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RESEARCH ARTICLE

QUANTUM YIELD MEASUREMENT OF PHOSPHORS USING AN INDIGENOUS INTEGRATING SPHERE

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ABSTRACT

This paper reports the measurement of photoluminescence quantum yield (PLQY) of well-known tri-color phosphors, namely $Y_2O_3:Eu^{3+}$ (red), $YVO_4:Eu^{3+}$, $YBO_3:Eu^{3+}$, $BaMgAl_{10}O_{17}:Eu^{2+}$ (blue), and $LaBaB_9O_{16}:Tb^{3+}$ (green), using an indigenously designed integrating sphere coupled with a fluorescence spectrophotometer. The system operates on the principle of collecting the total emitted and scattered radiation from the sample within the integrating sphere and determining the quantum yield by comparing the emission intensity with the incident excitation intensity. The optimum dopant concentrations for all the host materials were achieved using the solid-state diffusion method. The photoluminescence (PL) excitation and emission spectra of the synthesized phosphors were found to be in good agreement with those reported in the literature, confirming the reliability of the prepared samples. The quantum yield values measured using the developed setup were compared with those obtained from a standard FLS-1000 spectrometer (Edinburgh Instruments, UK), demonstrating close agreement and validating the performance of the indigenously designed system. The optical geometry of the Hitachi F-7000 spectrophotometer was modified such that the excitation beam enters the integrating sphere through one port, interacts with the sample, and both the reflected excitation light and the emitted luminescence are sequentially recorded by the detector.

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INTRODUCTION

The development of high-efficiency phosphors is a cornerstone in the advancement of solid-state lighting and modern display technologies, such as white light-emitting diodes, field emission displays, and plasma display panels (1, 2). Among these, tri-colour (red, green, and blue) phosphor systems are essential for achieving high colour rendering indices and tunable chromaticity (3). Specifically, europium-doped yttrium oxide ($Y_2O_3:Eu^{3+}$) (1, 2), yttrium vanadate ($YVO_4:Eu^{3+}$) (4) and yttrium borate ($YBO_3:Eu^{3+}$) (5) are widely recognized as a premier red-emitting phosphor due to its excellent chemical stability, low volatility under vacuum, and high luminescence efficiency. Similarly, materials such as BAM ($BaMgAl_{10}O_{17}:Eu^{2+}$) (6) and terbium-doped lanthanum barium borates ($LaBaB_9O_{16}:Tb^{3+}$) (7) serve as critical components for blue and green emissions, respectively. To optimize these materials for commercial applications, accurate characterization of their optical performance particularly their quantum yield is very important (8). Photoluminescence quantum yield (PLQY) is defined as the ratio of the number of photons emitted to the number of photons absorbed by the sample (8). It is the fundamental metric used to evaluate the efficiency of a luminescent material. While traditional relative methods compare emission spectra against known standards, they are often sensitive to scattering and the specific geometry of the sample. Integrating sphere spectroscopy has emerged as the most reliable method for determining absolute QY, particularly for scattering powder samples, because it collects the total emitted and scattered radiation, thereby eliminating anisotropy in the detection process (9, 10). Despite the importance of absolute QY measurements, the high cost and complexity of commercial stand-alone systems, such as the Edinburgh Instruments FLS-1000, can limit their accessibility in many research environments (11). This has led to an increased interest in modifying existing laboratory equipment to perform accurate absolute measurements (12). The Hitachi F-7000, a widely used fluorescence spectrophotometer, offers high sensitivity and a broad dynamic range, making it an ideal candidate for such modifications (8). In this study, we report the measurement of the quantum yield of a well-known tri-color phosphor set, including $Y_2O_3:Eu^{3+}$, $YVO_4:Eu^{3+}$, $YBO_3:Eu^{3+}$ (red), $BaMgAl_{10}O_{17}:Eu^{2+}$ (blue) and $LaBaB_9O_{16}:Tb^{3+}$ (green). The phosphors were synthesized via the solid-state diffusion method, with dopant concentrations optimized for maximum efficiency. To perform the measurements, we utilized an indigenously designed integrating sphere coupled with a modified Hitachi F-7000 spectrophotometer. The system's performance was validated by comparing results with those obtained from a standard FLS-1000 spectrometer, demonstrating the reliability of our indigenous setup for the accurate characterization of luminescent materials.

