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Insights into structural, luminescence and temperature-dependent emission characteristics of $\text{Ca}_2\text{Al}_2\text{O}_5:\text{Dy}^{3+}$ phosphors for advanced lighting applications†

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This research synthesized and thoroughly examined novel $\text{Ca}_2\text{Al}_2\text{O}_5:\text{Dy}^{3+}$ phosphors to assess their potential for solid-state lighting and temperature-sensing applications. X-ray diffraction (XRD) verified the formation of a cubic phase with Dy^{3+} ions successfully integrated into the $\text{Ca}_2\text{Al}_2\text{O}_5$ host lattice. Photoluminescence (PL) analysis showed distinct blue (483 nm), yellow (575 nm), and weak red (663 nm) emissions, corresponding to the ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{15/2}$, ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{13/2}$, and ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{11/2}$ transitions of Dy^{3+} , respectively. The study identified that a 2 mol% concentration of Dy^{3+} is the ideal doping to achieve optimal luminescence, and the emission falls in the cool white light region. The optical study was used to ascertain the optical band gap, and the band gap of the host matrix decreases upon doping (from 5.01 eV to 4.83 eV) as new defect energy levels appear between the valence band and the conduction band. Temperature-dependent photoluminescence (TDPL) studies demonstrated excellent thermal stability, with the phosphors retaining significant luminescence intensity even at elevated temperatures. These phosphors exhibit appreciable thermal quenching behaviour and possess an activation energy of 0.20051 eV, underscoring their resilience at high temperatures. These results highlight the promising optical performance and thermal durability of $\text{Ca}_2\text{Al}_2\text{O}_5:\text{Dy}^{3+}$ phosphors, making them strong candidates for white LEDs and temperature-sensitive optoelectronic devices.

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1. Introduction

Phosphor materials are vital in advancing light-emitting technologies, acting as a key element in fields such as solid-state lighting, display systems, and radiation detection. When activated by different energy sources, including ultraviolet light, heat, or electrical fields, these substances exhibit luminescence, effectively converting energy into visible light. Their high quantum efficiency and customizable emission characteristics make them indispensable in devices like light-emitting diodes

(LEDs), fluorescent lamps, and electroluminescent displays. With the increasing demand for energy-efficient lighting solutions, developing high-performance phosphors with robust thermal stability and strong emission intensity has become a primary focus in materials research.^{1,2}

The properties of a phosphor, including its emission wavelength, intensity, afterglow, and thermal quenching behaviour, are influenced by the combination of its host lattice and the dopant ions. Numerous host-dopant pairings have been developed to achieve superior emission qualities. For instance, YAG:Ce³⁺ is frequently utilized in white LEDs due to its broad yellow emission, which blends with blue light to create white light.^{3,4} Other significant systems include (Sr,Ca):Eu for red emission in fluorescent lamps,⁵ Zn₂SiO₄:Mn for use in flat panel displays,⁶ and BaMg-Al₁₀O₁₇:Eu²⁺ for blue emission in LED displays.⁷ Long-lasting phosphors like SrAl₂O₄:Eu²⁺,Dy³⁺ are particularly valued for emergency lighting and glow-in-the-dark uses.^{8–10} Moreover, phosphors based on nitrides^{11–13} and oxynitrides^{14–16} are noted for their high quantum efficiency and thermal stability, making them excellent red emitters in LEDs. Similarly, hosts made of silicate,^{17–19} borate,^{20–22} aluminat^e,^{23,24} vanadate,^{25–27} and fluoride^{28–30} when doped with rare-earth ions offer a variety of optical properties

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suitable for lasers,^{31,32} displays,^{33–35} bioimaging,^{36,37} and up-conversion applications.^{38–40}

Among these, aluminite-based phosphors have gained considerable attention due to their excellent chemical stability, long afterglow, and suitability for high-temperature applications.^{41,42} There are reports on structural and luminescence properties of Sr₄Al₁₄O₂₅:Eu/Dy,⁴¹ electronic structure and high-pressure luminescence studies of Sr₄Al₁₄O₂₅:Mn,⁴² JO analysis of LaAlO₃:Tm,⁴³ luminescence of LaAlO₃:Eu,⁴⁴ computational and spectroscopic study of Eu/Nd doped MAI₂O₄ (M = Ca, Sr, Ba), radioluminescence of SrAl₂O₄:Eu,Sm,Dy,⁴⁵ emission studies of SrAl₂O₄:Er,⁴⁶ Eu/Dy,^{47,48} CaAl₂O₄:Eu/Nd,⁴⁹ Pr,⁵⁰ Sm,⁵¹ thermoluminescence characteristics of CaAl₂O₄:Dy,Sm,Tm,⁵² trap depth analysis of CaAl₂O₄:Tb,⁵³ LaMgAl₁₁O₁₉:Eu,⁵⁴ temperature dependent luminescence studies of BaMgAl₁₀O₁₇:Ce,Tb,⁵⁵ luminescence features of BaMgAl₁₀O₁₇:Eu,^{56–58} Cr,⁵⁹ BaMgAl₁₀O₁₇:Mn,⁶⁰ BaMgAl₁₀O₁₇:Eu,Yb,⁶¹ BaMgAl₁₀O₁₇:Dy,⁶² SrMgAl₁₀O₁₇:Eu,Dy,⁶³ SrMgAl₁₀O₁₇:Mn,Eu,⁶⁴ Ca₂Al₂O₅:Eu,^{65,66} and mechanoluminescence studies of SrMgAl₁₀O₁₇:Eu⁶⁷ phosphors.

After thoroughly reviewing the literature, we selected Ca₂Al₂O₅ as the host matrix and Dy³⁺ as the dopant. Ca₂Al₂O₅ phosphors are renowned for their outstanding thermal and chemical stability, even in high-temperature and challenging environmental conditions. This characteristic makes them ideal for applications involving high-power or high-temperature lighting. The Ca₂Al₂O₅ system can be produced through a cost-effective solid-state method using readily available raw materials (CaCO₃ and Al₂O₃). In contrast to well-known hosts like SrAl₂O₄ or YAG, Ca₂Al₂O₅ has not been extensively studied, especially in Dy³⁺ doping. To our knowledge, only one prior study has explored Dy³⁺ in this host, and it did not provide a comprehensive analysis of structural, luminescence, and temperature-dependent emission properties. Since the studies are limited to Ca₂Al₂O₅:Dy, the present work focuses on the structural, morphological, and luminescence properties along with in-depth analysis of crystallite size, correlation between dopant concentration and luminescence features. Furthermore, the temperature-dependent luminescence characteristics are explored with critical insights into activation energy and FWHM variation. Thermal stability and activation energy contribute to designing and optimizing tools for elevated temperature applications such as solid-state lighting and display applications.

2. Experimental details

2.1 Sample synthesis

The Ca₂Al₂O₅:Dy (CAO-Dy) phosphors were prepared by varying the amount of Dy³⁺ from 1 mol% to 5 mol%. The precursors CaCO₃, Al₂O₃, and Dy₂O₃ were taken in stoichiometric ratios and mixed well in a mortar for 45 minutes. To ensure homogenous mixing, we added ethanol while grinding the sample. The mixture was calcined at 1300 °C for 8 hours at a heating rate of 5 °C min⁻¹. The samples were collected after attaining room temperature for further characterization.

2.2 Characterization techniques

The structural studies are conducted using a Rigaku Miniflex 600 (5th generation) device, which employs K- α radiation

(λ = 1.54 Å), voltage maintained at 40 kV and current at 15 mA. A SHIMADZU-IRSpirt ATR-FTIR spectrometer is utilized to identify functional groups. The morphology of the samples is analyzed with SEM technique using a Sigma Zeiss instrument. Diffuse reflectance data is gathered with a PerkinElmer Lambda 900 spectrophotometer. The emission characteristics are recorded utilizing a JASCO-FP 8500. The variation in luminescence properties with temperature is obtained with an Agilent Cary Eclipse Fluorescence Spectrophotometer.

