

Tb³⁺ doped Sr₂(BO₃)Cl green emitting phosphor for solid state lighting

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ABSTRACT

In this paper we present luminescence results on Tb³⁺ doped Sr₂(BO₃)Cl green phosphor. The vibrational properties of Sr₂(BO₃)Cl phosphor was studied by Fourier transform infrared spectroscopy. Photoluminescence studies have been carried out to understand the mechanism of excitation and the corresponding emission in the as prepared phosphor. As the Tb³⁺ ion is commonly used as an activator for the green emission, the excitation and emission spectra indicate that this phosphor can be effectively excited by 380 nm, to exhibit bright green emission centered at 546 nm corresponding to the f→f transition of Tb³⁺ ions. Copyright © 2014 VBRI press.

Keywords: Green emitting phosphor; borates; photoluminescence; XRD; SEM; FTIR.



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Introduction

Material based on the luminescent properties is widely used in daily life. Their best-known applications are solid state lighting application, CTV screen phosphors, projection TV phosphors, fluorescent lamps, scintillators, full color displays, X-ray storage, dosimetry of ionizing radiation screen intensifying phosphors, laser materials, etc. [1–2]. In recent years solid state lighting, particularly ultra violet light emitting diodes (UVLEDs) and their potential applications paying attention much interest for day-to-day lighting necessities. Light-emitting diode (LED)-based white light sources have already broadly used and have promising features such as low power consumption, high efficiency, longer lifetime, and mercury-free excitation [3,4], consequently they are expected to replace conventional incandescent and fluorescent lamps for general lighting application in the future [5].

Various researchers are engaged in studying the luminescent property of trivalent terbium ions in different compositions. Recently, Yerpude et al. reported Photoluminescence properties of $X_5\text{SiO}_4\text{Cl}_6:\text{Tb}^{3+}$ ($X = \text{Sr, Ba}$) green phosphor prepared via modified solid state method [6]. Research on tricolor phosphors suitable for near-ultraviolet/ ultraviolet excitation has paying considerable attention because of their significant applications in solid state lighting. Some of phosphors can be prepared using the rare earth ions such as Tb^{3+} . However, in these systems, the emission relies on D to f transitions which are parity forbidden and therefore rather inefficient. In addition, the lower lying f -orbitals are rather well-shielded from the coordination surroundings of the ion and, consequently, the emission arising from those D - f transitions are sharp and not very appropriate for covering a large region of the observable spectrum. The emission quantum effectiveness depends on the phonon energy of the host material in rare earth doped phosphor. Therefore, selection of host is very important for the examination of luminescence properties. Borate based phosphors show excellent chemical constancy; long duration and great brightness. Tb^{3+} ion in terms of practical applications are utilized in various fields in emissive products. The emission of Tb^{3+} ions is very much affected by the host lattice because of crystal-field effects. Borate based inorganic compounds doped with rare earth ions from significant family of phosphors for solid state lighting applications. Solid state lighting using light emitting diode and phosphor material to produce white light is the current research focus in the lighting industry. Inorganic luminescence materials have useful applications in many devices for simulated light manufacture [7].

Solids state lighting technology has several advantages over conventional fluorescent lamp such as reduced power consumption, compactness, efficient light output and longer lifetime. Solid state lighting will have its impact on reducing the global electricity consumption [1]. Recently, inorganic luminescence materials have practical applications in many devices for artificial light production. 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 tip to be the next generation solid state lighting, in the substitute of conventional incandescent and fluorescent lamps which are Hg pollutants, frangible and high energy consumption [8, 9]. In the recent years, research on the phosphors used for white LEDs has become a warm topic and gained growth. A large number of papers have been published in the related journals about the successful synthesis and luminescence characterization of rare earth doped phosphors with the orientation of exploring phosphors suitable for solid state lighting. As an important family of materials, related to luminescence, borates have been paid intense attention because of their excellent properties.

Rare earth doped borates have emerged as a original class of luminescence materials, showing merits of high stability, brightness, and flexible industrial processing ability that are suitable for solid state lighting and display devices [8,9]. Many phosphors such as $\text{BaAl}_2\text{B}_2\text{O}_7:\text{Tb}^{3+}$, and $\text{SrAl}_2\text{B}_2\text{O}_7:\text{Tb}^{3+}$, $\text{LiCaBO}_3:\text{Tb}^{3+}$ were developed for

their photoluminescence and high chemical stability. So far, green luminescence materials [10, 11] based on rare earth ions activated strontium borate has been manufactured by the solid-state reaction method. However, it should be noted that there is slight research on green light emitting phosphors based on strontium borate based host material. Trivalent terbium, the most stable state of terbium, is widely doped in several hosts for producing green color and bright photoluminescence. Due to these reasons; Tb^{3+} ion is the subject of numerous optical spectroscopic and luminescence investigation for the next generation of displays and lighting devices [12].

