

## Article

# Tailoring Optical Properties via Ru Doping and Magnetic Properties via Ce Doping in $\alpha\text{-Fe}_{2-4x}\text{Z}_{3x}\text{O}_3$ (Z = Ce, Ru) Solid-Solution Nanoparticles

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## Abstract

$\alpha\text{-Fe}_{2-4x}\text{Z}_{3x}\text{O}_3$  (Z = Ce, Ru) nanoparticles were synthesized via a conventional solid-state reaction route. X-Ray diffraction analysis confirmed that all compositions crystallize in the single-phase hexagonal hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) structure, with no detectable secondary phases. Cerium substitution resulted in a pronounced reduction in crystallite size accompanied by a progressive narrowing of the optical band gap, which decreased to approximately 1.73 eV at higher Ce contents. The optical properties were further investigated through absorption coefficient, optical transmittance, and complex refractive index analyses, revealing that cerium-doped hematite exhibits enhanced light-harvesting capability, highlighting its strong potential for optoelectronic and solar-energy conversion applications. Magnetic hysteresis measurements on  $\alpha\text{-Fe}_{2-4x}\text{Ru}_{3x}\text{O}_3$  samples showed a systematic increase in both coercive field ( $H_c$ ) and remanent magnetization ( $M_r$ ) with increasing Ru concentration. This magnetic hardening behavior is attributed to strengthened magnetocrystalline and shape anisotropy induced by Ru incorporation into the hematite lattice. Mössbauer spectroscopy confirmed the presence of  $\text{Fe}^{3+}$  and  $\text{Ru}^{4+}$  species, providing valuable insight into the oxidation states and local magnetic environments within the corundum-type structure.

**Keywords:** Ce/Ru-doped hematite; solid-solution nanoparticles; band gap engineering; oxygen vacancies; magnetic anisotropy; Mössbauer spectroscopy

## 1. Introduction

Oxides have attracted considerable research interest owing to their wide range of potential applications in optical devices, including telecommunications, optical filters, interference coatings, and solar cells [1,2]. Among these oxides, iron oxide is particularly attractive owing to its band gap within the visible spectral range and its remarkable chemical stability in aqueous environments, making it well suited for electrochemical applications [3]. These properties make iron oxide a promising candidate for a wide range of applications, particularly as a photoanode material for water oxidation reactions [4]. Among the various iron oxide polymorphs, hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) is considered the most stable and reliable phase [5]. As an n-type semiconductor, it offers several advantages, including low cost, wide availability, excellent thermal stability, and non-toxicity [6], as well as physical properties that are closely related to its magnetic and electronic behavior [7]. From this perspective, hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) exhibits a band gap of approximately 2.2 eV, enabling it to absorb nearly 40% of incident sunlight. This feature makes it highly attractive compared to conventional optoelectronic materials. In nanoparticle form, hematite



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is particularly interesting for magnetic studies, as it can exhibit antiferromagnetic, weak ferromagnetic, and superparamagnetic behaviors [8]. In its bulk form, hematite exhibits a Néel temperature of approximately 960 K and undergoes the Morin transition at around 263 K. Above the Morin transition, it behaves as a weak ferromagnet, whereas below this temperature, it adopts an antiferromagnetic state [9]. Substituting elements at the Fe site in  $\alpha\text{-Fe}_2\text{O}_3$  hematite—such as Sn, Zn, In, Co, Ni, Cr, and Mn—can significantly modify its physical properties [10]. In this context, lanthanide doping, such as  $\text{Ce}^{4+}$ , is particularly interesting because it can simultaneously alter the electronic structure and optical properties of hematite. Ce doping has been shown to narrow the effective bandgap and enhance visible-light absorption in  $\text{Fe}_2\text{O}_3$ , which is critical for solar energy applications [11]. Moreover, Ce incorporation can generate oxygen vacancies and defect levels, potentially modulating carrier density and electron–hole recombination.

In comparison, 4d transition metal dopants such as Ru have been reported to significantly modify the electronic and optical properties of hematite. Ru doping can reduce the effective bandgap, increase visible-light absorption, and improve electronic mobility by modifying the conduction band [12]. Some recent studies also suggest that  $\text{Ru}^{3+}$  dopants can affect the magnetic and electronic properties of  $\alpha\text{-Fe}_2\text{O}_3$ , although these effects remain underexplored [13].

Despite these efforts, a clear understanding of how Ce doping influences oxygen vacancies, strongly localized 4f electronic states, and visible-light absorption in hematite is still lacking. Furthermore, the relative effects of Ce versus Ru doping have not been systematically established.

In this context, the present study aims to investigate how cerium doping influences the structural, morphological, optical, and magnetic characteristics of hematite.

## 2. Experimental

$\alpha\text{-Fe}_2\text{O}_3$  nanoparticles (NPs) were synthesized using a solid-state technique [14], employing commercial  $\text{Fe}_2\text{O}_3$ ,  $\text{RuO}_2$ , and  $\text{CeO}_2$  powders of 99.9% purity (Sigma-Aldrich, St. Louis, MO, USA) as precursors. For the synthesis, Ce-doped  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles were prepared with  $x = 0, 0.067, 0.134$ , and  $0.2$ , corresponding to nominal Ce contents of 0%, 10%, 20%, and 30% in the Fe sublattice. Similarly, Ru-doped  $\alpha\text{-Fe}_{2-4x}\text{Ru}_{3x}\text{O}_3$  nanoparticles were synthesized with nominal compositions  $x = 0, 0.01, 0.0167$ , and  $0.0267$ . The nominal Fe and Ru contents were slightly adjusted (e.g.,  $\alpha\text{-Fe}_{1.97}\text{Ru}_{0.03}\text{O}_3$  for  $x = 0.01$ ) to ensure that the prepared compositions closely match the intended  $\alpha\text{-Fe}_{2-4x}\text{Ru}_{3x}\text{O}_3$  stoichiometry, maintaining controlled substitution of Ru for Fe in the hematite lattice. The precursors were thoroughly mixed in an agate mortar and subsequently ground in a planetary ball mill Pulverisette 7 (Fritsch, Idar-Oberstein, Germany) for 8 h at 800 rpm to achieve the desired stoichiometry. The resulting mixture was annealed at 900 °C in air for 3 h. The calcined powders were then pressed into pellets (~10 mm in diameter and 0.1 mm thick) using a uniaxial press (Specac, Orpington, UK) and further annealed at 1200 °C for 5 h to densify the samples.

