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A promising $Sr_5(PO_4)_3Br:RE^{3+}$ ($RE^{3+}= Dy^{3+}$, Eu^{3+}, and Tb^{3+}) Phosphors for Near UV Excited White LED and Display Devices

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***Abstract** Single phase $Sr_5(PO_4)_3Br:RE^{3+}$ ($RE^{3+}= Dy^{3+}$, Eu^{3+} or Tb^{3+}) phosphors were prepared by solid state reaction method and optical properties were investigated for the possible applications in white light emitting diodes (LED) and display devices. Prepared phosphors are characterized by X-ray diffraction, scanning electron microscopy, photoluminescence (PL) and FT-IR spectroscopy. The PL properties of $Sr_5(PO_4)_3Br:Dy^{3+}$ phosphor shows the characteristic emission of blue (479 nm) and yellow (575 nm) light under near UV excitation of 387nm, $Sr_5(PO_4)_3Br:Eu^{3+}$ shows characteristic orange (590 nm) and red (613 nm) under near UV excitation of 396 nm and $Sr_5(PO_4)_3Br:Tb^{3+}$ shows green (545 nm) emission under 379 nm excitation. The results indicate that the RE^{3+} activated bromoapatite phosphor could be promising candidate for near UV-based white LEDs and display devices.*

***Keywords:** Bromoapatite, FT-IR, Photoluminescence, Rare earth, SEM, XRD.*

I. INTRODUCTION

A phosphor is a luminescent material that absorbs radiation energy in a UV portion of the electromagnetic spectrum and emits energy in another visible portion of the electromagnetic spectrum. Phosphors are crystalline inorganic compounds in which small quantities of other elements, called “activators” are added to convert them into efficient fluorescent materials. With proper combination of activators and inorganic compounds, the color of the emission can be controlled. Most useful and well-known phosphors emit radiation in the visible portion of the electromagnetic spectrum in response to excitation by electromagnetic radiation outside the visible range. A light-emitting phosphor consists of a host lattice into which activator ions such as rare earths are doped at a few mol% [1]. White light-emitting diodes offer several advantages in terms of energy saving, reliability, and safety and therefore have become important in recent years. Nowadays, one of the most important part of optoelectronic devices are light emitting diodes (LEDs) because several factors are driving their development. The most important ones are brightness, efficiency flexibility, lifetime, rugged construction, low power consumption and suitable driving voltage. These properties are contributing to growth in markets such as traffic lights, automotive brake signals displays, decorative signs and the many uses of the new white LED-based products [2].

To increase the efficiency of white LEDs, special attention has recently been paid to the development of new phosphors with good luminescence properties that can be excited in the near UV range (350–420 nm) for display devices [3]. Apatite phosphors also known as alkaline-earth-metal halo phosphates, which are naturally occurring materials, have recently been used for field-emission displays (FEDs) and would be suitable for white LEDs in the near future because of their low price, environmental friendliness, thermal stability and good photoluminescence properties.

Apatites have the generic molecular formula $M_5(PO_4)_3X$ (M=Ca, Sr, Ba; X=F, Cl, Br, OH). Apatite is one of the most interesting types of new inorganic materials due to the ability of the tetrahedral PO_4^{3-} group to bond with other structural units. Apatites are well known phosphor materials for their applications as laser hosts, and biocompatible materials. Among these, $Ba_5(PO_4)_3Cl:Eu^{2+}$ is a promising material for X-ray imaging. $Sr_5(PO_4)_3Cl:Eu^{2+}$ is an efficient photo luminescent material, used in high efficiency compact fluorescent lamp. To the best of our knowledge the reports on optical properties of rare earth doped $Sr_5(PO_4)_3Br$ phosphors has not reported yet. Hence, in this work the PL study of Dy^{3+}, Eu^{3+} and Tb^{3+} doped $Sr_5(PO_4)_3Br$ phosphors are undertaken [3].

