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Structural Investigation, Physical and Optical Properties of Mixed Alkali Bismuth Borate Glasses

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A B S T R A C T

In this paper lithium sodium bismuth borate glasses have been studied to obtain information about the mixed alkali effect. Powder XRD pattern confirmed the amorphous nature of all prepared glasses. The physical parameters and optical properties of all the glasses have been evaluated.DSC characterization has been carried out to determine the glass transition temperature, and the non-linear behavior of density, molar volume, glass transition temperature, direct and indirect optical band gaps and refractive index confirmed the mixed alkali effect. The FTIR analysis revealed that the network structure consists of BO_3, BO_4 units and BiO_6 octahedral units and confirmed the absence of boroxyl rings in the prepared glasses. UV-vis spectroscopy showed no sharp absorption edges giving a clear indication of the amorphous nature of the glasses.

1. INTRODUCTION

A great attention has been drawn on glasses containing heavy metal oxides by several researchers for their excellent IR transmission [1-3]. Lead oxide is hazardous to health and environment. So, use of Lead oxide is eliminated from various applications. Bismuth oxide can replace the Lead oxide for its isoelectronic properties. Bismuth glasses are suitable in Lead-free, low-softening point dielectric glasses for plasma display panel, thick film conductors, sealing glasses for metals, etc. [4, 5]. Bi_2O_3 acts as non-conventional glass forming oxide and it also participates in the glass matrix as [BiO_3] pyramidal units and [BiO_6] octahedral units [6-9]. It has high refractive index, high optical basicity, large polarizability and large optical susceptibility values [10-12]. B_2O_3 consists of boroxyl rings, BO_3, BO_4 units and B-O-B linkages. The existence of structural groups containing both three and four coordinated boron was first postulated by Krogh-Moe [13].

When one alkali is progressively substituted for another in doped alkali oxide glasses, there would be a non linear variation which confirms the occurrence of mixed alkali effect (MAE).There would be a small non linear variation in molar volume, density, refractive index and thermal expansion coefficient. The non linear deviation is more for properties such as conductivity, dielectric relaxation and internal friction [14-17].

Bismuth borate glasses doped with two alkali oxides were prepared to investigate the occurrence of mixed alkali effect in their physical and optical properties. The structural investigation has been done by using FTIR spectroscopy. Till date, there has been ambiguity in the interpretation and understanding of MAE. Hence, this paper addresses the mixed alkali effect in bismuth borate glasses due to doping of two alkali oxides such as Sodium and Lithium oxides.

To the best of our knowledge, for the first time, we have reported the optical properties of lithium sodium bismuth borate glasses.

2. EXPERIMENTAL

2.1. Glass preparation

Glass samples of composition "$xLi_2O+(30-x)Na_2O+55B_2O_3+15Bi_2O_3$"(x=5,10,15,20,25) were prepared by melt quenching technique. All the glass compositions were prepared by mixing stoichiometric amounts of pure analar grade compounds; i.e., Bi_2O_3, H_3BO_3, Li_2CO_3 and Na_2CO_3.The mixture was homogeneously ground for 2 h in an Agate mortar and then melted in a silica crucible at 1100 ºC (in air). The

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melt was kept at this temperature for 30 min in furnace and was stirred to ensure homogeneity. The melt was rapidly quenched in a stainless steel mould and pressed with another disc to obtain glass. All the samples were transparent and light golden yellow in color. The glasses were annealed at 300 ºC for 3h to remove thermal stress and slowly cooled to room temperature.

