. Accordingly, these samples can indicate the existence of surface-level imbalances and the foremost agglomerated particles. The CZNF3 composition exhibits the minimal surface area and the maximal crystallite size observed in the study. The substantial values of S suggest the nanostructured nature of the ferrite, aligning with the determined crystallite size [41].

Furthermore, the X-ray density of the manufactured $\text{Co}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$, following the incorporation of Ni, was evaluated using the subsequent formula [37]:

$$d_x = \frac{ZM_{wt}}{N_A a^3} \quad (4)$$

In this context, Z signifies spinel ferrite's atoms or molecules per unit cell, where $Z=8$; M_{wt} refers to CZNF's molecular weight, and N_A symbolizes Avogadro's numeral. Table 3

Fig. 6 Williamson-Hall plots of CZNF0 ($x=0.0$), CZNF2 ($x=0.2$), CZNF4 ($x=0.4$) and CZNF5 ($x=0.5$) samples



demonstrate that the X-ray density values went down as the Ni content went up in the CZNF NPs. The determination of d_x is contingent upon the lattice parameter of the material as well as its atomic weight. Molar masses can be used to explain the drop in d_x that occurs as the concentration of Ni^{2+} in nanoparticles increases. The molar mass of substituted Ni, at 58.69 amu, is less than that of Zn, which is 65.39 amu. Consequently, its integration into nanoparticles leads to a lessening of the molecular weight of the specimens, which in turn causes deterioration in X-ray density.

The experimental bulk density is premeditated by assuming the pellets have a circular shape, utilizing the following relation:

$$d_b = \frac{m}{\pi r^2 h} \quad (5)$$

In this context, d_b denotes bulk density, m signifies the mass of the pellet, h symbolizes thickness and r indicates the radius of the pellet. The data presented in Table 3; Fig. 7 clearly indicates that the bulk density variation among the six co-doped samples lacks a consistent pattern. The initial increase is observed from 1.86 g/cm³ to 2.56 g/cm³ as x transitions from 0.0 to 0.3, followed by a decrease to 2.43 g/cm³ at $x=0.5$. This variable alteration could be linked to the differences in crystallite size resulting from nickel substitution [42]. An alternative explanation for the increase in 'bulk density' values with a rise in Ni^{2+} ions could be because Ni^{2+} ions have a higher density (8.90 g. cm⁻³) compared to that of Zn^{2+} ions (7.14 g. cm⁻³). Intergranular or intra-granular porosity brought on by irregular grain development could be the source of the density decrease [43]. Notably, the computed density of the X-ray exceeds the bulk density. The divergence among the theoretical and bulk densities suggests that the particles possess pores within their crystal

structure, a characteristic that arises during the manufacturing process [44].

The assessment of pore existence within the crystal structure is carried out by calculating the percentage porosity, referred to as 'P%' [45],

$$P\% = 100 \left(1 - \frac{d_b}{d_x} \right) \quad (6)$$

When nickel is utilized as a dopant in place of zinc ($x \leq 0.3$), the percentage porosity 'P%' diminishes. However, when the nickel level surpasses ($x = 0.3$), the percentage porosity increases as displayed in Fig. 7. The observed performance of P % compared to x is anticipated, given the composition's porosity mirrors their apparent density. The alteration in porosity may be analyzed in relation to crystallite size, as porosity is inversely correlated with crystallite size. Such a phenomenon might take place if, for example, the size of the crystallites increases while the grain boundaries of the particles diminish, leading to a decrease in porosity.

3.2 Structural parameters for tetrahedral and octahedral sites

The mean cationic radius at sites A (r_A) and B (r_B) can be expressed using the radius value of oxygen anions, $r_O = 1.32$ Å, through the succeeding formula:

$$r_A = \left(u - \frac{1}{4} \right) a\sqrt{3} - R_o \quad (7)$$

$$r_B = \left(\frac{5}{8} - u \right) a - R_o \quad (8)$$

The displacement parameter of oxygen ions associated with the A site, denoted as u , (~ 0.381 Å). The values were derived using the previously mentioned formulas, and they can be displayed in Table 3. The ionic radii (r_A and r_B) exhibited a non-monotonic pattern, firstly increasing because of augmented lattice parameters and cation rearrangement, followed by a decrease at elevated doping levels, likely attributable to unit cell contraction. In the tetrahedral site A, nominally inhabited by zinc and ferric ions in the CZNF formula, the ionic radius of Zn^{2+} is 0.80 Å, while that of Fe^{3+} is 0.645 Å, resulting in reduced overall r_A values. Conversely, in the optimal inverse spinel structure, the B octahedral site is filled by Ni^{2+} with an ionic radius of 0.72 Å, Co^{2+} at 0.73 Å, and Fe^{3+} at 0.645 Å, respectively, leading to larger r_B values. Due to their size differences, the ionic radii values for the B-site exceed those of the A-site [46].

