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IR analysis of borate glasses containing three alkali oxides

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ABSTRACT: FTIR investigations on borate glasses containing three alkali oxides, $x\,\mathrm{Li_2O}\cdot x\,\mathrm{Na_2O}\cdot x\,\mathrm{K_2O}\cdot(50-3x)\,\mathrm{PbO}\cdot 50\,\mathrm{B_2O_3}$ (x = 8, 10, 12, 14, and 16 mol%), $10\,\mathrm{Li_2O}\cdot 10\,\mathrm{Na_2O}\cdot 10\,\mathrm{K_2O}\cdot y\,\mathrm{PbO}\cdot(70-y)\,\mathrm{B_2O_3}$ (y = 25, 30, 35, 40, and 50 mol%), and $x\,\mathrm{Li_2O}\cdot x\,\mathrm{Na_2O}\cdot x\,\mathrm{K_2O}\cdot(50-3x)\,\mathrm{Bi_2O_3}\cdot 50\,\mathrm{B_2O_3}$ (x = 8, 10, 12, 14, and 16 mol%) are reported. The present glasses contain mainly BO_3 and BO_4 structural units. The presence of fingerprints of both BiO_3 and BiO_6 structural units in bismuth alkali borate glasses was confirmed. The presence of varied types like di, tetra, penta, and ortho borate groups are confirmed in the glass matrix. Glasses with low PbO content contained more BO_3 units than BO_4 units.

KEYWORDS: glass formers, glass modifier, borate groups, non-bridging oxygen atoms

INTRODUCTION

Alkali borate glasses have been studied for various technical and industrial applications. The insulating property of borate glasses turns into a semiconducting or electronic or ion conducting nature when metal oxides such as alkali and alkaline earth oxides are added to them. Alkali borate glass systems are good candidates for ion conduction and suitable for the fabrication of solid state batteries. Borate glasses with heavy metal ions such as Bi, Pb, Zn etc. exhibit good nonlinear optical properties[1].

Borate glasses containing lead oxide have several applications including radiation shields, optical, and thermal properties[2–4]. Similarly bismuth containing oxide glasses have advantages such as host materials for lasers, long IR cut-off, glass ceramics, memory, and switching devices[5,6]. PbO plays a role of glass former at higher composition while it exhibits glass modifier action at low concentration in the glass[3]. High content PbO glasses contain mixture of both PbO_3 and PbO_4 structural units[7]. Some authors proposed that Bi_2O_3 glasses contain both BiO_6 and BiO_3 groups[4,8].

Fourier transform infrared (FTIR) technique is one of the most sought spectroscopic investigations to probe the structural units present in the glass matrix. The structure of borate glass not only depends on the glass forming oxides, but also on the glass modifier oxides and dopant salts present in the glass composition. The present paper reports structural investigations on borates glasses containing three alkali oxides using FTIR technique. We present the action of PbO when the content of alkali oxides remains the same and when it varies in borate glass structure. The paper also reports the structural groups of Bi_2O_3 when the content of alkali oxides remains constant in the borate glass network.

EXPERIMENTAL

Glass systems $x\,\mathrm{Li_2O}\cdot x\,\mathrm{Na_2O}\cdot x\,\mathrm{K_2O}\cdot(50-3x)\,\mathrm{PbO}\cdot 50\,\mathrm{B_2O_3}$ (x = 8, 10, 12, 14, and 16 mol%), $10\,\mathrm{Li_2O}\cdot 10\,\mathrm{Na_2O}\cdot 10\,\mathrm{K_2O}\cdot y\,\mathrm{PbO}\cdot(70-y)\,\mathrm{B_2O_3}$ (y = 25, 30, 35, 40, and 50 mol%), and $x\,\mathrm{Li_2O}\cdot x\,\mathrm{Na_2O}\cdot x\,\mathrm{K_2O}\cdot(50-3x)\,\mathrm{Bi_2O_3}\cdot 50\,\mathrm{B_2O_3}$ (x = 8, 10, 12, 14, and 16 mol%) were prepared by conventional melt quenching technique. The starting chemicals of AnalaR grade, namely lithium carbonate (Li_2CO_3), sodium carbonate (Na_2CO_3), potassium carbonate (K_2CO_3), boric acid (H_3BO_3), bismuth oxide (Bi_2O_3), and lead monoxide (PbO) were weighed to get the required composition in mole percentage. After thorough grinding, these materials were transferred into a porcelain crucible and melted in an electric furnace at 1000 °C for about 30 min. For homogeneity of glass samples, the melt was frequently stirred. The melt was then quenched rapidly in air onto a stainless steel plate maintained at 373 K and kept at that temperature to relieve mechanical strains in the glass until the stainless steel plate returned to room temperature in ambient conditions. The glass samples thus prepared are clear with no air bubbles. The compositions of the present glass samples were given Table 1.

