

Characterization and photoluminescence of $\text{Sr}_2\text{CeO}_4: \text{Eu}^{+3}, \text{La}^{+3}$

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ABSTRACT

In this paper we report the synthesis and luminescence of strontium cerium oxide ($\text{Sr}_2\text{CeO}_4:\text{Eu}^{3+}(0.5\text{mol}\%),\text{La}^{3+}(0.5\text{mol}\%)$) prepared by solid state reaction method in air at 1200°C . These samples were characterized by X-ray diffraction, scanning electron microscope (SEM) and photoluminescence (PL) techniques, particle size analysis. The photo luminescence (PL) spectra of pure and $\text{Eu}^{3+}, \text{La}^{3+}$ codoped Sr_2CeO_4 were recorded at room temperature. Sr_2CeO_4 phosphor was effectively excited around 250 nm and observed a broad emission band peak around 467 nm. XRD data revealed that the structure of Sr_2CeO_4 is orthorhombic, and the mean crystallite size of Sr_2CeO_4 and $\text{Sr}_2\text{CeO}_4: \text{Eu}^{3+}, \text{La}^{3+}$ were 9 nm and 11nm respectively. The colour co-ordinates of Sr_2CeO_4 were $x = 0.16$ and $y = 0.18$ and CIE co-ordinates revealed that the present phosphors may be used in white light applications. Copyright © 2013 VBRI press.

Keywords: Phosphor; solid state reaction; X-ray diffraction; photoluminescence; SEM; CIE; particle size analysis.



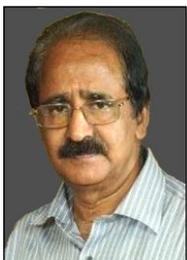
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Introduction

The search for blue phosphor emitters has been increasing due to their applicability in many fields, such as cathode ray tubes, projection televisions, fluorescent tubes, X-ray detectors and field emission displays [1]. Even in the paper industry, fluorescent dyes absorb ultraviolet light and emit blue color are widely used as organic optical brightening agents [2]. White light-emitting diodes (W-LEDs) offer benefits such as high luminous efficiency, low energy consumption, long lifetime, and environment friendly and so on. They are tipped to be the next generation solid state lighting, in the replacement of conventional incandescent and fluorescent lamps which are Hg pollutants, frangible and high energy consumption [3]. In the recent years, research on the phosphors used for white LEDs has become a hot topic and gained maturity. A large number of papers have been published in the related journals about the successful synthesis and luminescence of rare earth phosphors with the orientation of exploring novel phosphors suitable for solid state lighting [4]. Recently, Rao and coworkers reported synthesis and characterization of nano Sr₂CeO₄ doped with Eu and Gd phosphor [5] and some rare earth doped alkali earth sulfide phosphors for white-light LEDs [6]. Also Ping Zhang et al. reported some new methods for the preparation of phosphate based phosphor, which are used in the commercial application [7]. Sr₂CeO₄ crystals doped with rare-earth elements have been studied since an unusual blue phosphor Sr₂CeO₄ was prepared using the combinatorial techniques by Danielson et al. in 1998. Since then, lots of scientists have taken great interest in studying the special structure and fluorescent properties of this novel material [8-13]. Due to its broad emission band, Sr₂CeO₄ can be used as the matrix material, which may provide a new method to search for future attracting luminescence hybrids. Until now, the Sr₂CeO₄ lattice is the only host lattice in which charge-transfer (CT) luminescence of Ce⁴⁺ has been observed at room temperature [14]. Sr₂CeO₄ crystals doped with rare-earth elements have been studied earlier [15-17].

