

Article

Multifunctional characterization of pure and Ni-substituted ZnFe_2O_4 nanoparticles: Correlation of structural, Raman, morphological, and optical properties

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Abstract: Spinel zinc ferrite (ZnFe_2O_4) nanoparticles have been of high interest owing to their multifunctionality and wide variety of technological and biomedical uses. ZnFe_2O_4 nanoparticles in the current experiment were obtained through the solution combustion technique, which is a simple, quick, and energy-saving method. Zinc and iron metal nitrates were dissolved, and distilled water was used; urea was the fuel used in the combustion process. A solution-combustion reaction occurred upon heating, and an as-burnt powder was formed, which was porous. This was then subjected to calcination to get phase-pure ZnFe_2O_4 nanoparticles. The structural features were analyzed using X-ray diffraction, whereby it was found that the material formed a single-phase cubic spinel structure, which was of good crystalline nature. The average crystallite sizes were calculated as 34.9 nm for pure ZnFe_2O_4 and 15.08 nm for the doped sample, indicating a nanocrystalline nature. UV-Visible spectroscopy confirmed the semiconducting nature of the synthesized samples, with direct optical band gap values increasing from 2.14 eV for pure ZnFe_2O_4 to 2.25 eV for the Ni-doped ZnFe_2O_4 sample. The synthesized ZnFe_2O_4 nanoparticles are promising materials to be used in Structural, morphological, and optical applications due to the combined structural stability along with a tunable optical band gap.

Keywords: ZnFe_2O_4 ; nanoferrites; structural; FTIR; Raman spectroscopy; UV-visible properties

1. Introduction

Ferrites are one of the important classes of materials with numerous industrial applications [1]. Among different characterization techniques, Raman spectroscopy is widely employed to analyze the structural and vibrational characteristics of ferrite nanoparticles. The material provides valuable information on cation distribution, defect structures, and their connection to optical and other properties. This understanding is crucial for improving performance in applications like catalysis, sensing, and the biomedical field. ZnFe_2O_4 nanoparticles have attracted considerable attention in biomedicine because of their excellent chemical stability, tunable magnetic behavior, low toxicity, and favorable biocompatibility. These characteristics make them promising candidates for magnetic hyperthermia, targeted drug delivery, magnetic resonance imaging (MRI) contrast enhancement, biosensing, antimicrobial systems, and multifunctional theranostic platforms. Their performance in these applications is strongly influenced by structural parameters, including particle size, crystallinity, cation distribution, surface morphology, and defect concentration, which

govern magnetic response, surface reactivity, and interactions with biological systems. Therefore, comprehensive structural and optical characterization is essential for understanding and optimizing ZnFe_2O_4 nanoparticles for future biomedical applications. Structurally, ferrites are categorized into four broad groups, namely spinel ferrites, garnets, and ortho-ferrites. Spinel ferrites are widely used materials due to their stable crystal structure and favorable optical properties. Specifically, zinc ferrite (ZnFe_2O_4) nanoparticles exhibit a tunable optical band gap and excellent structural integrity, making them suitable for optoelectronic and technological applications [2,3]. High thermal stability and controlled magnetism also characterize the ferrite materials, which is why they can be utilized in any industry depending on the need to control heat in a more precise manner. They are significant as they have high Curie temperatures that make them effective as heating components in catalytic reactors, moderate magnetic losses in the kHz band, which allows them to sustain constant operation, and are more energy efficient in industrial applications [4]. Zinc ferrite (ZnFe_2O_4) and spinel ferrites, in particular, have become a major subject of research with their remarkable structural, morphological, and optical characteristics. Spinel ferrites tend to follow the MFe_2O_4 formula, and the oxygen ions tend to follow a face-centered cubic (fcc) lattice as a common characteristic of all spinel structures. Within the normal spinel, Zn^{2+} ions are most likely to occupy the tetrahedral (A) sites, and the Fe^{3+} ions are at the octahedral (B) sites of the lattice. But in the case of the synthesis of ZnFe_2O_4 in nanocrystalline form, particularly by the solution combustion technique, rearrangement of cations may take place partially. It leads to a mixed spinel structure, in which Zn^{2+} and Fe^{3+} are located on tetrahedral and octahedral sites, which eventually affects the optical properties of the material [5]. Ferrites are generally divided into three broad categories: spinel ferrites, garnet ferrites, and hexagonal ferrites, each with a distinct structural characteristic. Spinel ferrites have the general formula AB_2O_4 , with A^{2+} ions occupying the tetrahedral sites and B^{3+} ions occupying the octahedral sites. The crystal structure of these materials is cubic, with oxygen ions packed in a close-packed arrangement typical of the Fd-3m space group. Nickel-doped zinc ferrites crystallize in a cubic spinel structure, and zinc-based compositions, especially, were studied extensively. Even minor levels of Zn^{2+} replacement are also reported to create significant alterations in the structural and optical behaviour of these ferrite systems. Such cationic adjustment affects the lattice symmetry and particle size, which has rendered Zn-containing ferrites potential for applications where structural and optical properties are to be controlled [6,7]. New research studies have pointed out that the process of using natural plant extracts in developing nanoparticles can be a solution in providing an alternative to chemical methods. This solution combustion method makes the reaction occur simply and sustainably, and the reaction does not use harsh and hazardous chemicals. These techniques are valued because they are cost-effective, environmentally friendly, and can synthesize stable nanoparticles in a brief reaction period. Besides, they allow large-scale production with minimal toxic byproducts, which is quite consistent with the objectives of green nanotechnology and sustainable material synthesis [8]. Some research studies have been conducted to modify ferrite materials to improve their structural and optical characteristics. In the current research, ZnFe_2O_4 nanoparticles have been prepared using the solution combustion method to investigate the influence of synthesis factors on their

morphological, optical, and structural characteristics, providing further understanding of their capabilities for multifunctional use in technology [9]. The solution combustion method has proved to be one of the most promising and energy-saving techniques since it allows fast synthesis, uniform mixing at the molecular level, and formation of small and homogeneous nanoparticles at comparatively low temperatures. The structural, morphological, and optical properties of ZnFe_2O_4 nanoparticles can be fine-tuned by means of the critical adjustment of specific parameters [10]. Recent advancements in Raman spectroscopy have made it an effective technique for investigating semiconductor materials. This method provides detailed information on vibrational modes, crystal symmetry, and structural order, which are closely related to crystallite size and phase formation. Owing to its sensitivity and non-destructive nature, Raman analysis is particularly suitable for confirming the structural integrity of synthesized ZnFe_2O_4 nanoparticles [11]. Raman spectroscopy has emerged as a reliable tool for the characterization of nanomaterials. It enables the identification of lattice vibrations and symmetry-related features that reflect the internal arrangement of atoms within the crystal structure [12].