Host material selection and the concentration of rare earth ions i.e. dopant is very much important to be considered in developing more efficient results. Therefore, trivalent terbium plays very vital role to produce green emission so $\text{Sr}_2(\text{BO}_3)\text{Cl}$ with trivalent terbium doped is likely to be good green phosphors for solid state lighting industries. In this present work, we have prepared and investigate different concentration of Tb^{3+} doped strontium borate chloride phosphor. XRD, SEM FTIR and emission and excitation spectra were recorded for all concentration of Tb^{3+} . From the emission spectra, concentration dependent luminescence intensities are analyzed.

Experimental

The samples $\text{Sr}_2\text{BO}_3\text{Cl}$ and $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ were prepared by a step wise modified solid state reaction method, shown in Fig. 1. In this process the powders produced from solid state reaction is very fine as well as the cross contamination is very less, [b] while preparing the samples, the constituents SrCO_3 (Loba, 99%), NH_4Cl (Loba, 99%), H_3BO_3 (Loba, 99%) and Tb_4O_7 (Hi-Media, 99.9%) were taken in a weight of stoichiometric ratio and crushed in a mortar pestle for one hour. Then this material was heated at 400°C for 3 hour by slowly increasing the temperature in the step of 100°C , after 3 hour heating the material was again crushed for one hour and finally heated at 800°C for 12 hours resulting in the compounds $\text{Sr}_2\text{BO}_3\text{Cl}$ and $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ in powder form according to the following chemical reaction.

The reaction is as follows



Normally, the luminescence property of phosphor is known to be strongly affected by the concentration of the activator. This synthesis route is very easy and does not require expensive as well as sophisticated equipments. Such a high temperature leads to formation and crystallization of phosphor materials. The most important advantage of solid state reaction method is, the final product in solid form is structurally pure with the desired properties depending on the final sintering temperatures [13]. Until now, this technique has been employed to produce a variety of materials such as oxides, borates, silicates, and carbonates.

The resultant product is the phosphor which occupies the entire volume of the reaction vessel. The energy released from exothermic reaction between the starting compound and fuel can quickly heat the system to high temperatures without an external heat source. Synthesized

phosphors powders are generally more consistent, have fewer impurities, and have higher surface areas than powders prepared by conventional solid-state methods [2]. All the measurements were performed at room temperature. In this process the powders produced from solid state reaction method is very fine as well as the cross contamination is very less. This method is environment friendly and no toxic or unwanted waste is produced after the solid state reaction is complete. This method is also very convenient for large scale production on industrial scale [14].

