

# Photoluminescence properties of $\text{Eu}^{3+}$ , $\text{Ce}^{3+}$ doped $\text{LaPO}_4$ phosphors

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## ABSTRACT

Pure  $\text{LaPO}_4$  and  $\text{LaPO}_4$ : Eu (0.5 mol %) Ce (0.5 mol %) phosphors were synthesized by the solid-state reaction method. X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FT-IR), photoluminescence (PL) spectra and the particle size analysis were used to characterize these samples. The XRD results reveal that the synthesized  $\text{LaPO}_4$ :Eu (0.5 mol%) Ce (0.5 mol%) phosphors are well crystalline and assigned to the monoclinic structure with a main (120) diffraction peak. The calculated crystallite size of pure  $\text{LaPO}_4$  and  $\text{LaPO}_4$ :Eu, Ce phosphors were 67.6nm and 64nm respectively. Upon excitation at 254nm wavelength, the emission spectrum of pure  $\text{LaPO}_4$  phosphor emits a maximum intensity peak at 470 (blue) nm. In the emission spectrum of  $\text{LaPO}_4$ : $\text{Eu}^{3+}$  $\text{Ce}^{3+}$  phosphor, the low contributions of the red (613nm)  $^5\text{D}_0$ - $^7\text{F}_2$  emissions and the high intensity of the orange-red (589nm)  $^5\text{D}_0$ - $^7\text{F}_1$  emission results in high color purities. The most intense emissions appearing in the 580-620nm region is responsible for the strong orange-red luminescence observed in the Eu,Ce doped  $\text{LaPO}_4$  phosphor whose CIE colour coordinates are  $x = 0.57$  and  $y = 0.43$ . Thus the prepared phosphors can be used as an orange-red emitting material in the field of illuminations and display devices. Copyright © 2014 VBRI press.

**Keywords:** Photoluminescence; XRD; SEM; FTIR; phosphor; rare-earth ions; solid state reaction technique; CIE; particle size analysis.



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## Introduction

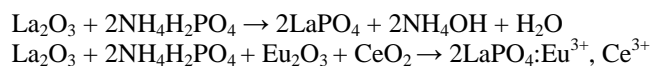
Rare-earth orthophosphates (REPO<sub>4</sub>) are a very interesting class of host lattices of activator ions due to their physico-chemical inercy (high insolubility, high thermal stability), thus providing durable phosphors [1]. The luminescent properties of rare-earth phosphates can be conferred by the presence of lanthanide (III) ions as activators due to their intense and narrow emission bands arising from f-f transitions, which are proper for the generation of individual colours in multiphosphor devices [2-4]. Thus, the red <sup>5</sup>D<sub>0</sub>-<sup>7</sup>F<sub>2</sub> (610nm), and blue <sup>1</sup>D<sub>2</sub>-<sup>3</sup>F<sub>4</sub> (450 nm) emissions of Eu<sup>3+</sup> and Ce<sup>3+</sup>, respectively, can be utilized for the design of novel phosphors. In recent years, lanthanide orthophosphates (LnPO<sub>4</sub>) have attracted much interest in the potential applications as phosphors, proton conductors, sensors, catalysts, ceramic materials, and heat-resistant materials [5, 6]. It is known that the LaPO<sub>4</sub> has a monoclinic phase of monazite structure crystallographically, wherein La<sup>3+</sup> ion is nine coordinated to oxygen atoms, four oxygens forming a distorted tetrahedron interpenetrating a quasiplanar pentagon formed by another five [7-10]. The La<sup>3+</sup> ion site in the monazite structure can be easily substituted by any other lanthanide ions. Many methods have been developed for the synthesis of LaPO<sub>4</sub> or doped LaPO<sub>4</sub> including solid-state reaction [11], combustion route, spray pyrolysis [12], coprecipitation and solvothermal route [13].