Recent advances in ferrite nanomaterials have expanded their use cases to include a wide range of biomedical applications beyond their traditional roles as magnetic or catalytic materials. There has been substantial interest in ZnFe_2O_4 nanoparticles due to their good chemical stability, biocompatibility, adjustable magnetic behavior, and semiconductor nature, which make them ideal for applications such as MRI, magnetic hyperthermia, drug delivery systems, biosensors, antimicrobial coatings, photocatalysis, and theranostics. Specifically, particle size, crystallinity, defects in the crystal lattice, and ion distribution are factors that affect the magnetic behavior, optical absorption, and biological behavior of ZnFe_2O_4 nanoparticles [13].

Despite the extensive literature on combustion-synthesized ZnFe_2O_4 nanoparticles, most previous studies have primarily focused on individual characterization techniques such as XRD, SEM, FTIR, Raman spectroscopy, or UV-Visible analysis [13]. Consequently, the relationship among crystallite size, lattice strain, vibrational characteristics, surface morphology, and optical band-gap evolution resulting from Ni substitution has not been systematically established. Furthermore, comprehensive investigations that correlate these interdependent properties using a single solution combustion synthesis route remain limited. To highlight this research gap, a comparative summary of representative studies has been presented in **Table 1**. The comparison demonstrates that, unlike earlier reports, the present work establishes a comprehensive correlation between structural, morphological, Raman, and optical characteristics, providing a clearer understanding of the influence of Ni incorporation on ZnFe_2O_4 nanoparticles. Patade et al. reported single-phase superparamagnetic MnFe_2O_4 nanoparticles with an average particle size of ~25 nm and excellent magnetic properties. Their high heating efficiency ($\text{SAR} = 217.62 \text{ W/g}$) enabled rapid attainment of the hyperthermia temperature, demonstrating promising applicability in magnetic fluid hyperthermia [13].

Several recent studies have shown that properties such as structural stability, particle size, cation distribution, and optical properties of spinel ferrite nanoparticles significantly influence their biomedical applications, including magnetic hyperthermia, drug delivery, MRI contrast agents, and theranostics [14]. While this study does not

focus on biological applications, understanding the structure-property relationship through detailed structural and optical characterization of zinc ferrite nanoparticles will be very useful for designing zinc ferrite-based multifunctional nanomaterials for biomedical applications in future research.

Establishing these structure-property relationships is important because changes in crystallite size, lattice strain, and cation distribution directly affect the functional performance of ferrite nanomaterials. Therefore, a systematic correlation among structural, vibrational, morphological, and optical characteristics provides a more comprehensive understanding of the role of Ni substitution and offers useful guidance for designing ferrite nanomaterials with tailored properties.

Hence, this work systematically investigates the influence of Ni substitution on the structural, vibrational, morphological, and optical properties of solution-combustion-synthesized ZnFe_2O_4 nanoparticles and establishes correlations among these characteristics to provide a comprehensive understanding of their structure-property relationship.

Table 1. Comparison of representative literature with the present work on ZnFe_2O_4 and Ni-substituted ferrite nanoparticles, highlighting the research gap and novelty.

Reference	Material	Synthesis method	Crystallite size (nm)	Band gap (eV)	Characterization	Major findings
Kombaiah et al. (2017) [15]	ZnFe_2O_4	Conventional & Microwave Combustion	28–35	2.05–2.18	XRD, SEM, UV–Vis	Compared combustion routes and optical properties
Sundararajan et al. (2022) [16]	$\text{ZnFe}_2\text{O}_4/\text{NiFe}_2\text{O}_4$	Sol–gel	20–45	2.10–2.30	XRD, FTIR, SEM	Comparative structural and magnetic investigation
Chandamma et al. (2017) [17]	Zn-doped Ni ferrite	Combustion	18–30	—	XRD, Electrical	Structural and electrical properties
Jogi et al. (2022) [18]	ZnFe_2O_4	Green synthesis	25–40	2.18	XRD, FTIR, UV–Vis	Structural and optical characterization
Albini et al. (2023) [19]	ZnFe_2O_4	Chemical synthesis	—	—	Raman	Raman modes of zinc ferrites
Present Work	Ni-doped ZnFe_2O_4	Solution Combustion	34.9 → 15.08	2.14 → 2.25	XRD, SEM, FTIR, UV–Vis, Raman	Demonstrates Ni-induced lattice contraction, crystallite size reduction, Raman confirmation of the cubic spinel structure, and enhanced optical band gap in solution-combustion-synthesized ZnFe_2O_4 .

2. Experimental

2.1. Materials

The synthesis of pure ZnFe_2O_4 and Ni-doped nanoparticles by the solution combustion method is shown in **Figure 1**. Analytically pure metal nitrates were used as the starting materials. The reagents were zinc nitrate [$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], nickel nitrate [$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], and ferric nitrate [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$] procured from Sigma-Aldrich, which served as the sources of Zn^{2+} , Ni^{2+} , and Fe^{3+} ions, respectively. Urea was used as a fuel component because it can facilitate an exothermic, self-sustaining burning process.

