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RESEARCH ARTICLE

ENHANCEDLUMINESCENCE PROPERTIES OF EU3+DOPED SR2CEO4 PHOSPHOR

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ABSTRACT

In this work, Enhanced Luminescence properties of Eu^{3+}doped Sr_2CeO_4 phosphor were prepared by Conventional Solid State Reaction (SSR) method. The obtained phosphors were characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Photoluminescence (PL) and The Commission International de l'Eclairage co-ordinates (CIE). XRD reveals that except Sr_2CeO_4, no other phase exists in the XRD spectra. SEM image is exhibits the grains like morphology with different sizes and shapes. PL emission spectrum of un-doped Sr_2CeO_4 phosphor shows broad emission from 350 – 650nm. This broad band is due to f→t_1g transition of Ce^{4+}. The emission spectra of Eu^{3+}doped Sr_2CeO_4 phosphor with(0.5mol%) with citric acid($C_6H_8O_7$) as flux shows peaks at 467, 491, 512, 537,557, 585, 590, 601, 611 and 616nm under 254 & 270nm excitation. The CIE co-ordinates were calculated by the Spectrophotometric method using the spectral energy distribution of un-doped Sr_2CeO_4 and Eu^{3+}doped Sr_2CeO_4phosphor with (0.5mol %) were analysized.

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INTRODUCTION

In addition, the rare-earth oxides have been perceived as a prominent replacement for currently available luorescent probes used in the fluorescent displays and magnetic resonance imaging (MRI) namely organic dyes, rare-earth doped oxysulfide, vanadates, ferrites and chelates [1-3]. Rare-earth luminescent materials exhibit a wide band gap and excellent luminescence properties arising from their intra 4f transitions, large anti-stokes shifts and stokes shifts (up to 500 nm). Then they have been widely used in several of areas, like biomarker, military, nuclear physics, radiation and so on. Especially, rare earth ions activated Strontium ceriumoxide (Sr_2CeO_4) has advantage of high light absorption, high energy transfer efficiency [4-6], high X-ray blocking ability, short attenuation time, good irradiation stability and good scintillation properties. Therefore, they make contribution in plasma display panel, X-ray phosphor, X-CT, positron emission computed tomography (PET), medical radiotherapy linear accelerator and so on. In recent years, rare earth luminescent materials with micro and nano hollow structure have arose far-reaching attention, so that it can contain a large number of guest molecules or large-size guest molecules [7]. Because of its high surface activity and good surface permeability, hollow sphere is widely used in drug release, magnetic storage materials, dye precipitation and catalytic applications [8]. For instance, a magnetic core can be covered with a porous silica shell that can be loaded with drugs and provide biocompatibility. From this point of view, no matter in terms of radiation efficiency, decay time, chemical stability or radiation damage resistance, Sr_2CeO_4 is a good host matrix suitable for doping rare earth ions to fabricate phosphors. Sr_2CeO_4 activated by trivalent europium ions (Sr_2CeO_4:Eu^{3+}) can effectively improve the luminescent center and make them become important red phosphors [9-12].

There is growing interest in the development of new full color emitting phosphor materials that combine thermal and chemical stability in air with high emission quantum yield at room-temperature. The search for blue phosphor emitters has been increasing due to their applicability in many fields, such as cathode ray tubes (CRTs), projection televisions (PTVs), fluorescent tubes, X-ray detectors and field emission displays (FED) [13]. It is well known that the phosphors for field emission displays (FEDs) are required to have a high efficiency at low voltages, a high resistance to current saturation, a long service time and equal or better chromaticity than cathode ray tube (CRT) phosphors [14]. Very satisfactory red and green commercial materials are being produced, but comparable materials for the blue emission are still lacking and are under development for practical applications. Even in the paper industry, fluorescent dyes that absorb UV and emit in blue color are widely used as organic optical brightening agents (OBA) and new inorganic ones have been under investigation. Concerning many of these applications, such as FED and OBA, 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. Most recently Danielson et al. [15] invented a new blue luminescent material, Sr_2CeO_4, using combinatorial techniques. Not only that the same phosphor was prepared by different routes, such as conventional solid state reaction, chemical co-precipitation, microwave calcinations, pulsed laser deposition, polymeric precursorsand ultrasonic spray pyrolysis methods. Sr_2CeO_4 was found to exhibit efficient blue–white luminescence under excitation with UV light, cathode rays or X-rays [16-18]. The excited-state lifetime, electron spin resonance, magnetic susceptibility and structural data suggest that luminescence of Sr_2CeO_4 originates from a ligand-to-metal Ce^{4+} charge-transfer (CT) [19-21]. The presents paper reports on a study of

