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Effect of Li_2O on structure and optical properties of lithium bismosilicate glasses

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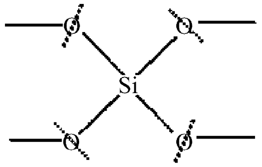
ABSTRACT

Bismuth–silicate glasses containing lithium oxide having composition $x\mathrm{Li_2O}\cdot(85-x)\mathrm{Bi_2O_3}\cdot15\mathrm{SiO_2}$ ($5 \le x \le 45$ mol%) were prepared by melt quench technique. Density, molar volume and glass transition temperature for all the glass samples were measured. IR spectroscopy was used for structural studies of these glasses in the range from 400 to 1400 $\mathrm{cm^{-1}}$. The increase of Li_2O content in glass matrix results in the decrease of the Si–O–Si bond angle and increase in the covalence nature of Bi–O bond. IR spectra suggest the presence of distorted $[BiO_6]$ octahedral units and the degree of distortion increases with the addition of Li_2O in these glasses. The optical transmission spectra in the wavelength range from 200 to 3300 nm were recorded and optical band gap (E_g) was calculated. The values of E_g lie in between 2.81 and 2.98 eV. The values of average electronic oxide polarizability as well as optical basicity in these glasses were found to be dependent directly on Bi_2O_3/Li_2O ratio.

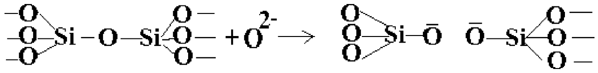
1. Introduction

Silicate glasses containing Bi_2O_3 are of great importance for their industrial and special applications as low-loss fiber optics, infrared transmitting materials or as active medium of Raman fiber optical amplifiers and oscillators [1–4]. These glasses have high nonlinear optical susceptibility and are used in all optical switching and in broadband amplification devices. Further, the high refractive index of these glasses makes them important for advanced optical telecommunication and processing devices [5]. The large polarizability and small field strength of Bi^{3+} in oxide glasses make them suitable for optical devices such as ultra fast all optical switches, optical isolators, optical Kerr shutters (OKR) and environmental guidelines [6]. After an appropriate annealing, these glasses are also used to produce high temperature superconductors with controllable microstructure [7–9]. In addition, these glasses have a very high secondary emission coefficient and used in the production of electron multipliers after reduction in hydrogen atmosphere [10–12]. Wide transmitting window in the optical region having sharp cut-off in both UV–vis and infrared region may make these glasses useful in spectral devices [13].

SiO_2 is one of the most common glass-former and its glass forming range can be extended by addition of alkali oxide. SiO_2 glasses exhibit the three dimensional network of tetrahedral $[SiO_{4/2}]^0$ units and all the four oxygens in SiO_4 tetrahedral are shared; therefore SiO_2 unit as such is neutral.



On addition of alkali oxide (R_2O) to SiO_2, the Si–O–Si linkage is broken (as shown below) and form $Si–O^-$ termination:



Thus, the structure is depolymerised or modified. The oxygen in the Si–O–Si linkage is known as bridging oxygen (BO) and oxygen in $Si–O^-$ is known as non-bridging oxygen (NBO). The alkali ions locate themselves in the structure near the NBO's. The degradation of network is assumed to be systematic as the alkali concentration increases. The modification results in the formation of meta, pyro and ortho-silicates in the order: $[SiO_{4/2}]^0$, $[SiO_{3/2}O]^-$, $[SiO_{2/2}O_2]^{2-}$, $[SiO_{1/2}O_3]^{3-}$ and $[SiO_4]^{4-}$ which are designed as Q_4, Q_3, Q_2, Q_1 and Q_0, respectively [14]. Tenny and Wong. [15] studied the infrared spectra of some alkali silicate glasses and observed a main band at about 1000 $\mathrm{cm^{-1}}$. This band shifts to higher frequencies and its intensity increases, when the alkali oxide content increases. They assigned this band to the formation of SiO_4 tetrahedra with NBOs [16].

