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A NOVEL WARM RED-EMISSION OF $SRLA_{0.9}EU_{0.1}AlO_4$ PHOSPHOR OBTAINED BY COMBUSTION METHOD

Nelson Oshogwue Etafo
Programa de Posgrado en Ciencia y Tecnología de Materiales, Facultad de Ciencias Químicas, Universidad Autónoma de Coahuila, Ing. J. Cárdenas Valdez S/N Republica, 25280 Saltillo, Coahuila México.

Abstract: In this study, A novel warm red-emitting phosphor Strontium Lanthanum Aluminate ($SrLaAlO_4$:xEu) doped with the trivalent Europium ions were synthesized via urea-based solution combustion method. The x mol% doping concentration of Eu^{3+} was varied in x = 0.0, and 0.01 mol%. X-ray diffraction analysis show that all samples exhibit the reflected peaks of the tetragonal structure $SrLaAlO_4$ matching with the JCPDS No 24-1125 standard card. The SEM analysis show irregular grains for all samples with mean sizes which reduces from 0.996 to 0.909 μm. The optical band gap of 4.47 eV and refractive of 1.79 were obtained from the Tauc plots from the absorbance spectra. Photoluminescence spectra of the $SrLaAlO_4$: 0.10Eu, under 279 nm excitation, show a very intense red emission peak band centered at 622 nm due to the $^5D_0$-$^7F_2$ transition of Eu^{3+}. The CIE chromaticity color coordinates were calculated and found to have a color purity and CCT with 53.1 % and 2057 K respectively. All the results clearly indicate that Eu-doped $SrLaAlO_4$ phosphors can be used as a red color component for solid state lighting devices, especially for the fabrication of near-UV based white LEDs, bio-imaging, and labeling.
Keywords: Combustion synthesis, red-emitting, optical band gap, Strontium Lanthanum Aluminate, Europium, Phosphors, Photoluminescence.

INTRODUCTION

Lanthanide-doped luminescent materials have gained a wide acceptance in application through optoelectronic, computer monitor, White light-emitting diodes (WLEDs), fluorescence labels, solid-state lasers etc. [1-3]. This is because lanthanide ions emit visible light after absorbing in the UV-region [2]. They also have special electronic properties that gives them better chemical and optical properties [3]. Trivalent Europium, as a lanthanide is one of the most investigated lanthanides because it has a forbidden 4f -4f transition and with red-emission spectra between $^5D_0$ -$^7F_{0,6}$ level to take up the suitable site symmetry in the host lattice [4,5]. The advantages of Eu^{3+}-activated emission include they are suitable for maximum profit in red-emitters, with unusual high luminescence capabilities in storage devices, optoelectronics and LEDs devices when doped with aluminates, Eu-doped with Lanthanides results in enhanced luminescence and ability to tune its properties to meet its specific requirement like florescence sensing, wavelength conversion etc. [6,7]. The recent Eu – doped phosphors are $NaYbF_4$:Eu^{3+}, $Ca_8NaBi(PO_4)_6F_2$:Eu^{3+}, $Sr_3LaNb_3O_{12}$:Eu^{3+} [8-10].The challenges of red emitting diodes are poor efficiency, poor color stability and limited brightness when compared to other colors of LEDs [11]. This has led to the search for a new phosphor. Strontium lanthanum Aluminate ($SrLaAlO_4$) has attracted so much attention because it has 5.92 gcm^{-3} density, enhanced quantum efficiency, good chemical stability and sustained afterglow [12]. $SrLaAlO_4$ belong to the $ABCO_4$- kind layered perovskite compound as a host lattice where A represents the alkaline earth divalent metallic ion, B represents the trivalent lanthanide ion while C represents any trivalent transition metal ion [13]. This research is novel and has not been investigated in contrast to the reports by Yan et al on Eu doped $SrLaAlO_4$ who used sol-gel as their synthesis method and lower Eu-dopant concentration (0.0 -0.025 mol.%) [14]. The aim of this research is to investigate the structural, morphological, optical and photoluminescence properties of 0.10 mol.% Eu doped $SrLaAlO_4$ phosphors by combustion synthesis method. We further investigated the effect of Sr instead of Ba since 0.10 mol.% produced the highest intensity in our previous investigation of $BaLaAlO_4$:Eu [15]. The synthesized was characterized

