Influence of Eu3+ Dopant on Physical and Optical Properties of Lithium-Borosulfophosphate Glasses

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ABSTRACT: Multi-components glass host with enhanced physical and optical features are greatly demanding for various photonics and optoelectronics devices. Selection of suitable glass former, modifier, and dopant ions with optimized composition is a key concern in the fabrication of novel optical glass materials for the aforementioned purpose. Thus, this work reports the convectional melt quench synthesis of europium (Eu^{3+}) doped lithium-borosufophosphate glasses with composition $15Li_2O$-$30B_2O_3$-$15SO_3$-$(40\text{-}x)P_2O_5$-xEu_2O_3 (where $x = 0.1$, 0.3, 0.5, 0.7 and 1.0 in mol%). The effect of P_2O_5 substitution by Eu_2O_3 on their physical (density, molar volume, ion concentration, polaron radius, inter-nuclear distance and field strength) and optical properties was examined. The amorphous nature of the samples was confirmed by XRD diffraction pattern. The density of glass samples was slightly increased with increasing concentration of Eu_2O_3. Such trend is attributed to the higher molecular weight of Eu_2O_3 (351.926 g/mol) than P_2O_5 (141.9445 g/mol). The room temperature absorption spectra revealed four weak bands in the UV-Vis range and two strong bands in the NIR range with 1.0 mol% glass sample possessing the highest intensity at $^7F_0 \rightarrow {}^7F_6$ (2095 nm). Incorporation of Eu^{3+} ion significantly enhanced the glass absorbance and the physical properties. The results on high refractive index (∼2.3), polarizability and non-linear physical features suggest that these glasses are potential for photonics and solid-state laser applications.

KEYWORDS: Borosulfophosphate glasses, Europium ions, physical properties, optical properties and photoluminescence.

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I. INTRODUCTION

The increasing demand for new, inexpensive and highly efficient optical glass host for optical device applications such as solid-state lasers, optical amplifiers, light emitting devices, display devices, sensors among others have capture the attention of many researchers in the search of novel glass host with simple synthesis procedure, excellent homogeneity and wide flexibility of chemical composition that can be customize to meet the aforesaid need.

The research interest on phosphate (P2O5) as the main network former in this study is due to its low melting points, high thermal expansion coefficient, low glass transition temperatures and softening temperature, and hence can be used as sealing having a high thermal expansion coefficient (Qian et al., 2013). However, these glasses have a relatively poor chemical durability that often limits their usability. Several authors attest that the chemical durability of phosphate glasses can be improve by addition of various oxides either network former, modifier or intermediate (Wong et al., 2014, Marimuthu, 2016).

The inclusion of borate which is another good glass former into phosphate glass network has been reported to increase the chemical durability of the phosphate glasses by contributing boron atoms into the system (Vijayakumar et al., 2015, Tonchev et al., 2015). This unified glass system called borophosphate glass matrix derived from the amalgamation of borate and phosphate exhibit enhanced and tailored physical and optical properties compare to the pure borate and phosphate glass system (Maheshvaran and Marimuthu, 2012, Gomes et al., 2017). In addition, the drawbacks such as high phonon energy and hydroscopic nature of borophosphate host matrix can be overcome by introducing modifier reagent into the host network to improve the intensity, disrupt the lattice, open the network structure, weakened the bond strength, and lowered the viscosity of host matrix (Pang et al., 2014). In this work, lithium is added as modifier to decrease the phonon energy, hygroscopic properties and improve the strength of the host (Wong et al., 2014).

In recent time, few researchers introduced sulfate as intermediate into borophosphate forming "Borosulfophosphate glasses" to further enhance the strength of the host matrix (Kumar et al., 2012a, Kumar et al., 2012b, Ravi Kumar, 2013). These materials revealed superior features than borophosphate glasses such as low strain birefringence, low melting point, low optical loss, low non-linear refractive index coefficient and superior refractory nature (Selvi et al., 2015). Despite the applied interest on these glass hosts, the information on physical and optical properties of borosulfophosphate glasses activated with rare earth ions is not fully understood.