2.2. Glass characterization

The XRD spectral profiles of prepared glass samples were obtained using *PANalyticalX'Pert Pro* powder X-ray diffractometer operated at 45 kV and 30 mA using Ni filtered Cu-K_α radiation (λ = 1.5418 A). The glassy phase of the samples was confirmed from XRD pattern in Fig.1 as it showed a broad hump at 2θ ~30º.The glass transition temperature of the present glasses was determined using Differential Scanning Calorimeter (*Model METTLER-TOLEDO DSC1*) at a heating rate of 10 ºC/min, temperature range: 25 to 550 ºC, purging gas and flow rate: Nitrogen @ 40 mL/min. FTIR spectra were recorded using *Thermo Nicolet 6700* FTIR spectrometer in the region of 400-4000 cm^{-1}. Optical absorption spectra of the polished glass samples were studied by using *Analytik Jena SPECORD S-600* Spectrophotometer in the region of 200–1020 nm. Density measurements were carried out at room temperature by standard principle of Archimedes with Xylene (density 0.86 gm/cc) as an immersion liquid. The density of prepared glass samples, ρ were calculated by,

$$\rho = \frac{W_a \times 0.86}{W_a - W_b} \quad (1)$$

where, W_a is the weight of the sample in air, W_b is the weight of the sample in Xylene. The molar volume V_m was calculated from

$$V_m = \frac{M}{\rho} \quad (2)$$

where, M is the molecular weight of the sample.

3. RESULT AND DISCUSSION

3.1. X-ray diffraction

The XRD patterns in Fig. 1 showed broad halo peak at around 30º, confirming the amorphous nature of the glasses. Amorphous materials do not possess long range order compared to the crystalline materials.

3.2. Density and molar volume

The density is a powerful tool capable of exploring the changes in the structure of glasses. The measured densities (ρ) of the present glasses with evaluated values of molar volume (V_m) are given in Table1. The compositional dependence of density and molar volume on Li_2O is shown in Fig. 2, Both density and molar volume vary non-linearly indicating the presence of mixed alkali effect in these glass systems. The measured physical parameter values show similar results which have been reported in the literature [18]. The density and molar volume values showed least non linear variation.

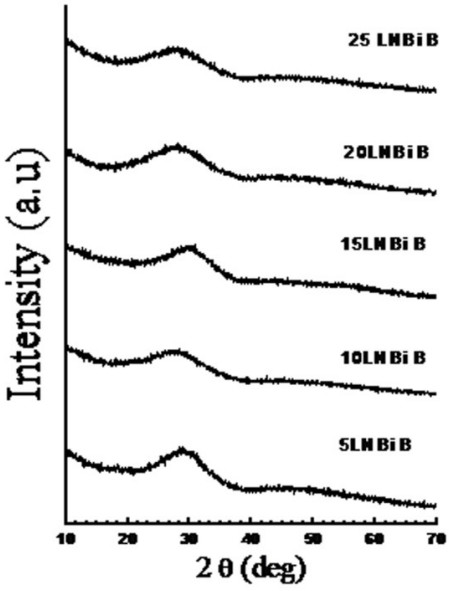


Figure 1. XRD patterns of LNBiB glasses at room temperature

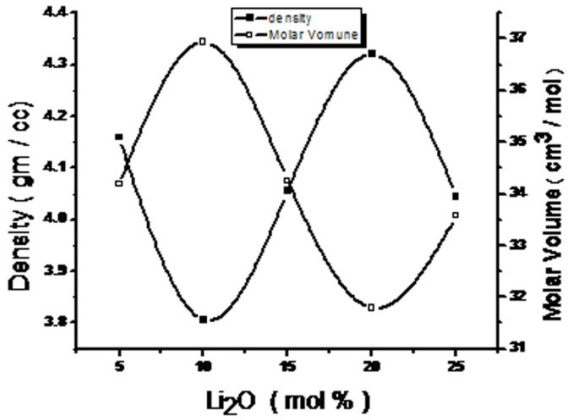


Figure 2. Compositional dependence of density and molar volume

3.2. DSC studies

The glass transition temperature (T_g) depends upon the coordination number of the network forming atoms and the number of non-bridging oxygen atoms (NBOs) [19]. T_g represents the strength of the glass structure. A change in T_g is always attributed to the variations occurring in the glass structure. A decrease in T_g indicates the decrease in oxygen packing density and that the structure becomes loosely packed [20]. The

DSC pattern of all the glasses is shown in Fig. 3, T_g in the present glass system varies in the range of 400 – 468 ºC. The non linear variation of T_g with Li_2O is shown in Fig. 4, which shows the presence of mixed alkali effect. A large non linear variation can be observed in the T_g of the prepared glass samples.

