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Combustion Synthesis and Photoluminescence Study of $Sr_3Y_{1-x}(BO_3)_3$: xPr^{3+} $(0.01 \le x \le 0.05)$ Red Borate Phosphor for Near Blue Pumped WLED

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Abstract: A red borate phosphor $Sr_3Y_{1-x}(BO_3)_3$: xPr^{3+} ($0.01 \le x \le 0.05$) was prepared by using a solution combustion reaction method, and its photoluminescence property is investigated. The synthesized phosphors were characterized by X-ray diffraction (XRD), scanned electron microscope (SEM) and photoluminescence (PL). Their excitation bands at 375, 384 and 395 nm are originated from the transitions of $^3H_4 \rightarrow {}^3P_2$, $^3H_4 \rightarrow {}^3P_1$ and $^3H_4 \rightarrow {}^3P_0$ of Pr^{3+} ion. Under375nm nm excitations, the emission of Pr^{3+} at 595, 614 and 632 nm are attributed to $^1D_2 \rightarrow {}^3H_5$, $^3P_0 \rightarrow {}^3H_6$ and $^3P_0 \rightarrow {}^3F_2$. The Commission International de Eclairage (CIE) chromaticity coordinates are ($X = 0.67745889$, $Y = 0.322362077$) for $Sr_3Y_{1-x}(BO_3)_3$: xPr^{3+} . The result shows that $Sr_3Y_{1-x}(BO_3)_3$: xPr^{3+} is promising candidate for red phosphors in the fabrication of pc-WLED and in blue LED chip WLED.

Keywords: Borate phosphors; Photoluminescence, Solution combustion, $Sr_3Y_{1-x}(BO_3)_3$: xPr^{3+}

I. INTRODUCTION

White light-emitting diodes (W-LEDs) are the new generation light source because of their high brightness, long lifetime, low power consumption, and environment friendliness (1) (2). One approach to get white light is using blue light (around 430-480 nm) chip coated with yellow phosphors (3). The other approach is mixed tri-color (red, green and blue) phosphors in which red phosphors have more effect on the luminous flux than green and blue phosphor (4). Shi-Zhang Ma et al reported $KSr_4(BO_3)_3:Pr^{3+}$ a new red-emitting phosphor for blue-pumped white light-emitting diodes (5). P Boutinaud et.al. Reported $NaNbO_3:Pr^{3+}$and $LiNbO_3:Pr^{3+}$polycrystalline samples as red phosphors which show persistence luminescence (6). A O Chauhan et.al. studied $LiSr_4(BO_3)_3:Gd^{3+}$, Pr^{3+} as narrow-band UVB-emitting phosphor. $LiSr_4(BO_3)_3$: Pr^{3+} phosphor shows broad excitation band from 210 to 260 nm with a maximum at 245 nm. The phosphors give strong and a much broader emission spectrum from 260 to 370 nm having two shoulder peaks at 280 and 325 nm under the excitation of 245 nm (7). Role of Pr^{3+} in various hosts is different due to crystal field energy. Maximum Pr^{3+}-doped materials show intense $^3P_0 \rightarrow {}^3H_6$ emission in the red spectral region. Pr^{3+} doped phosphors have been widely adopted as red emitting phosphors (8). In recent years, the Pr^{3+} doped borates have created much interest for their use in various optical and optoelectronic devices (9). Luminescence properties of Pr^{3+} doped $Sr_3Y_{1-x}(BO_3)_3$ have not been studied yet, though the red luminescence of Pr^{3+} ion has been mostly used in the lighting and laser (10) (11). Therefore, motivated by the above investigations and the attempt to develop novel phosphors for the applications of W-LED, we have investigated the photoluminescence (PL), photoluminescence excitation (PLE) of $Sr_3Y_{0.96}(BO_3)_3$: $0.04Pr^{3+}$

II. EXPERIMENTAL DETAILS

Given Inorganic borate phosphors were prepared by the solution combustion synthesis technique. Stoichiometric amounts of high purity starting materials, $SrNO_3$ (A.R.), Pr_2O_3 (high purity 99.9%), H_3BO_3 (A.R.), $CO(NH_2)_2$ (A.R.), $Y(NO_3)_3$ (A.R.) are used for phosphor preparation. The starting materials with little amount of double distilled water were mixed thoroughly in agate mortar to obtain a homogeneous solution.

