

Effect of Dysprosium Doping on the Structural, Morphological and Optical Properties of ZnO Nanostructures

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ABSTRACT

Zinc oxide (ZnO) is a promising optoelectronic material, yet its performance is often constrained by high charge recombination rates and a fixed band gap. To address these limitations, dysprosium (Dy)-doped ZnO nanocrystals (0–18 mol%) were synthesized via a co-precipitation method to evaluate the structural and optical effects of rare-earth doping. X-ray diffraction (XRD) confirmed a primary hexagonal wurtzite structure across all samples, with crystallite sizes ranging from 18 to 40 nm according to Scherrer formula calculations. Doped samples exhibited an additional diffraction peak at $2\theta = 29^\circ$ due to the formation of a monoclinic phase. Scanning electron microscopy (SEM) revealed a transition from the smooth spherical morphology of undoped ZnO to larger, varied particle structures at higher doping levels. Photoluminescence (PL) analysis showed key emission bands at 419 nm, 457 nm and 483 nm with the 6 mol% Dy-doped sample achieving the highest emission intensity. Furthermore, UV-Vis spectroscopy demonstrated an optical band gap tunable between 3.14 and 2.89 eV. These findings demonstrate that 6 mol% Dy doping effectively enhances luminescent intensity and offers band gap control for ZnO-based optoelectronic devices.

Keywords: Co-precipitation method; dysprosium doping; optical band gap; photoluminescence; zinc oxide

INTRODUCTION

For the past several years, considerable research interest has been directed toward ZnO due to its numerous advantageous properties, including high photosensitivity, a highly tunable specific surface area, low toxicity, good optical transparency in the visible region and excellent biocompatibility [1]. Furthermore, high electron mobility, strong thermal

conductivity, a wide direct band gap and high exciton binding energy give ZnO a distinct competitive edge, making it an ideal candidate for emerging optoelectronic applications [2]. Despite its numerous advantages, the optoelectronic performance of pristine ZnO remains constrained by several intrinsic limitations. For instance, the wide and relatively fixed band gap of ZnO primarily restricts its absorption to the UV region, limiting its interaction with a broader portion of the solar spectrum and reducing the flexibility of its optical response. Furthermore, defects and localized electronic states in ZnO can strongly influence charge-carrier dynamics and may provide pathways for electron–hole recombination. Such recombination processes can dissipate photo-generated energy through non-radiative or competing radiative pathways, reducing the efficiency of optical and optoelectronic processes. Consequently, modification of the ZnO electronic structure and band gap engineering through controlled doping have emerged as important approaches for overcoming these limitations [3]. Recent studies have explored rare-earth doping as an effective approach to modulate the optoelectronic properties of ZnO, enabling band gap engineering, enhanced carrier mobility and improved device performance [4]. The trivalent state of electrons in rare-earth ions causes the ions to have unique optical, chemical and electrical characteristics, which is why oxide nanocrystalline materials doped with rare-earth ions receive significant interest [5]. However, dopant concentration needs to be precisely controlled to achieve a desirable balance between structural integrity and optical functionality.

Rare-earth-doped ZnO nanostructures have attracted attention because of their significant fluorescence properties and high emission quantum yields. Luminescent materials development has expanded on rare-earth ions or rare-earth-based host lattices. For instance, the use of rare-earth phosphors greatly enhanced instrument performance [6]. Dy^{3+} is one of the most abundant rare-earth elements. In solid-state lighting applications, Dy^{3+} -doped phosphors only need a single dopant and a single host to provide white-light emission [7]. Dy^{3+} was chosen as the rare-earth ion to be doped into ZnO because dysprosium can effectively enhance luminescence intensity by acting as a sensitizer [8]. On the other hand, studies on Dy-doped ZnO nanocrystals are still limited. Researchers still face several difficulties when attempting to improve the structural and luminescent characteristics of rare earths. Therefore, further improvement of procedures is required to gain full knowledge of doping processes.

In this work, Dy-doped ZnO with Dy concentrations ranging from 0 to 18 mol% was synthesized using a chemical co-precipitation method. The structural, morphological and optical characteristics of the resulting materials were systematically investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM), UV–Visible spectroscopy and photoluminescence (PL) spectroscopy. Particular emphasis was placed on establishing the relationship between Dy concentration and the evolution of the ZnO crystal structure, surface morphology, optical band gap and luminescent behavior.