II. EXPERIMENTAL METHOD

Bromoapatite phosphors $Sr_5(PO_4)_3Br$ reported here were prepared by the solid state reaction method. The pure starting materials were $Sr(NO_3)_2$, $NH_4H_2PO_4$, NH_4Br, Dy_2O_3, Eu_2O_3 and Tb_2O_3 were used for preparation of $Sr_5(PO_4)_3Br$ materials. The formation of $Sr_5(PO_4)_3Br$ (pure) and $Sr_5(PO_4)_3Br:RE^{3+}$ are shown by following chemical reaction:

$$\mathbf{10Sr(NO_3)_2 + 6NH_4H_2PO_4 + 2NH_4Br = 2Sr_5(PO_4)_3Br + 19O_2\uparrow + 22H_2O\uparrow + 14N_2\uparrow}$$

In this synthesis the stoichiometric quantities of raw materials were weighted and mixed thoroughly by crushing mechanically for 1hr in agate mortar. These dry powders were then heated in a muffle furnace, at around 150^0C for 2hrs.

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The powders were again crushed and heated at 400^{0}C for 2hrs to remove all gases. The powders were crushed and finally annealed at about at 900^{0}C for 6 hours in air by slowly Increasing the temperature of muffle furnace, and then cooled down slowly to room temperature. The powder were crushed again in agate mortar as done previously. Finally the samples were quenched at 1100^{0}C after heating 1hour to observe the effect of raising the temperature on intensity of PL emission. Same method were utilized for the preparation of the rare earth (Dy^{3+}, Eu^{3+} and Tb^{3+}) doped phosphors in various molar concentration (0.1-2mol%).

The X-ray powder diffraction method using Cu Kα radiation was used for the prepared host lattice $Sr_5(PO_4)_3Br$ to characterized it for phase purity and crystallinity. Phosphors morphology was checked by a scanning electron microscope (SEM). Bruker Fourier transform infrared spectrometer was used to record infrared spectra. It was taken in the range of 500 to 4000cm^{-1}. The excitation and emission spectra, for the measurement of spectroscopic properties, were recorded at room temperature on Shimadzu make RF5301PC spectro-fluorometer using a spectral slit width of 1.5nm.for all samples [4]-[6].

III. RESULTS AND DISCUSSION

A. X-ray Diffraction (XRD)

X-ray diffraction analysis was carried out to confirm the forming of prepared host by solid state method. Figure 1 shows the diffraction data pattern for the prepared pure $Sr_5(PO_4)_3Br$ phosphor host. The sharp diffraction peaks of XRD patterns of strontium bromoapatite confirms the forming of a crystalline $Sr_5(PO_4)_3Br$ host. It shows good similarity with ICDD data file 89-5876 of $Sr_5(PO_4)_3Br$.

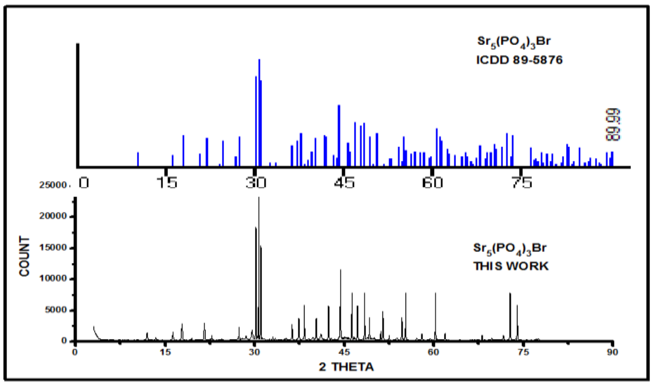

Figure 1: XRD pattern of synthesized Bromoapatite Sr5(PO4)3Br Phosphors.

The XRD diffraction pattern of prepared $Sr_5(PO_4)_3Br$ host did not indicate existence of the nitrate component and the remains of ammonia. This shows an indirect evidence for the homogeneous formation of the prepared $Sr_5(PO_4)_3Br$ host.

The crystal structure of prepared $Sr_5(PO_4)_3Br$ host was refined precisely using the single crystal structure model of $Sr_5(PO_4)_3Br$ from XRD data, earlier by the Rietveld method [7] . The prepared $Sr_5(PO_4)_3Br$ host has same structure with $Sr_5(PO_4)_3Br$ and belongs to P63/m space group with cell parameters are a0 = 9.9641(1)Å, c0 = 7.2070(1) Å, α=β=90°, γ=120°, Z=2 [8] [8]-[10].

