THE ROLE OF LEAD OXIDE IN PbO-B$_2$O$_3$ GLASSES FOR SOLID STATE IONIC DEVICES

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Abstract. Physical and optical properties of xPbO-(100-x) B$_2$O$_3$ (x=20-50 mol %) lead-borate glasses were investigated. The density of these glass samples increases from 4.267 to 5.960 g/cm^3 whereas the molar volume decreases initially and then increases continuously as the content of PbO increases. PbO's dual role (modifier and glass former) in the glassy matrix is responsible for the non-uniform variation in density and the uneven pattern in molar volume. The UV-visible spectra also revealed a non-uniform variation in optical band gap with increasing PbO content, confirming that PbO enters the glass network as a modifier at low concentrations and as a glass former at high concentrations. Other parameters, such as average boron-boron separation and oxygen packing density (OPD) decrease progressively as PbO content increases, while the refractive index, the molar volume of oxygen (V$_0$) and packing density (V$_t$) increase linearly. Because of the presence of lead, the ionic behaviour of the prepared glasses increases while covalency decreases, indicating that these can be used to make solid state ionic devices.

Keywords: Borate Glasses; Optical Properties; Density; Molar Volume

Citation: Singh G.Pal, Singh J., Kaur P., Singh T., Kaur R., Singh D.P. The Role of Lead Oxide in PbO-B$_2$O$_3$ Glasses for Solid State Ionic Devices // Materials Physics and Mechanics. 2021, V. 47. N. 6. P. 951-961. DOI: 10.18149/MPM.4762021_13.

1. Introduction

The importance of borate glasses as optical materials is attributed to their low melting point, high transparency, and high thermal stability [1]. These glasses are commonly used as insulating and dielectric materials [2]. Due to the high value of polarizability of Pb^{2+} ions in glass matrices, lead borate glasses have increased optical non-linearity [3]. They are appealing materials for luminescent applications [4-5], switches [6], and radiation shielding materials [7] ,etc.

Certain non-conventional network formers like lead oxide (PbO) after addition in glasses impart many extra-ordinary properties, for instance, large density, high refractive index, high non-linear optical susceptibility, and exceptional infrared transmission [8-10]. The

beauty of a glass system including a heavy metal oxide, such as PbO, originates not only from its different unique properties, such as low melting temperatures and wide glass formation regions but also from the fact that it shows binary behaviour when added to the glass[8-10]. It can be utilized as a network former and also as a glass modifier [11]. It is also worth noting that lead, in the form of Pb^{2+} ions, enters the glassy system as a modifier, favouring the formation of tetrahedral BO_4 units with bridging oxygens at two tetrahedral per oxygen atom rate. Nuclear magnetic resonance (NMR) investigations [12] demonstrated that as a glass former, lead penetrates the glass preferentially by covalent bonding. The quantity of PbO which has been added to the glass structure is sufficient enough to determine its dual purpose in the present case.

The objective of the current research work is to investigate the role of PbO in the altering borate network under the compositional constraint. Thereby, the structural and optical properties of these glasses have been studied. This paper also aims to learn more about the bond ionicity in the glass structure and check its suitability for ionic/photonic devices.

2. Experimental details

Preparation of Glasses. The conventional melt-quenching technique is used for the preparation of glass samples of the type x PbO–(100-x) B_2O_3 where x is the mole fraction of lead oxide (20-50%). Chemical data for all the above-mentioned glasses are shown in Table 1.

An electronic balance was used to take the weight measurements. After that, the chemicals were stirred for half an hour in a pestle mortar. Then this batch is transferred to a silica crucible. The silica crucible containing the batch was then placed in an electric furnace capable of reaching a temperature of 1400°C and was kept at a temperature above 1000°C for half an hour under normal atmospheric conditions. The melt was then poured into a preheated steel mould and annealed at a temperature of about 340°C for one hour to secure against the breaking of the sample by residual internal strains. The annealing furnace was then switched off and samples were cooled very slowly at room temperature. The prepared glass samples were polished with cerium oxide to obtain flatness.