3. Results and discussion

3.1 X-ray diffraction (XRD) study

The synthesized samples' phase confirmation and crystal structure are obtained by XRD analysis. Fig. 1(a) shows the XRD peaks of CAO-Dy samples, which match the COD code – 1525613 pattern. There is no appreciable peak shift with changing dopant concentration, and the crystal structure remains unaltered upon doping. Fig. 1(b) shows the crystal structure of the CAO unit cell. The Rietveld refinement was performed to ascertain the lattice parameters, volume, and phase purity (Fig. 1(c)). This analysis indicated the formation of a cubic lattice with space group $I\bar{4}3d$, crystal parameters $a = b = c = 11.9953$ Å, and $\alpha = \beta = \gamma = 90^\circ$, and the volume is 1725.97 Å³. No impurity peaks were observed in the XRD pattern. The reliability factors obtained are R_p : 32% and R_{wp} : 33%, with structural coordinates and occupation detailed in Table 1. As depicted in Fig. 2(b), the refinement data shows an excellent fit with $\chi^2 = 2.74$.

To explore the dopant's occupation site, we must focus on the dopant ion's ionic radii and the cation being replaced in the host matrix.⁶⁸ For each cation and dopant pair for a different coordination number (CN), the acceptable percentage difference value (R) is calculated as per the following equation.

$$R = \frac{|R_h(\text{CN}) - R_d(\text{CN})|}{R_h(\text{CN})} \times 100\% \quad (1)$$

where R_h is the host cation radius, CN is the coordination number, and R_d is the radius of the dopant ion. If R is well below 30%, the substitution of dopant to the respective cationic site is confirmed. The calculation for the R -value is given in the Table 2. From Table 2, $R < 30\%$ for Ca²⁺-Dy³⁺ combination; hence we can confirm the occupation of dopant ion in Ca²⁺ sites.^{69,70} The crystallite size of CAO-Dy samples is calculated using the Debye-Scherrer equation and size-strain plot (SSP) approach. The crystallite size affects the diffraction pattern and the luminescence features of phosphors. The crystallite size varies with dopant concentration and is calculated using the following Debye-Scherrer equation^{71,72}

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

λ is the X-ray wavelength (1.54 Å), β is the full-width half maximum of the XRD peak, and k is a constant taken as 0.9. The crystallite size variation is also determined using the SSP method, where the higher angle reflections are given less weight as they have lower precisions. The Lorentzian function

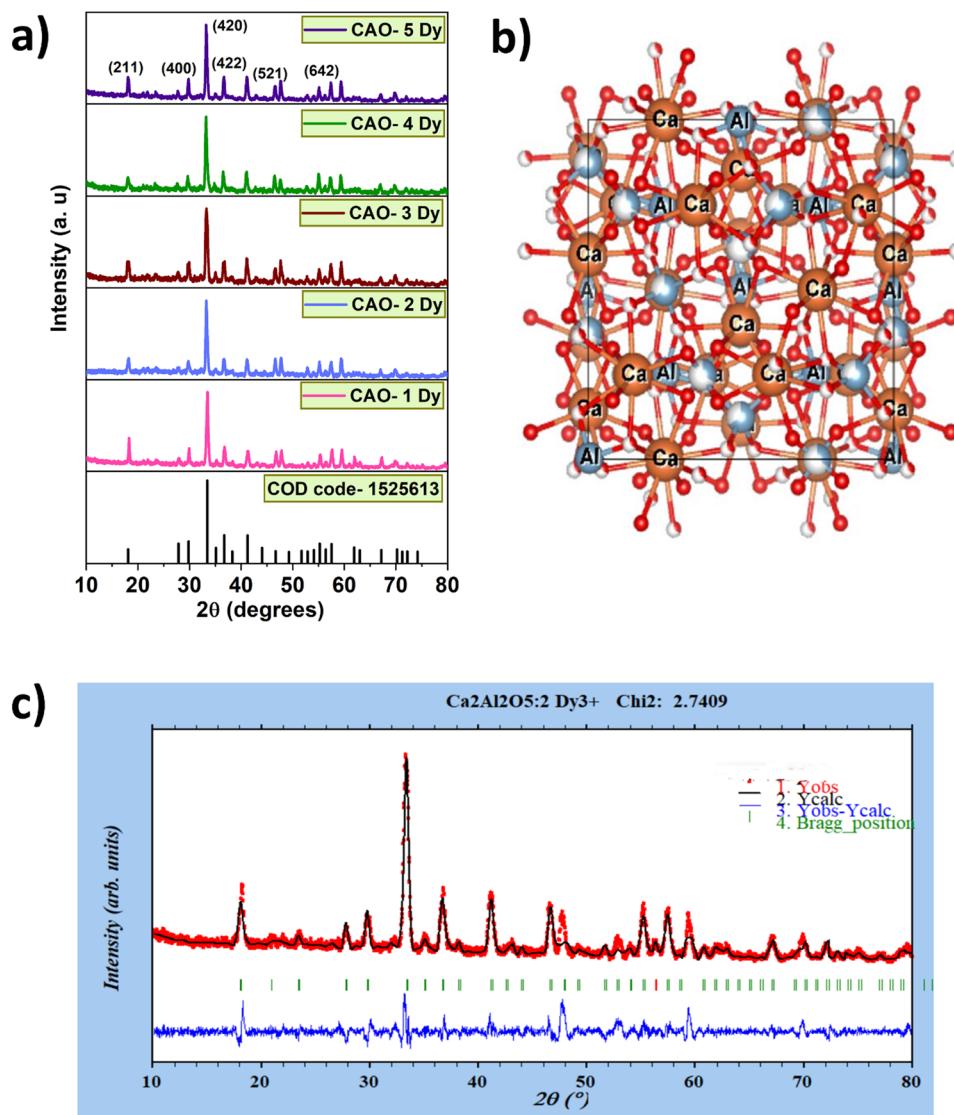


Fig. 1 (a) XRD pattern of CAO phosphor for varying Dy^{3+} concentration, and (b) CAO unit cell, (c) Rietveld refinement of CAO-2 Dy sample.

and Gaussian functions are used to illustrate the crystallite size profile and the strain profile, respectively, as per the following equation,^{73,74}

$$(d_{hkl}\beta_{hkl} \cos \theta)^2 = \frac{K}{D}(d_{hkl}^2\beta_{hkl} \cos \theta) + \left(\frac{\varepsilon}{2}\right)^2 \quad (3)$$

where K is 0.75, which depends on the shape of the particles, ε is the strain, d_{hkl} is the interplanar spacing. By taking the slope (K/D) of $(d_{hkl}\beta_{hkl} \cos \theta)^2$ vs. $(d_{hkl}^2\beta_{hkl} \cos \theta)$ graph, the crystallite size can be calculated (Fig. 2). Table 3 gives the variation in crystallite size of CAO-Dy phosphors calculated using Debye-Scherrer and SSP methods.

The average crystallite size of CAO-Dy samples was 18–23 nm using Scherrer's method and 66–75 nm range for the SSP method. This difference might be attributed to the inclusion of strain in the latter method.

3.2 Fourier transform infrared (FTIR) spectroscopy

The vibrational functional groups of phosphor samples are identified using FTIR spectroscopy. Fig. 3 shows the vibrational bands in the CAO host matrix and Dy^{3+} doped CAO. Region 1 (in Fig. 3) displays bands at 745 cm^{-1} and 806 cm^{-1} , corresponding to Ca-O vibrations.⁷⁵ The same bands appear in the Dy^{3+} doped sample as well. As the dopant occupies Ca^{2+} sites, there is no peak shift (shift is within error limit) or band appearance/disappearance upon Dy^{3+} doping, confirming the unaltered structure of the host matrix upon doping. The regions identified as 2 and 3 (Fig. 3) are assigned to Al-O vibrations, having bands at 514 and 571 cm^{-1} .⁷⁶

3.3 Scanning electron microscopy (SEM)

The morphological features of CAO-Dy samples are shown in the Fig. 4. Highly agglomerated microstructure accompanied by porous morphology is obtained for the prepared sample.⁷⁷ The



Table 1 Sample notation

| Chemical formula | Notation |
|---|----------|
| $\text{Ca}_2\text{Al}_2\text{O}_5$:1 mol% Dy | CAO-1 Dy |
| $\text{Ca}_2\text{Al}_2\text{O}_5$:2 mol% Dy | CAO-2 Dy |
| $\text{Ca}_2\text{Al}_2\text{O}_5$:3 mol% Dy | CAO-3 Dy |
| $\text{Ca}_2\text{Al}_2\text{O}_5$:4 mol% Dy | CAO-4 Dy |
| $\text{Ca}_2\text{Al}_2\text{O}_5$:5 mol% Dy | CAO-5 Dy |

shape of the particles is irregular, and accurate particle measurement is impossible.⁷⁸

