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Transmission and Absorption Properties in the Novel Ultra-optical TiO_2-Bi_2O_3-PbO Glass System

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ABSTRACT

Glasses with ultra-optical properties are most favored in all-optical communication devices, e.g. switches. Heavy polarizable Bi_2O_3 and PbO, in their high contents, have achieved the most high index of refraction and dispersion in oxide glasses, particularly in cooperation with relatively heavy glass conditional former, such as, TiO_2. In this research, transmission and absorption properties of novel TiO_2-Bi_2O_3-PbO (TBP) glass system were characterized by the spectrophotometry techniques in the near infera-red (2.5-10 μm) and ultraviolet/visible (0.25-1 μm) regions. The corresponding traces were explored with respect to the glass compositions. The Taus plot (method) was executed for the absorption coefficient (attenuation) measurements, and the subsequent related predictions in the uv/visible regions. Results indicated that TBP glasses claimed relatively appreciable absorption around, 7μm due to Ti-O bonds. Addition of TiO_2 shifted transmission cut off and the related absorption peaks to higher wavelengths and broadened the absorption region. In the uv/visible region, addition of TiO_2, as a conventional glass former, widened the transmission window in all by shifting attenuation to shorter wavelengths, where steeper absorption tails were observed. The overall attenuation in TBP glasses were more affected by Bi_2O_3 than PbO. Prog. Color Colorants Coat. 14 (2021), 67-78© Institute for Color Science and Technology.

1. Introduction

In all-optical communications, linear and non-linear optical properties, and in particular transmission and absorption are under prime consideration with respect to energy consumption and transmission limitation. The spectroscopy studies of transmission and absorption properties of optical glasses, in infra-red and UV/Visible regions, determines the media transmission characteristics and defines boundaries in the design of optical devices, particularly in applications concerning; optical fibers, all-optical switches, filters and etc. Since, in all-optical communication, transmission in infra-red regions, around 1.25 to 1.65 μm, was in the interest of optical fibers, attempts have been devoted to investigating desirable glasses with wide near infra-red (IR) transmitting windows. Fluoride, chalcogenide, phosphate based glasses are referred to IR transmitting glasses [1-4]. In the relevant all-optical communication systems concerning devices, such as; switches, ultra-optical and the consequent third non-linearity properties of glasses, along with the low attenuation (absorption coefficient) in UV/Visible and near infra-red (IR) transmission regions, are under particularly

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considerations. In correspondence with the mentioned requirements, heavy atom oxides with suitable glass formers may be tailored to propose appropriate glasses for all-optical based devices [5-7].

In discrete spectrophotometry experiments for the different stable glass compositions, dependence of near infra-red (0.78-1000 μm) transmission and uv/visible (0.25-1 μm) absorption on ionic bonds and electrons configurations have been studied, in order to detect a desired transmission window in association with the optical properties. Generally, absorption property of glass is characterized by the Beer,s Law expression, where the absorption coefficient or attenuation η cm^{-1}, is defined in the following equation (Eq. 1) [8]:

$$\frac{I}{I_o} = e^{-\eta t} \tag{1}$$

Where I_o and I are the incident and transmitted intensities through glass of t (cm) thickness, respectively.

Transmitted incident rays, in the infra-red region through a transparent glass, are normally absorbed by different resonances of constituting atomic bond vibrations, which can be bond stretching and bending. The latter acquire lower energy level. Resonance frequency decreases with the increase of; atomic sizes and weight, bond length, and ion coordination numbers in the glass network structure. Thus, heavy atoms bonded to oxygen, e.g., Pb-O and Bi-O, in comparison with small atoms, e.g., Si-O and B-O, tend to have lower absorption band frequencies or say longer wavelengths, [9-12]. However, ultra-violet absorption in glass as a transparent medium may occur, when the incident light rays are in resonance with the frequencies of electrons configured in the ions, constituting the glass [13].