Further, Hazra et al. [13] used the Raman and IR spectra to investigate the structure of unconventional lithium bismuthate glasses over the wide range of alkali oxide and found that the structure of this unconventional binary glass system changes systematically with the increase of Li_2O content. In Raman spectra, they observed a broad but strong band at ~380 $\mathrm{cm^{-1}}$ assigned to the Bi–O–Bi

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Table 1

Density (D), molar volume (V_M), glass transition temperature (T_g), cutoff wavelength (λ_{cutoff}), optical band gap (E_g), oxide ion polarisability ($\alpha_O{}^{2-}$) and optical basicity (Λ_{th}) for xLi$_2$O·(85 − x)Bi$_2$O$_3$·15SiO$_2$ glasses.

Glass code	x (mol%)	D (g/cm^{-3})	V_M (cm^3 mol^{-1})	T_g (°C)	λ_{cutoff} (nm)	E_g (eV)	$\alpha_O{}^{2-}(E_g)$	Λ_{th} (×10^{-24} cm^3)
LB1	5	6.27	61.13	470	436	2.84	2.887	1.09
LB2	10	6.15	58.77	462	440	2.81	2.962	1.10
LB3	15	6.07	55.95	454	426	2.90	2.750	1.06
LB4	20	5.92	53.69	442	438	2.83	2.926	1.09
LB5	25	5.84	50.69	435	422	2.93	2.690	1.04
LB6	30	5.72	47.94	428	428	2.89	2.760	1.06
LB7	35	5.48	46.05	412	424	2.92	2.750	1.06
LB8	40	5.73	40.25	393	430	2.88	2.580	1.02
LB9	45	5.61	37.22	375	436	2.84	2.600	1.02

vibration of [BiO$_6$] octahedral units, while a weak band at ~628 cm^{-1} was also observed and was assigned to the Bi–O–stretching vibration (i.e. vibration of non-bridging oxygen) of the [BiO$_6$] octahedral units modified in presence of Li$_2$O. Also it was established that the strength of the glass structure decreases with increase in Li$_2$O content in these glasses and the glass with 30Li$_2$O·70Bi$_2$O$_3$ is found to have extra stability. However, it has been reported [17–21] that the BiO$_6$ and BiO$_3$ units are present and the ratio changes with the other glass component oxides. Recently, Batal has studied [22] the IR spectra of the bismuth–silicate glasses over a composition ranging from 90Bi$_2$O$_3$·10SiO$_2$ (mol%) to 55Bi$_2$O$_3$·45SiO$_2$ (mol%). They observed two very prominent and sharp bands at 471–456 cm^{-1} and at 879–865 cm^{-1} in the mid-infrared region (2000–400 cm^{-1}). The band at about 457 cm^{-1} is attributed to Bi–O bonds in [BiO$_6$] octahedral and shifting to higher wave number (471 cm^{-1}) is due to the increase of the degree of distortion and the second band is due to stretching vibrations of Bi–O bonds in [BiO$_6$] octahedral [23,24]. These studies on bismuth–silicate glasses suggests the sharing of Bi^{3+} in network glass structure as octahedral BiO$_6$ groups and the possibility of the presence of BiO$_3$ units.

Thus, the structural role played by Bi$_2$O$_3$ in silicate glasses is complicated and imperfectly understood because the [BiO$_n$] polyhedrons are highly distorted due to lone pair electrons. As the bismuth oxide possess asymmetrical structural units in the crystalline state, it is possible to form bonds with different lengths in the distorted polyhedra. Hence the glass structure may be considerably different depending on their contents. Hazra et al. [13] claimed that [BiO$_6$] octahedral units were present as network former in bismuth containing glasses and influence of lithium ions in the glass matrix of these glasses was also confirmed from optical Raman and electrical properties. These results were further supported by the earlier studies of Moguš-Milankoviæ et al. [25] in which bismuth ions were assumed to be present in network forming units as very deformed BiO$_6$ groups. Bishay and Maghrabi. [26] claimed that BiO$_3$ groups were formed in Bi$_2$O$_3$ containing glasses. It has been indicated [25,27] that bismuth ions can participate both as network modifier and as network former and the ratio of these entities depends on the type and percentage of the other glass constituents. The main objective of this present work is to investigate the structural changes induced in bismuth–silicate glasses when the unconventional glass former Bi$_2$O$_3$ is gradually replaced by the modifier Li$_2$O in the presence of fix content of traditional glass former SiO$_2$ in the glass composition. For this a systematic study of the optical UV–vis, infrared spectra, density and thermal properties of some ternary lithium bismosilicate glasses was performed. Particular attention was devoted to the mid infrared region (400–1400 cm^{-1}) which provides the structural information.

