

Enhanced photoluminescence and structural properties of (Cr,Zn) co-doped Al₂O₃ phosphors synthesized via solution combustion

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ABSTRACT

Chromium-doped alumina (Cr-doped Al₂O₃) is a versatile material utilized in various applications such as sensors, phosphors, and bioimaging. It possesses attractive properties such as sharp photoluminescence (PL), long PL lifetime and excellent photostability. Enhancing its PL intensity is crucial for expanding its applicability. One approach to achieve this is by incorporating additional elements into the sample matrix. In this study, Cr-doped Al₂O₃ and (Cr,Zn)-co-doped Al₂O₃ samples were synthesized by solution combustion. X-ray diffraction (XRD) analysis showed that both samples displayed sharp, clear diffraction peaks consistent with the alpha-aluminum oxide (α -Al₂O₃) structure. Notably, the fundamental crystal structure did not change when Zn was introduced. The presence of Zn increased the crystallite size, suggesting that the zinc ions play a role in promoting the crystal growth and crystallization of Al₂O₃. Based on photoluminescence (PL) decay curves for (Cr,Zn)-co-doped Al₂O₃, the lifetime was estimated at around 3 ms. The energy band gap was calculated using UV-Vis spectroscopy data and was found to decrease from 4.31 eV to 4.23 eV after Zn addition, likely contributing to a 34% increase in PL intensity. This increment is attributed to the modification of the crystal field environment resulting from the addition of Zn²⁺ ions. A phosphor-converted light-emitting diode (pc-LED) was fabricated by depositing (Cr,Zn)-co-doped Al₂O₃ phosphor onto a 395 nm LED chip. The pc-LED with a 694 nm wavelength falls in the deep red spectrum, which is particularly beneficial for plant growth. The CCT of 2984 K suggests a warm light source, which could be useful for specific plant applications. However, the color purity of 24.6% indicates a mix of other wavelengths, which might affect efficiency depending on the targeted plant species.

Keywords: Photoluminescence, Chromium doped Alumina, Solution Combustion, Zinc co-doping, Phosphor

1. INTRODUCTION

The PL efficiency of certain materials can be improved by modifying their chemical compositions. Introducing dopants or co-dopants into the host material can modify the electronic structure, leading to improved radiative recombination rates and enhanced PL intensity [1-3]. Zn is one of the metal oxides that can give significant changes for the Cr-doped Al₂O₃. Based on a first-principles study on Bi₂WO₆, adding Zn can increase the electron density in the valence band maximum and narrow the energy band gap [4]. Thus, the Zn incorporation into Cr-doped Al₂O₃ host matrix could improve photoluminescence (PL) properties. Also, Zn ions have been incorporated in Al₂O₃:Er³⁺/Yb³⁺ [5]. Their study demonstrated that the near-infrared emission increased rapidly due to addition of Zn. As a result, the addition of Zn has increased the intensity of emission by approximately four times. Also, Zn incorporation in Yb³⁺/Er³⁺/Ho³⁺:Al₂O₃ has shown enhanced luminescence [6]. Anbarasu et al. investigated the effect of Zn doping to improve the visible light mediated photocatalytic activity of Al₂O₃ nanoparticles (NPs) [1]. The results show that Zn ions have a strong effect on the optical properties. The

substitution of Zn (4 wt%) ions has improved the photocatalytic performance of the doped Al₂O₃ NPs. Visible up-conversion and infrared luminescence investigations of Al₂O₃ powders doped with Zn ions have been reported by Singh et al. [7]. It shows, by adding the Zn ions in the Al₂O₃ host matrix able to enhance the emission band by 2.5 times. Another study of Zn ion addition has been done by researchers using different host. Study by Zhang et al., found that an increase in PL intensity around 46% for Cr³⁺ doped Li₂MgTi₃O₈ through Zn co-doping. The increase was most likely due to lattice distortion [8]. Li et al. reported the enhanced red photoluminescence in Yb³⁺/Er³⁺/Ho³⁺:Gd₂O₃ nanocrystals via 5% Zn co-doping. [6]. In addition, the effect of Zn concentration on the optical properties of LiNbO₃:Zn/Er crystals was reported by Qian et al. [9]. As the Zn ions are incorporated with LiNbO₃:Er crystals, it showed the enhancement of the green emission with respect to the red emission. Thus, based on promising previous findings, the utility of Zn as a co-dopant to improve the PL properties of Cr-doped Al₂O₃ is demonstrated for the first time in this work. While the influence of Zn has been documented in various alumina-based systems to enhance near-infrared and visible emissions, its specific synergy with chromium

