Optoelectronic characterization of Eu$^{3+}$ doped MLa$_2$O$_4$ (M = Sr, Ca, Mg) nanophosphors for display devices

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Abstract: Eu$^{3+}$ doped MLa$_2$O$_4$ (M = Mg, Ca, Sr) nanophosphors were synthesized by a rapid facile gel combustion route. Luminescence properties of these prepared nanophosphors were analyzed by their excitation and emission spectra. The excitation spectrum consisted of some peaks in the 350–410 nm range due to the f-f transitions. The emission spectra of prepared nanophosphors had transitions of Eu$^{3+}$ ions i.e. $^5$D$_0$ $\rightarrow$ $^7$F$_0$ (580 nm), $^5$D$_0$ $\rightarrow$ $^7$F$_1$ (594–596 nm), $^5$D$_0$ $\rightarrow$ $^7$F$_2$ (614–618, 628–629 nm), and $^5$D$_0$ $\rightarrow$ $^7$F$_3$ (650–651 nm). The main emission peak was observed at 614–618 nm of $^5$D$_0$ $\rightarrow$ $^7$F$_2$ transitions of Eu$^{3+}$ ions. The enhancement in optical properties was observed when materials were reheated at higher temperatures. The nanostructural morphology was confirmed with scanning as well as transmission electron microscopy. The prepared materials were having size in the range of 10–50 nm. X-ray powder diffraction (XRD) technique was used to determine the crystal structure and phase of the prepared phosphor materials. XRD measurements revealed that the crystallinity of MLa$_2$O$_4$ materials increased with increasing the sintering temperature. The prepared materials had bright red emitting optical properties that could be suitably applied in various display devices.

Subjects: Chemistry; Engineering & Technology; Material Science; Physics

Keywords: nanophosphor; MLa$_{2-z}$O$_4$:zEu$^{3+}$; f-f transitions; luminescence; combustion technique

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PUBLIC INTEREST STATEMENT
Europium-doped mixed oxide has received particular attention due to their efficient optical properties. These materials have bright red emission under the UV light source. Generally all the prime colors are important for the generation of the full color. This prepared series of compounds emit red luminescence (614–618 nm) that could be proficiently used for the generation of the white light for the various optoelectronic display applications.
1. Introduction

Technologically, rare earth-doped oxides nanophosphors are the strong motivators for the research due to their outstanding thermochemical and optoelectronic properties (Gouteron, Michel, Lejus, & Zarembowitch, 1981; Morais, Scalvi, Cavaleiro, Tabata, & Oliveira, 2008). The electronic structure of trivalent europium ion when used as an activator has a non-degenerate ground state $^7F_0$ which is well separated from the first excited $^5D_0$ multiplet (Venkatramu et al., 2010), providing unique properties to various host lattices (García-Hipólito et al., 2003; Joffin, Dexpert-Ghys, Verelst, Baret, & Garcia, 2005; Quan, Wang, & Lin, 2005). The emission spectrum of Eu$^{3+}$ ion is generally not affected by the influence of the surrounding due to the shielding effect of 5s, 5p electrons (Zhang, Lü, Xiu, & Wang, 2007). Hence, due to this excellent property of Eu$^{3+}$ ion, it is used as an activator for the synthesis of red phosphors having outstanding properties (Cong et al., 2008; Davolos, Feliciano, & Pires, 2003; Dhanaraj, Geethalakshmi, & Jagnanathan, 2004; Fragoso, de Mello Donégá, & Longo, 2003; Joly, Chen, Zhang, & Wang, 2007; Sun, Qi, Lee, & Lee, 2004). These Eu$^{3+}$ ions-doped materials have been used as various display materials (Rambabu, Khanna, & Rao, 1998; Rambabu, Mathur, & Buddhudu, 1999; Ronda, 1997; Tanner & Wong, 2004). Thermoluminescence, electroluminescence, and cathodoluminescence properties of these materials are significantly applied in radiation detector, display material as field emission and electroluminescent display (FED, ELD) and cathode ray tube (CRT) (Oshio et al., 1999; Ravichandran, Roy, & White, 1997; Shea, McKittrick, & Lopez, 1996).

