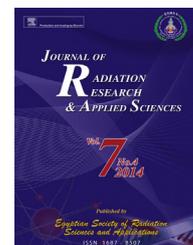


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Dosimetric and kinetic parameters of lithium cadmium borate glasses doped with rare earth ions

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ABSTRACT

Thermoluminescence (TL) characteristics of X-ray irradiated pure and doped with four different rare earth ions (viz., Pr³⁺, Nd³⁺, Sm³⁺ and Eu³⁺) Li₂O–CdO–B₂O₃ glasses have been studied in the temperature range 303–573 K; the pure glass has exhibited single TL peak at 466 K. When this glass is doped with different rare earth ions no additional peaks are observed but the glow peak temperature of the existing glow peak shifted gradually towards higher temperatures with gain in intensity of TL light output. The area under the glow curve is found to be maximum for Eu³⁺ doped glasses. The trap depth parameters associated with the observed TL peaks have been evaluated using Chen's formulae. The possible use of these glasses in radiation dosimetry has been described. The result clearly showed that europium doped cadmium borate glass has a potential to be considered as the thermoluminescence dosimeter.

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1. Introduction

Oxylithiumborate glasses are considered as good materials for dosimetry applications since they are relatively moisture resistant when compared with the pure borate glasses. The understanding of the glass structure by detailed studies on radiation induced defect centres has been an interesting subject of investigation in recent years. Recently some recommendable work has done on thermoluminescence mechanisms in borate based glasses. Hashim et al. (2014) reported the thermoluminescence dosimetry properties and kinetic parameters of lithium potassium borate glass co-

doped with titanium and magnesium oxides. Thermoluminescence study of MnO doped borophosphate glass samples for radiation dosimetry (Swamy et al., 2013) and the influence of copper ions on thermoluminescence characteristics of CaF₂–B₂O₃–P₂O₅ glass system (Swamy et al., 2014) reported by the same author. Thermoluminescence properties of CaO–B₂O₃ glass system doped with GeO₂ reported by Tengku Kamarul Bahri, Wagiran, Hussin, Hossain, and Kadni (2014).

It is well known that boric acid (B₂O₃) is one of the good glass formers and can form glass alone with good transparency, high chemical durability, thermal stability and good rare-earth ion solubility (Zhang, Lu, Sun, Xu, & Ni, 2009). Addition of modifier oxide CdO into these glass matrix, it is

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Table 1 – Various physical properties of $\text{Li}_2\text{O}-\text{MO}-\text{B}_2\text{O}_3:\text{Ln}_2\text{O}_3$ glasses.

Property/glass	CdB	CdBPr	CdBNd	CdBSm	CdB Eu
Refractive index, n_d	1.523	1.538	1.531	1.529	1.524
Density, ρ (g/cm^3)	2.799	3.443	3.329	3.221	3.118
Average molecular weight, \bar{M}	46.028	44.831	44.835	44.839	44.841
Rare earth ion concentration, N_i ($10^{22}/\text{cm}^3$)	–	4.63	4.47	4.33	4.19
Inter-ionic distance of rare earth ions, R_i (\AA)	–	2.79	2.82	2.85	2.88

expected to shorten the time taken for solidification of glasses during the quenching process. CdO mixed glasses are thermally stable, sublime and appreciably covalent in character (Lee, 1996). The glass containing Li_2O as network modifier was seen as bubble free, highly stable and moisture resistant, suitable for a systematic analysis (Rao, Reddy, Nazeer Ahammed, & Parandamaiah, 2000).

Lithium tetraborate glass system is a known and important starting material in the development of applications of radiation dosimetry for a long period, since its effective atomic number $Z_{\text{eff}} \approx 7.25$ has the property of being nearly tissue equivalent that makes it as a very promising material in the field of personal and clinical dosimetry and for other applications like X-ray phosphors, scintillators and thermoluminescent detectors (Chialanza, Castiglioni, & Fornaro, 2012; Kipke & Hofmeister, 2008; Ignatovych, Fasoli, & Kelemen, 2012; Swamy et al., 2013; Szumera & Walawska, 2012). However, pure borate glasses have certain disadvantages to use in radiation dosimetry since they are highly hygroscopic and exhibit weak glow peak at relatively low temperatures.