Excess water was removed by heating the samples at temperature 100 ^{O}C for about 30 min and the paste was then transferred directly to a pre-heated Muffle furnace, maintained at temperature 680^{O}C, for combustion. Following the combustion, the resulting foamy samples was crushed to obtain fine particles and then annealed for 3 h at temperature 950^{o}C. As prepared Borate phosphor material was characterized by powder XRD, SEM, PL and FT-IR techniques. Surface morphology and elemental analysis of the calcined powder sample was observed by scanning electron microscopy [SEM: Model JSM6100 (Jeol)].

TABLE 1: Molar concentration of ingredients

$3[Sr(NO_3)_2] + x[Pr(NO_3)_3] + (1-x)Y(NO_3)_3 + 3H_3BO_3 + CO(NH_2)_2 \xrightarrow{680\,^{\circ}C}$				
$Sr_3Y_{1-x}(BO_3)_3{:}xPr^{3+}$ + Gaseous products				
$Sr(NO_3)_2$	$Pr(NO_3)_3$	$Y(NO_3)_3$	$3H_3BO_3$	$CO(NH_2)_2$
6.3486	0.0669	1.13	1.855	4.80

1) *XRD:* Powder X-ray diffraction measurements were taken on a Rigaku Miniflex II X-ray Diffractometer and compared with those of ICSD files. Powder XRD pattern of the synthesized phosphor was recorded by X-ray diffraction using the Cu-Kα wavelength (λ= 1.54060 Å) and scanning in the 2θ range from 20°–80°. Figure [1] shows the powder XRD pattern of the phosphor $Sr_3Y(BO_3)_3$. The powder XRD pattern of synthesized phosphor agrees with standard pattern given by ICSD File NO. 246230. Space group is R-3; crystal system is trigonal (hexagonal axes).