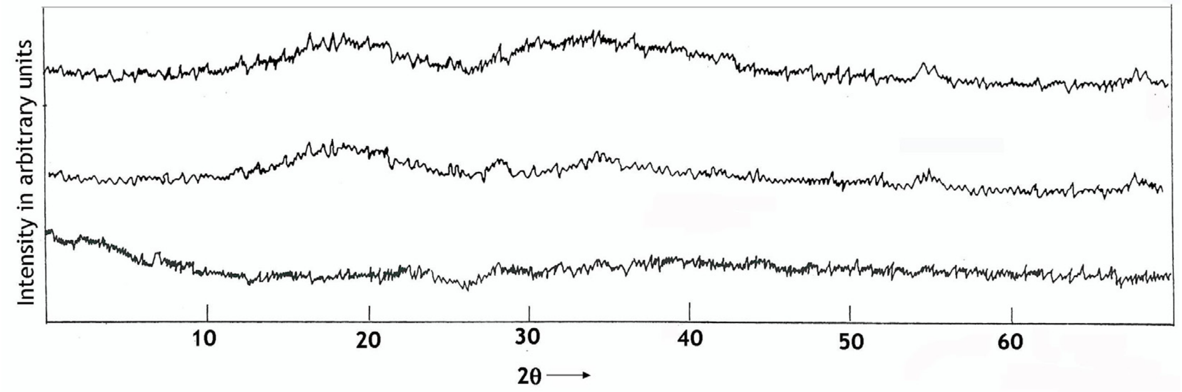

Fig. 1 Powder X-ray diffractograms of some glass samples.

Table 1 Glass compositions.

Glass	Composition
LP1	$8\,Li_2O \cdot 8\,Na_2O \cdot 8\,K_2O \cdot 26\,PbO \cdot 50\,B_2O_3$
LP2	$10\,Li_2O \cdot 10\,Na_2O \cdot 10\,K_2O \cdot 20\,PbO \cdot 50\,B_2O_3$
LP3	$12\,Li_2O \cdot 12\,Na_2O \cdot 12\,K_2O \cdot 14\,PbO \cdot 50\,B_2O_3$
LP4	$14\,Li_2O \cdot 14\,Na_2O \cdot 14\,K_2O \cdot 8\,PbO \cdot 50\,B_2O_3$
LP5	$16\,Li_2O \cdot 16\,Na_2O \cdot 16\,K_2O \cdot 2\,PbO \cdot 50\,B_2O_3$
HP1	$10\,Li_2O \cdot 10\,Na_2O \cdot 10\,K_2O \cdot 25\,PbO \cdot 45\,B_2O_3$
HP2	$10\,Li_2O \cdot 10\,Na_2O \cdot 10\,K_2O \cdot 30\,PbO \cdot 40\,B_2O_3$
HP3	$10\,Li_2O \cdot 10\,Na_2O \cdot 10\,K_2O \cdot 35\,PbO \cdot 35\,B_2O_3$
HP4	$10\,Li_2O \cdot 10\,Na_2O \cdot 10\,K_2O \cdot 40\,PbO \cdot 30\,B_2O_3$
HP5	$10\,Li_2O \cdot 10\,Na_2O \cdot 10\,K_2O \cdot 45\,PbO \cdot 25\,B_2O_3$
HP6	$10\,Li_2O \cdot 10\,Na_2O \cdot 10\,K_2O \cdot 50\,PbO \cdot 20\,B_2O_3$
BB1	$8\,Li_2O \cdot 8\,Na_2O \cdot 8\,K_2O \cdot 26\,Bi_2O_3 \cdot 50\,B_2O_3$
BB2	$10\,Li_2O \cdot 10\,Na_2O \cdot 10\,K_2O \cdot 20\,Bi_2O_3 \cdot 50\,B_2O_3$
BB3	$12\,Li_2O \cdot 12\,Na_2O \cdot 12\,K_2O \cdot 14\,Bi_2O_3 \cdot 50\,B_2O_3$
BB4	$14\,Li_2O \cdot 14\,Na_2O \cdot 14\,K_2O \cdot 8\,Bi_2O_3 \cdot 50\,B_2O_3$
BB5	$16\,Li_2O \cdot 16\,Na_2O \cdot 16\,K_2O \cdot 2\,Bi_2O_3 \cdot 50\,B_2O_3$

