



STRUCTURAL AND LUMINESCENCE PROPERTIES OF Sm³⁺/Eu³⁺ CO-DOPED 2CaO-B₂O₃-P₂O₅ PHOSPHORS: ENERGY TRANSFER MECHANISM AND ENHANCEMENT OF RED LUMINESCENCE BY TEMPERATURE

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ABSTRACT

Sm³⁺/Eu³⁺ co-doped calcium borophosphate phosphors were synthesized by solid state reaction method. 2CaO-B₂O₃-P₂O₅: Sm³⁺/Eu³⁺ co-doped phosphors were characterized by XRD, SEM, DRS, FT-IR, ³¹P solid state NMR, excitation, photoluminescence (PL) and decay profiles. XRD profiles showed that the prepared phosphors exhibit a hexagonal phase in crystal structure and SEM results showed that the particles are more irregular morphologies. FT-IR spectrum shows prominent band at 1029 cm⁻¹ which is due to the asymmetric stretching vibrations of [PO₄]³⁻ tetrahedrons. From ³¹P NMR spectra of Sm³⁺/Eu³⁺ co-doped 2CaO-B₂O₃-P₂O₅ (CBP) phosphors, it was observed that the chemical shifts are located in the positive frequency region indicating the presence of mono-phosphate complexes Q⁰-(PO₄³⁻). Photoluminescence spectra of Sm³⁺/Eu³⁺ co-doped CBP phosphors show increase in emission intensity of Eu³⁺ ion due to co-doping with Sm³⁺ ions due to energy transfer process. The energy transfer mechanism between Sm³⁺ and Eu³⁺ ions has been clearly explained. The energy transfer process has been evidenced by lifetime decay profiles. From CIE chromaticity diagram, pure red color emission was observed in 0.6Sm³⁺/0.6Eu³⁺ co-doped CBP phosphors. These results suggest that the prepared phosphors are potential for red luminescent optical materials.

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INTRODUCTION

Currently, great interest in phosphors has resulted in rapid developments in the solid state lighting sources, including LED display and illumination technologies. The applications of white light from combined blue emission of blue chip and yellow light emitted by YAG:Ce³⁺ have been limited in many fields due to the lack of red light harmonic [1, 2]. Thus, the lack of red phosphors for long term constitutes a serious obstacle to the development of advanced LED-based technologies. Therefore, one way of developing practical white LEDs is to search for both high efficiency and quality red phosphors to improve color rendering [3]. A series of red phosphors have been investigated using the compounds such as nitrides, silicates, sulfides, and molybdates et al. [4, 5].

Rare earth (RE) ion doped phosphors have considerable attention for diverse applications such as solid state lasers, waveguides and optical fibers. RE ions have 4f-4f and 4f-5d electronic transitions, which exhibit sharp emission lines from the ultraviolet (UV) to the infrared (NIR) regions [6]. Among RE ions, Eu³⁺ has good energy level structure and high luminescence efficiency of ⁵D₀→⁷F₂ transition, which is very sensitive to changes in chemical surroundings of the Eu³⁺ ion. On the other hand for Sm³⁺ ion, the ionic radius is nearly

same compared to Eu³⁺, gives a red color emission, sometimes shifted to longer wavelengths compared to Eu³⁺ [7]. It's emitting level; ⁴G_{5/2} gives relatively high quantum efficiency and also shows different emission quenching [8].

The energy transfer between two RE ions co-doped in phosphors has been studied in many systems. The reason is that the sensitized luminescence is not only of interest for applications but also for understanding the basic mechanisms involved [9-11]. In Sm³⁺ and Eu³⁺ co-doped phosphors, the excitation wavelength range of Eu³⁺ emission is broadened owing to the energy transfer from Sm³⁺ to Eu³⁺ [12]. Many researchers have discussed the phenomena of radiative and non-radiative transitions in Sm³⁺/Eu³⁺ co-doped phosphors and succeeded in realizing energy transfer in co-doped phosphors. Matwa et al. studied photoluminescence and energy transfer of Eu³⁺ and Sm³⁺ single-doped and co-doped BaB₈O₁₃ phosphors [13]. Hachani et al. discussed energy transfer between Sm³⁺ and Eu³⁺ in YPO₄, LaP₅O₁₄ and LaP₃O₉ phosphates: potential quantum cutters for red emitting phosphors [14]. Wembo et al. studied luminescent properties and energy transfer mechanism of NaGd(MoO₄)₂:Sm³⁺/Eu³⁺ phosphors [15]. Fengwen et al. reported luminescence and red long after glow investigation of Eu³⁺/Sm³⁺ co-doped CaWO₄ phosphor [16].