The region behind the ions in A- and B- locations is where ions swap locations due to exchange forces, referred

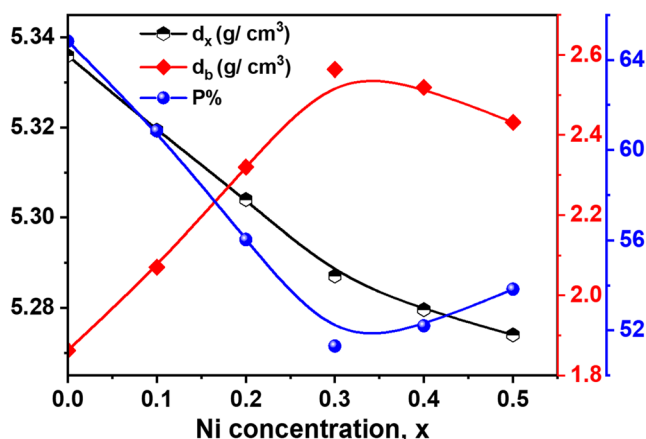


Fig. 7 The effect of Ni^{2+} ions on X-ray density, bulk density and percentage porosity

to as the tetrahedral shared length (L_{AE}) and octahedral shared length (L_{AB}), which were computed using the following formulas [47]:

$$L_{AE} = \sqrt{2a} \left(2u - \frac{1}{2} \right) \quad (9)$$

$$L_{BE} = \sqrt{2a} (1 - 2u) \quad (10)$$

The octahedral sites possess an unshared space between metal ions and ferrite oxygen ions, referred to as the octahedral unshared length (L_{BUE}), which was determined using Eq. (11):

$$L_{BUE} = a \left(4u^2 - 3u + \frac{11}{16} \right)^{1/2} \quad (11)$$

Table 3 indicates that the shared edge lengths of the A-site and B-site (L_{AE} and L_{BE}) as well as the unshared octahedral edge length (L_{BUE}) demonstrate a non-linear variation with elevated Ni^{2+} content, showing a noticeable peak at $x=0.3$ that is dependent on the doping content (x) and the lattice parameters. The alterations in these factors corresponded to variations in the lattice parameters of the constituents.

The tetrahedral (A-site) and octahedral (B-site) configurations' ion hopping lengths, denoted as " d_A " and " d_B ," were determined utilizing the lattice constant (a) values along with the subsequent Eq. [3]:

$$d_A = a\sqrt{3}/4 \quad (12)$$

$$d_B = a\sqrt{2}/4 \quad (13)$$

The numbers acquired for the hopping lengths d_A and d_B are systematically presented in Table 3. Ni^{2+} ion concentration obviously influences the spacing between magnetic ions. Upon elevating the Ni concentration from $x=0.0$ to 0.3, it is discovered that these " d_A and d_B " values increase as well. Subsequently, a reverse trend is observed, characterized by a continuous increase in the dopant levels from $x=0.4$ to 0.5. According to the variations that were witnessed in the lattice constant, the trend that was established is consistent. Since d_A is greater than d_B , as shown in Table 3, it is more

likely that ions in tetrahedral A and octahedral B sites will not hop electrons than ions in octahedral B-B sites.

3.3 Interionic distances

Utilizing the estimated lattice parameters, one can derive the cation-cation (Me-Me) distances (b , c , d , e , f) through the application of the subsequent formula [3], with the computed values illustrated in Table 4.

Me-Me cation-cation,

$$b = \frac{a\sqrt{2}}{4} \quad (14)$$

$$c = \frac{a\sqrt{11}}{8} \quad (15)$$

$$d = \frac{a\sqrt{3}}{4} \quad (16)$$

$$e = \frac{3\sqrt{3}a}{8} \quad (17)$$

$$f = \frac{a\sqrt{6}}{4} \quad (18)$$

The values of d exceed those of b , attributable to less effective covalent bonding in the A-location compared to the B-location. All of the composites that were studied retain this trend.

In a similar vein, one can ascertain the distance between cations and anions by utilizing the established framework of Stanley relations:

Me-O cation-anion:

$$p = a \left(\frac{1}{2} - u \right) \quad (19)$$

$$q = a \left(u - \frac{1}{8} \right) \sqrt{3} \quad (20)$$

$$r = a \left(u - \frac{1}{4} \right) \sqrt{11} \quad (21)$$

Table 4 Estimated distances between cation-anion (p , q , r , and s) and the cation-cation distances (b , c , d , e , and f) of $\text{Co}_{0.5}\text{Zn}_{0.5-x}\text{Ni}_x\text{Fe}_2\text{O}_4$ NPs

x	p	q	r	s	b	c	d	e	f
0.0	2.0489	1.9053	3.6483	3.6651	2.9688	3.4812	3.6360	5.4540	5.1421
0.1	2.0491	1.9054	3.6487	3.6654	2.9691	3.4815	3.6364	5.4545	5.1426
0.2	2.0491	1.9055	3.6487	3.6655	2.9691	3.4816	3.6364	5.4547	5.1427
0.3	2.0494	1.9057	3.6492	3.6660	2.9695	3.4820	3.6369	5.4553	5.1433
0.4	2.0484	1.9048	3.6474	3.6642	2.9681	3.4804	3.6351	5.4527	5.1409
0.5	2.0472	1.9037	3.6453	3.6620	2.9663	3.4783	3.6330	5.4495	5.1378