3.4 Optical studies

Fig. 5(a) gives the Diffuse reflectance (DR) spectra of CAO-2 Dy phosphor, the peaks are centred at 490 nm, 792 nm, 880 nm, 1060 nm, 1252 nm, 1380 nm, and 1650 nm attributed to transitions $^6\text{H}_{15/2} \rightarrow ^4\text{F}_{9/2}$, $^6\text{H}_{15/2} \rightarrow ^6\text{F}_{5/2}$, $^6\text{H}_{15/2} \rightarrow ^6\text{F}_{7/2}$, $^6\text{H}_{15/2} \rightarrow ^6\text{H}_{7/2}$, $^6\text{H}_{15/2} \rightarrow ^6\text{F}_{11/2}$, $^6\text{H}_{15/2} \rightarrow ^6\text{H}_{9/2}$, and $^6\text{H}_{15/2} \rightarrow ^6\text{H}_{11/2}$ respectively. These transitions are further used to

identify the nature of bonding between the dopant ion and the host matrix ligand. The nephelauxetic ratio (β) and bonding parameter (δ) are given using the following equations⁷⁹

$$\beta = \frac{\nu_c}{\nu_a} \quad (4)$$

ν_c and ν_a are the energies of the Dy^{3+} transitions in the host matrix and the aqueous solutions, respectively.⁸⁰

$$\delta = \frac{1 - \beta_{\text{avg}}}{\beta_{\text{avg}}} \quad (5)$$

where β_{avg} is the average value of β for observed transitions, if $\delta < 0$, the bonding is ionic, and $\delta > 0$ corresponds to covalent bonding. Table 4 gives the values of β and δ for the Dy^{3+} transitions.

Since the δ value is 0.00623, the Dy^{3+} -ligand bond is covalent for the prepared phosphor samples. A similar covalent

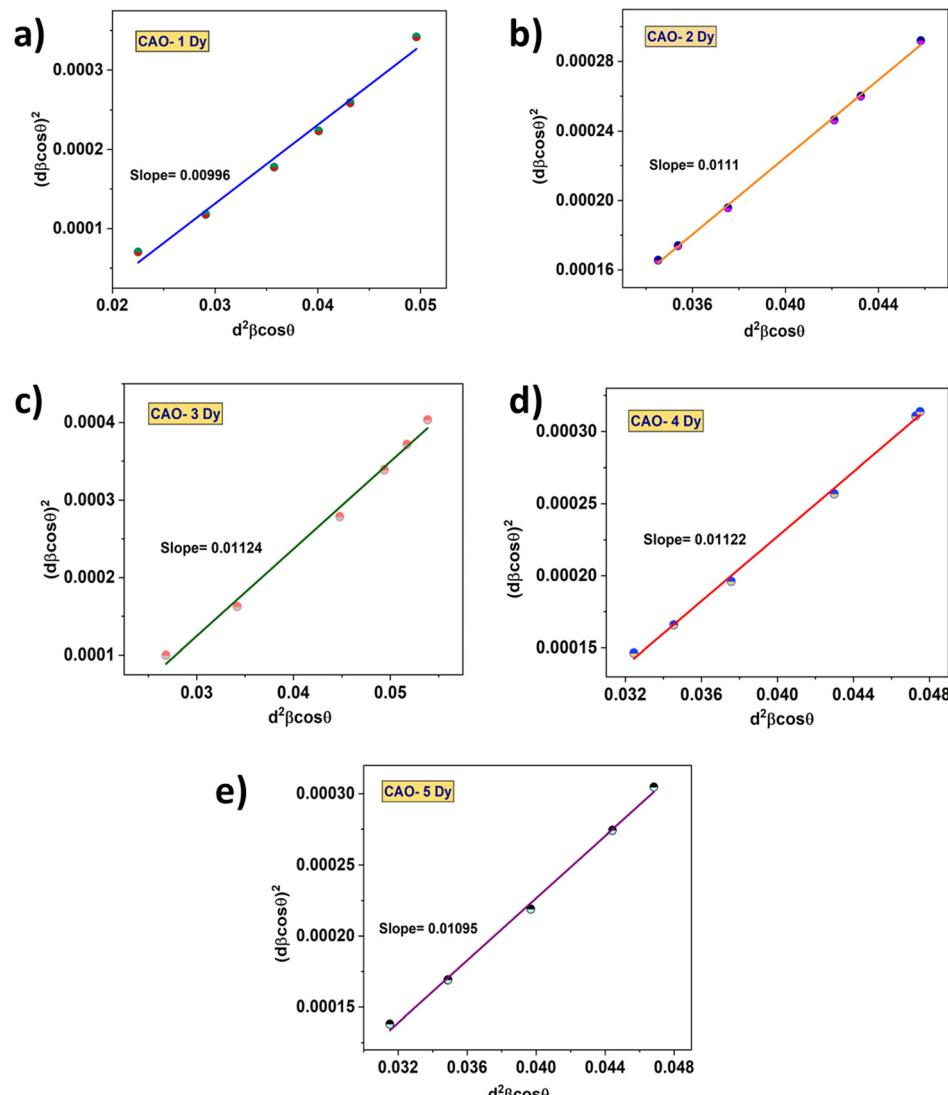


Fig. 2 (a)–(e) Crystallite analysis using the SSP method for CAO–Dy samples.



Table 2 *R* values for different cation-dopant combinations

| Ca ²⁺ -Dy ³⁺ pair | | | | |
|---|---------------------|------------------------|---------------------|--------------|
| CN of Ca ²⁺ | R _h (CN) | CN of Dy ³⁺ | R _d (CN) | <i>R</i> (%) |
| 6 | 1 | 6 | 0.912 | 8.8 |
| 6 | 1 | 8 | 1.027 | 2.7 |
| 8 | 1.12 | 6 | 0.912 | 18.57 |
| 8 | 1.12 | 8 | 1.027 | 20.30 |
| Al ³⁺ -Dy ³⁺ pair | | | | |
| CN of Al ³⁺ | R _h (CN) | CN of Dy ³⁺ | R _d (CN) | <i>R</i> (%) |
| 6 | 0.535 | 6 | 0.912 | 70.40 |
| 6 | 0.535 | 8 | 1.027 | 91.90 |

Table 3 Crystallite size variation using Scherrer's formula and SSP approach

| Sample name | 2θ (degrees) | FWHM (degrees) | Scherrer's method <i>D</i> (nm) | SSP method <i>D</i> (nm) |
|-------------|--------------|----------------|---------------------------------|--------------------------|
| CAO-1 Dy | 33.415 | 0.4124 | 20.10 | 75.30 |
| CAO-2 Dy | 33.2474 | 0.4477 | 18.50 | 68.20 |
| CAO-3 Dy | 33.2023 | 0.3594 | 23.05 | 66.72 |
| CAO-4 Dy | 33.2158 | 0.3692 | 22.44 | 66.85 |
| CAO-5 Dy | 33.1194 | 0.3928 | 21.10 | 68.50 |

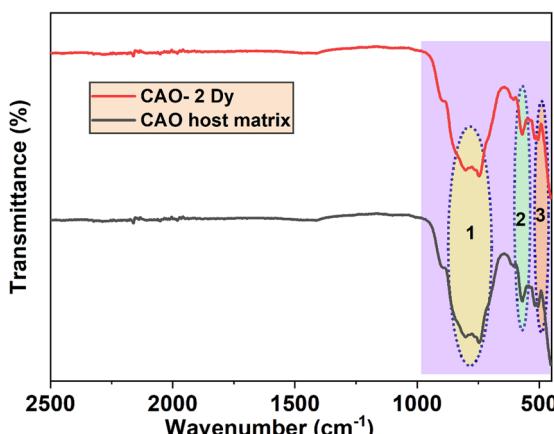
nature of dopant-ligand covalent band is reported in previous studies.⁸¹⁻⁸³