Schulman, Kirk, and West (1965) were the first to be acknowledged for starting the TL studies on lithium borate compounds and since then various details on TL studies of alkali and alkaline earth tetra borates continued up to present times especially on magnesium and lithium borate compounds. Several attempts were also made to enhance thermoluminescence (TL) sensitivity of these glass materials by adding different transition and rare earth [RE] or lanthanide [Ln] metal ions to these glass samples (Ivascu, Cozar, Daraban, & Damian, 2013; Kutub, Elmanhawaawy, & Babateen, 2007; Mady, Benabdesselam, & Blanc, 2010; Rojas, Yukimitu, & Hernandez, 2008; Sheng et al., 2012; Takam, Bezak, Liu, & Marcu, 2012).

The study on the influence of rare earth ions on thermoluminescence light output of these glasses is also carried out with a view to examine the suitability of these glasses in the radiation dosimetry.

2. Material and methods

Undoped and following rare-earth ion doped glasses in mole% are prepared by using standard melting and quenching techniques and used for the present study (Elliot, 1990; Paul, 1982; Shackl Ford, 1985).

CdB: 30 Li_2O –10 CdO –60 B_2O_3 ,
 CdBPr: 30 Li_2O –10 CdO –59 B_2O_3 :1 Pr_2O_3 ,
 CdBNd: 30 Li_2O –10 CdO –59 B_2O_3 :1 Nd_2O_3 ,
 CdBSm: 30 Li_2O –10 CdO –59 B_2O_3 :1 Sm_2O_3 and
 CdB Eu: 30 Li_2O –10 CdO –59 B_2O_3 :1 Eu_2O_3

Appropriate amounts of raw materials CdO , H_3BO_3 , Li_2CO_3 , Pr_2O_3 , Nd_2O_3 , Sm_2O_3 and Eu_2O_3 were thoroughly mixed and grounded in an agate mortar and melted in a platinum crucible. The chemicals used in the work were of high purity (99.9%). These compositions were heated in a PID temperature controlled furnace at 450 °C for 2 h for the decarbonization from Li_2CO_3 and then the temperature maintained within the range 1000–1050 °C and kept the melt at this temperature for an hour till a bubble free liquid was formed. The crucibles were shaken frequently for the homogeneous mixing of all the constituents. The resultant melt was poured on a rectangular brass mould held at room temperature. The samples were subsequently annealed at glass transition temperature in another furnace to remove mechanical stress and were polished.

The density ' ρ ' of these glasses was determined by the standard principle of Archimedes' using xylene (99.99% pure) as the buoyant liquid. The glass transition temperatures T_g and crystallization temperature T_c of these glasses were determined (to an accuracy of ± 1 °C) by differential scanning calorimetry (DSC) traces, recorded using universal V23C TA differential scanning calorimeter with a programmed heating rate of 15 °C per minute in the temperature range 30–750 °C.

Infrared transmission [IR] spectra for these glasses were recorded using a Perkin Elmer Spectrometer in the wavenumber range 400–4000 cm^{-1} by KBr pellet method. For recording thermoluminescence emission, the glasses were irradiated with X-rays for 1 h with Norelco X-ray Unit operated at 35 kV, 10 mA; thermoluminescence output of these glasses was recorded on a computerized Nucleonix-TL set up with a heating rate of 1 °C/s.

3. Results

From the measured values of density and the average molecular weight \bar{M} , various other physical parameters such as rare earth ion concentration N_i and mean rare-earth ion separation distance R_i are calculated and presented in the Table 1.

Our visual examination, absence of peaks in X-ray diffraction spectra, existence of glass transition temperature T_g and crystallization temperature T_c in differential thermal analysis curves, indicate that the glasses prepared were of amorphous in nature.