$$s = \frac{a}{3} \left(u + \frac{1}{2} \right) \sqrt{3} \quad (22)$$

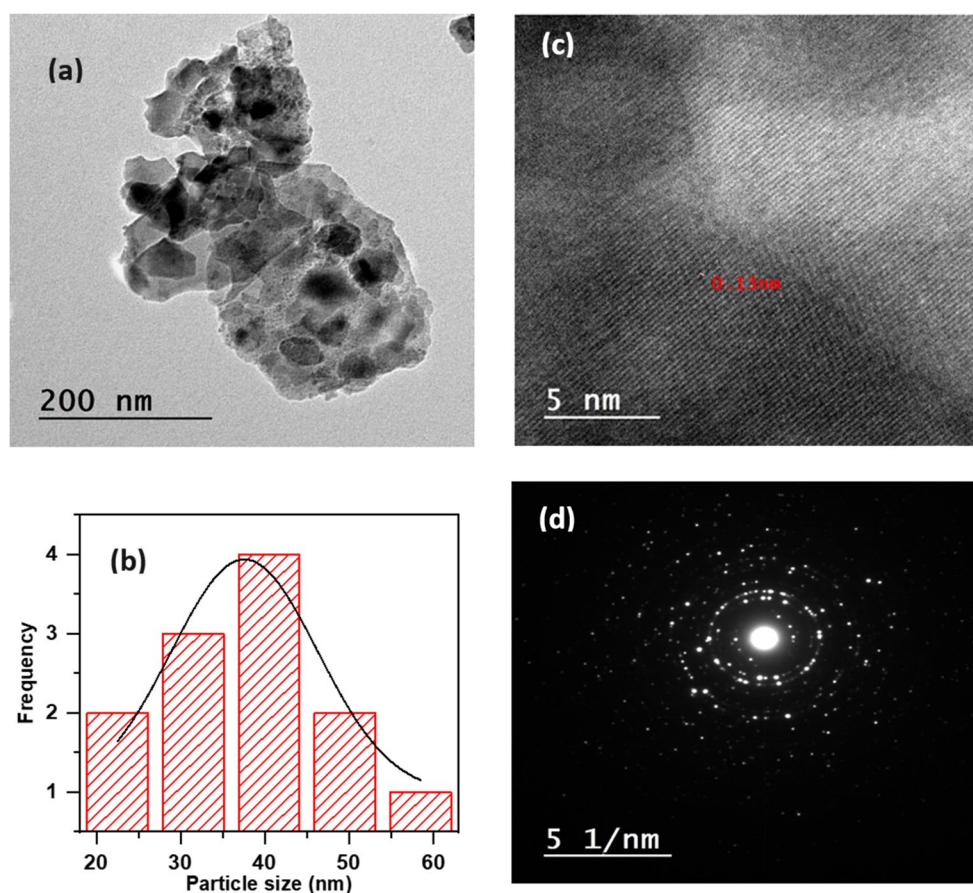
The distances among cations and anions, in addition to those between cations, increased to ($x=0.3$) before subsequently going back down when the Ni^{2+} content rose, exhibiting a variation akin to that of the lattice parameter.

3.4 High-resolution transmission electron microscopy characterization

The synthesized CZNF3 sample was examined by HR-TEM to see how big the particles were and what shape they were. The corresponding HR-TEM image of sample CZNF3 is presented in Fig. 8. The particles have nearly spherical forms with some agglomeration caused by magnetic contact between nanoparticles. The existence of certain agglomerations can improve the magnetic characteristics of the substance, including saturation magnetization and coercivity. Nonetheless, the increased level of agglomeration results in a diminished effective surface area, consequently reducing the electric and electrochemical possessions of the material. The citrate method of synthesis leads to uniform nucleation, generally producing a narrow distribution of particle sizes. The control of particle size and agglomeration is affected by

various critical parameters, containing the amount of precursor, temperature, pH, and the application of surfactants. It is possible to have more control over how fast the particles grow thanks to the homogenous nucleation that takes place throughout the citrate process. In order to restrict particulate growth and prevent agglomeration, surfactants like citric acid or oleic acid are frequently implemented. Reducing agglomeration may be possible by optimizing surfactant content. These agents stabilize nanoparticles by binding to their surfaces, thereby preventing them from adhering to one another as a result of magnetic or van der Waals forces. These forces are so powerful that they cause agglomeration in magnetic nanoparticles. Nevertheless, by fine-tuning parameters like the amount of precursor, temperature, pH, and surfactant sort and concentration, the substance's possessions can be improved. The histogram in Fig. 8b presents the dispersal of particle size for the CZNF3 specimen, which has been fitted with a Gaussian function. The histogram indicates that the sample is nanometer-sized, with a mean particle size of 37.59 nm. The particle size measured by TEM differs the one obtained by XRD, since TEM is capable of analyzing morphological characteristics, whereas XRD solely delivers insights regarding the crystallite component [48]. Utilizing HRTEM and selected area electron diffraction (SAED) patterns, we successfully

Fig. 8 (a) TEM image for CZNF3 NPs, (b) Distribution of particle size, (c) lattice fringe and (d) SAED pattern of the sample



acquired detailed information regarding the lattice fringes and crystalline phases of the samples. The lattice fringes perceived in the HRTEM, portrayed in Fig. 8c, affirm the emergence of the CZN ferrite phase, as evidenced by the distance of 0.13 nm between adjacent fringes, which aligns with the (442) crystalline planes. Figure 8d displays the SAED decoration of CNZF3 spinel ferrites, revealing vivid and intense rings composed of distinct spots. The SAED patterns are indexed through the calculation of d-values, which involves measuring the distance between the central spot and the examined ring. We noted the presence of various diffraction rings that correspond to the planes (220), (311), (400), (511), and (440), thereby affirming the spinel crystal structure. The presence of small spots arranged in a ring, in conjunction with the overlaid bright spots, underscores the nanocrystalline characteristics of the analyzed samples. The computed d-values from the SAED pattern exhibit a commendable alignment with those derived from Bragg's law, thereby corroborating the majority of the planes identified in the XRD pattern.