B. Surface Morphology

The surface morphology and crystallite sizes were investigated by scanning electron microscope analysis for the prepared $Sr_5(PO_4)_3Br$ host. It is shown in figure 2. The SEM observations of the sample shows highly agglomerated crystallite morphology. Estimation of particle size is uncertain as the particles are highly amassed together. At higher magnification, SEM analysis shows the presence of particles in the submicron range in some regions of the SEM micrographs with irregular spherical morphology [11].

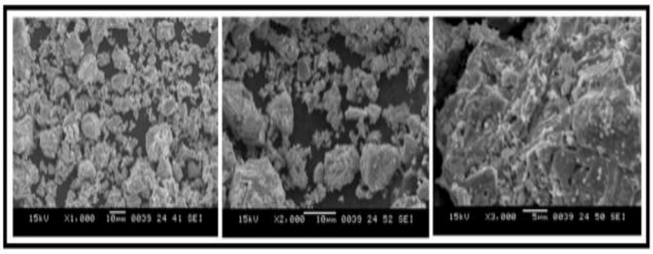

Figure 2: Scanning electron microscope (SEM) micrographs of synthesized Bromoapatite $Sr_5(PO_4)_3Br$.

C. FT-IR Analysis

The infrared spectrum for bromoapatite $Sr_5(PO_4)_3Br$ phosphors is as shown in figure 3. The phosphate group is the only and essential polyatomic ion in bromoapatite $Sr_5(PO_4)_3Br$ phosphors. A unit cell of phosphate group PO_4^{3-} contains three phosphorous atoms. Each phosphorous atom is surrounded by a tetrahedron of oxygen atoms. The most characteristic chemical group in the FTIR spectrum of synthesized bromoapatite is PO_4^{3-}.

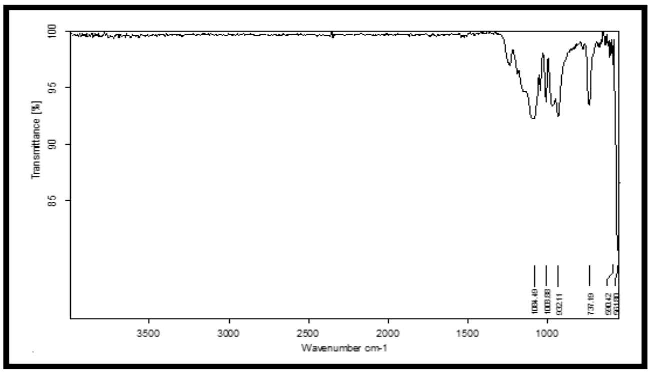

Figure 3: FT-IR spectrum of synthesised Bromoapatite Sr5(PO4)3Br phosphors.

The infrared spectra of bromoapatite show characteristic bands originating from P-O or O-P-O vibrations present in [PO_4] tetrahedral. These are the bands due to the symmetric stretching vibration ν_1 (933 cm^{-1}) and ν_3 asymmetric stretching modes (1084 cm^{-1} and 1008 cm^{-1}) vibrations as well as bands originating from the asymmetric vibrations ν_4 (590 cm^{-1}, 561 cm^{-1}) occurring within these tetrahedral. In addition, the peak at 737 cm^{-1}, implies the presence of $P_2O_7^{4-}$ group which is a characteristic to strontium bromo phosphate phase. It is further inferred that the prepared host is free from nitrate group (2213-2034 cm^{-1}).

It is also observed that OH- bands completely evaporated in a prepared bromoapatite host, implying that bromine ions have been substituted effectively and therefore the characteristic bands of the $(PO_4)^{3-}$ groups are observed [4].