TABLE 1. Density, Molar volume, Oxygen Packing Density, T_g for the LNBiB glasses

Sample	Density (g/cc)	Molar volume (cc/mol)	OPD (g-atom/l)	T_g(˚C)
05LNBiB	4.159	34.183	70.20	434
10LNBiB	3.806	36.938	87.33	468
15LNBiB	4.056	34.261	70.04	400
20LNBiB	4.321	31.790	75.49	449
25LNBiB	4.044	33.574	71.48	447

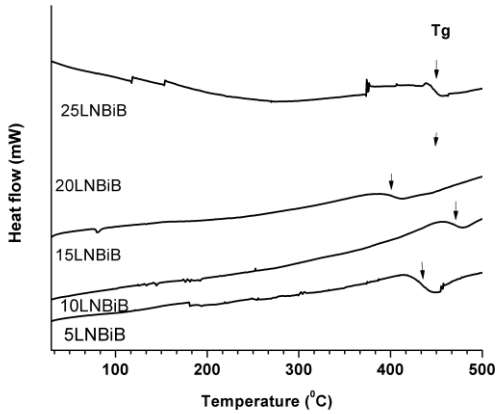


Figure 3. DSC thermograph of LNBiB glasses

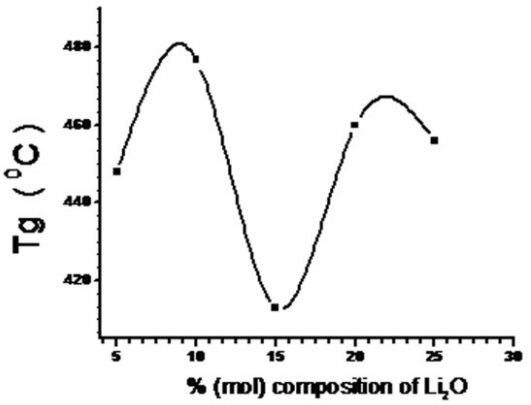


Figure 4. Variation of T_g with Li_2O content

3.3. Analysis of FTIR spectra

IR transmission spectra give information regarding the molecular vibrations or rotations. The FTIR transmission spectra of the present glasses at room temperature are shown in Fig 5. The borate network vibrational modes are active in three infrared spectral regions. The first group of bands which occur at 1200–1600 $cm^{-1}$is due to the asymmetric stretching relaxation of the B–O bond of trigonal BO_3 units, the second group lies between 800 and 1200 cm^{-1} and is due to the B–O bond stretching of the tetrahedral BO_4 units and the third group is observed at around 700 cm^{-1} and is due to bending of B–O–B linkages in the borate network [21]. The IR spectra data has been analysed by comparing the experimental data of glasses with literature data. The absorption peak at 806 cm^{-1} is absent and thereby shows that the glass matrix consists of randomly connected BO_3 and BO_4 groups instead of boroxyl rings [22]. In the literature, Bi_2O_3 of bismuth borate network is reported in the deformed BiO_6 groups [23,24].The band at around 480 cm^{-1} indicates the presence of Bi-O bonds in BiO_6 octahedra [25]. The absence of characteristic band at 830 cm^{-1}shows that BiO_3 polyhedra does not occur; there are only BiO_6 octahedral units present in the glasses [24,26].

The main IR absorption peaks of the present glass system shows three major bands at around 442-506, 961-992 and1371-1393 cm^{-1}.The bands present at around 442-506 cm^{-1} are assigned to the vibrations of Bi-O bonds in BiO_6 octahedral units. The bands observed in the region of 961-992 cm^{-1} are assigned to asymmetric stretching vibrations of B-O bonds in BO_4 units. The bands at around 1371-1393 cm^{-1} is attributed to the B-O bonds due to stretching vibration in the trigonal BO_3units in the glass sample. The observed peaks are in good agreement with the literature values [27-31]. The vibrational band shift is due to changes in the concentrations of alkali contents, Li and Na. These changes indicate the presence of MAE in the glasses.