The optical energy band gap of pure and optimized CAO-2 Dy samples is calculated using Tauc plot, using the following equation,^{84,85}

$$[F(R) \cdot E]^{\frac{1}{n}} = K(E - E_g) \quad (6)$$

$$F(R) = \frac{(1 - R)^2}{2R} \quad (7)$$

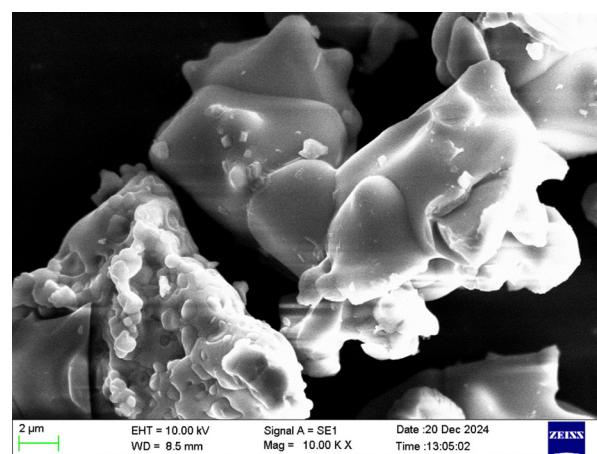
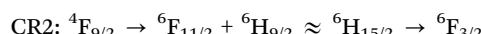
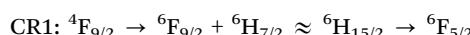
where $F(R)$ is the Kubelka Munk function, E_g is the energy band gap of the material, E is the energy of incident radiation, and R is the sample's reflectance. The value of n differs based on the type of bandgap observed. For direct band gap, $n = 0.5$ and for

**Fig. 3** FTIR spectra of CAO host and CAO-2 Dy samples.

indirect bandgap, $n = 2$. For the synthesized CAO and CAO-2 Dy samples, the best fit is observed for $n = 0.5$, and the x -intercept of $(F(R)hv)^2$ vs. photon energy plot gives the energy gap (Fig. 5(b)). The band gap of pure CAO is 5.01 eV; upon doping, the band gap decreases to 4.83 eV. This bandgap reduction is attributed to forming defect states between the forbidden gap by adding Dy³⁺ ions. Such a decreasing trend in the energy gap is observed and reported in the literature.⁸⁶⁻⁸⁸

3.5 Photoluminescence (PL) analysis

The luminescence properties of Dy-doped CAO phosphors are crucial for understanding their potential applications in solid-state lighting. The excitation spectrum of Dy-doped CAO samples is recorded for a fixed emission wavelength of 575 nm (given in Fig. 6(a)). There are excitation peaks observed at 295 nm, 325 nm, 351 nm, 387 nm, 426 nm, 454 nm, and 465 nm corresponding to the transitions $^6H_{15/2} \rightarrow ^4D_{7/2}$, $^6H_{15/2} \rightarrow ^6P_{3/2}$, $^6H_{15/2} \rightarrow ^6P_{7/2}$, $^6H_{15/2} \rightarrow ^4M_{21/2}$, $^6H_{15/2} \rightarrow ^4G_{11/2}$, $^6H_{15/2} \rightarrow ^4I_{15/2}$ and $^6H_{15/2} \rightarrow ^4F_{9/2}$ respectively.^{89,90} The 351 nm peak corresponding to $^6H_{15/2} \rightarrow ^6P_{7/2}$ is the excitation wavelength to record the emission spectra (Fig. 6(b)). Dy³⁺ characteristic emission peaks are obtained at 483 nm ($^4F_{9/2} \rightarrow ^6H_{15/2}$), 575 nm ($^4F_{9/2} \rightarrow ^6H_{13/2}$), and 663 nm ($^4F_{9/2} \rightarrow ^6H_{11/2}$).⁹¹ The emission intensity shows an increasing trend with Dy³⁺ concentration up to 2 mol%, beyond which the PL intensity reduces (Fig. 6(c)). The reason for the variation in PL intensity is correlated with dopant concentration, which is called concentration quenching. The quenching in PL intensity can be explained using cross-relaxation (CR) paths between neighbouring Dy³⁺ ions. Fig. 7 illustrates the energy level diagram and the potential cross-relaxation pathways for the Dy³⁺ ions.⁹² In this context, we identify three distinct energy transfer (ET) channels between identical Dy³⁺ ions within the CAO host, designated CR1, CR2, and CR3. The ET channels result in non-radiative energy transfer; hence, the emission intensity decreases. The ET transitions are given below.^{93,94}

**Fig. 4** Surface morphology of CAO-2 Dy phosphor.

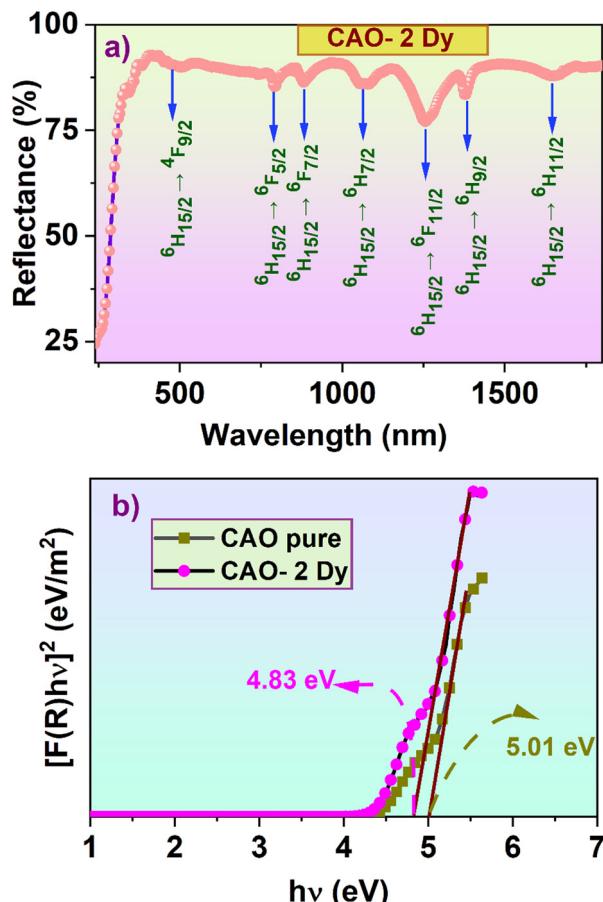
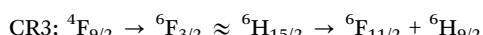


Fig. 5 (a) DR spectrum of CAO–2 Dy phosphor, (b) optical band gap of pure and doped CAO samples.



In addition to the cross-relaxation mechanism, the ET can be associated with the multipolar or exchange interactions among Dy^{3+} ions. The mode of ET is determined by estimating the critical radius R_c using the below equation,

$$R_c = 2 \left(\frac{3V}{4\pi X_c N} \right)^{1/3} \quad (8)$$

where the unit cell volume, $V = 1725.97 \text{ \AA}^3$, optimum concentration; $X_c = 0.02$, and $N = 2$. The value of $R_c = 43.517 \text{ \AA}$. As the critical distance exceeds 5 \AA , indicating that concentration

Table 4 Bonding parameter calculation for CAO–2 Dy

| Sl. number | Transition from ${}^6\text{H}_{15/2}$ to | $\nu_c \text{ (cm}^{-1}\text{)}$ | $\nu_a \text{ (cm}^{-1}\text{)}$ | β |
|----------------------|--|----------------------------------|----------------------------------|---------------|
| 1 | ${}^4\text{F}_{9/2}$ | 20 963 | 21 100 | 0.9935 |
| 2 | ${}^6\text{F}_{5/2}$ | 12 323 | 12 400 | 0.9938 |
| 3 | ${}^6\text{F}_{7/2}$ | 10 926 | 11 000 | 0.9932 |
| 4 | ${}^6\text{H}_{7/2}$ | 9014 | 9100 | 0.9905 |
| 5 | ${}^6\text{F}_{11/2}$ | 7667 | 7700 | 0.9957 |
| 6 | ${}^6\text{H}_{9/2}$ | 7637 | 7692 | 0.9928 |
| 7 | ${}^6\text{H}_{11/2}$ | 5835 | 5850 | 0.9974 |
| β_{avg} | | | | 0.9938 |

quenching results from multipolar interaction. Several multipolar interactions exist, including dipole–dipole, dipole–quadrupole, and quadrupole–quadrupole. Dexter's theory⁹⁵ can be employed to determine the specific multipolar interaction in CAO–Dy phosphors (eqn (9)).