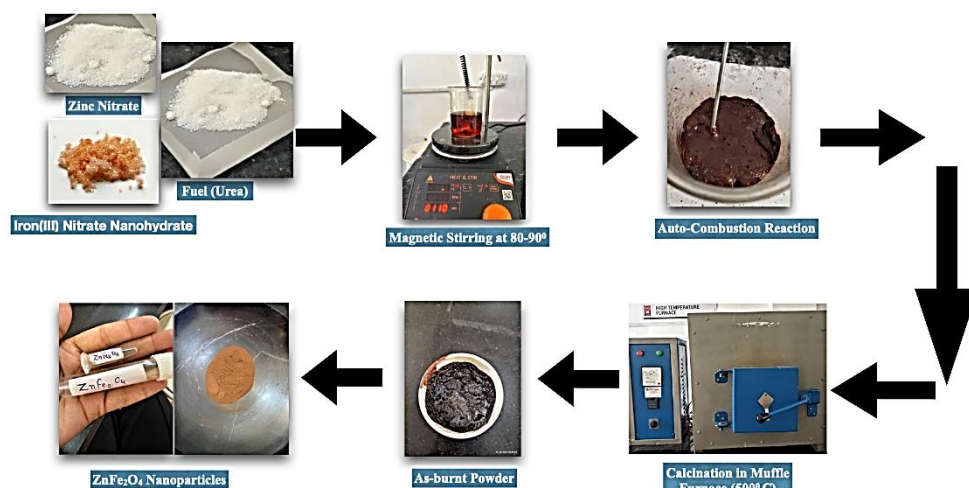


Figure 1. Synthesis of pure ZnFe_2O_4 and doped ZFO nanoparticles by the Solution Combustion method.

2.2. Synthesis of pure and Ni-doped ZnFe_2O_4 nanoparticles

All the metal nitrates were dissolved in a sufficient volume of distilled water and stirred continuously on a magnetic stirrer to ensure homogeneity. As the heating was continued, the clear solution gradually thickened and transformed into a porous, foam-like material, indicating that it was ready for the combustion stage. This beaker containing the material was placed in a pre-heated muffle furnace. When the as-formed product was exposed to high temperature, it ignited spontaneously and experienced a self-propagating combustion reaction at a high rate. This intense response caused a large amount of gases to be released, and the by-product of the combustion was a soft, porous, and foam-like ash. The reaction was followed by allowing the obtained powder to cool naturally. Gently, it was crushed in an agate mortar and pestle to a fine and uniform texture. The obtained powder was further heat-treated at 500 °C for a suitable period to improve the crystallinity of the synthesized nanoparticles.

The synthesis of pure and Ni- doped ZnFe_2O_4 Nanoparticles by the solution combustion method is illustrated in **Figure 1**.

3. Characterization

The structural and physical properties of the prepared ZnFe_2O_4 nanoparticles were examined using different characterization techniques. Before analysis, the sample was calcined at 500 °C for 5 h. The samples were used in powder form for all characterization studies. Powder X-ray diffraction (PXRD) analysis was performed using a Bruker D8 ADVANCE diffractometer with $\text{Cu K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) in the 2θ range of 20–80 °to study the crystal structure and phase formation. The obtained diffraction peaks indicated a cubic spinel structure, and the crystallite size was estimated using the Debye-Scherrer equation from the most intense peak. The surface morphology and particle distribution were analyzed using scanning electron microscopy (SEM, JEOL JSM-6510LV) to determine particle shape and agglomeration, while Fourier Transform Infrared (FTIR) spectroscopy (Bruker Tensor-27) was carried out across the 400–2000 cm^{-1} range to determine bonding characteristics and functional groups within the material. UV-Visible spectroscopy

(Shimadzu UV-2600) was also used to study the optical properties, and the absorption data were analyzed to determine the optical band gap. Vibrational modes and structural features were confirmed by micro-Raman spectroscopy (Renishaw in Via Raman microscope), and the formation of the spinel phase was confirmed by carrying out all measurements under standardized conditions.

4. Result and discussion

4.1. Powder X-ray diffraction analysis

Figure 2 shows the PXRD patterns of pure ZnFe_2O_4 and Ni-doped $\text{Zn}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ samples, enabling a direct comparison of their structural features. The diffraction patterns recorded in the $20\text{--}80^\circ(2\theta)$ range after calcination at 500°C were indexed to a face-centered cubic structure [20]. The diffraction patterns proved that all the reflections were similar to those of a cubic spinel lattice related to the $\text{Fd}3\text{m}$ space group [18]. The sharp and well-defined reflections indicate high crystallinity and phase purity of the synthesized samples [21]. The prominent (311) reflection was used for structural analysis. The interplanar spacing (d) was found to be $2.54\text{ \AA}$ for the pure sample and $2.52\text{ \AA}$ for the doped sample, calculated using Bragg's law. The lattice parameter (a) decreased from $8.43\text{ \AA}$ (pure ZnFe_2O_4) to $8.36\text{ \AA}$ (doped $\text{Zn}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$), and the unit cell volume decreased from $596.9\text{ \AA}^3$ to $584.2\text{ \AA}^3$ due to the substitution of larger Zn^{2+} ions ($0.74\text{ \AA}$) by smaller Ni^{2+} ions ($0.69\text{ \AA}$), leading to lattice contraction. The X-ray density (ρ) increased slightly from 5.36 g/cm^3 (pure) to 5.42 g/cm^3 (doped), likely reflecting compositional variation and cation redistribution within the spinel lattice. The crystallite size, estimated using the Debye-Scherrer equation, decreased significantly from 34.9 nm (pure) to 15.08 nm (doped), indicating that Ni substitution induces lattice strain and suppresses grain growth [22]. The calculated structural parameters are presented in **Table 2**. The characteristic diffraction peaks observed at approximately 30° , 35° , 37° , 43° , 53° , and $57^\circ(2\theta)$, corresponding to the (220), (311), (222), (400), (422), and (511) planes, confirm the formation of a cubic spinel structure with no detectable impurity phases [23]. Overall, all samples exhibit a single-phase cubic spinel structure consistent with the $\text{Fd-}3\text{m}$ space group. The PXRD patterns of pure and Ni-doped ZnFe_2O_4 nanoparticles are presented in **Figure 2**, while the corresponding structural parameters calculated from the diffraction data are summarized in **Table 2**.

The reduction in lattice parameter from $8.43\text{ \AA}$ to $8.36\text{ \AA}$ indicates lattice shrinkage resulting from the substitution of larger Zn^{2+} ions ($0.74\text{ \AA}$) with smaller Ni^{2+} ions ($0.69\text{ \AA}$). This ion substitution alters the cation distribution between the tetrahedral (A) and octahedral (B) sites of the spinel lattice, leading to local lattice distortion and increased internal strain. A comparable structural evolution has been reported by Somvanshi et al. [24], where cation engineering produced measurable changes in lattice parameters and crystallinity, highlighting the strong relationship between structural modification and the physicochemical properties of ferrite nanoparticles. The reduction in crystallite size from 34.9 nm to 15.08 nm further indicates that Ni incorporation suppresses grain growth during solution combustion synthesis by promoting nucleation. These observations confirm that the reduction in

lattice parameter and crystallite size originates from ion-radius mismatch, cation redistribution, and the resulting increase in lattice strain.

