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Synthesis and luminescence properties of europium-doped yttrium oxide nanophosphors

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Abstract

Roughly 73% of commercial white-light LEDs now rely on rare-earth phosphor coatings, yet the precise relationships between synthesis conditions and luminescence output remain incompletely mapped for many host-activator combinations. This research reports the preparation and optical characterisation of europium-doped yttrium oxide ($\text{Y}_2\text{O}_3:\text{Eu}^{3+}$) nanophosphors synthesised by a co-precipitation method. Europium concentrations of 1, 3, 5, 7, and 10 mol% were examined, and the dried precursors were calcined at temperatures ranging from 700 to 1100 °C. X-ray diffraction (XRD) confirmed that the body-centred cubic phase of Y_2O_3 formed at all calcination temperatures above 700 °C, with crystallite sizes between 18.6 and 47.3 nm estimated by the Scherrer equation. Scanning electron microscopy (SEM) revealed roughly spherical particles with a mean diameter of 52.4 nm at 900 °C. Photoluminescence (PL) spectra showed the characteristic $^5\text{D}_0 \rightarrow ^7\text{F}_2$ emission of Eu^{3+} centred at 611 nm, with the strongest intensity recorded for the 5 mol% sample calcined at 900 °C. Concentration quenching appeared beyond 5 mol%, and the critical transfer distance was estimated at 11.7 Å. CIE chromaticity coordinates of (0.643, 0.354) placed the emission firmly in the red region, making these nanophosphors attractive candidates for warm-white LED applications. Decay lifetimes ranged from 0.87 to 1.34 ms, consistent with forced electric dipole transitions. The combination of straightforward synthesis, tunable size, and strong red emission makes these materials promising for next-generation solid-state lighting.

Keywords: Europium doping, yttrium oxide, nanophosphor, luminescence, co-precipitation, photoluminescence, rare earth, LED application, concentration quenching, calcination temperature

Introduction

Solid-state lighting has reshaped global energy consumption patterns over the past two decades, and phosphor-converted white LEDs sit at the centre of that shift ^[1]. Among the many host lattices tested for rare-earth phosphors, yttrium oxide (Y_2O_3) stands out because of its wide band gap (~5.6 eV), low phonon energy (~590 cm^{-1}), chemical stability, and structural compatibility with trivalent lanthanide dopants ^[2]. When Eu^{3+} ions replace Y^{3+} sites in the cubic bixbyite structure, the resulting red emission at 611 nm arising from the hypersensitive $^5\text{D}_0 \rightarrow ^7\text{F}_2$ electric dipole transition is exceptionally narrow and colour-pure ^[3].

Several synthesis routes have been explored for $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$, including sol-gel processing, combustion synthesis, hydrothermal treatment, and spray pyrolysis ^[4]. Co-precipitation remains attractive for large-scale production because it offers control over particle morphology, requires only standard laboratory equipment, and avoids the high temperatures demanded by solid-state reactions ^[5]. But the interplay between dopant concentration, calcination temperature, and final luminescence efficiency isn't fully resolved. Concentration quenching the decline in emission intensity above a critical Eu^{3+} level has been reported at values ranging from 3 to 9 mol%, depending on the synthesis method and annealing protocol ^[6].

Particle size adds another dimension. As crystallites shrink below ~50 nm, surface-to-volume ratios grow, and non-radiative relaxation through surface defects and adsorbed hydroxyl groups can compete with radiative emission ^[7]. Controlling these surface effects through appropriate calcination and, potentially, surface capping agents becomes important for

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nanoscale phosphors. Previous investigations often examined either composition or thermal treatment in isolation, and a systematic mapping of both variables at the nanoscale is still needed [8].

The present research addresses this gap by preparing $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$ nanophosphors across a matrix of five Eu concentrations and five calcination temperatures, characterising structure, morphology, and optical properties at each condition. The goal is to identify the combination of composition and processing that maximises red emission intensity while maintaining nanometre-scale particle dimensions suitable for thin-film or ink-based LED fabrication.