3.5 Optical properties

Examining the optical properties of ferrites through UV-visible spectroscopy is essential for determining the optical energy gap, which is a crucial element for numerous possible uses [49]. Scientists can determine the band gap energy, which is the energy required for electrons to jump from the valence band to the conduction band, by examining absorption spectra in the UV-visible region. Understanding this attribute is essential for the advancement of photodetectors and photovoltaics, as it directly influences the substance's ability to effectively utilize and absorb light. Furthermore, the energy gap provides critical insights into the electrical structure and potential electronic transitions within the material. This information is instrumental in the advancement and improvement of ferrites for revolutionary requests in areas such as optoelectronics, catalysis, and sensors. As illustrated in Fig. 9, the produced ferrites' diffuse reflectance spectra (DRS) were taken between 200 and 2500 nm. Indeed, DRS is an effective technique for examining opaque objects. Absorptions in the visible and ultraviolet light bands (which scan from 250 to 800 nm) are broad and intense in the spectra of CZF and co-doped CZF. As a result, these synthesized materials have the potential to be utilized as effective photocatalysts in a variety of photocatalysis processes that make use of both naturally occurring sunlight and ultraviolet radiation. Adding Ni dopants marginally increased the absorption intensity, as shown in Fig. 9. Absorption of ultraviolet and visible light is thought to be triggered by electrons being stimulated from the valence band of the O-2p level to the conduction band of the Fe-3d level [50]. Kubelka-Munk

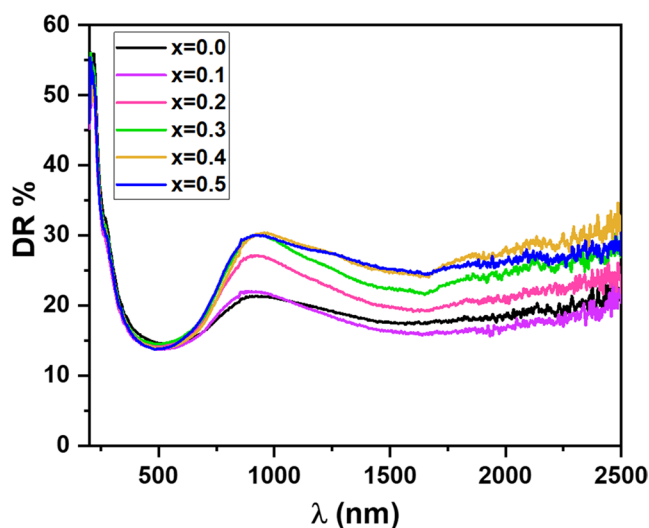


Fig. 9 UV-Visible diffuse reflectance spectra of $\text{Co}_{0.5}\text{Zn}_{0.5-x}\text{Ni}_x\text{Fe}_2\text{O}_4$ ferrites

indicated that for a semi-infinite thick material, which reflects equal quantities of light regardless of the substrate's reflectance, the absorption (α) and scattering (S) coefficients can be ascertained from the specimen's diffuse reflectance (R) using the ensuing relation [51, 52]:

$$F(R) = \frac{\alpha}{S} = \frac{(1-R)^2}{2R} \quad (23)$$

The modified Tauc's equation [53] has been utilized to evaluate the impact of Ni/Zn substitution on the energy band gap (E_g) of CZNf samples.

$$F(R) h\nu = A(h\nu - E_g)^n \quad (24)$$

Inside this particular framework, the band-gap energy is embodied by E_g , the photon energy is indicated by $h\nu$, and A measures disorder within the compound. When $n=1/2$, optical transitions are direct, and when $n=2$, they are indirect. The type of optical transition that occurs is controlled by the proponent n . By plotting $(F(R)h\nu)^2$ versus $h\nu$ for wholly CZNf x NPs, the extrapolation of the lined segments $(F(R)h\nu)^2 = 0$ facilitates the determination of the direct E_g values. According to the data presented in Fig. 10, the band gaps (E_g) for CZNf0, CZNf1, CZNf2, CZNf 3, CZNf 4, and CZNf 5 are calculated to be 1.451, 1.440, 1.441, 1.480, 1.542, 1.589 eV, the respective values. These values fall within the typical semiconductor band gap range and the variation with Ni content is shown in Fig. 11. The band gap values were influenced by an assortment of influences including the charging carrier concentration, the size of the crystallite, phase purity, lattice strain, and lattice parameters [54]. The observed pattern is influenced by two primary factors:

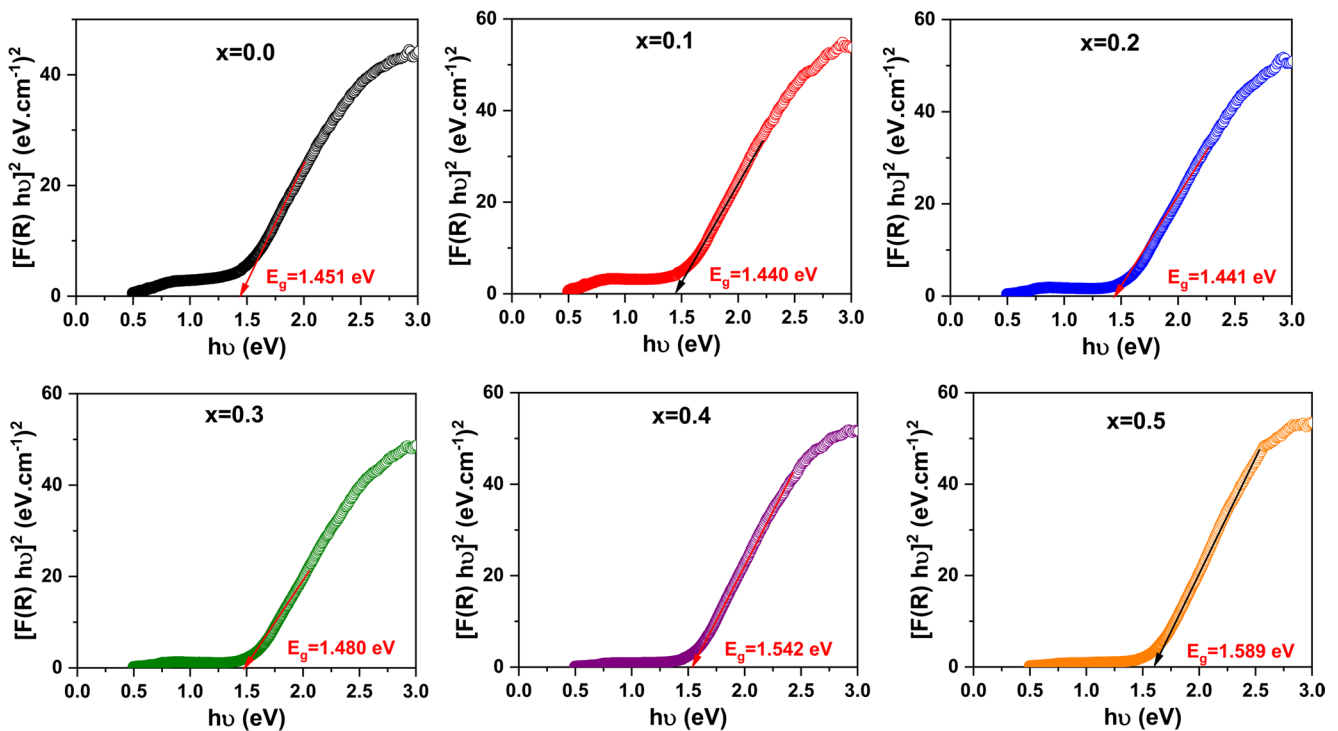


Fig. 10 Tauc's plot of $\text{Co}_{0.5}\text{Zn}_{0.5-x}\text{Ni}_x\text{Fe}_2\text{O}_4$ ($0 \leq x \leq 0.5$) nanoferrites

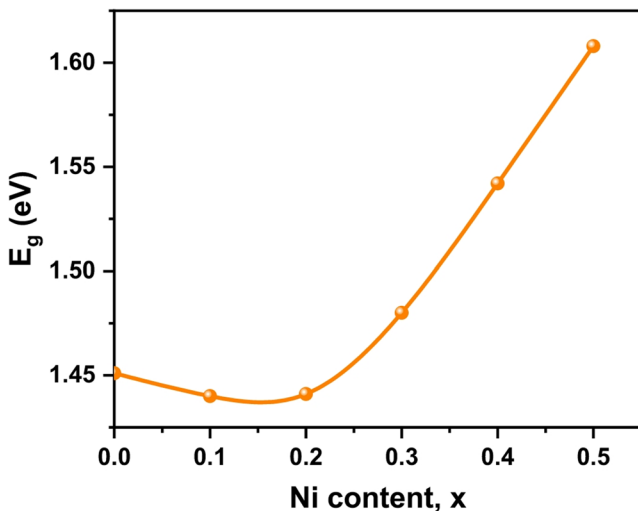


Fig. 11 Change in energy gap with nickel content (x)

the variation in crystallite size and the presence of localized electronic states within the substance. These elements contribute to the fluctuation in E_g as nickel ions are introduced. It is possible to attribute the reverse association between E_g numbers and the size of the crystallite to the influence of quantum size effects [55]. The noted reduction in E_g for $\text{Co}_{0.5}\text{Zn}_{0.4}\text{Ni}_{0.1}\text{Fe}_2\text{O}_4$ and $\text{Co}_{0.5}\text{Zn}_{0.3}\text{Ni}_{0.2}\text{Fe}_2\text{O}_4$, when compared to $\text{Co}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$, can be accredited to the enhancement in grain size. This increase results in a reduction in the quantity of grain boundaries, consequently diminishing the

scattering effects of carriers on the band gap. Furthermore, the observed reduction in the E_g could be attributed to the interaction amongst the localized d-electrons of Ni^{2+} ions and the band electrons of $\text{Co}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$. Consequently, the incorporation of Ni^{2+} might have diminished the energy gap by creating sub-bands inside the energy band gaps, which eventually align with the conduction band to establish a cohesive band [56]. It has been shown that there is a link between crystallite size and band gap energy. This means that when the size of the crystallite gets smaller, the band gap energy usually goes up. This could be attributed to the electron-hole pair coming very close together as the crystallite size approaches the nanoscale, where entirely components consist of a limited numeral of atoms. Consequently, the Coulombic force is too important to ignore for very long, which would cause the total amount of kinetic energy to rise [9]. Therefore, the observed increase in band gap with a reduction in crystallite size may be linked to the nano crystalline characteristics of the specimens. Furthermore, the electronic band structure is further affected by lattice distortions and modifications in bond lengths resulting from Ni^{2+} doping.

3.6 Biological evaluation on maize seed germination

One of the critical stages of plant seedling growth is seed germination, and the percentage of seeds that germinate is