Enhancedluminescence properties of Eu^{3+}doped Sr_2CeO_4 phosphor with (0.5mol%), citric acid as added with flux and prepared by the conventional solid state reaction method process.

Experimental Procedure

Analytical grade Strontium nitrate [Sr $(NO_3)_2$], Cerium oxide (CeO_2), Europium oxide (Eu_2O_3) of assay 99.9% were used as starting materials. All the phosphor samples are prepared via solid state reaction method. First we prepared un-doped Sr_2CeO_4 phosphor by weighing inorganic salts, Strontium nitrate [$Sr(NO_3)_2$], Cerium oxide (CeO_2) in 2:1 molar ratio. Then mixing and ground into fine powder using agate mortar and pestle about an hour. The sample was fired at 1200ºC for 3 hours in a muffle furnace with a heating rate of 5^oC/min by keeping in an alumina crucible closed with lid. In the same way Eu^{3+} (0.5mol %) and citric acid ($C_6H_8O_7$) at 10 wt % as flux were added in the base material of the Sr_2CeO_4 phosphor [22-24]. We investigated the phase purity, morphology, Photoluminescence excitation and emission and CIE colour coordinates (1931-Chart). Powder X-Ray Diffraction (PXRD) analysis was carried out with a powder diffractometer (Rigaku-D/max 2500) using Cu Kα radiation, microstructures/morphology of the samples were studied using a Scanning Electron Microscopy (SEM) (Philips XL-CP-30), Photoluminescence (PL) emission and excitation spectra were measured by Spectro fluorophotometer (SHIMADZU, RF5301 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 1.5nm. The Commission International de l'Eclairage (CIE-1931 Chromaticity diagram) colour co-ordinates were calculated by the spectrophotometric method using the spectral energy distribution [25-30]. The chromatic colour coordinates (x, y) of prepared materials were calculated with colour calculator version 2, software from Radiant Imaging.

RESULTS AND DISCUSSION

Powder X-ray diffraction Analysis: To determine the crystal structure and phase purity of the phosphors, XRD analysis was carried out. The crystal structure of the prepared silicate phosphor was determined by using X-ray diffraction analysis.The structure and the crystallite size were determined by means of X-ray diffraction technique.The powder X-ray diffraction (PXRD) profiles of Eu^{3+}doped Sr_2CeO_4 phosphor, calcined at 1200°C for 3h are shown in figure.1. From the figure, it is observed that the XRD pattern of Eu^{3+}doped Sr_2CeO_4 phosphor and was indexed to crystallize in the orthorhombic phase corresponding to JCPDS Card no - 89-5546 [31]. In addition, the refined crystallographic unit cell parameters were obtained and are listed in table 1. These values are compared with L. Li et al. [32] and R. Seema et al. [33]. The powder derived from the solid state reaction method was heated at 1200^0C in air for 3hrs shows the starting materials disappeared completely and Sr_2CeO_4 was formed as the predominant product with a tiny amount of $SrCeO_3$. Except this, no other phase exists in the XRD spectra, indicating the formation of the final product Table 1.

