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THERMALLY STIMULATED LUMINESCENCE IN CaB_6O_{10}:Cu PHOSPHOR VIA SOLUTION COMBUSTION SYNTHESIS METHOD

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Abstract: The thermoluminescence properties of a novel calcium borate CaB_6O_{10}: Cu inorganic phosphors has been prepared first time by solution combustion synthesis technique, are discussed. The single crystal X-Ray structural analysis showed that CaB_6O_{10} crystallizes in the monoclinic space group *P21/c* with a = 9.799(1) A°, b = 8.705(1) A°, c = 9.067 (1) A°, β = 116.65 (1), Z = 4. The structure of this prepared phosphor is characterized by using X-Ray diffraction. By exposing to gamma-rays its thermoluminescence properties is recorded. Thermally stimulated luminescence (TSL) glow curve of $Ca_{0.2}B_6O_{10}$: $Cu_{0.8}$ phosphor following irradiation with ^{60}Co gamma-ray source shows two peaks at 150 °C and 225 °C respectively for the same heating rate of 5 °C/sec. The phosphor is found to be about 84 % sensitive to that of commercially available LiF: Mg, Cu, P (TLD-100H) phosphor. The effect of dose variation on CaB_6O_{10}: Cu was found to be linear up to 25 Gy dose with fading of about 38 % in 60 days**.**

***Index Terms* - Thermally stimulated luminescence, Solution combustion method, Kinetic parameters.**

I. INTRODUCTION

All The calcium phosphate is well known synthetic biomaterials usually synthesized by precipitation, solid state reaction, sol-gel method and combustion synthesis [1]. $Ca_{10}(PO_4)_6(OH)_2$, β-$Ca_3(PO_4)_2$ and α-$Ca_3(PO_4)_2$ are some of the common bioactive calcium phosphate materials which is chemically similar to the mineral component of bones and hard tissues in mammals. These are classed as bioactive meaning that it will support bone ingrowths and osseointegration when used in orthopaedic, dental and maxillofacial applications [2, 3]. Various publications emphasizes on calcium phosphate materials especially on calcium phosphate cements applicable in therapeutic use [4]. Chen at al [5] studied the crystal structure of newly prepared CaB_6O_{10} by solid state reaction below 750 °C while novel CaB_6O_{10}: with different rare earth are prepared and studied in details for their structure and optical properties recently [6, 7]. Thermoluminescence of copper activated borates such as $Li_2B_40_7$: Cu [8], CaB_40_7: Cu [9] and $K_2B_40_7$: Cu [10] has been extensively investigated which are among the most sensitive known thermally stimulated luminescence phosphors [11] suggested to be used for dosimetry applications [12] In this paper synthesis we are discussed thermally stimulated luminescence in CaB_6O_{10}: Cu via solution combustion method.

II. EXPERIMENTAL

Polycrystalline Ca $_{(1-x)}$ B_6O_{10}: x Cu [x = 0.001, 0.002, 0.005, 0.01 and 0.02] phosphor is prepared by well-established modified solution combustion technique [13, 14]. During the reaction, the stoichiometric amounts of ingredients all precursor calcium nitrate, urea (fuel), $NH_4B_5O_8$ (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 ± 10) ˚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 1.1: Balance reaction for the phosphor

Product	Corresponding reaction with balance molar ratios of precursors
$Ca_{(1-x)}B_6O_{10}$:xCu	$Ca(NO_3)_2$ + **1.2** $NH_4B_5O_8$ + **10.2** $CO(NH_2)_2$ + **10.2** NH_4NO_3 + **x** $CuCl_2$ ⟶ **$Ca_{(1-x)}B_6O_{10}$:xCu** + Gaseous (H_2O, NH_4 and NO_2 etc.) [x=0.001, 0.002, 0.005, 0.01 and 0.02]

III. RESULTS AND DISCUSSION

3.1 XRD ANALYSIS

Fig. 1 shows the powder XRD pattern for polycrystalline sample of CaB_6O_{10}:Cu confirm on Rigaku Miniflex X-ray Diffractometer with scan speed of 2.00 deg / min by Cu K 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 Å, b = 8.542 Å and c = 5.315 Å with z = 4 and volume of unit cell 574.77 $Å^3$ and the values for α = 90˚, β = 90.30˚, γ = 90˚. The formed material has Centro-symmetric structure with space group: P21/n.