within an Al₂O₃ matrix remains underdeveloped. Despite these promising indicators, the specific utility of Zn as a co-dopant to amplify the red-near-infrared PL properties of Cr-doped Al₂O₃ for targeted applications has not been fully realized. This work marks the first demonstration of utilizing Zn co-doping to specifically optimize the PL performance of Cr-doped Al₂O₃ synthesized via the solution combustion (SC) method. We leverage the SC technique not only for its cost-effectiveness and speed but also for its superior ability to yield nanosized particles with excellent stoichiometric control and uniform dopant distribution [10]. By systematically investigating the structural and optical evolution of the (Cr,Zn) co-doped Al₂O₃ system, this study aims to engineer a high-intensity light source specifically tailored for plant growth applications, filling a critical gap in the development of efficient, narrow-band red-emitting phosphors.

2. THEORITICAL BACKGROUND

The crystallite size, D for both samples is determined from the full-width at half maximum (FWHM) using the Scherrer equation:

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

where, k represents the Scherrer's constant (typically 0.9), λ is the wavelength of the X-rays, β denotes the peak width as the Full Width at Half Maximum (FWHM), and θ is the Bragg's angle.

To acquire better comparison, Williamson-Hall (W-H) plots were used to calculate crystallite size using the equation below [11]:

$$\beta \cos \theta = \varepsilon(4 \sin \theta) + \frac{k\lambda}{D} \quad (2)$$

To acquire more information about the deformities present in the synthesized samples, Dislocation density, δ was evaluated using the equation below:

$$\delta = \frac{1}{D^2} \quad (3)$$

The optical energy gap for prepared Cr-doped Al₂O₃ powder with and without Zn co-doping was determined using the Tauc's equation:

$$(\alpha h\nu)^{1/n} = B(h\nu - E_g) \quad (4)$$

α is the absorption coefficient, h is Planck's constant, ν is the frequency of vibration, B is a constant known as band tailing parameter [12] and E_g is the optical energy gap. The α value of powder glass sample can be determined by converting diffuse reflectance to the Kubelka-Munk (K-M) function which is expressed as follows [13]:

$$F(R_\infty) = \frac{(1 - R_\infty)^2}{2R_\infty} \quad (5)$$

where R_∞ is the diffuse reflectance. The absorption - proportional parameter $F(R)$ was obtained by converting the vertical axis. $F(R)$ is used in place of α in the Tauc equation. Consequently, the relational expression turns into:

$$(F(R)h\nu)^{1/n} = B(h\nu - E_g) \quad (6)$$

3. EXPERIMENTAL SECTION

The preparation of Cr-doped Al₂O₃ powder co-doped with Zn (1.0 wt.%) was carried out using the SC method using Chromium Nitrate (Cr(NO₃)₃·9H₂O) (R&M, 99.98% purity), Aluminium Nitrate (Al(NO₃)₃·9H₂O) (Bendosen, 99.98% purity), Urea (CO(NH₂)₂) (R&M, 99.98% purity) and Zinc Nitrate (Zn(NO₃)₂·6H₂O) (Bendosen, 99.98% purity). The starting materials were weighed in appropriate amounts according to the stoichiometric ratio.

The synthesis began by combining all the chemicals with 20 mL of distilled water and stirring for 15 minutes to ensure homogeneity. The resulting solution was then calcined in a crucible at 500 °C for 1 hour. The initial product, a white foamy powder, was allowed to cool, followed by 30 minutes of grinding to achieve a fine pink powder. The preparation concluded with a final thermal treatment of the powder at 1000 °C for 2 hours.

A Scanning Electron Microscope (SEM-TESCAN VEGA3 SBU) was used to examine the morphology of Cr-doped Al₂O₃ powder. The elemental composition was analyzed using an EDX device (the Oxford INCA X-Max 51-XXM 0021) alongside SEM. Optical reflectance spectra were recorded with a Cary 5000 UV-Vis spectrophotometer over the wavelength range of 200–750 nm at ambient temperature. A fluorescence spectrophotometer (JASCO-FP-8000 series) equipped with a 450 W xenon lamp was utilized to measure the PL excitation and emission spectra, as well as the decay curves, of the Al₂O₃:Cr³⁺ powder. The same parameters were used for each sample, with a weight of 0.5 g.