Fig. 1 represents the thermograms of pure and $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3:\text{Ln}_2\text{O}_3$ glasses; the pure glass exhibits an endothermic effect due to the glass transition temperature T_g at 537 °C. Presence of single transition temperature T_g indicates homogeneity of the glass. At still higher temperatures an exothermic peak T_c due to the crystal growth followed by an endothermic effect due to the re-melting of the glass symbolized by T_m are

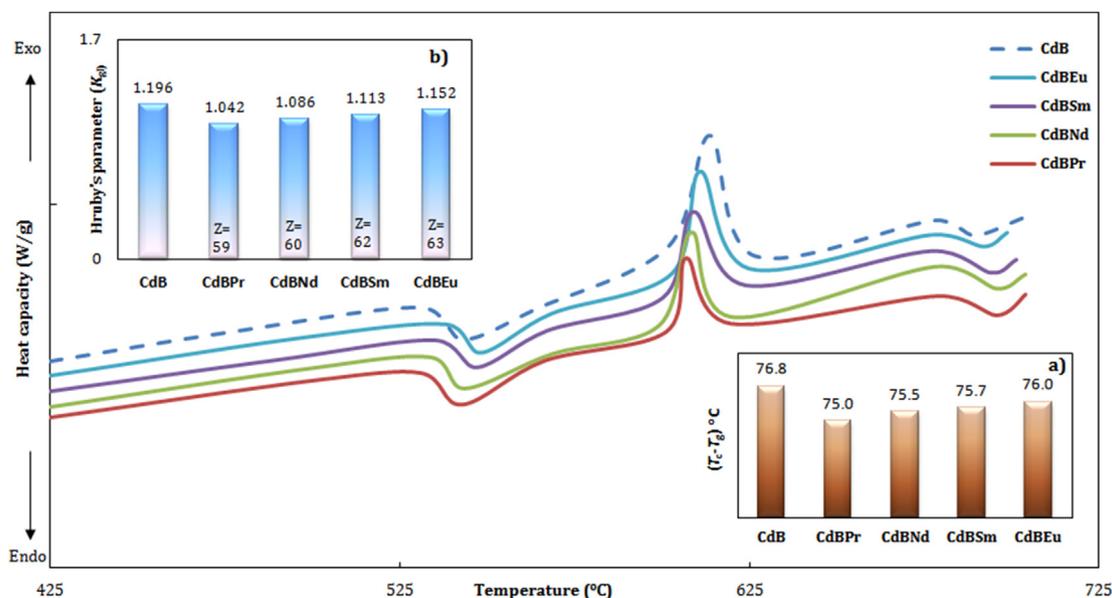


Fig. 1 – DSC patterns of pure and rare earth doped $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glasses. Insets a) the variation of $(T_c - T_g)$ with the atomic number, Z of the rare earth ion, b) the variation of Hruby's parameter.

observed. The glass forming ability (Hruby's) parameter $K_{gl} = (T_c - T_g)/(T_m - T_c)$ is calculated, which gives the information about the stability of the glass against devitrification (Dietzel, 1968; Hruby, 1972) are evaluated and presented in Table 2. Insets Fig. 1 represent the variation of Hruby's parameter for different rare earth ions and the variation of $(T_c - T_g)$ with the atomic number, Z of the rare earth ion.

For the rare earth doped glasses the glass transition temperature T_g is in between 532°C and 535°C . For all glasses with the increase in the atomic number Z of the rare earth ions the values of T_g and $T_c - T_g$ is found to increase gradually.

Fig. 2 represents IR spectra of the pure as well as rare earth doped $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glasses. The infrared transmission spectra of pure $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glasses exhibit three groups of bands: (i) in the region $1300-1340\text{ cm}^{-1}$, (ii) in the region $990-1040\text{ cm}^{-1}$ and (iii) a band at about 710 cm^{-1} . It is well known that the effect of introduction of alkali oxides into B_2O_3 glass is the conversion of sp^2 planar BO_3 units into more stable sp^3 tetrahedral BO_4 units and may also create non-bridging oxygens. Each BO_4 unit is linked to two such other units and one oxygen from each unit with a rare earth ion and the structure leads to the formation of long tetrahedron chains. The second group of bands is attributed to such BO_4 units where as the first group of bands is identified as due to the stretching relaxation of the B–O bond of the trigonal BO_3 units and the band at 710 cm^{-1} is due to the bending vibrations of

B–O–B linkages in the borate network (Ahmed, Abd ElShafi, & ElTohamy, 1998; Khalifa, El Batal, & Azooz, 1998; Qiu, Mori, Sakata, & Hirayama, 1995; Tandon & Hotchandani, 2001).