The remaining experimental techniques are the same as present in the previous study [13].

Table 1. Nominal composition (mole fraction), density, molar volume, boron–boron separation $<d_{B-B}>$, optical band gap, refractive index, Packing Density, V_0 and Bond Density of the glass samples

Glass	PbO (%)	B_2O_3 (%)	Density (D) (g/cm^3)	Molar Volume (V_m) (cm^3/mol)	$<d_{B-B}>$ (nm)	Optical Band Gap [eV]	Refractive Index	Packing Density (V_t)	V_0	Bond Density $(n_{b \times 10}^{29} \ m^{-3})$
Pb-20	20	80	4.267	22.05	0.451	2.90	2.424	0.151	8.48	1.0925
Pb-30	30	70	5.185	21.26	0.389	2.75	2.468	0.226	8.86	1.1331
Pb-40	40	60	5.618	22.49	0.360	2.59	2.517	0.278	10.22	1.0709
Pb-50	50	50	5.960	23.91	0.341	2.25	2.635	0.323	11.96	1.0075

3. Results and discussion

X-ray diffraction pattern of PbO-B$_2$O$_3$ glass. Figure 1 shows the X-ray diffraction pattern of PbO-B$_2$O$_3$ glass samples. The absence of sharp peaks in the X-ray diffraction pattern of samples Pb-20, Pb-30, Pb-40, and Pb-50 reveals that there is no crystalline phase in the manufactured glass samples, indicating and confirming their amorphous nature. The unavailability of a long-range atomic arrangement is a clear indicator of the samples' glassy nature [14].
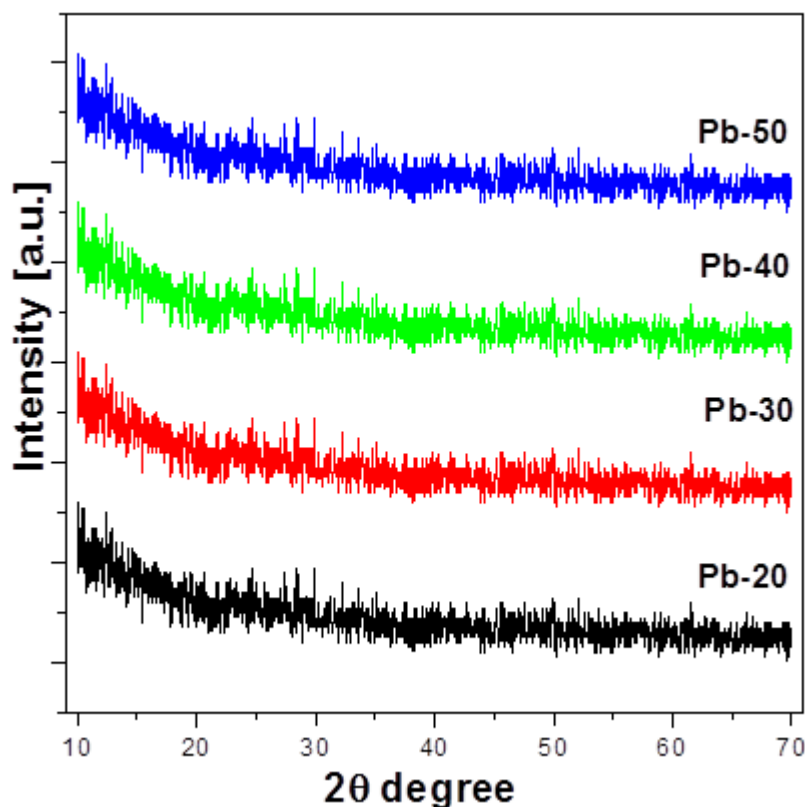
Fig. 1. X-ray diffraction pattern of PbO-B$_2$O$_3$glasses for Pb-20, Pb-30, Pb-40 and Pb-50 samples

Density and molar volume. The structural softness or compactness, change in geometrical configuration, cross-link density, coordination number and the dimension of the interstitial space of the glasses influence the density [15]. As a result, any variation in density with glass system composition is related to the glass structure [15].