In general, any glass hosts doped with rare earth ions has been a key to the development of several optical devices such

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as visible and infrared solid-state lasers, Light-emitting diodes (LEDs), optical amplifiers, sensors to mention but a few owning to their unique features such as high refraction with relatively low dispersion, high visible emission efficiency, inhomogeneous line width, stable oxidation state and several excited states for optical pumping (Janek et al., 2016). These unparallel features and the prominent applications of rare earth doped glass materials piloted our research interest in the study of their physical and optical characteristics.

Among different rare earth ions, trivalent europium (Eu3+) ion has been identified as the best dopant for inorganic lattice as well as pure red light-emitting center for display devices due to its dominant 5D0→7F2 electronic transition (Keshavamurthy and Eraiah, 2015a). The selection of Eu3+ ion has proved to deserve for the study of disordered materials, because of its relatively simple energy level structure (Venkateswarlu and Rudramadevi, 2015, Boonin et al., 2016). The good correlation in physical and optical properties of Eu3+-doped glasses dictates the number of readily available new glass materials for various optical applications (Swapna, 2015, Kaewkhao et al., 2015). Due to technological importance of Europium (Eu3+) ion and the advantages of above research, this study examines the influence of different concentration of Eu3+ ions on the physical and optical properties of lithium-borosulfophosphate glasses prepared by melt quenching method.

II. MATERIALS AND METHODS

A. Sample Preparation

Lithium- borosulfophosphate glasses doped with varying concentration of europium ions (Eu3+) defined by the chemical composition 15Li2O-30B2O3-15SO3-(40-x)P2O5-xEu2O3 (where x= 0.1, 0.3, 0.5, 0.7 and 1.0 in mol%) were prepared using convectional melt quenching procedure. The starting raw materials lithium carbonate (Li2CO3), boric acid (H3BO3), sulphuric acid (H2SO4) and phosphoric acid (H3PO4) and europium oxide (Eu2O3) of high purity (99.9%) in batches (20g) were measured via precision weighing balance and mixed thoroughly in a porcelain crucible, and then placed in an electrical furnace following the procedure reported in literature (Kumar et al., 2012a).

The chemical compositions of the samples are summarized in Table 1.

Table 1: Nominal composition of Li2O –B2O3–SO3–P2O5 glasses doped with different concentration of Eu2O3 (in mol %).

S/no	Sample code	Li_2O	B_2O_3	SO_3	P_2O_5	Eu_2O_3
1	0.1Eu	15	30	15	39.9	0.1
2	0.3Eu	15	30	15	39.7	0.3
3	0.5Eu	15	30	15	39.5	0.5
4	0.7Eu	15	30	15	39.3	0.7
5	1.0Eu	15	30	15	39.0	1.0

B. Glass Sample Characterizations

X-ray diffractograms of the samples were recorded at room temperature via Siemens X-Ray Diffractometer D5000 with Cukα (λ=1.54Å) having the power source of 40kV and 30mA, and 2θ was varied from 10° to 80° at scanning rate of 2° per minute to ascertain the amorphous nature of the synthesized samples.

Density measurements were made by using standard Archimedes method with toluene as immersing fluid (Hua et al., 2013, Wong et al., 2014, Reddy et al., 2016). The optical absorption spectra for all Eu3+-doped samples were obtained at room temperature using Shimadzu 3101 UV-Vis-NIR spectrophotometer in the range of 200 – 2400 nm.

C. Fundamental Formulae for Physical and Optical Parameters

The basic knowledge on physical and optical parameters of newly synthesized glass material foretells its prominent physical and optical response prospective for various optical applications. In this context, the essential physical and optical parameters employed in this research are given in Tables 2 and 3 respectively.

Table 2: Physical parameters of $15Li_2O$-$30B_2O_3$-$15SO_3$-$(40-x)P_2O_5$ glasses activated with varying concentration of Eu_2O_3 ions.