by X-ray diffraction (XRD) to study their crystalline structure. The morphology of the surface of the fabricated phosphors was studied by the instrumentality of the scanning electron microscope (SEM). The absorbance, optical gap (ε_g) and refractive index were studied by the DR spectroscopy. The down-conversion (DC) Photoluminescence spectra were measured with the CIE, Chromaticity coordinates, color purity and color correlated temperature (CCT) evaluated. The results showed that $SrLa_{0.9}Eu_{0.1}AlO_4$ phosphor is a promising candidate for bioimaging.

EXPERIMENTAL SECTION.

SYNTHESIS OF SrLaAlO4 AND SrLaAlO4: Tm^{3+}.

The reagents for the preparation of the phosphors were purchased from Sigma Aldrich and used as received: Lanthanum Nitrate hexahydrate ($La(NO_3)_3.6H_2O$, (99.0%), Aluminum Nitrate nonahydrate ($Al(NO_3)_3.9H_2O$ (98.0%)), Strontium Nitrate ($Sr(NO_3)_2$ (99.0%)), Europium Nitrate pentahydrate, ($Eu(NO_3)_3.5H_2O$ (99.99%)), and urea ((99.0%)). Samples were prepared with x = 0.0, and 0.10 mol.% and named as SLA, and SLA 1 respectively in a solution combustion synthesis (Eq.1) and thermal treatment to produce both pure $SrLaAlO_4$ and $SrLaAlO_4$:Eu^{3+}ceramic powders [16]. A typical procedure for the synthesis of Eu doped Strontium Lanthanum Aluminates was as follows: Certain amount of Lanthanum Nitrate hexahydrate, Aluminum Nitrate nonahydrate, Strontium Nitrate, Europium Nitrate, and urea were dissolved into 20 ml of deionized water, stirred and kept on a hot plate first and heated for 20 minutes to obtain a homogenous solution. Thereafter, it was given a heat treatment at 600 °C inside a muffle furnace for some minutes to remove moisture, N_2 and CO_2 as a result of which fluffy powder product was obtained and collected. This product was then grinded and compacted with a press to form pellets. These pellets were annealed in air at 1200 °C for a 6 h duration.

$$Sr(NO_3)_2 + Al(NO_3)_3.9H_2O + xEu(NO_3)_3.5H_2O + 0.97La(NO_3)_3.6H_2O + 6.67NH_2CONH_2 \rightarrow SrLa_{1-x}Eu_xAlO_4 + 28.33H_2O_{(g)} + 10.67N_{2(g)} + 6.67CO_{2(g)} \quad (1)$$

CHARACTERIZATION OF $SrLaAlO_4$ AND $SrLaAlO_4$:EU^{3+}PHOSPHORS.

The morphology of the SLA and SLA 1 samples was analyzed using scanning electron microscopy (JEOL ARM200F) and an energy of 15 KV. The X-ray diffraction (XRD) patterns of the SLAO 0-7 phosphors samples were obtained by using Bruker D8 equipment with a Cu-Kα radiation (λ= 1.54056 Å) in the 2θ range of 10–80°. The photoluminescence curves were recorded in the visible range by using a RF-6000 Shimadzu fluorometer and a scan rate of 0.1 nm/s. All measurements were done at room temperature.

RESULTS AND DISCUSSION

STRUCTURAL AND MORPHOLOGICAL CHARACTERIZATION.