The density of the glass samples at room temperature was obtained from standard Archimedes' principle by using benzene as buoyant. It is determined by using the following relation [13]:

$$\rho = \left[\frac{W_a}{W_a - W_b} \right] \times \rho_b,$$

where W_a is the glass sample weight in the air, W_b is the glass sample weight in buoyant and ρ_b is the density of buoyant benzene which is 0.865 g/cm^3 in the present case. According to the findings of this investigation, the density of glass samples increases as the lead oxide level increases. This could be attributed to the existence of lead oxide because of the higher molecular weight of PbO than that of borate. But this increase in density is not uniform. Initially, in sample Pb-20 and Pb-30, density shows a rapid increase from 4.267 to

5.185 g/cm^3 with an increase in lead oxide content from 20 to 30 mol% and then with further addition of PbO, the increase in density slows down from 5.185 to 5.960 g/cm^3 with an increase in lead oxide content from 30 to 50 mol% (as shown in Table 1). The reason for this non-uniform increase in density could be due to the formation of [BO$_4$] groups from [BO$_3$] groups which are responsible for the compaction of glass networks because [BO$_4$] groups are significantly denser than [BO$_3$] groups [16-17]; and back conversion of [BO$_3$] from [BO$_4$] groups with increasing PbO concentration, which is responsible for the expansion of glass networks. Another reason for this could be that PbO acts as a modifier when present in low concentrations, but as the concentration of PbO increases, it begins to act as a glass former causing initial compaction and then continuous expansion in the glass networks. This could also be due to creation of non-bridging oxygen (NBOs). NBOs break the bonds of the borate host glass, increasing free space in the network and thus decreasing the rate of increase in density with increasing concentration of lead oxide in glass networks [41].

Molar volume studies show that as PbO content increases, there is an initial increase followed by a progressive decrease in molar volume. This could be because at low PbO concentrations, the conversion to four-fold coordinated boron occurs, converting the boroxyl ring to pentaborate and diborate units. The back transition of four-fold to three-fold coordinated boron occurs at high lead content, and the network now consists primarily of orthoborate, pyroborate, and metaborate groups [14-16]. A reduction in molar volume results in a decrease in bond length, which causes compaction of glass networks, whereas an increment in molar volume results in an increase in bond length, which induces glass network expansion [14-16]. This non-uniform change may be associated with the formation of non-bridging oxygen (NBOs) in the glass network.

Average boron-boron separation <d$_{B-B}$>. Molar volume results are confirmed by calculating the average boron-boron separation $<d_{B-B}>$. It is calculated by using the relation:

$$<d_{B-B}> = [\frac{V_m}{2(1-X_b)N_A}]^{1/3},$$

whereV_m is molar volume, X_b is molar fraction of B$_2$O$_3$ and N_A is Avogadro's number.

Calculating the average boron-boron separation <d$_{B-B}$>[16] confirms the molar volume and density results. Value of <d$_{B-B}$>decreases as PbO content increases, but this decrease is not uniform (Table 1). The existence of Pb(2+) ions aids in the lowering of the average boron-boron separation. This non-uniform change confirms PbO's role in glass networks both as a modifier and a former. In low concentrations, the inclusion of lead oxide causes compaction and in high concentrations, it causes the expansion of glass networks which validates the results of density and molar volume.