3.4. Optical studies

The optical absorption spectra of the present glass samples are shown in Fig. 6, The absence of sharp absorption edges gives a clear indication of the amorphous nature of the glass samples. The non linear absorbance edge shows the MAE.

The absorption coefficient, α (υ) is determined near the absorption edge of different photon energies for all glasses and is given by the following relation [32, 33]:

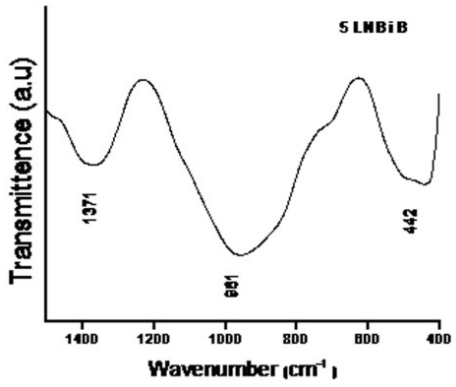
$$\alpha(\nu) = \frac{2.303 \times A}{d} \tag{3}$$

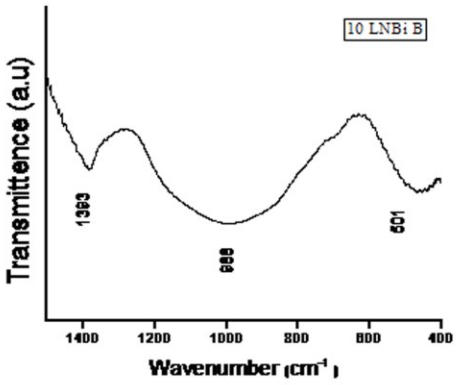
where 'A' is the absorbance and ‘d’ is the thickness of the sample.

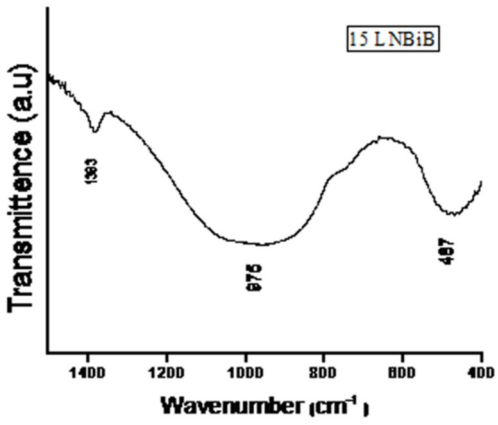
Direct and indirect are two types of optical transitions that can occur at the fundamental absorption edge of crystalline and non-crystalline materials which provides

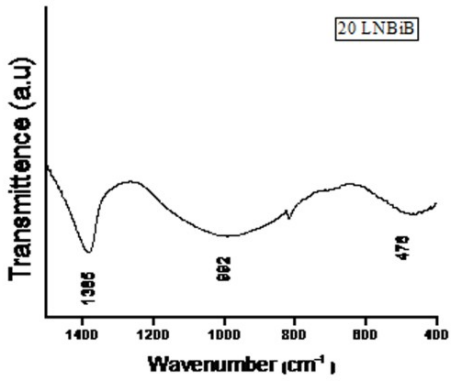
information about band structure and energy gap. In these cases, photons interact with the electrons in the valence band, which gives rise to the photoconductivity response. Davis and Mott [34] gave the following forms of absorption co-efficient as a function of photon energy for direct and indirect transitions.
For direct transition

$$\alpha(\upsilon) = \frac{B(h\upsilon - E_{opt})^n}{h\upsilon} \quad (4)$$









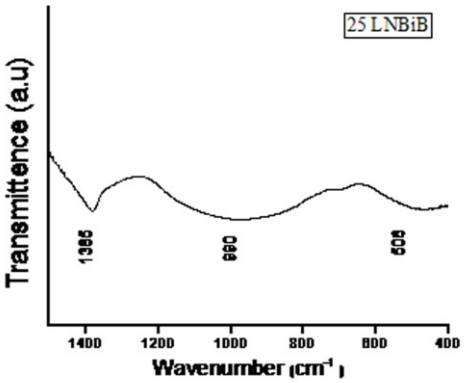


Figure 5. FTIR spectra [a, b, c, d, e corresponding to x = 5,10,15,20,25, respectively]