2. Experimental details

2.1. Synthesis

The lithium-bismosilicate glasses were prepared having composition xLi$_2$O·(85 − x)Bi$_2$O$_3$·15SiO$_2$ ($5 \le x \le 45$ mol%) by simple melt-quench technique. The 15 g batches of reagent grade Li$_2$CO$_3$, SiO$_2$ and Bi$_2$O$_3$ chemicals were taken in appropriate proportion and melted in porcelain crucible in air at 1150 °C temperature for 30 min. The melt was then poured on stainless steel plate and quenched quickly at room temperature (RT). Table 1 gives the composition of the prepared glass samples.

2.2. Density measurements

The density (D) of each glass sample was measured at RT using Archimedes method with xylene as the immersing liquid. The density was calculated according to the formula

$$D = \frac{W_{air}}{W_{air} - W_{xylene}} \times \rho_{xylene} \tag{1}$$

where W_{air} and W_{xylene} are the weight of glass sample in air and xylene, respectively and ρ_{xylene} is the density of xylene (=0.8645 g/cm^3). All measurements were repeated thrice. The accuracy of the results in triplicate measurements is approximately ±0.001 g/cm^3. The molar volume was calculated from the traditional relation $V_M = M/D$ where M is the molar mass of the glass.

2.3. DSC measurements

DSC for powder samples were made at heating rate of 10 °C/min. for the determination of glass transition temperature (T_g) using differential scanning calorimeter (Q10, TA Instruments). The estimated error in the measurements was ±5 °C.

2.4. IR transmission measurements

IR transmission spectra of the glasses were recorded at room temperature using KBr pellet technique on a Shimadzu FTIR 8001PC spectrometer in the range 400–4000 cm^{-1}. The samples were grinded and were mixed with pulverized KBr in the ratio 1:20 (by weight). The weighed mixture was then subjected to a pressure of 7–8 ton to produce clear homogenous discs. The infrared transmission spectra were measured immediately after preparing the desired discs.

2.5. UV–vis transmission measurements

The UV–vis transmission spectra of the glasses were recorded with a UV–vis–NIR spectrometer (Cary 5000, Varian) in the spectral range 200–3300 nm at RT.

3. Results and discussion

3.1. Density and molar volume

The variations of density as well as the molar volume of all the glasses under study are shown in Fig. 1 and their values are presented in Table 1. It is obvious that the density values decreases with the increase in Li$_2$O content. A similar result has been reported on binary lithium bismuthate glasses [13]. This data are expected in relation to the high mass of the heavy metal cation (Bi^{3+}). Qi et al. [28] have related the density of binary silicate, borate and phosphate oxides containing Bi$_2$O$_3$ to electronic polarizability of the oxide ion and therefore the data are much larger than those of binary oxide glasses containing monovalent or divalent metals. The anomalous behavior in the density change is observed at x = 35 mol% with lowest density value suggests some major structural changes at this composition in the studied glasses.

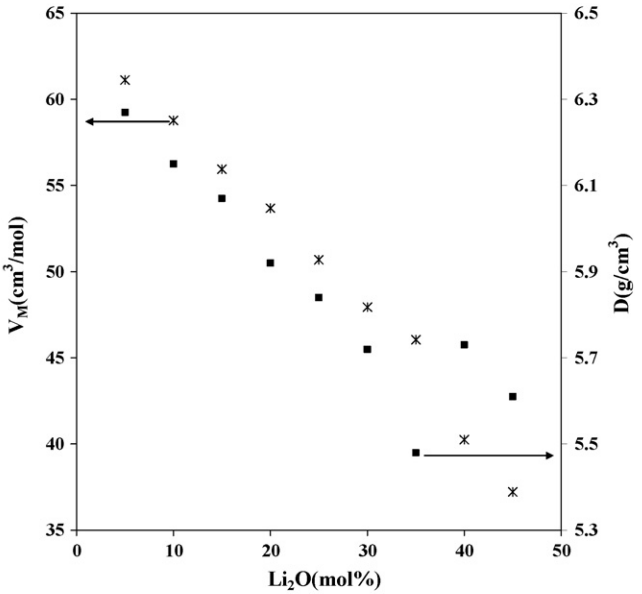

Fig. 1. Composition dependence of density and molar volume of lithium bismosilicate glasses.