Oxygen packing density(OPD) and molar volume of oxygen(V$_0$).OPD is calculated by using density and molar mass values in the following relation [13],

$$OPD = 1000 \times C \times (\frac{\rho}{M}).$$

The molar volume of the glasses has also been used to calculate oxygen molar volume (V_0) by using the relation,

$$V_0 = \frac{V_m}{\sum_i x_i n_i},$$

whereV_m is molar volume and n_i is the fraction of oxygen atoms present in each constituent element of prepared glass samples. The results obtained show that V$_0$ increases whereas oxygen packing density OPD decrease with the corresponding change in density and molar volume (Table 1). This increase in V$_0$ and decrease in OPD may be due to the creation of non-bridging oxygens which make structure loosely packed and leads to change in the physical and chemical properties of glasses.

Packing density (V$_t$) and bond density (n$_b$).

*Bond density (n$_b$).*It can be calculated by the following relation,

$$n_b = \frac{N_A}{V_m} \sum_i n_c x_i,$$

where N_A is the Avogadro number, n_c is the coordination number ofcation and x_i is a mole fraction of different oxides [13].

The number of bonds per unit volume i.e. bond density, is affected by the increment of lead oxide content in glass samples [16-17]. The results obtained show that firstly, the value of bond density increases from 1.093 to 1.133 m^{-3} with the increment in PbOcontent from 20-30 mol% (Table 1) and secondly, with further addition of PbO from 30-50 mol%, it starts decreasing from 1.133 to 1.007 m^{-3}. It reveals clearly that lead plays the role of network modifier when present in lower concentration by creating more B-O bond vibrations of [BO$_4$] group. But when the lead oxide is present in higher concentrations, it starts behaving like a network former.

Packing density (V$_t$). The molar volume has been used to calculate the packing density (V$_t$) of glass systems [13].

$$V_t = \sum_i \frac{V_i x_i}{V_m}.$$

Packing density is the measure of the tightness and hardness of glass systems [16-17]. Results obtained show a nonlinear increase in packing density which is in agreement with density and molar volume results.

UV-VIS absorption spectra. The UV absorption spectra of all glass samples were recorded at room temperature by using UV-Visible Spectrophotometer (Shimadzu, Japan) in the range 200-800 nm as shown in Fig. 2. The band edge continuously shifts towards the longer wavelength as the PbO concentration increases (408-489 nm). This may be because of more tetrahedral [BO$_4$] groups and [PbO$_4$ or PbO$_6$] groups forming in the glasses. With a rise in lead oxide content, a significant number of oxygen ions are becoming available in the glass networks, causing the network to shift from trigonal [BO$_3$] to tetrahedral [BO$_4$] units, resulting in a glass network compaction [10,19-20]. These advancements contribute to the shift of the band edge to a higher wavelength.
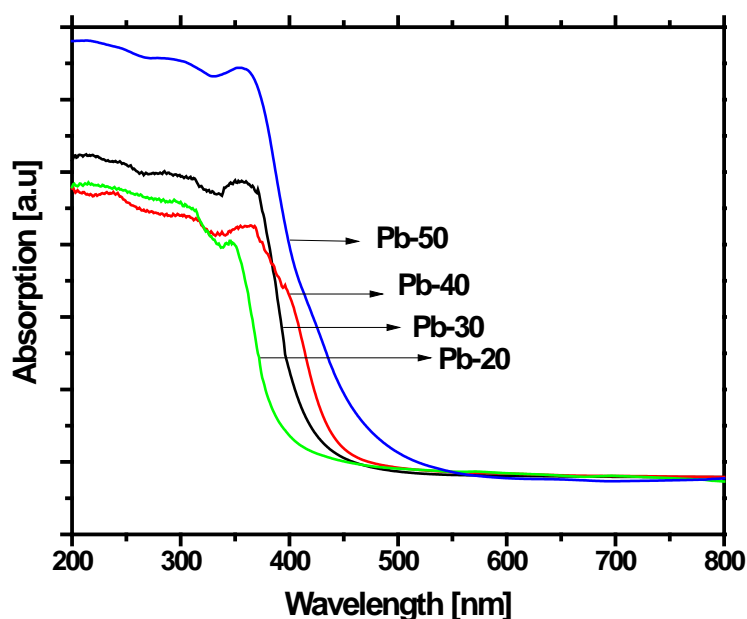
Fig. 2. Optical absorption of PbO-B$_2$O$_3$ glasses for Pb-20, Pb-30, Pb-40 and Pb-50 samples