When the glasses are doped with Ln_2O_3 , the intensity of the second group of bands (band due to the trigonal BO_4 units) is found to increase at the expense of first group of bands (bands due to tetrahedral BO_3 units) with the increase of atomic number of rare earth ions with the shifting of meta-centres of first and second group of bands, respectively towards slightly lower and higher wavenumber for all the glasses. (Inset of Fig. 2 represents the variation of intensities of BO_3 and BO_4 units with atomic number of rare earth ions.) No significant change in position and intensity of the other bands are observed in the spectra of the glass by introducing the rare earth ions. The summary of the data on the positions of various bands in the IR spectra of $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3:\text{Ln}_2\text{O}_3$ glasses are presented in Table 3.

Thermoluminescence glow curves of all the glasses doped with rare earth ions have shown in Fig. 3. Pure $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glass exhibits glow peak at 466 K . When these glasses are doped with different rare earth ions no additional peaks are observed but the glow peak temperature T_m of the existing glow peak shifted gradually towards higher temperatures with a gain in the intensity of TL light output. The glow peaks of rare earth doped glasses CdBPr, CdBNd, CdBSm, and CdBEu shifted to 469 K , 475 K , 482 K and 486 K respectively.

Table 2 – Data on differential scanning calorimetric studies of $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3:\text{Ln}_2\text{O}_3$ glasses.

Glass	T_g ($^\circ\text{C}$)	T_c ($^\circ\text{C}$)	T_m ($^\circ\text{C}$)	T_g/T_m	$(T_c - T_g)$ ($^\circ\text{C}$)	$(T_c - T_g)/T_m$	K_{gl}
CdB	537.0	613.8	678	0.792	76.8	0.113	1.196
CdBPr	532.0	607	679	0.784	75.0	0.110	1.042
CdBNd	533.0	608.5	678	0.786	75.5	0.111	1.086
CdBSm	533.3	609	677	0.788	75.7	0.112	1.113
CdBEu	535.0	611	677	0.790	76.0	0.112	1.152

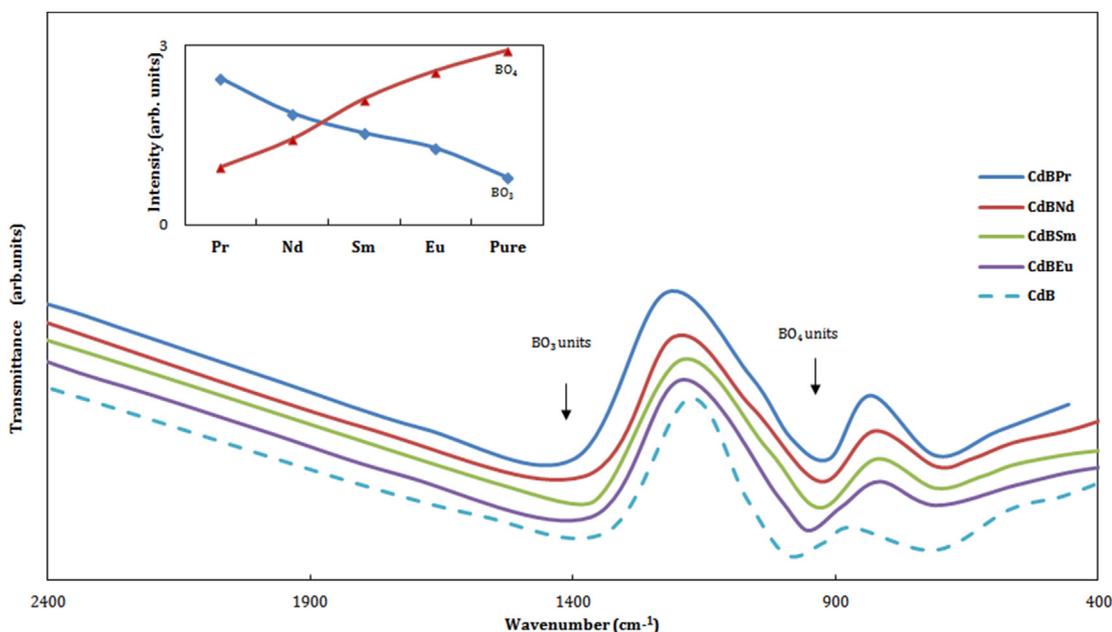


Fig. 2 – Infrared spectra of pure (dotted line) and rare earth doped (solid line) $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glasses. Inset figure represents the variation of intensities of BO_3 and BO_4 units for different glasses.