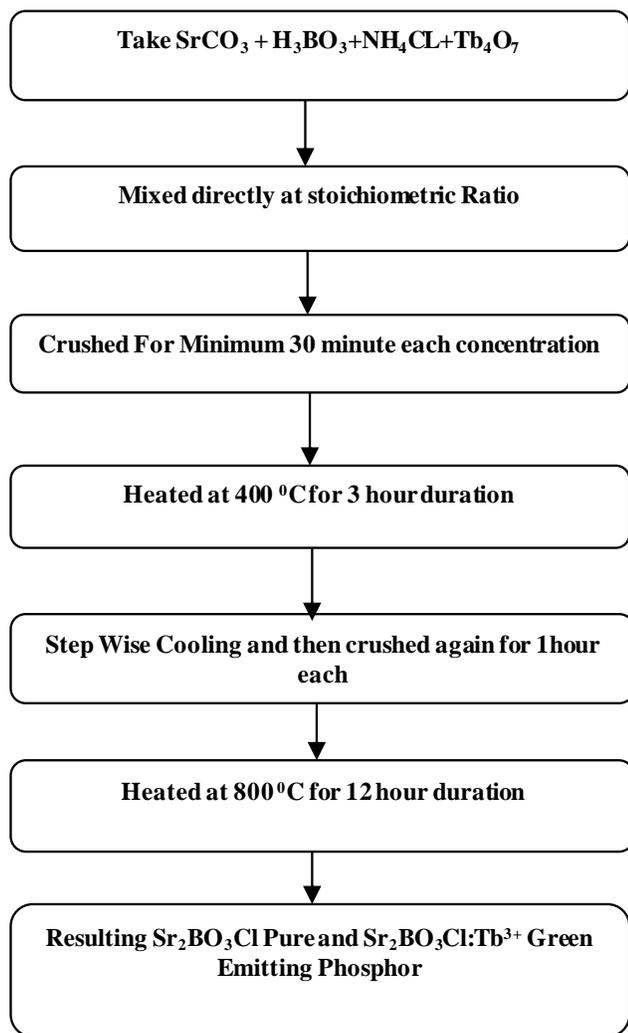


Fig. 1. Flow Chart of Preparation of $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ Phosphor.

The samples were then slowly cooled at room temperature, at cooling rate of $0.5\text{ }^\circ\text{C} / \text{min}$. The resultant polycrystalline material was crushed to fine powder in a mortar pestle; the resultant powder formed was used for further study. Several corresponding methods were used to characterize the prepared phosphors. SEM micrographs were obtained using a HITACHIS-4800 scanning electron microscope. The SEM micrographs were taken at 5000V accelerating voltage, 8300 μm working distance, 7800 nA emission current, at high lens mode with fast scan speed and gray scale color mode. The prepared host lattice was characterized for their phase purity and crystallinity by X-ray powder diffraction (XRD) using X'pert-PRO PAN

analytical diffractometer (Cu-K α radiation) at a scanning step of 0.001. The photoluminescence (PL) emission spectra of the samples were recorded using a Fluorescence spectrometer (Shimadzu, RF5301PC). The photoluminescence measurements of excitation and emission were recorded on the Shimadzu RF5301PC spectrofluorophotometer using a spectral slit width of 1.5nm. The same amount of sample was used for each measurement. All the measurements were performed at room temperature.

Results and discussion

XRD analysis

It is well established fact that every crystalline material gives a exceptional x-ray diffraction pattern (comparable with fingerprint) with a very few exceptions. A careful study of diffraction pattern of unknown material offers a powerful means of a qualitative identification of various phases present therein. It is therefore proved to be a quick and non-destructive method for an identification of compounds. In addition to this the information obtained on lattice parameter using the x-ray powder diffraction data gives better impending to structural changes produced in compound by physical or chemical process. These merits put this technique above all, which are, in general, used for structural characterization of luminescent material. Fig. 2 shows XRD-pattern of $\text{Sr}_2\text{BO}_3\text{Cl}$ phosphor there is no XRD-pattern of $\text{Sr}_2\text{BO}_3\text{Cl}$ phosphor is not available in the standard JCPDS data source.

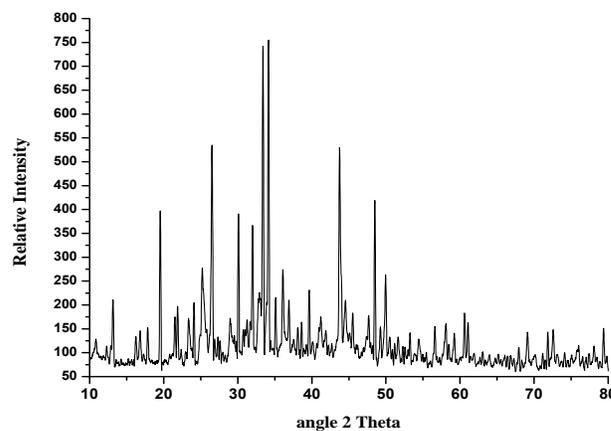


Fig. 2. XRD structure of $\text{Sr}_2\text{BO}_3\text{Cl}$ phosphor.

The crystal phase composition of the synthesized powder was analyzed by X-ray diffraction (XRD) pattern. Powder XRD pattern was recorded on X-ray diffractometer with using X-ray powder diffraction (XRD) using X'pert-PRO PAN analytical diffractometer (Cu-K α radiation) at a scanning step of 0.001. The high intensity of the peaks discovered the high crystallinity of the synthesized luminescent phosphor powder, and only the $\text{Sr}_2\text{BO}_3\text{Cl}$ phosphor phase was detected and no other products or starting materials were observed. Furthermore, we found that the little amount of doped rare-earth ions has almost no effect on the $\text{Sr}_2\text{BO}_3\text{Cl}$ phosphor phase composition. It observed that phosphor had good crystalline nature. The

dopants have no obvious influence on the crystalline structure of the host [15,16]. The XRD pattern did not show presence of the constituents and other likely phases which is a direct evidence for the formation of the desired compound. These results show that the final product was formed in crystalline and consistent form [17].