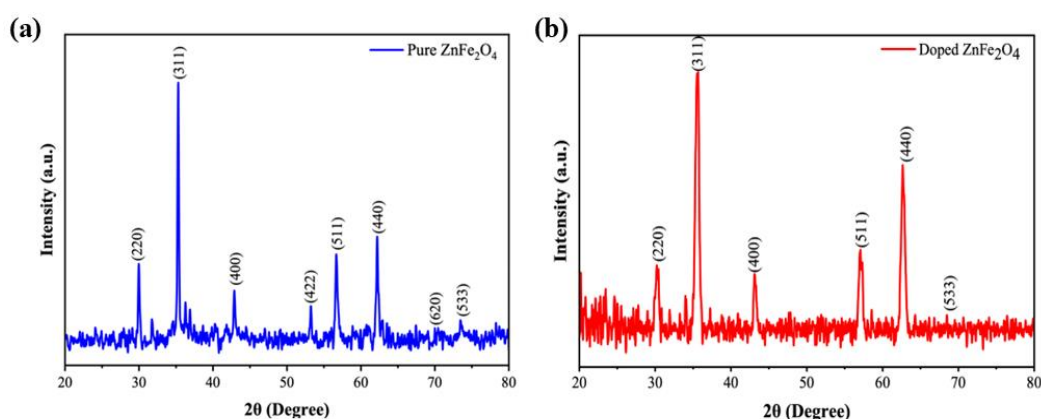


Figure 2. Powder XRD patterns of (a) pure ZnFe_2O_4 and (b) Ni-doped ZFO nanoparticles.

Table 2. Comparative XRD structural parameters, including lattice parameter(a), unit cell volume(V), X-ray density(ρ_x), and crystallite size(D) for pure and doped ZnFe_2O_4 samples.

Parameters	Pure	Doped
Plane (hkl)	311	311
d-spacing(Å)	2.54	2.52
Lattice Parameter, a(Å)	8.43	8.36
Unit Cell Volume, V(Å ³)	596.9	584.2
X-ray density(g/cm ³)	5.36	5.42
Crystallite size(nm)	34.9	15.08
Lattice strain ($\times 10^{-4}$)	9.33	12.58

The increase in lattice strain and the reduction in crystallite size are expected to increase the specific surface area and the number of active surface sites available for physicochemical interactions. These structural modifications can facilitate surface adsorption and improve charge transfer processes at the material surface. Consequently, Ni-substituted ZnFe_2O_4 nanoparticles exhibit enhanced potential for applications such as gas sensing, photocatalysis, and other semiconductor-based functional devices, where surface characteristics play a crucial role in determining performance.

Williamson-Hall (W-H) analysis

In order to separate the effects of crystal size and lattice strain in causing the broadening of XRD peaks, the Williamson-Hall (W-H) method using the Uniform Deformation Model (UDM) was performed. The Williamson-Hall plots of pristine ZnFe_2O_4 NPs and Ni-doped ZnFe_2O_4 NPs are shown in **Figure 3**. It was found that there is a linear dependence between $\beta \cos \theta$ and $4 \sin \theta$ for both materials, which proves that the XRD peaks are broadened due to crystal size and lattice strain. Lattice strain for the doped system was found to be 0.33 times greater (increased from 9.33×10^{-4}

to 1.26×10^{-3}) in comparison to the pure ZnFe_2O_4 sample. This increase in the value of lattice strain can be explained by the replacement of bigger cations Zn^{2+} (0.74 Å) with smaller cations Ni^{2+} (0.69 Å). This causes lattice distortion and leads to the appearance of internal compressive stresses in the cubic spinel structure. These results correspond to the decrease in lattice parameter (from 8.43 Å to 8.36 Å) and crystallite size (from 34.9 nm to 15.08 nm).

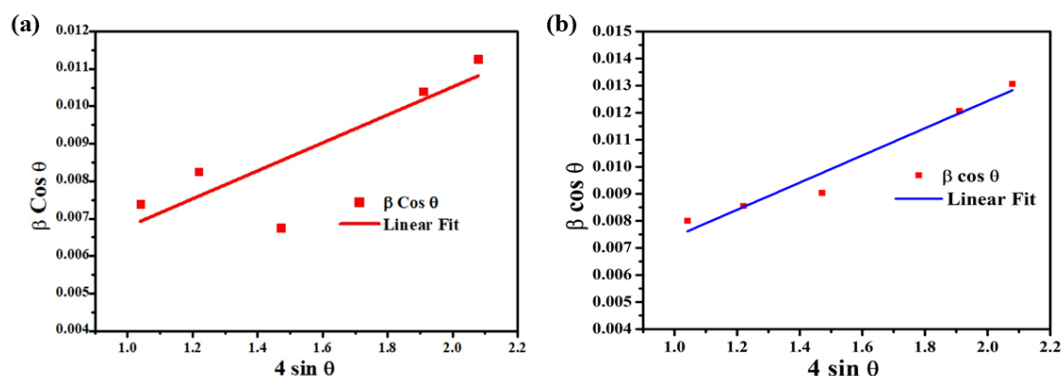


Figure 3. Williamson-Hall (W-H) plots of (a) pure ZnFe_2O_4 and (b) Ni-doped ZnFe_2O_4 nanoparticles showing the linear fitting used to determine crystallite size and lattice strain.