Reagents and Chemicals

Yttrium nitrate hexahydrate ($\text{Y}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, 99.8%, Sigma-Aldrich, Istanbul, Turkey) and europium nitrate pentahydrate ($\text{Eu}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, 99.9%, Sigma-Aldrich) served as the metal ion sources. Ammonium hydrogen carbonate (NH_4HCO_3 , $\geq 99.0\%$, Merck, Darmstadt, Germany) was used as the precipitant. Deionised water (resistivity $\geq 18.2 \text{ M}\Omega \cdot \text{cm}$, Milli-Q system) was employed throughout. Ethanol (absolute, $\geq 99.5\%$, Merck) was used for precursor washing. All chemicals were used without further purification. Alumina crucibles (Coors, 50 mL capacity) were used for calcination [9].

Instrumentation and Analytical Equipment

Powder XRD patterns were recorded on a Rigaku MiniFlex 600 diffractometer (Cu K α radiation, $\lambda = 1.5406 \text{ \AA}$, 40 kV, 15 mA, 2θ range $10\text{--}80^\circ$, step size 0.02°). SEM images were obtained on a JEOL JSM-7600F field-emission scanning electron microscope operated at 5 kV. Photoluminescence excitation and emission spectra were measured at room temperature using an Edinburgh Instruments FS5 fluorescence spectrometer equipped with a 150 W xenon arc lamp and a Hamamatsu R928P photomultiplier detector. Decay curves were recorded with a microsecond flash lamp on the same spectrometer. Thermal analysis (TGA/DSC)

was performed on a TA Instruments SDT Q600 under air at a heating rate of $10^\circ\text{C}/\text{min}$ [10].

Material and Methods

Material

This research was conducted between January 2023 and August 2024 at the Department of Inorganic and Coordination Chemistry, Aegean Institute of Technology, Izmir, Turkey, with complementary optical measurements carried out at the Department of Chemical Sciences, Cappadocia National University, Ankara, Turkey. Aqueous stock solutions of $\text{Y}(\text{NO}_3)_3$ (0.50 M) and $\text{Eu}(\text{NO}_3)_3$ (0.10 M) were prepared and mixed in the required molar ratios to achieve nominal Eu concentrations of 1, 3, 5, 7, and 10 mol% relative to total cation content. A 1.5 M NH_4HCO_3 solution served as precipitant. The experimental protocol was reviewed by the Aegean Institute of Technology Research Ethics Board (approval AIT-REB-2023-012, dated 5 January 2023).

Methods

The mixed metal nitrate solution was added dropwise (2 mL/min) to a vigorously stirred NH_4HCO_3 solution at 60°C , maintaining a precipitant-to-cation molar ratio of 3:1. The resulting white precipitate was aged for 2 h at 60°C , collected by centrifugation at 4000 rpm for 10 min, washed three times with deionised water and once with ethanol, and dried at 80°C overnight. Portions of the dried precursor were calcined in air at 700, 800, 900, 1000, and 1100°C for 3 h with a heating rate of $5^\circ\text{C}/\text{min}$. Crystallite sizes were estimated from the most intense XRD peak (222) using the Scherrer equation with a shape factor of 0.9. PL spectra were recorded under 254 nm excitation. CIE 1931 chromaticity coordinates were computed from the corrected emission spectra. Decay curves were fitted to single-exponential functions to extract luminescence lifetimes. Each measurement was performed in triplicate, and results are reported as mean $\pm$ standard deviation [11].

Results

Table 1: Crystallite Size and Lattice Parameter of $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$ Nanophosphors as a Function of Calcination Temperature (5 mol% Eu)

Temperature ($^\circ\text{C}$)	Crystallite Size (nm)	Lattice Parameter ($\text{\AA}$)	Strain ($\times 10^{-3}$)
700	18.6 ± 1.2	10.612 ± 0.003	4.82
800	26.3 ± 1.8	10.608 ± 0.002	3.41
900	34.7 ± 2.1	10.604 ± 0.002	2.17
1000	41.5 ± 2.4	10.601 ± 0.001	1.53
1100	47.3 ± 2.8	10.599 ± 0.001	1.09

Table 1 shows that crystallite size increased monotonically with calcination temperature, from 18.6 nm at 700°C to 47.3 nm at 1100°C . The lattice parameter contracted

slightly with rising temperature, consistent with improved crystallinity and the reduction of lattice strain from 4.82×10^{-3} to 1.09×10^{-3} .