3.2. *Glass transition temperature*

The values of glass transition temperature (T_g) obtained from DSC measurements are presented in Table 1. It is observed from the table that T_g of $x\mathrm{Li_2O}\cdot(85-x)\mathrm{Bi_2O_3}\cdot15\mathrm{SiO_2}$ glass system decreases with increase in Li_2O content. The rate of decrease in T_g is more pronounced for $x > 35$ mol%. This decreasing behavior of T_g with the increase of Li_2O content ensures the formation of non-bridging oxygen in the glasses and it depends on the interaction of Li_2O with matrix. A similar result has been reported in $\mathrm{Li_2O\cdot Bi_2O_3}$ glass system [13]. The change in the transition temperature of the studied glass samples with composition can be realized by considering that the strength of the network decreases as the somewhat stronger Bi–O bonds are replaced by the weaker Li–O bonds and further due to increase in NBOs by the transformation of SiO_4 units to SiO_2 units with increase in Li_2O content. This decrease in average bond strength, leads to a decrease in the glass transition temperature. Firstly, up to $x < 35$ mol% where, Li^+ cations are subtracted to their network modifying role owing to the need of charge compensation of the $[BiO_6]$ octahedron, a slow decrease in T_g occurs at lower content of Li_2O. However, for a particular ratio of Bi_2O_3/Li_2O when the charge compensation exceed, the Bi–O bonds are replaced by weak Li–O bonds and hence weaken the strength of network resulting in fast decrease in T_g at higher content of Li_2O.

3.3. *Infrared transmission spectra*

Infrared spectroscopy is one of the most useful experimental techniques available for easy structural studies of glasses [29]. As this technique leads to structural aspects related to both the local units constituting the glass network and the anionic sites hosting the modifying metal cations, infrared is a powerful tool for the structural studies of glasses modified by metal oxides. It is accepted that the main vibrational modes appeared above 400 cm^{-1} in mid infrared range are associated with structural chain in the glass network [30–32]. These network modes are well separated from the metal ion site vibrational modes active in the far infrared region [32,33]. The Fourier transform infrared (FTIR) spectrum for ternary lithium bismosilicate glasses of the composition $x\mathrm{Li_2O}\cdot(85-x)\mathrm{Bi_2O_3}\cdot15\mathrm{SiO_2}$ ranging from $5 \le x \le 45$ mol% were measured and are shown in Fig. 2. The mid and near infrared spectra of the studied lithium bismosilicate glasses show some resemblance to the spectra usually obtained from the traditional silicate glasses and crystals [30]. But the positions of absorptions bands are different due to the abundance of the heavy metal bismuth oxide (Bi_2O_3) and modifying cation (Li_2O). The mid region extending from 2000–400 cm^{-1} is characterized by the appearance of the characteristic absorption bands of the network forming groups [30].