crucial to the entire development of the plant. The breakdown of seed dormancy is the main method by which the use of NPs has been demonstrated to improve seed germination [57]. According to Yang et al. [27], nanoprimering is a cutting-edge seed priming technique that is highly regarded for its safety, efficacy, and environmental friendliness in advancing sustainable agriculture. The application of Ni-substituted Co-Zn spinel nanoferrites significantly influenced the germination percentage of *Zea mays* seeds (Fig. 12). Seeds treated with $\text{Co}_{0.5}\text{Zn}_{0.4}\text{Ni}_{0.1}\text{Fe}_2\text{O}_4$ (CZNF1), $\text{Co}_{0.5}\text{Zn}_{0.3}\text{Ni}_{0.2}\text{Fe}_2\text{O}_4$ (CZNF2), $\text{Co}_{0.5}\text{Zn}_{0.2}\text{Ni}_{0.3}\text{Fe}_2\text{O}_4$ (CZNF3) showed a notable improvement in germination percentage compared to the untreated control (Fig. 13a). For example, treatment with $\text{Co}_{0.5}\text{Zn}_{0.3}\text{Ni}_{0.2}\text{Fe}_2\text{O}_4$ (CZNF2) resulted in a germination rate of 90, 100 and 100%, relative to 60, 80 and 90% in the control after 3, 6 and 10 days. However, treatments with $\text{Co}_{0.5}\text{Zn}_{0.1}\text{Ni}_{0.4}\text{Fe}_2\text{O}_4$ (CZNF4), and $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ (CZNF5), showed lower germination with a percent similar, more or less, as control.

The average seedling length (sum of root and shoot) also followed the same trend in germination percentage (Table 5). Seeds nano-primed with $\text{Co}_{0.5}\text{Zn}_{0.3}\text{Ni}_{0.2}\text{Fe}_2\text{O}_4$ (CZNF2) and $\text{Co}_{0.5}\text{Zn}_{0.2}\text{Ni}_{0.3}\text{Fe}_2\text{O}_4$ (CZNF3) exhibited significantly

longer roots and shoots than the control, with increases of up to 45.5 and 18% in seedling total length relative to control (Fig. 13b).

Both fresh and dry weights of maize seedlings improved significantly in nano-primed treatments. The $\text{Co}_{0.5}\text{Zn}_{0.2}\text{Ni}_{0.3}\text{Fe}_2\text{O}_4$ (CZNF3) treatment yielded the highest fresh weight ($\sim 1.5 \pm 0.039\text{a}$ g/seedling) and dry weight ($\sim 0.2958 \pm 0.0078\text{a}$ g/seedling), compared to the control values ($\sim 0.992 \pm 0.028\text{d}$ g and $0.2432 \pm 0.0064\text{bc}$ g, respectively) (Table 5).

Concerning vigor index, both SLVI (Fig. 13c) and SWVI (Fig. 13d) was the highest in seeds treated with $\text{Co}_{0.5}\text{Zn}_{0.3}\text{Ni}_{0.2}\text{Fe}_2\text{O}_4$ (CZNF2) and $\text{Co}_{0.5}\text{Zn}_{0.2}\text{Ni}_{0.3}\text{Fe}_2\text{O}_4$ (CZNF3) over the control and other treatments, confirming the stimulatory effect of Ni-substituted nanoferrites on early plant vigor. The enhancement in vigor index reflects improved energy use efficiency and seedling robustness under NPs influence.

Our results are consistent with previous studies highlighting the crucial role of nano-priming in improving seed germination [57, 58]. Numerous researches have demonstrated that the application of nanoparticles positively influences plant growth, seed germination, and development.

Fig. 12 Effect of Ni substituted Co-Zn spinel nanoferrites on maize seed germination after 3, 6 and 10 days