For the preparation of a phosphor-converted LED (pc-LED), 0.5 g of (Cr,Zn) co-doped Al₂O₃ fine powder was mixed with a transparent epoxy resin and its hardener in a 1:1 ratio. The mixture was stirred thoroughly to achieve a homogeneous coating material. The phosphor-epoxy mixture was then carefully poured into a circular mold, forming a layer approximately 0.5 cm thick. The coating thickness was adjusted to ensure full LED coverage and maximum light conversion efficiency. Afterward, a 395 nm LED chip was dipped into the mould filled with the phosphor-epoxy mixture. The assembly was left overnight to ensure proper hardening.

4. RESULT AND DISCUSSION

4.1. X-ray diffraction

Figure 1 shows the comparison of X-ray diffraction (XRD) patterns for the Cr-doped Al₂O₃ powder at optimum Cr³⁺ concentration (0.8 wt.% of Cr³⁺) with and without Zn co-doping. In this work, 1.0 wt.% of Zn was added in the

sample. Both samples present well-defined diffraction peaks corresponding to α - Al_2O_3 structure and remains unchanged with Zn addition. No additional diffraction peaks are detected, suggesting the absence of impurities in the synthesized Zn co-doped Cr- Al_2O_3 . The diffraction peaks are identified from the plane directions (012), (104), (110), (113), (024), (116), (018), (214), (300), and (119) (JCPDS PDF No. 42-1468). This reveals a high level of crystallization, as evidenced by the sharp and intense peaks that indicate the material's crystallinity. Based on the XRD pattern, it is revealed that the Zn co-dopant does not give clear effect on the α - Al_2O_3 phase. This shows, Zn ion has diffused well into Al_2O_3 host. Based on the Figure 1, it can be observed that at (104) peak position is slightly shift toward higher 2θ angles when doped with Zn. The smaller Al^{3+} ionic radius (0.053 nm) was replaced with a larger Zn^{2+} ionic radius (0.074 nm) that might result in the stretching of bond length as well as lattice parameter which results in the increase of unit cell volume [14]. These outcomes stipulate that the change in the lattice parameters of Cr-doped Al_2O_3 is because of the addition of Zn^{2+} ions in the lattice structure.

All parameter values derived from the XRD data are shown in Table 1. As seen in the table, the sample with the Zn co-doping shows high crystallinity at 82.18 % compared to sample without Zn addition. The crystallite size without Zn co-doping was estimated to be 40.97 nm and 49.62 nm for the sample with Zn co-doping. The increment in crystallite size after Zn co-doping shows there is influence in crystal growth and also in crystallization of Al_2O_3 . The size increment might be due to the substitution of Al^{3+} ions with relatively larger ionic radius of Zn^{2+} ions. Similar trend was reported for the case of Zn-doped α - Fe_2O_3 samples [15]. The crystallite size was estimated to be 84.71 nm and 82.20 nm for sample with and without Zn co-doping respectively. From the WH plot in Figure 2, it clearly indicates the samples display positive slope which shows the existence of positive strain or tensile strain. The increase in crystallite size and decrease in dislocation density indicates the dopant atoms are incorporated in the lattice [16]. Micro strain is found to increase after the addition of zinc. This increase is due to the lattice distortion cause by zinc addition [17].