Referring to Babcock and Rawson [14-16] absorption can be considered as the imaginary component of refractive index variation with frequency, i.e., dispersion. Therefore, absorption may approaches maximum as dispersion increases. Urbach [17, 18] proposed an empirical relationship (equation 2) between the uv/visible absorption coefficient (attenuation), η and the incident photon energy, $E=h\nu$, $\nu=1/\lambda$, where, h is the planks number, and ν and λ are frequency and wavelength, respectively.

$$\eta = Ae^{[(E-E_g)/\Delta E]} \tag{2}$$

After rearranging equation 2:

$$\ln(\eta) = E/\Delta E-[E_g/\Delta E-\ln(A)] \tag{3}$$

Where, A is the proportionality constant, `$E_g$` is the band gap energy or optical gap which is the material dependent and is considered constant (3.5 eV or $\sim 1x10^5 cm^{-1}$), [19, 20]. It concerns the photon energy corresponding to uv absorption edge and is estimated by Tauc plot or method for a particular composition [19], and ΔE is referred to as the Urbach width. The latter is the material and temperature dependent and is considered constant. Hence, logarithm of the attenuation is expected to be in proportional to the incident photon energy, and is expected to vary linearly [21].

In these series of studies, attentions are drawn towards transmission and absorption characteristic behavior of highly content PbO and Bi_2O_3 heavy atom oxide glasses. These oxides are highly polarizable and acquire ultra-optical properties, in the favor of the above applications requirements. Absorption characteristic curve of these glasses in correlation with the optical properties, for instance the refraction and dispersion tend to move to longer wavelengths with the tail extended in the low spectra visible region of incident light. Since the above oxides cannot form glass alone, TiO_2, as a conditional glass former may be added, in order to make stable glass. TiO_2 introduces relatively high proportion of oxygen, as a strong anion, which induces structural distortion and causes high polarizable states in the glass network. Generally, the glass network polarization is particularly enhanced, when the probability of the non-bridging oxygen is expected to rise with the oxygen population. In addition, Ti^{4+} in comparison with other glass formers is heavier and larger, and so demanding higher polarisability [22, 23]. Thus, TiO_2 could compromise polarisability effects of the above heavy oxides on achieving the ultra-optical properties of the tailored glasses, when are substituted by TiO_2. Correspondingly, TiO_2 is a high photon energy absorber and would act as a photo-catalyst, in relation to ultra-violet light in different applications, [24, 25].

2. Experimental

Different batches of TiO_2, Pb_3O_4 and Bi_2O_3 of 99.9 wt % purity were prepared by tumbling in a mixing jar for three minutes. Referring to TiO_2-Bi_2O_3-PbO (TBP) ternary phase diagram in [22]. The melting processes were consistence and performed continuously in separate covered α-alumina crucibles heated at a maximum temperature 1100 °C for half an hour along with agitation. Suitable stable glasses for the spectrophotometry tests were attained around the eutectic and peritectic regions, (Table 1). Homogeneous glass samples, free from any cracks, inclusions or any defects, were chosen. Parallel-sided slabs of about 1±0.05 mm thickness were cut, ground and polished with SiC powders (200, 450 and 600 meshes) and cerium oxide power of particle size 0.35 µm in liquid paraffin, consecutively. Finally, appropriate specimens, with uniform, smooth and cleaned surfaces, were shaped and prepared for the spectrophotometry experiments. Perkin Elmer 683 and 330 Spectrophotometers were employed for infra-red transmission set in the spectrum ranges of wavenumbers (inverse of wavelength) between 4000 and 500 cm^{-1}. Figure 1 presents an example of the trace of the infra-red spectrophotometer, corresponding to the glass sample, in Table 1, $20TiO_2$-$10Bi_2O_3$-70PbO (20TBP70).

3. Results and Discussion

The IR traces for the stable TiO_2-Bi_2O_3-PbO glasses have been similar and displayed two absorption peaks at wavenumbers (1/λ) shown in Figure 1 and Table 1.

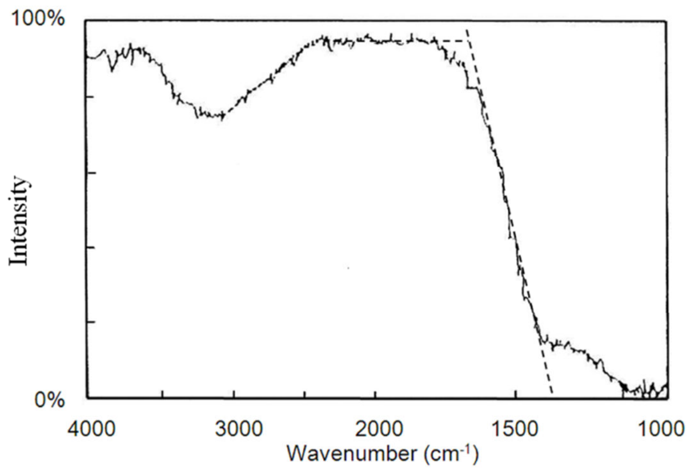

Figure 1: Infra-red spectrophotometer traces of 20TBP70 glass.