According to the literatures, the luminescence properties of rare-earth doped Sr₂CeO₄ have been affected by energy transfer from the triplet excited state of the metal to ligand charge transfer (MLCT) state for Sr₂CeO₄ to the rare-earth ions. Concerning many of these applications, the availability of systems consisting of uniform particles in size and shape is also an essential prerequisite for improved performance, and new synthetic routes are been developed in order to reach these systems [18-22]. This material has been found to exhibit luminescence under excitation with cathode and X-rays. In addition, it has also been established that Sr₂CeO₄ exhibits photoluminescence under excitation with irradiation of ultraviolet rays [23-25]. Therefore, it has been attracted that Sr₂CeO₄ has good potential for application as a blue phosphor in lamps and in field emission displays. The luminescence associated with Eu³⁺, La³⁺ contained in different host lattices has found applications related to its red light emission which is important in the fields of displays, sensors and lasers. The past few decades have seen a lot of work reported on the use of divalent/trivalent europium as a dopant in phosphors, as they have very good optical properties in the blue to red regions which make them part of many display devices.

Among all the rare-earth ions, Eu³⁺, La³⁺ is the most extensively studied owing to the simplicity of its spectra and also its use in commercial red phosphors. The luminescence spectrum of Eu³⁺, La³⁺ reveals spectroscopic transitions from the visible to the near-infrared region.

In this research article, we have studied on the synthesis, size, morphology and photoluminescence properties of pure and Eu³⁺, La³⁺ codoped Sr₂CeO₄ phosphors prepared by solid state reaction method in air at 1200°C. The prepared materials were characterized by XRD, SEM, Photoluminescence techniques. It is investigated the mean particle size from particle size distribution histograms of the pure and Eu, La (0.5mol %) co-doped Sr₂CeO₄ phosphor particles. The chromatic coordinates (x, y) of prepared materials was calculated with Color Calculator version 2, software from Radiant Imaging. PL studies and CIE co-ordinates of Eu:La(0.5mol%) co-doped Sr₂CeO₄ phosphor reveals that the emission colour varies from blue to white. This phosphor has a good potential for application in white light LED's.

Experimental

Materials

Sr₂CeO₄, pure and doped with Eu³⁺ and La³⁺ ions were synthesized by the conventional solid state reaction method. Strontium Nitrate Sr(NO₃)₂ assay (99.995%) Sigma-Aldrich Chemie, Inc, Germany, cerium oxide (CeO₂) assay (99.5%), europium oxide (Eu₂O₃) assay (99.9%) and lanthanum oxide (La₂O₃) assay (99.99%) National Chemicals, Nutan Gujarat Industrial Estate, Vadodara, India were used as starting materials to prepare Sr₂CeO₄ and Eu³⁺, La³⁺ doped phosphors. A stoichiometric mixture of these starting powders was thoroughly homogenized in an agent mortar for 1hr and then put into an alumina crucible. The homogenized mixture was heated in air at 1200°C for 3h in a muffle furnace with a heating rate of 5°C/min. Finally the samples were allowed to cool down to room temperature for about 20h. Sr₂CeO₄ codoped with rare earth ions Eu³⁺, La³⁺ of 0.5 mol% each. In the present investigation, materials are prepared according to the chemical reaction,



Synthesis and characterization

This synthesis route is very easy and does not require expensive as well as sophisticated equipments. The major advantage of SSR method is, the final product in solid form is structurally pure with the desired properties depending on the final sintering temperatures. This method is environment friendly and no toxic or unwanted waste is produced after the SSR is complete. In this process the powders produced from SSR method is very fine as well as the cross contamination is very less. This method is also very convenient for large scale production on industrial scale. Several complementary methods were used to characterize the prepared phosphors. To identify the crystal phase, X-ray diffraction (XRD) analysis was carried out with a powder X-ray diffractometer (Indus beam line-II(ADXRD BL-12), RRCAT, Indore, India). The electron source size at this port is approximately 0.5mm (H) X 0.5

mm (v). The beam acceptance of the beam line is 2mrad (H) X 0.2mrad (V). The microstructures of the samples were studied using a scanning electron microscopy (SEM) (XL 30 CP Philips). The scanning continues time is 10s, and 2θ range from 15 to 60° . The Photoluminescence (PL) emission spectra were measured by a spectrofluorophotometer (SHIMADZU, RF-5301 PC) using Xenon lamp as excitation source. All the spectra were recorded at room temperature. Emission and excitation spectra were recorded using a spectral slit width of 1.5 nm [26]. The Emission and excitation spectra of pure and Eu^{3+} (0.5mol %), La^{3+} (0.5mol %) codoped Sr_2CeO_4 phosphors were recorded for excitation at 250nm and 365nm, and for emission at 400nm.