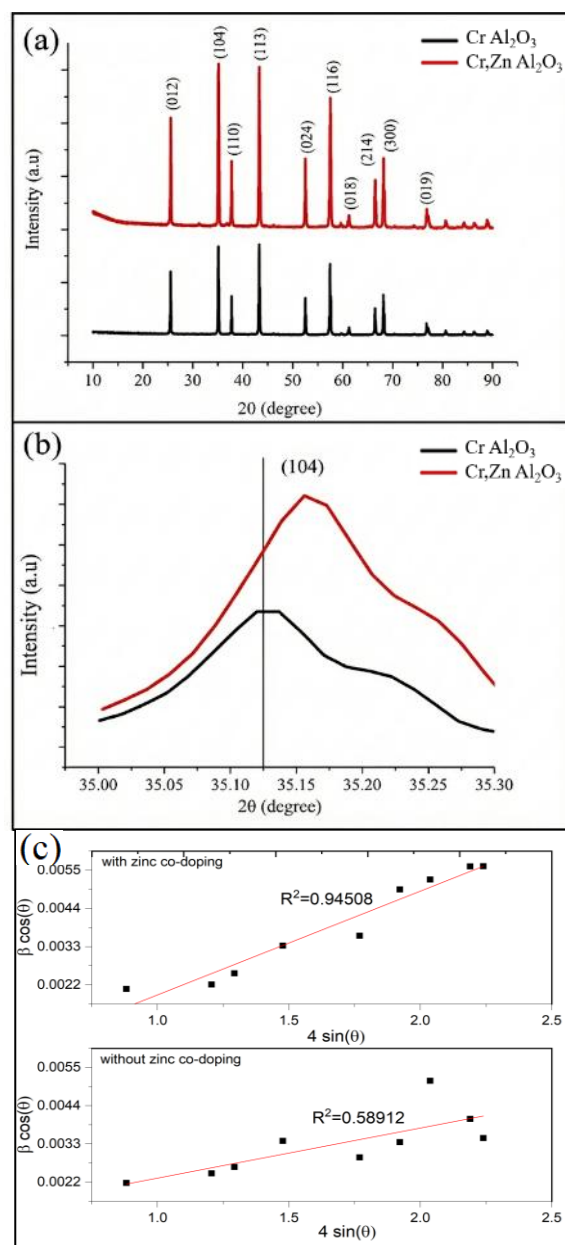


Figure 1. a) XRD pattern of Cr-doped Al_2O_3 (red) and (Cr,Zn)-co-doped Al_2O_3 (black) samples. b) The peak shift at (104) from XRD pattern. c) W-H plot for (Cr,Zn)-co-doped Al_2O_3 (top) and Cr-doped Al_2O_3 (bottom).

Table 1. The percentage of crystallinity, crystallite size, dislocation density and micro strain of Cr-doped Al_2O_3 with and (Cr,Zn) co-doped Al_2O_3 .

Samples	$[\text{Cr}^{3+}]$ (wt.%)	Crystallinity (%)	Crystallite size, D		Dislocation density, δ (nm^{-2}) ($\times 10^{-4}$)	Micro strain, ϵ ($\times 10^{-4}$)
			Scherrer formula	W-H plot		
1.0wt.% Zn	0.8	82.18	49.62	84.71	1.315	9.51 ± 2.87
Without Zn		69.75	40.97	82.20	3.341	8.42 ± 8.68

Further analysis on the crystal structure was performed using Rietveld refinement. The weighted profile R-factor (Rwp), the unweighted profile R-factor (Rp), and the chi-squared (χ^2) values are presented in Table 2. The lattice parameters of the Cr-doped Al₂O₃ materials almost same with the previous data. Figure 2 (a,b) shows that the observed and calculated profiles exhibit a strong agreement, indicating a close match between experimental data and theoretical predictions. Moreover, the lattice constants are accordance with the corresponding single crystal structure. The refined unit cell volume (V) shows small decrease after the addition of Zn. Although Zn²⁺ has a larger ionic radius than Al³⁺, the Rietveld refinement results

reveal a slight reduction in the unit-cell volume after Zn incorporation. This indicates that the structural response is not governed solely by ionic-size effects. The substitution of Zn²⁺ for Al³⁺ likely introduces local lattice distortions and defect-related charge compensation mechanisms, such as oxygen vacancies, which can induce structural relaxation and partial lattice contraction [18-20]. However, the addition of Zn in Cr-doped Al₂O₃ is at low amount which give small effect on Al₂O₃ lattice. For the c/a value shows an increment as the Zn addition that implies there is expansion in the crystallographic plane. The refined structure was produced with VESTA software, as illustrated in Figure 2(c).

Table 2. Crystallographic parameters of Cr-doped Al₂O₃ with and without zinc co-doping extracted from the Rietveld refinements of the XRD datasets and Cr-doped Al₂O₃.

[Zn ²⁺] (wt.%)	[Cr ³⁺] (wt.%)	Lattice parameters (Å)		2 θ (°)		Volume, (Å ³)	c/a	Rwp	Rp	χ^2
		a=b	c	$\alpha=\beta$	γ					
0.0	0.8	4.7607	12.9987	90	120	255.15	2.730	0.12	0.09	1.47
1.0		4.7604	12.9961	90	120	255.05	2.731	0.10	0.08	2.38