In this paper LaPO<sub>4</sub> and LaPO<sub>4</sub>:Eu<sup>3+</sup> (0.5 mol %) Ce<sup>3+</sup> (0.5 mol %) phosphors prepared by the solid state reaction method in air at 1200 °C, and their luminescent properties were studied. Optimization of the concentration of activator ions incorporated into the host lattice during the synthesis of the phosphor powders is essential for developing highly luminescent RE<sup>3+</sup> doped nanocrystalline phosphors as well as for the growth of grain particles. Photoluminescence studies and CIE co-ordinates of LaPO<sub>4</sub>:Eu<sup>3+</sup> (0.5 mol %) Ce<sup>3+</sup> (0.5 mol %) phosphors reveals that the emission colour varies from blue to orange-red. So this material may be a potential luminescent material.

## Experimental

### Synthesis

Pure LaPO<sub>4</sub> and LaPO<sub>4</sub>:Eu<sup>3+</sup> (0.5 mol %) Ce<sup>3+</sup> (0.5 mol %) phosphor powders were synthesized by using the conventional solid-state reaction method. The formation of the phosphor powders occurs according to the following chemical equation.



The starting materials were lanthanum oxide (La<sub>2</sub>O<sub>3</sub>), Diammonium Hydrogen Phosphate [(NH<sub>4</sub>)<sub>2</sub> H<sub>2</sub> PO<sub>4</sub>], and Europium Oxide (Eu<sub>2</sub>O<sub>3</sub>) of 99.9% purity and Ceric oxide (CeO<sub>2</sub>) of 99.9% purity. They were weighed with a certain stoichiometric ratio. The composite powders were grinded in an agate mortar and then placed in an alumina crucible with the lid closed. After the powders had been sintered at 1200 °C for 3 hr in a muffle furnace and then cooled to

room temperature. All the samples were again ground into fine powder using an agate mortar and pestle about an hour.

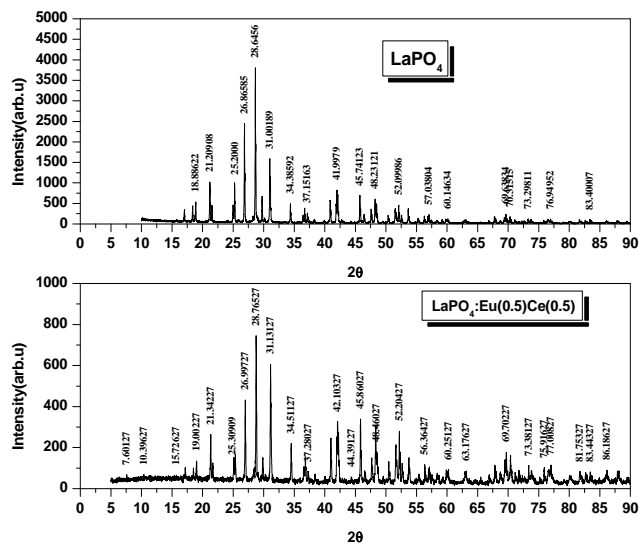
### Characterization

X-ray diffraction (XRD) pattern of the product was carried out on PANalytical's X-ray diffractometer X'Pert PRO (Cu K $\alpha$  radiation,  $\lambda=0.154\text{nm}$ ) employing a scanning rate of  $0.02\text{s}^{-1}$  and  $2\theta$  ranges from  $15^\circ$  to  $80^\circ$ . The morphology of the nanoparticles was observed by using a scanning electron microscope (TESCAN VEGA3 SEM) with a tungsten heated filament. The emission and the excitation spectra of the synthesized powders were characterized with a spectrofluorophotometer (Shimadzu RF – 5301 PC) with xenon lamp as excitation source. Infrared spectra for the prepared solid nano powders were recorded in the range between 400 and 4000  $\text{cm}^{-1}$  on a Fourier-transform spectrometer (Bruker Vector 22 FT-IR Spectrometer). The particle size was measured by using laser based system Malvern Instrument, U.K. The Commission International de l'Eclairage (CIE) co-ordinates were calculated by the spectrophotometric method using the spectral energy distribution. The chromatic coordinates ( $x$ ,  $y$ ) of prepared materials were calculated with colour calculator version 2, software from Radiant Imaging [14].