The value of the optical band is calculated from Urbach plots between $(\alpha h v)^{1/2}$ and energy (hv) of PbO-B_2O_3 glasses [19]; where α, h, and v are absorption coefficient, Planck's constant, and frequency respectively. The Urbach plots and optical band gap of glass samples are shown in Fig.3 and Table 1respectively. The variation of the optical band gap is studied as a function of the glass composition. The sensitivity of the method is visible in the different values of the optical band gap with different concentrations of PbO.The results show that when PbO is available in small concentrations, the optical band gap value decreases because the existence of a large number of oxygen ions in the glass network transforms the network unit from trigonal $[BO_3]$ to tetrahedral $[BO_4]$, resulting in a reduction in bandgap (Table 1). Lead oxide, at lower concentrations in sample Pb-20 and Pb-30, also functions as a network modifier in the form of $[PbO_6]$ or $[PbO_3]$ units. As the content of lead oxide increases in glass samples Pb-40 and Pb-50, the bandgap value increases, resulting in the formation of NBOs (non-bridging oxygens). As the concentration of lead rises, some of the NBOs form tetrahedral $[PbO_4]$ units causing lead oxide to begin acting as a network former. It indicates that PbO enters the glass network as a modifier in lower concentration and as glass former in higher concentration [10,13-14,19].
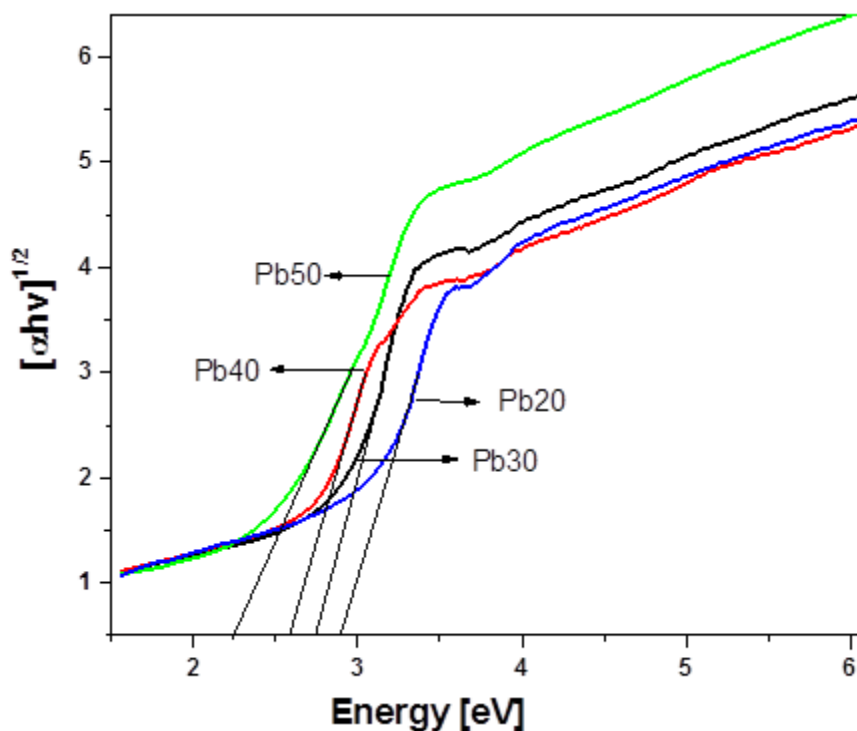
Fig. 3. The optical band gap of PbO-B_2O_3 glasses for Pb-20, Pb-30, Pb-40, and Pb-50 samples

Refractive index of PbO-B_2O_3 Glasses. The refractive index of glasses can be calculated by the following relation [13]:

$$\left(\frac{n^2-1}{n^2+2}\right) = 1 - \sqrt{\frac{E_g}{20}},$$

where$n$ and E_g are the refractive index and optical bandgap respectively.