Experimental

Design and Calibration of Integrating Sphere: An integrating sphere of nearly 100 mm diameter was mounted inside the sample chamber of the Hitachi F-7000 fluorescence spectrophotometer after removing the inbuilt sample holder along with its base assembly. The inner surface of the integrating sphere was coated with a highly reflective $BaSO_4$ layer to achieve efficient diffuse reflection of photons over the visible spectral region. The reflective coating was prepared by dissolving polyvinyl alcohol (PVA) in warm distilled water under continuous magnetic stirring until a clear viscous solution was obtained. Fine $BaSO_4$ powder was then gradually added to the solution and mixed thoroughly to form a homogeneous reflective paint. This paint was uniformly applied to the inner surface of the sphere using a soft brush in 8–10 thin successive coatings. Each coating layer was completely dried before applying the next layer to avoid cracking and to maintain uniform thickness. The final coating provided diffuse reflectance greater than 95% in the visible region ([13]), ensuring efficient photon scattering and accurate photoluminescence quantum yield (PLQY) measurements. The solid sample holder consisted of a circular metallic disc that could be fixed into a circular opening of approximately 2 cm diameter provided at the bottom of the integrating sphere, as shown in Fig. 1. A shallow groove was precisely made at the centre of the upper surface of the holder to accommodate the powder sample. The phosphor powder was gently pressed into this groove to obtain a smooth and plane sample surface, which is essential for uniform excitation and reproducible measurements. Two optical openings were provided at right angles to each other in the equatorial plane of the integrating sphere. These openings were aligned respectively with the excitation and emission windows of the fluorescence spectrophotometer. A small mirror mounted at the center of the integrating sphere was carefully aligned so that the excitation photons entering through the excitation window were directed onto the sample surface placed in the sample holder. Upon excitation, the sample emitted photoluminescence photons in all directions, while a portion of the excitation photons was also scattered by the sample surface. Both the emitted and scattered photons underwent multiple diffuse reflections from the highly reflective $BaSO_4$-coated inner surface of the integrating sphere. Due to these repeated reflections, the photon distribution inside the sphere became spatially uniform. Finally, the diffusely reflected photons emerged through the emission-side opening and were detected by the spectrophotometer detector system. This arrangement enabled accurate collection of both excitation and emission photon intensities, which is essential for reliable PLQY determination.

For calibration of the integrating sphere setup for powder samples, sodium salicylate powder was used as a standard reference material. A thin layer of sodium salicylate powder was uniformly spread in groove provided at the top surface of the powder sample holder and placed inside the integrating sphere. The emission spectrum of the standard sample was then recorded under UV excitation. The obtained spectrum was compared with the well-known emission characteristics of sodium salicylate to verify proper UV excitation, detector sensitivity, optical alignment, and overall response of the PLQY measurement system for solid-state phosphor samples. PLQY measurement involves the acquisition of an emission spectrum for no-sample condition and with the sample. The PLQY value measured using this set up is compared with the value measured by using FLS-1000 spectrometer (Edinburgh Instruments, UK).

Figure 1. Integrating sphere mounted inside the sample chamber of the Hitachi F-7000 fluorescence spectrophotometer (a) front view, (b) top view, (c) liquid sample holder, (d) solid sample holder and (e) spring arrangement to hold sample holder in the hole at the bottom of the Integrating sphere

Data processing was carried out through a combined software approach involving FL-Solutions for acquisition, a custom Scilab-based program for baseline correction, numerical integration, yield computation and visualization. Manual PLQY estimation was also performed using Origin Pro, confirming the numerical accuracy of the automated workflow.

Synthesis of Phosphors for PLQY Study: All the phosphors studied in this paper are synthesized by solid state diffusion method based on the principle of diffusion-controlled reactions between solid reactants at elevated temperatures. When heated, ions migrate across particle boundaries, leading to interdiffusion and formation of a new crystalline phase. The reaction rate depends on temperature, particle size, and the degree of mixing of the precursors ([14]). The stoichiometric amounts of precursors used for the synthesis of the phosphors with the optimum dopant concentrations are represented in the chemical reaction (Table 1). The photoluminescence (PL) excitation and emission spectra of the synthesized phosphors were found to be in good agreement with those reported in the literature, confirming the reliability of the prepared samples.

