Rare Earth doped LaPO₄ Phosphor Synthesis and Characterization

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ABSTRACT

The present paper reports the Synthesis and Photoluminescence (PL) studies of the LaPO₄ phosphor doped with Eu (0.5), Tb (1.0) and Ce with 0.5, 1.0, 1.5, 2.0, 3.0, 4.0 and 5.0 molar percentages respectively. The phosphor was synthesized using the standard solid state diffusion reaction technique. The mixture was fired at 1200°C for 3 hours with heating rate of 5°C/min in a muffle furnace and the received cake is ground using mortar and pestle. The powder phosphors were characterized by X-ray diffraction, FTIR and scanning electron microscopy. The crystallinity and phase purity of the phosphor was confirmed by XRD studies. The Infrared spectra for the prepared solid nano powders were recorded in the range between 400 and 4000 cm⁻¹ on a Fourier transform spectrometer. The particle morphology of the phosphor was characterized by SEM. The Photoluminescence properties of the materials were studied using Spectrofluorophotometer at room temperature. The Photoluminescence (PL) excitation spectra were recorded for different excitation wavelengths at 254 nm, 264 nm and 275 nm monitoring at 614 and 545 nm. The PL emission of Eu(0.5), Tb(1.0) and Ce(0.5, 1.0, 1.5, 2.0, 3.0, 4.0 and 5.0 mol%) doped LaPO₄ phosphor was recorded for different excitation wavelengths. The PL emission peaks are found at 364, 381, 415, 438, 470, 488, 545, 589, 594, 614 and 622 nm with good intensity.

Keywords: Photoluminescence [PL]; Rare Earth ions [RE ions]; XRD, Solid State Reaction [SSR].

I. INTRODUCTION:

Recently various phosphors like LaPO₄:Ce,Tb have been good commercial green phosphor materials which have been actively investigated to improve their luminescent properties and to meet the development of different display and luminescence devices. Inorganic compounds doped with rare earth ions form an important class of phosphors as they possess a few interesting characteristics such as excellent chemical stability, high luminescence efficiency, and flexible emission colors with different activators. As a new green luminescent material, LaPO₄ Ce,Tb phosphor has been widely studied since it was found by different preparation methods [1,2].

These phosphors are widely used in displays and lighting devices. The useful applications of rare earth element compounds, especially lanthanide phosphate doped inorganic materials, have been touched upon broadly [5]. Over the past a few years, they have been applied in many fields, such as optical display panels, cathode ray tubes, optoelectronic, sensitive device, electronic and plasma display panels due to their special chemical and physical properties. Various solution-phase routes, including solid state reaction, sol-gel, precipitation, water oil micro emulsion, polyol-mediated process, ultrasonification, hydrothermal, and mechanochemical method, have been tried to lower the reaction temperature and obtain high-quality LaPO₄ based nanoparticles. However, the simple and mass fabrication of LaPO₄ nanocrystals with narrow grain size distribution and uniform morphology still remains a challenge [3,4]. It appears that the best solution both to control powder morphology and to produce low cost thin films is the use of soft chemistry routes. We adopted the standard solid state reaction technique to prepare LaPO₄ with good morphologies and fine crystal structures, and its emission intensity of luminescence was also studied [5,6]. The present paper reports the Photoluminescence (PL) of the LaPO₄ phosphor doped with Ce, Eu and Tb rare-earth ions with different emission and excitation wavelengths, the doping concentration of Eu, Tb and Ce are 0.5, 1, 2, 3, 4 and 5 molar percentages respectively.