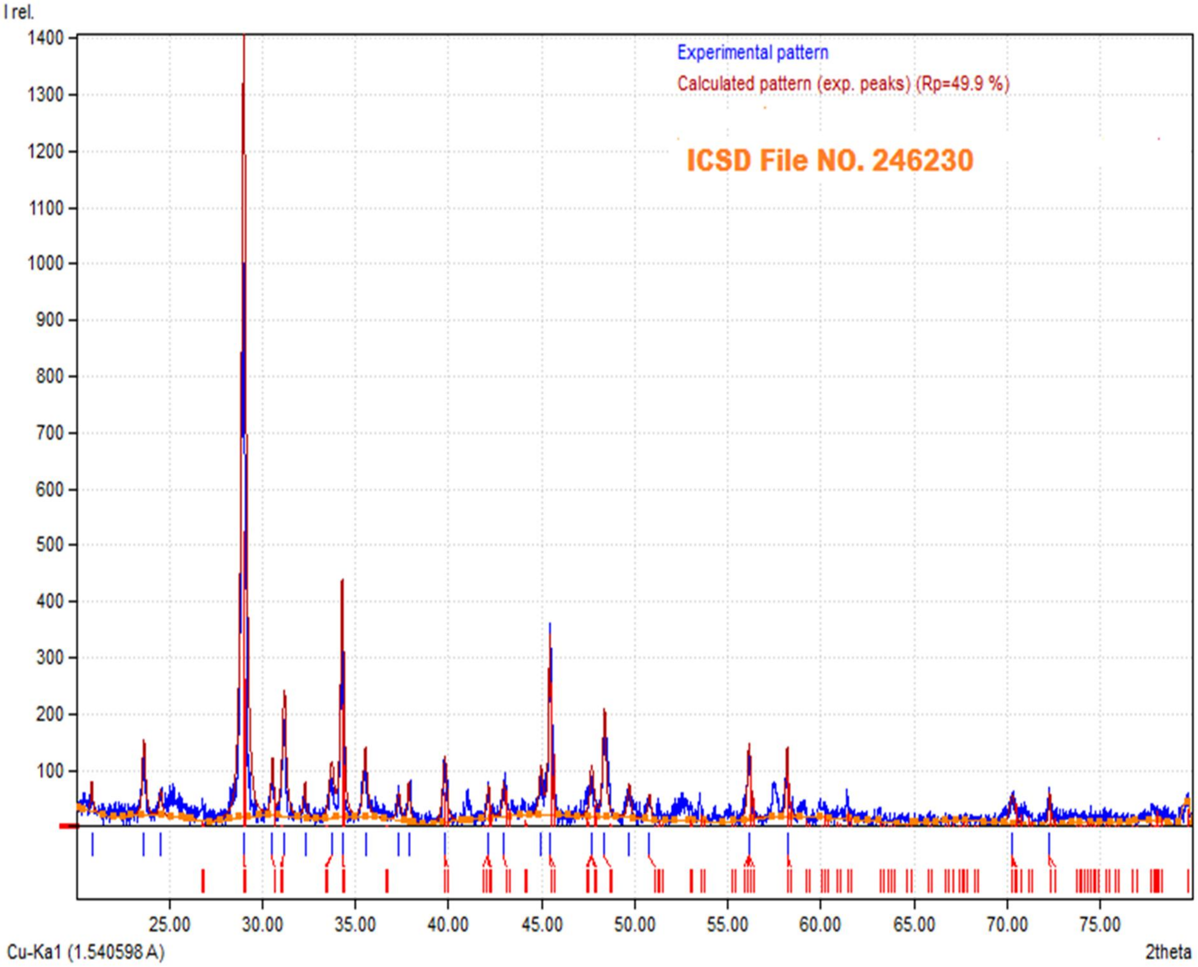

Figure 1 powder XRD pattern of the phosphor $Sr_3Y(BO_3)_3$

2) *FTIR:* FTIR of sample was done on F.T. Infra-Red Spectrophotometer Model RZX (Perkin Elmer). The coordination environment of B–O in $Sr_3Y_{0.96}(BO_3)_3{:}0.04Pr^{3+}$ structure is confirm using, the FTIR spectra measured at room temperature; which is shown in Figure(2). The IR absorption at wave numbers smaller than 500 cm^{-1} mainly originates from the lattice dynamic modes. The strong bands observed above 1100 cm^{-1} should be assigned to the B–O stretching mode of the triangular $[BO_3]^-$ groups, while the bands with maxima at about 700–800 cm^{-1} should be attributed to the B–O out of plane bending, which confirms the existence of the $[BO_3]^-$ groups (12). The absence of peaks in 1500–2000 cm^{-1} indicates the complete removal of nitrate and organic matter. The peak at 3300–3500 cm^{-1} corresponds the stretching mode of O–H are also absent.