Table 1. The stoichiometric amounts of precursors used for the synthesis of the phosphors

$Y_2O_3:Eu^{3+}$	$1.88\ Y(NO_3)_3 + 0.12\ Eu(NO_3)_3 \quad Y_{1.88}Eu_{0.12}O_3$ + Gases
$YVO_4:Eu^{3+}$	$0.95\ Y(NO_3)_3 + 0.05\ Eu(NO_3)_3 + NH_4VO_3 \quad Y_{0.95}Eu_{0.05}VO_4$ + Gases
$YBO_3:Eu^{3+}$	$0.97\ Y(NO_3)_3 + 0.03\ Eu(NO_3)_3 + 0.2\ NH_4B_5O_8 \quad Y_{0.97}Eu_{0.03}BO_3$ + Gases
$BaMgAl_{10}O_{17}:Eu^{2+}$	$0.92\ BaCO_3 + 5\ Al_2O_3 + MgO + 0.08\ Eu(NO_3)_3 \quad Ba_{0.92}Eu_{0.08}MgAl_{10}O_{17}$ + Gases
$LaBaB_9O_{16}:Tb^{3+}$	$0.96\ La(NO_3)_3 + BaCO_3 + 9\ H_3BO_3 + 0.01\ Tb_4O_7 \quad La_{0.96}Tb_{0.04}BaB_9O_{16}$ + Gases

Steps involved in the synthesis of the phosphors by solid state diffusion method are depicted in the (AI generated) Fig. 2. The process involves repeated cycles of heating and grinding to achieve the phase purity and high degree of crystallinity in the synthesized phosphors. Extra step is performed for reduction of Eu^{3+} to Eu^{2+} in BAM, by heating it in reduced atmosphere created using burning of activated charcoal ([15]).

Figure 2: Steps involved in the synthesis of the phosphors by solid state diffusion method

The X-ray diffraction analysis was carried out on Rigaku Miniflex II X-ray Diffractometer. The PL excitation and emission spectra was obtained from the Hitachi F7000 fluorescence spectrophotometer. The PLQY was measured using indigenously designed QY set up.

RESULTS AND DISCUSSION

X-ray diffraction analysis: The crystallinity and phase purity of the synthesized phosphors were verified using X-ray diffraction (XRD) analysis. The obtained XRD patterns (Fig. 3 (a), (b), (c), (d) and (e)) showed close agreement with the corresponding ICDD/JCPDS reference files of the host materials, confirming the successful formation of the desired phase.