In the trivalent rare earth ions, the luminescence arises mainly due to transactions within the 4f shell. The efficiency of emission depends on the number of electrons in the 4f shell. The Tb³⁺ ion has 8 electrons in the 4f shell, which can be excited in the 4f-5d excitation band. The electron in the excited 4f⁰ - 5d state remains at the surface of the ion and comes under the strong influence of the crystal field resulting in the splitting of the excitation band. The excitation Spectra thus has multiple peaks. The excited ion in the 4f⁰ – 5d state decays stepwise from this state to the luminescent levels 3D₄-F₄ or 3F₄ by giving up phonons to the lattice. Luminescence emission occurs from either of these states, with the ion returning to the ground state. The emission line in the green region lying at 545 nm is due to the transition 3D₄ - F₄. There are in fact multiple emission lines at each of these due to the crystal field splitting of the ground state of the emitting ions [7,8].
2. MATERIALS AND METHODS
All the chemical reagents were analytically pure and used without further purification. LaPO₄ phosphor doped with Eu, Tb and varying concentrations of Ce (0.5, 1.0, 1.5, 2.0, 3.0, 4.0 and 5.0 mol %) rare-earth ions prepared using solid state diffusion reaction method. Stoichiometric proportions of raw materials namely, Lanthanum Oxide (La₂O₃), Diammonium Hydrogen Phosphate [(NH₄)₂ H₂PO₄], Cerium Oxide (CeO₂), Europium Oxide (Eu₂O₃) and Terbium Oxide (Tb₂O₃) of assay 99.9% were used as starting materials and grounded in an agate motor and pestle, mixed and compressed into a alumina crucible and heated at 1200°C for 3 hours with heating rate of 5°C/min in the muffle furnace [9, 10]. The prepared samples were again grounded in to powder for taking the characteristic measurements.

All the phosphor samples were characterized by X-ray diffraction (Synchrotron Beam Indus -II), The Photoluminescence (PL) emission and excitation spectra were measured by Spectrofluorophotometer (SHIMADZU, RF-5301 PC) using 150 watts Xenon lamp as excitation source. The emission and excitation slit were kept at 1.5 nm, and recorded at room temperature. The Infrared spectra for the prepared solid nano powders were recorded in the range between 400 and 4000 cm⁻¹ on a Fourier transform spectrometer. The particle morphology of the phosphor was characterized by SEM.

3. RESULTS AND DISCUSSION
3.1 X-ray Diffractometry (XRD)
The crystallinity and phase purity of the phosphors were firstly examined by XRD analysis. fig.1 shows the typical X-ray diffraction (XRD) patterns of synthesized samples of LaPO₄ doped with Eu(0.5), Tb(1.0) and Ce(2.0 mol%). The XRD patterns of the phosphor is in good agreement with the values from JCPDS no.35-731 of LaPO₄, which conform the phosphor is in monazite LaPO₄ with monoclinic structure. All diffraction patterns were obtained using CuKα radiation (λ = 1.540598 Å) at 40 kv and 30 mA, and divergence slit fixed at 1.52 mm. Measurements were made from 2θ = 15° to 50° with steps of 0.008356°. When crystallites are less than approximately 100 nm in size, appreciable broadening in X-ray diffraction lines occurs. The crystallite size of powder sample were calculated by using Scherer equation D= 0.9 λ / βcosθ Where β represents full width at half maximum (FWHM) of XRD lines (0.112), λ = Wavelength of the X-rays(0.154 nm in the present case), θ = Braggs angle of the XRD peak. The calculated crystallite size for LaPO₄: Eu (0.5), Tb(1.0)&Ce(2.0mol%) phosphors is 73.14 nm, and from the XRD study it conforms the formation of phosphor in single phase and matched with JCPDS card no 35-731 of LaPO₄.

Fig.1: XRD of LaPO₄ doped with Eu³⁺, Tb³⁺ and Ce³⁺

3.2 SEM Study:
Figure 2 and 3 show SEM image of LaPO₄: Eu (0.5), Tb (1.0), Ce(2.0 mol%). From SEM images it is observed that the particles look irregular shape and agglomerated having particle size of 1 micron to 5 microns.

Fig.2. SEM Image of LaPO₄: Eu, Tb, Ce.(8.45 KX)
3.3 FTIR study of LaPO4: Eu, Tb, Ce.