Results and discussion

Crystal structure of Sr_2CeO_4

The typical XRD pattern of Sr_2CeO_4 is shown in **Fig. 1** and all the diffraction peaks can be well indexed based on the JCPDS No. 89-5546. All the prepared samples are characterized to be single phase Sr_2CeO_4 of orthorhombic structure. Except this, no other phase exists in the XRD spectra, indicating that the as-prepared Sr_2CeO_4 phosphor is single phase. XRD study confirms the single phase Sr_2CeO_4 with the orthorhombic structure, which agrees with the findings of previous research works of Danielson et al [27], Sankar et al [28] and Shu-Jian Chen et al [29]. By using the Scherer's formula, the calculated mean crystallite size of pure and Eu^{3+} , La^{3+} codoped Sr_2CeO_4 phosphors were around 9 nm and 11 nm, respectively.

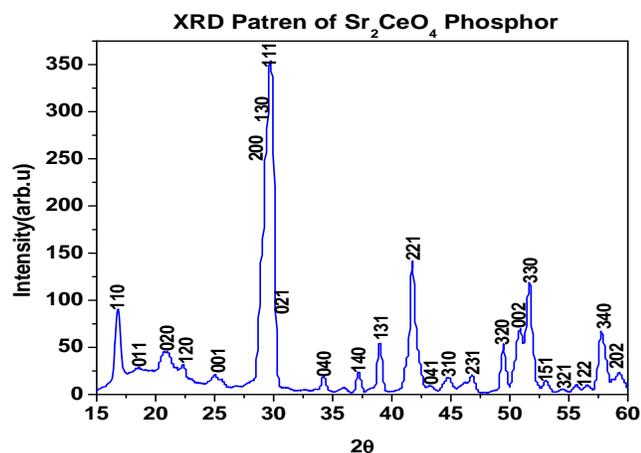


Fig. 1. X-ray powder diffraction pattern of Sr_2CeO_4 .

Morphology of Sr_2CeO_4

The SEM image of pure Sr_2CeO_4 phosphor in **Fig. 2 (a)** clearly show good dispersion and small particle size of the Sr_2CeO_4 sample at 1200°C . It indicates that the particles has accelerated aggregation and sintering. The sample exhibit highly agglomerated particles. It confirms that the calcinations temperature can be reduced in order to get a higher specific surface area and a smaller crystallite size. **Fig. 2 (b)** is the typical SEM photograph of the synthesized Sr_2CeO_4 : Eu^{3+} , La^{3+} phosphor. The multi layered structure was observed and the prominent characteristic of Sr_2CeO_4 : Eu^{3+} , La^{3+} crystal growth is the serious anisotropic growth

rate. The growth rate along the *ab* plane is ~ 5 times larger than that along the *c*-axis. This phenomenon can be easily understood from the periodic bond chain (PBC) theory. The fundamental building units are in the orthorhombic structure.