La$_2$O$_3$ is renowned as an outstanding host lattice for rare earth-doped oxide phosphors due to their good photoluminescence properties and moderately low cost. RE ions-activated metal lanthanates phosphors having general formula MLn$_2$O$_4$ (where M = Sr, Ba, and Ln = Y, La, Gd) have been proved outstanding phosphor materials (Yang, Xiao, Ding, Yang, & Wang, 2009; Zhou, Shi, & Gong, 2007). Different synthetic methods such as spray pyrolysis (Medina, Orozco, Hernandez, Hernandez, & Falcony, 2011), precipitation (Gunawidjaja, Diez-y-Riega, & Eilers, 2015), sol–gel Pechini method (Méndez et al., 2010), glycine nitrate solution combustion synthesis (Shang, Jiang, Shang, Li, & Zhao, 2011), etc. are used for the preparation of lanthanates-based phosphor materials. In our present work, we have synthesized and investigated the luminescence properties of these nanocrystalline MLa$_2$O$_3$:Eu$^{3+}$ (M = Sr, Ca, Mg) phosphors with facile rapid combustion synthesis process. The prepared phosphors are having very fine particle size because of the released heat energy and gases of exothermic reaction of fuel and metal nitrates (Lou & Chen, 2008). The synthesized series of phosphors are characterized by PL, X-ray powder diffraction (XRD), SEM, and TEM analysis. The synthesized nanomaterials are having better optical properties than the La$_2$O$_3$:Eu$^{3+}$ that could be effectively used in various display devices.

2. Experimental details

2.1. Syntheses of nanomaterials

High-purity chemicals [Eu(NO$_3$)$_3$.6H$_2$O], [M(NO$_3$)$_2$.xH$_2$O] (M = Sr, Ca, Mg), [La(NO$_3$)$_3$.6H$_2$O] and hexamethylenetetramine [C$_6$H$_{12}$N$_4$] were taken as raw materials. Nanophosphors having general formula MLa$_{2-z}$O$_4$:zEu$^{3+}$ (where z = 1–5 mol%) were synthesized (Singh et al., 2015) by combusting a hydrated concentrated mixture having a considered stoichiometric amount of metal nitrates and hexamethylenetetramine as fuel. The amount of hexamethylenetetramine fuel was calculated by considering oxidizing and reducing valencies (Ekambaram & Patil, 1997). All these chemicals were mixed with deionized water and heated on hot plate till a semisolid paste was prepared. This gel mixture was then subjected to muffle furnace set already at 500°C. The material underwent rapid dehydration followed with degradation and production of numerous gases. These combustible gases ignited and burnt rapidly leaving a fluffy white colored fine powder. The solid powder obtained was reheated at 750 and 950°C for 1 h to study its effect on materials (Figure 1). The equation for the synthesis of this series of nanophosphor is shown as:

$$M(NO_3)_3 \cdot xH_2O + (2 - z)La(NO_3)_3 \cdot 6H_2O + zEu(NO_3)_3 \cdot 6H_2O + [C_6H_{12}N_4] \rightarrow MLa_{2-z}O_4zEu^{3+} + \text{gaseous products and water}$$
where $M$ may be Sr/Ca/Mg, $x$ is the number of moles of water in accordance to the metal nitrates and $z$ is the number of moles of Europium nitrates ($z$ varies from 1 to 5 mol%).

### 2.2. Instrumentation

Nanophosphors were characterized by X-ray diffraction profiles of the samples by the use of Rigaku Ultima IV X-ray diffractometer with Cu–Kα radiation for 2$\theta$ = 20–80°. Transmission electron micrographs (TEM) were taken using the Hitachi F-7500 transmission electron microscope. Scanning electron microscopy was carried out using JEOL JSM-6360LV scanning electron microscope. Emission and excitation spectra were measured with a Fluorimeter SPEX Fluorolog 1680 (USA) equipped with the SPEX 1934 D phosphorimeter having Xenon lamp as excitation source at room temperature in the UV–Visible region. All the characterizations were done at room temperature.