4.2. Scanning Electron Microscopy (SEM) analysis

The SEM micrographs of the synthesized ZnFe_2O_4 nanoparticles are presented in **Figure 4**. SEM analysis was performed to investigate the surface morphology and microstructural characteristics of the synthesized nanoparticles. The micrographs reveal that the particles exhibit a quasi-spherical and irregular morphology with relatively uniform distribution throughout the sample. A porous, sponge-like surface texture is also observed, indicating the formation of interconnected particles with appreciable surface roughness. These grains are nanosized, as the fine structural features contribute to the formation of well-structured nanoparticles. The level of agglomeration is also moderately high, as it is common in the case of ferrite-based nanomaterials and can be attributed to strong magnetic dipole-dipole interaction between the particles and the high surface energy associated with nanoscale systems [25,26]. Comparable morphological characteristics have been reported by Pandhare et al. [23] for polyol-crafted ferrite nanoparticles, where quasi-spherical particles with controlled morphology and moderate agglomeration were observed, supporting the present SEM results. Other than establishing the particle size, SEM images also provide overall information about the morphology, distribution pattern, and surface texture of the nanoparticles. The homogeneity of the micrographs indicates that the synthesis process was selected to enable the growth of particles of a homogeneous shape, which had minimal structural aberration [27]. Structural data analyzed with XRD give a good match with the morphology of the surface based on SEM. The diffraction peaks confirm a crystalline, pure cubic spinel with the index to the $\text{Fd}3\text{m}$ space group, which indicates that the ZnFe_2O_4 nanoparticles were in good crystalline purity without the formation of secondary phases [28]. The phase-pure PXRD patterns along with the uniform morphology observed in SEM analysis indicate that the solution combustion technique is highly effective for the synthesis of structurally

stable ferrite nanoparticles. These homogeneous morphological attributes also affirm that the synthesis attributes have been effective in controlling the growth of uniformly dispersed and structurally sound nanoparticles.

Figure 5 shows the size distribution of the particles analyzed using ImageJ on the SEM micrographs. It can be observed that the distribution is normally distributed (Gaussian) with an average size of 61.70 nm and a standard deviation of 5.20 nm, which indicates uniform growth of the particles. The narrow size distribution of the particles clearly shows that the solution combustion synthesis technique successfully synthesized ZnFe_2O_4 nanoparticles with controlled morphology [29].

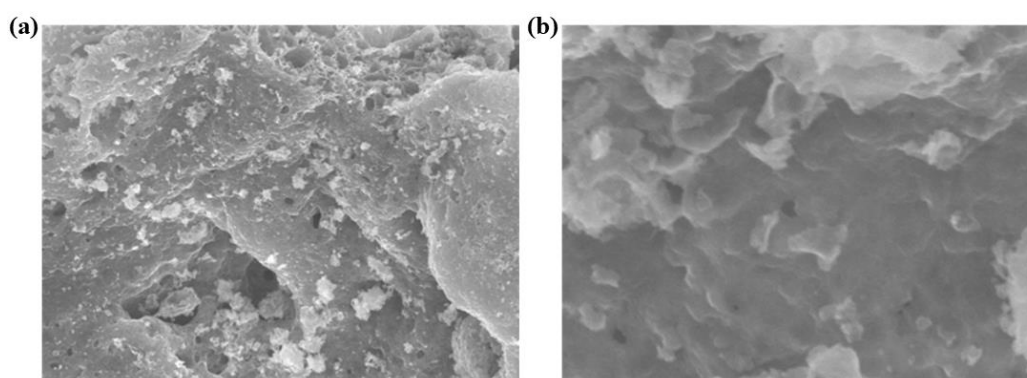


Figure 4. SEM micrographs of (a) pure ZnFe_2O_4 and (b) Ni-doped ZFO nanoparticles.

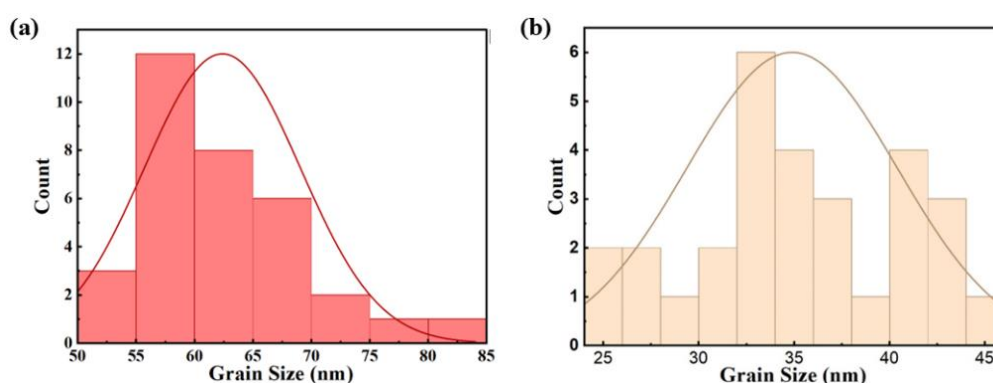


Figure 5. Histogram SEM micrographs of (a) pure ZnFe_2O_4 and (b) Ni-doped ZFO nanoparticles.

4.3. FTIR spectral analysis

The FTIR spectra of pure and Ni-doped ZnFe_2O_4 nanoparticles are presented in **Figure 6**. The spectra reveal the characteristic vibrational modes of metal–oxygen (M–O) bonds in the spinel lattice, which provide valuable information regarding the bonding environment, cation distribution, and structural modifications induced by Ni substitution. Absorption peaks corresponding to lattice vibrations in the FTIR spectrum are associated with the distribution of metal ions and are influenced by changes in the bonding environment, such as the position and strength of the bands, which are altered by changes in the arrangement of cations, as well as the mass of the cation and the strength of the metal-oxygen bond [30,31]. The FTIR spectrum shows characteristic bands of the spinel structure. In the pure sample, the principal band at 497 cm^{-1} assigned to metal-oxygen stretching at tetrahedral (A) sites, shifts slightly to 498 cm^{-1} after Ni doping; another band near 740 cm^{-1} shifts to about 737 cm^{-1} , which

is related to metal-oxygen vibrations. These small changes suggest structural adjustments induced by Ni, implying proper arrangement of cations within the lattice [32]. Minor changes in peak position and intensity are linked to variations in cation arrangement, which affect lattice strain and vibrational behavior of the metal-oxygen bonds [33,34]. Kharat et al. explained that the FTIR spectrum confirms the formation of the spinel ferrite structure through the characteristic metal-oxygen stretching vibrations observed below 700 cm^{-1} [32]. The shift in peaks is due to the change in metal-oxygen bonds after Ni replacement. This is because the ionic radius of Ni^{2+} ions is smaller compared to that of Zn^{2+} ions; therefore, replacement results in a variation in the M-O bond length and force constant, resulting in the frequency of vibration for both the tetrahedral and octahedral sites.