MATERIALS AND METHODS

The materials used in this study were 0.05 M zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) as an initial reagent, Dy_2O_3 (dysprosium(III) oxide) as the dopant source and 0.1 M NaOH as the precipitating agent. First, the starting material 0.05 M zinc nitrate hexahydrate was weighed according to stoichiometric ratios and dissolved in 100 mL of DI water. Next,

the solution was stirred at room temperature on a hot plate and 0.1 M NaOH was added dropwise into the solution until precipitation occurred. The mixture was stirred until it turned sticky or formed a gel. The gel was kept at room temperature overnight. After that, the precipitate was collected and dried in a hot-air oven at 90 °C for 3 h. Next, the sample was weighed, placed in a crucible and calcined in a furnace at 700 °C for 2 h. After calcination, the sample was weighed again and mass changes were noted. The steps were repeated using different volumes of NaOH and changes in mass were observed. For doped samples, the starting materials ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and Dy_2O_3) were weighed stoichiometrically and underwent the same procedure. The Dy content was varied between 2 and 18 mol%. After completing sample preparation, characterization was carried out using X-ray diffraction (XRD), UV-Vis spectroscopy, scanning electron microscopy (SEM) and photoluminescence (PL) spectroscopy.

RESULTS AND DISCUSSION

The sample yield for the undoped sample synthesis at different NaOH volumes is depicted in Figure 1(a). Figure 1(b) shows the sample yield for different Dy concentrations. It shows that the yield of the sample increased at higher Dy concentrations. The influence of NaOH volume on yield exhibits a bimodal trend, with peak outputs at 20 mL (2.898 g) and 60 mL (2.97 g). Conversely, Dy concentration shows a consistent positive correlation with yield, surging significantly to 0.5722 g at 18 mol%. The observed increase in yield may arise from the formation of Dy_2O_3 as a secondary phase in Dy-doped ZnO at higher dopant concentrations. This interpretation is consistent with the additional diffraction feature observed in the Dy-doped samples in the present study [9]. For maximum efficiency, a synthesis configuration of 60 mL NaOH and a Dy concentration of 18 mol% is recommended. Therefore, 60 mL NaOH was used to synthesize further samples.

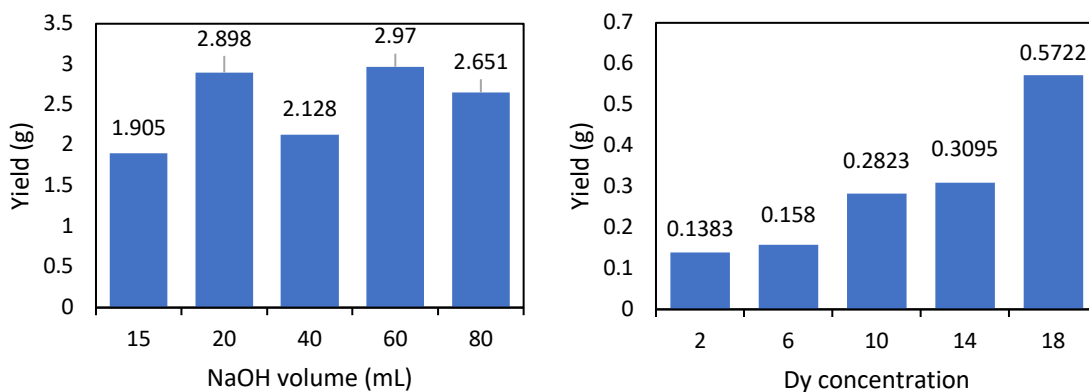


Figure 1. Yield of pure and Dy doped ZnO

The structural analysis was performed using an XRD instrument. From the XRD diffraction pattern, the crystal structure can be identified. The crystalline nature of the prepared samples was confirmed from the XRD analysis, as shown in Figures 2 and 3. Crystallinity is related to the degree of order and the crystal size of a given crystalline substance. Good crystallinity can also be demonstrated by calculating the Crystallinity Index (CI), which is a quantitative indicator of crystallinity. There are many different

methods to calculate the CI, one of which is by analyzing the XRD data. According to Islam et al. (2024), the crystallinity index is the ratio of the area of the crystalline peaks to the total area of crystalline and amorphous peaks [10]:

$$\text{Crystallinity index} = \frac{\text{Area of all the crystalline peaks}}{\text{Area of all the crystalline and amorphous}}.$$