### 3. Results and discussion

#### 3.1. Optical characterization

MLa$_2$O$_4$:Eu$^{3+}$ ($M$ = Mg, Ca, Sr) materials show different peaks (350–410 nm range) in their excitation spectra. These excitation lines are due to $^7\!F_{0,1} \rightarrow ^{5}D_{4}$ (360–362 nm), $^7\!F_{0,1} \rightarrow ^{5}L_{7}$ (377–380 nm), and $^7\!F_{0,1} \rightarrow ^{5}L_{6}$ (394–396 nm) transitions (f–f) of Eu$^{3+}$ ion. The $^7\!F_{0,1}$ to $^5L_{6}$ at 394–396 nm is observed as the strongest absorption for the synthesized materials. The emission spectra of MLa$_2$O$_4$:Eu$^{3+}$ upon excitation with 395 nm show a series of emission lines in visible red region as shown in Figures 2–5. These emission lines are assigned to $^5\!D_{0} \rightarrow ^{7}F_{J}$ ($J$ = 0–3) transitions of the 4f$^6$ configuration of Eu$^{3+}$ ion present in their lattices. All the emission peaks are positioned in 560–660 nm range and the most intense emission band is observed at around 613–618 nm ascribed to the $^5\!D_{0} \rightarrow ^{7}F_{2}$ transition of Eu$^{3+}$ ion for prepared series of nanophosphors (MLa$_2$O$_4$:Eu$^{3+}$). The orange emission is observed at 595 nm which appears due to the magnetic dipole $^5\!D_{0} \rightarrow ^{7}F_{1}$ transition of europium ion and this transition rarely varies with the crystal field.
The emission at 614–616 and 628–629 nm is ascribed to the electric dipole transition of $^5D_0 \rightarrow ^7F_2$ transition of Eu$^{3+}$, and this transition depends on the crystal field (Liu & Wang, 2007) of the prepared lattice. As excitation and emission overlay graphs (Figure 5) are showing the maximum intensity of emission in Eu$^{3+}$-doped SrLa$_2$O$_4$ lattice.

Luminescence properties of nanophosphors frequently depend on activator concentration and crystallinity of the material. Luminescence intensity varies with Eu$^{3+}$ ions concentration in MLa$_2$O$_4$:Eu$^{3+}$ samples ($z = 1$–5 mol%), synthesized at 500°C which are presented in Figures 2(b), 3(b), 4(b). It is analyzed that emission intensity in prepared host lattice increased with the increase of europium ion concentration and reached at maximum when $z$ is doped up to 4 mol% in the host lattice. Further increase in activator concentration results in overdoping, which with further increase of non-radiative energy transfer between adjacent Eu$^{3+}$ ions, resulting in lowering of their luminescence intensity (Blasse & Grabmaier, 1994). Figures 2(a), 3(a), 4(a) show the emission spectra of MLa$_2$O$_4$ doped with 4 mol% of europium ions, synthesized at 500°C, sintered at 750 and 950°C temperatures, upon excitation at 395 nm. The increase in the intensity of emission spectral lines is accredited to the increase of crystallinity of nanophosphors particles with the increase of sintering temperature.
The powders sintered at high temperatures show better red emission and this intensity of red color also increased with increase in dopant concentration. The best sample is observed when concentration of dopant is taken 4 mol% and calcined at 950°C. This is well described by color coordinates given in Table 1. The ratio $R$ of the intensities $I(\mathbf{D}_0 \rightarrow \mathbf{F}_1)/I(\mathbf{D}_0 \rightarrow \mathbf{F}_2)$ provides the percentage purity of the red luminescent color of phosphors. As confirmed by XRD, an additional phase La$_2$O$_3$ along with the main MLa$_2$O$_4$ phase is also present. These two phases in the lattice resulted into crystal field splitting of $\mathbf{D}_0 \rightarrow \mathbf{F}_2$ into two lines at 613–618 nm and 628–629 nm. Figures 2–5 reveal that the intensity of $\mathbf{D}_0 \rightarrow \mathbf{F}_2$ transitions is more; as a result, $R > 1$ is obtained. So the red color is more prominent in MLa$_2$O$_4$Eu$^{3+}$ phosphors than the La$_2$O$_3$:Eu$^{3+}$.