Eq. No	Parameter	Formula	Description
	Physical parameters		
1)	Density (ρ)	$\rho = \frac{a}{a-b} \times \rho_t$	$a \rightarrow$ weight of glass sample in air $b \rightarrow$ weight of sample in toluene $\rho_t \rightarrow$ density of toluene
2)	Molar volume (V_m)	$V_m = \frac{M}{\rho}$	$M \rightarrow$ molecular weight of the glass sample
3)	Ion Concentration (N_i)	$N_i = \frac{x\rho N}{M_a}$	$x \rightarrow$ mole fraction of dopant $N \rightarrow$ Avogadro's number $M_a \rightarrow$ Average molecular weight of sample
4)	Polaron radius (r_p)	$r_p(\text{Å}) = \frac{1}{2}(\frac{\pi}{6N_i})^{1/3}$	$N_{i\rightarrow}$ Ion concentration
5)	Inter-nuclear distance (r_i)	$r_i(\text{Å}) = (\frac{1}{N_i})^{1/3}$	$N_{i\rightarrow}$ Ion concentration
6)	Field strength	$\mathrm{F} = \frac{Z}{r_p^2}$	$Z \rightarrow$Atomic number of the dopant

Table 3: Optical parameters of $15Li_2O$-$30B_2O_3$-$15SO_3$-(40-x) P2O5 glasses activated with varying concentration of Eu_2O_3 ions.

Eq. No	Parameter	Formula	Description
	optical parameters		
1)	Tau's Relation	$(\alpha h\nu)^{\frac{1}{r}} = A(h\nu - E_g)$	$\alpha \rightarrow$ Absorption coefficient $h\nu \rightarrow$ photon energy A → band tailing parameter $r = 1/2$ → Direct transition $r = 2$ →Indirect transition
2)	Urbach Relation	$\ln\alpha = (\frac{h\nu}{E_{Urb}}) - c$	$E_{Urb} = 1/slope$:Urbach energy c → Constant
3)	Refractive index (n)	$\frac{n^2 - 1}{n^2 + 2} = 1 - \sqrt{\frac{E_g}{20}}$	$E_g \rightarrow$ optical band gap
4)	Dielectric constant (ε)	$\varepsilon = n^2$	$n \rightarrow$ Refractive index
5)	Optical dielectric constant (ϵ)	$\epsilon = n^2 - 1$	$n \rightarrow$ Refractive index
6)	Reflection loss (R)	$R = (\frac{n-1}{n+1})^2$	$n \rightarrow$ Refractive index
7)	Molar refractivity(R_M)	$R_M = V_m(\frac{n^2-1}{n^2+2})$	$V_m \rightarrow$ Molar volume
8)	Molar polarizability(α_M)	$\alpha_M = \frac{3}{4\pi N_i} R_M$	$N_i \rightarrow$ Ion Concentration

III. RESULTS AND DISCUSSION

A. XRD Analysis

The X-ray diffraction patterns of the studied glasses depicted in Figure 1 show no discrete or distinguish sharp crystalline peaks but the pattern reveal a broad hump, confirming that all our synthesized samples are fully amorphous (glassy) in nature rather than crystalline.

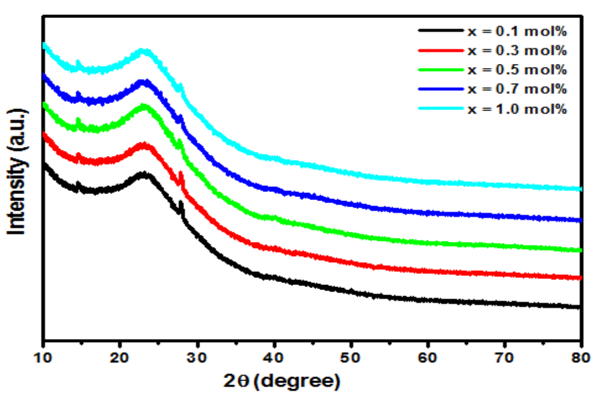

Figure 1: X-Ray Diffraction pattern of $15Li_2O$-$30B_2O_3$-$15SO_3$-(40-x) P_2O_5 glasses activated with varying concentration of Eu_2O_3 ions.

B. Physical Properties Analysis

The composition dependency on density and other vital physical parameters is significant for exploring the changes in the structure of glasses. These parameters are highly influenced by structural compactness, change in geometrical configuration, coordination number, cross-link density and dimension of interstitial spaces of the glass.

Based on the measured glass density (ρ), other physical parameters such as molar volume (V_m), Ion concentration (N_i), Polaron radius (r_p), inter-nuclear distance (r_i), field strength (F) were computed using the standard formulae described earlier in Table 1 and are presented in Table 4.