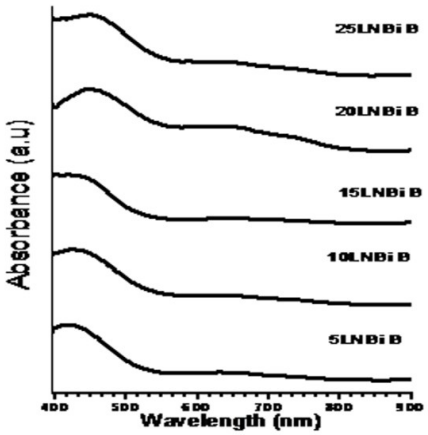


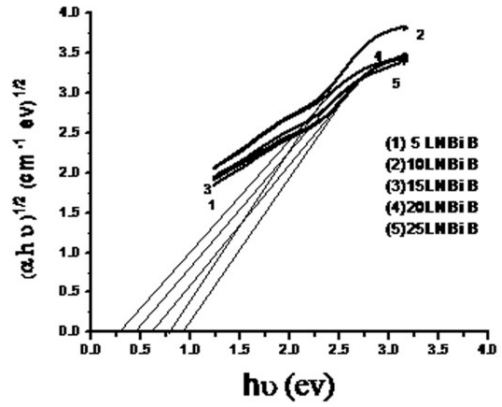
Figure 6. Optical absorption of LNBiB glasses

where n=1/2 for allowed direct transition, B is a constant and E_{opt} is optical band gap. For indirect transitions, n=2.

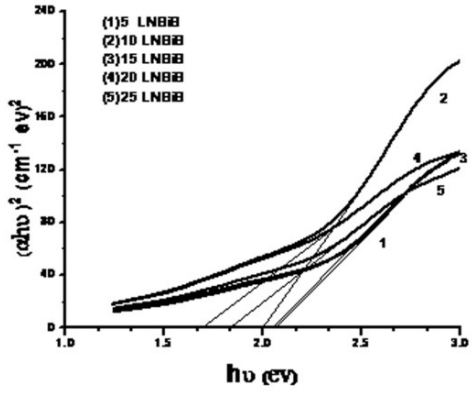
$$\alpha(\upsilon) = \frac{B(h\upsilon - E_{opt})^n}{h\upsilon} \quad (5)$$

Plotting $(\alpha h\upsilon)^{1/2}$ and $(\alpha h\upsilon)^2$ as a function of photon energy hυ, optical band gap, E_{opt} can be obtained for indirect and direct transitions, respectively. E_{opt} values obtained for indirect and direct transitions by extrapolating to $(\alpha h\upsilon)^{1/2}=0$ and $(\alpha h\upsilon)^2=0$, respectively. Plots were drawn between $(\alpha h\upsilon)^{1/2}$,$(\alpha h\upsilon)^2$ vs hυ and are shown in Fig. 7. The direct and indirect optical band gap energy initially decreases till x=20 and thereafter increases. Variation of direct and indirect optical band gap energy is oscillatory in nature. Effect of Li_2O content on the optical band gap energy is as shown in Fig. 8. Theortical optical band gap can be calculated using the following relation;

$$E_{opt} = \frac{hc}{\lambda_0} \quad (6)$$



(a)



(b)

Figure 7. (a) $(\alpha h\upsilon)^{1/2}$ Vs. hυ (b) $(\alpha h\upsilon)^2$ Vs. hυ of LNBiB glasses

where λ_0 is optical absorption edge. Compositional variation of optical absorption edge with Li_2O is shown in Fig. 10, Optical absorption edge initially decreases, reaches minimum at x=15 then further increases. This clearly indicates the presence of MAE. In the prepared glasses, both the direct and indirect band gaps are involved as mentioned in the literature [35].The observation made in the present investigation agrees well with the literature values. [36-39]