The Fig.1a shows a simulation (made from Diamond software) of the SrLaAlO4, which consists of octahedrons of AlO6 as shown by light green substructures. The ionic radii of the atoms are Sr^{2+}= 1.26 Å and La^{3+}= 1.30 Å, O^{2-}= 1.24 Å [12]. The X-ray diffraction (XRD) spectra were obtained for the SLA powders synthesized with 0.00, and 0.10 mol.% of Eu^{3+} and depicted in Fig.1b. It was observed that the samples have the tetragonal pure phase with diffracted peaks matching with

the pure $SrLaAlO_4$ (JCPDS 24-1125) with no observable additional phase [17]. The most intense peak corresponds to (101), and (110) orientations which are the angles $2\theta = 24.9^{0}$, and $2\theta = 33.8^{0}$, respectively. The crystalline size of each sample of SLAO was calculated using the renowned Scherrer formula (Eq.2).

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (2)$$

Where k is the shape factor (0.9), λ is the wavelength =1.5418 Å (CuKα radiation), β is the full width at half maximum (FWHM), θ is the diffraction angle and it was observed from an XRD plane (103) to obtained the crystallite size of 11.01, and 10.99 nm for SLA and SLA 1 respectively. It was observed that as the concentration of Eu^{3+} increases in the host, the crystallite size decreases as seen in Table 1. There were no observable impurity diffraction peaks that show the Eu doping did not affect the crystalline phase of the SrLaAlO4. The a, b, and c lattice parameters were calculated using the diffraction peaks located at $2\theta = 48.72^{0}$, and 59.51^{0}, which corresponds to (200), and (213) orientations respectively. This generally shows that a, b, and c lattice parameters increase their values as the Eu^{3+} concentration is raised from 0.00 to 0.10 mol.%. Similarly, the volume of the unit cell also increases as the Eu^{3+} concentration increases as seen in Table 1. This agrees with the reports given by Cantelar E. *et al* that shows that an increase in unit cell volume enhances the photoluminescence properties of Eu-doped host [18]. The radius percentage (D_r) (Eq. 3) between substituted ions and dopants ions was calculated to determine the possibility of the formation of a new solid solution and the value of $D_r < 15$ % can be defined [19].

$$D_r = \frac{R_1(CN) - R_2(CN)}{R_1(CN)} \text{ X } 100\ \% \quad (3)$$

Where $R_1(CN)$ and $R_2(CN)$ and are assigned to the radii of the substituted ions (Sr^{2+} *or* La^{3+}) and dopants ions (Eu^{3+}) respectively. They have ionic radii and coordinate numbers (CN) as follows 1.26 Å (CN = 8) or 1.16 Å (CN = 8) and 1.066 Å (CN = 8) respectively. The estimated value of D_r between Eu^{3+} and $Sr^{2+}(La^{3+})$ ions was found to be 15.36 % and 8.10 % respectively. This shows that the Eu^{3+}will substitute the La^{3+} ions site in this study because the Dr of La^{3+} and Eu^{3+} were found to less than 15 % in the SrLaAlO4 host material when compared with Sr^{2+} and Eu^{3+}.

The SEM images show the evolution of the morphology of the samples of SLA:Eu, for 0.00, and 0.10 mol.% of Eu^{3+} dopant concentration respectively. The average size of the microparticles of the different SLA Eu were carried out by employing the Image J software. The average of 10 microparticles of the SEM images of each sample was obtained carefully. Fig. 2a) shows the pure SLAO where no Eu^{3+} was incorporated into the SLA host to give a microporous network of coalesced particles with uneven tubular shape with an average size of 0.996 μm observed (as seen in green circle). If the concentration of Eu^{3+} dopant is increased to 0.10 mol.%, a higher microtubular shape was observed with an average size of 0.909 μm (see blue circles in Fig. 2b). In general, the SEM results prove that raising the concentration of Eu^{3+} dopant increases the conglomeration among the SLA Eu, microparticles. This agrees with the phenomenon of agglomeration of dopant concentration observed in rare-earth doped materials [20]. There is an imbalance of charges that causes a random growth of the microparticles with big sizes and irregular shapes because of the high content of Eu^{3+} [20].