The dependences of density and molar volume of $15Li_2O$-$30B_2O_3$-$15SO_3$-(40-x)P_2O_5 glasses on Eu^{3+} content shown in Figure 2 exhibit non-linear behaviour which is in agreement with other studies on Eu^{3+} -doped glasses (Razak et al., 2016, Boda et al., 2016) and (Wagh et al., 2015). The account for the increase in density at the expense of europium content is due to the successive replacement of phosphate atom having less atomic weight by the europium atom with higher atomic. In addition, the inclusion of Eu_2O_3 to host network causes some type of structural rearrangement of the atoms. Doubtlessly Eu^{3+} will enter the host network by breaking up the phosphate double bond and consequently lead to the formation of more bridging oxygen (BO). This explained the modifying role of Eu^{3+} in the glass structure by transforming PO_3 to PO_4. Conversely, the decrease in molar volume implies a decrease in bond length and inter-atomic distance between the atoms. Therefore, the compactness of the glass increased and more bridging oxygen (BO) are generated which in turn increase the rigidity of the glass.

Figure 3 on the other hand, demonstrates variation of polaron radius and field strength of $15Li_2O$-$30B_2O_3$-$15SO_3$-(40-x)P_2O_5 glasses on Eu^{3+} content. It was observed that the polaron radius decreases proportionally with increase in Eu_2O_3 content. This decrease may be due to the increase in Eu^{3+} ion concentrations (N_i) which give rise to high field strength

Table 4: Physical properties of $15Li_2O$-$30B_2O_3$-$15SO_3$-$(40-x)P_2O_5$ glasses doped with different concentration of Eu_2O_3 ions.

Glass sample	*x* = 0.1	*x* = 0.3	*x* = 0.5	*x* = 0.7	*x* = 1.0
Physical parameters					
M_a (g)	101.934	102.042	102.150	102.258	102.420
ρ (gcm^{-3})	1.786	1.996	2.117	2.119	2.210
v_m (cm^3mol^{-1})	59.080	51.126	48.241	48.205	46.354
Eu^{3+} $N_i 10^{21}$ ionscm^{-2}	1.055	3.534	6.242	8.730	12.991
r_p(Å) $\times 10^{-8}$	3.965	2.650	2.193	1.961	1.717
r_i(Å) $\times 10^{-8}$	9.838	6.576	4.540	4.464	4.261
$F \times 10^{16} cm^{-2}$	4.006	8.969	13.105	16.389	21.137

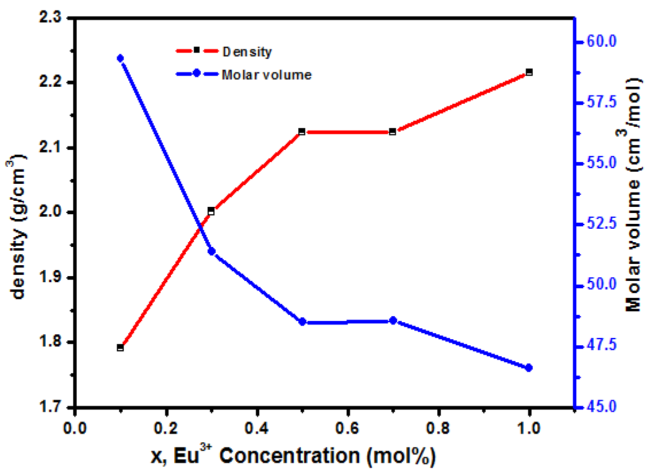

Figure 2: Density and molar volume relation of $15Li_2O$-$30B_2O_3$-$15SO_3$-$(40-x)P_2O_5$ glasses doped with varying concentration of Eu_2O_3 ions.