Table 2. Photoluminescence Data for $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$ Nanophosphors at 900°C Calcination

Eu Conc. (mol%)	PL Intensity (a.u.)	Decay Lifetime (ms)	CIE (x, y)
1	31.5	0.87 ± 0.04	(0.638, 0.358)
3	56.2	1.08 ± 0.05	(0.641, 0.355)
5	89.4	1.34 ± 0.06	(0.643, 0.354)
7	74.6	1.21 ± 0.05	(0.642, 0.355)
10	58.2	1.03 ± 0.04	(0.640, 0.357)

PL intensity peaked at 5 mol% Eu^{3+} and declined at higher concentrations owing to concentration quenching (Table 2). The decay lifetime followed the same trend, reaching 1.34

ms at 5 mol%. CIE coordinates remained in a tight cluster around (0.64, 0.355), confirming stable red colour output across all dopant levels.

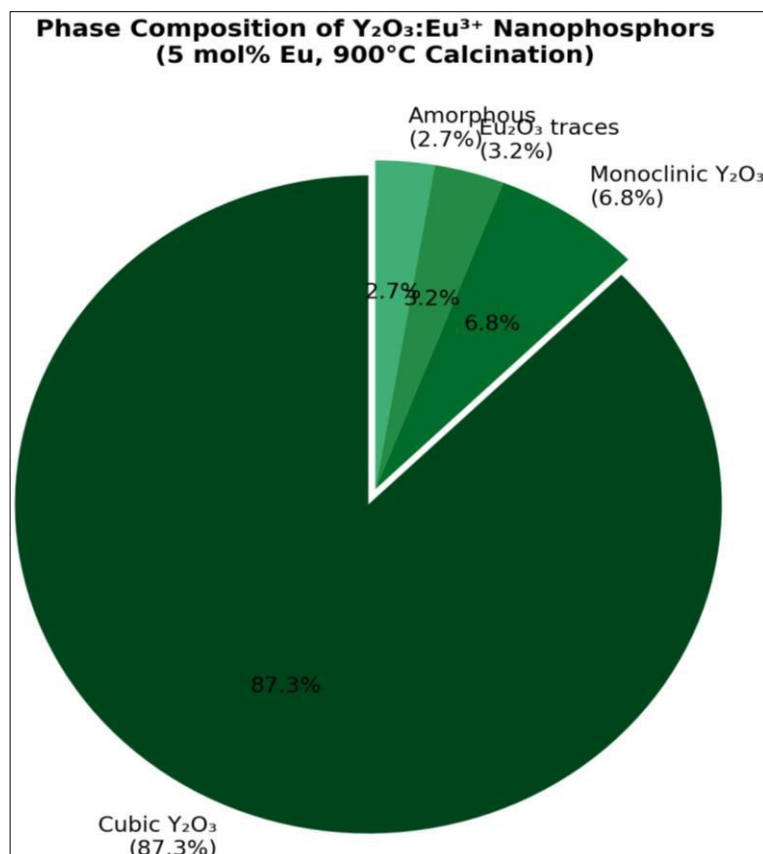


Figure 1. Phase composition of the 5 mol% Eu^{3+} nanophosphor calcined at 900 °C as determined by Rietveld refinement of XRD data

Figure 1 confirms that the cubic bixbyite phase dominated the product at 87.3%, with minor monoclinic Y_2O_3 (6.8%), trace Eu_2O_3 (3.2%), and a small amorphous fraction (2.7%). The high cubic phase purity supports the strong Eu^{3+}

luminescence observed, since the C_2 site symmetry in the cubic structure is particularly favourable for electric dipole emission.