$$\log \frac{I}{x} = c - \frac{\theta}{3} \log x \quad (9)$$

The relationship between $\log I/x$ and $\log x$ exhibit a slope of $-(\theta/3)$, where θ serves as an indicator of the multipolar interaction type (Fig. 8(a)). Specifically, θ values of 6, 8, and 10 correspond to dipole–dipole, dipole–quadrupole, and quadrupole–quadrupole reactions. An analysis of the graph depicting $\log(I/x)$ against $\log(x)$ reveals a slope of -1.73537 . Given that $\theta \approx 5.20$ approximates 6, the data suggests that dipole–dipole interactions predominantly govern the energy transfer mechanism.^{96,97} The reduction in PL intensity could be related to substituting the Dy^{3+} ion into the Ca^{2+} site. The charge imbalance leads to the formation of defects. The probable defects introduced to maintain charge neutrality are oxygen vacancies (V_o) and cationic vacancies (V_{Ca}). These defects act as electron traps, reducing the emission intensity.^{98,99}

The intense yellow emission (${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{13/2}$) due to electric dipole is more intense than the magnetic dipole transition corresponding to the blue emission (${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{15/2}$). Thus, we can specify that the Dy^{3+} ions do not occupy inversion symmetry sites as the hyper-sensitive electric dipole transition is dominant (Fig. 8(b)). The magnetic dipole transition will be predominant if the dopant ions occupy inversion symmetry sites.¹⁰⁰ The Y/B ratios for CAO–Dy samples are 2.15, 2.24, 2.32, 2.37, and 2.32 for 1, 2, 3, 4, and 5 mol% of dopant concentration (Fig. 8(c)). As the (Y/B) > 1 , the emission appears to be yellowish-white light rather than pure white light.^{101,102}

3.6 Photometric analysis

The tristimulus values determine the colour coordinates of the prepared phosphor sample (refer to eqn (S1)–(S6), ESI†),¹⁰³

In the Fig. 9, the colour coordinates fall in the yellowish-white region, and the colour purity is estimated using the below formula¹⁰⁴

$$\text{Colour purity (CP)} = \frac{\sqrt{(x_s - x_0)^2 + (y_s - y_0)^2}}{\sqrt{(x_d - x_0)^2 + (y_d - y_0)^2}} \quad (10)$$

The coordinates (x_0, y_0) , (x_s, y_s) , and (x_d, y_d) represent the epicentre of convergence (0.332, 0.186), coordinates of the phosphor sample, and coordinates of the dominant emission, respectively. The correlated colour temperature is determined using McCamy's formula¹⁰⁵

$$\text{CCT} = -449n^3 + 3525n^2 - 6823n + 5520.33 \quad (11)$$

where n is the slope of the inverse line, $n = (x - x_0)/(y - y_0)$.

The colour rendering index (CRI) measures how accurately a light source displays colours by comparing it to an ideal or natural lighting benchmark. The quality of light source is evaluated depending on the range of CRI value. The CRI range is 68–72, corresponding to a good light source with high accuracy in colour appearance.

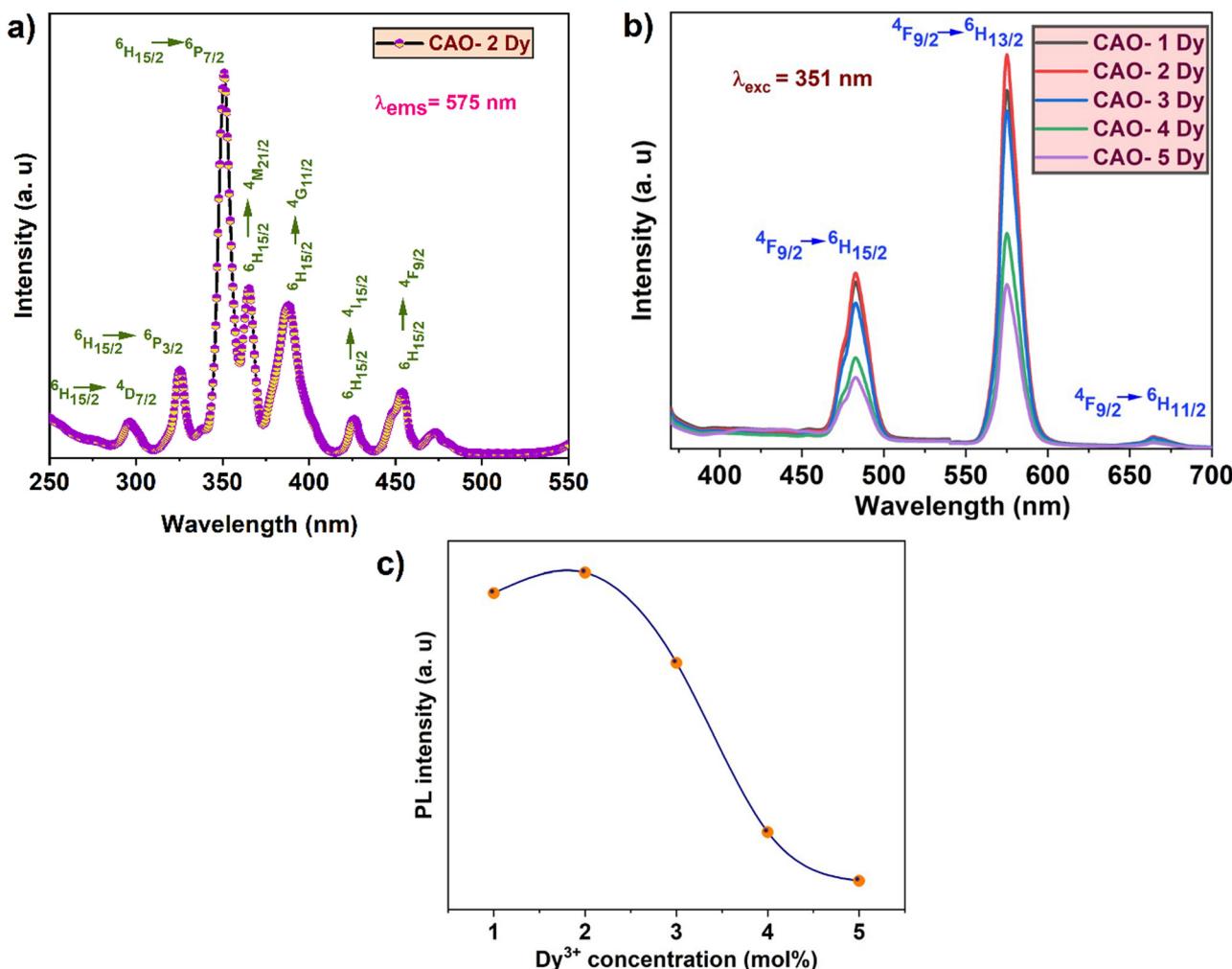


Fig. 6 (a) Excitation spectrum of CAO–2 Dy phosphor, (b) emission spectra of CAO samples for different dopant concentrations, and (c) variation in PL intensity as a function of Dy³⁺ concentration.

Table 5 gives the colour coordinates, colour purity, and CRI and CCT values of CAO–Dy phosphors. Table 6 compares emission wavelength, Colour coordinates, CCT, CP, and CRI values of other phosphors and the present work. The synthesized CAO–Dy phosphors emit cool white light (CCT > 4000 K) with good CRI value; hence, they have potential cool/neutral light generation applications.

3.7 Lifetime analysis

Monitoring the excitation wavelength at 351 nm and the emission wavelength at 575 nm allowed the determination of the luminescence lifetime of the Ca₂Al₂O₅:xDy³⁺ [x = 1, 2, 3, 4, and 5 mol%] phosphors. Fig. 10(a) displays the decay profile, which displays the intensity as a function of decay lifetime. A single exponential decay function fit was used to further examine the experimental data, and equation was represented as follows,^{116,117}

$$I(t) = Ae^{-\frac{t}{\tau}} + I_0 \quad (12)$$

Here τ is the component of the decay lifetimes, A is the fitting

parameter and I_0 the initial fluorescence intensity. The single exponential fit is done and shown in Fig. 10(b).