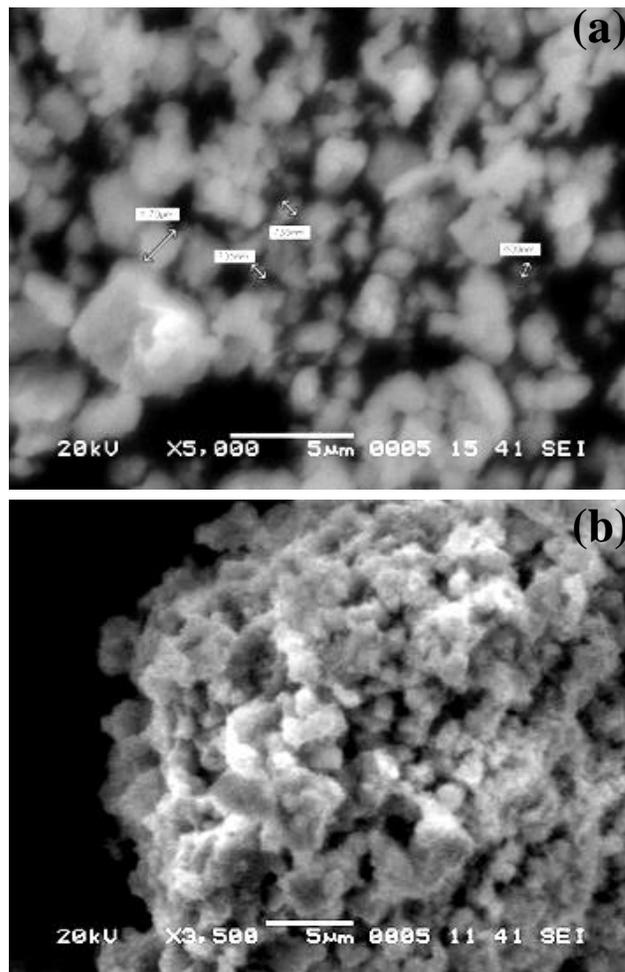


Fig. 2. (a) SEM image of pure Sr_2CeO_4 phosphor and (b) Sr_2CeO_4 : Eu^{3+} , La^{3+} phosphor.

Photoluminescence of pure and Eu^{3+} , La^{3+} co-doped Sr_2CeO_4

The excitation spectra of pure and Eu^{3+} (0.5mol %), La^{3+} (0.5mol %) co-doped Sr_2CeO_4 phosphors were monitored under 400nm wavelength are shown in **Fig. 3a**. It is observed that a broad excitation range from 220 to 370nm shows peaks around 250 and 365nm. The 365nm peak belongs to crystal field. The excitation spectrum is same for both samples and shows peaks at 250 and 365nm. But the emission spectrum of Sr_2CeO_4 shows a broad band due to $f \rightarrow t_{1g}$ transitions of Ce^{4+} . When the excitation was varied from 250 to 365 nm, the observed emission is a broad band with a peak at 470nm. This is mainly due to the charge transfer position of the $\text{Ce}^{4+} - \text{O}^{2-}$ ligand as described by Danielson et al [27]. The two excitation peaks may be assigned to the two kinds of Ce^{4+} ions present in Sr_2CeO_4 . There are two different $\text{Ce}^{4+} - \text{O}^{2-}$ bond lengths in the lattice and hence two different charge transfer transitions takes place.

The PL emission spectrum of Sr_2CeO_4 is characterized by a broad band ranged from 350 to 650 nm with a peak position at 475 nm (**Fig. 3b**), which can be ascribed to the CT transition of $\text{Ce}^{4+}-\text{O}^{2-}$. The PL spectra of Eu^{3+} (0.5 mol %), La^{3+} (0.5 mol %) codoped Sr_2CeO_4 consists of the characteristic lines of Eu^{3+} , La^{3+} corresponding to transitions from the excited $^5\text{D}_0$ level: $^5\text{D}_2 \rightarrow ^7\text{F}_{1,2,3}$, $^5\text{D}_1 \rightarrow ^7\text{F}_{1,2}$ and $^5\text{D}_0 \rightarrow ^7\text{F}_{0,1,2}$ located at 467, 490, 512, 537, 556, 587, 614 and 616 nm respectively. The red emission intensity of the transition $^5\text{D}_0 \rightarrow ^7\text{F}_2$ was the strongest, so it seems that there is no inversion symmetry at the site of the rare-earth ion Eu^{3+} . As everyone knows, if Eu^{3+} occupies in the crystal lattice a site with inversion symmetry, the magnetic-dipole transitions $^5\text{D}_0 \rightarrow ^7\text{F}_1$ of Eu^{3+} dominate and if there is no inversion symmetry at the site of Eu^{3+} , the main emission is the electric-dipole transition $^5\text{D}_0 \rightarrow ^7\text{F}_2$ [30]. The peak around 610- 620 nm is due to the electric dipole transition of $^5\text{D}_0 \rightarrow ^7\text{F}_2$. When the Eu^{3+} , La^{3+} concentration is 0.5 mol %, the broad band emission originates from Sr_2CeO_4 host itself and Eu^{3+} , La^{3+} codoped sample. The emission lines in the blue, green, and red regions coexist.