OPTICAL PROPERTIES

The Fig. 3a shows the absorbance spectra for the undoped SLA and the dopant SLA:Eu samples. The absorbance spectrum of the undoped SLA sample (as seen in the green

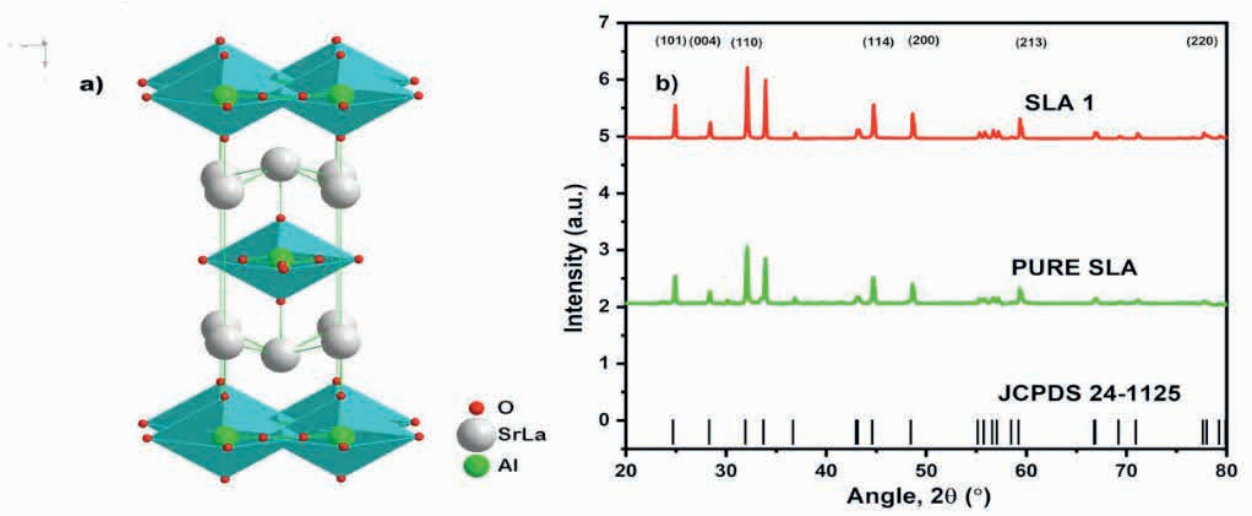


Fig. 1 a)- Visualization of the crystalline structure of the pure orthorhombic $SrLaAlO_4$. b) X-ray diffraction patterns of $SrLaAlO_4$ and different Eu dopant concentration.

Sample	a (Å)	b (Å)	c (Å)	Volume (Å^3)	Crystallite size (nm)
0.00	3.7348	3.7348	12.426	173.327	11.01
0.10	3.7437	3.7437	12.456	174.574	10.99

Table 1- The volume and crystallite size of the SLAO phosphors doped at different concentrations of Eu^{3+}.

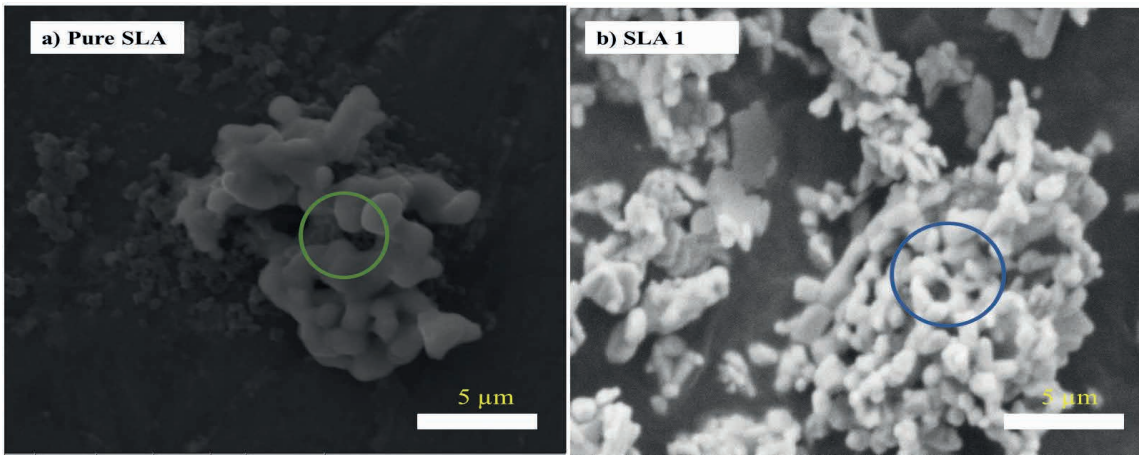


Fig. 2- The SEM micrographs of $SrLaAlO_4$:xEu phosphor powders.