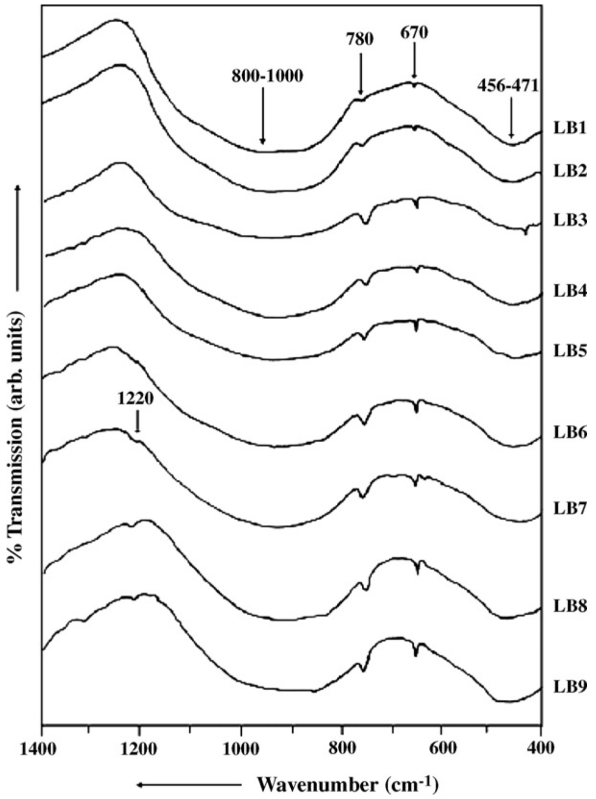

Fig. 2. Infrared transmission spectra for $x\mathrm{Li_2O}\cdot(85-x)\mathrm{Bi_2O_3}\cdot15\mathrm{SiO_2}$ glasses.

3.3.1. *Contribution of IR transmission spectra*

There are three fundamental vibration bands for the silica structure in the wave number range of 450–1200 cm^{-1} observable both by IR and Raman spectroscopy [34–36]. First is the asymmetric stretching (AS) mode observed in the wave number range of 1050–1100 cm^{-1}. Second is the symmetric stretching (SS) mode observed in the wave number range of 790–810 cm^{-1} and the third vibration band of silica is the bending-rock mode (R) observed in the wave number range of 440–470 cm^{-1}. The wave number of silica structural bands shifts with the Si–O–Si bond angle changes. It is [34–36] known that as the Si–O–Si bond angle increases the band at ~1050 cm^{-1} shifts to higher wave number while the other two bands at ~790 and 440 cm^{-1} shifts to lower wave number. A frequency increase in the band from 456 to 471 cm^{-1} with increase in Li_2O content denotes the increasing depolymerization of the silicate network. The infrared bands in the 420–480 cm^{-1} region were reported to the characteristic absorption bands of $[BiO_6]$ polyhedra [4]. Also Bi–O bonds are expected to have different degrees of covalent or ionic in nature, the more covalent one corresponds to higher frequencies [4]. The detailed infrared absorption spectra obtained can be realized and interpreted on the following basis:

(a) The compositional variations extending from $5\mathrm{Li_2O}\cdot80\mathrm{Bi_2O_3}\cdot15\mathrm{SiO_2}$ to $45\mathrm{Li_2O}\cdot40\mathrm{Bi_2O_3}\cdot15\mathrm{SiO_2}$ shows high bismuth content in the studied bismuth–silicate glasses. With the high Bi_2O_3 content ratio, it is expected to reflect the effect of the high mass cations of Bi^{3+} on the infrared absorption spectra.

(b) In the IR spectra of studied glasses a broad but strong band at 456–471 cm^{-1} was observed and shifts towards longer wave number as bismuth decreases and Li_2O increases suggesting loosening of the glass network leading to decrease in glass transition temperature (DSC results). This shifting with Li_2O content is related to the change of local symmetry of the polyhedra. It has been accepted by various authors [20,22] that this band originates from Bi–O bands in BiO_6 octahedra and the shifting to higher wave number (471 cm^{-1}) is due to the increase of the degree of distortion [20]. Dimitriev and Mihallova [37] have also attributed the shift in band from 482–520 cm^{-1} to the variation in the local symmetry of highly distorted BiO_6 polyhedra. It has been shown [34–36] that the band observed in the wave number range of 440–470 cm^{-1} is attributed to the bending-rock mode (R) BO's bonding. Therefore, the band near 456–471 cm^{-1} may composes of two bands attributed to Bi–O in BiO_6 and bending-rock mode (R) BO's bonding. The same integrated intensity ratio between the band near 1000 cm^{-1}(stretching mode of silicate network) and the band near 456–470 cm^{-1} in the studied glasses suggests that most of the intensity of band near 456–470 cm^{-1} comes from bending vibrations of the silicate network. The shifting to higher wave number is due to depolymerization of the network and decrease in bond angle of Si–O–Si which is also supported by the fact of shifting of the band at 1050 cm^{-1} to lower wave number [34–36].