SEM analysis

The Scanning electron microscopy has been the most adaptable technique, judged by its widespread use in almost all branches of science and more appropriately by the importance of the information, which it has provided. The SEM is claimed as a considerable scientific technique for characterization. In addition to this, the most important advantage of SEM is due to its outstanding resolution when compared with that of the optical microscope. Vastly enlarged depth of focus in this technique is also of special value. Indeed, the advantages highlighted above, of SEM have covered the way exceptionally to study the morphology of material. It was carried out to investigate the surface morphology of the phosphor and crystallite sizes of the synthesized phosphor powder but morphology of the $\text{Sr}_2\text{BO}_3\text{Cl}$ phosphor which has small crystal grains. The particles possess lathered like morphology formed from highly agglomerated crystallites. The average crystallite size is in sub-micrometer range as shown in Fig. 3 a crystallite sizes are nearly same for all the compositions prepared. An average crystallite size is in the ten micrometer range. It can be seen clearly from these images that the sample exhibits very excellent shape.

Photoluminescence study

A series of $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ phosphors with various Tb^{3+} concentrations were prepared and the effect of doped Tb^{3+} concentration on the emission intensity is investigated. The photoluminescence excitation and emission spectra were recorded at room temperature. The emission spectrum of trivalent terbium doped sample $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ phosphor are shown in Fig. 4 and the excitation spectrum of $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ phosphor are shown in Fig. 5 by monitoring the emission at 545 nm. When the emission wavelength is monitored at 545 nm, the excitation spectrum consists band, due to the d-f transition of trivalent terbium in the wavelength 380 nm. Actually rare earth ions have been considered the most important optical activators for luminescent devices. The aim is to develop luminescent materials of three basic colors with higher radiative efficiency.

The spectrum of Tb^{3+} doped $\text{Sr}_2\text{BO}_3\text{Cl}$ phosphor consist of nine groups of emission lines peaking at about 425, 437, 450, 475, 500, 545, 580, 586, 625 and 635 nm the groups are due to the transitions of the $^5\text{D}_3 \rightarrow ^7\text{F}_5$, $^5\text{D}_3 \rightarrow ^7\text{F}_4$, $^5\text{D}_3 \rightarrow ^7\text{F}_3$, $^5\text{D}_3 \rightarrow ^7\text{F}_{1/2}$, $^5\text{D}_4 \rightarrow ^7\text{F}_6$, $^5\text{D}_4 \rightarrow ^7\text{F}_5$, $^5\text{D}_4 \rightarrow ^7\text{F}_4$, and $^5\text{D}_4 \rightarrow ^7\text{F}_3$, $^5\text{D}_4 \rightarrow ^7\text{F}_2$, $^5\text{D}_4 \rightarrow ^7\text{F}_1$ of the trivalent terbium respectively. When the emission spectra was monitored at 545 nm, it consisted of one band, due to the $^5\text{D}_4 \rightarrow ^7\text{F}_5$ 5d transition of Tb^{3+} in the excitation wavelength at 380 nm. The obtained emission spectrum of $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ originates from D-F transition lines within electronic configuration of Tb^{3+} , i.e., in the blue region $^5\text{D}_3 \rightarrow ^7\text{F}_5$ (425 nm), in the blue region $^5\text{D}_3 \rightarrow ^7\text{F}_4$ (437 nm), $^5\text{D}_3 \rightarrow ^7\text{F}_3$ (450

nm), $^5\text{D}_3 \rightarrow ^7\text{F}_{1/2}$ (475 nm), $^5\text{D}_4 \rightarrow ^7\text{F}_6$ (500 nm), and the green region $^5\text{D}_4 \rightarrow ^7\text{F}_0$ (545 nm), $^5\text{D}_4 \rightarrow ^7\text{F}_4$ (580 nm), $^5\text{D}_4 \rightarrow ^7\text{F}_3$ (625 nm), $^5\text{D}_4 \rightarrow ^7\text{F}_5$ (545 nm). The strongest emission peak is located at 545 nm corresponding to $^5\text{D}_4 \rightarrow ^7\text{F}_5$ transition of Tb^{3+} .