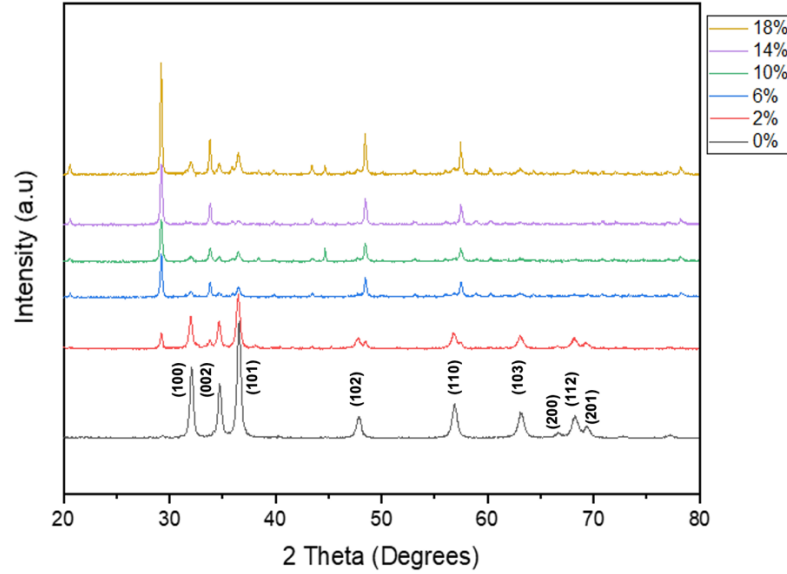


Figure 2. XRD patterns measured locally for different concentrations on Dy doped ZnO nanocrystals in the range of 20° to 80°

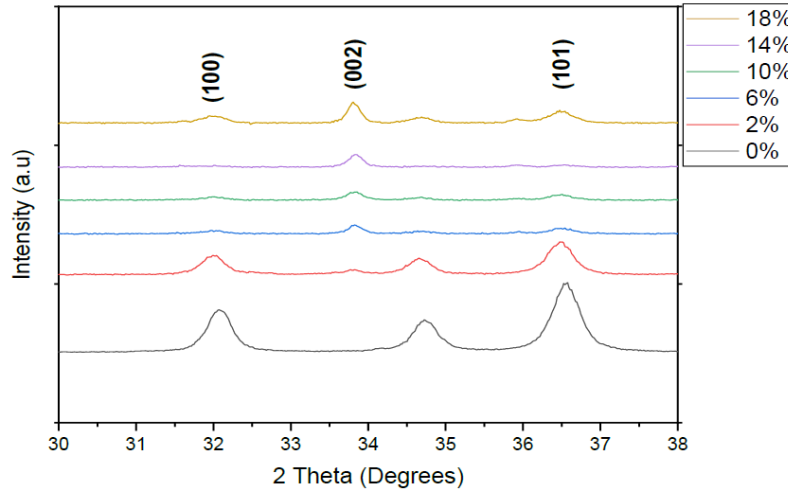


Figure 3. XRD patterns measured locally for different concentrations on Dy doped ZnO nanocrystals in the range of 30° to 38°

Based on Figure 2, the crystallinity and crystallite size for varied mol% of Dy are tabulated in Table 1. Table 1 shows that the crystallinity index of the 2 mol% Dy-doped sample is the highest among all samples, while the 18 mol% Dy-doped sample shows the lowest CI value. Furthermore, the results from the Crystallinity Index (CI) can be related

to the peaks in Figure 2, as the peaks of the 2 mol% Dy-doped sample are consistent with hexagonal wurtzite ZnO. It was reported that increasing the annealing temperature can improve crystallinity [11]. The diffraction peaks for the sample without Dy (pure ZnO) matched the standard Joint Committee on Powder Diffraction Standards (JCPDS) data card no. 36-1451. For comparison, the XRD pattern for undoped ZnO shows diffraction peaks corresponding to the (100), (002), (101), (102), (110), (103), (200), (112) and (201) planes of the hexagonal wurtzite structure. However, the XRD patterns of ZnO nanocrystals doped with 2, 6, 10, 14 and 18 mol% Dy³⁺ show differences compared to pure ZnO. After ZnO was doped with Dy, the diffraction pattern changed and the 18 mol% sample shows good agreement with results reported in a previous study [12]. According to that study, the XRD pattern for 20 mol% Dy-doped ZnO is similar to that of 18 mol% Dy. Several additional diffraction peaks from secondary phases were detected, indicating the presence of ZnO nanocrystals with amorphous components and additional Dy₂O₃ crystalline phases. When additional peaks are detected in the XRD spectrum, it indicates that Dy ions are not fully incorporated into the crystal lattice of ZnO. Additional phases can occur due to the large amount of dopant used during synthesis [13]. Figure 2 shows the formation of a monoclinic phase as the peak at 29° intensifies with increasing Dy³⁺ concentration. In addition, Figure 3 shows that the position of the (0 0 2) peak is slightly shifted to lower angles as Dy³⁺ ions substitute into the ZnO lattice or locate on the surface of the ZnO nanocrystals [14].