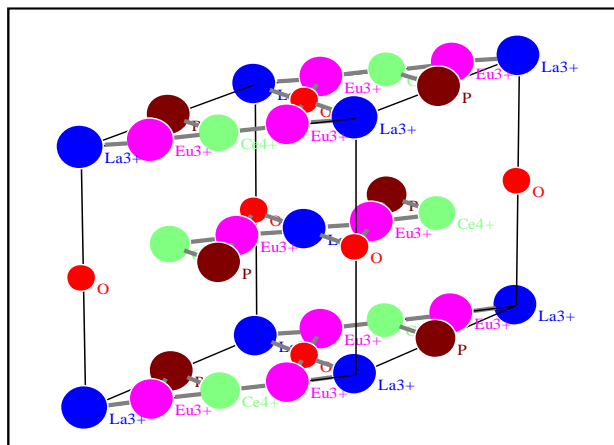
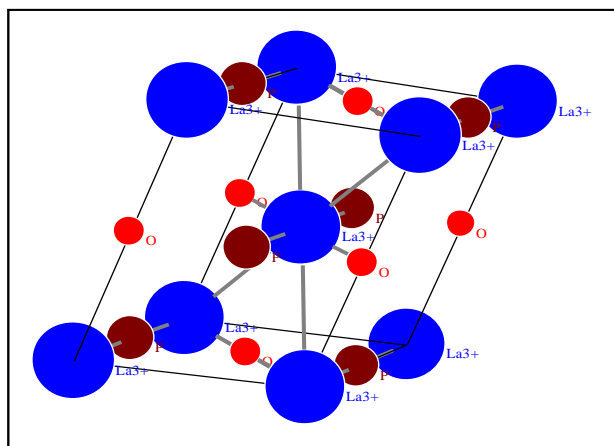
## Results and discussion

### Crystal structure of LaPO<sub>4</sub> and LaPO<sub>4</sub>:Eu<sup>3+</sup>, Ce<sup>3+</sup> phosphors

**Fig. 1 (a)** show the X-ray diffraction (XRD) patterns of synthesized samples of LaPO<sub>4</sub> and LaPO<sub>4</sub>:Eu<sup>3+</sup> (0.5 mol %) Ce<sup>3+</sup> (0.5 mol %) phosphor powders. The XRD spectra consist of three strong peaks and several weak peaks: The three main peaks occur at  $2\theta=26.86$ ,  $28.65$  and  $31.0018^\circ$ . These peaks correspond to the diffractions from the (200), (120), and (012) planes of LaPO<sub>4</sub> respectively. The relatively weak multi peaks centered at  $21.29$ ,  $34.30$ ,  $41.99$  and  $48.23^\circ$  are attributed to the diffraction from the (111), (202), (311), and (132) planes, respectively. The intensity of peaks reflected the high degree of crystallinity of the nanoparticles. However, the diffraction peaks are broad which indicating that the crystalline size is very small. All the diffraction peaks could be well indexed to JCPDS: 84-0600, which indicated a monoclinic structure LaPO<sub>4</sub> (space group P2<sub>1</sub>/n) with a main diffraction peak (120). No spurious diffractions due to crystallographic impurities are found. **Fig. 1 (b)** show the structure of LaPO<sub>4</sub> and structure of LaPO<sub>4</sub> doped with the Eu and Ce. The average particle size has been estimated by using the Debye-Scherrer formula  $D=0.9\lambda/\beta\cos\theta$ , where  $\lambda$  is the wavelength of the X-ray ( $\lambda=1.54\text{\AA}$ ),  $\beta$  is FWHM (full width at half maximum),  $\theta$  is the diffraction angle and  $D$  is the particle diameter size. The average size of a particle of pure LaPO<sub>4</sub> phosphor and LaPO<sub>4</sub>:Eu<sup>3+</sup>, Ce<sup>3+</sup> phosphor are 67.6 nm and 64nm respectively. This confirms the formation of nano crystalline size phosphor, via solid state reaction method. Structure of LaPO<sub>4</sub> and Structure of LaPO<sub>4</sub> doped with the Eu, Ce shown in **Fig. 1 (b)**. Unit cell parameters of the sample powders were shown in **Table 1**.