curve in Fig. 3a) shows a strong absorption at the UV region and there is no absorbance for Eu^{3+} in the range of 400 -700 nm. This proves the absence of dopants in the SLAO host. The absorbance spectra of SLA:Eu doped samples exhibit the peaks attributed to the transition lines of Eu^{3+} centered at 222 nm which is attributed to charge transfer band (CTB) reported in where CTB is between the range of 220 -280 nm [21]. It can also be seen that the excitation spectra of SrLaAlO4:Eu phosphor neither has a broad band from 222 nm to 1000 nm nor any maximum intensity. It was observed that 0.10 mol.% of Eu^{3+} doping concentration gives a maximum intensity of the absorption lines (see 214 nm peaks). This fact indicates a good incorporation of the Eu^{3+} into the SrLaAlO4 host lattice with a potential application in near-infrared emission. The optical band gap values for both the pure SLA and the SLA 1 samples were calculated from the absorbance spectra using the Tauc plot procedure. The Tauc plot are graphs in which the y-axis is plotted $(\alpha hv)^{1/n}$ and in the x-axis is plotted energy (eV). The band gap was obtained by extrapolating the curve to the energy axis ($\alpha hv = 0$). The α is the absorption coefficient, hv is the photon energy, with *h* the plank constant and v frequency of light, n was taken as ½ due to the SrLaAlO4 is a host material with a direct band gap with a value of 5.00 eV in Fig. 3 b which agrees reported values of the undoped $SrLaAlO_4$ [13]. The optical band was found to decrease for the 0.10 mol.% of Eu concentration to a value of 4.47 eV as shown in Fig. 3c. The decrease in the optical band gap was observed to result in warm red emission in 0.10 mol.% of Eu^{3+}.

Furthermore, the refractive index of the microphosphors were calculated using the formula in Eq (4) [13].

$$\frac{n^2-1}{n^2+1} = 1 - \sqrt{\frac{\varepsilon_g}{20}} \qquad (4)$$

Where n = refractive index and E_g = optical band and we obtained the refractive index of the SLA 1 with a value of 1.79 respectively. Unlike the reports by the Yan and his group who did not report on absorbance, optical band and refractive index of Eu-doped $SrLaAlO_4$ sample, both the optical band and refractive index of our sample enhanced its the photoluminescence properties.

Then the linear dielectric constant and the susceptibility of the phosphor was further obtained from the formulae in Eq. (5) and (6) respectively [22].

$$n^2 = \epsilon_r \qquad (5)$$

$$\chi = 1 + \epsilon_r \qquad (6)$$

where n = refractive index, ϵ_r = linear dielectric constant and χ = susceptibility respectively. We obtain $\epsilon_r = 3.20$ which means that the Eu-doped $SrLaAlO_4$ phosphor can store electrical energy moderately in compare to the vacuum. The $\chi = 4.20$ proves that Eu-doped $SrLaAlO_4$ phosphor has a relatively high response when exposed to an external magnetic field.

DOWN-CONVERSION LUMINESCENCE PROPERTIES OF SLA:EU

Luminescence properties of the SLA:Eu phosphors were measured for SLA 1 for 0.10 mol.% concentration of Europium as the dopant. Fig. 4a shows down-conversion excitation spectrum of SLA: Eu excited at 622 nm. It shows a broad band emission ranging from 240-340 nm but peak at 279 nm. This 279 nm correlates with the charge transfer band (CTB) state of electrons between the occupied 2p orbitals of O^{2-} to the vacant 4f-orbital of Eu^{3+} which confirms reports [23]. The SLA:Eu^{3+} was excited at 279 nm to produce a down-conversion emission as shown in Fig. 4b to give 3 main red emission bands observed at 537 nm, 593 nm, and 622 nm which corresponds to $^5D_1 \rightarrow {}^7F_1$, $^5D_0 \rightarrow {}^7F_1$, and