All the glass samples were characterized (2θ values between 0 and 80°) by Siemens D-5000 model X-ray diffractometer to check their amorphous nature. The powder X-ray diffractograms of few glass samples are shown in Fig. 1. The FTIR vibration spectra of the present glasses were recorded at room temperature using KBr pellet technique in the wavenumber range 400–4000 cm^{-1} using Perkin-Elmer 467 spectrometer which has a resolution of 1 cm^{-1}.

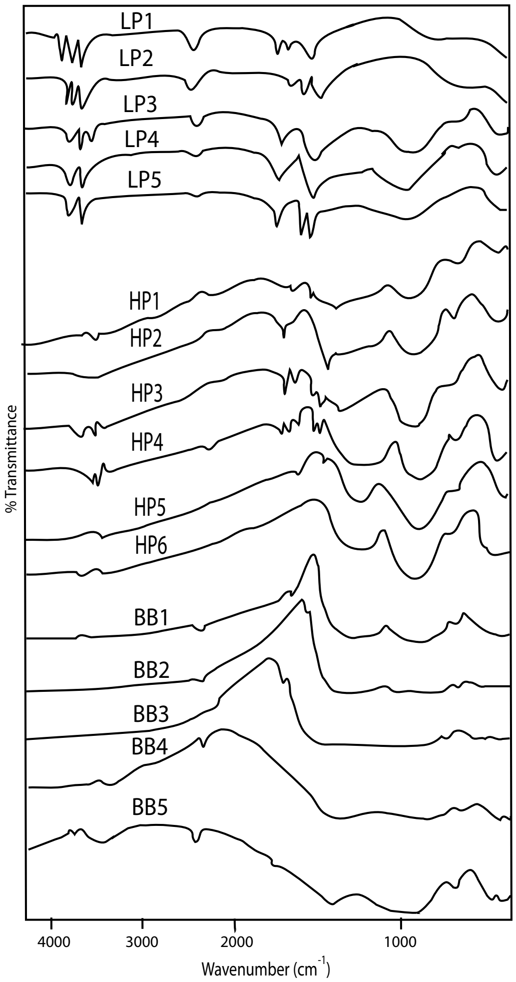

Fig. 2 FTIR spectra of present glass samples.

RESULTS AND DISCUSSION

The amorphous nature of the glass samples was confirmed from the peak free X-ray diffractograms (Fig. 1). Fig. 2 shows the FTIR spectra of glass samples. The peaks corresponding to the infrared bands observed in these glasses are given in Table 2. The main active vibrational modes of the borate network observed in the present glasses are in three infrared groups (1200–1600 cm^{-1}, 800–1200 cm^{-1}, and around 700 cm^{-1}) are similar to the values re-

Table 2 Infrared absorption band positions of LP, HP, and BB glass series.

Glass	IR band positions in wavenumber (cm^{-1})					
LP1	475	667	–	1554	1613	1648
LP2	470	726	–	1506	1615	1694
LP3	473	761	–	1540		1684
LP4	468	726	996	1506		1684
LP5	473	738	1008	1507	1615	1684
HP1	472	726	1008	1388	1506	1684
HP2	473	726	961	1231	1507	1684
HP3	473	720	972	1396	1506	1684
HP4	479	726	944	1255	1508	1684
HP5	473	720	956	1301	1508	1684
HP6	473	726	948	1273	1508	
BB1	497	544	714	954	1320	1649
BB 2	473	520	726	1026	1372	1560
BB 3	462	515	720	979	1407	1654
BB 4	462	509	726	981	1407	1470
BB 5	479	515	726	1012	1425	1713

Table 3 IR band assignments for the glass samples $x\,\mathrm{Li_2O} \cdot x\,\mathrm{Na_2O} \cdot x\,\mathrm{K_2O} \cdot (50-x)\,\mathrm{PbO} \cdot 50\,\mathrm{B_2O_3}$.