**Fig. 1** (a) XRD of  $\text{LaPO}_4$  and doped with  $\text{LaPO}_4:\text{Eu}$  (0.5 mol %)  $\text{Ce}^{3+}$  (0.5 mol %).



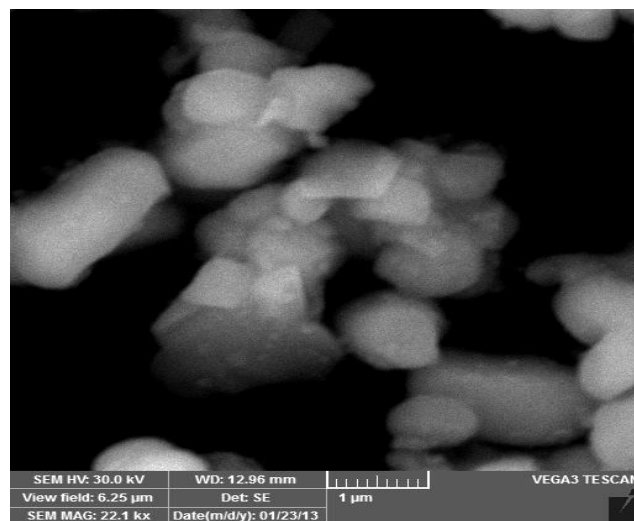
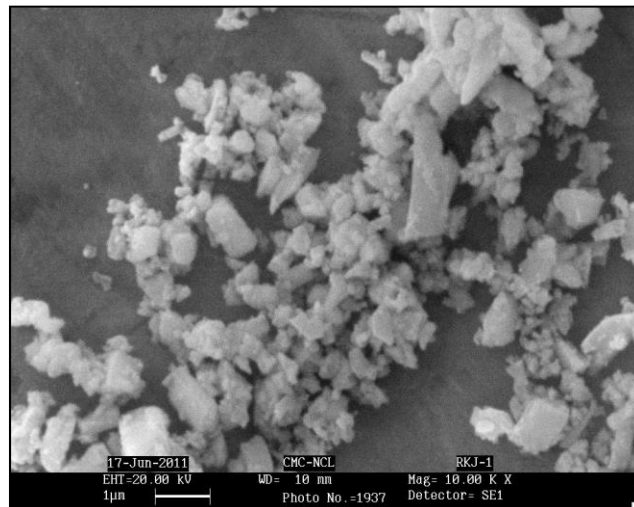
**Fig. (b)** Structure of  $\text{LaPO}_4$  and Structure of  $\text{LaPO}_4$  doped with Eu and Ce

**Table 1.** Unit cell lattice constants of the sample powders.

Samples	a (nm)	b (nm)	c (nm)	$\beta$ angle (deg)	Cell volume (nm <sup>3</sup> )
JCPDS 84-0600	0.6825	0.7057	0.64822	103.210	0.3039
$\text{LaPO}_4$ phosphor	0.6827	0.70477	0.63968	103.210	0.3077
$\text{LaPO}_4:\text{Eu}^{3+}, \text{Ce}^{4+}$	0.6779	0.7032	0.6371	103.66	0.3037