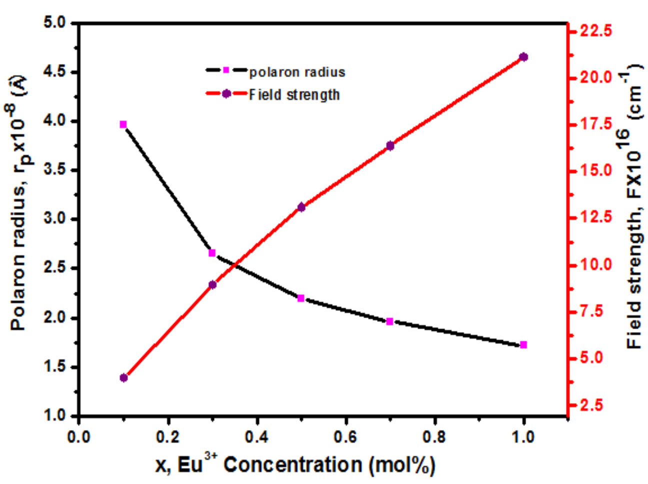

Figure 3: Variation of polaron radius and field strength of $15Li_2O$-$30B_2O_3$-$15SO_3$-$(40-x)P_2O_5$ glasses activated with varying concentration of Eu_2O_3 ions

C. Optical Properties Analysis

The absorption spectra of lithium-borosulfophosphate glasses activated with varying concentration of Eu^{3+} ions in the UV-Vis regions (350 - 650 nm) and NIR regions (2000 - 2400 nm) are shown in Figure 4 and 5 respectively. The observed absorption spectra in this work are in consistent with other report on Eu^{3+} doped glass system (Manasa and Jayasankar, 2016, Boonin et al., 2016). The absence of well-defined sharp absorption edges as shown in Figure 4 further reinforce XRD studies, confirming that the current samples are fully amorphous in nature. The observed UV absorption edges (cut-off wavelengths) are red shifted in the range of 369 – 392 nm with increasing Eu^{3+} content. This slight shift to higher wavelength may be attributed to the lower rigidity of the glass from higher Eu^{3+} content.

The absorption spectra revealed total of six bands in all regions which are separated into two regions. Figure 4 exhibits four bands due to $^7F_0 \rightarrow ^5L_6$ (391 nm), $^7F_0 \rightarrow ^5D_2$ (463 nm), $^7F_0 \rightarrow ^5D_1$ (541 nm) and $^7F_0 \rightarrow ^5D_0$ (580 nm) in the UV-Vis regions whereas NIR regions (Figure 5) shows two intense bands due to $^7F_0 \rightarrow ^7F_6$ (2095 nm) and $^7F_1 \rightarrow ^7F_6$ (2207 nm) transitions. It is observed that1.0 mol% of Eu^{3+} content at $^7F_0 \rightarrow ^7F_6$ (2095 nm) is found to be more intense than all other transitions. Therefore, 1.0 mol% of Eu^{3+} doped lithium-borosulfophosphate glass may be used as potential optical glass host for optical device applications.

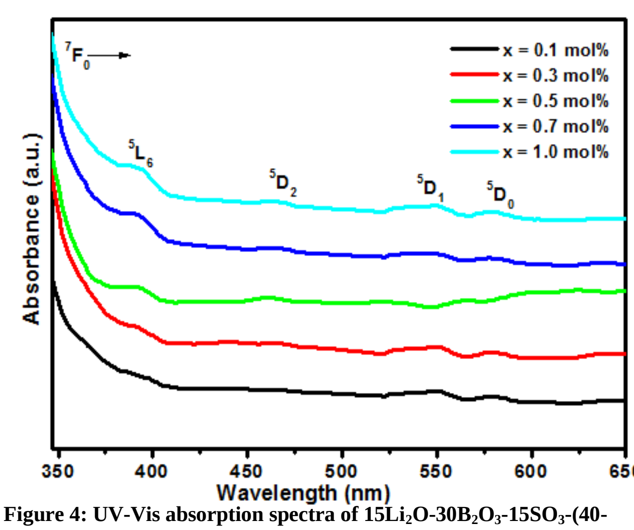

Figure 4: UV-Vis absorption spectra of $15Li_2O$-$30B_2O_3$-$15SO_3$-$(40-x)P_2O_5$- xEu_2O_3 glasses