Crystal name	a (Å)	b(Å)	c(Å)	Cell volume($Å^3$)
JCPDS No. 89-5546[13]	6.119	10.350	3.597	227.79
Our sample	5.964	8.279	4.472	220.81
L.Li et al[14]	6.1153	10.3473	3.5957	227.52
R.Seema et al[15]	6.07	10.32	3.62	226.76

The average crystallite size was calculated from the XRD pattern using Debye Scherer's formula. Debye Scherer's formula is $D = K \lambda / \beta \cos \theta$, Where D = crystallite size for the (hkl), K = constant, λ = X-ray wavelength of incident radiation [Cu Ka (λ=1.5405Å], β = Full width at half maxima (FWHM), θ = Angle of the big peak. Based on the Debye-Scherer's formula, the crystallite size is calculated at calcined at 1200°C for 3hours.The average crystallite size of un-doped Sr_2CeO_4 phosphor is around ~9nmand Eu^{3+}doped Sr_2CeO_4 phosphor sample with (0.5mol%) is around is ~10nm.The reduction of unit cell volume was observed compared with previous workers as shown in table 1. Therefore this confirms the formation of nano crystallite phosphor, via solid state method. This may conclude that the formation of nano crystallites in the phosphors calcined at1200°C for 3hours are more sufficient in good heating treatment.

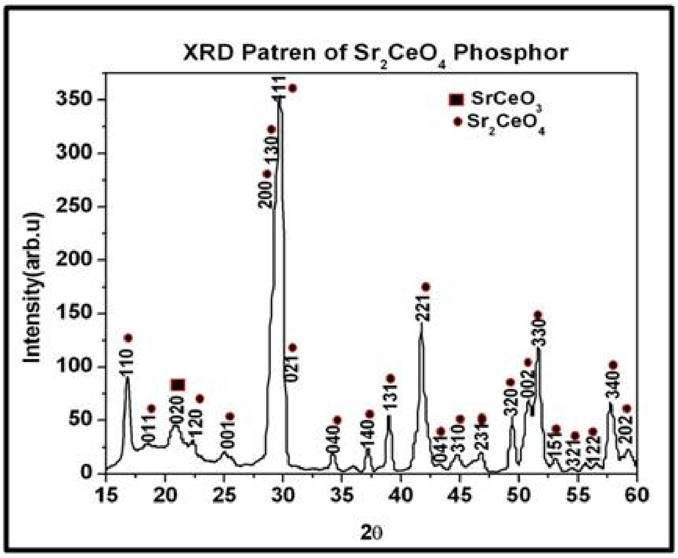

Fig. 1. XRD Pattern of Eu^{3+}doped Sr_2CeO_4 phosphor (0.5mol %) calcined at 1200^0C for 3hours

Scanning electron microscopy Analysis (SEM): Scanning electron microscopy (SEM) was used to study the morphology of the sample. Particle size of phosphor plays an important role in deciding the luminescence quality of the material. Figure.2.(a&b) shows the SEM micrograph of un-doped and Eu^{3+}doped Sr_2CeO_4 phosphor sample with (0.5mol%) iscalcined at 1200^0C for 3hours prepared by conventional solid state reaction method. The entire sample exhibits grain like morphology with different sizes and shape. At high magnification calcined at 1200^0C for 3hours prepared by conventional solid state reaction method, the particles were appeared to be agglomerated and the nature of individual crystallites is evident. Particle size is estimated through this analysis as uniformity of the particle shape and size affects the luminescence efficiency of phosphor materials [34-37].SEM image is exhibits the grains like morphology with different sizes and shapes and are highly agglomerated. The system undergoes violent combustion since the redox reaction mixture with nitrate salts is strongly exothermic. The pores formed are sparse and irregular owing to the uncontrolled dynamics of the process. However, the porous structure is distributed non-uniformly in the matrix. Average particles of phosphor with less than 3 μm are a good sign of lamp phosphor for coating purpose. Also, this can be helpful for display device and technological applications in LEDs.