Furthermore, the higher-frequency absorption band is primarily due to the stretching vibration of metal-oxygen bonds at tetrahedral (A) sites, whereas the lower-frequency band mainly arises from octahedral (B) site vibrations in the spinel lattice. Partial substitution of Zn^{2+} with Ni^{2+} alters the local coordination environment and redistributes cations between these crystallographic sites, resulting in subtle changes in bond strength and vibrational energy. The observed FTIR peak shifts therefore provide additional evidence of local structural rearrangement induced by Ni incorporation, consistent with the lattice contraction and increased lattice strain observed in XRD analysis. A comparable correlation between metal-oxygen vibrational characteristics and structural modification in zinc ferrite nanoparticles has also been reported by Somvanshi et al. [24], where changes in the surface and local bonding environment were reflected through variations in the vibrational spectra, supporting the present FTIR observations.

4.4. UV visible spectroscopy

The UV-Visible absorption spectra of pure and Ni-doped ZnFe_2O_4 nanoparticles are presented in **Figure 7**, while the corresponding Tauc plots used to estimate the optical band gap are shown in **Figure 8**. The optical properties of the synthesized nanoparticles were investigated using UV-Visible spectroscopy. The optical band gap was calculated using the Tauc relation:

$$\alpha h\nu = A(h\nu - E_g)^n, \quad (1)$$

where α is the absorption coefficient, $h\nu$ is the photon energy, A is a proportionality constant, and E_g is the optical band-gap energy. The band-gap values were obtained by extrapolating the linear region of the $(\alpha h\nu)^2$ versus $h\nu$ plot to the energy axis, as illustrated in **Figure 8** [33,35]. The optical band gap increased from 2.14 eV to 2.25 eV upon Ni doping, demonstrating the modification of optical properties in ZnFe_2O_4 nanoparticles. The observed increase suggests a change in the electronic structure [36,37]. The increase in the optical band gap owing to Ni incorporation into the compound is linked to changes in the electronic band structure of ZnFe_2O_4 . Ni^{2+} incorporation alters the electronic states of Fe-O and Ni-O, shifting the valence and conduction band edges. Partial cation inversion associated with Ni incorporation may further modify the distribution of Zn^{2+} and Fe^{3+} ions between the tetrahedral and octahedral sites, thereby influencing orbital interactions and contributing to the

observed widening of the optical band gap. Additionally, the decrease in crystallite size creates weak quantum confinement effects, whereas lattice distortion decreases the defect-related localized states within the forbidden energy band. Thus, the widening of the optical band gap requires higher photon energy for electronic excitation and suppresses defect-assisted electronic transitions, thereby improving optical stability. These optical characteristics indicate the potential suitability of Ni-substituted ZnFe_2O_4 nanoparticles for photocatalytic, optoelectronic, and gas sensing applications, where improved optical stability and efficient charge transport are desirable [38–40]. A decrease in absorbance accompanied by a blue shift of the absorption edge toward lower wavelengths further confirms the increase in the optical band gap following Ni substitution. This band-gap widening is primarily attributed to the replacement of Zn^{2+} ions by Ni^{2+} ions, which modifies the electronic band structure through changes in the Fe–O and Ni–O electronic states. In addition, partial cation inversion associated with Ni incorporation may alter the distribution of cations between the tetrahedral and octahedral sites, thereby influencing the valence and conduction band edges. The reduction in crystallite size further contributes through weak quantum confinement effects, while lattice distortion suppresses defect-related localized states within the band gap. These combined effects are consistent with recent reports on Ni-substituted ferrite nanomaterials [41,42] and indicate that controlled Ni incorporation effectively tailors the optical properties of ZnFe_2O_4 nanoparticles for advanced semiconductor-based applications. A similar relationship between structural modification and functional performance has also been reported by Phalake et al. [20], where controlled tuning of the physicochemical properties of ferrite nanoparticles was shown to play a key role in determining their application potential. These observations demonstrate a clear correlation between the structural modifications induced by Ni substitution and the optical response of ZnFe_2O_4 nanoparticles, confirming that controlled cation substitution is an effective approach for tailoring their physicochemical properties for advanced functional applications.

Figure 9 schematically summarizes the effect of Ni substitution on the structural and optical properties of ZnFe_2O_4 nanoparticles. The substitution of Zn^{2+} ions by smaller Ni^{2+} ions leads to lattice contraction, increased lattice strain, and reduced crystallite size, which subsequently modifies the electronic structure and results in optical band-gap widening. The proposed mechanism highlights the correlation between the structural and optical characteristics observed in the present study and demonstrates how controlled Ni substitution can be used to tailor the physicochemical properties of ZnFe_2O_4 nanoparticles for potential functional applications [43].

4.5. Raman spectroscopy

The Raman spectra of pure and Ni-doped ZnFe_2O_4 nanoparticles are presented in **Figure 10**. ZnFe_2O_4 crystallizes in a cubic spinel structure belonging to the Fd-3m space group [44]. The Raman spectra exhibit the characteristic vibrational modes of the spinel lattice, confirming the formation of the cubic phase and supporting the structural results obtained from XRD analysis. Raman spectroscopy was further employed to investigate the lattice vibrations, cation distribution, and local structural modifications induced by Ni substitution [45]. The observed Raman peaks indicate

good crystallinity and support the structural results obtained from XRD analysis [46]. Raman spectroscopy was employed to examine materials prepared via the solution combustion method, enabling the evaluation of structural features and lattice vibrations.

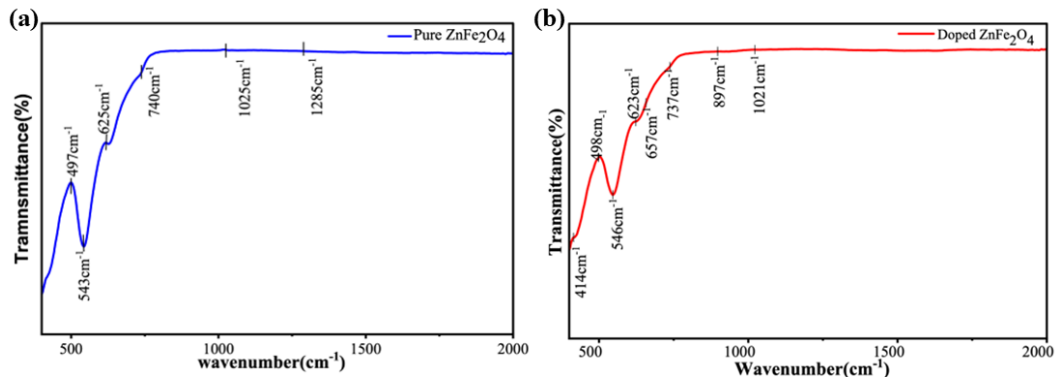


Figure 6. FTIR spectroscopy of pure (a) ZnFe_2O_4 and (b) Ni-doped ZFO nanoparticles.