CBP phosphors show significance for white LED (WLED) applications and some research work on Sm³⁺ and Eu³⁺ singly

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doped phosphors was done recently [17]. In the present paper, an attempt is made to report the influence of different concentrations of Sm³⁺ ion on the Eu³⁺ ion photoluminescence in Sm³⁺/Eu³⁺ co-doped CBP phosphors. Changes in spectroscopic properties as a function of concentration were observed and discussed in terms of local environment of Sm³⁺ and Eu³⁺ ions. In the present work, the structural, thermal and luminescence properties of new red emitting CBP phosphor co-doped with Sm³⁺/Eu³⁺ ions was reported. Eu³⁺ has been taken as acceptor ion and Sm³⁺ as donor ion. Surprisingly, four common excitation bands are observed in Eu³⁺ and Sm³⁺ consisting phosphors, with these excitations the emission performances have been observed and the energy transfer phenomenon has been explained by increasing the donor (Sm³⁺) concentration. The energy transfer mechanism has clearly explained between Sm³⁺ and Eu³⁺ ions with lifetime decay profiles.

Experimental

Using solid state reaction method, Sm³⁺/Eu³⁺ co-doped 2CaO-B₂O₃-P₂O₅ (CBP) phosphors ((1.4-x)CaO-B₂O₃-P₂O₅-xSm₂O₃-0.6Eu₂O₃ (where x=0.2, 0.4, 0.6, 0.8 and 1.0 mol%)) were prepared using raw materials, CaCO₃, H₃BO₃, NH₄H₂PO₄, Sm₂O₃ and Eu₂O₃ with 99.9 % purity. The batch of 10 gm was weighed as per the composition given above. These materials were taken in an agate mortar and after ground they are preheated at 650 °C for 2 h in porcelain crucible and then cooled down to room temperature. The preheated batches were again heated at 950 °C for 4 h in an electrical furnace. The obtained phosphor powders were pulverized for further characterization and analysis.

Phase purity of Sm³⁺/Eu³⁺ co-doped CBP phosphors were characterized by X-ray diffraction (XRD) with Philips PW 1050 instrument, using Ni filtered Cu tube with K α radiation. SEM images of Sm³⁺/Eu³⁺ co-doped phosphors were taken by Carl Zeiss EVO-MA15 scanning electron microscope. The UV-visible diffuse reflectance spectra (DRS) were measured for undoped and co-doped Sm³⁺/Eu³⁺ phosphors using JASCO V570 UV-Vis-NIR spectrometer. The thermal decomposition of CBP phosphors was performed by thermo gravimetry differential scanning calorimetry (TG-DSC; Model NETZSCH STA 449C, Selb, Germany) in air atmosphere with a heating rate 10 °C/min. The fourier transform infrared spectra were recorded at room temperature with 4 cm⁻¹ spectral resolution between 400 and 4000 cm⁻¹ by a BRUKER FTIR spectrometer. Solid state ³¹P NMR spectra were obtained at 400 MHz using a JOEL ECX400 DELTA2 NMR spectrometer with a 4 mm probe. Excitation, photoluminescence, decay lifetimes and quantum yield were acquired using FLS-920 Edinburgh-fluorimeter (Horiba FL3-22iHR320).

RESULTS AND DISCUSSION

X-ray diffraction spectra (XRD)

Sm³⁺/Eu³⁺ co-doped XRD patterns of CBP phosphors were measured in the range, 10°-80° and were shown in Fig. 1. By comparing the obtained XRD patterns of present co-doped phosphors with host pattern, all samples showed the same diffraction peaks reported in JCPDS card No: 0-018-0283 and are in good agreement. It was also found that, with the increase of doping concentrations, showed same JCPDS

structure, thus implying the formation of pure crystalline phase of CBP: Sm³⁺/Eu³⁺ co-doped phosphors. Generally, borophosphate crystal contains CaO₆, BO₄ and PO₄ polyhedron, generating three-dimensional composite frame work with large tunnels inside [18]. In CBP crystal, insertion of Sm³⁺/Eu³⁺ took in the nine-fold coordinate calcium site due to similar ionic radii of Sm³⁺, Eu³⁺ and Ca²⁺ ions. Sm³⁺/Eu³⁺ ions occupy a site in nine coordinated large polyhedron occurring in tortuous vertical columns formed by BO₄ and PO₄ tetrahedra [19]. A well crystallized hexagonal symmetry with group space P6cc (184) was observed from figure 1.