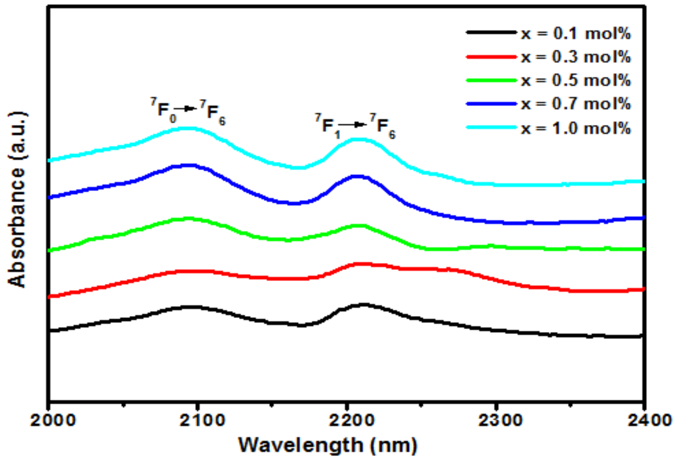

Figure 5: NIR absorption spectra of $15Li_2O$-$30B_2O_3$-$15SO_3$-$(40-x)P_2O_5$-xEu_2O_3 glasses.

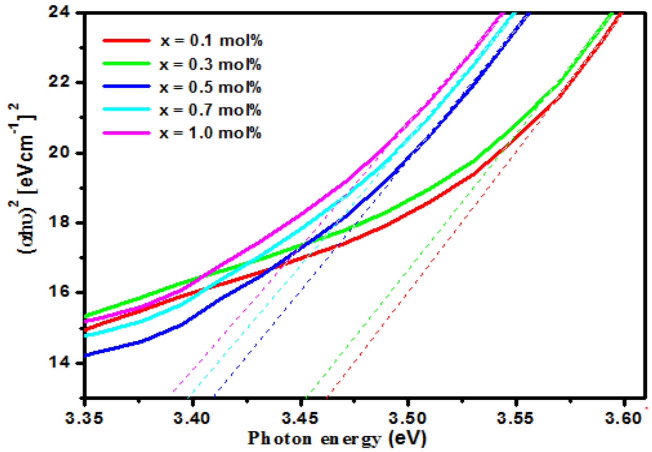

Figure 6: Direct optical energy band gap of $15Li_2O$-$30B_2O_3$-$15SO_3$-$(40-x)P_2O_5$- xEu_2O_3 glasses.

D. Optical Band Gap Analysis

The energy band gap is an important feature of glassy materials which determines their optical applications. From the obtained absorption spectra and Tau's relation mentioned earlier in equation (9) of Table 3, the plots of $(\alpha h\nu)^2$ and $(\alpha h\nu)^{1/2}$ versus photon energy at $(\alpha h\nu)^2 = 0$ and $(\alpha h\nu)^{1/2} = 0$ are presented in Figure 6 and Figure 7 respectively. The values of direct and indirect optical band gap are estimated by extrapolating a linear part of the aforesaid Figures to x-axis. The meeting points of the straight line on x- axis (photon energy) indicate the respective values of energy band gap and are tabulated in Table 5. As noticed from Table 5, the values of both direct and indirect optical band gap decrease as the concentration of Eu^{3+} increases from 0.1 to 1.0 mol%. This decrease in optical band gap arises from the red-shift in absorption edge resulting to the decrease in non-bridging oxygen (NBO) and hence making the glass structure more compact.

Table 5: Optical properties of $15Li_2O$-$30B_2O_3$-$15SO_3$-$(40-x)P_2O_5$ glass samples doped with varying concentration of Eu_2O_3 ions.

Glass sample	***x* = 0.1**	***x* = 0.3**	***x* = 0.5**	***x* = 0.7**	***x* = 1.0**
Optical parameters					
Cut-off wavelength (nm)	369	377	384	388	392
Direct optical band gap (eV)	3.499	3.489	3.459	3.439	3.436
Indirect optical band gap (eV)	3.468	3.449	3.398	3.380	3.351
Urbach's energy (eV)	0.289	0.308	0.313	0.323	0.333
refractive index n	2.275	2.279	2.283	2.287	2.290
Dielectric constant	5.176	5.194	5.212	5.230	5.244
Optical dielectric constant	4.176	4.194	4.212	4.230	4.244
Reflection loss	0.151	0.152	0.153	0.153	0.154
Molar refractivity (cm^{-3})	44.381	29.806	28.174	28.250	27.157
Molar polarizability10^{-23}(ions cm^{-1})	1.363	1.182	1.117	1.110	1.077