CIE invented $\bar{x}(\lambda)$, $\bar{y}(\lambda)$, and $\bar{z}(\lambda)$ [three color corresponding functions] subsequent to three primary colors i.e. blue, green, and red. By the use of these three functions, calorimetric coordinates ($x$, $y$) are calculated from the tristimulus value as shown in Equations (1) and (2).

$$x = \bar{x}(\lambda)/\bar{x}(\lambda), \bar{y}(\lambda), and \bar{z}(\lambda)$$

(1)

$$y = \bar{y}(\lambda)/\bar{x}(\lambda), \bar{y}(\lambda), and \bar{z}(\lambda)$$

(2)

Figure 3. Photoluminescence spectra of CaLa$_2$O$_4$:Eu$^{3+}$ excited at 395 nm. (a) Temperature variation (b) Concentration variation.
Figure 4. Photoluminescence spectra of MgLa$_2$O$_4$:Eu$^{3+}$ excited at 395 nm. (a) Temperature variation (b) Concentration variation.

Figure 5. Photoluminescence spectra of La$_2$O$_3$:Eu$^{3+}$ and MLa$_2$O$_4$:Eu$^{3+}$ (M = Sr, Ca, Mg) nanophosphor showing variation of metal ions in host lattice.
The calculated values of color co-ordinates are shown in Table 1 corresponding to red color of the visible region on the color gamut calculated from the emission spectra of MLa$_2$O$_4$:Eu$^{3+}$ nanophosphor prepared at various annealing temperatures and at different concentrations. The color co-ordinates of MLa$_2$O$_4$:Eu$^{3+}$ (0.04 mol) materials calcined at 950°C are shown in color triangle in Figure 6. Samples heated at 750 and 950°C show the CIE co-ordinates in the intense red color region of the color triangle.

### 3.2. Morphological study of the phosphor

The morphology of Eu$^{3+}$-doped MLa$_2$O$_4$ nanoparticles was studied by scanning electron micrographs and transmission electron micrographs. The surface morphology of Eu$^{3+}$ (4 mol%)-doped MLa$_2$O$_4$ nanoparticles prepared by present facile combustion method were studied using SEM. SEM photomicrographs of MLa$_2$O$_4$:Eu$^{3+}$ samples are presented in Figures 7(a, b, c). The SEM micrographs showed that as-synthesized phosphors have spherical agglomerated nanocrystalline particles. As in combustion synthesis, numerous gasses evolved within very short period that generally produced nanosized materials with pores and

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**Table 1. Color co-ordinates of phosphors at varying concentrations and at varying calcination temperatures**