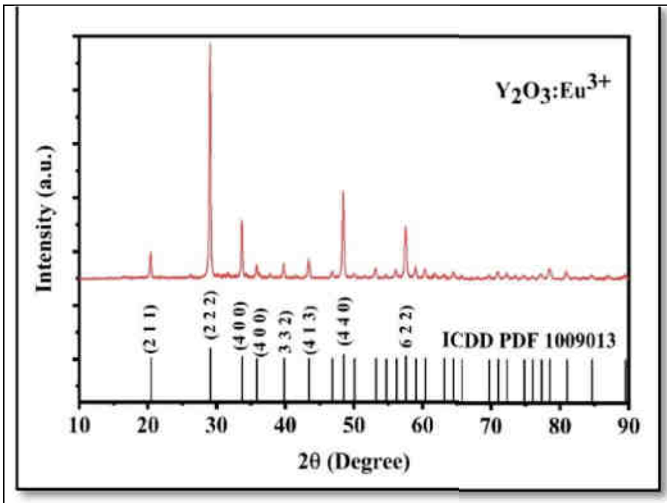


Figure 3(a). XRD patterns of $Y_2O_3:Eu^{3+}$

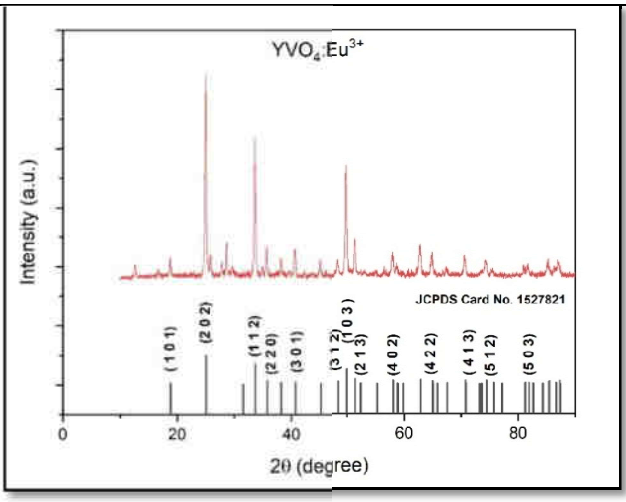


Figure 3(b). XRD patterns of $YVO_4:Eu^{3+}$

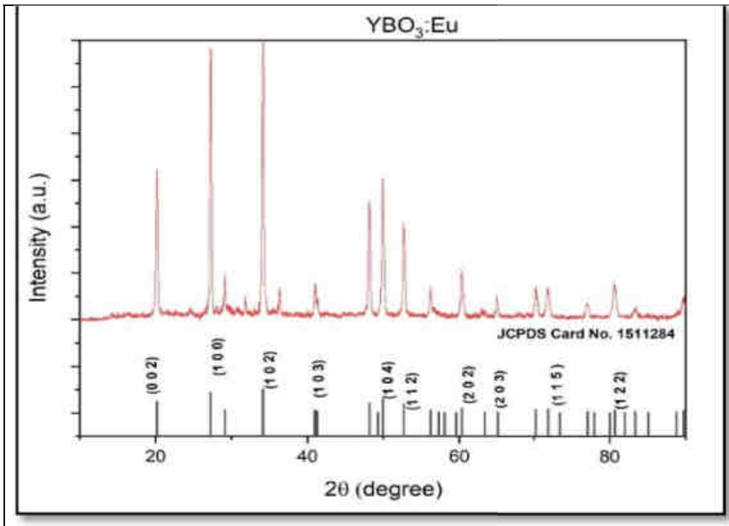


Figure 3(c): XRD patterns of $YBO_3:Eu^{3+}$

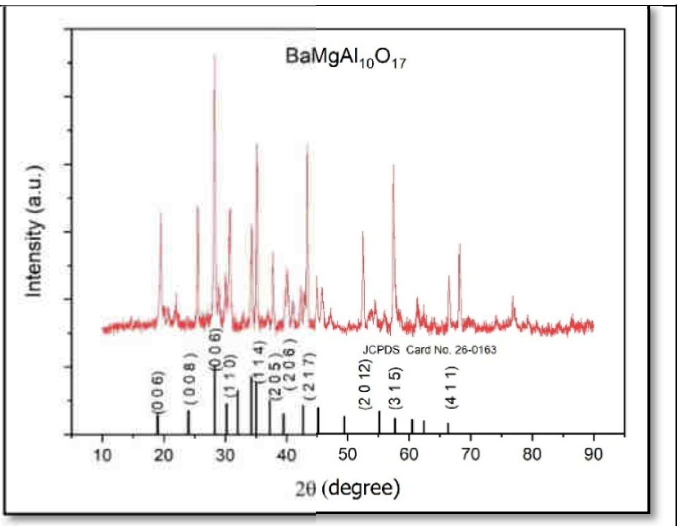