The crystal structure of the samples was characterized by X-Ray diffraction (Siemens D501, Munich, Germany) in the  $2\theta$  range of 20–80°, using  $\text{Cu K}\alpha$  radiation, a step size of 0.0167°, and a counting time of 18 s per step. The phase purity and uniform incorporation of the dopants were confirmed by XRD analysis and Rietveld refinement, which showed no secondary phases and consistent lattice parameter changes with increasing dopant content. Magnetic measurements were performed with a vibrating sample magnetometer (VSM, 9600-1, LDJ Electronics, Troy, MI, USA) only for the Ru-doped  $\alpha\text{-Fe}_{2-4x}\text{Ru}_{3x}\text{O}_3$  nanoparticles, while the optical properties of the Ce-doped  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles were investigated using UV–Vis spectrophotometry (PerkinElmer PE 1050, Waltham, MA,

USA). To ensure the reliability of the optical measurements despite the small pellet thickness, repeated UV–Vis measurements were performed on several pellets prepared under identical experimental conditions. The obtained spectra showed good reproducibility, with a standard deviation lower than 3%, confirming the consistency and reliability of the measured data.

### 3. Results and Discussion

#### 3.1. Structural and Morphological Analysis

Figure 1a shows the XRD patterns of cerium-doped  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.2$ ). All samples exhibited only the hematite-like phase ( $\alpha\text{-Al}_2\text{O}_3$ -type corundum), in agreement with standard reference data. The shift in the most intense diffraction peak corresponding to the (104) plane toward lower angles with increasing Ce content clearly indicates the successful incorporation of Ce ions into the  $\alpha\text{-Fe}_2\text{O}_3$  lattice. The crystallite size ( $D$ ) of the nanoparticles was estimated from the (104) diffraction peak using the Debye–Scherrer formula [15]:

$$\beta \cos(\theta) = k\lambda / D \quad (1)$$

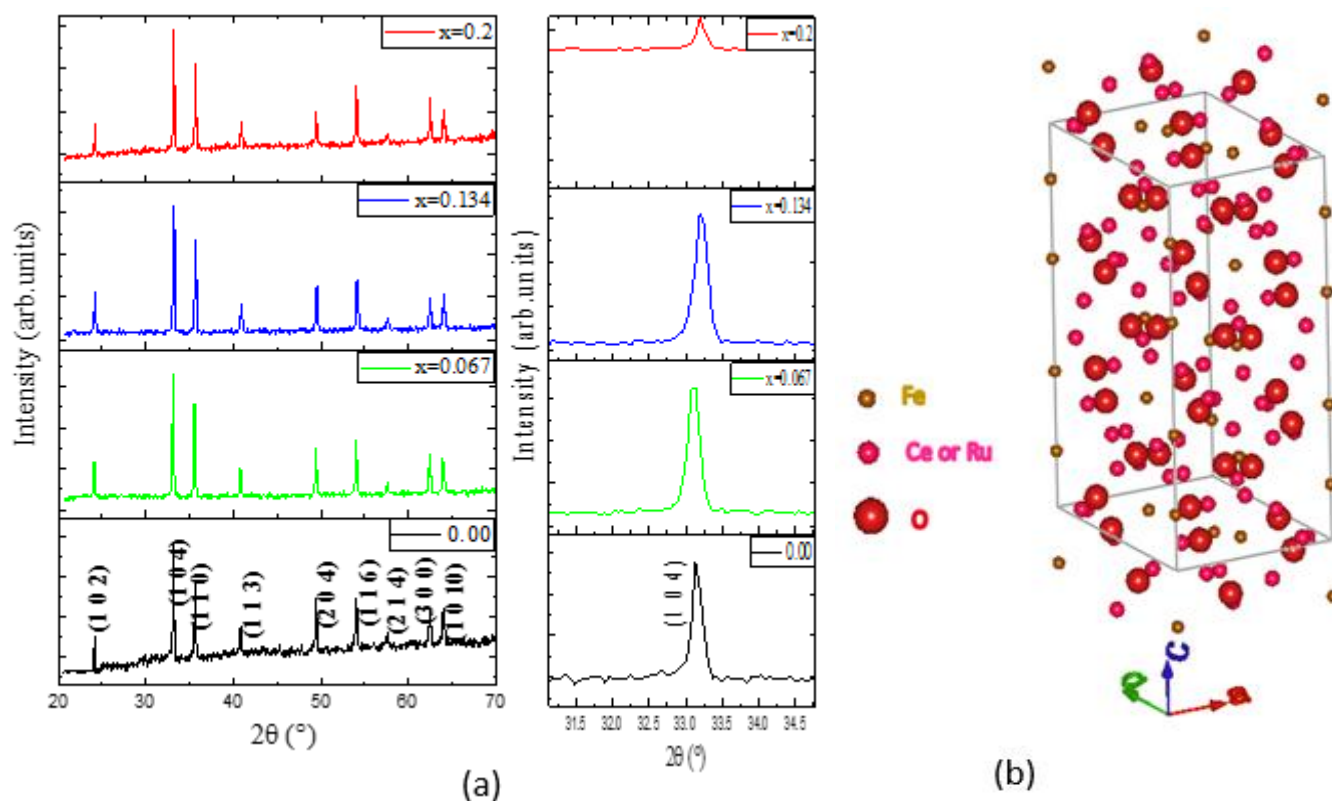


**Figure 1.** (a) X-Ray diffraction patterns of  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  ( $0 \leq x \leq 0.2$ ) nanoparticles, (b) the structure of  $\alpha\text{-Fe}_{2-4x}\text{Z}_{3x}\text{O}_3$  with ( $Z = \text{Ru}, \text{Ce}$ ), respectively.

In the Debye–Scherrer equation,  $\beta$  represents the full width at half maximum (FWHM) of the diffraction peak,  $\theta$  corresponds to the peak position,  $k$  is a constant equal to 0.94, and  $\lambda$  is the wavelength of the X-Ray radiation. The peak broadening parameter  $\beta$  was calculated using FullProf 8.0 software [16]. As the Ce content ( $x$ ) increases from 0 to 0.2, the crystallite size decreases from 141 to 95 nm. This reduction is attributed to the substitution of larger  $\text{Ce}^{4+}$  ions ( $R = 0.97 \text{ \AA}$ ) for smaller  $\text{Fe}^{3+}$  ions ( $R = 0.645 \text{ \AA}$ ), demonstrating the effect of Ce doping on the crystallite size.

