



CUPPER DOPED STRONTIUM HEXABORATE FOR DOSIMETRY APPLICATIONS

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Abstract

The polycrystalline sample of $\text{Sr}_{(1-x)}\text{B}_6\text{O}_{10}: x \text{Cu}$ was prepared by employing modified solution combustion synthesis method. The prepared sample was confirmed by X-ray diffraction (XRD) technique. The Scanning Electron Microscope (SEM) shows the sizes of particles from 0.1 μm to 2 μm range. By exposing with gamma-rays its thermoluminescence properties were recorded. Thermoluminescence (TL) glow curve of $\text{Sr}_{0.3}\text{B}_6\text{O}_{10}: \text{Cu}_{0.7}$ phosphor following irradiation with ^{60}Co gamma-ray source shows a single peak at 229 °C for the heating rate of 5 °C/sec. The phosphor is found to be about 90 % sensitive to that of commercially available LiF: Mg, Cu, P (TLD-100H) phosphor. The effect of dose variation on $\text{SrB}_6\text{O}_{10}: \text{Cu}$ was found to be linear up to 25 Gy dose with fading of about 33 % in 25 days.

Keywords: Thermoluminescence; Combustion Synthesis; Strontium Hexaborate.

1. Introductions

The strontium borate polycrystalline compound $\text{SrB}_4\text{O}_7: \text{Dy}$ exhibit thermoluminescence which acts as perspective material for solid state dosimetry [1]. The mechanism of incorporation of divalent rare-earth ions into $\text{SrB}_6\text{O}_{10}$ obtained by solid state synthesis in the air was discussed for Eu [2]. Hydrous red phosphor for the same host $\text{SrB}_6\text{O}_{10}$ was found in literature for its nano structure [3]. Some other activator like Tb was also tried by the researcher in $\text{SrB}_6\text{O}_{10}$ with co-dopant like Ce and Li for its thermoluminescence properties with gamma

irradiations. [4] Thermoluminescence of borates containing various dopants, such as $\text{Li}_2\text{B}_4\text{O}_7: \text{Cu}$ [5], $\text{CaB}_4\text{O}_7: \text{Cu}$ [6] and $\text{K}_2\text{B}_4\text{O}_7: \text{Cu}$ [7] has been extensively investigated by the researcher in last decade. The copper containing materials are among the most sensitive known thermoluminescence (TL) phosphors [8]. These phosphors are suggested to be used for dosimetry applications [9, 10]. We have already synthesized phosphors in this category [11-15]. In this paper we have thoroughly studies the thermoluminescence properties of copper doped $\text{SrB}_6\text{O}_{10}$ phosphor.

2. Experimental

Polycrystalline $\text{Sr}_{(1-x)}\text{B}_6\text{O}_{10}: x \text{Cu}$ [$x = 0.001, 0.002, 0.005, 0.01$ and 0.02] phosphor is prepared by well-established modified solution combustion technique [16]. During the reaction, the stoichiometric amounts of ingredients all precursor strontium nitrate, urea (fuel), $\text{NH}_4\text{B}_5\text{O}_8$ (oxidizer) and CuCl_2 (activator) calculated on the basis of molar ratio and were thoroughly mixed in the agate mortar by adding little amount of double distilled water, an aqueous homogeneous solution was obtained. The solution was then transferred into a china basin. The china basin was then kept into preheated muffle furnace maintained at $(550 \pm 10)^\circ\text{C}$. The solution boils foams and ignites to burn with the flame and a voluminous, foamy powder was obtained. The entire combustion was over in 5 minutes. Following the combustion, the resulting fine powder was annealed in open air at 750°C for 3 hours and allowed to cool at room temperature and the prepared sample is ready for the different characterizations.