(c) The spectrum of vitreous SiO_2 shows in addition to the band at 450 cm^{-1}, a weak band at about 800 cm^{-1} and a strong one at about 1080 cm^{-1}. The later is accompanied by a broad shoulder centered around 1200 cm^{-1}. These bands are attributed to different vibrational modes of Si–O–Si links [38]. It is reported that [39–41] when a network modifying oxide is added the bands associated with glass network shifts towards lower wave numbers and broadens. This is due to [39–41] the build up of SiO_4 tetrahedral units bearing a progressively higher number of non bridging oxygens. According to Handke and co-workers [42,43] the cation-modifiers induce the appearance of a band due to the symmetric stretching vibrations of the terminal $Si–O^-$ bond which occurs through breaking of parts of the oxygen bridges under the influence of these modifiers. In sodium silicate glasses [16] the weak band around 800 cm^{-1} shift to about 780 cm^{-1} and its position seems independent of the alkali oxide concentration. In the spectrum of present glass system therefore, the broad band in the higher wave number range (800–1000 cm^{-1}) is linked to the stretching vibrations modes of the SiO_4 tetrahedra, it partially overlaps the band at 780 cm^{-1} due to bending vibration modes of Si–O–Si [20]. However as the substitution proceeds, overlaps with the broad band at 800–1000 cm^{-1} due to the tetrahedral units stretching modes, progressively reduces owing to a sharpening of the later. The infrared band at 1220 cm^{-1} corresponds to Si–O–Si stretched vibrations [4] and appears in the present glasses when $Bi_2O_3 \leq 55$ mol%. In the mid infrared region, the strong and broad band centered about 880 cm^{-1} shifts to higher wavenumber when the bismuth content increases and may be related to Bi–O–Si stretched vibrations [4].

(d) A sharp peak at 670 cm^{-1} in all the glasses were observed with increase in intensity as the Bi_2O_3 content decreases or Li_2O increases. Betsch and White [44] reported detailed data on IR and Raman spectra of α-Bi_2O_3 and bismuthate phases $Bi_{12}MO_{20}$ having the silenite structure. With these investigations, band at 670, 620, 580 and 390 cm^{-1} in the spectrum of α-Bi_2O_3 were interpreted as vibrations of Bi–O bonds of different lengths in the distorted BiO_6 polyhedra. It was also found that there is no infrared band corresponding to the existence of $[BiO_3]$ polyhedra which are at 840 cm^{-1} [20]. These experimental observations suggest that the $[BiO_3]$ polyhedra are absent in the present glasses and therefore Bi^{3+} cations are interpreted in $[BiO_6]$ groups only [20]. It was also found that there is no infrared bands which corresponds to Si–O–Si stretched vibrations at 1124 and 1220 cm^{-1}(may be overlapped) but as bismuth decreases the weak band at 1220 cm^{-1} is just start to appear at $x \geq 35$ mol% in the glass composition. The structures of bismuth oxygen polyherda are more flexible owing to the more ionic nature of the Bi–O bonds; therefore this network can incorporate the SiO_4 tetrahedra to a certain extent.

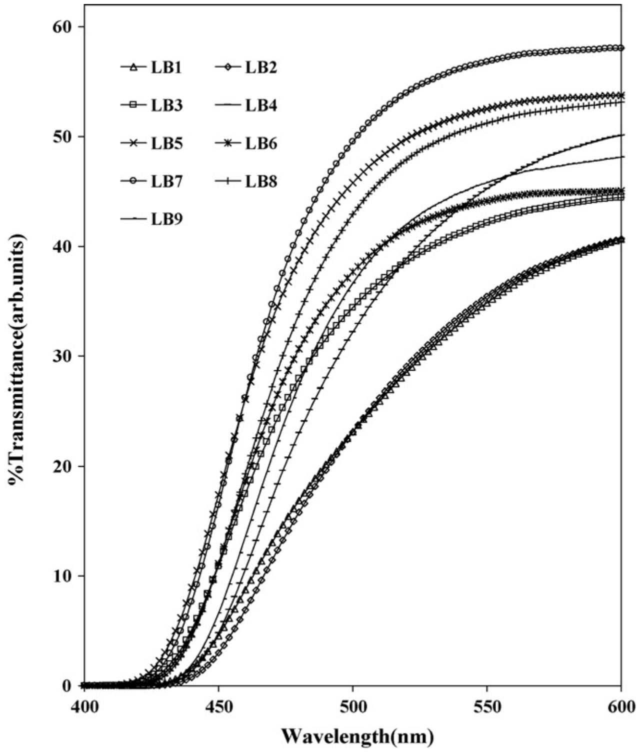

Fig. 3. Optical absorption spectra for $xLi_2O{\cdot}(85-x)Bi_2O_3{\cdot}15SiO_2$ glasses.