To evaluate the effect of defects on the structure of  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  ( $0 \leq x \leq 0.2$ ), the dislocation density ( $\delta$ ), defined as the total length of dislocation lines per unit volume ( $\text{lines}/\text{m}^2$ ), can be calculated. For the powders, the dislocation density ( $\delta$ ) is given by [17]:

$$\delta = \frac{n}{D^2} \quad (2)$$

When the dislocation density reaches its minimum, the number of dislocations ( $n$ ) can take a value of 1. As shown in Table 1, the dislocation density increases with decreasing crystallite size. Additionally, the high-energy milling at 800 rpm for 8 h used to prepare the Ce-doped  $\alpha\text{-Fe}_2\text{O}_3$  nanoparticles may introduce lattice strain and defects, which can slightly reduce crystallinity and broaden the XRD peaks. To evaluate the internal stresses induced in the crystal lattice by doping, the microstrain ( $\varepsilon$ ) was considered as a key parameter. The relationship governing this parameter is given by the following equation [18]:

$$\varepsilon = \frac{\beta_D}{4\tan(\theta)} \quad (3)$$

The morphology and particle size of cerium-doped  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.2$ ) were examined using scanning electron microscopy (FEI Quanta 450 FEG, Hillsboro, OR, USA), as shown in Figure 2a,b. The microstructure is predominantly composed of large, grey, spherical grains. The average grain size decreases from 1.5  $\mu\text{m}$  for undoped hematite to 0.95  $\mu\text{m}$  for  $x = 0.2$  Ce-doped hematite. It should be noted that the grain sizes observed via SEM are larger than those estimated from XRD, likely due to the agglomeration of multiple crystallites within each grain. Nonetheless, the SEM observations are consistent with the XRD results, confirming the trend of grain size reduction with increasing Ce doping.

**Table 1.** Some structural parameters of un-doped and Ce-doped  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  powders ( $0 \leq x \leq 0.2$ ).

| Sample      | Lattice Parameter                                 | Crystallite Size (nm) | Dislocation Density $\times 10^{13}$ Lines/ $\text{m}^2$ | Strain [%] |
|-------------|---------------------------------------------------|-----------------------|----------------------------------------------------------|------------|
| $x = 0.00$  | $a = 5.03 \text{ \AA}$<br>$c = 13.74 \text{ \AA}$ | 141                   | 5.029                                                    | 0.138      |
| $x = 0.067$ | $a = 5.03 \text{ \AA}$<br>$c = 13.74 \text{ \AA}$ | 134                   | 5.569                                                    | 0.147      |
| $x = 0.134$ | $a = 5.03 \text{ \AA}$<br>$c = 13.75 \text{ \AA}$ | 118                   | 7.181                                                    | 0.156      |
| $x = 0.2$   | $a = 5.03 \text{ \AA}$<br>$c = 13.75 \text{ \AA}$ | 95                    | 11.080                                                   | 0.183      |

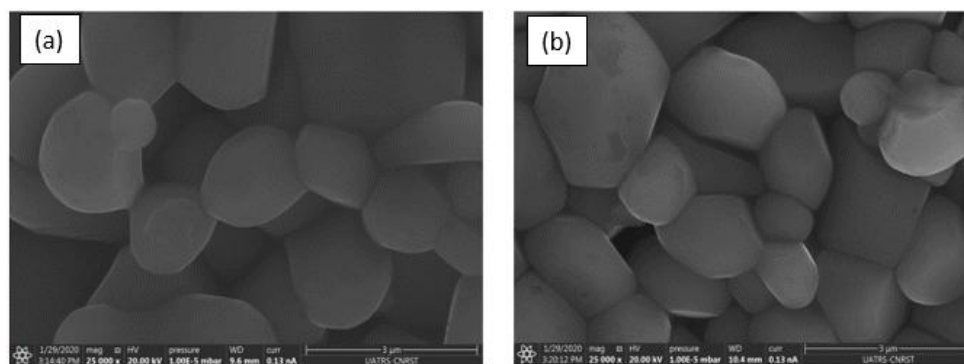


**Figure 2.** Typical scanning electron microscopy (SEM) micrographs of  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles with (a)  $x = 0$  and (b)  $x = 0.2$ , respectively.

### 3.2. Optical Transmission and Reflection Spectra

The absorbance spectra,  $A(\lambda)$ , of  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.2$ ) are presented in Figure 3a. All samples exhibit significant optical absorption throughout the visible region. The undoped hematite shows comparatively lower absorbance, whereas the Ce-doped samples display enhanced absorption intensity, indicating improved light-harvesting capability upon cerium incorporation. A pronounced absorption enhancement is observed in the 410–580 nm region for the Ce-doped compositions. This behavior can be attributed to the formation of impurity-induced localized energy levels within the band gap, which facilitate additional electronic transitions and promote electron–hole pair generation.