The relative TL light outputs (area under the glow curve) of pure and rare earth doped $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glasses have shown in Fig. 4a. Pure glass has the TL light output intensity area under the glow curve is 1142. In addition of RE ions to the pure glass network, the TL light output is increased by 4.20% in CdBPr glass, 9.63% in CdBNd glass, 12.61% in CdBSm glass and 18.65% in CdBEu glass and shown in Fig. 4b. The area under the glow curve is also found to be maximum for Eu^{3+} doped glasses.

The trap depth parameters for these glow peaks are computed using Chen's formulae.

The activation energies for these glow peaks are computed using Chen's formulae (Chen, 1969):

$$E_r = 1.52(k_B T_M^2 / \tau) - 1.58(2k_B T_M),$$

$$E_a = 0.976(k_B T_M^2 / \delta), \quad \text{for the first order kinetics.}$$

In the above equation k_B is Boltzmann constant, $\tau = T_M - T_1$, $\delta = T_2 - T_M$, where T_M is the glow peak temperature and T_1 (rising end) and T_2 (falling end) are the temperature at the half widths of the glow peaks. The summary of the

data on thermoluminescence peaks with corresponding trap depth parameters of the present glasses is furnished in Table 4. The trap depth parameters are found to be ~ 0.42 eV and observed to increase gradually with increase in the atomic number of rare earth ions. Such value of trap depth indicates that the lifetime (τ) of electron in these traps is of the order of several months (Gartia, Rey, Tejkumar Singh, & Basanta Singh, 2012; Swamy et al., 2013).

Prior to TL measurements, the optical absorption spectra of all the glasses before and after X-ray irradiation are recorded. After the X-ray irradiation no additional absorption bands are observed other than those obtained in non-irradiated glasses; however the relative intensities of these bands are slightly affected (Anjaiah, Laxmikanth, & Kistaiah, 2011). Fig. 5 shows the optical absorption spectra of Nd^{3+} doped $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glasses before and after X-ray irradiation. A similar behaviour is exhibited by all other glasses.

4. Discussion

The action of X-ray irradiation on glasses is to produce secondary electrons from the sites where they are in a stable state and have an excess energy. Such electrons may traverse in the glass network depending upon their energy and the composition of the glass and are finally be trapped, thus forming colour centres (or alternatively they may form excitons with energy states in the forbidden gap). The trapping sites may be the rare earth ions which constitute the glass structure, ions of admixtures to the main composition and the structural defects due to impurities in the glass. Thus this process leads to the formation of 1) boron electron centres, 2) non-bridging oxygen hole centres and 3) boron oxygen hole centres (Del Nery, Pontusuchka, Isotani, & Rouse, 1994; Pontusuchka,

Table 3 – Peak positions (cm^{-1}) of IR spectra of Ln^{3+} doped $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glasses.

Glass	Band due to B–O bond stretching in BO_3 units	Band due to B–O bond stretching in BO_4 units	Band due to B–O–B linkage in borate network
CdB	1336	992	710
CdBPr	1329	1008	710
CdBNd	1323	1019	710
CdBSm	1316	1027	710
CdBEu	1304	1036	710