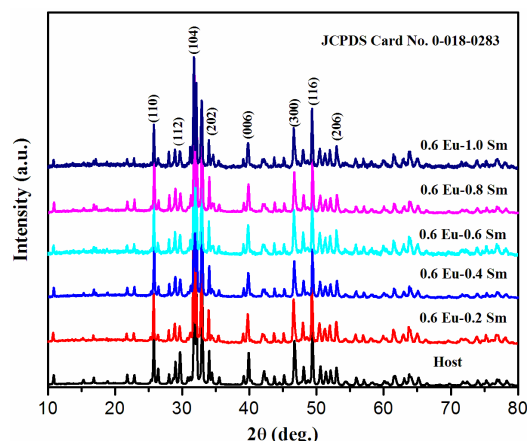


Fig 1 XRD profiles of Sm³⁺/Eu³⁺ co-doped 2CaO-B₂O₃-P₂O₅ phosphors.

SEM morphology

SEM images are shown in Fig. 2 (a-e) for different concentrations of Sm³⁺/Eu³⁺ co-doped CBP phosphors (a:0.2Sm/0.6Eu, b:0.4Sm/0.6Eu, c:0.6Sm/0.6Eu, d:0.8Sm/0.6Eu, e:1.0Sm/0.6Eu). From the figure, it was found that the particle size has increased as the Sm³⁺ concentration increased from 0.2 to 1.0 mol% with 0.6 mol% of Eu³⁺, and the particles

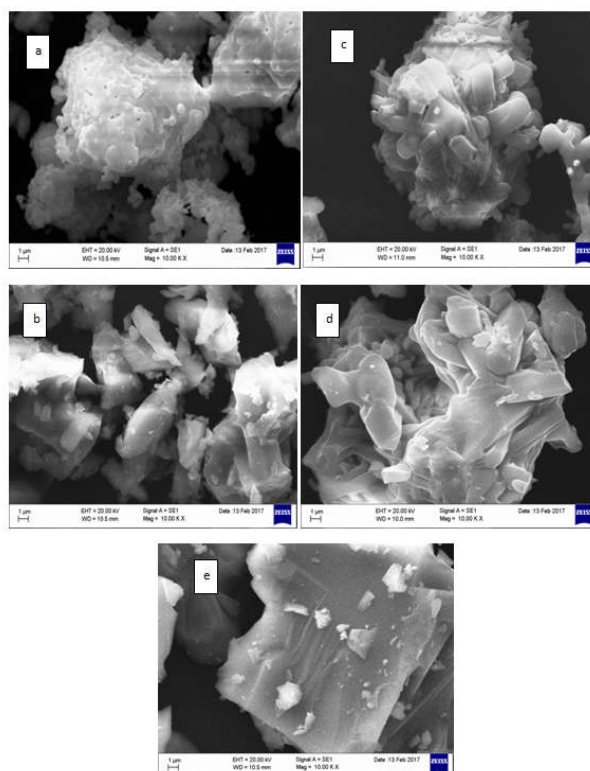


Fig 2 SEM images of Sm³⁺/Eu³⁺ co-doped 2CaO-B₂O₃-P₂O₅ phosphors.

have more irregular morphologies due to $\text{Sm}^{3+}/\text{Eu}^{3+}$ incorporation. In the present work, the sizes of the particles are between few microns and several tens of microns and they are suitable for use in the WLEDs applications.

Diffuse reflection spectra (DRS)

The diffuse reflection spectra (DRS) of $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped and undoped CBP phosphors in the visible range are shown in Fig. 3. From the reflection spectra it was observed that undoped and co-doped spectra have similar in shape indicating that the effect of co-doping of $\text{Sm}^{3+}/\text{Eu}^{3+}$ ions on the reflection spectra is negligible. The band gaps were obtained from the diffuse reflection spectra of both the phosphors using the formula given in ref. [20] and these values are 4.72 eV and 4.78 eV for undoped and $0.6\text{Sm}^{3+}/0.6\text{Eu}^{3+}$ co-doped phosphors, respectively.