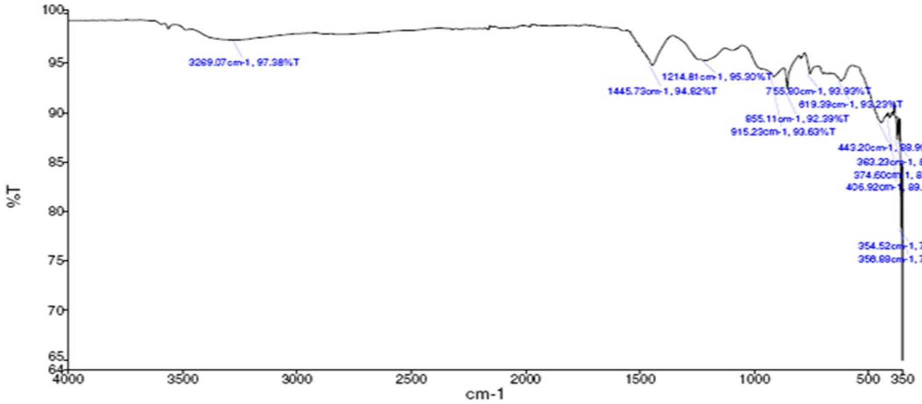

Figure 2 FTIR of $Sr_3Y_{0.96}(BO_3)_3$:0.04Pr^{3+}

III. PHOTOLUMINESCENCE STUDY

PL and PLE measurements at room temperature were performed on a Hitachi F-7000 spectrophotometer with spectral resolution of 2.5 nm and is given in figure (3). In PLE the excitation bands at 375, 384 and 395 nm are originated from the transitions of $^3H_4 \rightarrow {}^3P_2$, $^3H_4 \rightarrow {}^3P_1$ and $^3H_4 \rightarrow {}^3P_0$ of Pr^{3+} ion. Under375nm nm excitations, the emission of Pr^{3+} at 595, 614 and 632 nm are attributed to $^1D_2 \rightarrow {}^3H_5$, $^3P_0 \rightarrow {}^3H_6$ and $^3P_0 \rightarrow {}^3F_2$. Energy level diagram of Pr3+ is shown in figure (6). At 614nm characteristic PL the intense PLE is noted on 375nm spectral line. The broad excitation band from 290nm to 440nm is very important in view of application of the said phosphor material. Above 400nm Photo luminescence excitation role of phosphor in BLUE-CHIP-WLED increases.