Photoluminescence studies Analysis (PL): PL spectroscopy study is an important tool to understand the optical properties of the Eu^{3+}doped Sr_2CeO_4 phosphor sample with (0.5mol%) iscalcined at 1200^0C for 3hours prepared by conventional solid state reaction method.Fig.3 shows the PL excitation and emission spectrum of un-doped and Eu^{3+}doped Sr_2CeO_4 phosphor sample with (0.5mol%). The undoped sample shows broad emission (curve 2) from 350 – 650nm peaks at 470nm which is attributed to the energy transfer between the molecular orbital of the ligand and charge transfer state of the Ce^{4+} ion. The emission band can be assigned to the $f \rightarrow t_{1g}$ transitions of Ce^{4+} ions. The excitation spectra were studied under 260 and 270nm, the observed emission peak was same but intensity is high for 270nm excitation. However our results were compared with the previous workers [38-41]. The Eu^{3+}doped Sr_2CeO_4 phosphor sample with (0.5mol %) without and with flux; under 270nm excitation (curve 3 & 4) shows peaks at 467, 491, 512, 537, 557, 585, 590, 601, 611 and 616nm with good intensity. Under 270nm excitation, the samples show same emission spectra with increase in the intensity by 60%.

The observed peaks are from the transitions $^5D_2 \rightarrow {}^7F_{0,\ 2,\ 3}$, $^5D_1 \rightarrow {}^7F_{1,\ 2}$ and also from $^5D_0 \rightarrow {}^7F_{1,\ 2,}$ respectively. The peak around 610-620nm is due to the electric dipole transition of $^5D_0 \rightarrow {}^7F_2$, which is induced by the lack of inversion symmetry at the Eu^{3+} sites. It is well known that the $^5D_0 \rightarrow {}^7F_2$ / $^5D_0 \rightarrow {}^7F_1$ intensity ratio is a good measure of the site symmetry of rare-earth ions in a doped material. This is because the hypersensitive transition $^5D_0 \rightarrow {}^7F_2$ tends to be much more intense at a site with no inversion symmetry, while the magnetic dipole transition $^5D_0 \rightarrow {}^7F_1$ is constant, regardless of the environment. The result indicated the presence of energy transfer is taking place from Ce to Eu ion. It is also observed that broad emission from Ce ion is not completely quenched even when the dopant are added to the host material. This may be because of the low concentration of the dopant [42,43]. It is concluded that may be at high concentrations of Eu (5-10%) in Sr_2CeO_4 phosphor one can get red color emission. When compared with the sample without flux, it is also observed that the citric acid added sample shows the enhancement of 611nm peak, increased by 50% hence we can conclude that the citric acid plays an important role.