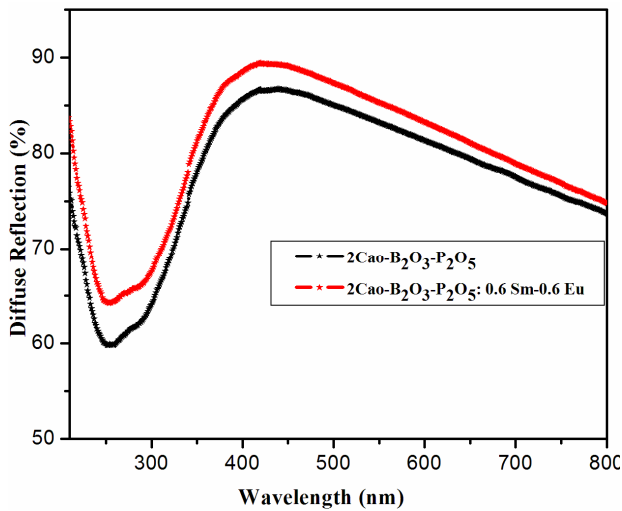


Fig 3 DRS spectra of $2\text{CaO}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5$ and $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped $2\text{CaO}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5$ phosphors.

FT-IR spectra

Fourier transform infrared spectrum of present phosphor was measured in the range $500\text{-}3800\text{ cm}^{-1}$ and shown in Fig. 4.

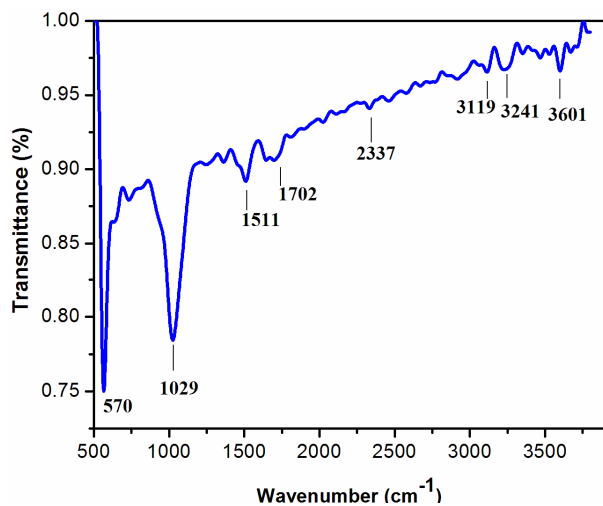


Fig 4 FT-IR spectrum of $2\text{CaO}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5$ phosphor

From the figure, it is observed that the spectrum consists of eight bands centered at ~ 570 , ~ 1029 , ~ 1511 , ~ 1702 , ~ 2337 , ~ 3119 , ~ 3241 and $\sim 3601\text{ cm}^{-1}$. The bands at 570 , 1029 and 1511 cm^{-1} are attributed to the $(\text{PO}_4)^{3-}$ vibrational bands [21] and correspond to asymmetric stretching vibrations. The band

at 570 cm^{-1} is due to O-P-O asymmetric bending vibrations, while the band at 1029 cm^{-1} is due to O-P-O asymmetric stretching vibrations of $[\text{PO}_4]^{3-}$ tetrahedrons [21]. The band at 1511 cm^{-1} is attributed to the asymmetric stretching of C-O bonds [22]. The bands observed between the energy range $1702\text{-}3601\text{ cm}^{-1}$ are assigned to the H-O-H bending vibrational mode of the H_2O molecule.

^{31}P solid state NMR spectra:

^{31}P solid state NMR is one of the important tool in characterizing the structures of phosphate-type phosphors due to the chemical shifts being sensitive to the phosphorus environment. The phosphate bonding is explained through Q^n species, where the superscript n refers to the number of bridging oxygens per tetrahedron [23]. ^{31}P NMR spectra of CBP and $\text{CBP}:0.6\text{Sm}^{3+}/0.6\text{Eu}^{3+}$ phosphors are shown in Fig. 5. In the present work, the obtained spectra (from Fig. 5) are a single, symmetric Gaussian peak and position of line for CBP and $\text{CBP}:0.6\text{Sm}^{3+}/0.6\text{Eu}^{3+}$ phosphors are at $+3.9\text{ ppm}$. This value indicates the presence of mono-phosphate complexes $\text{Q}^0\text{-(PO}_4^{3-})$ (phosphate tetrahedral with zero bridging oxygens) in both these phosphors network [24].