D. PL Emission Of RE^{3+} In $Sr_5(PO_4)_3Br$

i) Luminescence Studies Of $Sr_5(PO_4)_3Br:Dy^{3+}$

The PL excitation and emission spectra of the as prepared Dy^{3+} activated $Sr_5(PO_4)_3Br$ phosphor are shown in figure 4. The excitation spectrum for all the $Sr_5(PO_4)_3Br$ phosphors with various Dy^{3+} concentrations was observed at 575nm emission corresponding to the $^4F_{9/2}\rightarrow{}^6H_{13/2}$ transition of Dy^{3+}. The excitation peaks observed at 350nm ($^6H_{15/2}\rightarrow{}^6P_{7/2}$), 365nm ($^6H_{15/2}\rightarrow{}^6P_{5/2}$) and 387nm ($^6H_{15/2}\rightarrow{}^4I_{13/2}$) were due to f-f transition [12]. We chose 387nm as excitation wavelength amongst all these three near UV excitation peaks, because it is more intense and suitable for solid state lighting for display devices [13].

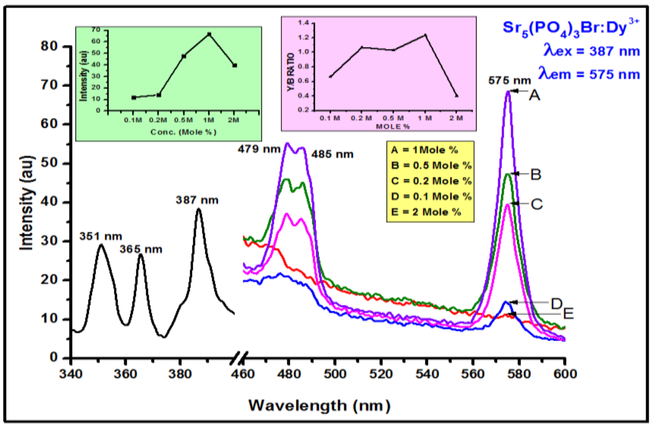

Figure 4: Excitation and emission spectra of Sr5(PO4)3Br:Dy3+ phosphors.

The emission spectra of $Sr_5(PO_4)_3Br:Dy^{3+}$ (Dy^{3+} mol% = 0.1, 0.2, 0.5, 1 and 2) phosphors excited at 387nm range display two predominant peaks at 479nm (blue, $^4F_{9/2}\rightarrow{}^6H_{15/2}$) and 575 nm (yellow, $^4F_{9/2}\rightarrow{}^6H_{13/2}$) zone[11]. The blue emission is due to magnetic dipole transition, and the yellow emission belongs to the hypersensitive (forced electric dipole) transition following the selection rule, $\Delta J = 2$ [4]. The magnetic dipole transition gives blue emission and the yellow emission is due to hypersensitive forced electric dipole hypersensitive transition. It follows $\Delta J = 2$ selection rule. It is known that the blue emission ($^4F_{9/2}\rightarrow{}^6H_{15/2}$) at 479nm is insensitive to the crystal field around the Dy^{3+} ions and the yellow emission ($^4F_{9/2}\rightarrow{}^6H_{13/2}$) at 575nm is hypersensitive transition and it is strongly influenced by the surrounding environment around Dy^{3+} [12]-[14]. In this prepared host, the stronger yellow emission implies that low symmetry sites (without inversion symmetry) have been occupied by Dy^{3+} ions. Due to the porosity of spinal structure, the Dy^{3+} ion may enter into the host lattice to substitute Sr^{2+} ions as the ionic radii of Dy^{3+} ions (912 pm) are much smaller than that of Sr^{2+} ions(118pm)[15].

Dependence of luminescence intensity on concentration of Dy^{3+} ions for $Sr_5(PO_4)_3Br:Dy^{3+}$ prepared phosphor is shown in inset of figure 4. The optimum concentration is 1mol% and decreases for higher concentration due to concentration quenching.

The yellow to blue (Y/B) emission intensity ratio is depicted in inset of figure 4. By carefully observing the emission spectra it is found that the yellow to blue (Y/B) emission intensity ratio is greater than 1. This confirms that Dy^{3+} ions occupy low symmetry site with no inversion symmetry in $Sr_5(PO_4)_3Br$ prepared host [16].

ii) Luminescence Studies Of Sr5(PO4)3Br:Eu3+

The PL excitation and emission spectra of the prepared $Sr_5(PO_4)_3Br:Eu^{3+}$ phosphors are shown in figure 5. It is obtained by tracking the red emission at 613nm and near UV excitation at 396nm, respectively. The excitation band observed at 396nm ($^7F_0\rightarrow{}^5L_6$) was sharp and caused by f-f transition.