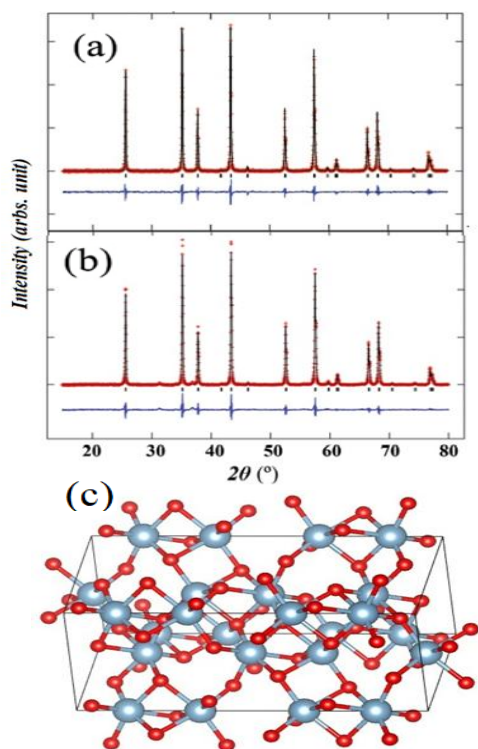


Figure 2. The Rietveld refinements of XRD patterns for Cr-doped Al₂O₃ (a) and (Cr,Zn)-co-doped Al₂O₃ (b) samples. (c) The corundum structure of Cr-doped Al₂O₃ with Zn co-doping produced by VESTA software.

4.2. SEM and EDX analysis

The surface morphology of the Cr-doped Al₂O₃ powder was examined by SEM. The SEM micrographs of Cr-doped Al₂O₃ (a) and (Cr,Zn)-co-doped Al₂O₃ are shown in Figure 3. Both samples consist of large chunks of crystals around 10 μ m, and small particle around 1 μ m. It is believed more smaller particle can be obtained by increase grinding time during sample preparation. The sample co-doped with Zn does show less smaller particles compared with Cr-doped Al₂O₃ sample. Figures 4(a,b) and 4(d,e) display the elemental

distribution mapping of the sample surface for (Cr,Zn)-co-doped Al₂O₃ and Cr-doped Al₂O₃ samples respectively. The colors denote as Al (red), Cr (blue), and Zn (purple). The mapping confirms that Zn ions are present in the system and demonstrates a homogeneous dispersion of Cr, Al, and Zn elements within the sample. Figures 4(c) and 4(d) show the corresponding typical EDX spectra. The spectra indicate the presence of Al, O, Cr and Zn in the samples. It is clearly seen that most intense peaks are aluminium and oxygen. Table 3 presents the EDX results of Cr-doped Al₂O₃ powder element composition. Chemical analysis of the (Cr,Zn)-co-doped Al₂O₃ sample showed a Zn content (1.01 wt.%) that closely to the 1.0 wt.% starting amount. The Cr content (0.87 wt.%) was also found to be incorporated, being only marginally higher than the initial 0.8 wt.%.

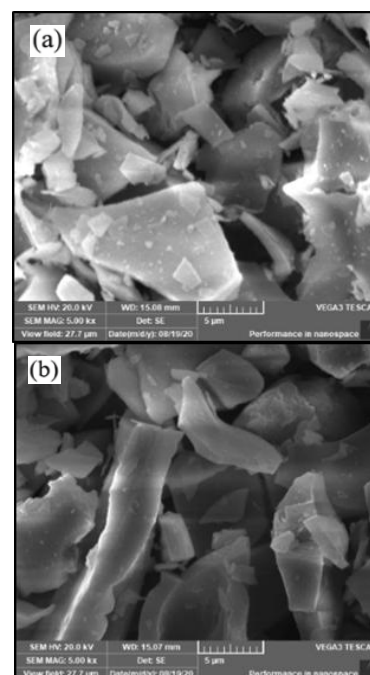


Figure 3. The SEM micrographs of Cr-doped Al₂O₃ (a) and (Cr,Zn)-co-doped Al₂O₃ (b) samples.