The average crystallite size was calculated via the peak broadening method. The full width at half maximum (FWHM) of the peak was measured. The average nanoparticle sizes were determined according to the Scherrer formula:

$$D = 0.89\lambda / \beta \cos\theta \quad (2)$$

where D is the crystallite size, λ is the wavelength (0.154 nm), β is the full width at half maximum and θ is the diffraction angle in radians. The terms β and 2θ were obtained from the XRD pattern. From Table 1, the average crystallite size decreased from 24.50 to 18.02 nm for samples with 0 to 14 mol% Dy doping. This phenomenon occurs due to the formation of Dy–O–Zn bonds on the sample surface [14]. This limits the growth of Dy-doped ZnO crystallites, making their crystallite size smaller than that of pure ZnO. On the other hand, the average crystallite size of the 18 mol% Dy-doped sample increased to 39.31 nm, which is larger than pure ZnO. A similar result for a 20 mol% Dy-doped sample was reported in another study [12]. According to Thangeeswari et al. (2015), crystallite size can increase at higher Dy concentrations [8].

Table 1. Crystallinity and crystallite size

Sample ZnO: Dy at mol%	Crystallinity Index, CI %	Crystallite size, D (nm)
0	78.51	24.50
2	85.31	23.11
6	59.12	19.95
10	41.27	19.31
14	53.15	18.02
18	38.29	39.31

SEM analysis was carried out to observe particle size and morphology. The morphological analysis was performed for 0, 2, 6, 10, 14 and 18 mol% Dy-doped ZnO, as presented in Figure 4. Imaging was carried out at different magnifications: 2000 $\times$, 5000 $\times$ and 10,000 $\times$. As depicted in Figures 4(a–c), the undoped ZnO surface is more spherical compared to doped ZnO, while the 2 mol% Dy-doped ZnO sample mainly consists of irregular plate-like particles ranging approximately from 1 μm to 3 μm .

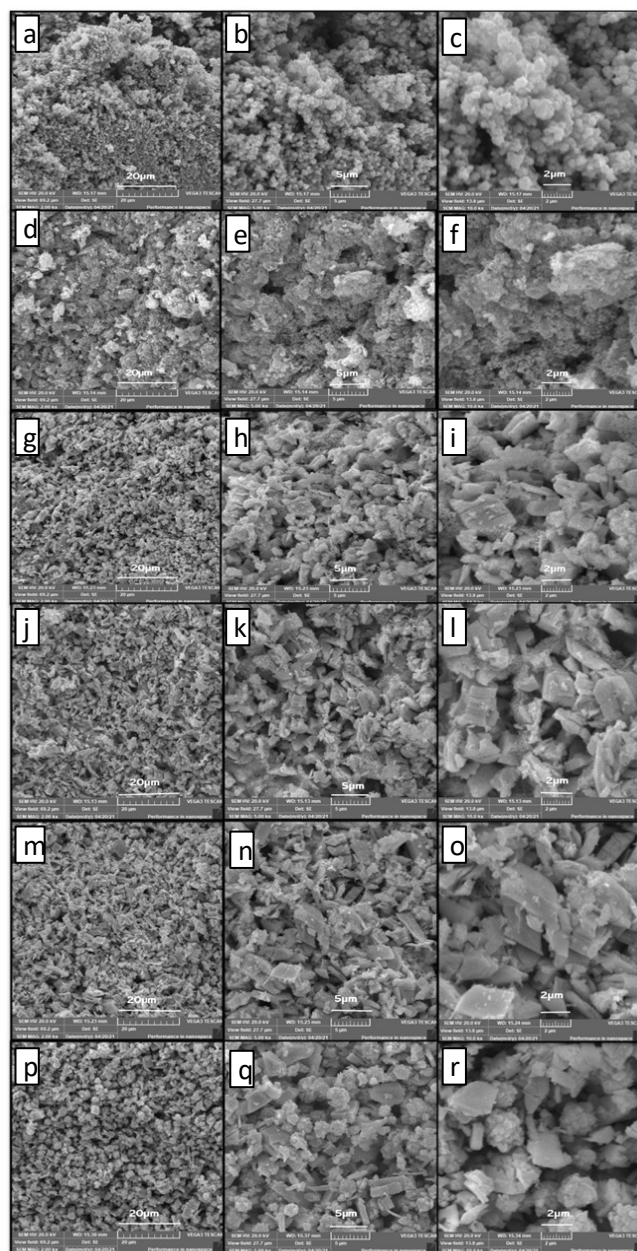


Figure 4. Scanning electron microscopy (SEM) images of Dy-doped ZnO nanocrystals with doping concentrations of (a–c) 0 mol%, (d–f) 2 mol%, (g–i) 6 mol%, (j–l) 10 mol%, (m–o) 14 mol% and (p–r) 18 mol%. Columns 1, 2 and 3 show magnifications of 2000 $\times$ (scale bar = 20 μm), 5000 $\times$ (scale bar = 5 μm) and 10,000 $\times$ (scale bar = 2 μm), respectively

The significant changes in particle size and morphology observed with increasing Dy concentration may be attributed to the higher dopant content introduced during synthesis, which may promote the formation of Dy–O–Dy bonds on the surface of the ZnO particles and consequently inhibit crystal grain growth [16]. Histograms of the particle size distribution for all samples are presented in Figure 5. The average particle sizes for 0, 2, 6, 10, 14 and 18 mol% Dy-doped ZnO were found to be 0.90 ± 0.18 , 1.52 ± 0.35 , 2.10 ± 0.37 , 2.70 ± 0.34 , 3.10 ± 0.40 and 3.18 ± 0.43 μm , respectively.