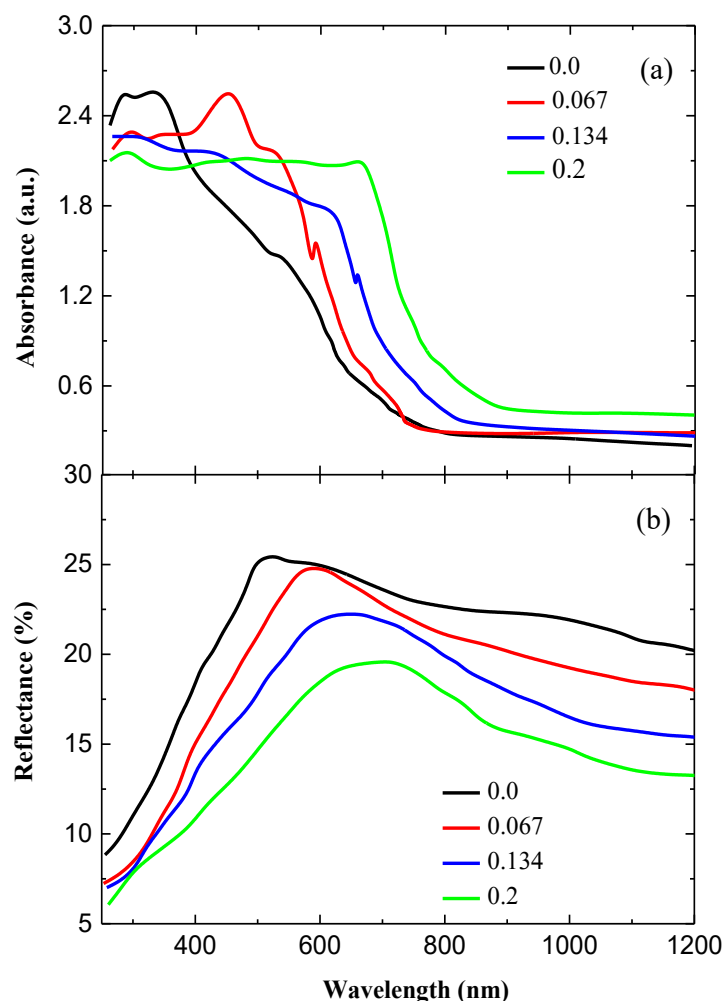


**Figure 3.** (a) Absorbance and (b) reflectance spectra of  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.2$ ).

The increase in absorbance with Ce content suggests that cerium doping introduces donor-type states near the conduction band, leading to a higher density of free charge carriers capable of absorbing lower-energy photons. Consequently, Ce incorporation effectively narrows the optical transition pathway and improves the visible-light absorption efficiency of  $\alpha\text{-Fe}_2\text{O}_3$ .

The reflectance,  $R(\lambda)$ , was determined from the transmittance,  $T(\lambda)$ , and the absorption coefficient,  $\alpha(\lambda)$ , using the appropriate relation between these optical parameters [19]:

$$\alpha = \frac{1}{d} \ln \left[ \frac{(1-R)^2}{T} \right] \quad (4)$$

The calculated reflectance,  $R(\lambda)$ , of  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.2$ ) is shown in Figure 3b. The spectra reveal two distinct regions. In the 400–610 nm range, the electronic excitation transitions from  $^1\text{A}_g \rightarrow ^1\text{A}_g$  to  $^4\text{T}_1 \rightarrow ^4\text{T}_1$ , occurring at approximately 580 nm for the undoped sample; this transition shifts to 500 nm for  $x = 0.2$  upon  $\text{Ce}^{4+}$  doping. Additionally, the maximum reflectance increases from 18% to 27% as the  $\text{Ce}^{4+}$  content rises from 0 to 0.2, which can be attributed to changes in the refractive index induced by Ce incorporation. The 610–900 nm region corresponds to the ligand field transition from  $^1\text{A}_g$  to  $^4\text{T}_2$ , observed around 700 nm.

### 3.3. Direct and Indirect Optical Band-Gap Energies

The optical band gap of the nanoparticles can be determined using the Tauc relation, which correlates the absorption coefficient ( $\alpha$ ) with the photon energy ( $h\nu$ ) as follows [20]:

$$\alpha h\nu = \alpha_0 (h\nu - E_g)^m \quad (5)$$

- $h\nu$  corresponds to the photon energy;
- $m$  equals 1/2 or 2 for direct or indirect transition and  $\alpha_0$  an optical constant.

In this study, the absorption coefficient ( $\alpha$ ) was calculated from the absorbance spectra ( $A$ ) using the relation  $\alpha = 2.3026 \times A/d$ , where  $d = 0.1$  mm is the thickness of the pellet. Although both direct ( $m = 1/2$ ) and indirect ( $m = 2$ ) optical transitions have been reported in the literature for  $\alpha\text{-Fe}_2\text{O}_3$ , both contributions can occur depending on experimental conditions [21]. In this work, the analysis focuses primarily on the direct transition, which provides a consistent estimation of the optical band gap for the Ce-doped samples. The optical band gap ( $E_g$ ) was determined by extrapolating the linear portion of the  $(\alpha h\nu)^2$  versus  $h\nu$  plot to  $(\alpha h\nu)^2 = 0$ . As shown in Figure 4b, the band gap of  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.2$ ) decreases from 2.22 eV for the undoped sample to 1.73 eV for the sample with  $x = 0.2$ . The  $E_g$  value of undoped hematite is in good agreement with previously reported literature [22,23]. The reduction in band gap with Ce doping is attributed to the presence of defects and unoccupied 4f orbitals of the lanthanide ions, which lie at lower energy compared to the Fe 3d orbitals, thereby decreasing the energy required for electron transitions from  $\text{O}^{2-}$  to these unoccupied orbitals. More generally, doping introduces additional tail states, which contribute to the observed decrease in  $E_g$ .

### 3.4. Urbach Energy

Substitution of Fe by Ce introduces Urbach tails due to the generation of localized electronic states. The Urbach energy ( $E_U$ ) exhibits an inverse relationship with the optical band gap,  $E_g$ , as shown in Figure 4a. The presence of Urbach tails provides valuable information about structural defects and disorder induced by Ce doping [24]. The Urbach energy ( $E_U$ ) was estimated from the linear fit of  $\ln(\alpha)$  versus  $h\nu$  using the relation:

$$\alpha = \alpha_0 \exp\left(\frac{h\nu}{E_U}\right) \quad (6)$$

Or

$$\ln(\alpha) = \ln(\alpha_0) + \left(\frac{h\nu}{E_U}\right) \quad (7)$$

It can be observed from Figure 4b that in Ce-doped hematite, the substitution of  $\text{Ce}^{4+}$  for  $\text{Fe}^{3+}$  generates oxygen vacancies to maintain charge balance, leading to the formation of localized states. The Urbach energy ( $E_U$ ) increases steadily with Ce concentration, while, inversely, the optical band gap ( $E_g$ ) decreases correspondingly. The formula used in this process involves a constant  $\alpha_1$  and the Urbach energy  $E_U$ .