Wavenumber (cm^{-1})	IR band assignments
∼470	Pb−O bond vibrations O−B−O bond bending vibrations
∼726	B−O−B linkages bending and isolated BO_3^{3-} groups
∼1000	B−O bonds stretching vibrations in $[BO_4]$ units from diborate groups
∼1230–1270	Asymmetric stretching vibrations of B−O bonds from pyro and ortho borate groups in $[BO_3]$ units
1300	Vibrations of boron oxygen rings
∼1506	$B-O^-$ bond stretching in $[BO_3]$ units due to varied groups
∼1684	Crystal water with H−O−H bending mode

Table 4 IR band assignments for the glass samples $10\,\mathrm{Li_2O} \cdot 10\,\mathrm{Na_2O} \cdot 10\,\mathrm{K_2O} \cdot y\,\mathrm{PbO} \cdot (70-y)\,\mathrm{B_2O_3}$.

Wavenumber (cm^{-1})	IR band assignments
∼470	Pb−O bond vibrations O−B−O bond bending vibrations
∼760	B−O−B linkages bending in borate network
∼1000	B−O bonds stretching vibrations in $[BO_4]$ units from diborate groups
∼1200–1237	Asymmetric stretching vibrations of B−O bonds from pyro and ortho borate groups in $[BO_3]$ units
∼1506–1554	$B-O^-$ bond stretching in $[BO_3]$ units due to varied groups
∼1613–1684	Crystal water with H−O−H bending mode

ported in the literature[9]. In pure B_2O_3 glass, the IR band at 806 cm^{-1} corresponding to characteristic of boroxol ring was not observed in all the present glass samples. The broad composite bands extending in the near infrared region 3200–3800 cm^{-1} are due to hydroxyl or water or B-OH groups present in the samples. The presence of these hydroxyl groups or water groups could be attributed to the (KBr technique) experimental procedure and may also arise due to the presence of water in H_3BO_3. IR band assignments are presented in Tables 3–5.

The intensity of the IR peak ∼2350 cm^{-1} decreased with increasing alkali oxide content and decreased with PbO content in the glass system $x\,\mathrm{Li_2O} \cdot x\,\mathrm{Na_2O} \cdot x\,\mathrm{K_2O} \cdot (50-3x)\,\mathrm{PbO} \cdot 50\,\mathrm{B_2O_3}$ (x = 8, 10, 12, 14, and 16 mol%). The broadening of peaks between 600 and 1000 cm^{-1} was also observed in these glasses.

With increasing PbO content in $10\,\mathrm{Li_2O} \cdot 10\,\mathrm{Na_2O} \cdot 10\,\mathrm{K_2O} \cdot y\,\mathrm{PbO} \cdot (70-y)\,\mathrm{B_2O_3}$ (y = 25, 30, 35, 40, 45, and 50 mol%) glass system, broadening of peaks was observed. The intensity of the peak around 726 cm^{-1} was decreased with increasing PbO content in the glass matrix. With increase in PbO content, the positions of peaks between 1000 and 948 cm^{-1} were shifted to lower wavenumbers in this system of glasses.

In the glass system $x\,\mathrm{Li_2O} \cdot x\,\mathrm{Na_2O} \cdot x\,\mathrm{K_2O} \cdot (50-3x)\,\mathrm{Bi_2O_3} \cdot 50\,\mathrm{B_2O_3}$, the peak intensity around 700 cm^{-1} has grown. Bands around 540 cm^{-1} were shifted to lower wavenumbers while the peaks around 1300–1425 cm^{-1} were shifted to higher wavenumbers. The weak bands around 1613–1684 cm^{-1} indicates existence of crystal water with H-O-H bending mode in the samples[10].

The general identification of finger print IR bands in borate glasses in the group 1200–1600 cm^{-1} occur due to the asymmetric stretching relaxation of the B-O bond of trigonal BO_3 units. The peak positions that lie between 800 and 1200 cm^{-1} are attributed to the B-O bond stretching of the tetrahedral BO_4 units and the bands around 700 cm^{-1} are due to the bending B-O-B linkages in the borate networks[11,12]. IR peaks between 2343 and 2367 cm^{-1} are attributed to the presence of –OH group in the glasses[13,14].

In the glass system $x\,\mathrm{Li_2O} \cdot x\,\mathrm{Na_2O} \cdot x\,\mathrm{K_2O} \cdot (50-3x)\,\mathrm{PbO} \cdot 50\,\mathrm{B_2O_3}$ the infrared transmission bands are centred at 470, 700, 100, 1200, 1510, and 1684 cm^{-1}. The band at 470 cm^{-1} corresponds to

Table 5 IR band assignments for the glass samples $x\,Li_2O \cdot x\,Na_2O \cdot x\,K_2O \cdot (50-3x)\,Bi_2O_3 \cdot 50\,B_2O_3$.