In order to determine the chemical bonds of the present studied phosphor FTIR analysis was carried out. Figure - 4 is the FTIR of the Eu, Tb and Ce doped LaPO4 phosphor, the main absorption around 3600 are assumed H-O-H stretching followed by other bonds of C-H bending, C-O stretching and CO-OH stretching. CO-OH and H-O-H stretching are due to absorbed CO2 and H2O molecules from atmosphere.

3.4 Photoluminescence Study:

Figure 5 and 6 are the excitation spectra of LaPO4 doped with Eu(0.5), Tb(1.0) Ce (0.5 to 5%) when monitored at 614 and 545nm. From figure 6 it is observed that the excitation spectra consist of two absorption maxima at 264 and 275nm. Fig.7,8, 9, 10 and 11 are the emission spectra of LaPO4 doped with Eu(0.5), Tb(1.0) and Ce(0.5,1.0,1.5,2.0,3.0,4.0 and 5.0 mol%) phosphor under different excitations at 254 and 275 nm wavelengths. The phosphor shows the PL peaks at 364, 381, 415, 438, 470, 488, 545, 589, 594, 614 and 622 nm with good intensity. The excitation spectrum monitored with the emission of Tb3+ (545 nm) consists of a broad band peaking at 275 nm with two shoulders at 241, 264 nm, which correspond to the transitions from the ground state F2 to the different components of the excited Ce3+ 5d states split by the crystal field [22]. Excitation into the Ce3+ band at 290 nm yields both the weak 5d→f emission of Ce3+ (300–400 nm) and the strong emission of Tb3+ (488, 545, 583 and 622 nm, corresponding to 5D4→7F6,5,4,3 respectively), as shown in Fig. 6b. This indicates that an efficient energy transfer from Ce3+ to Tb3+ occurs in LaPO4 doped with Eu(0.5), Tb(1.0) Ce (2) phosphor particles, as reported previously[11,12].

From the emission figures, as increase the excitation wavelength from 254 to 275nm, the emission peaks intensities decreased except 488 and 545 nm peaks, the intensity of 488 nm peak slightly increased and 545nm peak intensity is increased by two times which is shown in fig. 13 and table-3 for better comparison.
Fig-7,8 are the emission spectrum of LaPO$_4$: Eu(0.5), Tb(1.0) and Ce(0.5,1.0,1.5,2.0,3.0,4.0,5.0 mol%) under 254nm excitation wavelength. From the graphs it is observed that the peaks at 364,381,415,438,470, 614,622nm with lower intensity and 488,545,589 and 594nm are with high intensity. As increasing the Ce concentration in LaPO$_4$: Eu(0.5), Tb(1.0) from 0.5 to 5.0 mol %, the 545nm peak intensity increases up to 2.0 mol% of Ce doping further increasing the Ce concentration the 545nm peak intensity gradually decreases. When concentration of Ce is more than 2.0 mol% in LaPO$_4$: Eu(0.5), Tb(1.0) the quenching effect started and maximum quenching at 5% of Ce ions in LaPO$_4$: Eu(0.5), Tb(1.0) is observed. The other major peaks at 589nm and 594nm peaks intensity is gradually decreases as Ce concentration increases and the remaining peaks at 364nm,381nm,438nm and 470nm peaks intensity increases as Ce concentration increases up to 1.5 mol% and further increase of Ce decreases the PL intensity.

Fig-9,10,11 are the emission spectrum of LaPO$_4$: Eu(0.5), Tb(1.0) and Ce(0.5,1.0,1.5,2.0,3.0,4.0,5.0 mol%) under 275nm excitation wavelength. The same PL pattern is observed like 254nm excitation except the emission intensity of 545nm increases by 60% when compared to 254nm excitation when the Ce concentration is at 2%. However as Ce concentration increases the PL peak intensities of 364nm, 381nm, 415nm, 438nm and 470nm peaks increase marginally as Ce concentration increases up to 1.5 mol% and further decreased their intensity when excited with 275nm. Table-1 and Table-2 are the emission intensities of various peaks, they are observed in LaPO$_4$: Eu(0.5), Tb(1.0) and Ce for different concentrations when excitation with 254 and 275nm. The same is shown in fig-12 for better comparison.