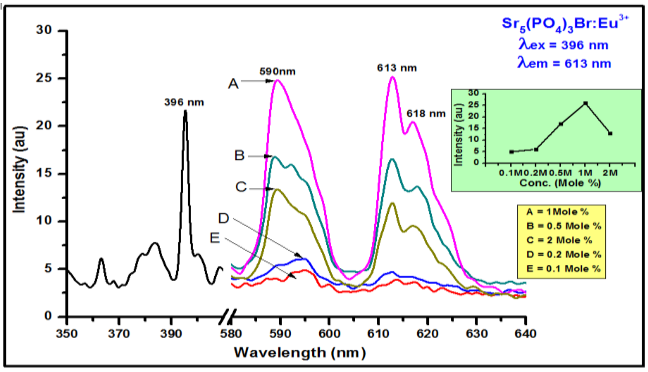

Figure 5: Excitation and emission spectra of Sr5(PO4)3Br:Eu3+ phosphors.

The characteristic emission bands of $Sr_5(PO_4)_3Br:Eu^{3+}$ phosphor are in the region of 580 to 640nm corresponding to intra 4f-shell transitions of $^5D_0\rightarrow{}^7F_J$ (J=0, 1, 2, 3, 4) of Eu^{3+}. With λ_{ex}= 396nm, the emissions at 590nm (Orange, $^5D_0\rightarrow{}^7F_1$, magnetic dipole) and 613 nm (Red, $^5D_0\rightarrow{}^7F_2$, electric dipole which is hyper sensitive) were found with emission peaks differing only in their relative intensity values. The intensity of emission at 590nm and 613nm was found to be almost equal.

Dependence of luminescence intensity on concentration of Eu^{3+} ions for $Sr_5(PO_4)_3Br:Eu^{3+}$ prepared phosphor is shown in inset of figure 5. The highest luminescent intensity is observed at 1 mol% and then decreases with the increase in concentration of the Eu^{3+} activator ions due to concentration quenching effect [4][17].

iii) Luminescence Studies Of Sr5(PO4)3Br:Tb3+

The PL excitation and emission spectra of the prepared Tb^{3+} activated $Sr_5(PO_4)_3Br$ phosphors are shown in figure 6. The excitation peaks associated with f-f transitions were observed at 351nm ($^7F_6\rightarrow{}^5L_9$), 371nm ($^7F_6\rightarrow{}^5G_5$) and 379nm ($^7F_6\rightarrow{}^5G_6$). Amongst these, excitation wavelength above 379nm is taken which is more suitable for solid state lighting and display devices [7][18].

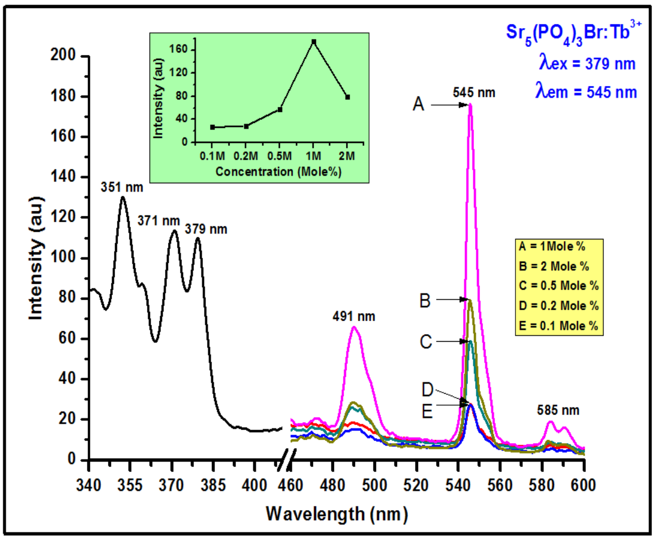

Figure 6: Excitation and emission spectra of Sr5(PO4)3Br:Tb3+ phosphors.