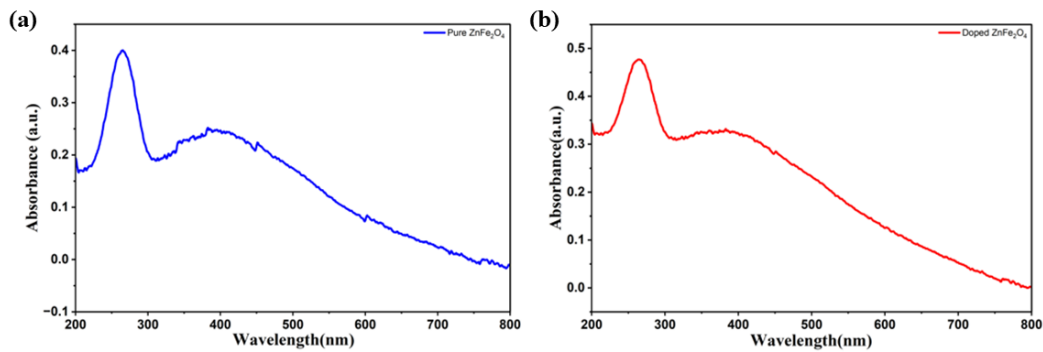


Figure 7. UV-Visible absorbance spectra of (a) pure ZnFe_2O_4 and (b) Ni-doped ZFO nanoparticles.

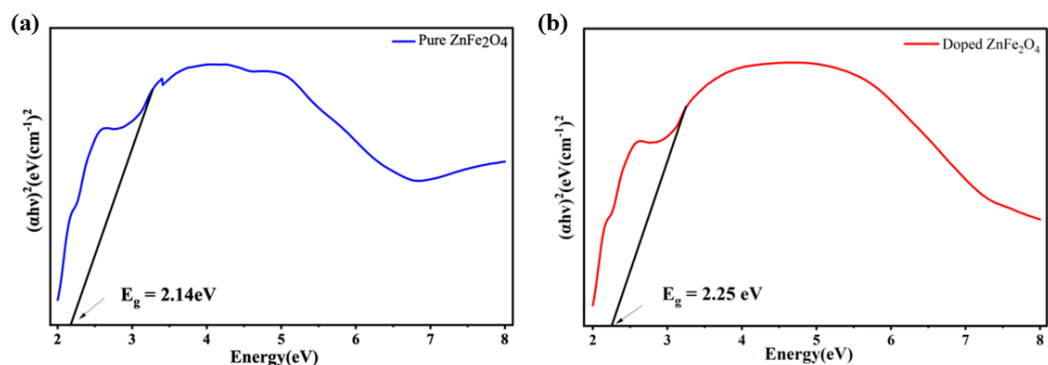


Figure 8. Tauc's Plot for optical band gap of (a) pure ZnFe_2O_4 and (b) Ni-doped ZFO nanoparticles.

Group theory predicts that the cubic spinel structure (space group Fd-3m) has five active Raman phonon modes: $A_{1g} + E_g + 3T_{2g}$. Out of these, the mode A_{1g} corresponds to the symmetric stretching vibration of oxygen in the vicinity of the tetrahedrally coordinated cations, the E_g mode corresponds to symmetric bending vibrations, and the T_{2g} modes correspond to asymmetric stretching and translational vibrations due to both tetrahedral and octahedral metal-oxygen bonds. The Raman modes in the cubic spinel structure are highly sensitive to the distribution of cations and structural distortions [47].

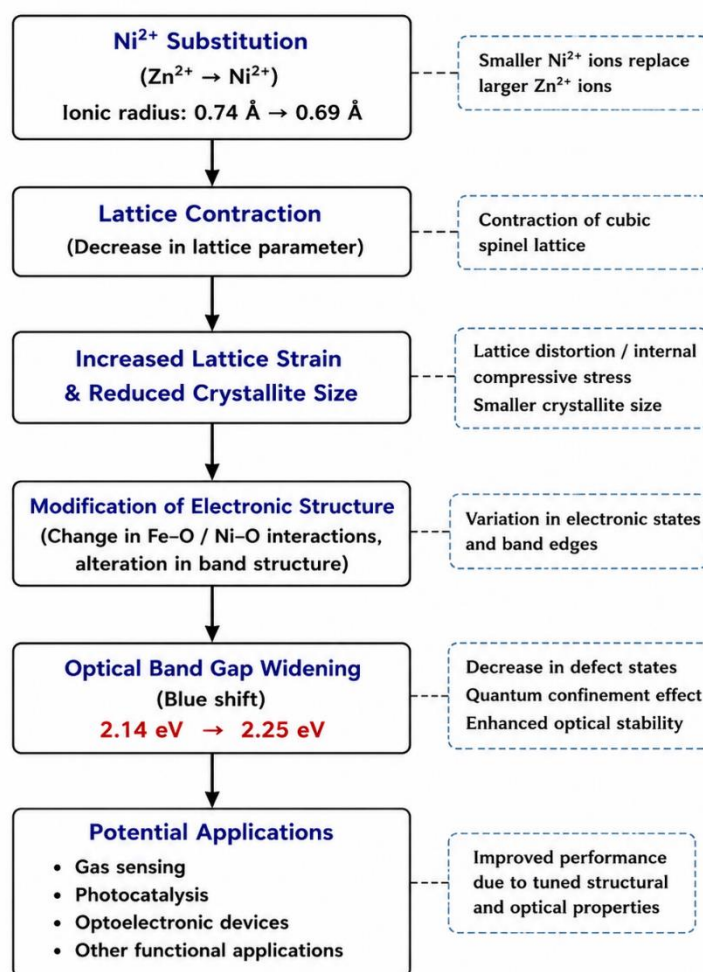


Figure 9. Schematic illustration showing the influence of Ni substitution on the structural modifications and optical properties of ZnFe₂O₄ nanoparticles.