### Morphology of $\text{LaPO}_4$ and $\text{LaPO}_4:\text{Eu}^{3+}, \text{Ce}^{3+}$ phosphors

**Fig. 2** (a, b) are typical SEM images of the morphology of the synthesized nanoparticles of pure  $\text{LaPO}_4$  and  $\text{LaPO}_4:\text{Eu}^{3+}, \text{Ce}^{3+}$  phosphors. SEM images attest for the obtaining of homogeneous solids, which occur as micrometric aggregates of nanosized structures. The particles composing these micro aggregates have a spherical shape and sizes ranging from 0.15-0.2  $\mu\text{m}$ . The grain sizes of the samples estimated from the SEM picture is larger than that obtained from XRD analysis. The observation of some larger nanoparticles may be attributed to the fact that the nanoparticles have the tendency to agglomerate due to their high surface energy.



**Fig. 2.** SEM images of pure  $\text{LaPO}_4$  and  $\text{LaPO}_4:\text{Eu}^{3+}$ (0.5mol%)  $\text{Ce}^{3+}$  (0.5 mol %) phosphor.

### FTIR analysis of $\text{LaPO}_4$ and $\text{LaPO}_4:\text{Eu}^{3+}, \text{Ce}^{3+}$ phosphors

FTIR analysis was carried out to determine the chemical bonds in a molecule. **Fig. 3** (a, b) shows the FTIR spectrum of pure  $\text{LaPO}_4$  and  $\text{LaPO}_4:\text{Eu}^{3+}, \text{Ce}^{3+}$  phosphor. The absorption bands at 3269 and 2013  $\text{cm}^{-1}$  can be assigned to physical adsorbed OH and  $\text{H}_2\text{O}$ . The characteristic vibrations of phosphate ( $\text{PO}_4^{3-}$ ) are obvious. The two bands located at 621.55 and 491  $\text{cm}^{-1}$  are clearly observed in the  $\nu_4$  region of the vibrations of  $\text{PO}_4^{3-}$  groups. The bands

at  $1056\text{cm}^{-1}$  can be attributed to the  $\nu_3$  anti-symmetric stretching of P-O band. The shoulder at  $773\text{cm}^{-1}$  can be assigned to the  $\nu_1$  vibration of  $\text{PO}_4^{3-}$  groups [15-17]. The  $\nu_2$  vibration at low wave number is not observed in the studied range of wave numbers. Furthermore, C-H stretching vibration at  $1909\text{cm}^{-1}$  can be detected.

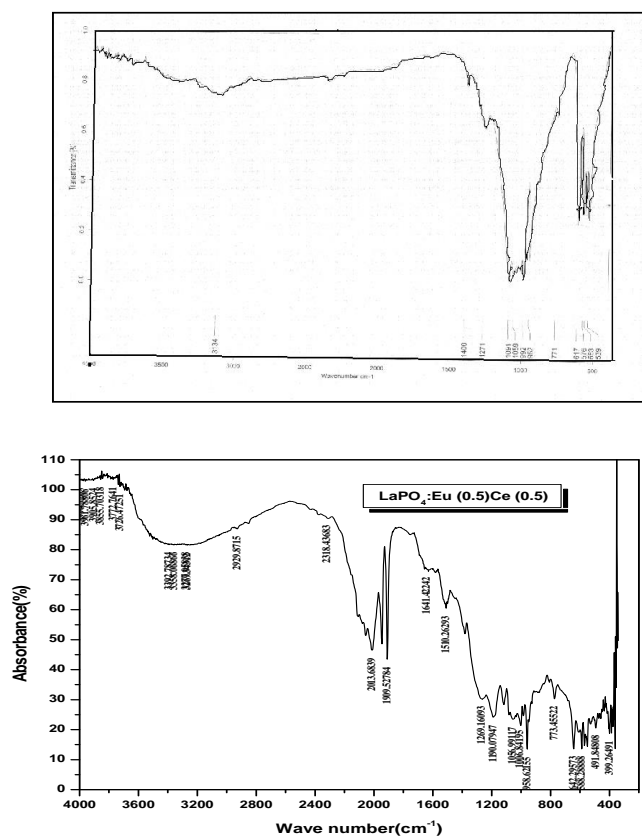


Fig. 3. FTIR Spectrum of  $\text{LaPO}_4:\text{Eu}(0.5)\text{Ce}(0.5)$

Fig. 3. FTIR spectrum of pure  $\text{LaPO}_4$  and  $\text{LaPO}_4:\text{Eu}^{3+}$  (0.5 mol %)  $\text{Ce}^{3+}$  (0.5 mol %).