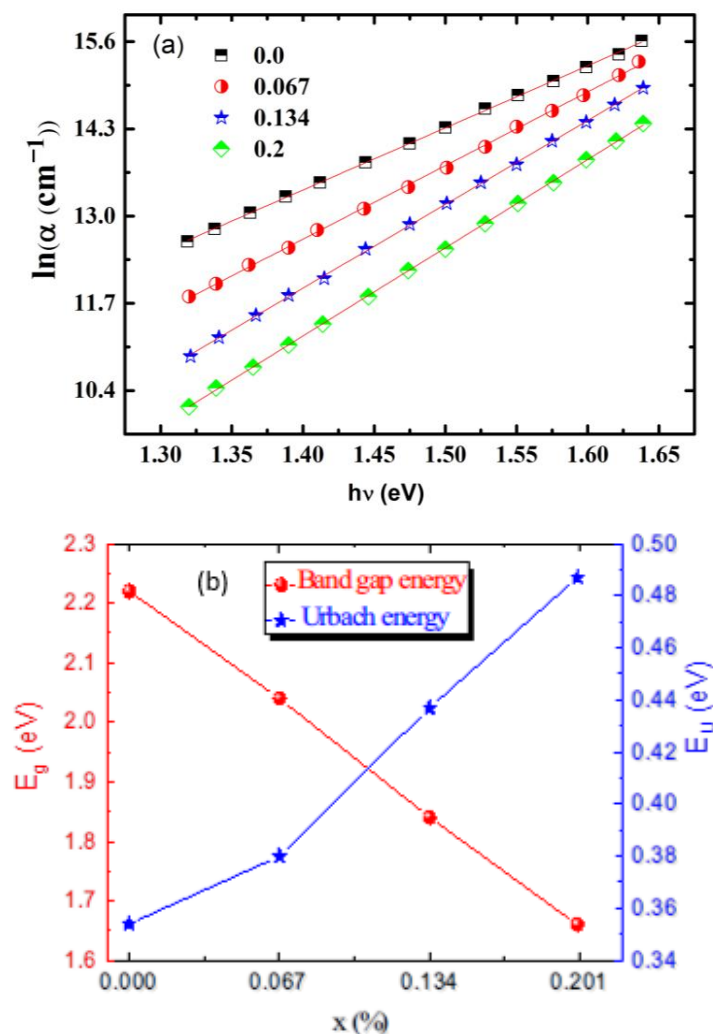


**Figure 4.** (a) Representation of  $\ln(\alpha)$  versus  $(h\nu)$  to evaluate the Urbach energy or the band tail width ( $E_U$ ) of  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.2$ ). (b) Change in band gap ( $E_g$ ) and Urbach ( $E_U$ ) energies as a function of Ce content( $x$ ) in  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.2$ ).

### 3.5. The Complex Optical Refractive Index

The propagation of light through a material and its wavelength influences the refractive index, which can be expressed as a complex quantity using the relation [25]:

$$\hat{n}_{\text{complex}} = n(\lambda) + ik(\lambda) \quad (8)$$

The refractive index of a material depends on both its intrinsic physical properties and the wavelength of the incident light, and it is expressed as a complex quantity with a real part,  $n(\lambda)$ , and an imaginary part,  $k(\lambda)$ . For the optically isotropic and homogeneous nanoparticles studied here, the real part of the refractive index was determined using the Fresnel equation:

$$n(\lambda) = \left( \frac{1+R}{1-R} \right) + \sqrt{\frac{4R}{(1-R)^2} - k^2} \quad (9)$$

Figure 5a shows the variation in the refractive index,  $n(\lambda)$ , for  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles. The results indicate that Ce-doped hematite exhibits a higher refractive index compared to undoped hematite. This increase is attributed to the higher density and enhanced polarizability arising from the Ce 4f and 5d orbitals, which contribute to stronger interaction with and attenuation of the propagating light [26].

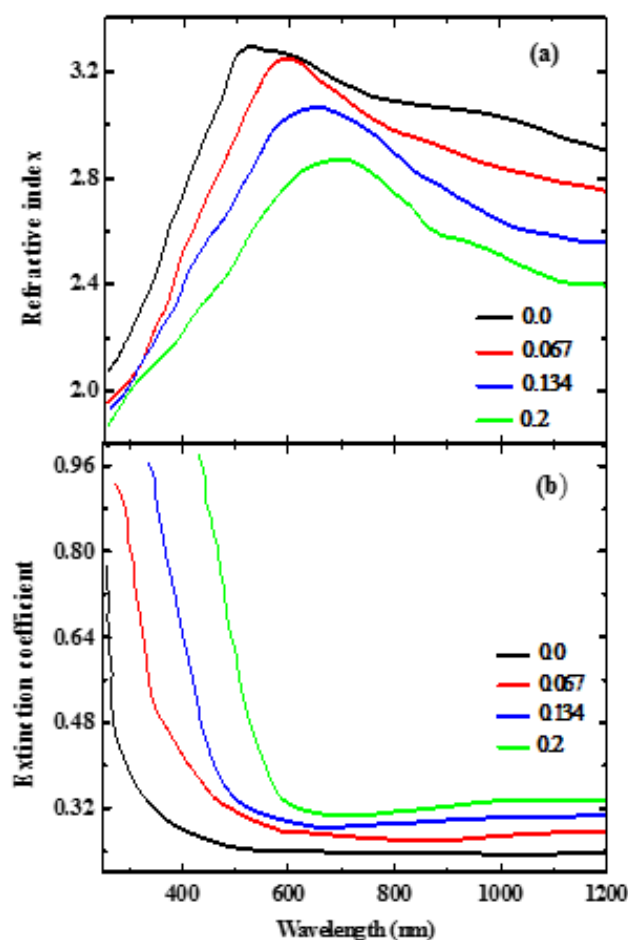


**Figure 5.** (a) Variation in the refractive index  $n(\lambda)$  of  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.2$ ) with the incident wavelength. (b) Corresponding extinction coefficient  $k$  change spectra.

The refractive index exhibits a peak in the 580–610 nm range upon Ce doping, indicating anomalous dispersion associated with energy transfer between two electronic states. At longer wavelengths, the refractive index follows normal dispersion behavior, reflecting the optical homogeneity of the material [27].

The extinction coefficient,  $k(\lambda)$ , is a key parameter that quantifies the amount of energy absorbed by a material during the propagation of electromagnetic waves. Its value is influenced by the density of free charge carriers and structural defects within the material. The extinction coefficient can be related to the absorption coefficient,  $\alpha(\lambda)$ , and the wavelength,  $\lambda$ , through the following equation:

$$k(\lambda) = \frac{\alpha\lambda}{4\pi} \quad (10)$$

As shown in Figure 5b, the extinction coefficient,  $k(\lambda)$ , increases in Ce-doped hematite compared to pure hematite, indicating enhanced light absorption at the nanoparticle surface. Doping with  $\text{Ce}^{4+}$  partially substitutes  $\text{Fe}^{3+}$  in the lattice, creating oxygen vacancies and crystalline defects (as commonly observed in rare-earth doping of  $\alpha\text{-Fe}_2\text{O}_3$ ) [28]. These defects can increase local polarizability and modify the refractive index ( $n$ ), while localized electronic states introduced by the dopant influence the extinction coefficient ( $k$ ) by enhancing absorption in the visible range. Consequently, increasing the Ce doping level leads to a slight increase in  $n$  and corresponding changes in the absorption features observed in  $k$ . This behavior facilitates greater light–matter interaction, improving light penetration and the overall optical performance of the material.