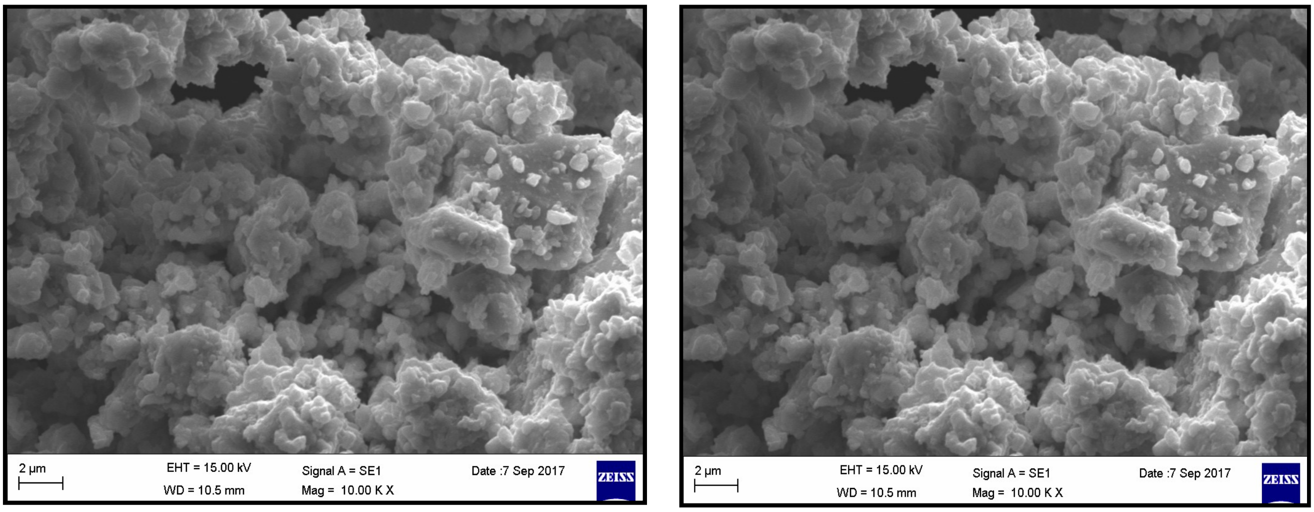

Fig.2 (a&b) SEM micrographs of (a) Undoped and (b) Eu^{3+}doped Sr_2CeO_4 phosphor with (0.5mol%) iscalcined at 1200^0C for 3hours

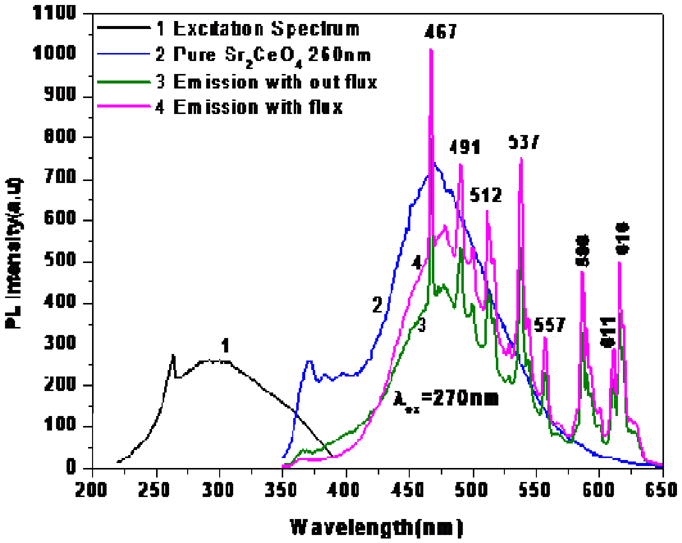

Fig. 3. Excitation and Emission spectrum of un-doped and Eu^{3+}doped Sr_2CeO_4 phosphor sample with (0.5mol%) with and without fluxesarecalcined at 1200^0C for 3hours

CIE Colour Coordinates analysis (CIE-1931Chart): In order to further improve the CIE Colour Co-ordinates and achieve good quality blue, yellow and red light emission, the attributed luminescence colour of the sample has been characterized by the CIE-1931 chromaticity diagram. Generally, monochromatic sources (Δλ-0) are located on the perimeter of the chromaticity diagram. The colour location moves towards the center of the chromaticity diagram, when the spectral bandwidth of a source gets broader. Fig. 4 shows the CIE Coordinates ofEu^{3+}doped Sr_2CeO_4 phosphor sample with (0.5mol%) with and without fluxesarecalcined at 1200^0C for 3hours presented in CIE-1931 chromaticity diagram [44].