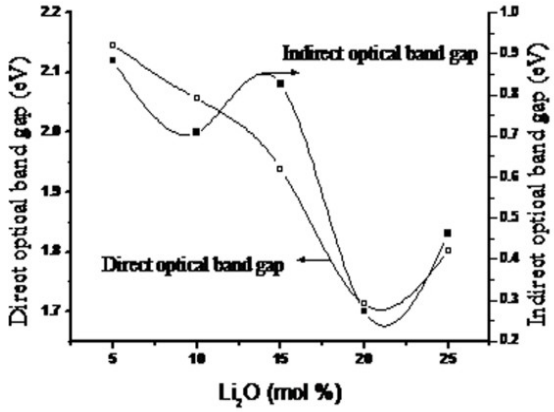


Figure 8. Variation of band gap energy with Li_2O content

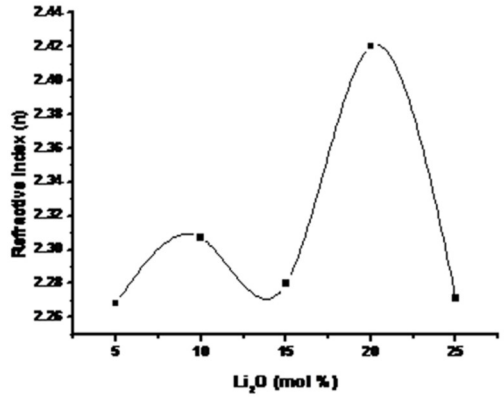


Figure 9. Effect of Li_2O content on R. I

TABLE 2. Optical absorption edge, band gap, R. I. values for LNBiB glasses

Sample	λ_0 (nm)	Band gap (eV)		Theoretical	R. I.
		Indirect	Direct		
05LNBiB	423	0.92	2.07	2.93	2.268
10LNBiB	422	0.79	2.01	2.94	2.307
15LNBiB	417	0.62	2.05	2.97	2.280
20LNBiB	448	0.29	1.70	2.76	2.420
25LNBiB	451	0.42	1.83	2.75	2.271

Refractive index (n) is determined from optical band gap energy using the formula proposed by Dimitrov and Sakka [40, 41].

$$\frac{n^2-1}{n^2+1} = 1 - \left[\frac{E_{opt}}{20}\right]^{1/2} \quad (7)$$

Calculated values of direct, indirect optical band gap energy, theoretical optical band gap energy and absorption edge values of LNBiB glasses are shown in Table 2. Effect of Li_2O content on the refractive index is shown in Fig. 9. Refractive index also shows non linear variation. This may be due to mixed alkali effect.

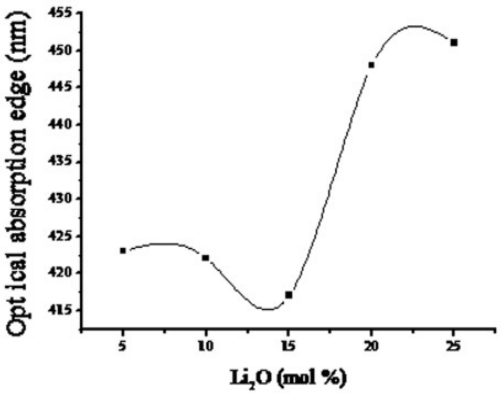


Figure 10. Variation of optical absorption edge with Li_2O content

4. CONCLUSIONS

Powder XRD pattern confirms the glassy nature of all the samples. Density, molar volume and glass transition temperature, direct optical band gap, indirect optical band gap, refractive index and absorption edge shows oscillatory behavior hence confirming the presence of mixed alkali effect. The FTIR analysis of the glasses revealed that the network structure consists of BO_3 and BO_4 units and BiO_6 octahedral bands present in the glasses. The corresponding vibrational band shift is due to change in the concentrations of alkali contents. These changes give the indication of MAE in the glass. The non sharp absorption edges give a clear indication of the amorphous nature of the sample.

5. ACKNOWLEDGMENTS

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