### 3.6. Real and Imaginary Dielectric Coefficient

The dielectric constant reflects the response of a material to an applied electric field and is related to the electrical polarizability of the constituent atoms or molecules. While it is generally a tensor quantity that depends on the crystallographic orientation, it reduces to a scalar in isotropic media. The dielectric constant is typically expressed as a complex quantity, with the imaginary component representing the absorption or dissipation of energy from the electromagnetic field.

The complex dielectric function,  $\epsilon_{\text{complex}}$ , consists of two components: the real part,  $\epsilon_1$ , and the imaginary part,  $\epsilon_2$ , which are related to the real refractive index,  $n(\lambda)$ , and the extinction coefficient,  $k(\lambda)$ , respectively. These relationships can be expressed as [29]:

$$\epsilon_1(\lambda) = n^2(\lambda) - k^2(\lambda) \quad (11)$$

$$\epsilon_2(\lambda) = 2n(\lambda)k(\lambda) \quad (12)$$

Figure 6a,b show the variation in the real ( $\epsilon_1$ ) and imaginary ( $\epsilon_2$ ) parts of the dielectric function as a function of wavelength. At longer wavelengths, both  $\epsilon_1$  and  $\epsilon_2$  remain relatively constant, whereas a pronounced increase is observed at shorter wavelengths, reaching a maximum at  $\lambda = 648$  nm. This peak becomes more prominent with increasing Ce doping. Both components are in good agreement with previously reported values for hematite [30]. The enhancement of the dielectric response is closely linked to the increase in optical conductivity, which is governed by the density of free charge carriers [31]. In hematite, oxygen vacancies act as shallow donor defects, creating electronic states near the bottom of the conduction band [32]. The formation of an oxygen vacancy releases two electrons per missing oxygen atom, thereby increasing the concentration of majority carriers. This simultaneously enhances electrical conductivity and interfacial polarization, leading to higher dielectric permittivity and dielectric losses [33,34].

### 3.7. The Magnetic Hysteresis Cycles

The magnetic properties of  $\alpha\text{-Fe}_{2-4x}\text{Ru}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.0267$ ) were investigated by measuring hysteresis loops under an applied magnetic field ranging from  $-10$  to  $10$  kOe, as shown in Figure 7. From these curves, the remanent magnetization ( $M_r$ ), saturation magnetization ( $M_{\text{sat}}$ ), and coercive field ( $H_c$ ) were determined at room temperature. The results indicate that all prepared samples exhibit ferromagnetic behavior. The coercive fields measured were 794, 1316, 1371, and 2783 Oe for  $x = 0.00, 0.01, 0.0167$ , and  $0.0267$ , respectively (see Table 2). The highest coercivity observed for  $\alpha\text{-Fe}_{1.92}\text{Ru}_{0.08}\text{O}_3$  is attributed to its shape and enhanced magnetocrystalline anisotropy. Notably, magnetization did not reach saturation even at the maximum applied field of  $10$  kOe. The undoped  $\alpha\text{-Fe}_2\text{O}_3$  sample exhibits weak ferromagnetism, with  $M_r = 0.434$  emu/g and  $H_c = 794$  Oe. The observed magnetic behavior can be explained by the presence of oxygen vacancies in the hematite lattice. These defects induce local lattice distortions, modifying the Fe–O–Fe bond angles and exchange pathways, and consequently altering the superexchange interactions that govern the intrinsic antiferromagnetic order of  $\alpha\text{-Fe}_2\text{O}_3$  [35]. Furthermore, oxygen vacancies facilitate the partial reduction of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  to maintain charge neutrality, creating mixed-valence states that enhance ferromagnetic exchange interactions [36]. As a result, the magnetocrystalline anisotropy is altered, and the local spin–orbit coupling is enhanced, contributing to the observed increase in net magnetization and the changes in coercivity [37]. In other studies, Hill et al. investigated the magnetic properties of both solid and mesoporous  $\alpha\text{-Fe}_2\text{O}_3$  (hematite) at room and low temperatures [38]. For bulk  $\alpha\text{-Fe}_2\text{O}_3$ , Hill et al. observed a non-hysteretic loop at  $12$  K, consistent with the behavior of a typical antiferromagnet, and a hysteresis loop at  $295$  K, indicative of weak ferromagnetism. At

room temperature, the remanent magnetization and coercive field for solid and mesoporous  $\alpha\text{-Fe}_2\text{O}_3$  were reported as  $M_r = 0.28$  emu/g and  $H_c = 0.17$  emu/g, respectively. These values differ from those obtained in the present study for  $\alpha\text{-Fe}_2\text{O}_3$ , likely due to differences in nanoparticle morphology. In a separate study, Varshney et al. investigated the effect of Ti doping on hematite and reported that Ti-doped samples exhibited increased coercivity compared to undoped hematite, demonstrating the influence of dopants on the magnetic properties [39]. In the present study, Ru doping of  $\alpha\text{-Fe}_{2-4x}\text{Ru}_{3x}\text{O}_3$  ( $0 \leq x \leq 0.2$ ) induces gradual changes in the hysteresis behavior, indicating that the magnetic properties are highly sensitive to the doping concentration. It is well established that the magnetization of ferromagnetic materials is strongly influenced by the sample's morphology and structure, which are determined by the synthesis method. Accordingly, the enhanced remanent magnetization and increased coercivity observed in the  $x = 0.0267$  Ru-doped  $\alpha\text{-Fe}_{2-4x}\text{Ru}_{3x}\text{O}_3$  superstructures can be attributed to shape anisotropy, which restricts the magnetization along the easy axes, thereby leading to higher remanence and coercivity. The Mössbauer spectra were analyzed using a single symmetric Lorentzian sextet model, corresponding to (Ru-doped) hematite. Room-temperature spectra were fitted with the NORMOS<sup>TM</sup>-90 software (R. A. Brand, University of Duisburg, Germany) [40], and all samples exhibit a well-defined magnetic ordering (Figure 8). The obtained chi-square ( $\chi^2$ ) values are minimal, indicating that the NORMOS-SITE fitting procedure provides an excellent match to the experimental data. The extracted Mössbauer parameters correspond to natural iron and are in good agreement with previously reported results [41]. For the Ru-doped samples, additional fitting models were employed to achieve the best agreement with the experimental Mössbauer spectra.