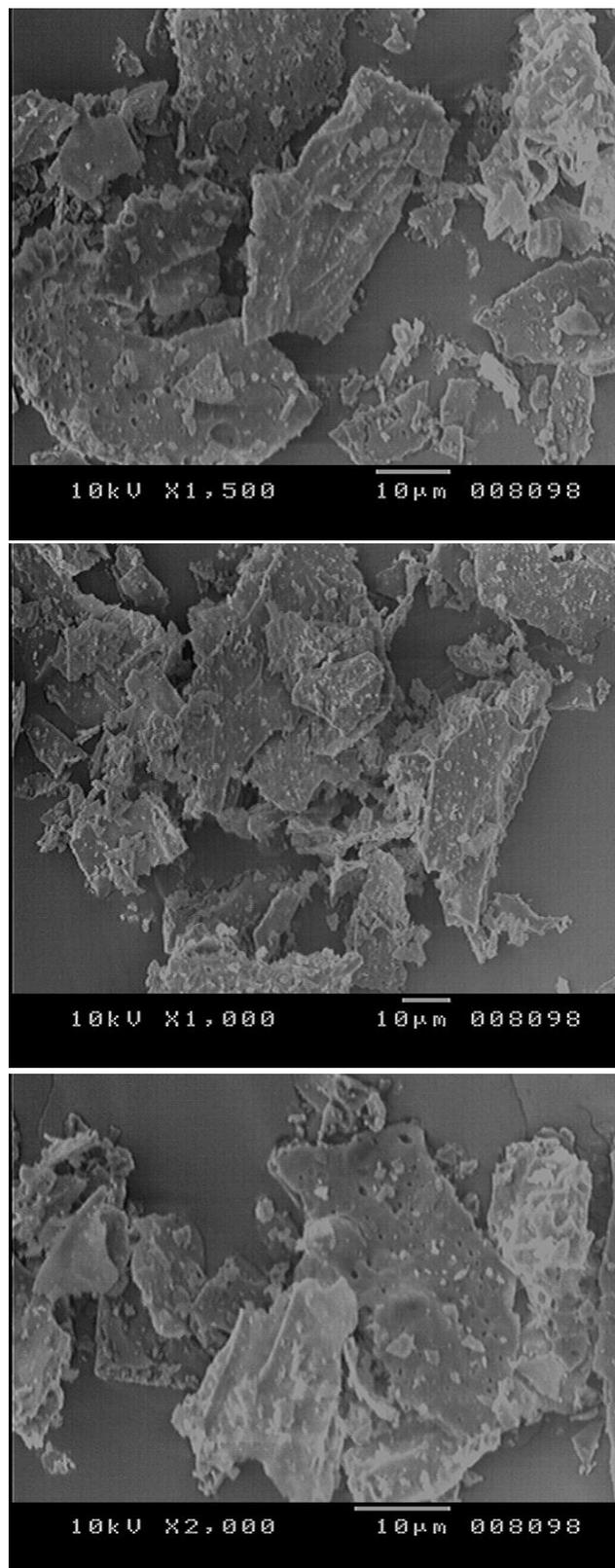


Fig. 3. SEM Morphology of $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$

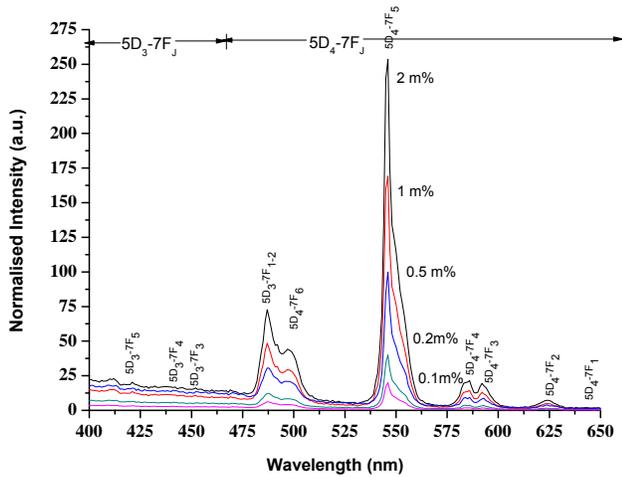


Fig. 4. Emission spectra of $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ under excitation at 380 nm.