Refractive index values show a continuous increase (2.425-2.635)with the increase in PbO content in glass samples and show a direct relationship with density. Glass samples with high density values have a high refractive index. This may be due to the activation of more ionic dipoles when placed in an electric field [15,21-22]. This explains the increase in value of the refractive index from 2.425 to 2.635 with an increase in PbO content from 20-50 mol%

(Table 1). The formation of non-bridging oxygen forms more ionic bonds.NBOs are more polarizable than BOs, so the increase in the refractive index of glasses can be attributed to the compositional changes which lead to the formation of NBOsand the increase in refractive index [21-23].

Refractive index v/s optical bandgap. The optical Band Gap of glasses strongly depends upon their Refractive Index. The plot of Band gap vs refractive index shows that band gap decreases whereas the refractive index increases continuously with the increase in PbO content in the glass samples. This leads to the shift of valence band, accordingly resulting in the shift of band gap. The variation of refractive index with band gap is shown in Fig. 4.
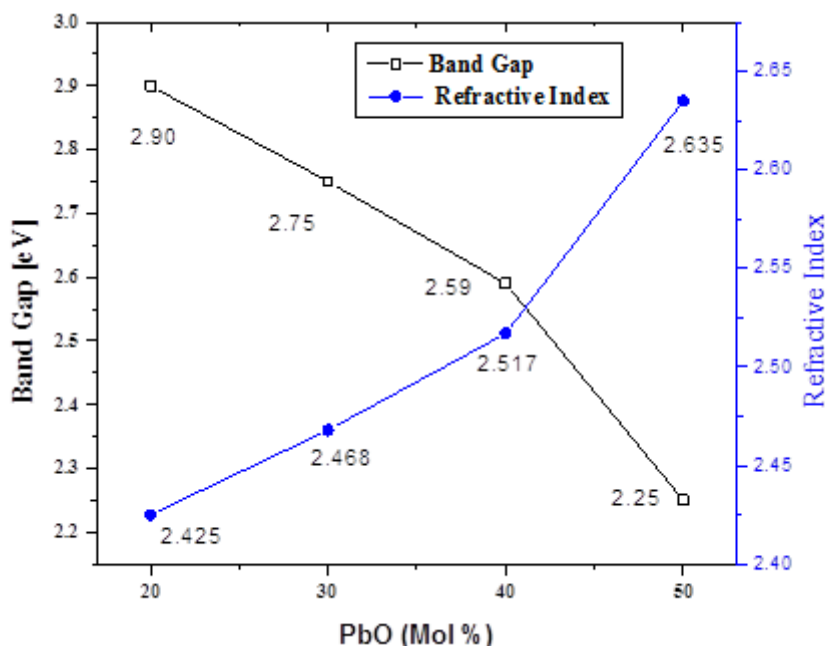

Fig. 4. Density vs Refractive Index Variation

Molar refraction and Molar polarizability. Molar refraction is the sum of contributions of cationic refraction and oxygen ionic refraction. As shown in Table 2, the values of molar refraction firstly increase upto 40% of PbO because it acts as modifier at low content as $[PbO_6]$ group but as the content of PbO increases from 40 to 50 mol%, the value of molar refraction decreases due to change in behavior of PbO from modifier to glass former as $[PbO_4]$ group [15]. Molar polarizability also shows a similar behavior as molar refraction which confirms the role of PbO both as glass network modifier and network former.

Covalency and Ionicity of PbO-B$_2$O$_3$ Glasses. It can be calculated from the electronegativity difference and Pauling's bond ionicity values. The electronegativity difference of glass may be calculated as follows:

$\Delta\chi = \sum X_i \, \Delta\chi_i,$

where$\Delta\chi_i$is the electronegativity difference of glass constituting oxides and is calculated by the relation:

$\Delta\chi_i = \chi_A - \chi_C,$

where$\chi_A$and$\chi_C$are the Pauling electronegativity of anion and cation respectively.