Table-1: Balance reaction for the phosphor

Product	Corresponding reaction with balance molar ratios of precursors
$\text{Sr}_{(1-x)}\text{B}_6\text{O}_{10}:\text{xCu}$	$\text{Sr}(\text{NO}_3)_2 + 1.2 \text{NH}_4\text{B}_5\text{O}_8 + 10.2 \text{CO}(\text{NH}_2)_2 + 10.2 \text{NH}_4\text{NO}_3 + \text{x}$ CuCl_2 $\text{Sr}_{(1-x)}\text{B}_6\text{O}_{10}:\text{xCu} + \text{Gaseous } (\text{H}_2\text{O}, \text{NH}_4 \text{ and } \text{NO}_2 \text{ etc})$ $[x=0.001, 0.002, 0.005, 0.01 \text{ and } 0.02]$

3. Results and Discussion

3.1 XRD Analysis and Crystal Structure of $\text{SrB}_6\text{O}_{10}:\text{Cu}$

Fig. 1 shows the powder XRD pattern for polycrystalline sample of $\text{SrB}_6\text{O}_{10}:\text{Cu}$ confirm on Rigaku Miniflex X-ray diffractometer with scan speed of 2.00 deg / min by Cu K α radiations. The results were confirmed by comparing the observed XRD spectra with the standard International Centre for Diffraction Data (ICDD) file (00-020-1190) which are in good agreement and show peak to peak matching. The reported lattice constants are $a = 12.66 \text{ \AA}$, $b = 8.542 \text{ \AA}$ and $c = 5.315 \text{ \AA}$ with $z = 4$ and volume of unit cell 574.77 \AA^3 and the values for $\alpha = 90^\circ$, $\beta = 90.30^\circ$, $\gamma = 90^\circ$. The formed material has Centrosymmetric structure with space group: P21/n.

The structure of $\text{SrB}_6\text{O}_{10}$ was discussed here analogy with $\text{CaB}_6\text{O}_{10}$. A $[\text{B}_3\text{O}_7]^{5-}$ tri-

borategroup is basic structural unit in $\text{SrB}_6\text{O}_{10}$. It has a six member ring in which twoboron atoms are surrounded by three oxygen atoms and oneboron atom coordinated by four oxygen atoms; these BO_3 (Δ) and BO_4 (T) groups are linked by vertices; the shorthand notation of the $[\text{B}_3\text{O}_7]^{5-}$ group is 3:2_ $_+$ T. There are two crystallographically independent $[\text{B}_3\text{O}_7]^{5-}$ groups, one formed by B_1 , B_2 and B_3 -centered polyhedra and another by B_4 , B_5 and B_6 -centered polyhedra. They are connected via the O_6 atom to form a $[\text{B}_6\text{O}_{13}]^{8-}$ group that contains six terminal oxygen atoms when it exists as an isolated ion as shown in Fig. 2. Each $[\text{B}_6\text{O}_{13}]^{8-}$ group is linked to six other similar groups through sharing all of the terminal O atoms to result in the formation of a 3D $3\infty[\text{B}_6\text{O}_{10}]^{2-}$ network. [17, 18]