#### Particle size analysis

The Particle size distribution histogram of  $\text{LaPO}_4:\text{Eu}^{3+}$ ,  $\text{Ce}^{3+}$  phosphor particles synthesized using the solid state reaction method illustrated in Fig. 4. The prepared phosphor specimen particle size was measured by using laser based system Malvern Instrument, U.K. The mean diameter of particle size of  $\text{LaPO}_4:\text{Eu}^{3+}$ ,  $\text{Ce}^{3+}$  phosphor is  $1.95\mu\text{m}$ .

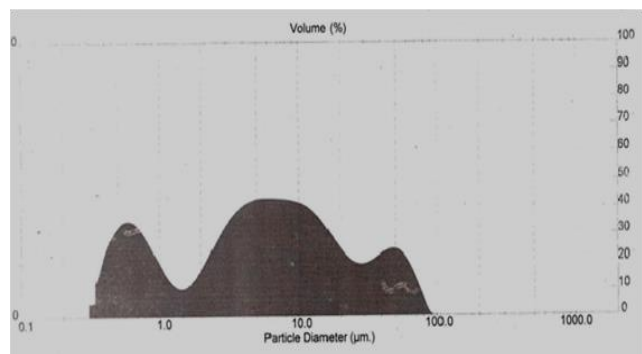


Fig. 4. Particle size distribution histogram of  $\text{LaPO}_4:\text{Eu}^{3+},\text{Ce}^{3+}$  phosphor.

#### Photoluminescence of $\text{LaPO}_4$ and $\text{LaPO}_4:\text{Eu}^{3+},\text{Ce}^{3+}$ phosphors

A series of  $\text{LaPO}_4:\text{Eu}^{3+}$  (0.5 mol %)  $\text{Ce}^{3+}$  (0.5 mol %) phosphors heated at  $1200^\circ\text{C}$  were prepared and the effect of  $\text{Eu}^{3+}$ ,  $\text{Ce}^{3+}$  concentration on the emission intensity was investigated. Fig. 5 (a) exhibits the PL excitation and emission spectra of pure  $\text{LaPO}_4$  phosphor. In the excitation spectrum monitored under 400nm wavelength, the broadband ranging from 220-300nm with peaks at 249 nm. The shape of the emission spectra and emission peak wavelength is independent of the excitation wavelengths. Upon excitation at 254nm wavelength, the emission spectrum of pure  $\text{LaPO}_4$  phosphor emits a broad band range from 300-600nm with maximum intensity peak at 470 (blue) nm as shown in Fig. 5a.

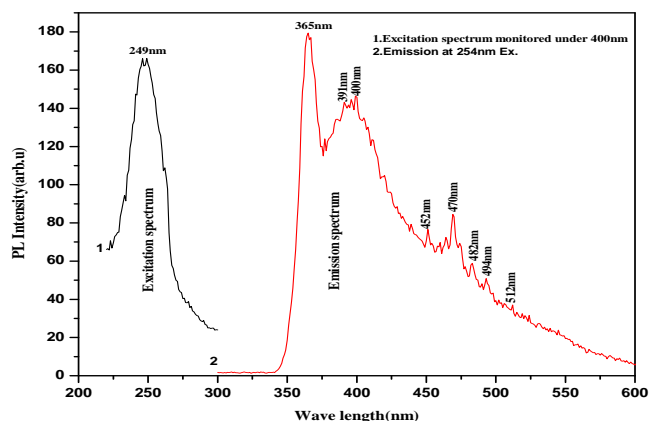


Fig. 5 (a). Excitation and Emission spectrum of  $\text{LaPO}_4$ .