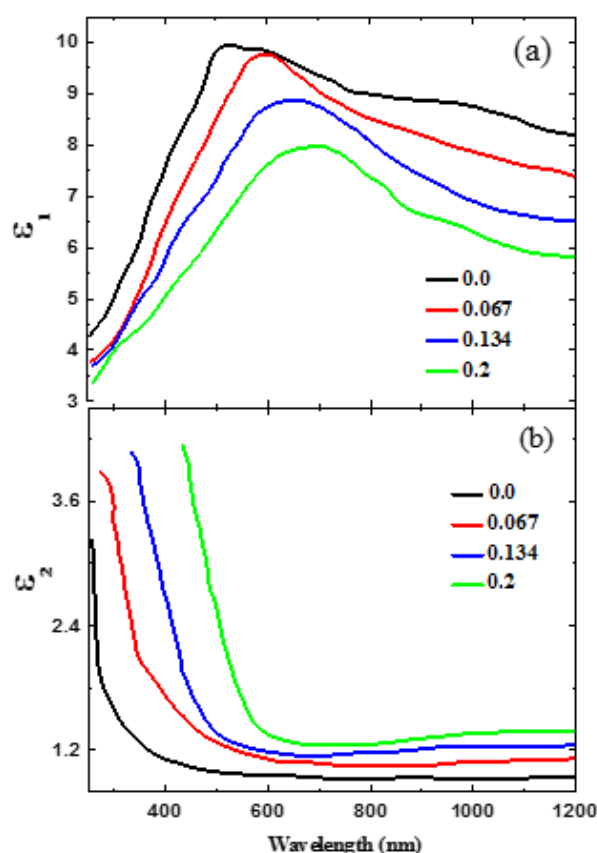


**Figure 6.** Real (a) and imaginary (b) contributions of the dielectric function,  $\epsilon_1(\lambda)$  and  $\epsilon_2(\lambda)$  versus wavelength ( $\lambda$ ) of  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.2$ ).

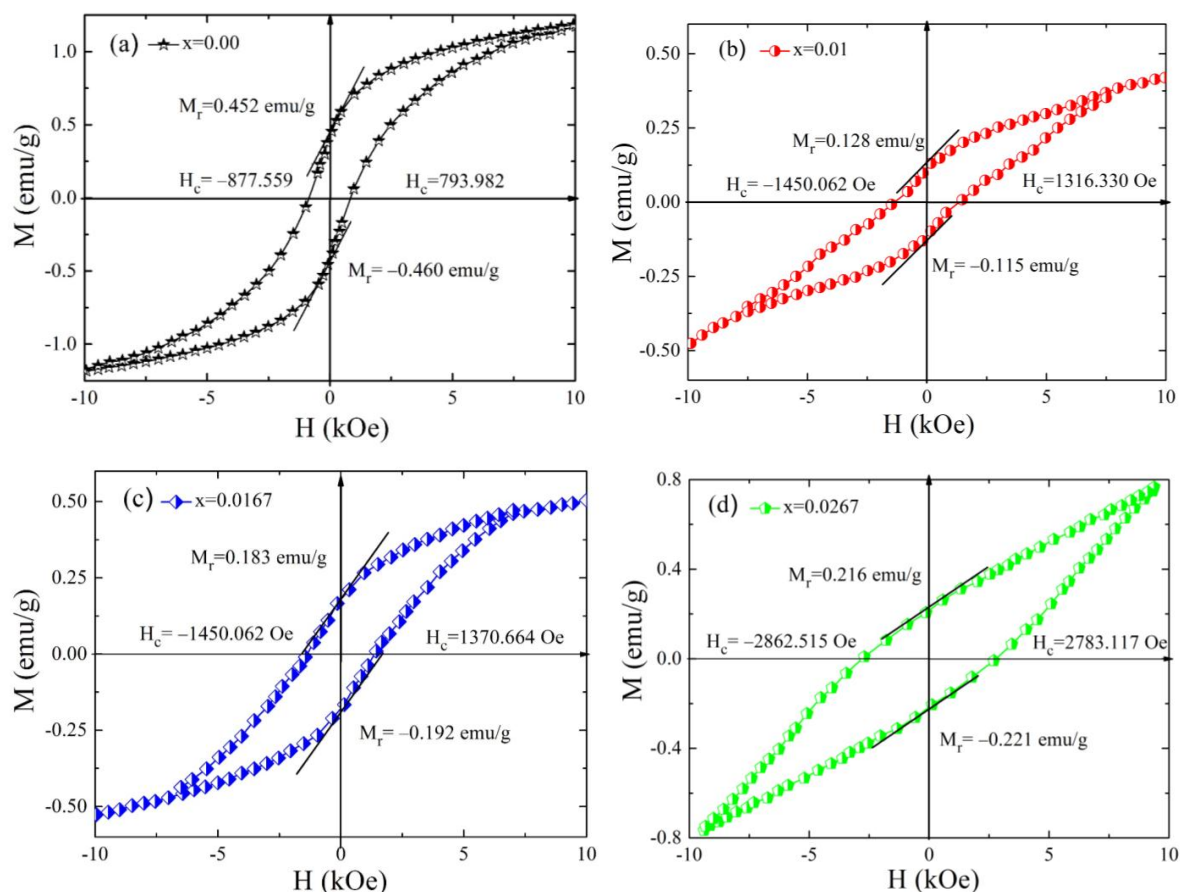


**Figure 7.** Hysteresis loops of Ru-doped  $\alpha\text{-Fe}_{2-4x}\text{Ru}_{3x}\text{O}_3$  with  $x =$  (a) 0.00, (b) 0.01, (c) 0.0167, and (d) 0.0267 measured at room temperature (300 K).