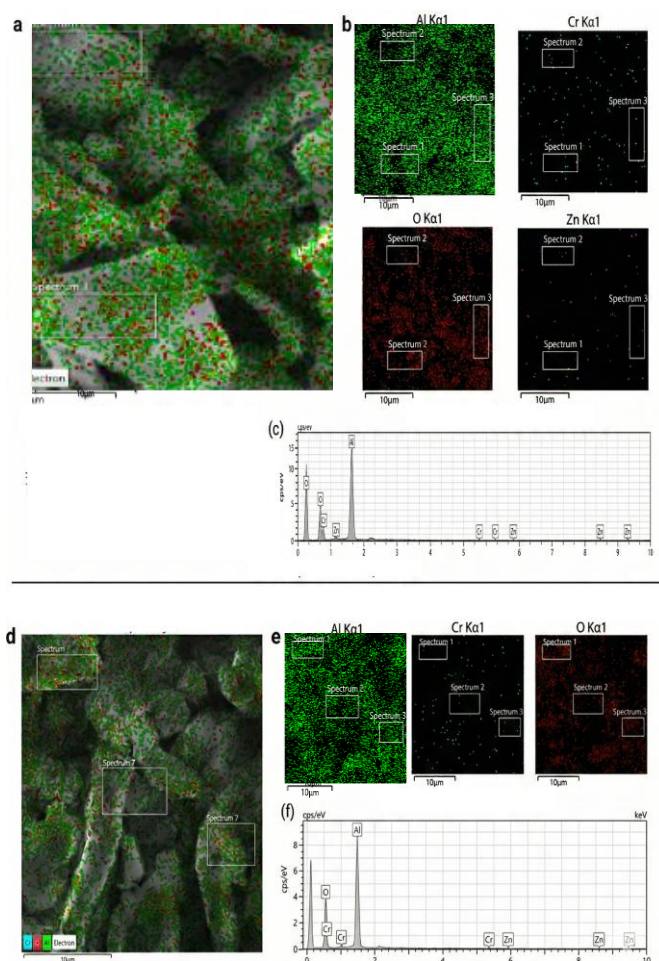


Figure 4. EDX mapping analysis-coloured squares represent the distribution of individual elements to the corresponding images of (Cr,Zn)-co-doped Al_2O_3 (top panel) and Cr-doped Al_2O_3 (bottom panel) samples. Aluminium (green), oxygen (red), chromium (blue) and Zinc (purple). Figure (c) and (f) show corresponding EDX spectrum.

4.3. Photoluminescence Analysis

Figure 5(a) shows the absorption spectra of Cr-doped Al_2O_3 (a) and (Cr,Zn)-co-doped Al_2O_3 samples. The optical energy gap, E_g was evaluated from the linear region of the curve of $(\alpha h\nu)^{1/n}$ interception on the y-axis versus photon energy ($h\nu$) plots on the x-axis. When $(\alpha h\nu)^{1/n}$ equal to zero, the E_g is equivalent to $h\nu$. Figure 5(b,c) display the Tauc plot for both samples. It is found that, the optical band gap for the sample with Zn co-doping is 4.23 eV while without Zn 4.31 eV. This shows that Zn content has lowered the energy band gap. This decline in band gap might be attributed to an increment in structural disorder or defects with Zn co-doping. Moreover, this decrease in band gap may also be due to partial hybridization between Zn t_{2g} and oxygen 2p states to empty aluminium t_{2g} 3d orbitals [21]. Similar trend was reported in previous work for the case of Zinc co-doped $\text{Mg}_{28}\text{Ge}_{10}\text{O}_{48}:\text{Mn}^{4+}$ phosphors. By improving material's energy structure can enhance photoluminescence signal [22].