Fig. 5 (b) exhibits the PL excitation and emission spectra of  $\text{LaPO}_4:\text{Eu}^{3+},\text{Ce}^{3+}$  phosphor. In the excitation spectrum monitored under 612 nm wavelength, the broad and intensive band ranging from 225-350nm with maximum peak at 264 nm, related to a ligand-metal charge transfer between  $\text{PO}_4^{3-}$  groups and  $\text{Eu}^{3+}$  ions.

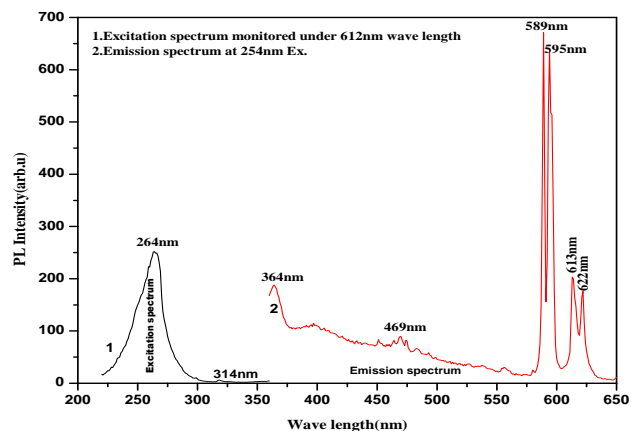


Fig. 5 (b). Excitation and Emission spectrum of  $\text{LaPO}_4:\text{Eu},\text{Ce}$ .

The emission spectrum of  $\text{LaPO}_4:\text{Eu}^{3+},\text{Ce}^{3+}$  phosphor under 254 nm excitation wavelength displays the characteristic red light with a number of narrow lines due to  ${}^5\text{D}_0 \rightarrow {}^7\text{F}_{J=0,1,2,3,4}$  transitions of  $\text{Eu}^{3+}$ . The  ${}^5\text{D}_0 \rightarrow {}^7\text{F}_0$  transition occurs as a unique, sharp and intense line, indicating the

occupation of a site of low symmetry, in agreement with the monoclinic structure of this orthophosphate. The predominance of the hypersensitive, magnetic dipole transition  ${}^5D_0-{}^7F_1$  (589nm) transition in the emission spectrum is determinant for the applicability of this material; high emission orange-red colour purity is achieved. In the present case, the low contributions of the red (613 nm)  ${}^5D_0-{}^7F_2$  emission originate from the electric dipole transition and the high intensity of the  ${}^5D_0-{}^7F_1$  (589 nm) emission results in high color purities. The electric dipole transition is allowed when  $\text{Eu}^{3+}$  ion occupies a site without an inversion center and is sensitive to local symmetry. When  $\text{Eu}^{3+}$  ion occupies inversion center sites, the  ${}^5D_0-{}^7F_1$  transition should be relatively strong, while the  ${}^5D_0-{}^7F_2$  transition should be relatively weak [18].

#### CIE coordinates

The CIE co-ordinates of (chart -1931) were calculated by the Spectrophotometric method using the spectral energy distribution of pure  $\text{LaPO}_4$  phosphor and  $\text{LaPO}_4:\text{Eu}^{3+}, \text{Ce}^{3+}$  phosphor as shown in Fig. 5. The color co-ordinates for pure  $\text{LaPO}_4$  sample (A) are  $x = 0.16$  and  $y = 0.03$  and  $\text{Eu}, \text{Ce}$  doped  $\text{LaPO}_4$  sample (B) sample are  $x = 0.57$  and  $y = 0.43$ . From the Fig. 6, it was observed that the emission varies from blue to orange-red region. CIE 1931 chromaticity coordinates of prepared samples are acceptable for many optical applications [19, 20].