Figure 3(d): XRD patterns of $BaMgAl_{10}O_{17}:Eu^{2+}$

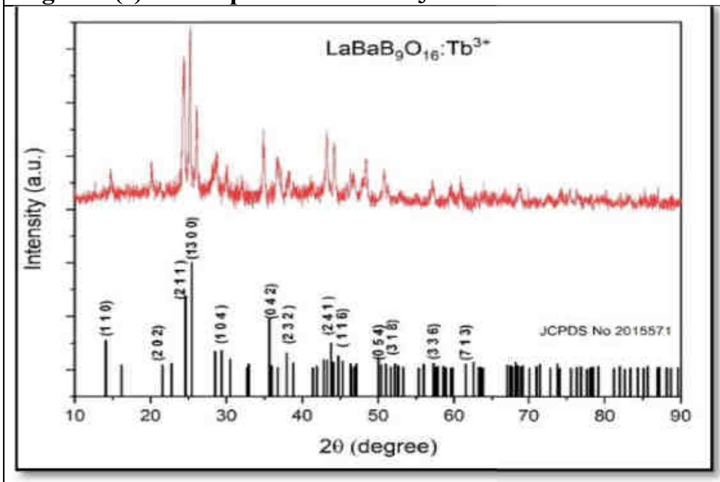
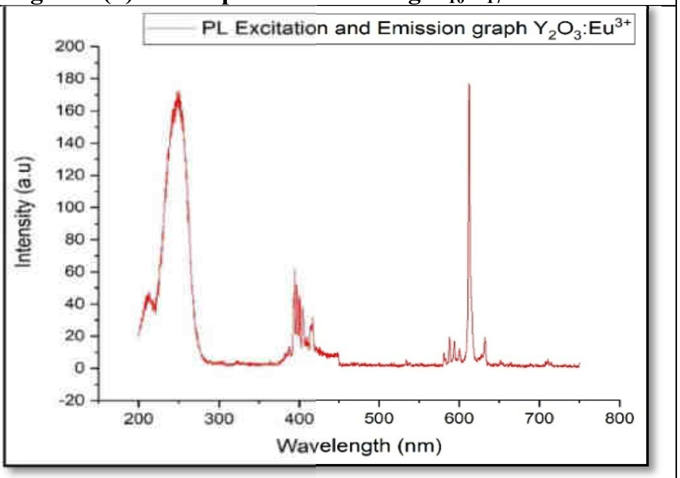
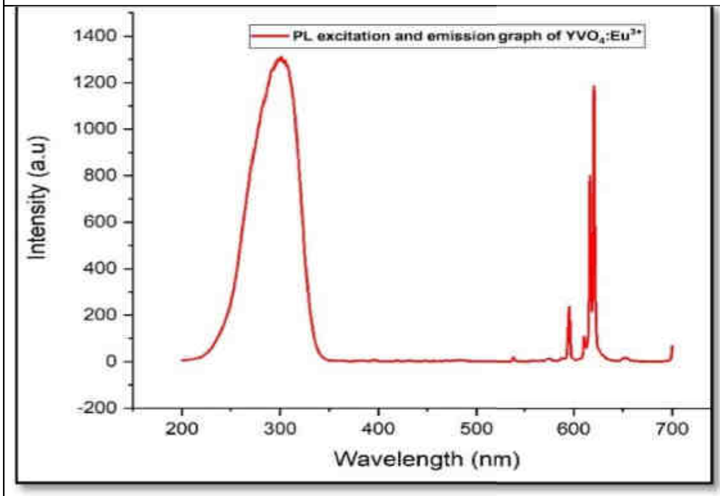


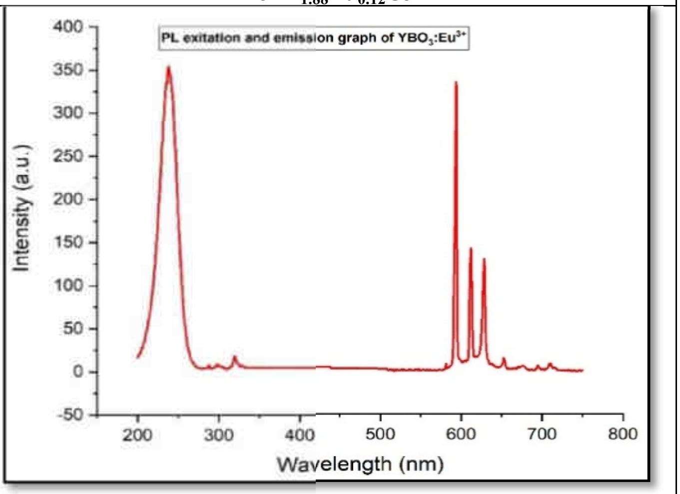
Figure 3(e): XRD patterns of $LaBaB9O_{16}:Tb^{3+}$



Figures 4: Photoluminescence excitation and emission spectra of $Y_{1.88}Eu_{0.12}O_3$