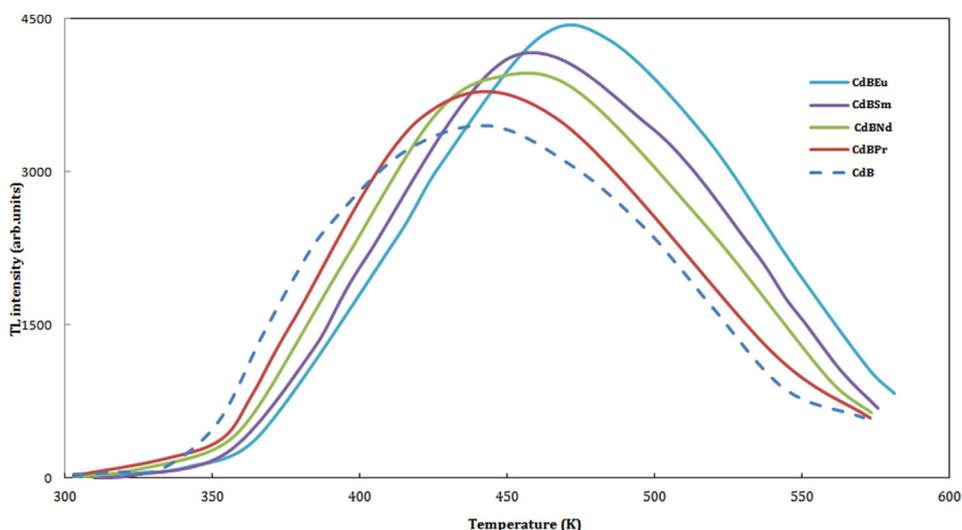


Fig. 3 – Thermoluminescence emission of pure (dotted line) and rare earth doped (solid line) $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glasses.

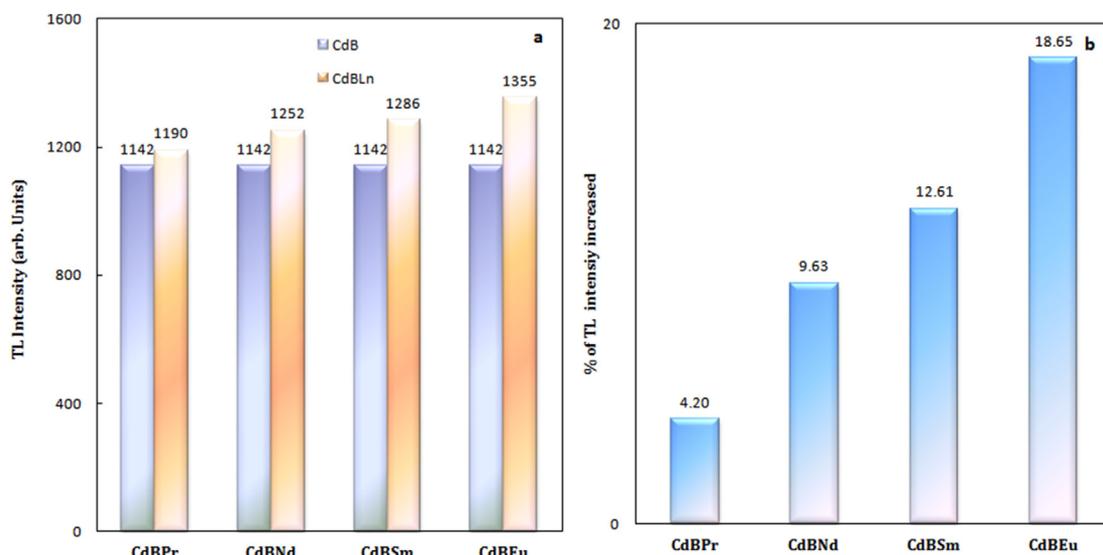


Fig. 4 – The relative TL light output of $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glasses: (a) pure and rare earth doped glasses and (b) percentage of excess TL light output due to rare earth doping.

Isotani, & Piccini, 1987; da Rocha, Pontusuchka, & Blak, 2003). Thermoluminescence is a consequence of radiative recombination between the electrons (released by heating from electron centre) and an anti-bonding molecular orbital of the nearest of the oxygen hole centres. The observed TL peaks in the present glasses can be attributed due to such radiation.

The Li^+ ions have closed structure, do not have energy levels within 10 eV of the ground state and hence these ions do not participate directly in luminescence but may act as activator ions (Van Der Ziel, 1971). Let us assume that the Ln^{3+} ions are uniformly distributed throughout the sample. In the absence of Ln^{3+} ion in the network, each electron released by heating from electron centre would be caught by an anti-bonding molecular orbital of the nearest of the oxygen hole centre. The process is followed by a radiative recombination. The observed TL peak in the present glasses is attributed to

such radiation. If Ln^{3+} ion is present in the glass network, we have observed such a radiative recombination to enhance with respect to that of corresponding pure glass indicating that the rare-earth ions are acting as TL activators in all the glasses. The comparison of TL emission of Ln^{3+} doped glasses

Table 4 – Data on various trap depth parameters of $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3:\text{Ln}_2\text{O}_3$ glasses.