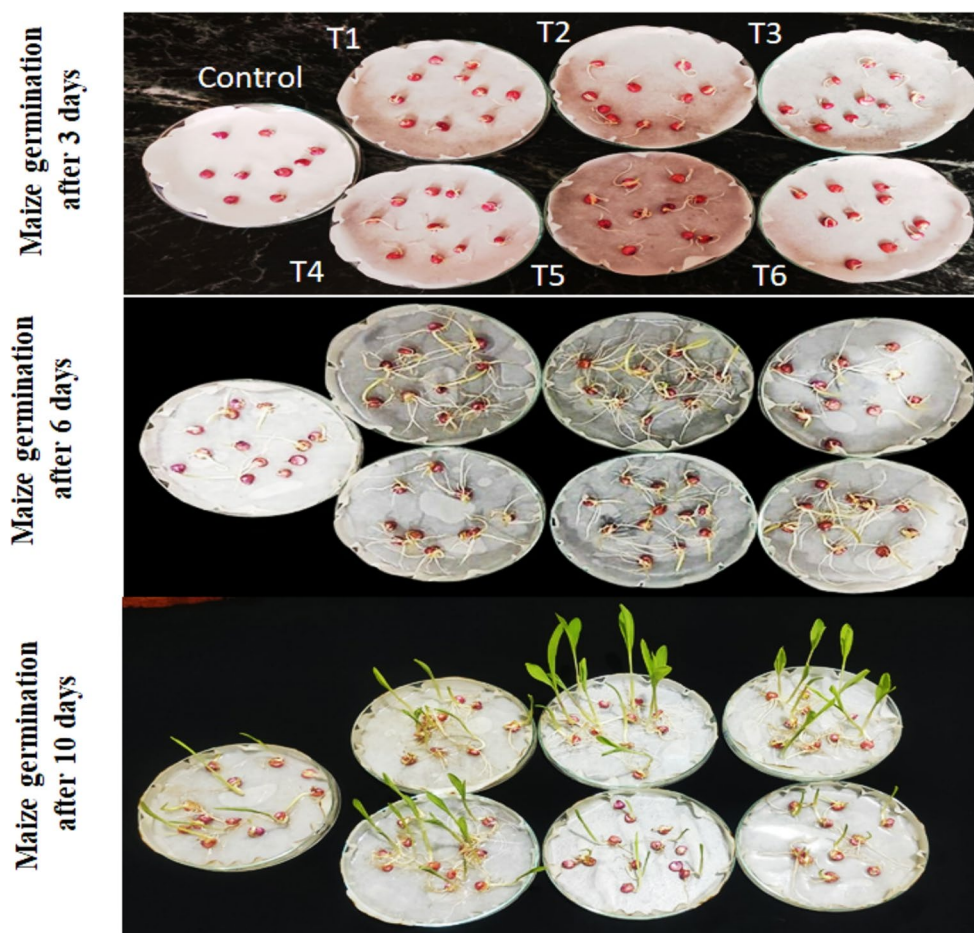


Fig. 13 Effect of Ni substituted Co-Zn spinel nanoferrites on (a) maize seed germination (after 3, 6 and 10 days), (b) enhancement percent in seedling parameters: treatments T5 and T6 are not shown in panel (b) because they did not exhibit any enhancement, (c) the seedling length vigor index (SLVI) and (d) seedling weight vigor index (SWVI). Data represent means $\pm$ standard errors (error bars) of three biological replicates. Different letters indicate significant difference ($p < 0.05$), according to a Duncan multiple range test

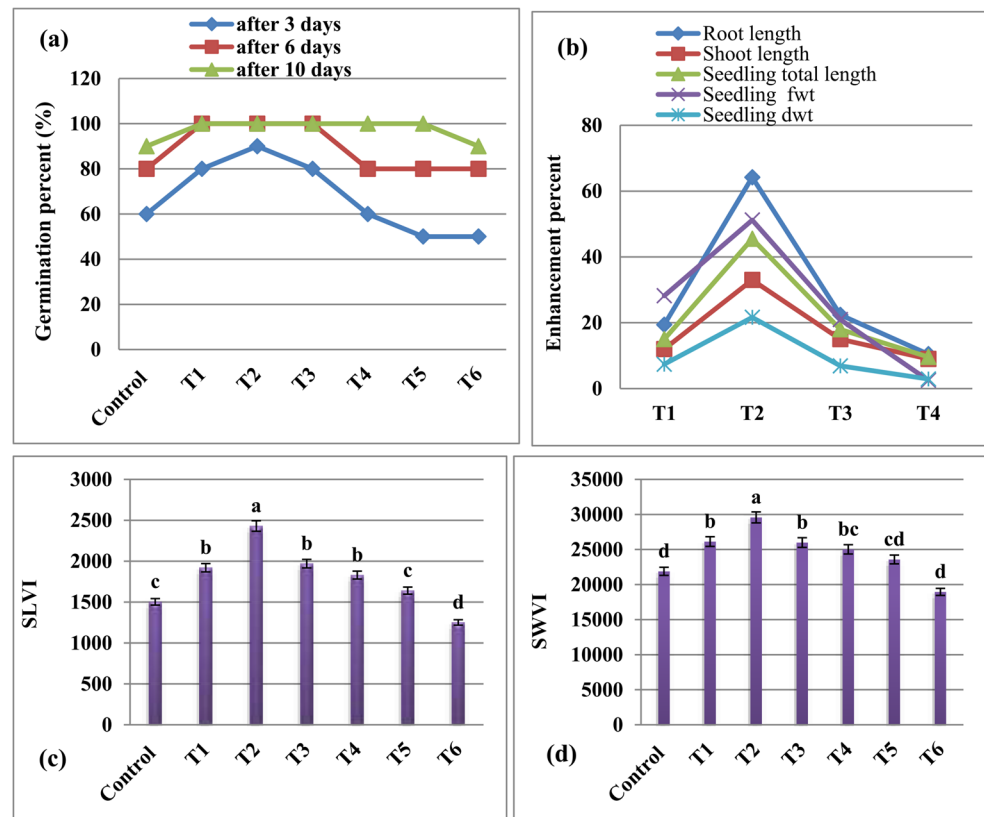


Table 5 Effect of Ni substituted Co-Zn spinel nanoferrites on germination parameters of maize seedlings

Data represent means $\pm$ standard errors of three biological replicates. Different superscripted letters indicate significant difference ($p < 0.05$), according to a Duncan multiple range test

Treatments	Root length (cm)	Shoot length (cm)	Seedling total length (cm)	Seedling total fresh weight (g)	Seedling total dry weight (g)
Control	6.70 $\pm$ 0.18 ^d	10.00 $\pm$ 0.27 ^c	16.70 $\pm$ 0.44 ^c	0.992 $\pm$ 0.028 ^d	0.2432 $\pm$ 0.0064 ^{bc}
T1	8.00 $\pm$ 0.21 ^{bc}	11.20 $\pm$ 0.29 ^b	19.20 $\pm$ 0.51 ^b	1.272 $\pm$ 0.034 ^b	0.2613 $\pm$ 0.0056 ^b
T2	11.00 $\pm$ 0.29 ^a	13.30 $\pm$ 0.35 ^a	24.30 $\pm$ 0.43 ^a	1.500 $\pm$ 0.039 ^a	0.2958 $\pm$ 0.0078 ^a
T3	8.20 $\pm$ 0.22 ^b	11.50 $\pm$ 0.30 ^b	19.70 $\pm$ 0.52 ^b	1.200 $\pm$ 0.032 ^{bc}	0.2599 $\pm$ 0.0068 ^b
T4	7.40 $\pm$ 0.19 ^c	10.90 $\pm$ 0.29 ^b	18.30 $\pm$ 0.48 ^b	1.116 $\pm$ 0.029 ^c	0.2502 $\pm$ 0.0066 ^{bc}
T5	6.50 $\pm$ 0.17 ^{de}	9.90 $\pm$ 0.26 ^c	16.40 $\pm$ 0.43 ^c	0.952 $\pm$ 0.025 ^{de}	0.2358 $\pm$ 0.0062 ^c
T6	5.90 $\pm$ 0.16 ^e	8.00 $\pm$ 0.22 ^d	13.90 $\pm$ 0.39 ^d	0.928 $\pm$ 0.024 ^e	0.2106 $\pm$ 0.0056 ^d

For example, Ghafari and Razmjoo [59] and Abbasi Khalaki et al. [60] reported that nano-priming improves seed germination, seedling growth and development, vigor, and dry weight in many plant species. This effect is attributed to the nanoscale size and unique physicochemical properties of NPs, which make them an effective seed priming strategy [61]. Compared to bulk materials, NPs, molecular or atomic aggregates with at least one dimension between 1 and 100 nm, exhibit enhanced chemical and physical properties [62, 63].