$Sr_5(PO_4)_3Br:Tb^{3+}$ phosphor shows sharp and intense narrow green emission ($^5D_4 \rightarrow {}^7F_5$, magnetic dipole transition ($\Delta J = \pm 1$)) at 545nm with other weak blue emission ($^5D_4 \rightarrow {}^7F_6$) at 491nm, and very weak yellow emission ($^5D_4 \rightarrow {}^7F_4$) at 585nm due to 4f - 4f transitions of Tb^{3+} ions. The PL emission peaks differs only in their relative intensity. The emission from the higher energy level ($^5D_3 \rightarrow {}^7F_J$ (J = 3, 4, 5, 6)) in the blue spectral region is not observed. The reason for this may be the formation of terbium ions pairs (Tb-Tb) and because of this the excitation energy of the $^5D_3$ level relaxes to the $^5D_4$ level by cross relaxation. Another possible reason for this may be due to fast relaxation from $^5D_3$ level since its lifetime is very small [19]-[21].

Dependence of luminescence intensity on concentration of Tb^{3+} ions for $Sr_5(PO_4)_3Br:Tb^{3+}$ prepared phosphor is shown in inset of figure 6. Up to 1 mol%, the PL intensity rises with increase of Tb^{3+} ion concentration and thereafter falls at higher concentrations due to concentration quenching [22].

iv) Effect Of Temperature On PL Intensity Of Sr5(PO4)3Br:RE3+ (Where RE3+= Dy3+, Eu3+, Tb3+)

The prepared samples of $Sr_5(PO_4)_3Br:RE^{3+}$ were quenched at 1100^0C for 1 hour and their excitation and emission spectra were compared with the samples annealed at 900^0C for 6 hours. The two spectra i.e. one annealed at 900^0C for 6 hours and the as-quenched one are identical but with a large difference in intensity. It implies that the local environment for the RE^{3+} ($RE^{3+} = Dy^{3+}$, Eu^{3+}, Tb^{3+}) ions remains same in both. A rise in emission intensity in as quenched sample is due to the better crystallinity of $Sr_5(PO_4)_3Br$, which further could be attributed to the reduced surface area due to the reduction of the concentration of inherent defective states or adsorbed species [23][24].

a) Effect Of Temperature On PL Intensity Of Sr5(PO4)3Br:Dy3+

In figure 7, excitation and emission spectra of a quenched sample of $Sr_5(PO_4)_3Br:Dy^{3+}$ is shown which indicates the radical change in the yellow to blue (Y/B) ratio. The yellow to blue (Y/B) ratio for $Sr_5(PO_4)_3Br:Dy^{3+}$ as quenched sample is found to be less than one indicating that magnetic dipole transition (Blue emission, $^4F_{9/2} \rightarrow {}^6H_{15/2}$) of Dy^{3+} take up high symmetry site (with an inversion centre), prominently [25]-[27].

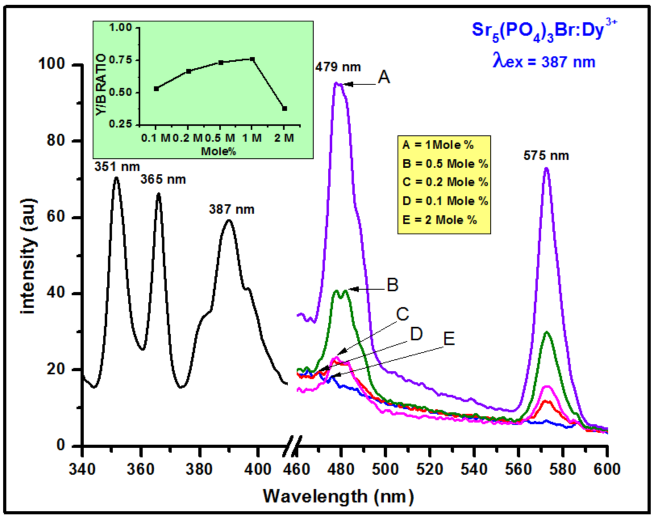

Figure 7: Excitation and emission spectra of quenched Sr5(PO4)3Br:Dy3+ phosphors.

b) Effect Of Temperature On PL Intensity Of Sr5(PO4)3Br:Eu3+

In figure 8, excitation and emission spectra of a quenched sample of $Sr_5(PO_4)_3Br:Eu^{3+}$ is shown which indicates the drastic change in the red to orange colour emission. It suggests that the local environment for the Eu3+ ion is same in both. The ration of red to orange colour emission for annealed and as quenched sample is found to be greater than one and hence we can infer that Eu3+ ions did not take up inversion symmetry site in $Sr_5(PO_4)_3Br$ [4].