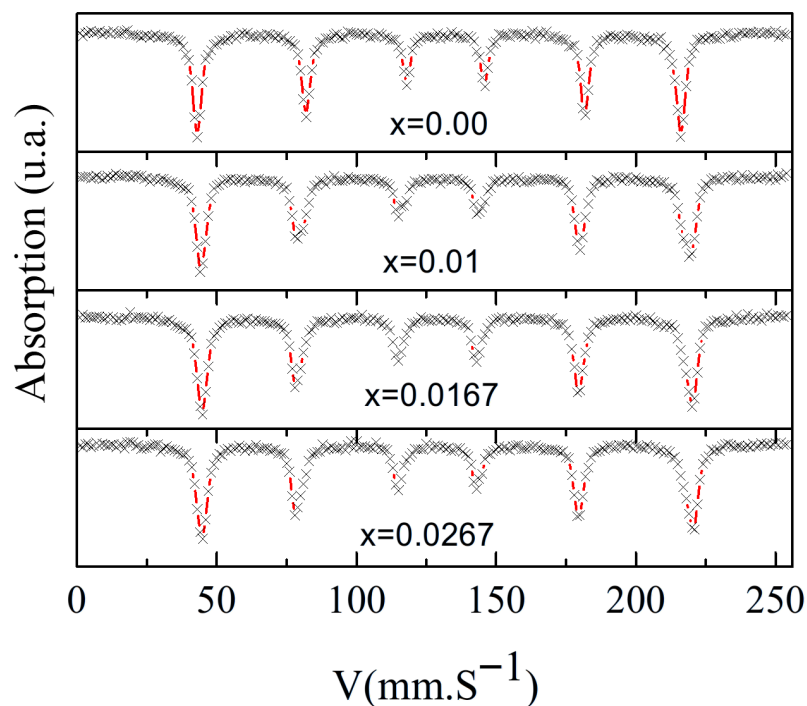


**Figure 8.** Room-temperature Mössbauer spectra of  $\alpha\text{-Fe}_{2-4x}\text{Ru}_{3x}\text{O}_3$  as a function of ruthenium doping ( $0 \leq x \leq 0.0267$ ).

**Table 2.** Remanent magnetization ( $M_r$ ) and coercive field ( $H_c$ ) measured for  $\alpha\text{-Fe}_{2-4x}\text{Ru}_{3x}\text{O}_3$  nanoparticles ( $0 \leq x \leq 0.0267$ ).

| Sample       | $M_r$ (emu/g) | $H_c$ (Oe) |
|--------------|---------------|------------|
| $x = 0.00$   | 0.452         | 793.98     |
| $x = 0.01$   | 0.183         | 1316.33    |
| $x = 0.0167$ | 0.128         | 1370.66    |
| $x = 0.0267$ | 0.216         | 2783.12    |

Figure 8 shows the room-temperature Mössbauer spectra of  $\alpha\text{-Fe}_{2-4x}\text{Ru}_{3x}\text{O}_3$  nanoparticles with varying Ru concentrations. All spectra display a magnetic sextet with narrow, nearly symmetric lines, indicating the presence of a single crystalline phase. Accordingly, the spectra were refined using a single magnetic sextet, with all hyperfine parameters allowed to vary freely. As summarized in Table 3, the extracted Mössbauer parameters are characteristic of the hematite phase and are in good agreement with previously reported values [42,43].

**Table 3.** Refinement parameters of the Mössbauer spectra of hematite nanoparticles at different ruthenium doping levels.

| Sample       | $\delta$ (mm/S) | $\Gamma/2$ (mm/S) | $2\epsilon$ (mm/S) | $\langle H_{\text{hyp}} \rangle$ (T) $\pm 2$ |
|--------------|-----------------|-------------------|--------------------|----------------------------------------------|
| $x = 0.00$   | 0.36            | 0.15              | −0.21              | 51.7                                         |
| $x = 0.01$   | 0.36            | 0.15              | −0.20              | 51.6                                         |
| $x = 0.0167$ | 0.36            | 0.14              | −0.20              | 51.4                                         |
| $x = 0.0267$ | 0.36            | 0.13              | −0.20              | 51                                           |

It should be noted that the hyperfine magnetic field remains practically constant within experimental accuracy ( $\pm 2$  T) for all Ru-doped samples, although a slight broadening of the Mössbauer lines is observed with increasing Ru content. This behavior can be attributed to a reduction in the crystallinity of the hematite grains. This interpretation is consistent with X-Ray diffraction results, which indicate that the average crystallite size decreases from 141 nm to 84 nm as the Ru content increases from  $x = 0.00$  to  $x = 0.0267$ . These observations suggest that the hematite phase is already stabilized at  $x = 0.0167$ , and that further Ru incorporation leads to a reduction in both particle size and crystallinity of the hematite nanoparticles.

#### 4. Conclusions

In summary,  $\alpha\text{-Fe}_{2-4x}\text{Ce}_{3x}\text{O}_3$  ( $0 \leq x \leq 0.2$ ) nanoparticles were successfully synthesized via a solid-state route. X-Ray diffraction analysis confirmed the formation of the hexagonal  $\alpha\text{-Fe}_2\text{O}_3$  (hematite) phase, with the crystallite size decreasing from 141 nm to 95 nm as the Ce content increased. UV–Visible spectroscopy revealed a decrease in the optical band gap from 2.22 eV for undoped  $\alpha\text{-Fe}_2\text{O}_3$  to 1.73 eV for  $x = 0.2$ , indicating that Ce doping enhances the optical response, making these materials promising candidates for photovoltaic applications. Magnetic hysteresis measurements showed systematic increases in remanent magnetization and coercivity with Ru doping, which were attributed to the influence of oxygen vacancies and associated defects. Mössbauer spectroscopy confirmed the presence of  $\text{Fe}^{3+}$  and  $\text{Ru}^{4+}$  ions, providing insights into the oxidation states and local magnetic environments. The Mössbauer results confirm the presence of  $\text{Fe}^{3+}$  ions in a single hematite phase. Subtle changes in hyperfine parameters, such as hyperfine field and quadrupole splitting, correlate with the observed variations in coercivity and remanence.

This emphasizes the connection between the local magnetic environments and the weak ferromagnetic behavior in Ru-doped  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles. Overall, Ce incorporation into  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> effectively tailors the structural and optical properties, while Ru incorporation modifies the magnetic properties, highlighting the potential of these dopants for multifunctional applications.

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