Figure 5. Particle size distribution in the form of histograms for undoped and Dy (2, 6, 10, 14 and 18 mol%)-doped ZnO nanocrystals

The weight percentages of Zn, Dy and O elements were confirmed by EDX, as shown in Table 2. The percentage of Dy increased as the molarity of Dy₂O₃ increased. The EDX spectra were recorded from various points on each sample to acquire accurate data. Figure 6 shows the elemental composition of undoped ZnO and Dy-doped ZnO obtained using EDX chemical mapping analysis. EDX analysis confirmed that Dy, representing the rare-earth element, was detected in all Dy-doped ZnO samples. The EDX analysis shows the presence of Dy, Zn and O elements with no impurity peaks detected, confirming the purity of the samples. In the mapping images, elements are represented by red, green and purple for Zn, O and Dy, respectively.

Table 2. Weight percentage of elements for pure and Dy-doped ZnO nanocrystals measured from energy-dispersive X-ray (EDX) analysis

Sample ZnO:Dy at mol%	Weight %		
	Zn	Dy	O
0	78.08±1.13	0	14.63±0.78
2	56.85±1.15	18.21±0.96	24.94±1.00
6	32.55±0.97	46.76±1.02	20.69±0.87
10	12.63±0.72	66.44±0.98	11.42±0.53
14	12.60±0.87	76.22±1.05	11.19±0.78
18	7.65±0.68	80.87±0.91	11.48±0.71

Optical absorption measurements were carried out by placing samples in 1 mm path-length cuvettes after dispersing them in distilled water. The analysis was performed in the wavelength range from 400 nm to 800 nm. Figure 7 shows the absorption spectra of pure ZnO and Dy-doped ZnO nanocrystal samples. The peak near 400 nm is expected to show broadening due to overlapping and interactions of excitonic oscillations with the excited states of Dy³⁺ ions [17]. However, the spectra differ slightly from previous studies due to the limitations of the UV-Vis spectrometer, which only measures wavelengths starting from 400 nm. The excitonic absorption features reflect the smaller particle size of the ZnO nanocrystals.

The Tauc method was used to obtain optical band gap energies from the UV-Vis absorption spectra. This method assumes that the absorption coefficient, α , as a function of photon energy can be described by the following equation [18]:

$$(\alpha h\nu) = B(h\nu - E_g)^n \quad (3)$$

where h denotes Planck's constant, ν represents photon frequency, E_g is the optical band gap energy and B is a constant related to the material. The exponent n is determined by the nature of the electronic transition, taking values of 1/2 for a direct allowed transition and 2 for an indirect allowed transition. By extrapolating the linear portion of $(\alpha h\nu)^2$ versus $h\nu$ as shown in Figure 8, the optical band gap energy of the ZnO samples can be determined [19]. The optical band gaps for 0, 2, 6, 10, 14 and 18 mol% Dy-doped ZnO are 3.08, 3.14, 3.04, 3.07, 3.05 and 2.98 eV, respectively. These values are slightly lower than the band gap of intrinsic bulk ZnO (~3.37 eV). Slight narrowing of the band gap

enhances optical absorption efficiency, making higher Dy-doped ZnO compositions advantageous for optoelectronic applications [20].