$\text{SrB}_6\text{O}_{10}$

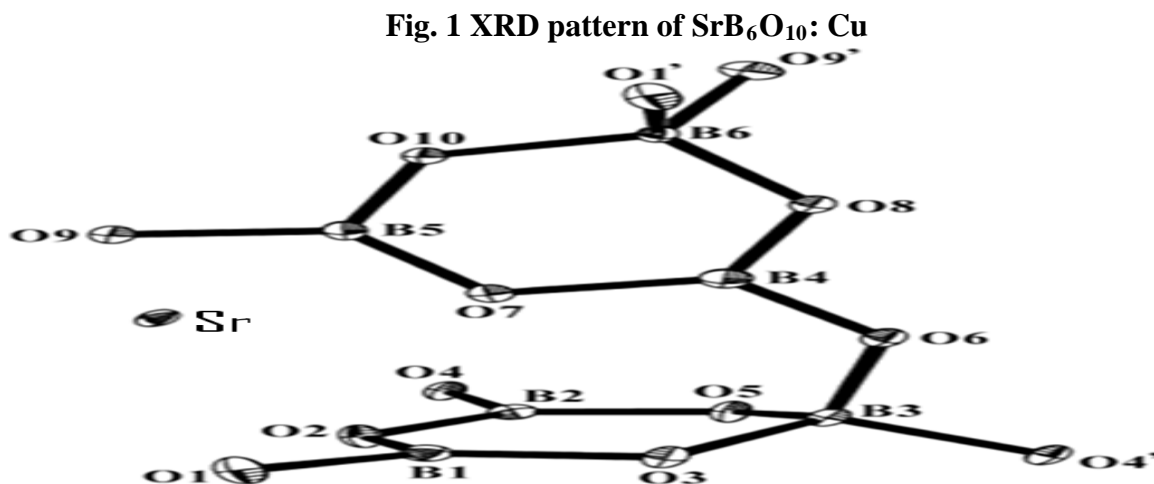


Fig. 2 Crystal Structure of SrB₆O₁₀**3.2 SEM Analysis**

SEM image of this phosphor SrB₆O₁₀:Cu was taken from Synthetic and Art Silk Mills Research Association (SASMIRA), Mumbai (Fig. 3). The material shows irregular spherical as well cylindrical shape particles with canal

like structure. The powder sample shows the sizes of particles ranging from 0.1 μm to 2 μm. This irregularity in masses may be caused due to the irregular mass flow during combustion process.

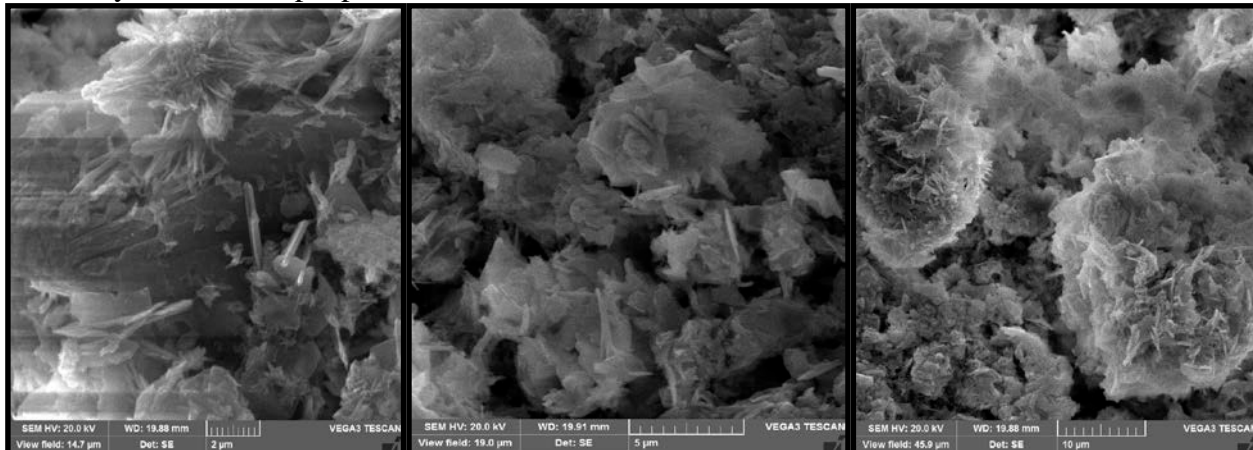
**Fig. 3 SEM image of SrB₆O₁₀****3.3 FTIR Analysis**

Fig. 4 represents the Fourier transformed infrared spectroscopy (FTIR) spectra for SrB₆O₁₀:Cu material. The FTIR revealed prominent absorption with peaks for SrB₆O₁₀:Cu at 1357, 1253, 1112, 1011, 881, 682, 662, 601, 576, 563, 533, 521 and 511 cm⁻¹ as shown in figure. The IR peak at about 1357 is due to presence of orthoborates groups containing BO₃⁻ while asymmetric stretching vibrations of B-O bonds from orthoborates groups identifies by 1253 cm⁻¹. The peak at 1011 cm⁻¹ is for Pentaborate group used for the preparation of SrB₆O₁₀:Cu. 881 cm⁻¹ is representative for