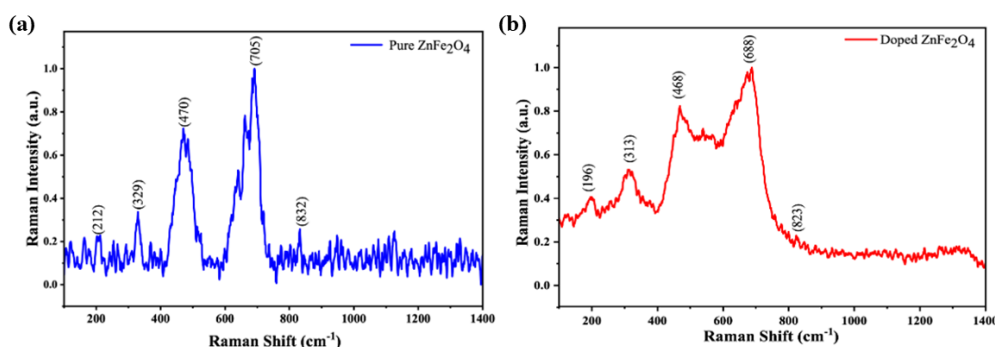


Figure 10. Raman Spectroscopy of (a) pure ZnFe₂O₄ and (b) Ni-doped ZFO nanoparticles.

In the present study, the prominent Raman peak observed at ~705 cm⁻¹ in the pure sample, attributed to symmetric stretching vibrations of metal-oxygen bonds at tetrahedral sites (A_{1g} mode), shifts to ~688 cm⁻¹ in the Ni-doped sample. A secondary Raman band observed near 470 cm⁻¹, corresponding to one of the T_{2g} vibrational modes of the spinel lattice, shifts slightly to approximately 468 cm⁻¹ after Ni substitution. This shift reflects changes in the local metal–oxygen bonding environment resulting from the redistribution of cations within the spinel lattice. The observed peak shifts indicate lattice distortion induced by Ni incorporation and further

confirm modifications in the local crystal structure. The shift of the Raman bands toward lower wavenumbers (red shift) suggests weakening of the metal–oxygen (M–O) bonds accompanied by a slight increase in bond length, thereby reducing the vibrational frequency through a decrease in the bond force constant [48].

Minor variations in peak position and intensity are attributed to changes in cation arrangement, which influence lattice strain and vibrational behaviour of the spinel framework, highlighting the sensitivity of the ZnFe_2O_4 structure to internal structural modifications. Red-shifting of the Raman bands due to Ni incorporation suggests that there is a change in the local metal–oxygen environment due to partial cationic distribution within the tetrahedral and octahedral sites. Introduction of Ni^{2+} brings about localized distortion of the lattice due to the difference in ionic radius and bond characteristics of Ni^{2+} relative to Zn^{2+} . Changes in structure bring about alteration of the force constants of metal–oxygen bonds, which change the vibrational frequency of the bonds [47].

In addition to lattice distortion, Ni incorporation introduces local structural disorder arising from partial cation redistribution within the tetrahedral and octahedral sites of the spinel lattice. The coexistence of Zn^{2+} and Ni^{2+} ions modifies the local crystal symmetry and produces slight variations in the metal–oxygen coordination environment, leading to subtle changes in phonon behaviour. These observations further support the conclusion that the Raman peak shifts arise from both lattice distortion and localized structural disorder while preserving the overall cubic spinel framework. Similar Raman behaviour associated with structural modification and changes in the local metal–oxygen environment has been reported by Somvanshi et al. [21], where the surface transformation of zinc ferrite nanoparticles influenced their physicochemical properties and magnetic performance. The present Raman results are consistent with these reported structure–property relationships.

The combined effects of lattice contraction, reduced crystallite size, improved crystallinity, and optical band gap widening demonstrate that Ni substitution effectively tailors the structural and electronic properties of ZnFe_2O_4 nanoparticles. These modifications promote enhanced surface reactivity, increased oxygen adsorption, and improved charge transport, which are critical parameters for high-performance gas sensors. Furthermore, the improved optical characteristics indicate the potential applicability of the synthesized nanoparticles in photocatalysis, environmental monitoring, and other multifunctional semiconductor-based devices [48].

5. Conclusion

In this study, pure and Ni-substituted ZnFe_2O_4 nanoparticles were successfully synthesized using an energy-efficient solution combustion technique. The comparative analysis of the structural, vibrational, morphological, and optical properties revealed that substitution of Ni greatly affects the lattice parameter, crystallite size, lattice strain, Raman properties, and optical bandgap of ZnFe_2O_4 . X-ray diffraction (XRD) structural analysis showed the presence of a cubic spinel structure, which was a single-phase structure, and the space group of the single-phase structure was Fd-3m , with no impurities detected. The calculated average crystallite

sizes of pure and doped ZnFe_2O_4 were found to be 34.9 nm and 15.07 nm, respectively, indicating the formation of nanostructured ferrite particles. Williamson–Hall analysis further confirmed that Ni substitution increased the lattice strain, supporting the observed lattice contraction and reduction in crystallite size. The morphological analysis of the nanoparticles through SEM showed that the nanoparticles are mostly in the form of spheres, which are uniformly dispersed. Despite some agglomeration, which was caused by strong dipole-dipole magnetic interaction, the general microstructure was very structured and aligned with the XRD data. Besides, the effective construction of the spinel lattice was confirmed by FTIR and Raman spectroscopy, which revealed the typical metal-oxygen vibrational modes at tetrahedral and octahedral sites. A direct band gap was observed under optical studies through UV-Visible spectroscopy; this proved the semiconducting property of the material synthesized. It is possible to fine-tune the optical properties through the structural changes that were observed in the form of a noticeable blue shift in the absorption edge. To sum up, structural stability, controlled morphology, and tunable optical properties make these ZnFe_2O_4 nanoparticles very promising in the future for multifunctional applications.

Although the present work is limited to the structural, vibrational, morphological, and optical characterization of pure and Ni-substituted ZnFe_2O_4 nanoparticles, magnetic characterization, including saturation magnetization, coercivity, remanence, and magnetic heating behavior, was beyond the scope of the present study. The observed improvements in lattice characteristics and tunable optical properties indicate their potential suitability for future investigations in magnetic, photocatalytic, gas sensing, and biomedical applications, including magnetic hyperthermia and theranostic platforms. Future studies involving magnetic, biological, and application-specific evaluations will be necessary to validate these potential applications.

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