The prepared Ca₂Al₂O₅:xDy³⁺ [x = 1, 2, 3, 4, and 5 mol%] phosphor samples had calculated average lifetimes of 0.8722 ms, 0.9925 ms, 0.9276 ms, 0.8928 ms, and 0.8827 ms respectively. The average lifetime increases for 2 mol% and gradually decreases as the doping concentration rises, indicating that the energy transfer activities between Dy³⁺ ions. Auzel's hypothetical model^{118,119} was used to assess this trend, and Fig. 10(c) shows the fitted profile.

$$\tau_c = \frac{\tau_0}{\left(1 + \frac{c}{c_0}\right)e^{-N/3}} \quad (13)$$

In this relation, τ_c is the lifetime at doping concentration c , c_0 is the critical concentration, and N is the number of phonons generated. 1.073 ms is the intrinsic radiative lifetime (τ_0). The following formula may be used to determine the non-radiative relaxation rate (k_{nr}) given the radiative lifespan (τ_0) and the

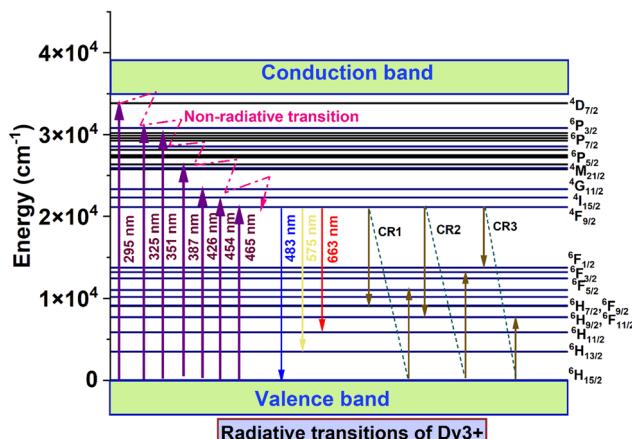


Fig. 7 Energy level diagram of Dy^{3+} ion showing radiative transitions and cross-relaxation paths.

empirically obtained average lifetime (τ_{avg}).¹²⁰

$$\frac{1}{\tau_c} = \frac{1}{\tau_0} + k_{\text{nr}} \quad (14)$$

One important metric for evaluating the optical performance of rare earth doped phosphors is quantum efficiency. It may be quantitatively stated in terms of radiative and non-radiative transition rates, as well as the excited-state lifespan, and is defined as the ratio of emitted to absorbed light intensity. The efficiency of energy conversion in luminous materials is

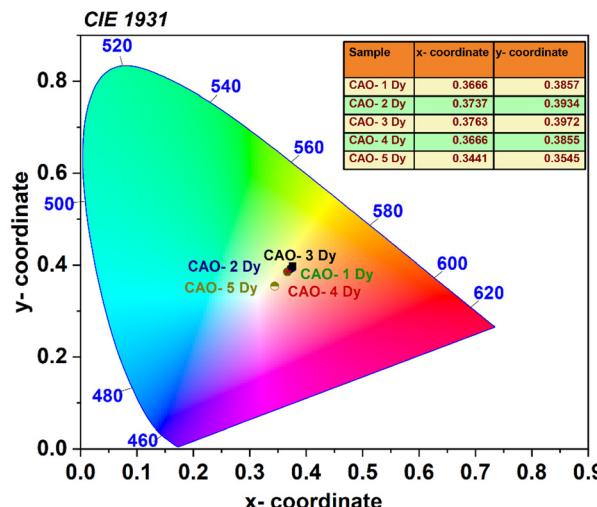


Fig. 9 Chromaticity diagram of CAO-Dy samples.

determined by this metric, which offers a direct measurement of quantum efficiency. The quantum efficiency can be expressed numerically as,¹²¹

$$\eta = \frac{I_{\text{em}}}{I_{\text{ab}}} = \frac{k_R}{k_R + k_{\text{nR}}} = \frac{\tau_c}{\tau_0} \quad (15)$$

The calculated average lifetime, nonradiative relaxation rate, and quantum efficiency are listed in Table 7.

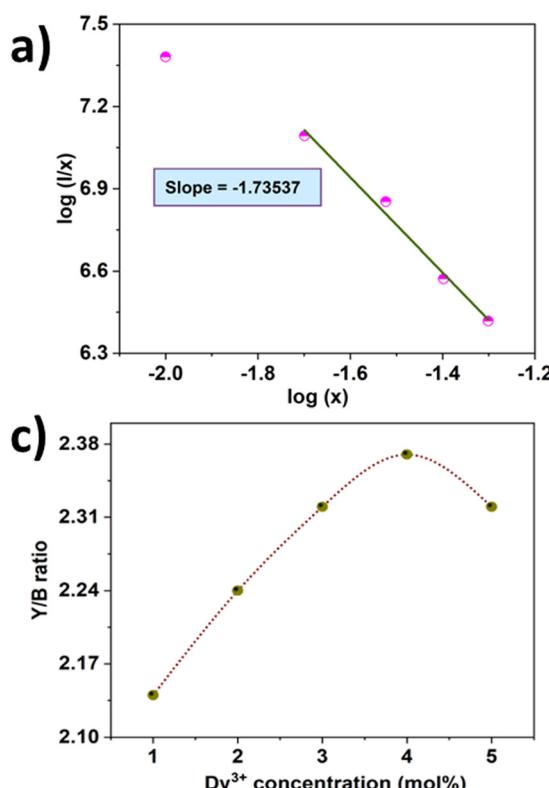


Fig. 8 (a) $\log(l/x)$ versus $\log(x)$ plot for CAO-Dy samples, (b) relative intensity comparison of blue and yellow emission, and (c) Y/B ratio of CAO-Dy phosphors.



Table 5 CIE coordinates and CCT values of CAO–Dy phosphors

| Sample code | CIE coordinates (x_s, y_s) | Colour purity (%) | CRI | CCT (K) |
|-------------|--------------------------------|-------------------|-----|---------|
| CAO-1 Dy | (0.3666, 0.3858) | 54 | 71 | 4445 |
| CAO-2 Dy | (0.3737, 0.3934) | 57 | 69 | 4291 |
| CAO-3 Dy | (0.3763, 0.3972) | 57 | 68 | 4244 |
| CAO-4 Dy | (0.3666, 0.3855) | 54 | 69 | 4443 |
| CAO-5 Dy | (0.3441, 0.3545) | 42 | 72 | 5047 |

3.8 Temperature dependent photoluminescence (TDPL)

Furthermore, the TDPL properties of the optimized CAO–2 Dy were extensively examined to evaluate its thermal stability and emission behaviour at elevated temperatures. The TDPL spectra have the characteristic emission peaks of Dy^{3+} recorded from 303 K to 483 K. A gradual reduction in photoluminescence intensity was observed as the temperature increased (Fig. 11(a)). Thermal quenching is mainly attributed to the increased

Table 6 Comparison of CIE coordinates, colour purity, CRI and CCT values of the optimized phosphor with previously reported works

| Phosphor | Emission wavelength (nm) | CIE coordinates (x_s, y_s) | Colour purity (%) | CRI | CCT (K) | Ref. |
|-----------------------------|--------------------------|--------------------------------|-------------------|-----|---------|-----------|
| $Ba_2TeP_2O_6:Dy^{3+}$ | 573 | (0.3981, 0.4333) | 55.3 | — | 3926 | 106 |
| $CaZn_2(PO_4)_2:Dy^{3+}$ | 572 | (0.3251, 0.3482) | — | 80 | 5815 | 107 |
| $Y_2CaB_{10}O_{19}:Dy^{3+}$ | 577 | (0.3188, 0.3233) | 16.2 | 77 | 6209 | 108 |
| $Y_2O_3:Dy^{3+}$ | 575 | (0.2650, 0.3880) | — | 33 | 8199 | 109 |
| $GdSr_2AlO_5:Dy^{3+}$ | 582 | (0.3396, 0.3851) | 17.6 | — | 5272 | 110 |
| $NaSrPO_4:Dy^{3+}$ | 576 | (0.2700, 0.3000) | — | — | 10150 | 111 |
| $CaLiLa(PO_4)_2:Dy^{3+}$ | 573 | (0.2750, 0.3006) | — | — | — | 112 |
| $SrLu(PO_4)_3:Dy^{3+}$ | 575 | (0.3740, 0.4070) | — | — | — | 113 |
| $K_3ZnB_5O_{10}:Dy^{3+}$ | 575 | (0.2560, 0.2580) | — | — | — | 114 |
| $Y_2MoO_6:Dy^{3+}$ | 575 | (0.3391, 0.3458) | 91 | — | 5218 | 115 |
| $Ca_2Al_2O_5:Dy^{3+}$ | 575 | (0.3737, 0.3934) | 57 | 69 | 4291 | This work |