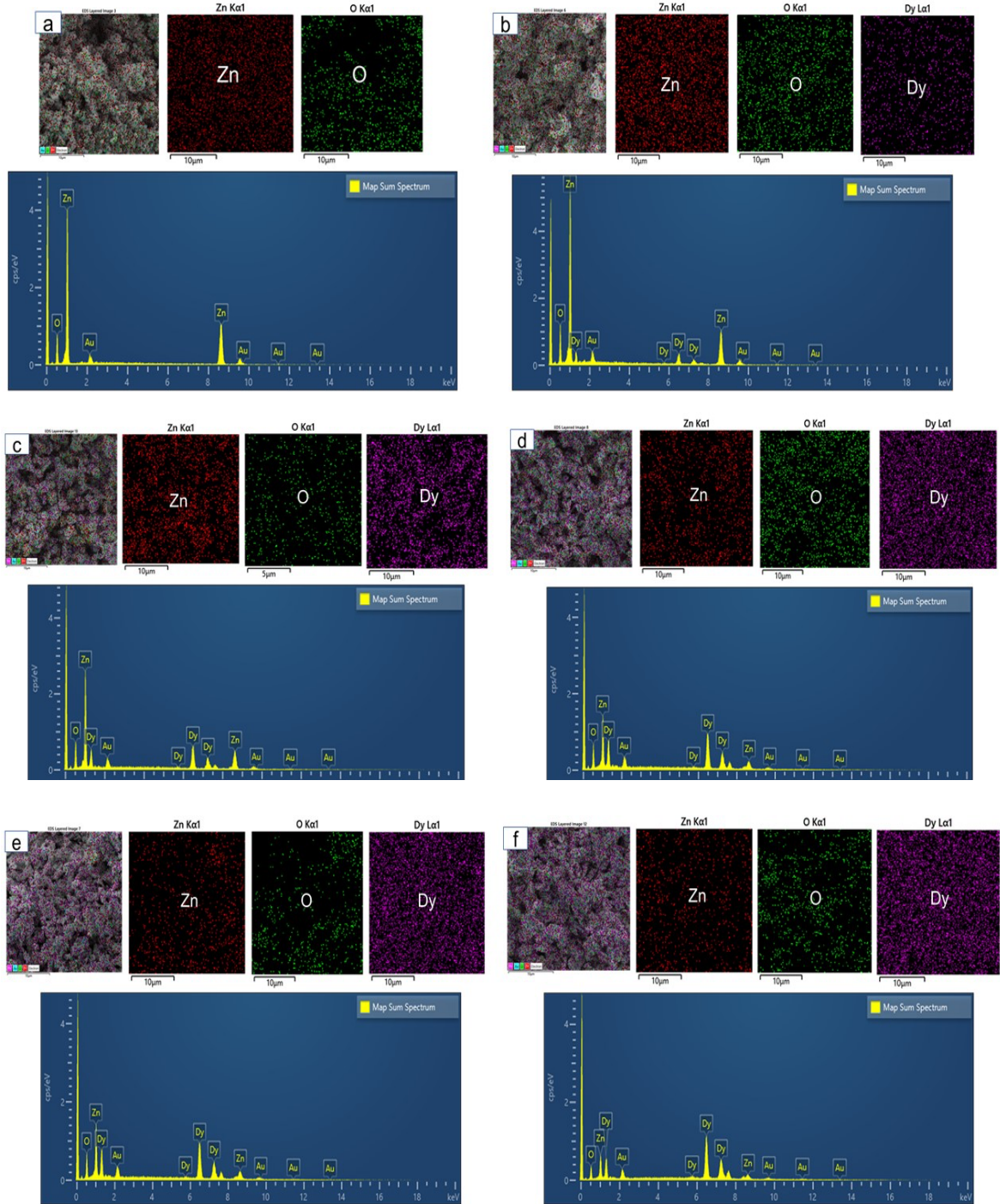


Figure 6. Energy-dispersive X-ray (EDX) mapping and spectra analysis of (a) undoped ZnO, (b) 2 mol%, (c) 6 mol%, (d) 10 mol%, (e) 14 mol% and (f) 18 mol% Dy-doped ZnO nanocrystals

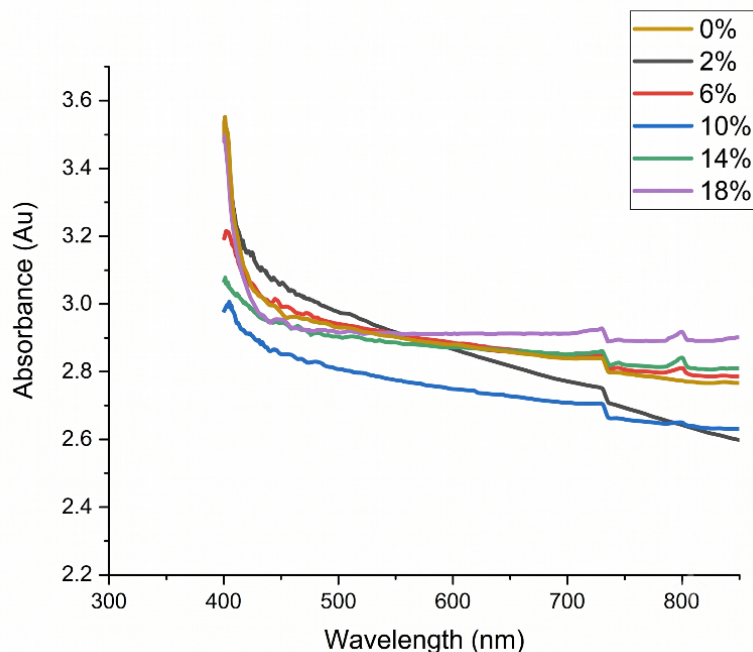


Figure 7. UV-Vis absorption spectra of undoped and Dy (2, 6, 10, 14 and 18 mol%)-doped ZnO nanocrystals