Wavenumber (cm^{-1})	IR band assignments
∼400–600	Bi−O−Bi and Bi−O in BiO_6 octahedral
∼470	Total symmetric bending vibrations of BiO_3 units
∼520	Bi−O bonds in BiO_6 units
∼540	Doubly degenerating stretching vibrations of BiO_3 units
∼726	B−O−B linkages
∼950–980	Stretching vibrations of B−Ø bonds in BØ$_4$ units from tri, tetra, and penta borate groups
1176–1500	B−O stretching vibrations of trigonal BO_3 units only
∼1320–1470	B−O symmetric stretching vibrations of varied borate groups in BO_3 units $B-O^-$ symmetric stretching vibrations of varied borate groups in BO_2O^-

the overlapping of O−B−O bond bending vibrations and Pb−O bond vibration[6]. The weak bands around 1000 cm^{-1} indicate B−O bonds stretching vibrations in BO_4 units from diborate groups. Asymmetric vibrations of B−O bonds from pyro and ortho borate groups in BO_3 trigonal units are confirmed from the peaks in the range 1200–1554 cm^{-1}.

In general, it was observed that the alkali oxides in borate glasses form di, tetra, pyro, penta borate groups. It was reported in the literature that in lead borate glasses with low PbO content (less than 20 mol%), the Pb−O bond is ionic and Pb^{2+} acts as modifier. As the PbO content in the present glass (B-series) is less, an increase in x mol%, decreases the PbO content in the glass composition, and as a result these glasses contain more BO_3 trigonal units rather than BO_4 units. Lead borate glasses show similar IR absorption spectra as that of alkali and alkaline earth borate glasses[12,15,16]. Oxides such as PbO, Bi_2O_3 etc play glass network former/modifier role in the borate glass matrix and enhance the stability of the borate glasses. PbO modifies B_2O_3 back bone structure by forming BO_3 or BO_4 tetrahedral structural units in the glass matrix. Similarly Bi_2O_3, well known glass network former, forms BiO_3 pyramidal and BiO_6 octahedral units in the glass[16,17].

The broadening of peaks between 600 and 1000 cm^{-1} in low lead content LP glass series, i.e., $x\,Li_2O \cdot x\,Na_2O \cdot x\,K_2O \cdot (50-3x)\,PbO \cdot 50\,B_2O_3$ (x = 8, 10, 12, 14, and 16 mol%) with increase in alkali oxide content could be assigned to the modifier action and formation of non-bridging oxygen atoms (NBOs). The decreasing PbO content is favourable to form BO_3 units rather than BO_4 units. The assignment of IR peaks to various vibrational frequencies in the present glass samples indicates that the glass matrix consists of variable borate groups belongs to BO_3 units. Hence, these glasses are rich in BO_3 units.

In contrast to the above LP glasses, HP series glasses, i.e., $10\,Li_2O \cdot 10\,Na_2O \cdot 10\,K_2O \cdot x\,PbO \cdot (70-x)\,B_2O_3$ are rich in lead content ($\geqslant$ 30 mol%). The infrared peak centred around 470 cm^{-1} is a sign of Pb−O bond vibrations present in HP series glass samples (Fig. 2 and Table 2). The peak around 726 cm^{-1} corresponds to vibrations of various borate segments. 948–1008 cm^{-1} bands can be attributed to B-O bonds stretching vibrations of BO_4 units from diborate groups[6,16]. Asymmetric stretching vibrations of B-O bonds from pyro and ortho borate groups in BO_3 trigonal units are established with the presence of peaks between 1200 and 1554 cm^{-1}. Thus HP series glasses contain both BO_3 trigonal and BO_4 tetrahedral units. As the PbO content increases in the glass, some of the BO_3 trigonal units might have converted into BO_4 tetrahedral units and non-bridging oxygen atoms might have formed.

The broadening of bands around 726 cm^{-1} with increasing PbO content in $10\,Li_2O \cdot 10\,Na_2O \cdot 10\,K_2O \cdot y\,PbO \cdot (70-y)\,B_2O_3$ (y = 25, 30, 35, 40, 45, and 50 mol%) might be due to the increasing role of PbO as glass network former rather than glass network modifier. As the alkali content remains constant the increase in PbO content, IR band positions between 1000 and 948 cm^{-1} were shifted to lower wavenumbers and this could be attributed to the formation BO_4 units at the expense of BO_3 units.