It is clearly observed that the PL emission intensity of 545nm, 589nm and 594nm peaks are affected with respect to excitation wavelengths, which is as shown in fig-13. From the figure 545nm peak’s intensity increased and 589nm, and the 594nm peak’s intensity decreased as increasing excitation wavelengths. Table-3 gives the emission peak intensity for better comparison.
The excitation spectra of LaPO₄: Eu, Tb, Ce phosphor was recorded by monitoring the "D₀ → F₂" transition of Eu³⁺ at 614nm is shown in Fig. 5. The excitation spectra show broad absorption peak in the range 220-270nm. The excitation spectra of LaPO₄: Eu, Tb, Ce phosphor was recorded by monitoring the "D₀ → F₂" transition of Eu³⁺ at 614nm is shown in Fig. 5. The excitation spectra show broad absorption peak in the range 220-270nm. The

Table-1: Emission intensity for different Ce concentrations under 254nm Excitation.

<table>
<thead>
<tr>
<th>S No</th>
<th>Peak Wavelength(nm)</th>
<th>Emission Intensity for different Ce concentrations under 254nm Excitation</th>
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<tr>
<td></td>
<td>0.5%</td>
<td>1.0%</td>
</tr>
<tr>
<td>1</td>
<td>364</td>
<td>54</td>
</tr>
<tr>
<td>2</td>
<td>381</td>
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<td>3</td>
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<td>5</td>
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<td>614</td>
<td>70</td>
</tr>
<tr>
<td>11</td>
<td>622</td>
<td>65</td>
</tr>
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</table>

Table-2: Emission peaks intensity for different Excitation wavelengths of LaPO₄:Eu(0.5),Tb(1.0) and Ce(2.0 mol %)

<table>
<thead>
<tr>
<th>S No</th>
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<th>Emission peak intensity for different Excitation wavelengths</th>
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<tr>
<td>11</td>
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<td>48</td>
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</table>

Table-3: Emission peaks intensity for different Excitation wavelengths of LaPO₄:Eu(0.5),Tb(1.0) and Ce(2.0 mol %)

![Fig-12: Variation of PL Intensity with Ce concentration for different emission peaks under 275nm Ex](image1)

![Fig-13: Variation of emission intensity with Excitation wavelength for different peaks](image2)
prominent excitation band was observed at 254nm due to the transition of Eu$^{3+}$ and this clearly indicates that in LaPO$_4$: Eu, Tb, Ce phosphor where in Eu stabilized as Eu$^{3+}$ ion. Fig. 7 and 9 are the emission spectra of LaPO$_4$: Eu, Tb, Ce phosphor at room temperature having 254 and 275nm excitation.

Emission spectrum consists of emission peaks in the range of 550–650 nm, which result from $^5D_0 \rightarrow ^7F_J$ (J=1, 2) transitions of Eu$^{3+}$ ion respectively. For an excitation wavelength of 254nm, the emission spectrum of LaPO$_4$: Eu, Tb, Ce consists of sharp emission lines at 589,594,614 and 622nm. The emission at 589nm and 594 nm originates from the allowed magnetic dipole (MD) transition $^5D_0 \rightarrow ^7F_1$. The $^5D_0 \rightarrow ^7F_1$ magnetic dipole transition emissions are split at 589 nm and 594 nm. The peak was observed at 614nm due to the electric dipole $^5D_0 \rightarrow ^7F_2$ transition, which has the less emission intensity. From the emission spectrum it is clearly observed that the emission intensity of magnetic dipole was higher than that of electric dipole transition, due to this Eu$^{3+}$ ions occupy a low symmetry site in LaPO$_4$: Eu, Tb, Ce host. Both magnetic dipole transition and electric dipole transitions are shown in the emission spectra. If the magnetic dipole transition $^5D_0 \rightarrow ^7F_1$ having the highest intensity then Eu$^{3+}$ ions in host lattice occupies an inversion centre [15,17]. If the emission intensity of magnetic dipole transition was lower than that of electric dipole transition, which indicates that Eu$^{3+}$ ions occupied without an inversion symmetric centers in the host.