The Pauling's bond ionicity relation I_b can be calculated as:

$I_b = [1 - e^{(-0.25(\chi^2))}].$

Table 2. The oxygen packing density (OPD), covalency, ionicity, Molar refraction, Molar Polarizibility and TPA of the glass samples respectively

Glass	OPD	Covalency	Ionicity	Molar refraction (cm^3/mol)	Molar polarizibility	TPA (cm/GW)
Pb-20	117.892	0.877	0.122	15.130	0.60002	13.27
Pb-30	112.880	0.873	0.126	15.885	0.62984	14.49
Pb-40	97.799	0.869	0.130	16.109	0.63885	15.78
Pb-50	83.642	0.865	0.134	15.457	0.6130	18.54

The covalent and Ionic behaviour of glasses is studied to confirm the effect of PbO on optical absorption of glass samples [14]. The Ionicity of glass samples increases whereas covalency decreases progressively with the increase in PbO content (Table 2). This confirms the conversion of covalent to ionic behaviour of glasses which is responsible for decreasing the optical band gap of glasses with an increase in the concentration of lead oxide.

Photonic View of Glasses. The oxide doped borate glasses are of great interest because of their non-linear optical properties and their applications in photonic devices. Two Photon Absorption (TPA) is one of the most important mechanisms in the field of interactions of radiations with the matter. The two photon absorption coefficient (β) shows non-linear variation with variation in band gap and is related with band gap as follows:

$$TPA[\beta(cm/GW)] = 36.74 - 8.1E_g(eV),$$

where E_g is optical band gap [24].

The obtained value of the TPA has been given in Table 2. The results obtained from TPA values reveal that the incorporation of lead concentration (20-50 mol%) to borate leads to an increase in TPA from 13.27 to 18.54 cm/GW. It clearly indicates the applications of these glasses in photonic devices and this can be achieved by controlling the composition of lead oxide and hence band gap of these glasses.

4. Conclusion

The xPbO-(100-x)B$_2$O$_3$glasses (where x=20-50 mol %) were successfully fabricated using the conventional melt quenching technique. Various parameters such as density, optical band gap, molar volume refractive index, bond ionicity and bond covalency have been found in strong correlation with each other indicating that PbO acts dually: as a network former and a network modifier. The following conclusions can be drawn on the basis of the research:

1. X-ray diffraction analysis confirmed the non-crystalline structure of the prepared glass samples.

2. The density of the glass samples increases non-linearly with increasing PbO content which leads to a non-linear change in the molar volume. When lead oxide is present in small quantities, it causes initial compaction of glass networks due to the transformation of BO$_3$ to BO$_4$ groups, whereas at higher concentrations, PbO causes glass network expansion due to the formation of NBOs and [PbO$_4$] units.The reduction in average boron-boron separation with PbO incorporation confirms the existence of the [BO$_4$] group and lead ions

within the glass network which eventually results in glass network compaction and thus confirms the density and molar volume results.

3. The optical band gap initially decreases when PbO is present in lower concentrations. However, as the concentration of PbO is increased with a corresponding decrease in B_2O_3 content, the optical band gap begins to increase, confirming PbO's role as a network modifier in lower concentrations and a network former in higher concentrations.

4. The addition of PbO helps to increase the refractive index, which supports the density, optical band gap, and molar volume results.

5. The increase in bond ionicity, accompanied by a decrease in bond covalency, confirms the transition of glasses from covalent to ionic.

6. The variation in two photon absorption coefficients reveals that these glasses can be used in photonic devices.

Hence, it can be concluded that the compositional values of PbO and B_2O_3 affect the density, optical band gap, molar volume, TPA and refractive index of glasses. As a result, by controllingthe material composition, all the parameters can be fine-tuned for specific applications.

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