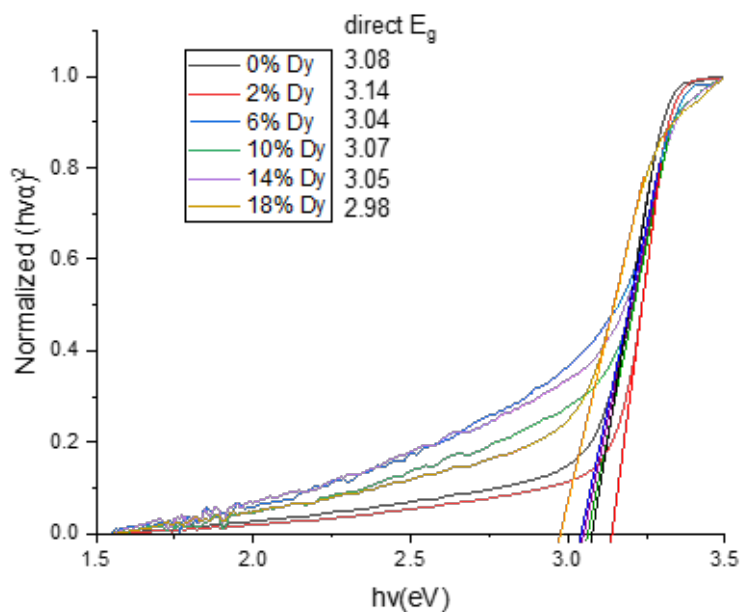


Figure 8. Tauc plots for the calculation of band gap for undoped and Dy (2, 6, 10, 14 and 18 mol%)-doped ZnO nanocrystals

Figure 9 shows the PL spectra of pure ZnO and Dy (2, 6, 10, 14 and 18 mol%)-doped ZnO nanocrystals under 370 nm excitation. The excitation wavelength was selected based on the excitation spectrum obtained from the samples. Under this excitation, all doped samples exhibit similar emission centers around 419 nm, whereas pure ZnO shows an emission centered at a longer wavelength. Emission centered around

419 nm is usually attributed to oxygen vacancies (V_O), surface dangling bonds and zinc interstitials. The spectra consist of emission peaks recorded at 419 nm ($^4I_{13/2} \rightarrow ^6H_{15/2}$), 457 nm ($^4I_{15/2} \rightarrow ^6H_{15/2}$) and 483 nm ($^4F_{9/2} \rightarrow ^6H_{15/2}$), which correspond to Dy^{3+} cation energy transitions, except for the absence of the yellow emission transition ($^4F_{9/2} \rightarrow ^6H_{13/2}$) typically observed near 573 nm. Dy^{3+} concentration does not alter peak positions significantly, as emission frequencies are mainly governed by the local site symmetry of the rare-earth ions [21]. According to Jayachandraiah et al. (2015), carrier trapping at defect states near the band edges can also cause emitted wavelengths to shift from expected values (e.g., 487 nm and 574 nm) [22].

The emission intensity of the 2 mol% Dy-doped sample increased significantly nearly threefold compared to pure ZnO while the 10 and 14 mol% samples exhibited similar intensities. A comparison of emission intensity among the different Dy doping levels is shown in Figure 9. The emission intensity of the 6 mol% Dy-doped sample was the highest, whereas undoped ZnO showed the lowest intensity. At higher concentrations (>6 mol%), emission intensity decreased due to concentration quenching [22]. Specifically, cross-relaxation between adjacent Dy^{3+} ions and energy transfer from the excited $^4F_{9/2}$ state to the $^6H_{15/2}$ ground state of neighboring Dy^{3+} ions increase at higher dopant levels, triggering concentration quenching [23]. Provided that the intensity of the peaks is consistently decreased due to the higher Dy^{3+} concentration will reduce the blue emission broadband, but the yellow emission will be intensified [24]. CIE chromaticity results are shown in Figure 10, illustrating the emission colors of all samples under 370 nm excitation. Evaluating phosphor performance relies on CIE 1931 chromaticity coordinates as key indicators. From the CIE diagram, all chromaticity coordinates fall within the blue emission region.