3.4. *Optical band gap*

The optical transmission spectra of the samples were recorded at room temperature and the effect of lithium oxide on the optical absorption edge in bismosilicate glasses is shown in Fig. 3. It is observed that optical absorption edge is not sharply defined, which indicates the amorphous nature of the samples. It is also observed that the cutoff wavelength shifts towards longer wavelength as the content of Li_2O increases beyond 35 mol%. This shift may be attributed to the increase in number of the nonbridging oxygen ions beyond a particular ratio of Bi_2O_3/Li_2O. The optical band gap in amorphous system is closely related to the energy gap between the valence band and conduction band [45,46]. Although the conduction band is mainly influenced by the glass forming anions, the cations also play a significant role indirectly. The variations in optical band gap (E_g) values (Table 1) with the alkali content may be attributed to indirect influence of Li_2O on the band gap [47]. The value of E_g for all the samples lies between 2.81 and 2.98 eV. The values for average electronic oxide polarizability ($\alpha_{O^{2-}}$) of the glasses are calculated by using the relation [47]:

$$\alpha_{O^{2-}}(E_g) = \left[\left(\frac{V_M}{2.52} \right) \left(1 - \frac{(E_g)^{1/2} - 1.14}{0.98} \right) - \sum_i p_i \alpha_i \right] q^{-1} \qquad (2)$$

where p and q denote the number of cations and oxide ions, respectively in the chemical formula A_pO_q. It is observed that the average electronic oxide polarizability directly depends upon Bi_2O_3/Li_2O ratio which is an expected result due to the presence of lone pair in the valence shell of Bi^{3+} ions having high polarizability. Since the lone-pair–bond-pair repulsion is greater than the bond-pair–bond-pair repulsion, the distance Bi–O_{ax} (axial bonds) and Bi–O_{eq} (equatorial bonds) becomes larger which increases the polarizing effect of Bi^{3+} cation on oxide ions. In the present glass system, the existence of lone pair is reduced as the negative charge is compensated by the successive addition of Li_2O in the glass matrix results in a decrease of the polarizing effect of Bi^{3+} cations on oxygen and hence $\alpha_{O^{2-}}$ is decreased. The theoretical optical basicity of these glasses has also been obtained using the relation established by Duffy [48]:

$$\Lambda_{\mathrm{th}} = 1.67\left(1-\frac{1}{\alpha_{O^{2-}}}\right) \qquad (3)$$

and its values are given in Table 1. The optical basicity decreases with Bi_2O_3/Li_2O ratio causes a shifting from ionic to more covalent character of the bonding between cation and oxide ion in these glasses.

4. Conclusions

The decrease in the density and molar volume with the addition of Li_2O indicates that some the structural changes occur in bismosilicate glasses. The interaction of Li_2O with glass matrix weakens the strength of glass network and hence causes decrease in glass transition temperature. In IR studies, shifting of band around 456–471 cm^{-1} to higher wave number and of the band at 800–1100 cm^{-1} towards lower wave number suggest the decrease in O–Si–O bond angle. The presence of distorted $[BiO_6]$ octahedral units in the glass network was observed and the degree of distortion was found to be increasing on progressive substitution of bismuth by Li^+ ions. The shifting of absorption edge towards longer wavelength for $Li_2O > 35$ mol%, suggests the formation of NBOs in the glass structure. Optical band gap lies between 2.81 and 2.98 eV. The smaller average electronic oxide polarizability as well as optical basicity of the glasses supports the assumption that the introduction of Li_2O in the glass matrix results a change in local symmetry of $[BiO_6]$ octahedral and increases the covalence nature of Bi–O bond.

Acknowledgements

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