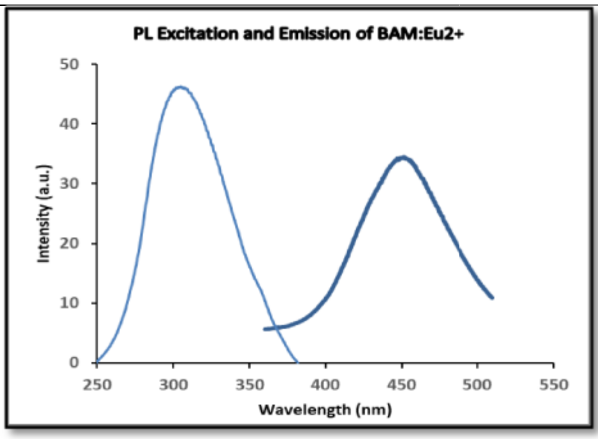


Figures 5: Photoluminescence excitation and emission spectra of $Y_{0.95}Eu_{0.05}VO_4$

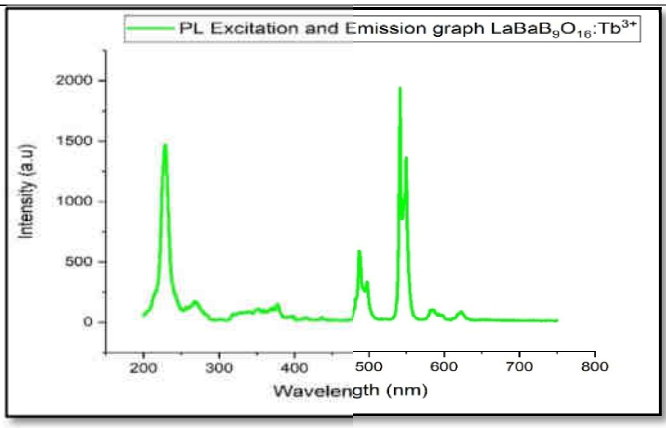


Figures 6: Photoluminescence excitation and emission spectra of $Y_{0.97}Eu_{0.03}BO_3$

Photoluminescence (PL) Excitation and Emission spectrums: The PL excitation and emission spectrum monitored at 254 nm excitation wavelength for the Eu^{3+} activated Y_2O_3, YVO_4 and YBO_3 are depicted in the Fig. 4, Fig. 5 and Fig. 6. The emissions peaks in orange-red region, in all three cases corresponds to characteristics transitions $^5D_0$ – ^7F_J of Eu^{3+} with difference in intensities. The variation in PL emission intensity of Eu^{3+} activated Y_2O_3, YVO_4 and YBO_3 is mainly due to differences in host lattice structure, site symmetry, phonon energy, and host-to-Eu^{3+} energy transfer efficiency. YVO_4 generally shows stronger emission because the VO_4^{3-}group efficiently absorbs UV energy and transfers it to Eu^{3+} ions. Differences in crystal field environment and local symmetry around Eu^{3+} ions also affect the intensity of characteristic $^5D_0$ – ^7F_J transitions. Additionally, variation in crystallinity, defects, and non-radiative losses among the hosts contributes to the observed intensity differences ([16], [17], [18], [19]). In $Ba_{0.92}Eu_{0.08}MgAl_{10}O_{17}$, Eu^{2+} ions substitute Ba^{2+} sites due to similar ionic radii and valency. The crystal field environment around Eu^{2+} affects the position and width of the emission band. Unlike Eu^{3+}, Eu^{2+} exhibits broad-band emission because the 5d levels are strongly influenced by the surrounding lattice. In this case broad-band emission centered around 450 nm at 300 nm excitation ([20]). As usual, UV absorption and excitation spectroscopic features in some Tb^{3+} activated materials are due to transitions from the lower levels of the $4f^n$ single configuration to the levels of the $4f^{n-1}5d$ mixed configuration of Tb^{3+}. The excitation band peaking at 237 nm associated with spin allowed $4f^8$-$4f^75d^1$ transitions of Tb^{3+} ions. $La_{0.96}Tb_{0.04}BaB_9O_{16}$ phosphor emits intense green colour, which corresponds to a group of $^5D_4 - {}^7F_J$ (J= 6, 5, 4, 3) transitions of Tb^{3+} ion with the predominant $^5D_4 - {}^7F_5$ transition peaking at 544 nm ([21]).