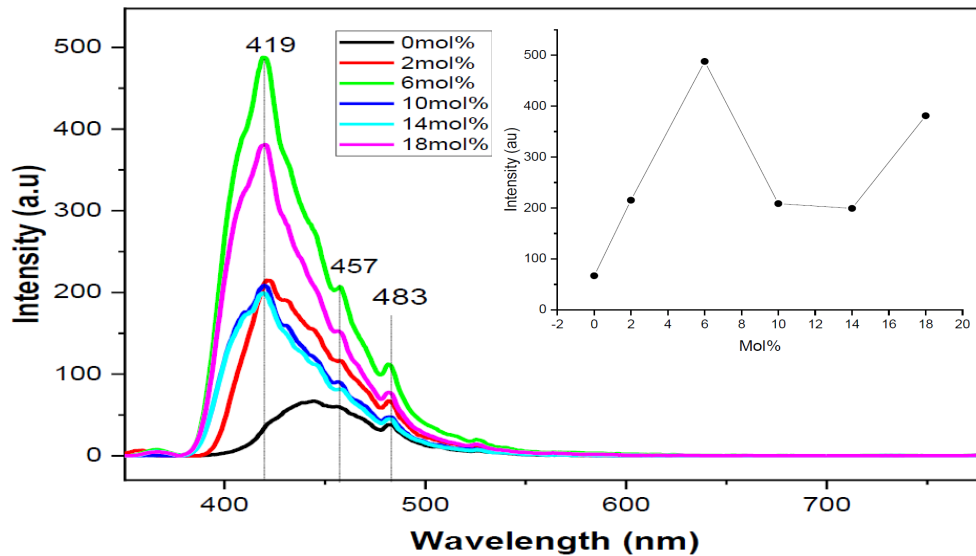


Figure 9. Photoluminescence emission spectra of pure and Dy (2, 6, 10, 14 and 18 mol%)-doped ZnO nanocrystals under 370 nm excitation (inset: emission intensity versus Dy mol%)

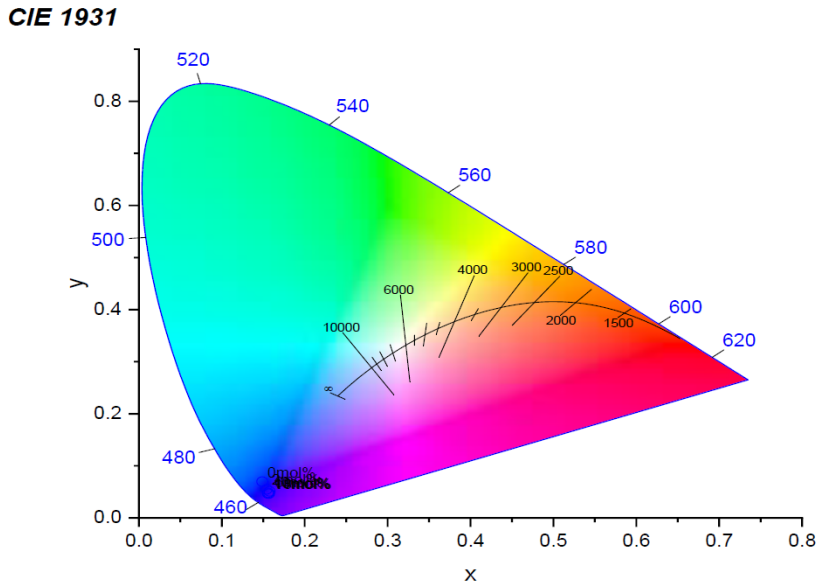


Figure 10. CIE 1931 chromaticity diagram showing chromatic coordinates of samples with different Dy^{3+} concentrations under 370 nm excitation

CONCLUSIONS

In this study, Dy-doped ZnO nanocrystals were successfully synthesized via a co-precipitation method using zinc nitrate hexahydrate as the precursor, NaOH as the precipitating agent and dysprosium(III) oxide as the dopant source. XRD analysis confirmed the crystal structure, crystallinity and average crystallite sizes. The 2 mol% Dy-doped sample exhibited the highest CI value (85.31%), whereas the 18 mol% Dy-doped sample showed the largest crystallite size (39.31 nm). SEM images revealed that undoped ZnO comprises spherical particles, the 2 mol% Dy-doped sample consists of plate-like structure and higher-doped samples exhibit varied, larger microstructures. Average particle sizes increased with higher Dy doping levels. EDX analysis confirmed an increase in Dy content corresponding to higher nominal Dy doping levels, with no elemental impurities detected. UV-Vis spectra showed optical band gaps ranging from 3.14 to 2.98 eV. Photoluminescence spectra displayed characteristic Dy^{3+} emission bands at 419 nm ($^4\text{I}_{13/2} \rightarrow ^6\text{H}_{15/2}$), 457 nm ($^4\text{I}_{15/2} \rightarrow ^6\text{H}_{15/2}$) and 483 nm ($^4\text{F}_{9/2} \rightarrow ^6\text{H}_{15/2}$), placing overall emissions in the blue region as indicated by the CIE chromaticity coordinates.

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AUTHORS' CONTRIBUTIONS STATEMENT

The manuscript was written through the contributions of all authors. MFO and NR designed and organized this work. SNMR, HJMR and SAS contributed to editing. NR

and WAWR drafted the main ideas of the work. WAWR provided supervision. AH conducted the final review of the manuscript.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

DATA AVAILABILITY

The datasets used and/or analyzed during the current study are available from the corresponding author upon reasonable request.

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