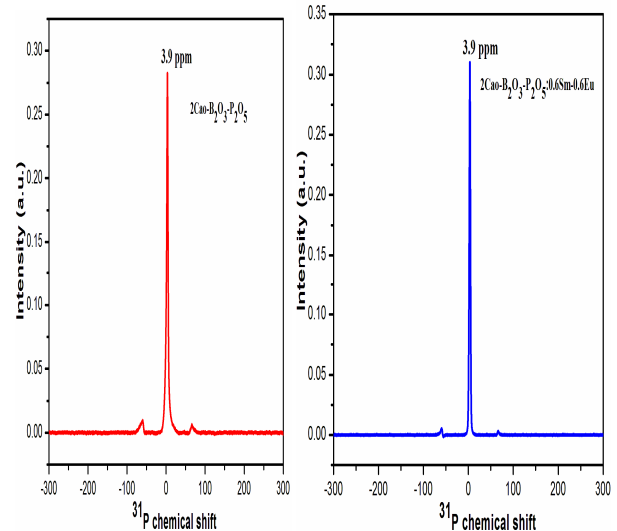


Fig 5 ^{31}P NMR spectra of $2\text{CaO}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5$ and $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped $2\text{CaO}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5$ phosphors.

Photoluminescence spectra

Fig. 6 shows the comparison of excitation spectra of Eu^{3+} singly doped [17] and $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped phosphors monitoring the Eu^{3+} emission wavelength at 613 nm .

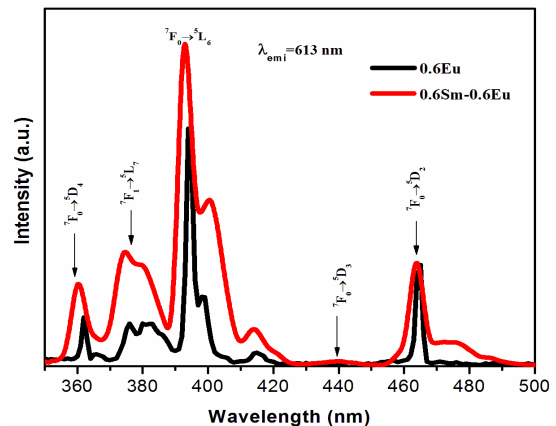


Fig 6 Excitation spectra of Eu^{3+} doped and $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped $2\text{CaO}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5$ phosphors.

The excitation spectra of Eu³⁺ doped phosphors display several excitation bands associated with transitions from the ⁷F₀ and ⁷F₁ states to ⁵D₄, ⁵L₇, ⁵L₆, ⁵D₃ and ⁵D₂ states as reported in previous paper [17]. It is observed that the spectrum of co-doped Sm³⁺/Eu³⁺ phosphor also shows the same excitation bands which are observed for Eu³⁺ doped phosphors.

The photoluminescence spectra of Sm³⁺/Eu³⁺ co-doped CBP phosphors excited with 393 nm for different Sm³⁺ concentrations are shown in Fig. 7.

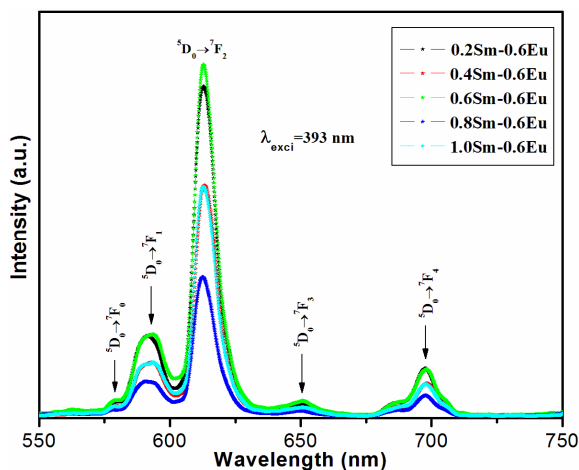


Fig 7 Emission spectra of Sm³⁺/Eu³⁺ co-doped 2CaO-B₂O₃-P₂O₅ phosphors for different concentrations.