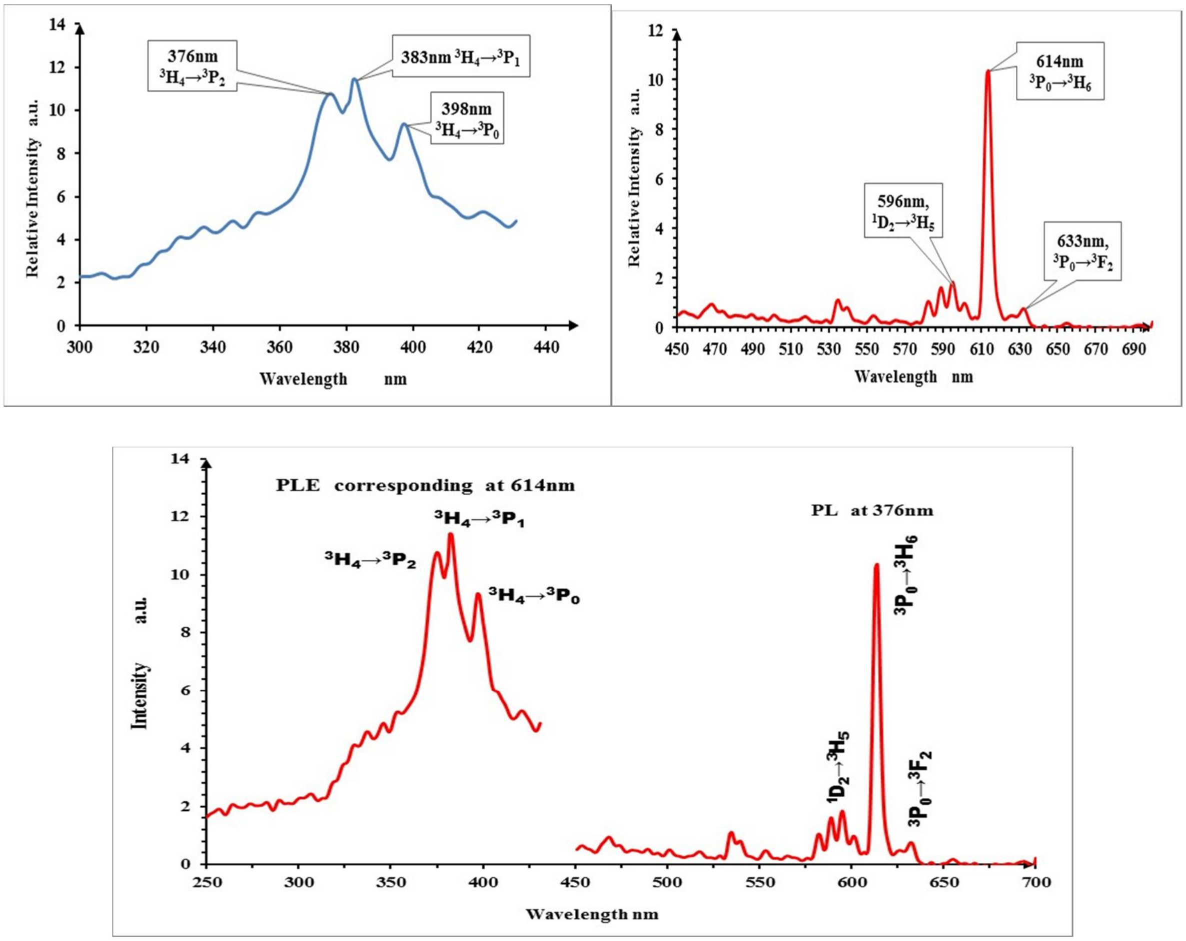

Figure 3 PL and PLE OF $Sr_3Y_{0.96}(BO_3)_3$:0.04Pr^{3+}

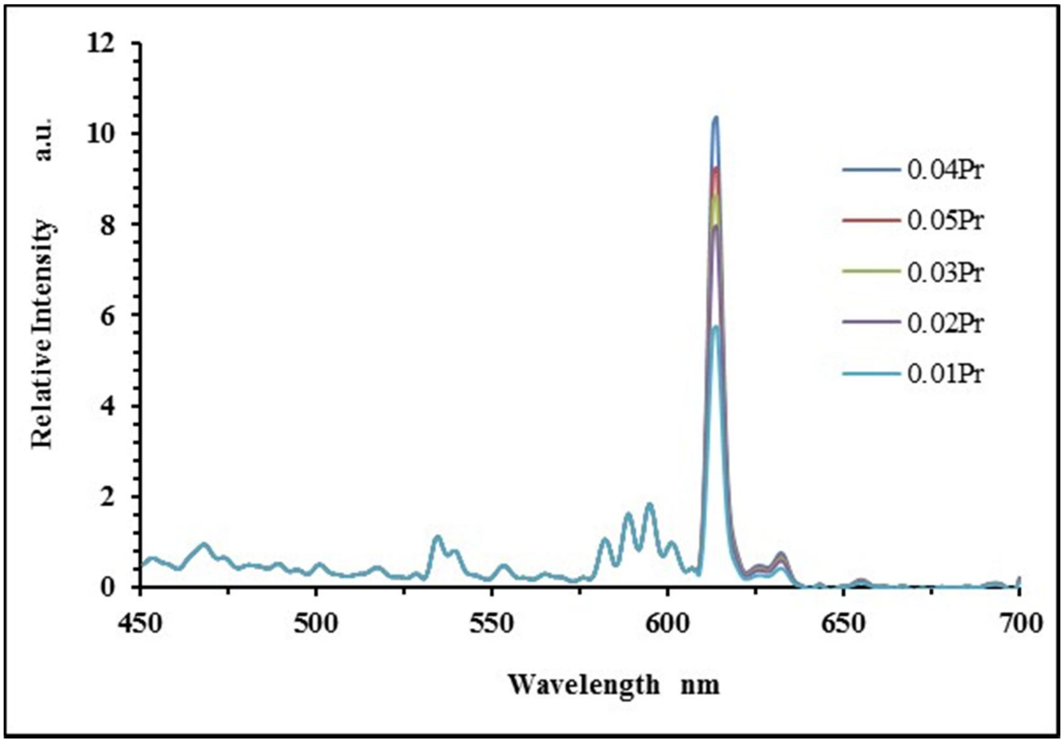

Figure 4 PL of $Sr_3Y_{1-x}(BO_3)_3$: xPr^{3+} ($0.01 \leq x \leq 0.05$)

Photoluminescence of $Sr_3Y_{1-x}(BO_3)_3$: xPr^{3+} ($0.01 \leq x \leq 0.05$) at 375nm excitation is shown in figure (4). At 4mole percent of Pr^{3+} PL intensity is maximum. Concentration quenching curve is given in figure (5). It indicates as concentration of Pr^{3+} increases, PL intensity increases upto 4 Mole % and then decreases.