| Phosphor compounds      | Color co-ordinates |
|-------------------------|--------------------|
|                         | 500°C             | 750°C             | 950°C             |
| SrLa$_2$O$_4$:Eu$^{3+}$ (0.01) | x-0.5086          | x-0.5178          | x-0.5245          |
|                         | y-0.2878          | y-0.2838          | y-0.2829          |
| SrLa$_2$O$_4$:Eu$^{3+}$ (0.02) | x-0.5184          | x-0.5250          | x-0.5363          |
|                         | y-0.2868          | y-0.2824          | y-0.2804          |
| SrLa$_2$O$_4$:Eu$^{3+}$ (0.03) | x-0.5354          | x-0.5463          | x-0.5563          |
|                         | y-0.2846          | y-0.2831          | y-0.2816          |
| SrLa$_2$O$_4$:Eu$^{3+}$ (0.04) | x-0.5446          | x-0.5856          | x-0.5909          |
|                         | y-0.2783          | y-0.2734          | y-0.2723          |
| SrLa$_2$O$_4$:Eu$^{3+}$ (0.05) | x-0.5261          | x-0.5310          | x-0.5423          |
|                         | y-0.2853          | y-0.2843          | y-0.2807          |
| CaLa$_2$O$_4$:Eu$^{3+}$ (0.01) | x-0.5289          | x-0.4335          | x-0.4382          |
|                         | y-0.2838          | y-0.2765          | y-0.2766          |
| CaLa$_2$O$_4$:Eu$^{3+}$ (0.02) | x-0.5301          | x-0.5376          | x-0.5423          |
|                         | y-0.2786          | y-0.2698          | y-0.2665          |
| CaLa$_2$O$_4$:Eu$^{3+}$ (0.03) | x-0.5324          | x-0.5423          | x-0.5489          |
|                         | y-0.2764          | y-0.2709          | y-0.2679          |
| CaLa$_2$O$_4$:Eu$^{3+}$ (0.04) | x-0.5347          | x-0.5434          | x-0.5567          |
|                         | y-0.2738          | y-0.2643          | y-0.2635          |
| CaLa$_2$O$_4$:Eu$^{3+}$ (0.05) | x-0.5310          | x-0.5406          | x-0.5468          |
|                         | y-0.2745          | y-0.2712          | y-0.2658          |
| MgLa$_2$O$_4$:Eu$^{3+}$ (0.01) | x-0.5235          | x-0.5307          | x-0.5398          |
|                         | y-0.2769          | y-0.2669          | y-0.2650          |
| MgLa$_2$O$_4$:Eu$^{3+}$ (0.02) | x-0.5276          | x-0.5328          | x-0.5409          |
|                         | y-0.2749          | y-0.2726          | y-0.2713          |
| MgLa$_2$O$_4$:Eu$^{3+}$ (0.03) | x-0.5293          | x-0.5346          | x-0.5476          |
|                         | y-0.2736          | y-0.2702          | y-0.2649          |
| MgLa$_2$O$_4$:Eu$^{3+}$ (0.04) | x-0.5321          | x-0.5387          | x-0.5439          |
|                         | y-0.2766          | y-0.2734          | y-0.2665          |
| MgLa$_2$O$_4$:Eu$^{3+}$ (0.05) | x-0.5289          | x-0.5327          | x-0.5412          |
|                         | y-0.2735          | y-0.2716          | y-0.2709          |
voids. So it is analyzed from SEM images that the powders show large voids, cracks, and pores. The TEM Figures 8(a, b, c) are showing highly agglomerated nanoparticles with an average size of ~10–50 nm. TEM analysis has provided further additional microstructural details of these nanostructures. Furthermore, the crystallite size according to Scherrer's equation is much closer to TEM observation.

3.3. X-ray diffraction study
The powder XRD patterns of the MLa$_2$O$_4$:Eu$^{3+}$ (M = Sr, Ca, Mg) nanophosphors prepared by facile combustion synthesis at 500°C, calcined at 750 and 950°C, are shown in Figures 9–11. By using JCPDS data, multiphase components are observed at 500°C because of the occurrence of cubic La$_2$O$_3$ phase attributed as JCPDS No. 005-0602. In SrLa$_2$O$_4$:Eu$^{3+}$, numerous supplementary peaks consequent to
Figure 9. XRD patterns of synthesized SrLa$_2$O$_4$:Eu$^{3+}$ phosphors.

Figure 10. XRD patterns of synthesized CaLa$_2$O$_4$:Eu$^{3+}$ phosphors.

Figure 11. XRD patterns of synthesized MgLa$_2$O$_4$:Eu$^{3+}$ phosphors.
Sr(NO₃)₂ phase (JCPDS No. 004-0310) are also found. The XRD patterns of SrLa₂O₄:Eu³⁺ powder sintered at 750 and 950°C are assigned to SrLa₂O₄ phase matched with JCPDS card No. 042-0343. XRD pattern of sintered CaLa₂O₄ nearly matched with SrLa₂O₄ but no further records are found for CaLa₂O₄ and MgLa₂O₄. The as-prepared CaLa₂O₄ and MgLa₂O₄ samples also showed many additional peaks for M(NO₃)₂ and cubic La₂O₃ phase. But with the increase of calcination temperature, these additional peaks diminished. So it appears that 500°C was not an adequate temperature for the degradation of M(NO₃)₂ to prepare pure MLa₂O₄ phase. It is observed from XRD graphs that the intensities of peaks increased with increase of sintering temperature, which specify the improvement of crystallinity and also assign the increase in size of particles. With the increase of temperature of calcination, the intensity of XRD pattern gets increased and full with half the maximum of peaks slightly decreased. All this indicated the enhancement in crystalline nature of the prepared compounds. Eu³⁺ ions (0.95 Å) replaced La³⁺ (1.061 Å) ions in the host lattices instead of Mg²⁺ (0.72 Å), Ca²⁺ (0.99 Å) and Sr²⁺ (1.12 Å) ions. Here due to both size and ionic charge issues, it is difficult for Eu³⁺ to replace M²⁺ ions, though size of Ca²⁺ is nearly same to Eu³⁺, but here also charge compensation issue predominate. Because the differences in sizes of ionic radii of La³⁺ and Eu³⁺ is small and the charges of both ions are also same. Therefore, Eu³⁺ ions easily substitute La³⁺ ions.