stretching vibrations of tetrahedral BO₄⁻ units. The IR absorption at wave numbers smaller than 500 cm⁻¹ mainly originates from the lattice dynamic modes. No clearly distinguished peak at 630 cm⁻¹ from the OH hydroxyapatite group is visible in spectrum. The absence of peaks in the range of 1550-2500 cm⁻¹ supports the complete removal of residual nitrate and organic matter. (19, 20) The IR spectrum confirms the existence of both trigonally and tetrahedrally coordinated boron atoms, consistent with the results obtained from the crystallographic study.

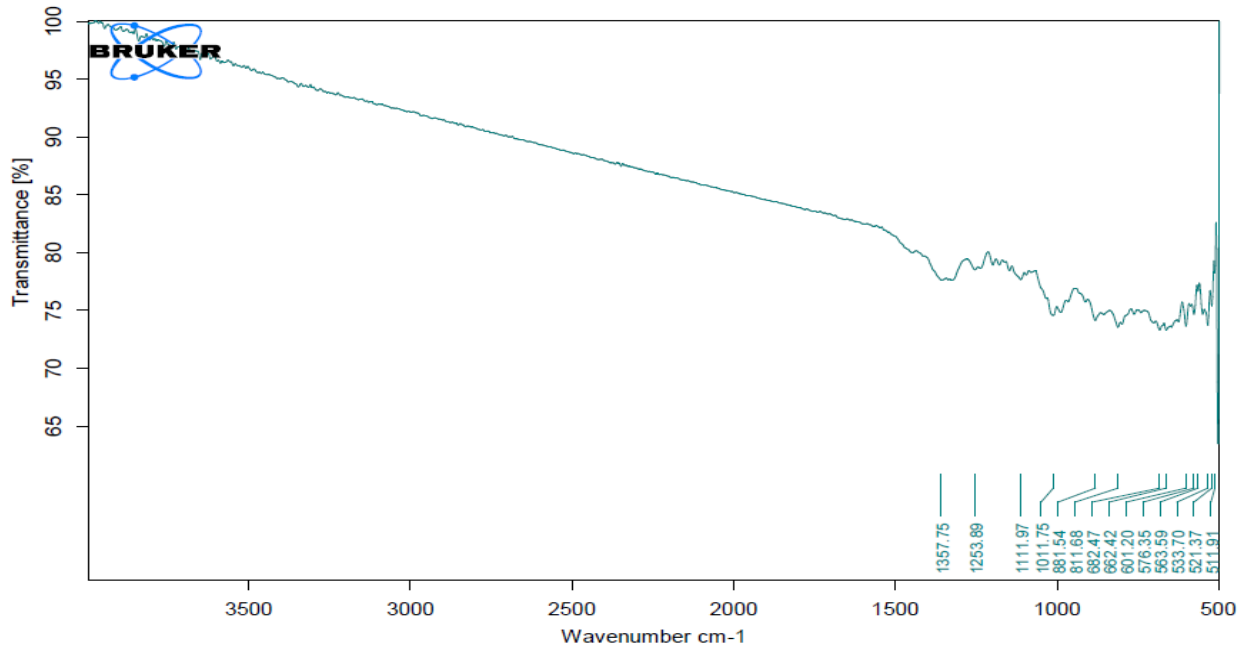


Fig. 4 FTIR analysis of SrB₆O₁₀: Cu phosphor

3.4 Thermoluminescence Studies

The prepared sample SrB₆O₁₀: Cuphosphor exposed to ⁶⁰Co gamma-ray radiation source at RTM Nagpur University with dose rate of 0.3712 kGy / hr. The TL glow curve of newly developed Sr_{0.3}B₆O₁₀: Cu_{0.7} for a test dose of 15 Gy is shown in the Fig.5 obtained at heating rate of 5°C /sec and compared with commercially available phosphor TLD-100 (Harshaw) with same

weight and dose as that of prepared phosphor. It was observed that the phosphor is sensitive to gamma-rays and exhibits TL intensity about 90 % to that of commercially available LiF: Mg, Cu, P (TLD-100 H) phosphor present in our laboratory as shown in Fig. 6. Glow curve for Sr_{0.3}B₆O₁₀: Cu_{0.7} has the main TL peak at about 229°C which is good sign for this phosphor to be used as good TLD material. [21-24]