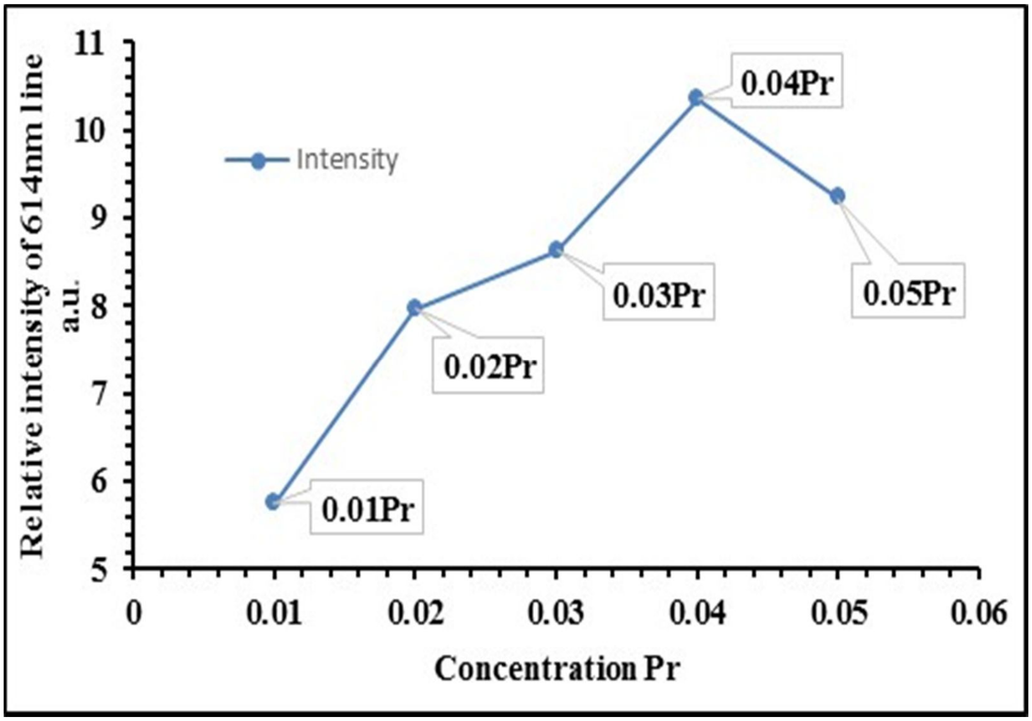

Figure 5 Concentration Quenching Curve of $Sr_3Y_{1-x}(BO_3)3$: xPr^{3+} ($0.01 \leq x \leq 0.05$)

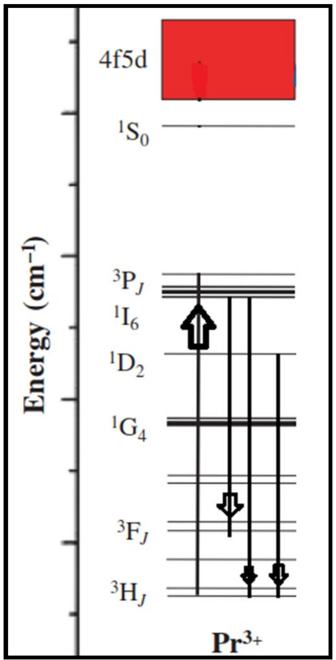

Figure 6 Energy levels of Pr^{3+}

1) *CIE- Diagram:* Figure (7) Shows the Commission International del Eclairage (CIE) chromaticity coordinates diagram of $Sr_3Y_{0.96}(BO_3)_3{:}0.04Pr^{3+}$ phosphor. The chromaticity coordinates of the phosphor $Sr_3Y_{0.96}(BO_3)_3{:}0.04Pr^{3+}$ for fixed concentration of Pr^{3+} was computed using LEDTUNING.NL Software [https://www.ledtuning.nl/en/cie-convertor]. The chromaticity coordinates are used in 'GO-CIE' CIE plot utility software developed by organic material laboratory of IIT Roorkee for 1931 CIE Chromaticity diagram [http://faculty.iitr.ac.in/~krjt8fcy/gocie.html]. The coordinates obtained were X=0.67745889 Y=0.322362077, which fall into the red region in the CIE 1931 chromaticity diagram and shown as black spot in figure (7). As seen in the PL spectra, the $Sr_3Y_{1-x}(BO_3)_3{:}xPr^{3+}$ phosphor has red emission under violet light at 375nm. This means that the $Sr_3Y_{1-x}(BO_3)_3{:}xPr^{3+}$ phosphor is a good red phosphor candidate for solid-state lighting applications and in blue-chip WLED.