From the fig. 4 it is observed that the CIE-1931 chart chromaticity diagram of the colour co-ordinates for the un-doped Sr_2CeO_4phosphor sample (A) are X = 0.158 and Y = 0.192 (Blue emission λ_{em}=467nm), Eu^{3+}(0.5mol%) doped Sr_2CeO_4phosphor sample (B) are X=0.313 and Y=0.325 (Yellow emission λ_{em}=537nm) and Eu^{3+}(0.5mol%) doped Sr_2CeO_4phosphorwith flux added sample (C) are X=0.469 and Y=0.295 (Red emission λ_{em}=616nm). From fig.4 it is observed that the emission varies from blue to yellow and then to red with increasing of the flux concentration. This particular colours are especially decorated lightings and display devices [45-48].

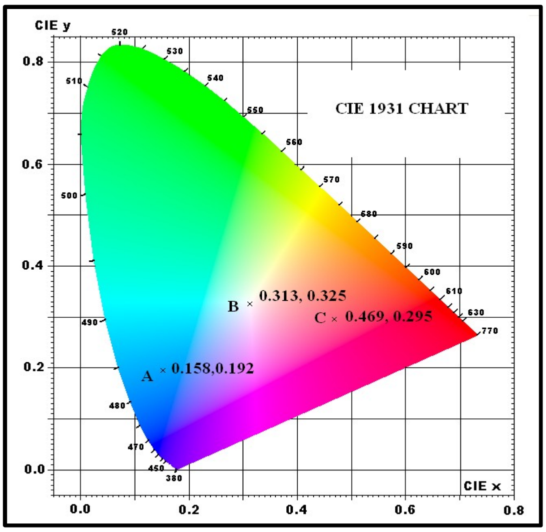

Fig. 4. CIE Coordinates ofEu^{3+}doped Sr_2CeO_4 phosphor sample with (0.5mol%) with and withoutfluxesarecalcined at 1200^0C for 3hours

CONCLUSIONS

- In this colclusion, we successfully synthesized and Enhanced the luminescence properties of Eu^{3+}doped Sr_2CeO_4 phosphor with (0.5mol%), citric acid as added with flux and prepared by the conventional solid state reaction method.

- Based on the Debye-Scherer's formula, the crystallite size is calculated at calcined at 1200°C for 3hours.The average crystallite size of un-doped Sr_2CeO_4 phosphor is around ~9nmand Eu^{3+}doped Sr_2CeO_4 phosphor with (0.5mol%) is around ~10nm.
- SEM micrograph of un-doped and Eu^{3+}doped Sr_2CeO_4 phosphor sample with (0.5mol%) iscalcined at 1200^0C for 3hours prepared by conventional solid state reaction method. The entire sample exhibits grain like morphology with different sizes and shape.
- The Eu^{3+}doped Sr_2CeO_4 phosphor sample with (0.5mol%) without and with flux; under 270nm excitation (curve 3 & 4) shows peaks at 467, 491, 512, 537, 557, 585, 590, 601, 611 and 616nm with good intensity. Under 270nm excitation, the samples show same emission spectra with increase in the intensity by 60%.
- The observed peaks are from the transitions $^5D_2 \rightarrow {}^7F_{0,\ 2,\ 3}$, $^5D_1 \rightarrow {}^7F_{1,\ 2}$ and also from $^5D_0 \rightarrow {}^7F_{1,\ 2,}$ respectively. The peak around 610-620nm is due to the electric dipole transition of $^5D_0 \rightarrow {}^7F_2$, which is induced by the lack of inversion symmetry at the Eu^{3+} sites.
- CIE-1931 chart chromaticity diagram of the colour co-ordinates for the un-doped Sr_2CeO_4 sample (A) are X = 0.158 and Y = 0.192(Blue emission λ_{em} =467nm), Eu^{3+}(0.5mol%) doped Sr_2CeO_4phosphor sample (B) are X=0.313 and Y=0.325(Yellow emission λ_{em} =537nm) and Eu^{3+}(0.5mol%) doped Sr_2CeO_4phosphor sample (C) are X=0.469 and Y=0.295(Red emission λ_{em} =616nm).This particular colours are especially decorated lightings many display devices, technological applications and in the fluorescent lamps.

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