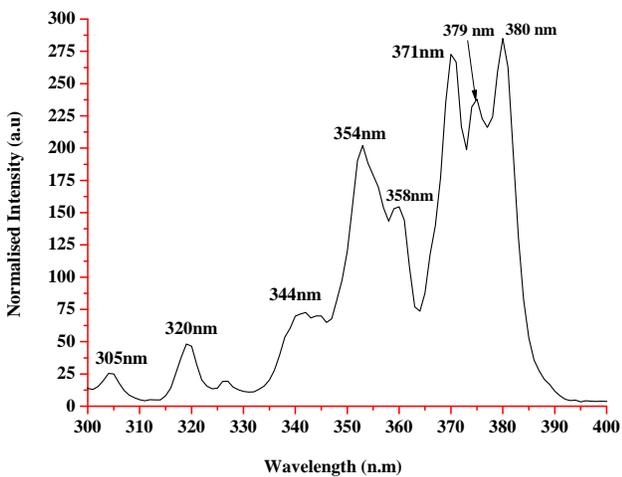


Fig. 5. Excitation spectra of $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ monitoring the emission at 545 nm.

The strongest $^5\text{D}_4$ to $^7\text{F}_5$ transition due to its major probability for both electric-dipole and magnetic-dipole induced transitions make the sample intense green-light emitting. In the emission spectra, the peak positions did not change as Tb^{3+} concentration varied. The Tb^{3+} ion shows luminescence professionally in borate compounds. The Tb^{3+} ion shows acceptable optical transitions in absorption and emission which are of the f-d type. When the $\text{Sr}_2\text{BO}_3\text{Cl}$ is doped with 2 mole % Tb, the intensity of observed emission spectra reaches the utmost value. The Photoluminescence emission properties of $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ were observed at 545 nm. Excitation band 380 nm in $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ was observed at 545 nm. The 545 nm emission observed in green region of the spectrum. All recorded spectra were corrected consequently to the apparatus characteristic. The emission spectrum of Tb^{3+} is strongly dependent on the Tb^{3+} concentration. The Photoluminescence results show that $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ phosphor posses the maximum emission intensity at 2 m%. The main emission band, located at 545 nm, gives rise to the Tb^{3+} well-known intense green luminescence, which indicates that it could be a good green phosphor candidate for solid state lighting industry applications. Green

luminescent $\text{Sr}_2(\text{BO}_3)\text{Cl}:\text{Tb}^{3+}$ powder phosphor under an UV source shown in following Fig. 6. The energy level diagram trivalent terbium, with labeled transitions that correspond to observed emission in the given phosphor material are shown in Fig. 7. As an important family of luminescent materials, borates have attracted much attention, not only owing to their excellent thermal stability, environmental benignity and potential low-cost synthesis, but also because a number of compounds with different structure can be selected [18].



Fig. 6. Green luminescent $\text{Sr}_2(\text{BO}_3)\text{Cl}:\text{Tb}^{3+}$ powder phosphor under an UV source.

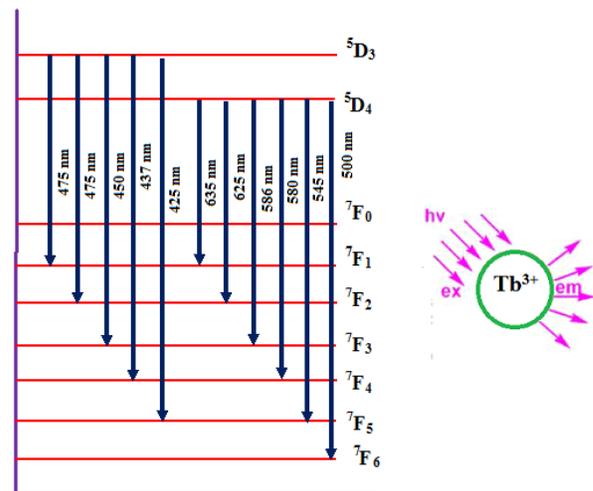


Fig. 7. Energy level diagram of trivalent terbium showing the energy level transitions.

The energy level transitions of Tb^{3+} ions are represented in Fig. 7. In general at lower doping concentrations of Tb^{3+} ions, the phosphor shows governance in the in the blue

region $^5D_3 \rightarrow ^7F_5$ (425 nm), in the blue region $^5D_3 \rightarrow ^7F_4$ (437 nm), $^5D_3 \rightarrow ^7F_3$ (450 nm), $^5D_3 \rightarrow ^7F_{1-2}$ (475 nm), $^5D_4 \rightarrow ^7F_6$ (500 nm), and the green region $^5D_4 \rightarrow ^7F_0$ (545 nm), $^5D_4 \rightarrow ^7F_4$ (580 nm), $^5D_4 \rightarrow ^7F_3$ (625 nm), $^5D_4 \rightarrow ^7F_5$ (545 nm). The strongest emission peak is located at 545 nm corresponding to $^5D_4 \rightarrow ^7F_5$ transition of Tb^{3+} , however with increasing concentrations of Tb^{3+} ions, the green peaks becomes stronger.