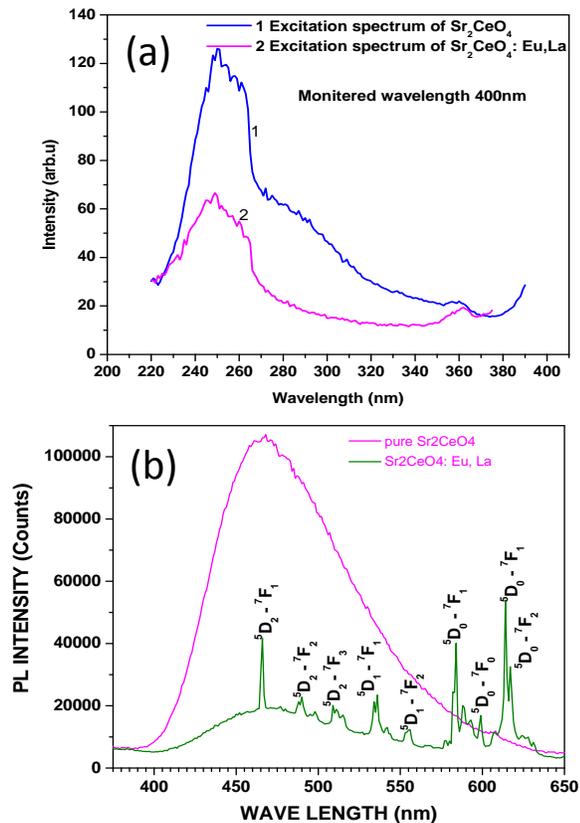


Fig. 3. (a) Excitation spectra of Sr_2CeO_4 phosphor and (b) Emission spectra of Sr_2CeO_4 : Eu^{3+} (0.5%) La^{3+} (0.5%).

Particle size analysis

The Particle size distribution histograms of the un-doped and Sr_2CeO_4 : Eu , La (0.5 mol %) co-doped particles as shown in **Fig. 4** (a) and (b). The prepared phosphor specimen particle size was measured by using laser based system Malvern Instrument U.K. The mean diameter of the particle size of pure and Eu , La codoped Sr_2CeO_4 were

26 μm and 27.28 μm respectively. And the average particle diameter and crystallite size of Eu , La doped Sr_2CeO_4 phosphor were 27 μm and the 11 nm. As such many molecular particles agglomerate and form as a crystallite and many crystallites together become a particle. In the present case approximately 0.5897 specific surface area/gm, 2000 crystallites of size of 11 nm together forms a particle with a diameter of 27 μm in Eu , La codoped Sr_2CeO_4 system.