This emission spectra consist five emission bands at 578, 592, 613, 650 and 698 nm, related to the ⁵D₀→⁷F_J (J= 0, 1, 2, 3 and 4) transitions respectively. From the figure, it is observed that the intensity of emission peaks of Eu³⁺ (613 nm) increased with Sm³⁺ concentration upto 0.6 mol% and then decreased with increasing Sm³⁺ concentration. The optimum concentration of Sm³⁺ ion in Sm³⁺/Eu³⁺ co-doped CBP phosphor was 0.6 mol% and it can be called concentration effect. In general, the sensitizer (donor) emission intensity decreases and activator emission intensity increases if energy transfer suited from sensitizer to activator (acceptor) in co-doped phosphors [25].

The energy transfer phenomenon from Sm³⁺ to Eu³⁺ can be explained using the energy level diagram as shown in Fig. 8.

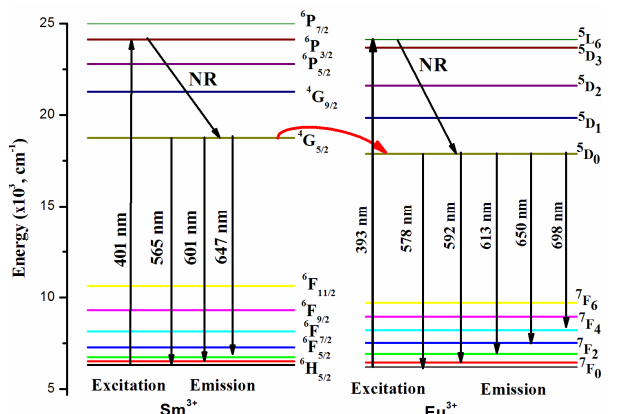


Fig 8 Partial energy level scheme of energy transfer from Sm³⁺ to Eu³⁺ in 2CaO-B₂O₃-P₂O₅ phosphors.

From the figure, under the excitation of 401 nm, an electron in the ground state, ⁶H_{5/2} of Sm³⁺ are stimulated to the excited

state ⁶P_{3/2} and then relaxes finally to the ⁴G_{5/2} level non-radiatively. The energy in the ⁴G_{5/2} level of Sm³⁺ is transferred to the ⁵D₀ level of Eu³⁺ due to resonance (the ⁴G_{5/2} energy level of Sm³⁺ is approximately 600 cm⁻¹ higher than the ⁵D₀ level of Eu³⁺) between the two energy levels. The energy transfer from Sm³⁺ to Eu³⁺ is almost irreversible, because the ⁴G_{5/2} level in Sm³⁺ is higher than the ⁵D₀ level in Eu³⁺, and the probability of emitting phonons in Sm³⁺:⁴G_{5/2}→Eu³⁺:⁵D₀ is higher than that of capturing phonons for Eu³⁺:⁵D₀→Sm³⁺:⁴G_{5/2} process. The resonance energy transfer increased the population of Eu³⁺: ⁵D₀ level and it was due to the co-doping with Sm³⁺ caused the sensitization of Eu³⁺ emission under certain excitation conditions and lead to the expansion of the exciting range in Eu³⁺ luminescence.

Effect of thermal treatment on luminescence spectra

Sm³⁺/Eu³⁺ co-doped present phosphor at optimum concentration are treated with different temperatures in the range 900 °C – 1100 °C with the step size of 50 °C to study the effect of temperature on Eu³⁺ emission intensity. The measured emission spectra are shown in Fig. 9. From the figure it was observed that the Eu³⁺ emission intensity increased from 900 °C to 950 °C then decreased upto 1050 °C and further increased at 1100 °C. The Eu³⁺ emission intensity was maximum at 1100 °C and minimum at 900 °C. The splitting of magnetic dipole (⁵D₀→⁷F₁, 592 nm) and electric dipole (⁵D₀→⁷F₄, 698 nm) transitions is observed, for the 950 °C and 1100 °C temperatures. The splitting of these transitions might be due to change in the chemical environment surrounding and local environment of Eu³⁺ ions [26].