Figures 7: Photoluminescence excitation and emission spectra of $Ba_{0.92}Eu_{0.08}MgAl_{10}O_{17}$



Figures 8: Photoluminescence excitation and emission spectra of $La_{0.96}Tb_{0.04}BaB_9O_{16}$

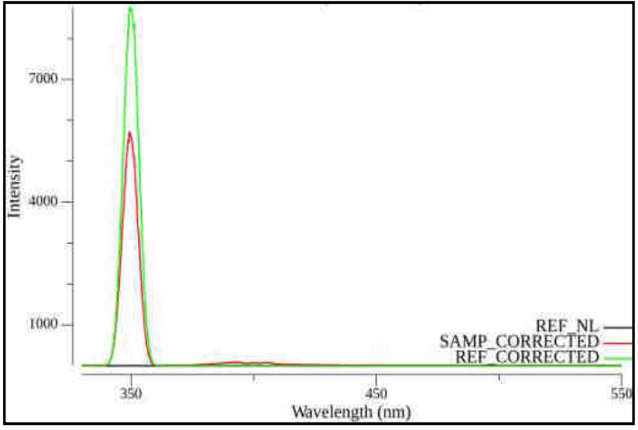


Figure 9. The overlayed spectrums from the integrating sphere with and without the sodium salicylate sample

Photoluminescence Quantum Yield Study

PLQE of sodium salicylate: The internal PLQY of sodium salicylate was measured as a standard material to validate the custom integrating-sphere setup. Fig. 9 shows the overlayed spectrums from the integrating sphere with and without the sodium salicylate sample. The PLQY of the sample is given by the emission area (under the red curve, after the excitation curve) divided by the light absorbed area (difference of areas under the green and red curves centered at 350 nm wavelength). The measured quantum yield was found to be 39.48%. To cross-verify this result, the PLQY of the same sample was also measured using the commercial FLS1000 spectrometer, yielding a value of 45.97%, which lies within the expected range for internal quantum yield measurements. Since sodium salicylate is known to exhibit a quantum yield in the range of 40–50% ([22]), the value obtained using the custom setup confirms the reliability and accuracy of the developed integrating-sphere system for internal PLQY measurements. Similar method is used for measuring the PLQY values with the indigenously designed integrating sphere coupled with fluorescence spectrophotometer for synthesized phosphors. Table 2 shows that the PLQY values measured using this setup are in close agreement with the values measured using FLS-1000 spectrometer.

Table 2: Comparison of PLQY values

Phosphor	PLQY measured using our set up	PLQY measured using FLS-1000 spectrometer
Sodium Salicylate	39.48%	45.9%
$Y_{1.88}Eu_{0.12}O_3$	55.76%	57.60%
$Y_{0.95}Eu_{0.05}VO_4$	41.71%	43.50%
$Y_{0.97}Eu_{0.03}BO_3$	49.58%	52.59%
$Ba_{0.92}Eu_{0.08}MgAl_{10}O_{17}$	72.03%	74.51%
$La_{0.96}Tb_{0.04}BaB_9O_{16}$	39.58%	41%

CONCLUSIONS

An indigenously designed integrating sphere coupled with a modified Hitachi F-7000 fluorescence spectrophotometer was successfully developed and utilized for the measurement of photoluminescence quantum yield (PLQY) of different tri-colour phosphors. The synthesized phosphors, namely $Y_2O_3{:}Eu^{3+}$, $YVO_4{:}Eu^{3+}$, $YBO_3{:}Eu^{3+}$, $BaMgAl_{10}O_{17}{:}Eu^{2+}$ and $LaBaB_9O_{16}{:}Tb^{3+}$, prepared by the solid-state diffusion method, exhibited phase purity and characteristic luminescence behaviour. The integrating sphere coated with highly reflective $BaSO_4$ enabled efficient collection of both excitation and emission photons, leading to reliable PLQY measurements for powder phosphor samples. Calibration using sodium salicylate confirmed the proper functioning and accuracy of the developed setup. The PLQY values obtained using the indigenous system showed close agreement with those measured using the commercial FLS-1000 spectrometer, validating the performance of the developed instrument. The study demonstrates that the proposed low-cost and easily adaptable setup can serve as an effective alternative for accurate PLQY measurements of luminescent materials in research laboratories.

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