The average crystallite sizes (D) are calculated using Scherrer’s formula from the line broadening obtained from the X-ray powders.

\[ D = \frac{K \lambda}{\beta \cos \theta} \]

where, “K” is constant, “\( \lambda \)” is wavelength of X-rays, and “\( \beta \)” is FWHM.

Tables 2–4 provided the detailed description related to size of particles and phase of prepared phosphors.

**Table 2. Detailed description of size and phase of particle of SrLa₂O₄:Eu³⁺**

| Nanophosphor | SrLa₂O₄:Eu³⁺ |
|--------------|--------------|
| Temperature°C | 500 | 750 | 950 |
| Phase | Major La₂O₃ + Minor La₂SrO₅ | La₂SrO₅ | La₂SrO₅ |
| 2θ value | 18.32 | 30.06 | 30.06 |
| FWHM (radian) | 0.02067 | 0.01135 | 0.00697 |
| Particle size (nm) | 7.07 | 14.12 | 19.91 |

**Table 3. Detailed description of size and phase of particle of CaLa₂O₄:Eu³⁺**

| Nanophosphor | CaLa₂O₄:Eu³⁺ |
|--------------|--------------|
| Temperature°C | 500 | 750 | 950 |
| Phase | Major La₂O₃ matches with JCPDS No. 005-0602 | Nearly matched with XRD of SrLa₂O₄:Eu³⁺ | No further records are found up to now |
| 2θ value | 29.498 | 30.06 | 30.06 |
| FWHM (radian) | 0.016048 | 0.01186 | 0.00942 |
| Particle size (nm) | 9.927 | 13.0493 | 16.0228 |

**Table 4. Detailed description of size and phase of particle of MgLa₂O₄:Eu³⁺**

| Nanophosphor | MgLa₂O₄:Eu³⁺ |
|--------------|--------------|
| Temperature°C | 500 | 750 | 950 |
| Phase | Major La₂O₃ matches with JCPDS No. 005-0602 | No records are found up to now |
| 2θ value | 29.498 | 30.036 | 30.036 |
| FWHM (radian) | 0.019223 | 0.01238 | 0.009507 |
| Particle size (nm) | 8.296 | 12.938 | 16.848 |
4. Conclusions

Structurally and morphologically well-defined nanophosphor compounds were efficiently prepared by present facile rapid gel combustion process. XRD patterns showed the presence of multiphase components at 500°C, but on increasing sintering temperature, crystallinity of phosphors increased and single phase of phosphors was obtained at 750 and 950°C. Crystallite size of MLa$_2$O$_4$:Eu$^{3+}$ phosphor particles were found below 50 nm as calculated from XRD using Scherrer equation. On increasing sintering temperature, sizes of particles were increased, which showed enhancement in the crystallinity of phosphors. Moreover, SEM and TEM characterization confirmed nanocrystalline size of prepared particles. Photoluminescence spectra obtained for all MLa$_2$O$_4$:Eu$^{3+}$ phosphors had dominant red emission peak at 613–618 nm. The photoluminescence intensity of all the prepared nanomaterials was found to be increased with the increasing of the sintering temperature. Intensity of luminescence of SrLa$_2$O$_4$:Eu$^{3+}$ nanophosphor was found maximum when sintered at 950°C. All prepared nanomaterials had tremendous red photoluminescence properties so they could be made functional in different optoelectronic display applications.

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