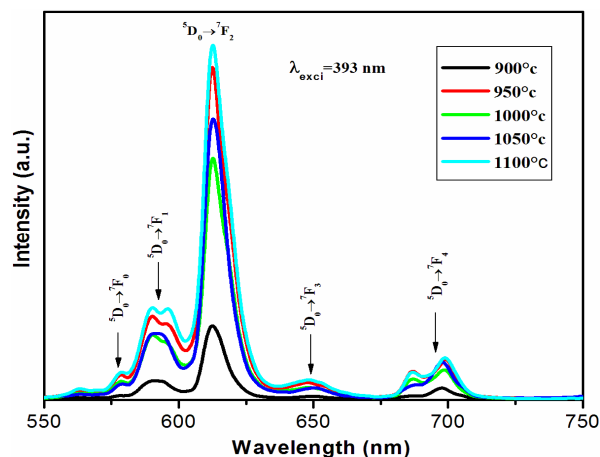


Fig 9 Emission spectra of 0.6Sm³⁺/0.6Eu³⁺ co-doped 2CaO-B₂O₃-P₂O₅ phosphors at different temperatures.

Fluorescence decay

The fluorescence lifetime decay profiles of Sm³⁺/Eu³⁺ co-doped CBP phosphor were measured with excitation wavelength 393 nm and emission wavelength 613 nm and are shown in Fig. 10. From the figure, it is noticed that the decay profiles exhibit non-exponential nature. The lifetimes (τ_{meas}) of the excited state, ⁵D₀ of Eu³⁺ are obtained in this Sm³⁺/Eu³⁺ co-doped CBP phosphors and are presented in Table 1. From the table, it is observed that the lifetime is higher (2.50 ms) for 0.6Sm³⁺/0.6Eu³⁺ co-doped phosphor than the remaining phosphors and also higher than Eu³⁺ singly doped CBP phosphor (2.30 ms) [17] indicating the energy transfer between the acceptor and donor ions. In the present work, the

concentration of Sm^{3+} (donor) ions per Eu^{3+} (acceptor) ion was found more, therefore the average distance between the Eu and Sm ions has decreased.

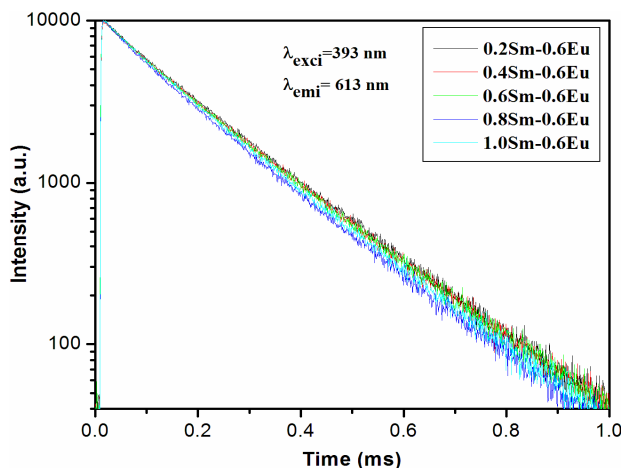


Fig 10 Decay curves of $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped $2\text{CaO}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5$ phosphors.

Table 1 Measured lifetimes (τ_{cal} , ms), absolute quantum efficiencies (Q_E , %) and color coordinates of $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped $2\text{CaO}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5$ phosphor.

Formula	τ_{meas}	Q_E	Color coordinates	
			x	y
$2\text{Ca}_{0.9}\text{O}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5:0.2\text{Sm}^{3+}/0.6\text{Eu}^{3+}$	2.21	97.22	0.59	0.30
$2\text{Ca}_{0.8}\text{O}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5:0.4\text{Sm}^{3+}/0.6\text{Eu}^{3+}$	2.25	75.18	0.62	0.33
$2\text{Ca}_{0.7}\text{O}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5:0.6\text{Sm}^{3+}/0.6\text{Eu}^{3+}$	2.50	59.38	0.65	0.32
$2\text{Ca}_{0.6}\text{O}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5:0.8\text{Sm}^{3+}/0.6\text{Eu}^{3+}$	2.32	42.50	0.63	0.31
$2\text{Ca}_{0.5}\text{O}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5:1.0\text{Sm}^{3+}/0.6\text{Eu}^{3+}$	2.29	28.02	0.60	0.32

Energy migration took place due to electronic multipole-multipole interactions and as a result, decay profiles exhibited non-exponential nature. The absolute quantum efficiencies (AQE) of $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped CBP phosphors were measured under excitation 393 nm with the fluorescence spectrometer using an integrating sphere and the AQE values are presented in Table 1. From the table, it is observed that AQE values have decreased with increasing Sm^{3+} concentration in $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped CBP phosphors.