Glass	T_M (K)	τ (K)	δ (K)	μ_g	E_c (eV)	E_δ (eV)	TL light output (rel. units)
CdB	466	76	44	0.367	0.244	0.409	1142
CdBPr	469	74	43	0.368	0.258	0.424	1190
CdBNd	475	72	40	0.357	0.277	0.468	1252
CdBSm	482	72	38	0.345	0.287	0.507	1286
CdBEu	486	68	37	0.352	0.318	0.530	1355

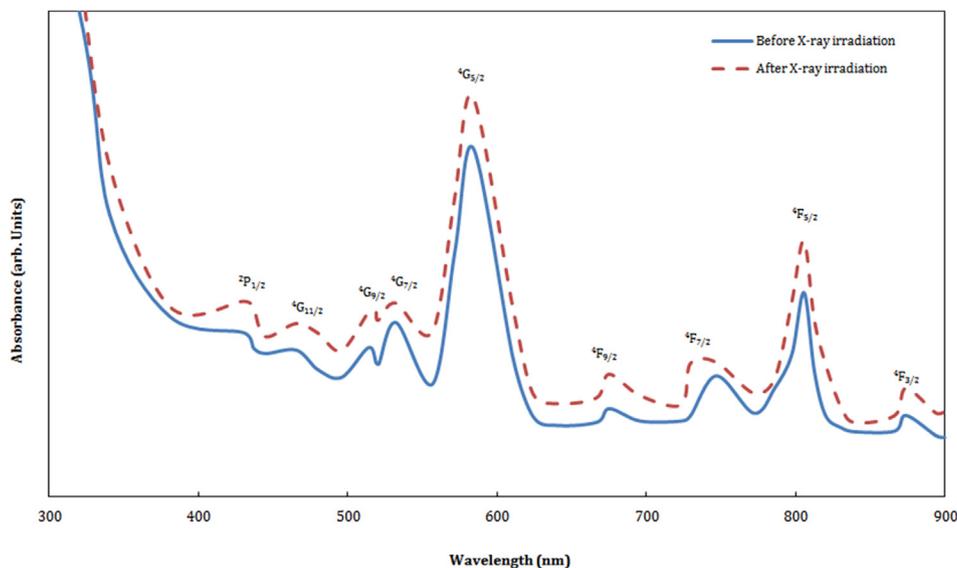


Fig. 5 – Optical absorption spectra of Nd^{3+} ions doped $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glasses before and after X-ray irradiation.

shows a low percentage of enhancements of TL light output for CdBPr glasses (Fig. 4).

The vibrational transitions are less intense in optical absorption process for Eu^{3+} ions in the present series of glasses, which may be more favourable for the formation of high concentration of colour centres at deeper depths in these glasses; the maximum TL output at higher temperatures obtained for the Eu^{3+} ions doped glasses can be attributed due to these reasons. It is well known that the effect of introduction of alkali oxides into B_2O_3 is the conversion of sp^2 planar BO_3 units into more stable sp^3 tetrahedral BO_4 units and may also create non-bridging oxygens. Each BO_4 unit is linked to two such other units and one oxygen from each unit with a metal ion and the structure leads to the formation of long chain tetrahedron. The presence of such BO_4 units in the present glasses is evident from the IR spectral studies.

5. Conclusions

Finally our studies on properties of $\text{Li}_2\text{O}-\text{CdO}-\text{B}_2\text{O}_3$ glasses doped different rare earth ions indicate that i) Differential scanning calorimetric studies indicate high glass forming ability is for CdBEu glass. ii) The IR spectral studies indicate relatively less disorder in CdBEu glass network. iii) The analysis of the TL data suggests that the Eu^{3+} ions doped glasses can be used more effectively in radiation dosimetry since they exhibit high TL light output in high temperature region.

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