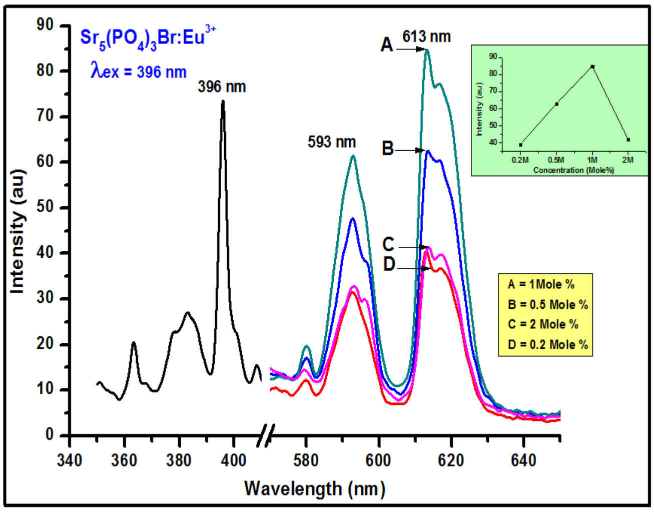

Figure 8: Excitation and emission spectra of quenched Sr5(PO4)3Br:Eu3+ phosphors.

c) Effect Of Temperature On PL Intensity Of Sr5(PO4)3Br:Tb3+

Figure 9 shows the excitation and emission spectra of a quenched sample of $Sr_5(PO_4)_3Br:Tb^{3+}$. The change in the intensity of excitation and emission spectra implies that the local environment for the Tb^{3+} ion is same in both.

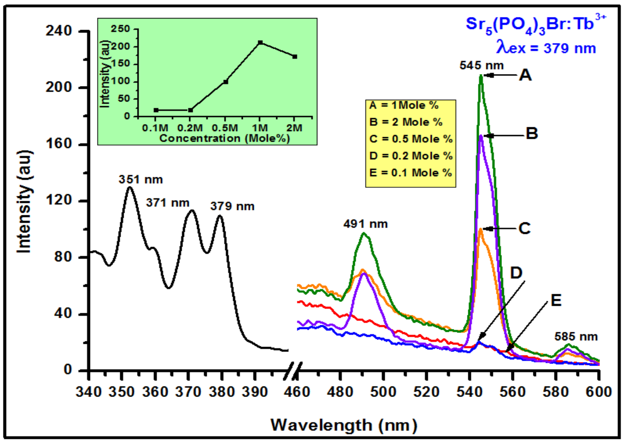

Figure 9: Excitation and emission spectra of quenched Sr5(PO4)3Br:Tb3+ phosphors.

E. Chromatic Properties

The figure 10 shows the CIE (Commission International de l Eclairage) chromaticity co-ordinates of the prepared phosphor $Sr_5(PO_4)_3Br:RE^{3+}$ (RE= Dy^{3+}, Eu^{3+}, Tb^{3+}). The (X, Y) chromatic co-ordinates are calculated using the colour calculator radiant imaging software for $Sr_5(PO_4)_3Br:RE$ are summarized in table III [4][13].

The comparison of percentage change in excitation and emission intensity of PL spectra is summarized in table I and table II, respectively.