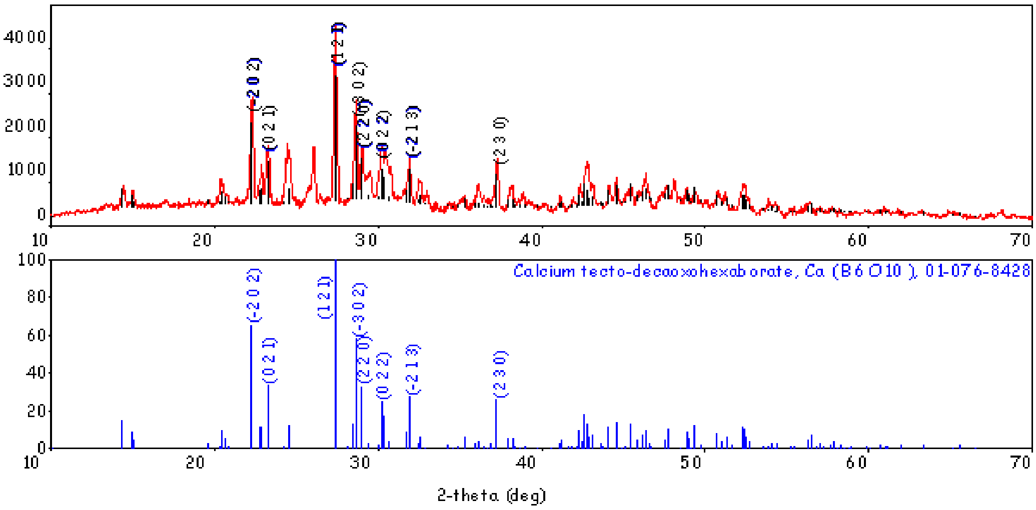

Fig. 1 XRD pattern of CaB_6O_{10}: Cu

3.2 SEM ANALYSIS

SEM image of this phosphor CaB_6O_{10}: Cu was taken from Synthetic and Art Silk Mills Research Association (SASMIRA), Mumbai (Fig. 2). 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.

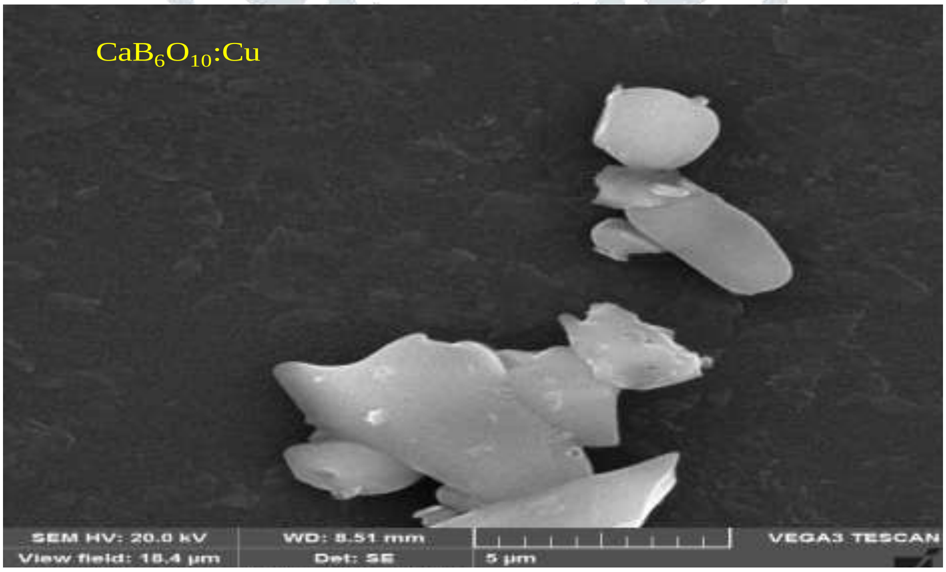

Fig. 2 SEM image of CaB_6O_{10}

3.3 THERMOLUMINESCENCE STUDIES

The prepared sample CaB_6O_{10}: Cu phosphor exposed to ^{60}Co gamma-ray radiation source at RTM Nagpur University with dose rate of 0.3712 kGy / hr. The TL glow curve of newly developed $Ca_{0.3}B_6O_{10}$: $Cu_{0.7}$ for a test dose of 15 Gy is shown in the Fig. 3 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-100H) phosphor present in our laboratory. Glow curve for $Ca_{0.3}B_6O_{10}$:$Cu_{0.7}$ has the main TL peak at about 229 °C which is good symptom for this phosphor to be used as good TLD material. [15, 16]