In the emission spectra we got peaks at 425 nm, 437 nm, 450 nm, 475 nm, 500 nm, 545 nm, 580 nm and 625 nm. A series of $Sr_2(BO_3)Cl$ terbium doped phosphors have been synthesized with the dopant concentration ranging from 0.1 to 2 mol % and maximum intensity is observed at 2 mol % molar concentration of Tb^{3+} ion.

FTIR analysis

The vibrational properties of Sr_2BO_3Cl phosphor was studied by Fourier transform infrared spectroscopy, which is shown in Fig. 8 consists of four peaks with energies ranging from 643 to 1451 cm^{-1} such as 1451.1903, 1239.3066, 747.7806, 642.6501 correspond to the formation of the Sr_2BO_3Cl phosphor and areas signed to the characteristic metal-oxygen vibrational modes in its structure. The trivalent terbium ion shows luminescence efficiently in strontium borate based compounds. The trivalent terbium ion shows allowed both electric-dipole and magnetic-dipole induced transitions make the sample show intense green-light emitting light seems to indicate that the Sr_2BO_3Cl phosphor is suitable for solid state lighting.

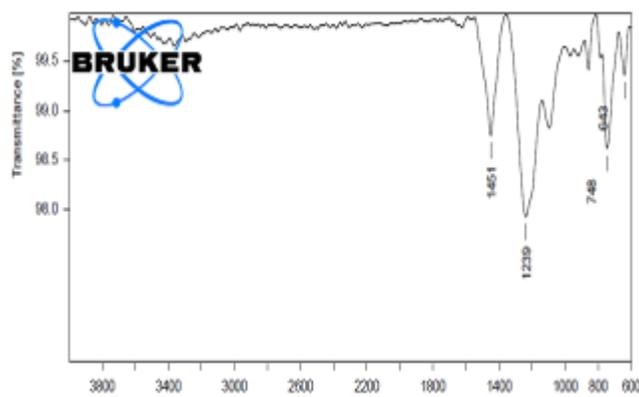


Fig. 8. Fourier Transform Infrared spectroscopic representation of phosphor.

Chromatic properties of $Sr_2BO_3Cl:Tb^{3+}$ phosphor

Chromaticity is an objective specification of the quality of a color regardless of its luminance, that is, as determined by its hue and colorfulness (or saturation, chroma, intensity, or excitation purity). Chromatic properties of Tb^{3+} singly doped Sr_2BO_3Cl phosphor. Generally lighting specifications refer to color in terms of the 1931 CIE chromatic color coordinates which recognizes that the human visual system uses three primary colors: red, green, and blue [19, 20]. All the results calculated from the emission spectra in Fig. 04 are plotted in the Commission International de l'Éclairage (CIE) 1931 chromaticity diagram, as shown in Fig. 09 It indicates that present

phosphors are close to the edge of CIE diagram, which indicates the high color purity of these phosphors [21]. In general, the color of any light source can be represented on the coordinates in this color space. The CIE chromaticity diagram of trivalent terbium doped Sr_2BO_3Cl phosphor is shown in Fig. 9. The chromatic coordinates (x, y) were calculated using the color calculator program radiant imaging [22]. The coordinates of the $Sr_2BO_3Cl:Tb^{3+}$ phosphor are, $C_x=0.269$, $C_y=0.717$. It indicates that the present phosphors are close to the edge of CIE diagram, which indicates the high color purity of these phosphors.

Fig. 9 shows the Commission International del O Eclairage (CIE) chromaticity coordinates of the $Sr_2BO_3Cl:Tb^{3+}$ phosphor. The chromaticity coordinates $Sr_2BO_3Cl:Tb^{3+}$ phosphor are $C_x=0.269$, $C_y=0.717$, which exactly fall into the green region in the CIE 1931 chromaticity diagram. As seen in the Photoluminescence spectra, the $Sr_2BO_3Cl:Tb^{3+}$ phosphor have green emission under the UV light. It means the $Sr_2BO_3Cl:Tb^{3+}$ phosphor could be a good green phosphor candidate for solid state lighting applications.