The excitation spectra of LaPO$_4$: Eu, Tb, Ce extended from 250-280nm when monitored at 545nm which is shown as 6. The emission transitions in the wavelength range of 320–400 nm are characteristic of forbidden 4f-4f transitions within the Tb$^{3+}$ configuration. The peaks with the position from 300 to 400 nm belong to the transitions between energy levels of the 4f$^8$ configuration of Tb$^{3+}$ (E-f transitions)[16,18]. Among these, the 381 nm transition has the maximum intensity compared to the other transitions.

The emission spectra of LaPO$_4$: Eu, Tb, Ce phosphors (x = 0.5 -5mol %) are compared in fig. 7,8,9,10,11. Each spectrum could be divided into two parts. The emission between 400–450 nm is due to transitions from the $^5D_3$ excited state. Above 480 –630nm, the emission peaks are originated from $^5D_0$ excited states of Tb$^{3+}$. The $^5D_4 \rightarrow ^7F_J$ (J = 6, 5, 4, 3) characteristic emissions of Tb$^{3+}$, at 415, 438, 488, 545 and 622 nm, correspond to the transitions of $^5D_3$ to $^7F_5$, $^7F_4$ and $^5D_4$ to $^7F_6$, $^7F_5$, and $^7F_3$, respectively. Under an ultraviolet lamp a strong greenish yellow luminescence is observed. The corresponding emission spectra of LaPO$_4$: Eu, Tb, Ce indicate narrow emissions which arise from 4f-4f transitions within Tb$^{3+}$ ions as shown in fig.9. Among the measured emission transitions, the green-emission transition $^5D_4 \rightarrow ^7F_5$ at 545 nm has more intense in nature due to the nature of the dopant Tb$^{3+}$ ion in the host matrix. Emission intensities from $^5D_1$ excited state decrease with the increase of the cerium concentration and show a maximum intensity at 545 nm when Ce is 2% when excited with 275nm. Comparing the emission intensities of the different doping concentrations of Ce, it can be observed that in the range of 0.5 –5 mol% for doping concentrations the luminescent intensities originating from the Tb$^{3+}$, $^5D_4 \rightarrow ^7F_J$ (J = 6, 5, 4, 3) transitions increase with the increase of concentration, as a result, the luminescent intensity reaches maximum at 2 mol% of Ce$^{3+}$. When the concentration of Ce$^{3+}$ increases more than 2 mol%, emission intensity decreases which is due to the concentration quenching and also Ce may be stabilizing in Ce$^{4+}$ state. The decrease in the intensity with an increase in concentration was presumably due to the well-established theory of concentration quenching [9]. It can be seen from fig.9, a prominent and significant greenish yellow emission has been noticed for 1 mol% Tb$^{3+}$, which implies that this concentration in LaPO$_4$:Eu, Tb, Ce phosphor could be found as a potential and efficient phosphor for decorative CFL lamps.

The absorption band of Ce$^{4+}$ is expected to be an efficient killer. Therefore, it is likely that Ce$^{4+}$ is present in the LaPO$_4$: Eu, Tb, Ce lattice when the Ce concentration is more than 3%. The absorption band of Ce$^{4+}$ is ascribed to a second phase containing Ce$^{4+}$. The Ce$^{3+}$ emission in La PO$_4$ is double band, peaking at 250 and 275 nm, respectively resulting from the splitting of the ground states $^2F_{5/2}$ and $^2F_{7/2}$. It is assumed that the cerium atoms have a good random distribution in the crystals, the inter-distance between Ce$^{3+}$ and Ce$^{4+}$ should also be in this range. It is known that the energy transfer between Ce$^{3+}$ in La PO$_4$ is through dipole-dipole interaction [5].