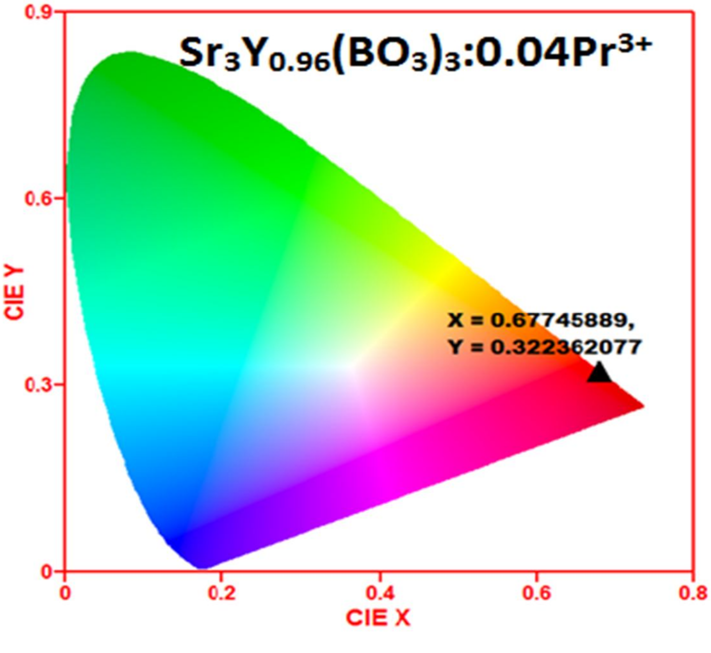

Figure 7 CIE) chromaticity coordinates diagram of $Sr_3Y_{0.96}(BO_3)_3{:}0.04Pr^{3+}$

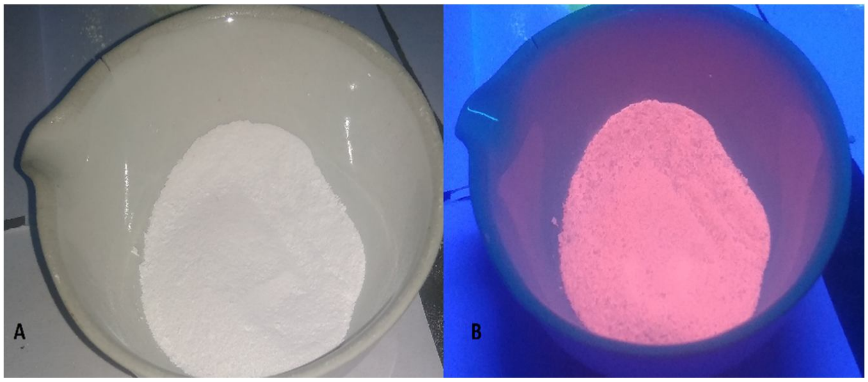

Figure 8 (A) White powder under day light (B) PL emission under Blue excitation

IV. CONCLUSION

The $Sr_3Y_{1-x}(BO_3)_3$:xPr^{3+} polycrystalline phosphor was synthesized by solution combustion method using urea as a fuel and nitrates as a oxidiser. Using XRD formation of compound is confirmed and it matches with standard ICSD File NO. 246230. Using FTIR presence of $[BO^3]^-$ groups, coordination environment of B-O in structure and Complete combustion of nitrates and organic matter were confirmed. Phosphor shows broad excitation band from 350 to 420 nm with prominent peak at 375, 384 and 395 nm. PLE for characteristic emission wavelength 614 nm was found to be 375 nm. At 375 nm violet light excitation $Sr_3Y_{1-x}(BO_3)_3$:xPr^{3+} phosphor emits red light. As Pr^{3+} is sensitive to surrounding crystal field environment it shows more absorption peak at 384nm, 395nm which gives emission at 595 nm and 632nm. Hence $Sr_3Y_{1-x}(BO_3)_3$:xPr^{3+} is new violet light or near blue light excited red emitting phosphor useful for UV/blue-chip based WLEDs.