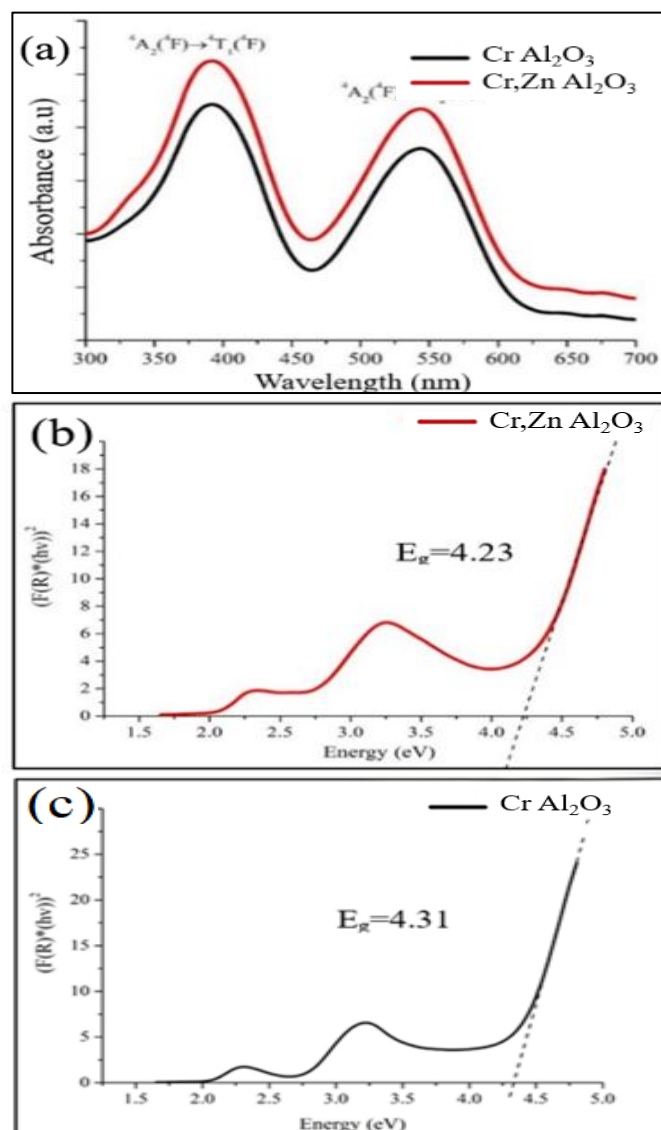


Figure 5. (a) The absorption spectra of Cr-doped Al_2O_3 (black) and (Cr,Zn)-co-doped Al_2O_3 (red) sample. (b,) Tauc plot for (Cr,Zn)-co-doped Al_2O_3 sample. (c) Tauc plot for Cr-doped Al_2O_3 sample.

The excitation and emission spectrum of of Cr-doped Al_2O_3 (a) and (Cr,Zn)-co-doped Al_2O_3 are shown in Figure 6 (a) and Figure 6 (b) respectively. Figure 6 (a,b) shows there two major absorption peaks at 405 nm and 557 nm. Based on Figure 6 (b), it is show that the PL intensity of Cr-doped Al_2O_3 powder is enhanced after Zn co-doping. The PL intensity is estimated to increase around 34%. The enhancement is likely due to the modification of the crystal field environment by Zn^{2+} ions which alters the emission properties [23]. Furthermore, lower energy band gap in sample with Zn also contribute to the PL enhancement by increasing the transition probability of emitting states [24]. This finding proved that the addition of Zn can enhance the PL intensity of Cr-doped Al_2O_3 powder.

The PL decay curves for both Cr-doped Al_2O_3 samples (with and without Zn co-doping) were recorded (shown in Figure 7). By analyzing these curves, the sample lifetime was calculated. It was determined that the PL decay accurately

adhered to the single-exponential decay function described in Equation (7):

$$I_t = I_o e^{-\left(\frac{t}{\tau}\right)} \quad (7)$$

where I_t is the intensity at time t , I_o is the initial intensity at $t=0$, and τ is the decay lifetime. The fitted emission lifetime values of the Cr-doped Al₂O₃ sample were found to be 3.26 ms and 3.27 ms for the samples with and without Zn co-doping, respectively. This lifetime value demonstrates good agreement with previously reported literature [25].

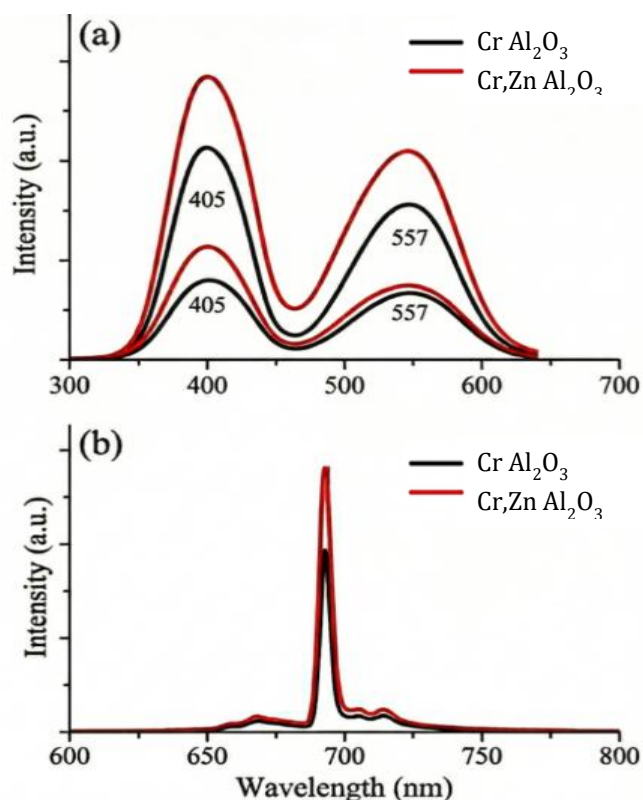


Figure 6. (a) PL Excitation (b) PL Emission spectra of Cr-doped Al₂O₃ (red) and (Cr,Zn)-co-doped Al₂O₃ (black line) samples.