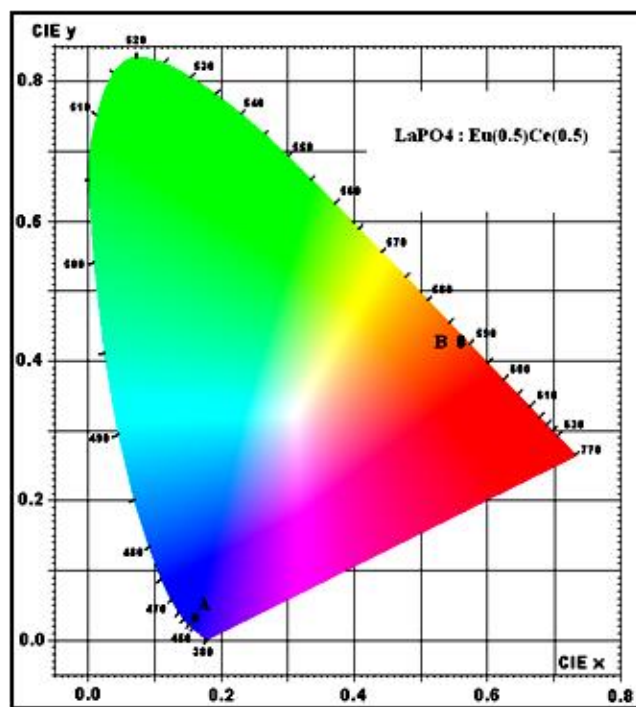


Fig. 6. CIE Co-ordinates of pure  $\text{LaPO}_4$  and  $\text{LaPO}_4:\text{Eu}^{3+}, \text{Ce}^{3+}$  phosphor depicted on the 1931 chart.

#### Conclusion

Pure  $\text{LaPO}_4$  and  $\text{LaPO}_4:\text{Eu}^{3+}$  (0.5 mol %)  $\text{Ce}^{3+}$  (0.5 mol %) phosphor powders were successfully synthesized by the high temperature solid state reaction method and the luminescent properties of samples was studied. The XRD results reveal that the synthesized samples phosphors are

well crystalline and assigned to the monoclinic crystal structure with a main (120) diffraction peak. No spurious diffractions due to crystallographic impurities are found. The width of diffraction peaks is broadened because of the small size of the crystallites. The grain sizes of the samples estimated from the SEM picture is larger than that obtained from XRD data.  $\text{LaPO}_4:\text{Eu}^{3+}$  (0.5 mol %)  $\text{Ce}^{3+}$  (0.5 mol %) phosphor powders exhibit the characteristic emission  $\text{Ce}^{4+}$  lines and also exists energy transfer process between  $\text{LaPO}_4$  and  $\text{Eu}^{3+}, \text{Ce}^{3+}$  ion. The luminescence intensity of  $\text{Eu}$  is found to improve when  $\text{Ce}$  is co-doped as a sensitizer. Under excitation, the intensity of transition from  ${}^5D_0-{}^7F_1$  is higher than that from  ${}^5D_0-{}^7F_2$ . The intensity variation of blue to orange-red emission is high in  $\text{Ce}$  sensitized  $\text{LaPO}_4:\text{Eu}^{3+}$ . The Stoke shift and the FWHM of the emission were characteristic of a ligand-to-metal charge transfer (CT) emission. The photoluminescence results indicate that the  $\text{LaPO}_4: 0.5 \text{ mol } \% \text{ Eu}^{3+}, \text{Ce}^{3+}$  (0.5 mol %) phosphor have a strong orange-red  ${}^5D_0-{}^7F_1$  transition. The Commission International de l'Eclairage [CIE] co-ordinates of pure  $\text{LaPO}_4$  phosphor exhibit the excellent colour tunability of blue, and  $\text{Eu}, \text{Ce}$  doped  $\text{LaPO}_4$  phosphor reveals that the emission varies from blue to orange-red may make it to be a potential luminescent material.

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