Based on the above discussions, we may conclude that the Ce$^{3+}$ is an efficient killer for the Ce$^{3+}$ emission and does play an important role in the luminescence process of LaPO$_4$:Eu, Tb, Ce. Since the energy transfer between Ce$^{3+}$ and Ce$^{4+}$ is an inter-valence charge transfer, the efficiency of this energy transfer depends not only on the concentration of the Ce$^{3+}$ but also on the special distribution of the cerium atoms in the material. The energy transfer between Ce$^{3+}$ and Ce$^{4+}$ reduces the energy transfer probability between Ce$^{3+}$ and Tb$^{3+}$, and subsequently reduces the emission intensity of Tb$^{3+}$. The transfer of energy from sensitizer to activator occurs without the appearance of a photon and is the primary result of the multipole interactions between the sensitizer and activator [19, 20].

The Cerium emission $^5d (^5D_{3/2}) \rightarrow 4f(^2F_{5/2}$ and $^2F_{7/2})$ involves pure electric dipole transitions but $4f \rightarrow 4f$ transitions in Tb$^{3+}$ and Eu$^{3+}$ have simultaneously a dipolar and a quadric polar character. Therefore two possible transfer mechanisms for Ce$^{3+}$ to Tb$^{3+}$ energy transfer are dipole-dipole and dipole- quadrupole interactions may be present. In double phosphates, the presence of PO$_4^{3-}$ groups, in which P-O bonds are strongly covalent, leads to relatively weak crystal field at the rare earth sites. The covalent nature of Ce-O bond is sufficient to shift the Ce$^{3+}$ emission in these phosphates towards the higher wavelength. This results are in overlapping of Ce$^{3+}$ emission (330–470 nm) and Tb$^{3+}$ excitation (250–290) spectrum. Thus, it is possible to sensitize Tb$^{3+}$ emission by Ce$^{3+}$ emission. We got a strong intensity for $^5D_4 \rightarrow ^7F_5$.
From the above graphs it is observed that many peaks are with less intensity at 364, 381, 415, 438, 470, 614, 622nm and with good intensity 488, 545, 589, 594nm. Out of these the two peaks at 364 and 470nm are due to electron transitions of Ce" 5D3 "→" 7F2 , 5D4 "→" 7F4 and 5D0 "→" 7F1 . The red emission transition 5D0 "→" 7F2 of Tb3+ ions in LaPO4: Eu(0.5%), Tb(1%), Ce(2%) is less over that of 5D3 "→" 7F2 because the presence of Eu(0.5%) in LaPO4: Tb(1%), Ce(2%) sensitizes the basic emissions of Tb3+ as well as emits its own emissions in Eu3+ state. The PL characteristics are concern the present studied phosphors can be used as phosphor in CFL’s. Because the results obtained are comparable with the commercially available phosphors.

CONCLUSION:
LaPO4: Eu, Tb, Ce phosphors were successfully synthesized by using solid state diffusion reaction method at low temperature. The main peak in XRD pattern was found around 28.6° corresponding to a d-value of about 3.15Å, followed by other less intense peaks corresponds to the monoclinic system of crystal structure of Lanthanum Phosphate. The calculated crystallite size for LaPO4: Eu(0.5%), Tb(1.0) & Ce(2.0mol%) phosphors is 73.14 nm, and from the XRD study it conforms the formation of phosphor is mostly in single phase. This method is easy for the preparation of Eu, Tb and Ce doped LaPO4 phosphors and can be potentially applied to the synthesis of other high quality rare earth ions doped phosphate phosphor materials with micro/nano structure. LaPO4: Eu, Tb, Ce phosphor powder was successfully synthesized using a modified solid state method. LaPO4: Eu, Tb, Ce phosphor shows green, orange-red emission under 254nm excitation. The photoluminescence study shows that the emissions from electric dipole transition (5D0 "→" 7F2) is less over that of magnetic dipole transition (5D0 "→" 7F1). The optimum concentration of Eu3+ in LaPO4: Eu, Tb, Ce was 2 mol%. The PL spectra also show a strong emission at 545nm (5D0 "→" 7F1) of Tb3+. The LaPO4: Eu, Tb, Ce phosphors can be easily applied in various types of lamp and display due to its good PL performance. The results indicated that present phosphor could find application in white light emitting CFLs.

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