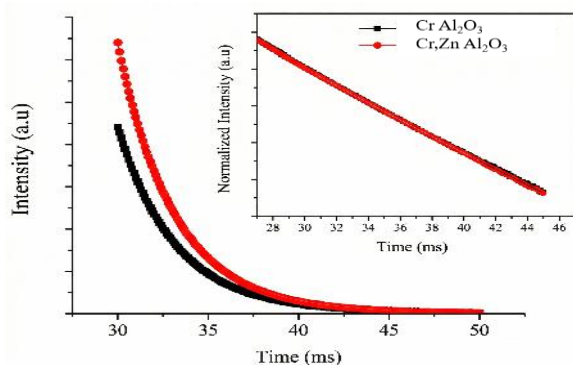


Figure 7. PL decay curves for Cr-doped Al₂O₃ (black) and (Cr,Zn)-co-doped Al₂O₃ (red), illustrating the effect of Zn co-doping on the emission lifetime. The inset shows the normalized PL intensity as a function of time

4.4. Chromatic properties and application

Figure 8(a) shows the CIE diagram of the (Cr,Zn) co-doped Al₂O₃ phosphor combined with the 395 nm LED chip. The chromaticity coordinate is (0.372, 0.31). Using this coordinate, the color purity (CP) and correlated color temperature (CCT) values were calculated. The CCT obtained via equations (8) [26].

$$CCT = -449n^3 + 3525n^2 - 6823.3n + 5520.33 \quad (8)$$

Where $n = (x - 0.3320)/(y - 0.1858)$. While CP value was obtained via equation (9) [25].

$$Colour Purity = \frac{\sqrt{(x-x_w)^2 + (y-y_w)^2}}{\sqrt{(x_s-x_w)^2 + (y_s-y_w)^2}} \times 100\% \quad (9)$$

where x and y are the measured colour chromaticity coordinate, x_w and y_w are reference white points based on illuminant C (0.3101, 0.3162) and x_s and y_s are the dominant wavelength point. Fig. 8(b) shows the fabricated pc-LED that generates bright pink light. The emitted light, with a wavelength of 694 nm, falls within the deep red spectrum and overlaps with the 500–700 nm absorption range of plant pigments [28, 29]. The calculated CCT of 2984 K, categorized as a warm light source [30]. The CP value of approximately 24.6% indicates the presence of other wavelengths, which may influence efficiency depending on the targeted plant species. Thus, this pc-LED has potential applications in plant growth.

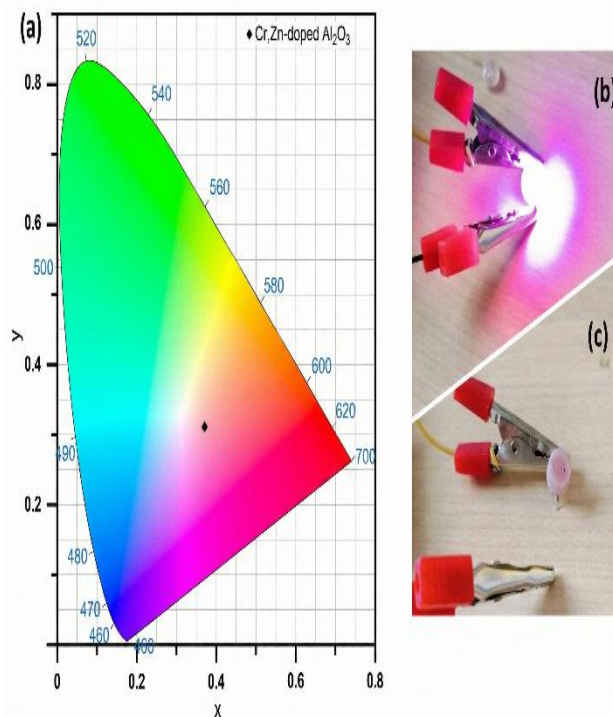


Figure 8. CIE chromaticity diagram of the (Cr,Zn) co-doped Al₂O₃ phosphor and the fabricated phosphor-converted LED, which consists of a 395 nm LED chip coated with (Cr,Zn) co-doped Al₂O₃ phosphor (b) connected and (c) unconnected to a 5 V power supply.

5. CONCLUSION

(Cr,Zn) co-doped Al₂O₃ was successfully synthesized using the solution combustion method. Rietveld refinement confirmed the phase of the sample and revealed increases in lattice parameter which is due to the replacement of aluminium with zinc. Zn addition also leads in energy band gap reduction from 4.31 eV to 4.23 eV. More importantly, the PL intensity was found to enhance by 34% after adding zinc element. Phosphor converted LED was demonstrated by depositing (Cr,Zn) co-doped Al₂O₃ on commercial 395 nm UV LED. The fabricated pc-LED produced light at 694 nm wavelength which can be potentially utilized for plant growth applications.

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