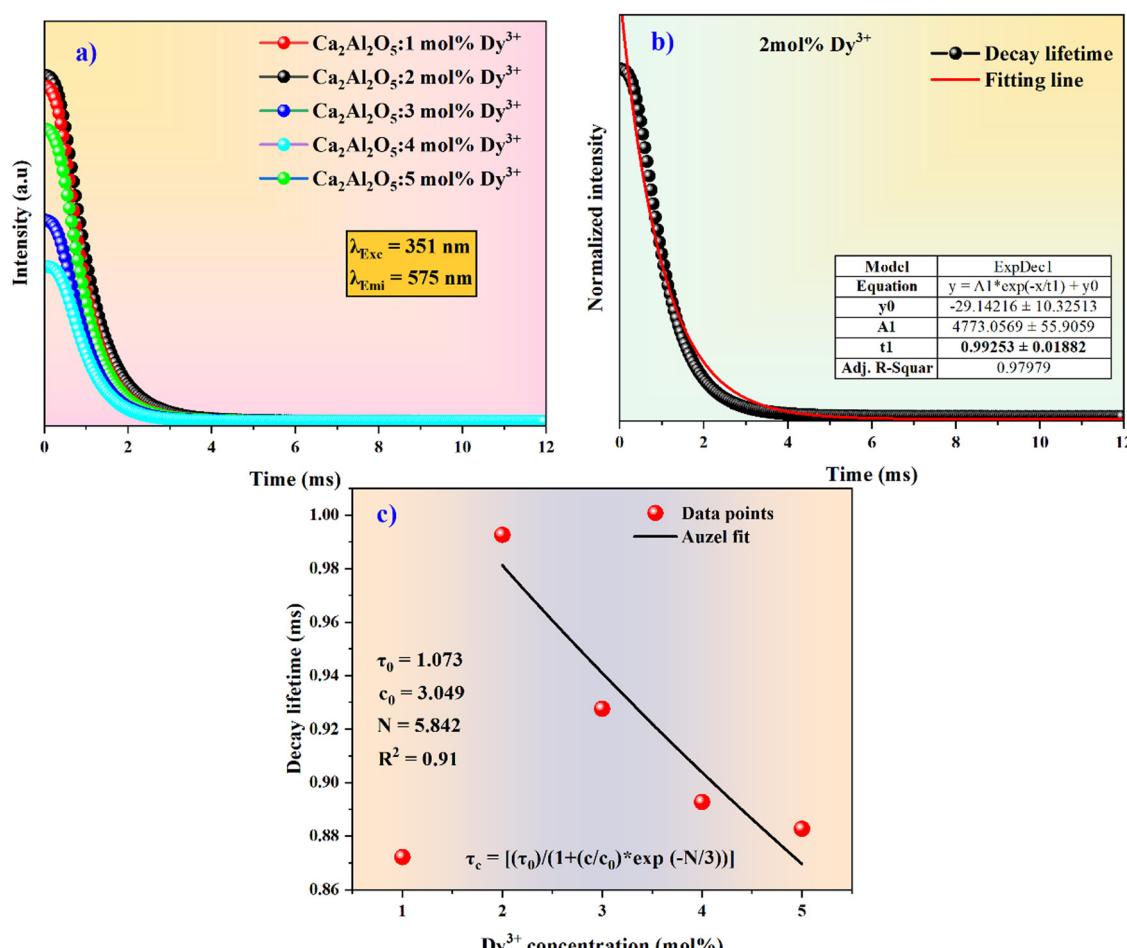


Fig. 10 (a) Fluorescence decay lifetime spectra for different concentration of Dy^{3+} in $Ca_2Al_2O_5$ phosphors. (b) Single exponential fit for the 2 mol% Dy^{3+} . (c) Auzel's fitting curve showing variation of fluorescence lifetime with Dy^{3+} concentrations.



Table 7 Decay time, non radiative relaxation rate and quantum efficiency of prepared $\text{Ca}_2\text{Al}_2\text{O}_5:\text{xDy}^{3+}$ [$x = 1, 2, 3, 4$, and $5 \text{ mol}\%$] phosphors

| Dy ³⁺ concentration (mol%) | τ_c (ms) | k_{nr} (s^{-1}) | η (%) |
|---------------------------------------|---------------|-------------------------------------|------------|
| 1 | 0.87224 | 214.507 | 81.28 |
| 2 | 0.99253 | 75.55977 | 92.50 |
| 3 | 0.9276 | 146.0844 | 86.44 |
| 4 | 0.89284 | 188.0551 | 83.20 |
| 5 | 0.88279 | 200.8058 | 82.27 |

formation of defect states at higher temperatures, facilitating non-radiative relaxation routes. As a result, non-radiative recombination processes become more dominant than radiative ones, reducing emission intensity.¹²² Fig. 11(b) illustrates the variation in normalized emission intensity at wavelengths of 483 nm, 575 nm, and 663 nm as a function of temperature. The normalized intensities exhibit a consistent decline, indicative of the quenching trend observed in the TDPL spectra, thereby confirming the temperature sensitivity of the luminescence process.

Notably, the emission peaks at 483 nm and 576 nm exhibit almost identical quenching behaviour, suggesting that thermal disturbances similarly affect these transitions and may originate from closely related energy levels within the Dy³⁺ ion.^{123,124} Fig. 11(c) illustrates how the full width at half maximum (FWHM) of the same emission bands varies with temperature. As the temperature increases, the FWHM values for the 483 nm and 575 nm emissions increase, whereas the 663 nm emission

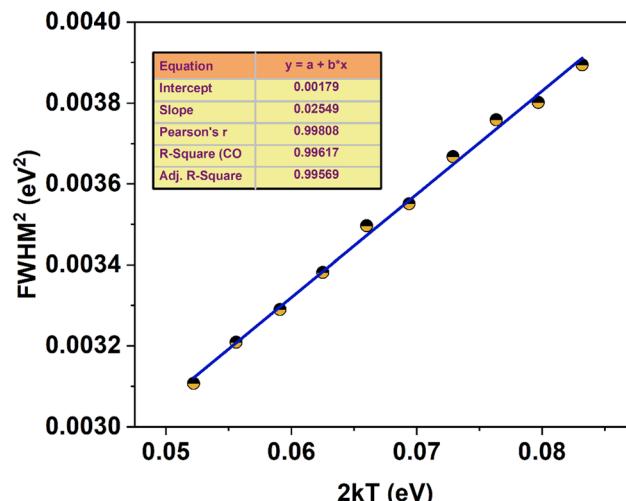


Fig. 12 The linear fit of FWHM^2 as a function of $2kT$ for 575 nm peak.

experiences a narrowing of FWHM. The spectral broadening is correlated with the increased phonon interactions at higher temperatures. There are reported models explaining the spectral broadening and enhanced phonon interactions in rare earth-doped materials.¹²⁵ The dependence of FWHM on temperature is described using the following equation,