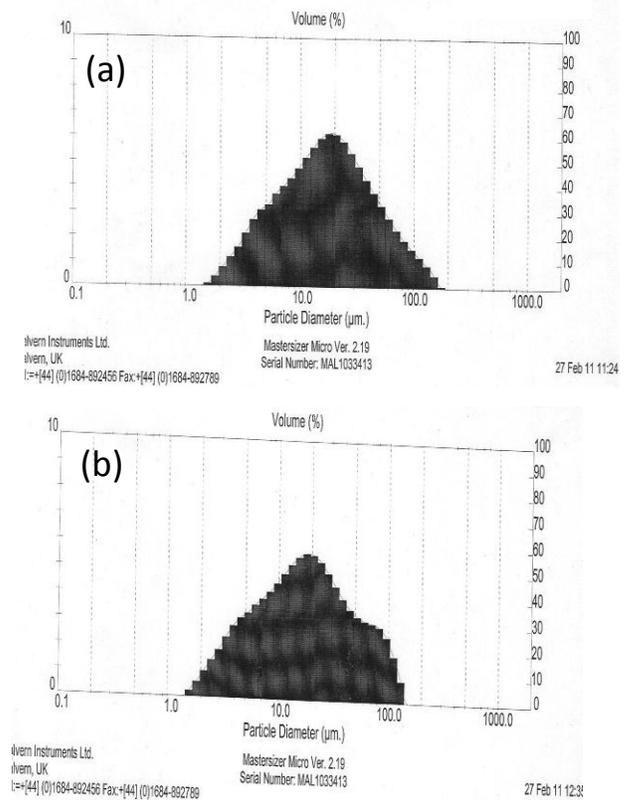


Fig. 4 (a). Particle size of pure Sr_2CeO_4 and (b) particle size of (0.5 mol %) doped Sr_2CeO_4 Eu : La .

CIE coordinates

Most lighting specifications refer to colour in terms of the 1931 CIE chromatic colour coordinates which recognize that the human visual system uses three primary colours: red, green, and blue. In general, the colour of any light source can be represented on the (x, y) coordinate in this colour space. The colour purity was compared to the 1931 CIE Standard Source C (illuminant Cs (0.3101, 0.3162)). The chromatic coordinates (x, y) were calculated using the colour calculator program radiant imaging. The dominant wavelength is the single monochromatic wavelength that appears to have the same colour as the light source. The dominant wavelength can be determined by drawing a straight line from one of the CIE white illuminants (Cs (0.3101, 0.3162)), through the (x, y) coordinates to be measured, until the line intersects the outer locus of points along the spectral edge of the 1931 CIE chromatic diagram [31-35].

All the results calculated from the emission spectra were

plotted in the Commission International de l'Éclairage (CIE) 1931 chromaticity diagram, as shown in Fig. 5. It indicates that present phosphors are close to the edge of CIE diagram, which indicates the high color purity of these phosphors. By connecting these two points as a triangle (included white light point (0.31, 0.32)) the intermediate compositions can generate warm white light with a particular ratio of this phosphor.

The colour co-ordinates for the pure Sr_2CeO_4 were $x = 0.16$ and $y = 0.18$ and Eu, La co-doped Sr_2CeO_4 phosphor were $x=0.32$ and $y=0.33$. Fig. 5 illustrates the CIE chromaticity diagram for the emissions of pure and Eu^{3+} , La^{3+} (0.5 mol %) codoped Sr_2CeO_4 . In the Fig.5, points 'A' and 'B' refers the coordinates of pure and Eu, La co-doped Sr_2CeO_4 phosphor. For the dopant concentration of Eu (0.5mol%), La (0.5mol %), the point at 'B' in the CIE chromaticity diagram is exactly in white. The emission varied from blue to white light with doping of Eu^{3+} , La^{3+} . This phosphor having excellent colour tunability from blue to white light and this phosphor has an application in the white lighting system.