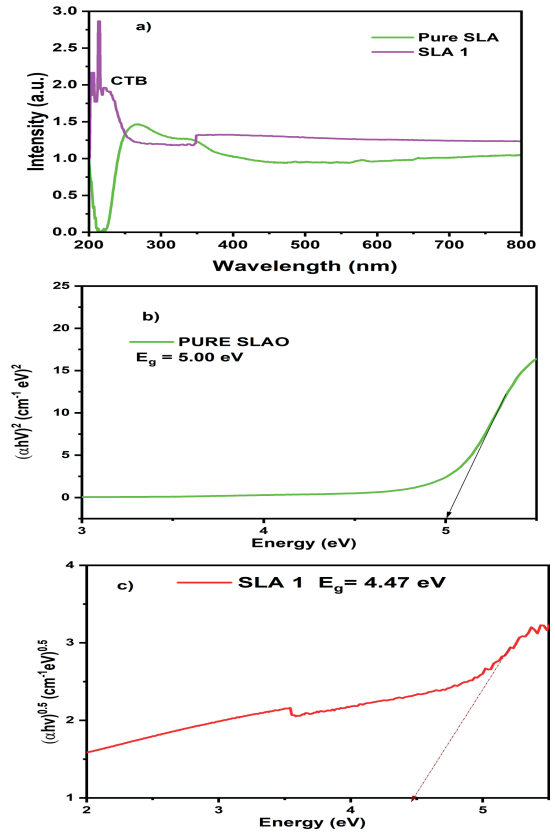


Fig. 3 a) The absorbance spectra of the pure $SrLaAlO_4$ and the Eu-doped $SrLaAlO_4$ samples. b) Tauc plot showing the optical band gap of pure SLAO c) Tauc plot showing the optical band gap of SLAO:xEu.

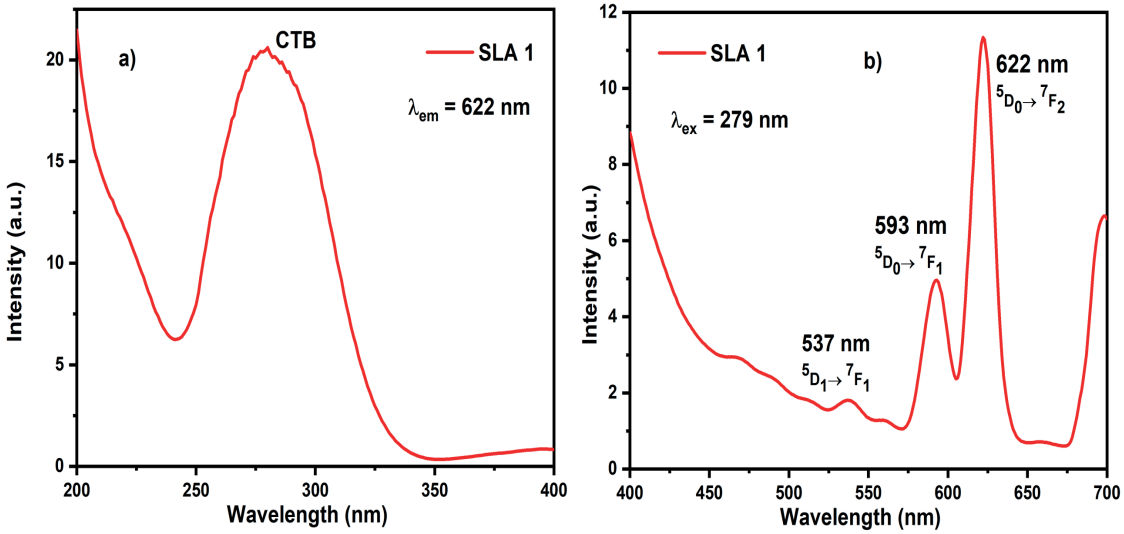


Fig. 4 - PL by down-conversion a) excitation b) emission of Intensity as a function of wavelength.