Both LP and HP glasses contain bands between 470 and 497 cm^{-1}. The bands arise due to the presence of Pb^{2+} or Pb−O vibrations. Similarly bands around 1300 cm^{-1} correspond to vibration of boron oxygen rings[18,19].

The infrared peaks below 610 cm^{-1} in $x\,Li_2O \cdot x\,Na_2O \cdot x\,K_2O \cdot (50-3x)\,Bi_2O_3 \cdot 50\,B_2O_3$, i.e., in BB series of glass samples arise due to cationic vibrations in the network and various modes of Bi-O vibrations in BiO_6[20–22]. The pure B_2O_3 glass mainly contains boroxol ring $B_3O_{9/2}$ with a trigonal BO_3 unit. When Bi_2O_3 is added, BO_3 units will be converted into BO_4 structural units[22]. According to Bishay and Maghrabi[23] bismuth oxide can take part in the structure of Bi_2O-B_2O_3 glasses in three different ways: (i) forming four coordinated boron atoms by giving its oxygen, (ii) forming BiO_3 units of pyramidal point group C_{3v}, and (iii) creates some non-bridging oxygen atoms.

The vibrations of various borate units in the glass matrix can be studied with the appearance of absorption peaks between 700 and 1550 cm^{-1}. The bands around 470 cm^{-1} are due to doubly degenerate bending vibrations of $[\mathrm{BiO}_6]$ units. The bands around 720 cm^{-1} are due to B−O−B bending vibrations in $[\mathrm{BO}_3]$ triangles. The characteristic Bi−O stretching vibrations of BiO_3 units correspond to bands around 840 cm^{-1}. As no such bands are observed, it can be concluded that BiO_6 units could outnumber BiO_3 units in the glass network[20]. The bands around 1250 cm^{-1} in these glass samples are due to the formation of Bi−O− of $[\mathrm{BiO}_6]$ groups. The infrared band in the range 1170–1260 cm^{-1} can be attributed to the Bi−O−Bi vibrations of $[\mathrm{BiO}_6]$ octahedral units. The presence of bands between 880 and 920 cm^{-1} are evidence for existence of $[\mathrm{BiO}_6]$ octahedral units in the glass samples. Hence, in present $x\,\mathrm{Li_2O}\cdot x\,\mathrm{Na_2O}\cdot x\,\mathrm{K_2O}\cdot(50{-}3x)\,\mathrm{Bi_2O_3}\cdot 50\,\mathrm{B_2O_3}$ glass system, IR bands between 460 and 520 cm^{-1} indicate coexistence of BiO_3 and BiO_6 groups[4,24].

The BB series glasses also contain varied types like tri, tetra and penta borate groups. Stretching vibrations of B−Ø bonds in $\mathrm{BØ}_4$ units (Ø = oxygen atom bridging two boron atoms) from tri, tetra and penta borate groups corresponds to ∼950–980 cm^{-1} band positions. B−O stretching vibrations of trigonal BO_3 units corresponds to 1176–1500 cm^{-1} while bands ∼1320–1470 cm^{-1} signifies B−O symmetric stretching vibrations of varied borate groups in BO_3 units[25]. The increased alkali oxide content played a role in broadening IR bands.

Infrared investigations on present glass systems reveal no significant changes in the overall structure of the glasses with composition. These glasses contained BO_3 and BO_4 units of varies types such as di, tetra, penta and ortho borates groups.

CONCLUSIONS

The characteristic boroxol ring (806 cm^{-1}) was not observed in the present glass samples. The lead glass samples (LP and HP glasses) contained triangular and tetrahedral (BO_3 and BO_4) groups along with Pb−O vibrations. In the bismuth borate glass samples (BB glasses) bands around 1250 cm^{-1} confirmed the existence of Bi−O−Bi vibrations of $[\mathrm{BiO}_6]$ octahedral units. The IR bands between 460 and 520 cm^{-1} in BB glasses indicates the coexistence of BiO_3 and BiO_6 groups. The low lead content LP glasses contained more BO_3 structural units than that of BO_4 units signifying the action of PbO as glass network modifier. Whereas in high PbO content HP glasses conversion of BO_3 structural units into BO_4 units took place and hence PbO acted as network former. As most of the band positions remained the same in the infrared spectra, it can be concluded that the structure of the glasses is stable and the impact of variation of composition on the structure is not much significant.

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