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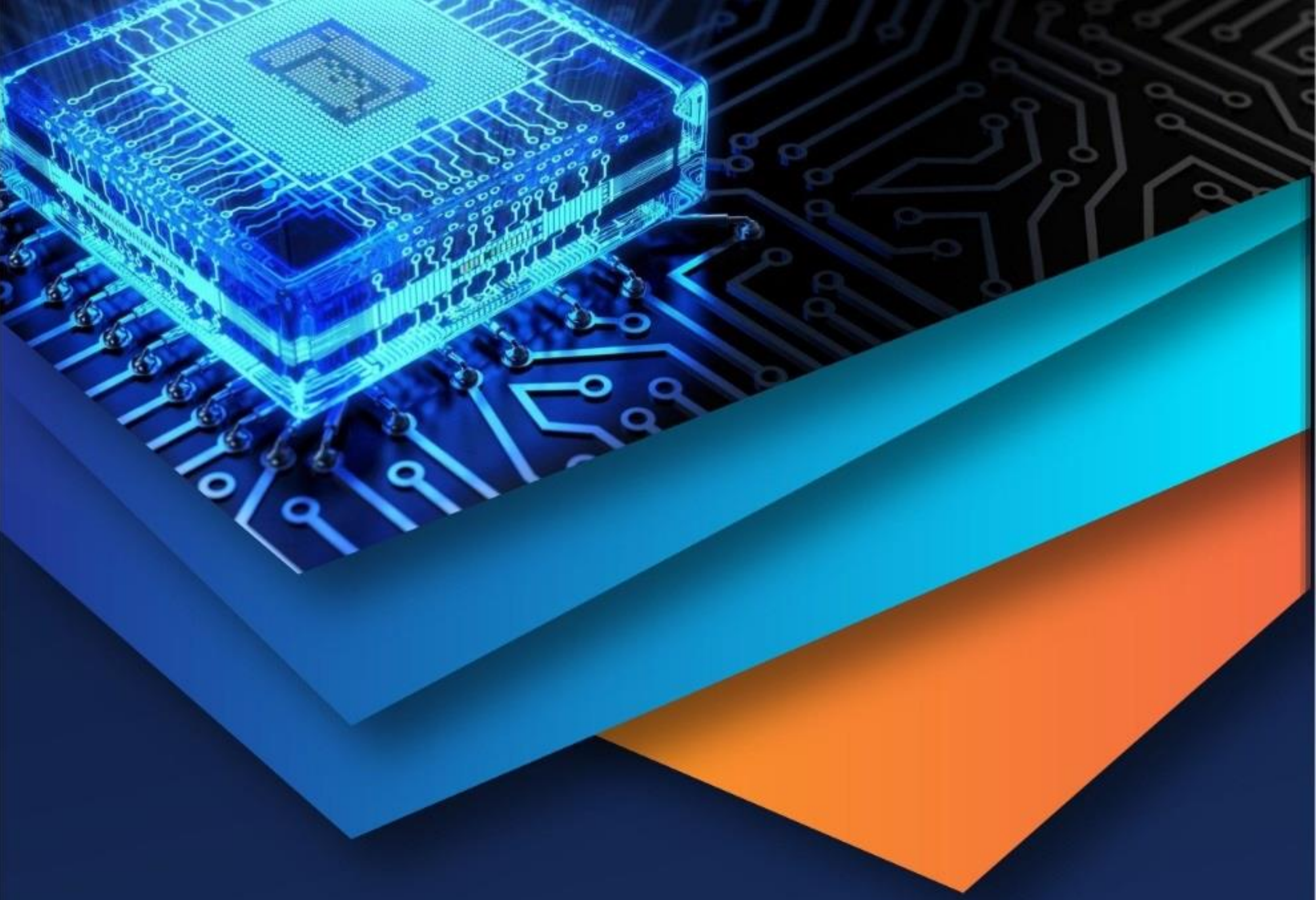

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