$^5D_0 \rightarrow {}^7F_2$ transitions respectively which agrees with reports [24]. The most intense emission was observed to be 622 nm shows a red band due to the existence of the f-f transitions which was responsible for luminescence properties in trivalent rare earth ions (Eu^{3+}) [24] which is attributed to the dipole of $^5D_0 \rightarrow {}^7F_2$ transition which is mainly by crystal environment and predominant emission with ions only occupies the sites with low symmetry [19]. The 593 nm is a strong magnetic and the intensity are strongly dependent on the Eu^{3+} ion environment [24]. The 537 nm is an electric and weak transition with absorptivity values reported to be less than 1 $Lmol^{-1}cm^{-1}$ which occur due to the UV light [23]. The sample that present the highest PL intensity under 279 nm excitation was the doped with x= 0.10 mol.% (SLA 1), the inset in Fig. 31 show an inset of the BAL1 sample under UV irradiation with a deep red emission. In comparing with our previous investigation using $BaLa_{0.9}Eu_{0.1}AlO_4$ phosphor with intensity of 617 nm [15], we observed that $SrLa_{0.9}Eu_{0.1}AlO_4$ phosphor has a higher emission of 622 nm. This suggest that Sr contributes to the photoluminescence properties of the sample despite having the same amount and atom present. This is could as a result of the change in crystalline structure where Sr forms a tetragonal structure in $SrLa_{0.9}Eu_{0.1}AlO_4$ phosphor while Ba forms orthorhombic structure in $BaLa_{0.9}Eu_{0.1}AlO_4$ phosphor.

The Commission International de I´ Eclairage, CIE, shows the emissive phosphors of SLA:Eu is in the light red region as shown in Fig. 5 a. with color coordinates (0.499, 0.324), for 0.10 mol.% which supports that the Eu favor only the red emission. The color correlated temperature (CCT) is expressed for the SLA:Eu using the McCamy empirical formula (Eq. 7) [19].

$$CCT = -449\,n^3 + 3525n^2 - 6823n + 5520.33 \quad (7)$$

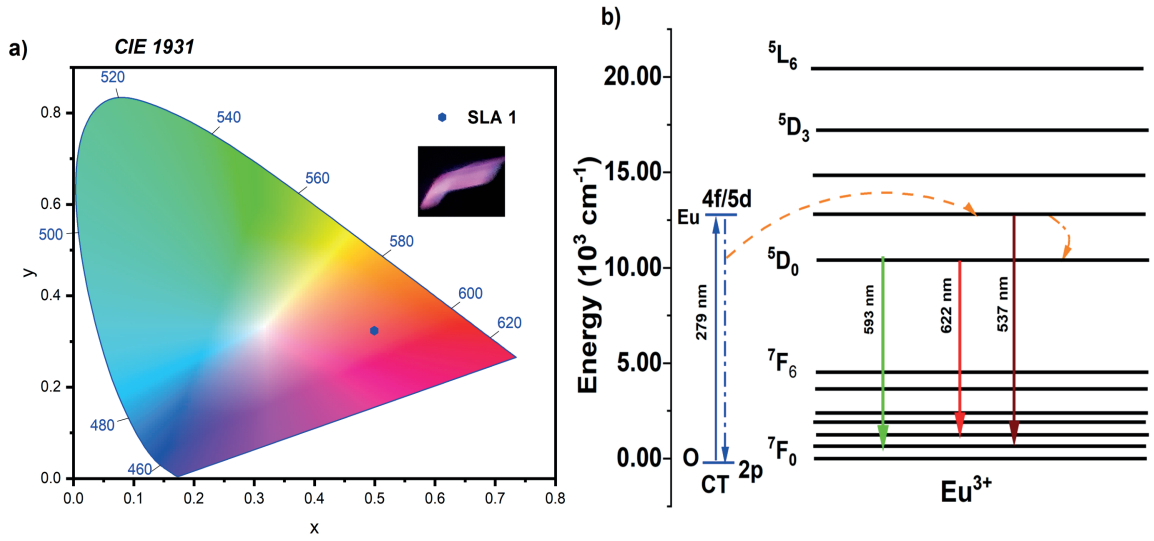
where $n = (x - x_e)/(y - y_e)$ is the reciprocal slope and x_e = 0.312 and y_e= 0.323) is the epicenter of the iso-temperature line. The CCT values of the SLA 0.1 Eu is 2057 K as shown in Table 2 which shows the Eu doped $SrLaAlO_4$ phosphors could emit a warm red light (CCT < 4500 K) which makes it applicable as indoor illuminant [19, 25]. The color purity (CP) was further investigated from the CIE coordinates using the formula (Eq. 8) [26].