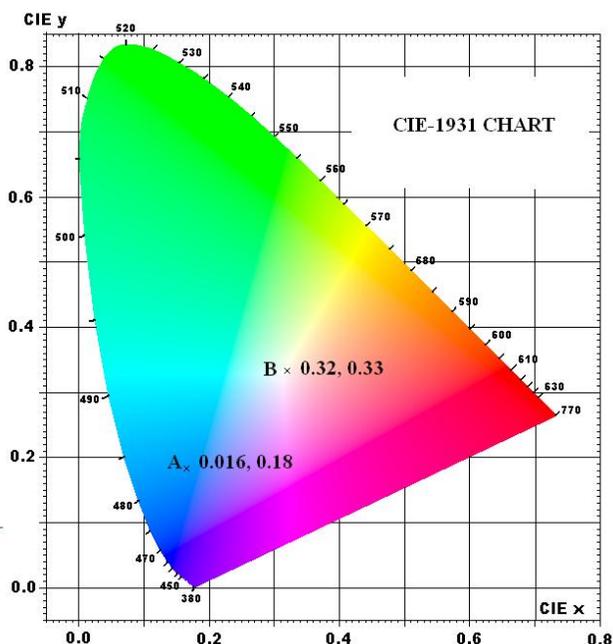


Fig. 5. CIE co-ordinates of pure and Eu^{3+} , La^{3+} codoped Sr_2CeO_4 depicted on 1931 chart.

Conclusion

Pure and Eu^{3+} , La^{3+} (0.5%) codoped Sr_2CeO_4 phosphors were successfully prepared by the solid state reaction method in the present work. The XRD spectra indicating that the as prepared Sr_2CeO_4 phosphor is single phase and confirm the structure of the system as orthorhombic. SEM image revealed that the particles have shown accelerated aggregation and sintering of Sr_2CeO_4 at 1200°C . The fundamental building units are in the orthorhombic structure. A broad emission band originates from Sr_2CeO_4 host and Eu^{3+} , La^{3+} (0.5%) emission lines in the blue, green, and red regions coexist. The emissions from the higher excited state $^5\text{D}_i$ ($i=1, 2$) apart from $^5\text{D}_0$ of Eu^{3+} ions are observed. The PL emission spectrum of pure Sr_2CeO_4 shows

a broad band due to $f \rightarrow t_{1g}$ transitions of Ce^{4+} . The two excitation peaks may be assigned to two kinds of Ce^{4+} ions present in pure Sr_2CeO_4 . There are two different $\text{Ce}^{4+}-\text{O}^{2-}$ bond lengths in the lattice and hence $^5\text{D}_2 \rightarrow ^7\text{F}_{1,2,3}$, $^5\text{D}_1 \rightarrow ^7\text{F}_{1,2}$ charge transfer transitions obtained. By studying the optical properties of codoped sample, we found that the emission transition of $^5\text{D}_0 \rightarrow ^7\text{F}_2$ intensity was the strongest among the main lines and the emission from $^5\text{D}_{1,2}$ was also observed due to the low-energy vibration of the host lattice. The Sr_2CeO_4 phosphor and $\text{Sr}_2\text{CeO}_4: \text{Eu}^{3+}$ (0.5mol%), La^{3+} (0.5mol%) shows highest PL Intensity when excited with 254nm. The $\text{Sr}_2\text{CeO}_4: \text{Eu}^{3+}$ (0.5%), La^{3+} (0.5%) emission could be tuned from blue to white and then to red light. The mean diameter of the particle size of pure and Eu,La codoped Sr_2CeO_4 were $26\mu\text{m}$ and $27.28\mu\text{m}$. The average particle diameter and crystallite size of Eu, La doped Sr_2CeO_4 phosphor were $27\mu\text{m}$ and the 11nm . The Commission International de l'Éclairage co-ordinates were $x = 0.16$ and $y = 0.18$ for the pure Sr_2CeO_4 phosphor and $x = 0.32$ and $y = 0.33$ for the Eu:La(0.5%) co-doped Sr_2CeO_4 phosphor. PL studies and CIE co-ordinates revealed that the Eu:La(0.5%) co-doped Sr_2CeO_4 phosphor can be a good blue emitting phosphor material for lamps and white light emitting phosphor for many display devices.

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