Color chromaticity co-ordinates

The CIE chromaticity diagram of $0.6\text{Sm}^{3+}/0.6\text{Eu}^{3+}$ co-doped CBP phosphor under the excitation of 393 nm was shown in Fig. 11.

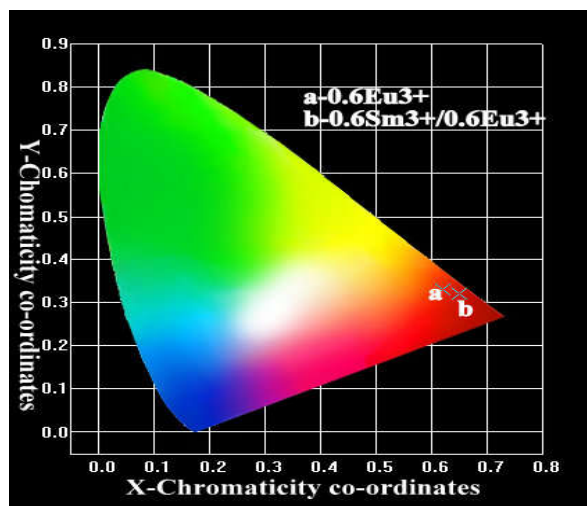


Fig 11 Color chromaticity diagram of Eu^{3+} doped and $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped $2\text{CaO}\cdot\text{B}_2\text{O}_3\cdot\text{P}_2\text{O}_5$ phosphors.

The chromaticity coordinates of present phosphor with co-doped $\text{Sm}^{3+}/\text{Eu}^{3+}$ ions for different concentrations were calculated and are presented in Table 1. The emitting color of Eu^{3+} (0.6 mol%) singly doped CBP phosphor [17] was in pale red region, and the corresponding color coordinates are 0.62 and 0.33. By doping 0.6Sm^{3+} into the CBP phosphor, color coordinates shifted towards pure red region and the color coordinates was changed to 0.65 and 0.32. Hence, $0.6\text{Sm}^{3+}/0.6\text{Eu}^{3+}$ co-doped CBP phosphor may be used as a potential red emitting material.

CONCLUSIONS

$\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped calcium borophosphate phosphors were synthesized by solid state reaction method. XRD shows $\text{Sm}^{3+}/\text{Eu}^{3+}$ ions occupy a site in nine coordinated large polyhedron occurring in tortuous vertical columns formed by BO_4 and PO_4 tetrahedra. The SEM images showed the particles were more irregular morphologies due to $\text{Sm}^{3+}/\text{Eu}^{3+}$ incorporation. From the FT-IR spectrum, the prominent band at 1029 cm^{-1} was due to the asymmetric stretching vibrations of $[\text{PO}_4]^{3-}$ tetrahedrons. From ^{31}P NMR spectra of $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped calcium borophosphate phosphors, the component lines corresponding to the chemical shifts located in the positive frequency region indicated the presence of mono-phosphate complexes $\text{Q}^0\text{-(PO}_4^{3-})$. From the photoluminescence spectra of $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped CBP phosphors, enhancement of emission intensity of Eu^{3+} was observed due to co-doping with Sm^{3+} ions and due to energy transfer process. The energy transfer mechanism, from Sm^{3+} to Eu^{3+} was clearly explained. Eu^{3+} ion emission intensities have significantly been enhanced due to sensitizer effect of Sm^{3+} ions in the $\text{Sm}^{3+}/\text{Eu}^{3+}$ co-doped CBP phosphors. This phenomenon was clearly explained by partial energy level diagram. The energy transfer process was also evidenced by lifetime decay profiles. From CIE chromaticity diagram, pure red color emission was observed at $0.6\text{Sm}^{3+}/0.6\text{Eu}^{3+}$ doped CBP phosphors. These results suggest that the prepared phosphors are the potential red luminescent optical materials.

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