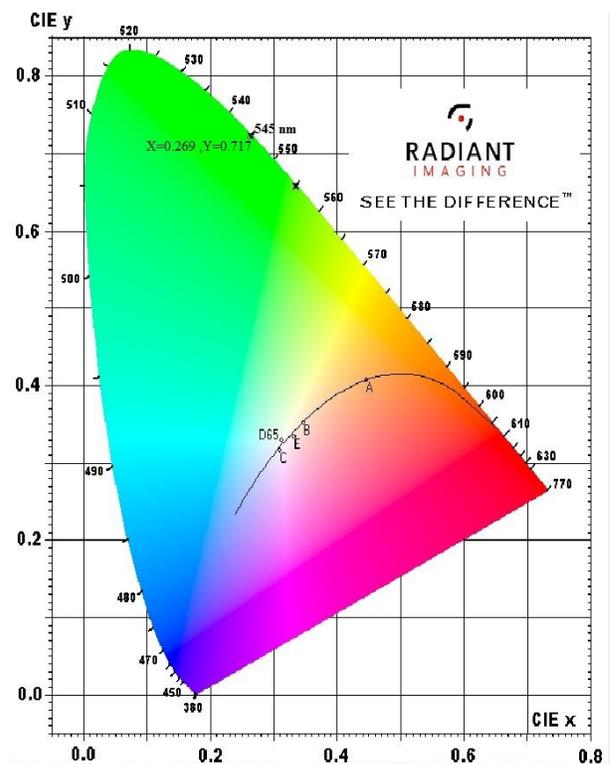


Fig. 9. Chromatic properties of $Sr_2BO_3Cl:Tb^{3+}$ phosphor.

Effect of Concentration of trivalent terbium ion on emission intensity of host lattice

In order to optimize the luminescence properties of Tb^{3+} in Sr_2BO_3Cl phosphor, the relationship between emission intensity and concentration of Tb^{3+} ion is shown in Fig. 10. The effect of doped Tb^{3+} concentration on emission of Sr_2BO_3Cl was also studied. The emission spectra of $Sr_2BO_3Cl:Tb^{3+}$ phosphor prepared at various concentration of trivalent terbium excited by 380 nm is shown in fig. 04. From Fig.10, it is observed that with the increase of Tb^{3+}

concentration, the intensities of the emission lines are enhanced considerably and reaching a maximum at a concentration of 2 mole % and then decrease.

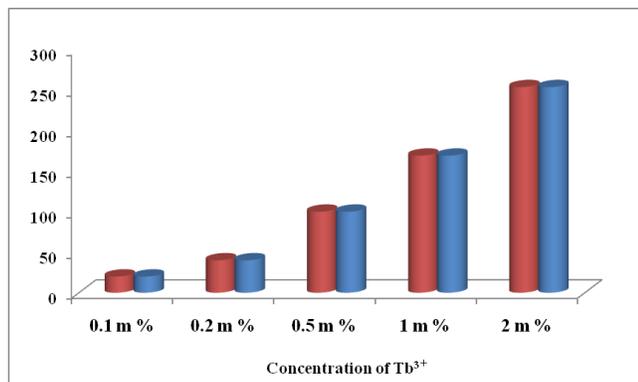


Fig. 10. Effect of Concentration of trivalent terbium ion on emission intensity of host lattice.

Conclusion

The trivalent terbium doped $\text{Sr}_2\text{BO}_3\text{Cl}$ green emitting phosphors synthesized by step wise solid state reaction method and their luminescent properties were investigated. The green emission from ${}^5\text{D}_4 \rightarrow {}^7\text{F}_5$ at 545 nm showed the utmost intensity. A single phase phosphor product was obtained evaluated from x-ray diffractometer. The photoluminescence results show that $\text{Sr}_2\text{BO}_3\text{Cl}:\text{Tb}^{3+}$ phosphors posses the highest emission intensity, that means it shows strong green emission at ${}^5\text{D}_4 \rightarrow {}^7\text{F}_5$ at 545 nm. Prepared terbium activated $\text{Sr}_2\text{BO}_3\text{Cl}$ phosphors having strong prospective application for solid state lighting application. Photoluminescence, XRD, scanning electron microscopic behavior, FTIR and color chromaticity of terbium doped strontium borate based phosphor was studied.

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