$$CP = \frac{\sqrt{(X_S - X_i)^2 + (y_S - y_i)^2}}{\sqrt{(X_d - X_s)^2 + (y_d - y_s)^2}} \times 100\ \% \quad (8)$$

(X_s, y_s) – color coordination of light source = (0.312, 0.323) (X_i, y_i) – color coordination of CIE illuminate (X_d, y_d) = (0.664, 0.334) – color coordination of dominant wavelength (615 nm) of the light source. The color purity showed that an intense red color was emitted for Eu-doped $SrLaAlO_4$ phosphors with 53.1 % for 0.10 mol.% concentrations of Eu^{3+}. In comparing with the investigation by Yan et al [14], we observed increasing the Eu^{3+} dopant concentration to 0.10 mol.% reduces the chromaticity coordinates, color purity and CCT as observed in Table 2. The proposed energy diagram of SLA Eu in Fig. 5b shows that there are three main steps: first, after the 279 nm excitation the $^5D_1$ level is populated when either the Eu^{3+} ions or $SrLaAlO_4$ host absorbs specific photons. Second, the electrons are relaxed non-radiatively to $^5D_0$ level and finally, Eu^{3+} ions emit visible red light (see the inset in Fig. 5b) for the electrons to undergo transition from $^5D_0$ level to the lower energy levels [27].

CONCLUSION

In this work, the synthesis of $SrLa_{0.9}Eu_{0.1}AlO_4$ phosphor was fabricated for the first time by a simple combustion method at 1200 ^{0}C. It was visualized by simulating with diamond software and characterized by SEM, absorbance, optical band by Tauc plot, refractive index and down-conversion (DC) photoluminescence (including the



The Fig. 5a) The CIE map showing the color coordinates of $SrLa_{0.9}Eu_{0.1}AlO_4$ phosphor with an inset photograph excited at 279 nm. b) the proposed schematic (illustrative) energy level diagram of Eu^{3+} in the $SrLaAlO_4$ host depicting the different emission bands.

Eu^{3+} dopant concentration	x	y	CCT (K)	Color purity (%)	Reference
0.025	0.670	0.311	-	97.5	[14]
0.10	0.499	0.324	2057	53.1	This work

Table 2- The table of the chromatic properties of various work on Eu-doped $SrLaAlO_4$ phosphors.

color purity and CCT) were measured. We obtained a tetragonal crystalline phase with no additional peaks observed from the XRD. The optical band gap (E_g) and refractive index were evaluated to be 4.47 eV and 1.79 respectively from the DR spectroscopy. The Down-conversion photoluminescence shows an intense warm red-emission at 622 nm due to the $^5D_0 \rightarrow {}^7F_2$ transition of Eu^{3+} when excited at 279 nm. The color purity and CCT were evaluated from the CIE 1931 to be 53.1 % and 2057 K respectively. This makes $SrLa_{0.9}Eu_{0.1}AlO_4$ phosphor a potential candidate for bioimaging. We reported also that at a higher concentration of 0.10 mol.% Eu in $SrLaAlO_4$ there is a lower PL emission with lower chromatic properties (CCT and color purity) but better properties than in $BaLaAlO_4$ due to change in the crystallographic phase.

DECLARATION OF COMPETING INTEREST

The authors declare that they have no competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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