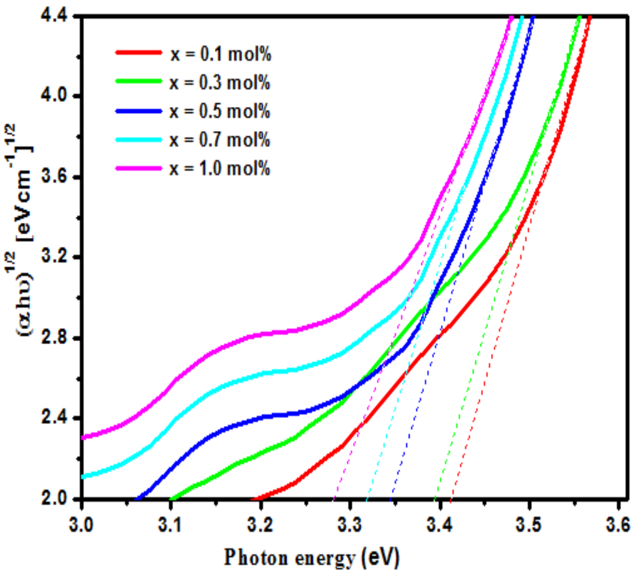

Figure 7: Indirect optical energy band gap of 15Li_2O-30B_2O_3-15SO_3-(40-x)P_2O_5- xEu_2O_3 glasses.

E. Urbach's Energy Analysis

In this work, the logarithm of absorption coefficient ($\ln\alpha$) was plotted as a function of $h\nu$. As can been seen from Figure 8, the Urbach's law is clearly verified via the linear Urbach plots of all the studied glass samples. The values of Urbach's energy given in Table 5 were evaluated by determining the slopes of the linear portion in the lower photon energy region of the curves and taking their reciprocals. The observed Urbach's energy in the range of 0.289 sto 0.333 eV indicates the increase in structural disorder of the glasses. This results is in agreement with the value obtained for amorphous semiconductor (0.046 – 0.66 eV) (Keshavamurthy and Eraiah, 2015b).

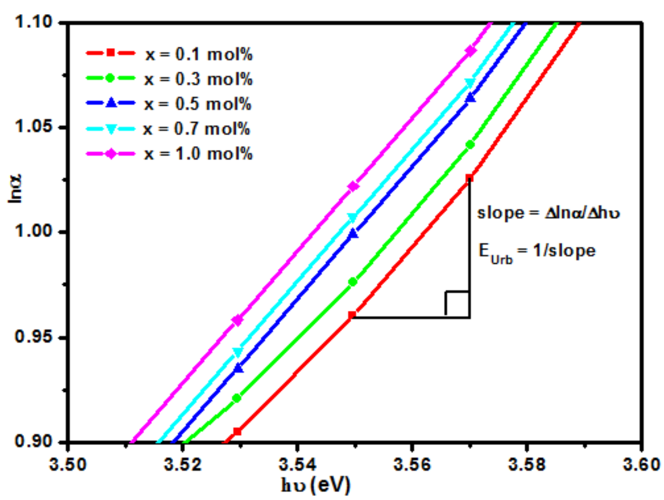

Figure 8: Urbach's energy of 15Li_2O-30B_2O_3-15SO_3-(40-x) P_2O_5- xEu_2O_3 glasses.

IV. CONCLUSION

A new class of transparent and good optical europium doped lithium- borosulfophosphate glasses were successfully synthesized by melt quenching. X-ray diffraction studies confirm the amorphous nature of the glasses. Based on the physical parameters, the increase in density and decrease in molar volume with increase in dopant concentration explained the modifying role of Eu_2O_3 in lithium-borosulfophosphate host structure. The optical absorption was slightly influenced by the variation of Eu_2O_3 concentration. Successive replacement of P_2O_5 by Eu_2O_3 leads to red shift in the fundamental absorption edge of the absorption spectra and consequently, decreases the optical band gap. 1.0 mol% Eu3+ doped lithium- borosulfophosphate glass revealed prominent intensity at $^7F_0 \rightarrow {}^7F_6$ (2095 nm). In this view, 1.0 mol% contained glass sample obtained from this study can be used as potential optical glass host for solid state lasers and light emitting devices.

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