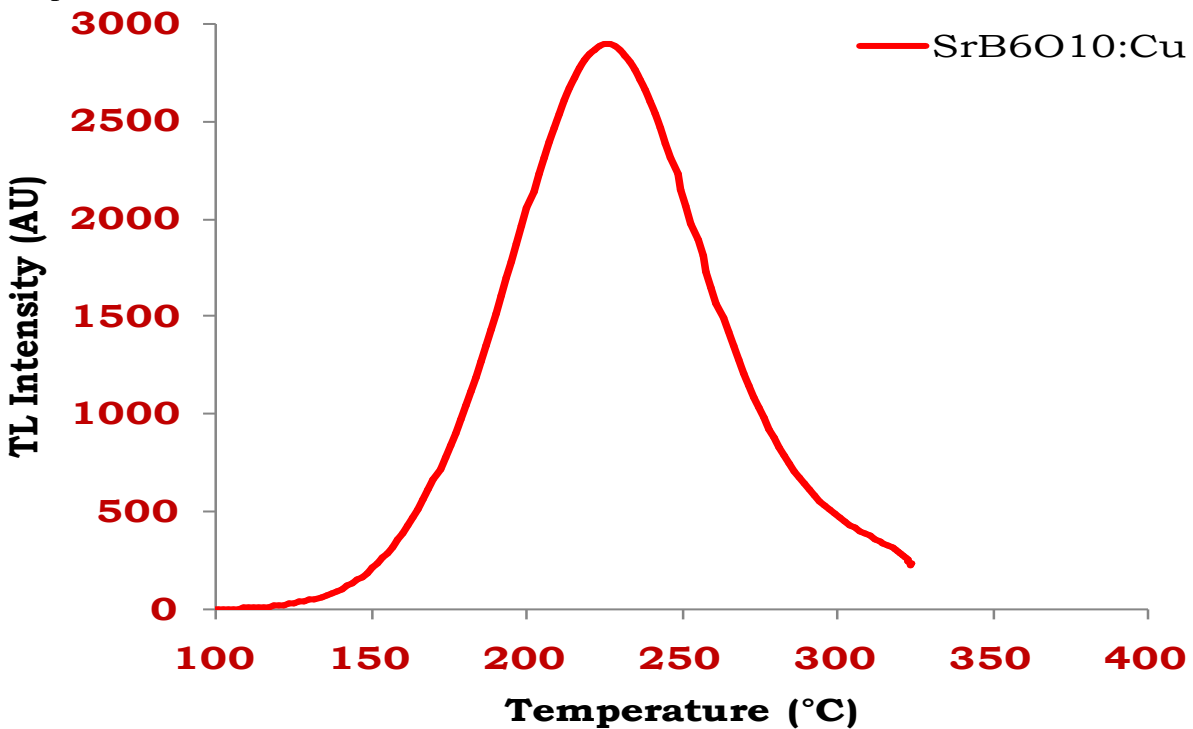


Fig.5 TL glow curve of Sr_{0.3}B₆O₁₀: Cu_{0.7} phosphor

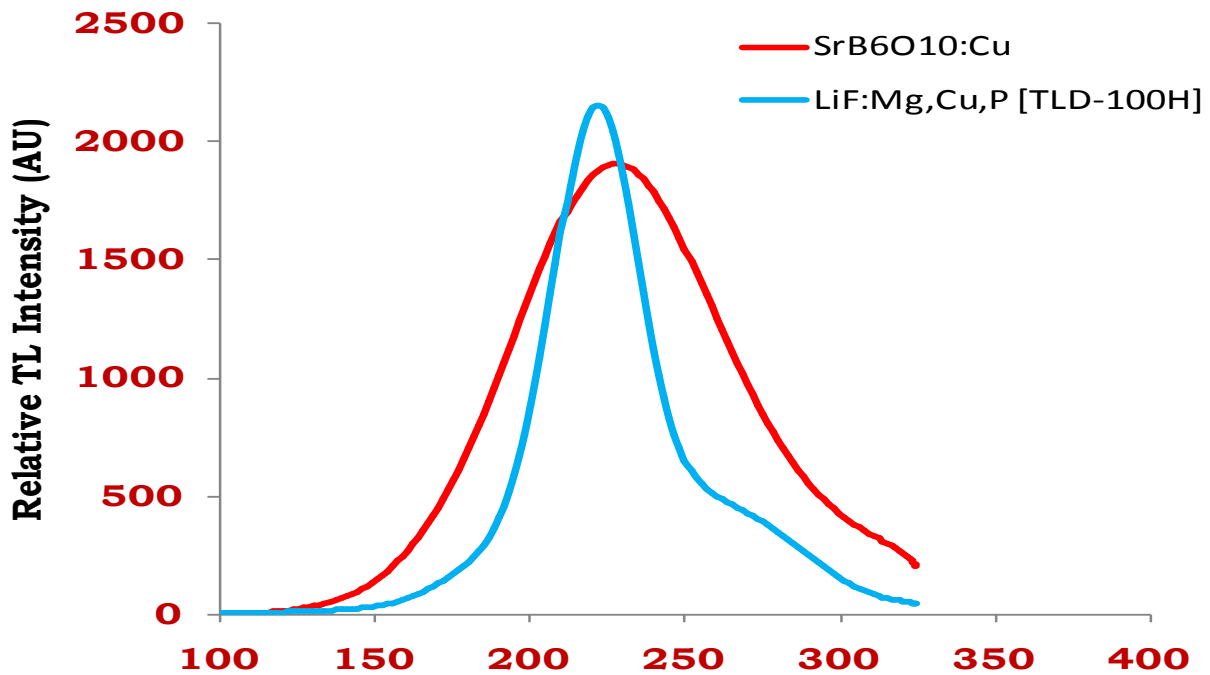


Fig. 6 TL glow curve for SrB₆O₁₀: Cu Compared with LiF: Mg, Cu, P (TLD-100H)

3.5 Dose Response for SrB₆O₁₀: Cu under gamma rays

The thermoluminescence (TL) material is said to be good dosimetric material when its response to absorbed dose is linear over the wide range. Prior to TL characterization, the sample powder was sintered in air at 500 °C in an alumina crucible for 1 hr and suddenly quenched at room temperature on the thick aluminum block. To study the linearity five samples were irradiated simultaneously for each level of dose. Each data point corresponds to the mean of the five readings. Depending upon the minimum availability of dose at the resource centre, 5Gy to 25Gy dose was decided for

irradiations. After about five hours, TL reading was taken with TL readout heating rate of 5 °C/sec on TL 1009I reader designed by Nucleonix system at SGB Amravati University with the temperature range of integration of the TL signal from 40 °C to 400 °C. The linearity was observed for the first peak in the range from 5Gy to 25Gy. The relationship between the TL response of the first high intensity peak and the absorbed dose for SrB₆O₁₀: Cu phosphor was shown in Fig. 7 and it was found to be linear. The fading of 25 Gy dose sample was found to be about 33 % in 25 days as shown in Fig. 8.