$$\Gamma = hv \sqrt{8 \ln(2) S \coth\left(\frac{hv}{2kT}\right)} \quad (16)$$

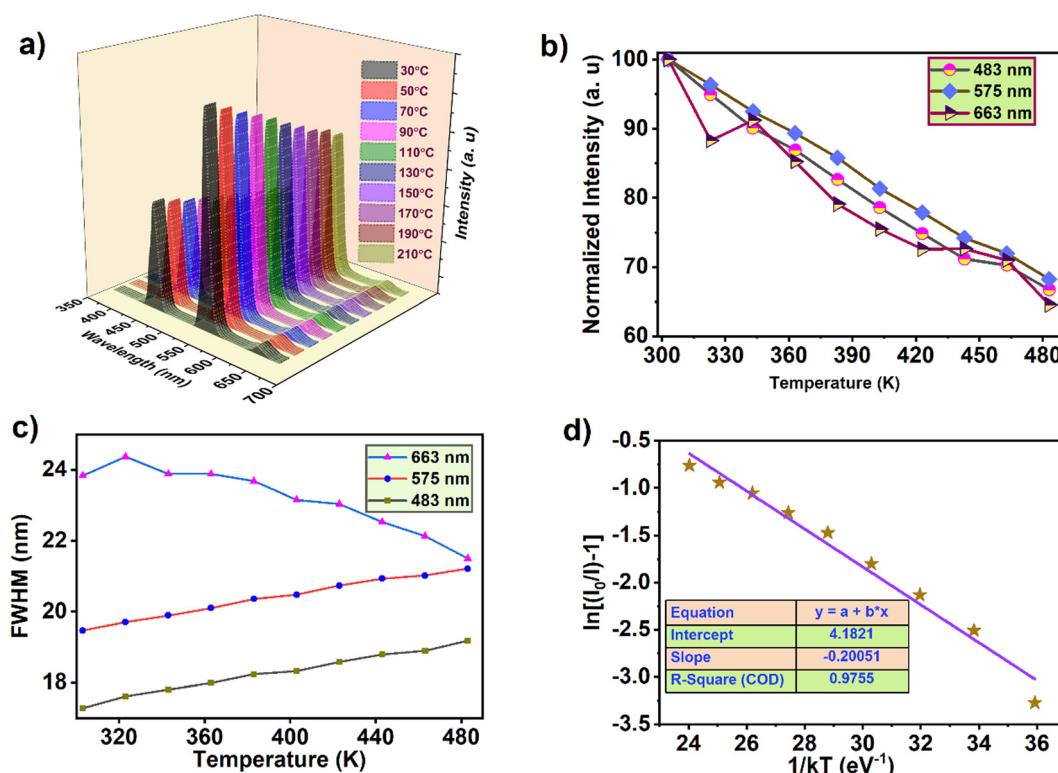


Fig. 11 (a) TDPL spectra of CAO-2 Dy sample, (b) normalized intensity variation, (c) FWHM variation for different emissions, and (d) activation energy graph of CAO-2 Dy phosphor.



Γ represents the full width at half maximum that varies with temperature, S is the Huang–Rhys parameter, k is the Boltzmann constant, $h\nu$ is the effective phonon energy, and T signifies the temperature. The extent of thermal quenching varies from one material to another. The thermal quenching is described as an effect of electron–phonon interaction. It depends on the phonon energy ($h\nu$) and S . If the phonon energy and S are larger, the stronger the electron–phonon interaction results in increased non-radiative relaxations. The thermal stability is associated with these values, and we simplify eqn (16) by expanding $\coth\left(\frac{h\nu}{2kT}\right)$ as $\frac{e^{\frac{h\nu}{kT}} + e^{-\frac{h\nu}{kT}}}{e^{\frac{h\nu}{kT}} - e^{-\frac{h\nu}{kT}}}$ and further simplified, and eqn (16) is squared,

$$\Gamma^2 = 5.57 \times S \times (h\nu)^2 \left(1 + \frac{2}{e^{\frac{h\nu}{kT}} - 1}\right) \quad (17)$$

Approximating $\frac{h\nu}{kT} \approx 10^{-3}$ and $\left(e^{\frac{h\nu}{kT}} - 1\right) \approx \frac{h\nu}{kT}$.

Eqn (17) is simplified into eqn (18),

$$\Gamma^2 = 5.57 \times S \times (h\nu)^2 \left(1 + \frac{1}{\frac{h\nu}{2kT}}\right) \quad (18)$$

Linearizing the above equation by taking $y = a + bx$, Γ^2 or FWHM^2 is taken along y -axis, kT along x -axis, $b = 5.57 \times S \times h\nu$, is the slope, and $a = 5.57 \times S \times (h\nu)^2$ is the intercept. Thus, the phonon energy and the S values are obtained by plotting the FWHM^2 vs. kT graph (Fig. 12). The phonon energy is 0.07022 eV, and $S = 0.06517$ for the 575 nm peak. On comparing the obtained values with the literature available, we can confirm that synthesized CAO–2 Dy samples show excellent thermal stability due to weaker electron–phonon interaction (lower value of S and $h\nu$).^{126,127}

The activation energy must be calculated using the Arrhenius equation to explore the thermal quenching properties.¹²⁸

$$I = \frac{I_0}{1 + Ce^{\frac{-\Delta E}{kT}}} \quad (19)$$

The initial intensity is denoted as I_0 at the starting temperature, while I represent the intensity at temperature T . Here, C stands for a constant, ΔE signifies the activation energy, and k is the Boltzmann constant. On linearizing the equation,

$$\ln\left(\frac{I_0}{I} - 1\right) = \frac{-\Delta E}{kT} + \ln C \quad (20)$$

The slope of $\ln\left(\frac{I_0}{I} - 1\right)$ versus $\frac{1}{kT}$ graph (Fig. 11(d)) gives $\Delta E = 0.20051$ eV.

Table 8 compares the reported systems with CAO–2 Dy phosphors regarding their thermal stability and activation energy. The data indicate that the CAO–2 Dy phosphor demonstrates superior thermal stability and activation energy, rendering it a promising candidate for optoelectronic applications.

Table 8 Comparison of optimized phosphor's thermal stability and activation energies with previously reported phosphors

| Phosphor | Temperature range (K) | Thermal stability (%) | Activation energy (eV) | Ref. |
|---|-----------------------|-----------------------|------------------------|--------------|
| CaLiLa(PO ₄) ₂ :Dy ³⁺ | 303–553 | 65 | 0.250 | 112 |
| SrLu(PO ₄) ₃ :Dy ³⁺ | 298–473 | 68 | 0.214 | 113 |
| K ₃ ZnB ₅ O ₁₀ :Dy ³⁺ | 303–483 | 82 | 0.520 | 114 |
| K ₃ Y(PO ₄) ₂ :Dy ³⁺ | 303–483 | 75 | 0.370 | 129 |
| LiCaBO ₃ :Dy ³⁺ | 100–480 | — | 0.420 | 130 |
| Li ₃ Ba ₂ Gd ₃ (WO ₄) ₈ :Dy ³⁺ | 298–523 | 62 | 0.352 | 131 |
| Na ₂ Y ₂ TeB ₂ O ₁₀ :Dy ³⁺ | 300–475 | 75 | 0.230 | 132 |
| NaGdTiO ₄ :Dy ³⁺ | 298–633 | — | 0.200 | 133 |
| Y ₂ CaB ₁₀ O ₁₉ :Dy ³⁺ | 303–663 | 84 | — | 134 |
| Ca ₃ LuAl ₃ B ₄ O ₁₅ :Dy ³⁺ | 300–500 | 85 | — | 135 |
| Ca ₂ Al ₂ O ₅ :Dy ³⁺ | 303–483 | 68 | 0.200 | Present work |

4. Conclusions

This work effectively synthesized Ca₂Al₂O₅ phosphors doped with Dy³⁺ through the conventional solid-state reaction technique. XRD analysis confirmed the formation of a pure phase, with no significant shifts in peak positions observed upon Dy³⁺ doping, indicating that the host lattice retained its structural integrity across all doping levels. SEM analysis of the surface morphology revealed agglomerated particle formations, characteristic of high-temperature solid-state synthesis processes. Optical characterization via DRS identified distinct Dy³⁺ absorption peaks and a reduction in the optical band gap upon introducing the dopant into the host matrix, underscoring the impact of Dy³⁺ incorporation. PL spectra exhibited the characteristic emission peaks of Dy³⁺ ions, corresponding to the $^4F_{9/2} \rightarrow ^6H_{15/2}$ (blue) and $^4F_{9/2} \rightarrow ^6H_{13/2}$ (yellow), and $^4F_{9/2} \rightarrow ^6H_{11/2}$ (red) transitions. 2 mol% Dy³⁺ doping yielded the highest emission intensity, with chromaticity coordinates within the cool white light region, rendering it suitable for lighting applications. TDPL studies demonstrated significant thermal stability, with the phosphor maintaining substantial luminescence at elevated temperatures. The calculated activation energy was determined to be 0.200 eV, highlighting the lower probability for non-radiative losses. In conclusion, the exceptional optical properties, robust thermal stability, and emission in the visible white-light spectrum position make Dy³⁺-doped Ca₂Al₂O₅ is identified as a promising phosphor candidate for applications in solid-state lighting and other optoelectronic devices.

Author contributions

Vidya Saraswathi. A: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Validation, Writing - original draft, Writing - review & editing; Tejas: Synthesis, Formal analysis, Writing, review; S. Masilla Moses Kennedy: Instrumentation, Formal analysis; A. Princy: Instrumentation, Formal analysis; M. I. Sayyed: Proofreading, Review & editing; Aljawhara H. Almuqrin: Review & editing; Vikash Mishra: Formal analysis, Review & editing; Sudha. D. Kamath: Supervision, review and editing. All authors have read and agreed to the published version of the manuscript.



Conflicts of interest

There are no conflicts to declare.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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