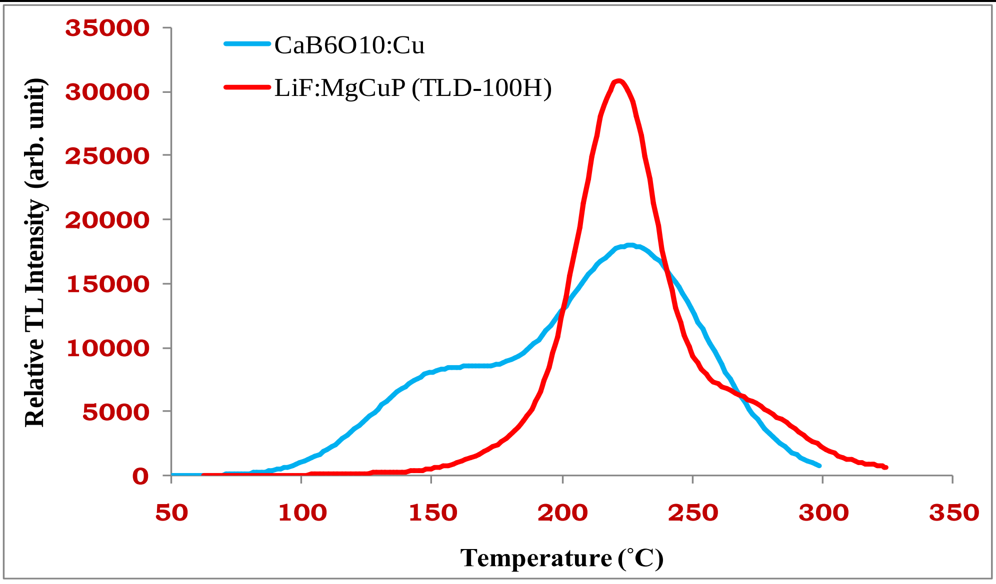

Fig. 3 TL glow curve of CaB_6O_{10}:Cu phosphor and its comparison with LiF: Mg,Cu,P (TLD-100H)

3.4 DOSE RESPONSE FOR CaB_6O_{10}: 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 hour 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, 5 Gy to 25 Gy 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 5 Gy to 25 Gy. The relationship between the TL response of the first high intensity peak and the absorbed dose for CaB_6O_{10}:Cu phosphor was shown in Fig.4 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. 5.

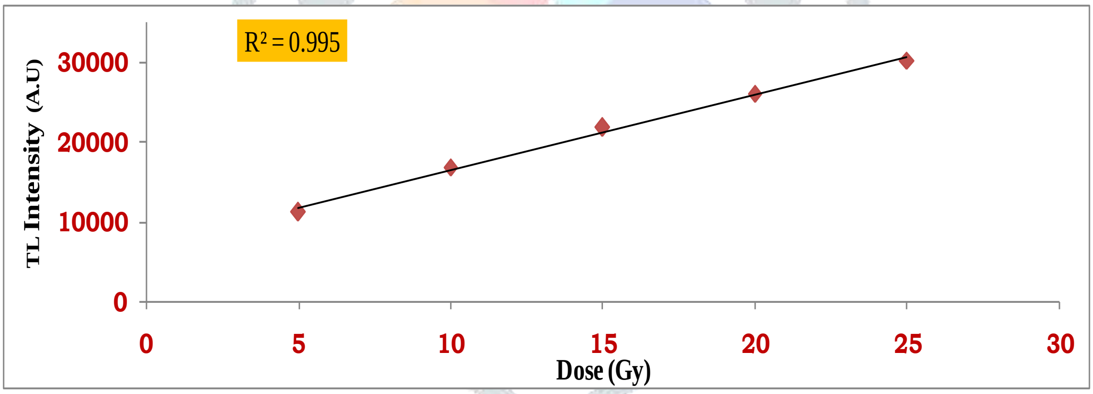

Fig.4 Dose response of CaB6O10: Cu

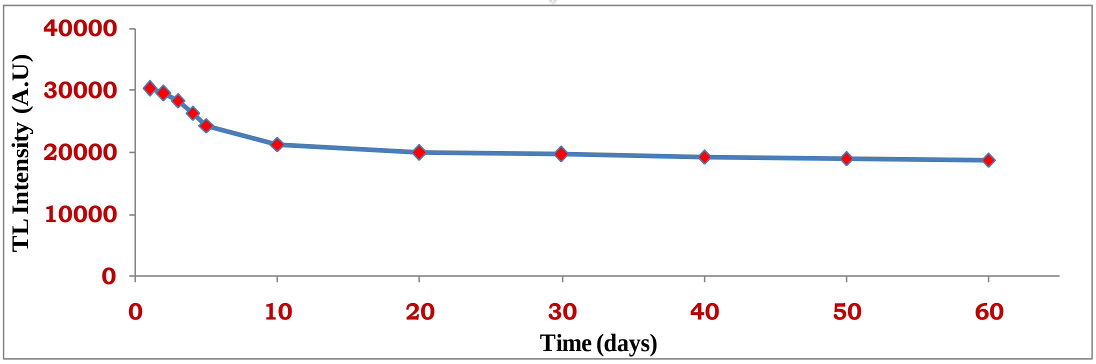

Fig. 5 fading response of CaB6O10: Cu phosphor

CONCLUSIONS

In current report X-ray diffraction result support the complete crystalline formation of CaB_6O_{10}: Cu by combustion synthesis. The thermoluminescence (TL) intensity of CaB_6O_{10}: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 CaB_6O_{10}:Cu was in scale range from 0.1 µm to 2 µm. The phosphor shows linear dose response from 5 Gy to 25 Gy dose. The fading of this sample material is about 38 % in 60 days. From these thermoluminescence studies this phosphor CaB_6O_{10}:Cu is said to be a good candidature for TLD phosphor.

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