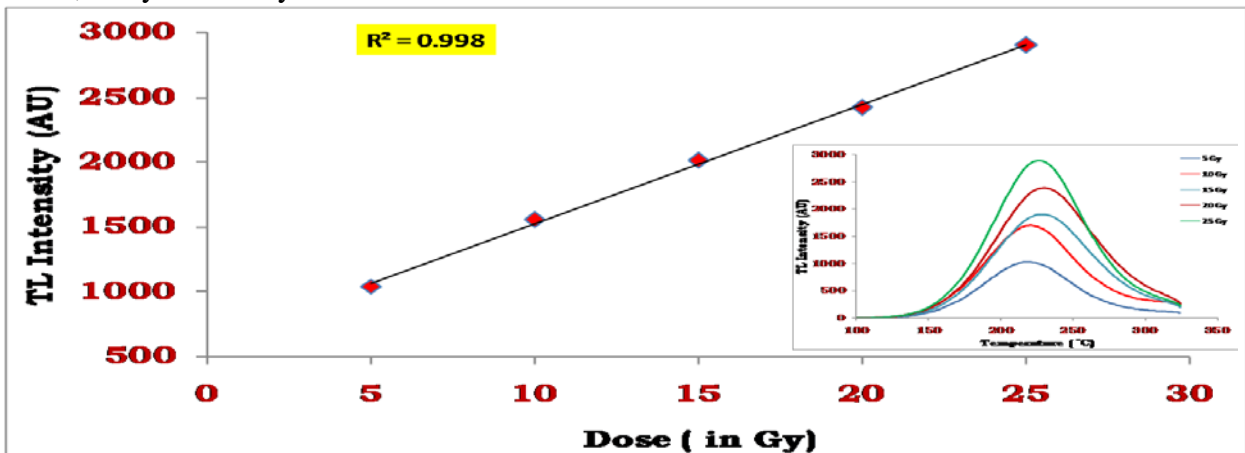


Fig.7 Dose response of SrB₆O₁₀: Cu

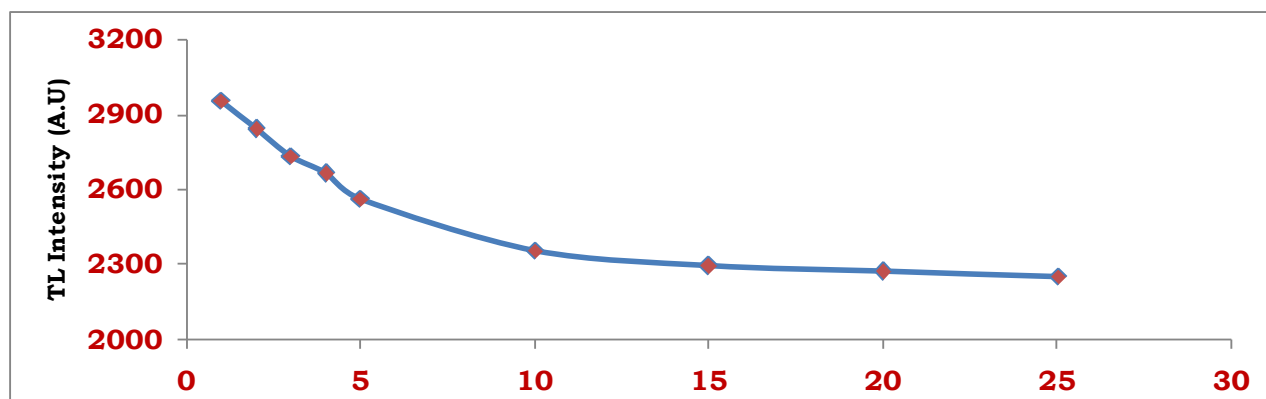


Fig. 8 Fading response of SrB₆O₁₀: Cu phosphor

4. Conclusions

In current report X-ray diffraction result support the complete crystalline formation of SrB₆O₁₀: Cu by combustion synthesis. The thermoluminescence (TL) intensity of SrB₆O₁₀: Cu phosphor was found to be about 90 % of the commercially available LiF: Mg,Cu,P (TLD-100H) phosphor available at our laboratory. The particle sizes in phosphor SrB₆O₁₀: Cu was in scale range from 0.1 μm to 2 μm. The phosphor shows linear dose response from 5 Gy to 25Gy dose. The fading of this sample material is about 33 % in 25 days. From these thermoluminescence studies this phosphor SrB₆O₁₀: Cu is said to be a good candidature for TLD phosphor.

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