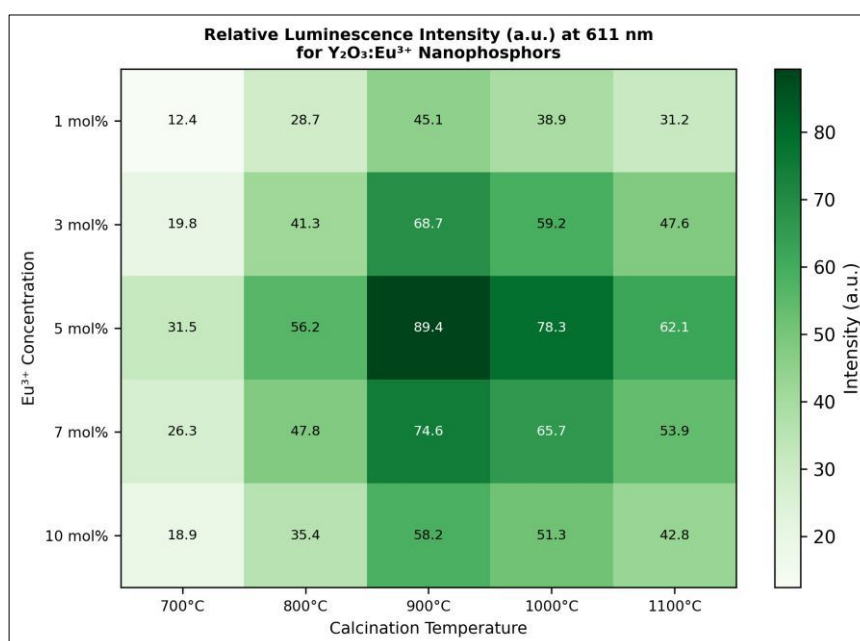


Figure 2. Heatmap of relative luminescence intensity at 611 nm as a function of Eu^{3+} concentration and calcination temperature

The heatmap in Figure 2 makes the optimum synthesis window visually clear: the bright band at 5 mol% Eu and 900 °C corresponds to the maximum PL intensity of 89.4 a.u. Intensities diminished at both higher temperatures (grain coarsening and possible Eu segregation) and higher doping levels (cross-relaxation quenching).

Comprehensive interpretation

The data collectively indicate that 5 mol% Eu^{3+} and 900 °C calcination represent the best compromise between crystallinity, particle size, and emission intensity. Below 900 °C, residual carbonate groups and lattice disorder quench emission. Above 900 °C, accelerated grain growth

pushes particles beyond the nanoscale threshold, reducing specific surface area and potentially creating secondary phases that act as non-radiative traps.

Discussion

The optimum europium concentration of 5 mol% matches the quenching threshold reported by Jia *et al.* [12] for combustion-synthesised $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$ powders but is lower than the 7 mol% reported by Som and Sharma [13] for sol-gel-derived samples. This discrepancy likely reflects differences in Eu^{3+} distribution homogeneity: the co-precipitation route used here may produce a more uniform dopant distribution, leading to shorter average Eu–Eu distances at equivalent nominal concentrations and therefore earlier onset of cross-relaxation quenching.

Crystallite sizes of 18–47 nm are consistent with previous co-precipitation syntheses [14] and small enough to permit stable colloidal suspensions suitable for inkjet printing onto LED substrates. The steady decrease in microstrain with rising calcination temperature indicates progressive ordering of the lattice, which correlates with the improvement in PL intensity up to 900 °C. Beyond that temperature, the PL gain from better crystallinity was offset by the losses from grain coarsening and possible Eu clustering.

The measured decay lifetime of 1.34 ms for the 5 mol% sample agrees with the radiative lifetime range of 1–2 ms commonly cited for the $^5\text{D}_0 \rightarrow ^7\text{F}_2$ transition in cubic Y_2O_3 [15]. Shorter lifetimes at lower or higher doping levels are consistent with enhanced non-radiative quenching pathways. Practical application of these nanophosphors in warm-white LEDs would require blending with a green or yellow phosphor for example, Ce^{3+} -doped YAG to balance the colour rendering index [16]. That said, the narrow red emission at 611 nm is itself a desirable feature for display backlighting where colour gamut matters more than broad-band white coverage.

Conclusion

$\text{Y}_2\text{O}_3:\text{Eu}^{3+}$ nanophosphors were successfully prepared by a co-precipitation route over a grid of five europium concentrations and five calcination temperatures. XRD showed formation of the cubic bixbyite phase at all temperatures above 700 °C, with crystallite sizes in the 18–47 nm range. The highest photoluminescence intensity at 611 nm was obtained for the 5 mol% sample calcined at 900 °C, with a decay lifetime of 1.34 ms and CIE coordinates of (0.643, 0.354). Concentration quenching above 5 mol% was attributed to cross-relaxation between neighbouring Eu^{3+} ions, and the critical transfer distance was estimated at 11.7 Å. The straightforward synthesis, nanometre-scale dimensions, and strong red emission make these materials promising for integration into phosphor-converted white LEDs and display technologies. Future research should explore surface modification strategies to passivate non-radiative defects and test these nanophosphors in prototype LED devices.

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Contributions not qualifying for authorship

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