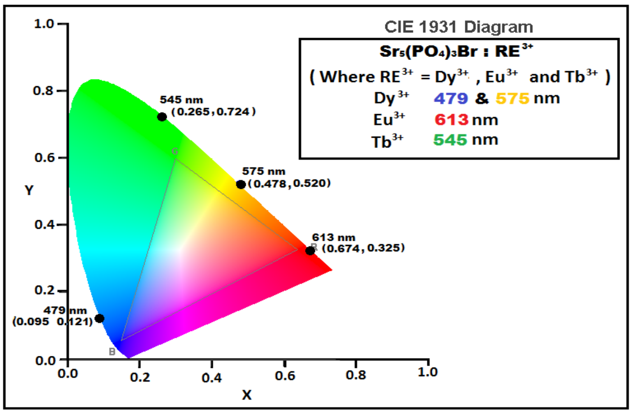

Figure 10: Chromatic graph of Sr5(PO4)3Br:RE3+ (RE3+ = Dy3+, Eu3+, Tb3+) phosphor excited near UV excitation.

Table I: % increase in intensity of excitation spectra of $Sr_5(PO_4)_3Br:RE^{3+}$ phosphors.

PHOSPHOR	Conc. (Mol%)	Excitation Spectrum (900⁰C, 6 Hours.)		Excitation spectrum (1100⁰C, 1 Hours.)		Increase in intensity (%)
		λex (nm)	Intensity (au)	λex (nm)	Intensity (au)	
$Sr_5(PO_4)_3Br:Dy^{3+}$	1	387	38	387	59	**55.26**
$Sr_5(PO_4)_3Br:Eu^{3+}$	1	396	22	396	74	**236.36**
$Sr_5(PO_4)_3Br:Tb^{3+}$	1	379	110	379	110	**00.00**

Table II: % increase in intensity of emission spectra of $Sr_5(PO_4)_3Br:RE^{3+}$ phosphors.

PHOSPHOR	Conc. (Mol%)	Emission Spectra (900⁰C, 6 Hours)		Emission spectra (1100⁰C, 1 Hours)		Increase in intensity (%)
		λ_{em} (nm)	Intensity (au)	λ_{em} (nm)	Intensity (au)	
$Sr_5(PO_4)_3Br:Dy^{3+}$	1	479 575	85 69	479 575	95 73	**11.76** **5.79**
$Sr_5(PO_4)_3Br:Eu^{3+}$	1	613	25	613	85	**240.00**
$Sr_5(PO_4)_3Br:Tb^{3+}$	1	545	177	545	210	**18.64**

Table III: The (X, Y) chromatic co-ordinates for synthesised $Sr_5(PO_4)_3Br:RE^{3+}$ phosphors.

PHOSPHOR	Excitation (nm)	Strong Emission (nm)	(X,Y) Coordinate	Colour Region
$Sr_5(PO_4)_3Br: Dy^{3+}$	387	479 575	(0.095, 0.121) (0.478, 0.520)	Blue Yellow
$Sr_5(PO_4)_3Br: Eu^{3+}$	396	613	(0.674, 0.325)	Red
$Sr_5(PO_4)_3Br: Tb^{3+}$	379	545	(0.265, 0.724)	Green

IV. CONCLUSION

The bromoapatite $Sr_5(PO_4)_3Br$ doped with different concentrations of RE^{3+} ($RE^{3+} = Dy^{3+}$, Eu^{3+}, Tb^{3+}) ions have been synthesized by solid state diffusion method and are characterized for their physical properties through XRD, SEM, FT-IR, absorption and luminescence spectra measurements. Solid state lighting phosphors uses Hg free excitation in the 300 to 400 nm range. The excitation spectra of $Sr_5(PO_4)_3Br$: RE3+ has shown good absorption in near UV region. The emissions of prepared $Sr_5(PO_4)_3Br$:RE^{3+} (RE= Dy^{3+}, Eu^{3+}, Tb^{3+}) phosphors are observed at 479 nm (Yellow), 575nm (Blue) for Dy3+ ions, 593nm (Orange), 613 nm(Red) for Eu^{3+} ions and 545 nm (Green) for Tb^{3+} ions, when excited by near UV excitation source. Based on the results obtained in the present work, we propose that, these optoelectronic luminescent phosphors have possible applications as a near UV convertible phosphor for white light emitting diodes and display devices.

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3. Spectroscopy of Lanthanide Doped Oxide Materials (Publisher: Elsevier)
4. Phosphate phosphors for solid state lighting (Publisher: Springer)
5. Phosphors for energy saving and conversion technology (Publisher:Taylor & Francis Group)
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