The pronounced enhancement of germination and seedling growth parameters observed in maize seeds nano-primed with CZNF1 and CZNF2 may be attributed to the synergistic effects of its optimized metal ion composition and nanoscale properties. The presence of Zn and Fe, which are essential micronutrients involved in enzyme activation,

chlorophyll biosynthesis, and electron transport, may enhance early metabolic activity during germination. Additionally, low levels of Ni and Co are known to act as cofactors for key enzymes related to nitrogen metabolism and cellular respiration, thereby promoting seedling vigor when supplied within a narrow concentration range. It was found that NPs may penetrate the seed coat and act as micronutrient sources, promoting cell division and elongation. Recent studies further indicate that doping ferrites with transition metals (e.g., Ni, Zn, Mn) alters their structural and surface properties, thereby modifying biological interactions [30]. These doped NPs can serve both as micronutrient sources (Fe, Zn, Co, Ni) and as biostimulants [28, 29].

At the nanoscale, the high surface area of the spinel nanoferrite likely facilitates controlled ion release and improved interaction with the seed coat, enhancing water uptake and

activating hydrolytic enzymes necessary for reserve mobilization. This can accelerate radicle protrusion and subsequent seedling development. Furthermore, nano-priming activates specific metabolic processes naturally triggered during the early stages of germination, resulting in improved germination rates, seedling emergence, growth, productivity, and crop quality [64]. Moreover, it influences plant–environment interactions at cellular and molecular levels [65] and has been shown to upregulate aquaporin gene expression, thereby enhancing water uptake, increasing leaf chlorophyll content, and improving overall plant growth and physiology [64]. However, when concentrations exceed the optimal threshold, these beneficial effects may be diminished due to ionic imbalance or oxidative stress, explaining the reduced response observed at higher dosages.

4 Conclusion

Utilizing the citrate-mediated sol-gel auto-combustion method, we have effectively produced CZNF nanoparticles. The physical characteristics of these ferrites were evaluated using XRD, TEM, and UV–visible diffuse reflectance spectroscopy. The manufactured ferrites reveal a singular phase FCC structure, with no detectable impurities, as corroborated by the XRD spectra. Our investigation shed light on the structural alterations brought about by Ni^{2+} substitution, emphasizing variations to the crystal structure and lattice parameters that affect the functional characteristics of the material. The behavior of the lattice parameters exhibits a strong correlation with the site preferences of Zn^{2+} and Ni^{2+} and their concentration in $\text{Co}_{0.5}\text{Zn}_{0.5-x}\text{Ni}_x\text{Fe}_2\text{O}_4$. At minimal substitution levels ($x=0.0\text{--}0.3$), Zn^{2+} ions integrate into the spinel lattice, occupying the unoccupied A- and B-locations, substituting for Fe^{3+} ions in B-locations. As a consequence, the lattice parameter is enlarged. The elevation of substitution levels beyond $x=0.3$ compels the Zn^{2+} ions to undergo a redistribution within the A-sublattice, specifically at the A- positions. The lattice value goes down because of this. The mean crystallite size was assessed through analyzing peak broadening at (311), showing an initial increase from about 42.57 to 55.63 nm as the Ni^{2+} ratio (x) rose from 0.0 to 0.3, shadowed by a decrease with growing Ni^{2+} ratio. The reduction in crystallite size can be ascribed to the reduced ionic radii of Ni^{2+} ions relative to Zn^{2+} . The specific surface area of the synthesized nanoparticles was determined to be between 2.66×10^8 and 2.05×10^8 cm^2/g . The TEM studies established the nanoscale structure of the synthesized ferrites. Through the analysis of optical measurements obtained from a UV–visible microscope, it was witnessed that the energy band gap derived from Tauc's plot varied from 1.440 to 1589 eV, thereby confirming the

semiconducting physiognomies of the synthesized ferrite nanoparticles. Biological assays revealed that low concentrations of Ni-substituted Co-Zn spinel nanoferrites positively influenced seed germination, root and shoot elongation, fresh and dry weights and overall seedling vigor in *Zea mays*, suggesting their potential use as nano-priming agents in agriculture. These findings highlight the dual functional role of engineered spinel ferrites as both structurally interesting nanomaterials and biologically active agents. Further research is needed to explore the underlying mechanisms of plant-nanoferrite interactions and to optimize safe dosage ranges for practical agricultural applications.

Author contributions N.I. Abu-Elsaad: Conceptualization, Visualization, Software, Writing - original draft, Software, Validation, Writing - review & editing. Reda E. Abdelhameed: Methodology, Software, Data curation, Writing - original draft, Writing - review & editing. A.S. Nawara: Methodology, Software, Data curation, Writing - original draft, Writing - review & editing.

Funding Open access funding provided by The Science, Technology & Innovation Funding Authority (STDF) in cooperation with The Egyptian Knowledge Bank (EKB).

Data availability Data supporting the findings of the current study are available from the corresponding authors upon request.

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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