Table 1: Measurements of transmission cut-off and absorption peaks positions in infra-red transmission curve for TiO_2-Bi_2O_3-PbO (TBP) glasses.

No.	Sample	Compositions, mole%	OH absorption peak, cm^{-1}	Transmission cut-off, cm^{-1}	Absorption peak, cm^{-1}
1	5TBP60	$5TiO_2$-$35Bi_2O_3$-60PbO	3108	1401	1345
2	5TBP70	$5TiO_2$-$25Bi_2O_3$-70PbO	3108	1451	1370
3	10TBP50	$10TiO_2$-$40Bi_2O_3$-50PbO	3129	1362	1303
4	10TBP60	$10TiO_2$-$30Bi_2O_3$-60PbO	3143	1412	-
5	10TBP70	$10TiO_2$-$20Bi_2O_3$-70PbO	-	1407	1325
6	15TBP55	$15TiO_2$-$30Bi_2O_3$-55PbO	3129	1371	1229
7	20TBP70	$20TiO_2$-$10Bi_2O_3$-70PbO	3129	1263	1143

The relatively shallow peak 1 observed in Figure. 1, corresponds to the -OH absorption band. It is ascribed to the presence of low water entrapped impurity, existing in the glass samples. In the plot of -OH absorption peak against TiO_2 content, (Figure 2), the shift and depth of -OH peaks of different compositions are not appreciable, yet it has apparently depicted a maximum around 10TBP50 and 15TBP55 glasses in which the oxygen concentrations in the glass network are the most. Correspondingly, the absorption resonances tend to lower frequencies (wavenumbers) TiO_2 concentration. The second peak, which is composed of two overtone absorptions, may correspond to TiO_4 and TiO_6 [26], where the latter attributes to the lower frequency peak, i.e. in the region of 1100-1400 cm^{-1}.

In Figure 3 with the addition of TiO_2, the major absorption peak, corresponding to TiO_6, shifts to lower frequencies. This may be attributed to the increase of structural distortion effects on Ti-O, which can be imposed by the presence of large modifying cations, such as; Pb^{2+} and Bi^{3+}. The appearance of a relatively steep cut-off edge with a weak shoulder, and its shift to lower frequencies is noteworthy, [27, 28], (Table 1). The trace shape of the transmission cut-off shoulder may suggest an impurity at low levels. Other possibility is that it can be an overtone band corresponding to Al_2O_3 impurities from the little dissolutions of the alumina crucibles, during melting process in glass sample preparation [29].

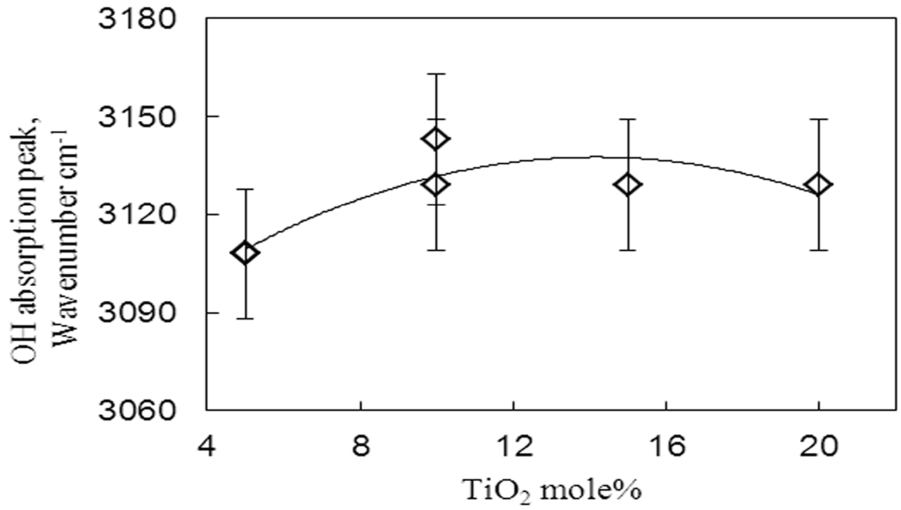

Figure 2: Effect of TiO_2 content on the absorption OH peak shift concerning to the water impurity.

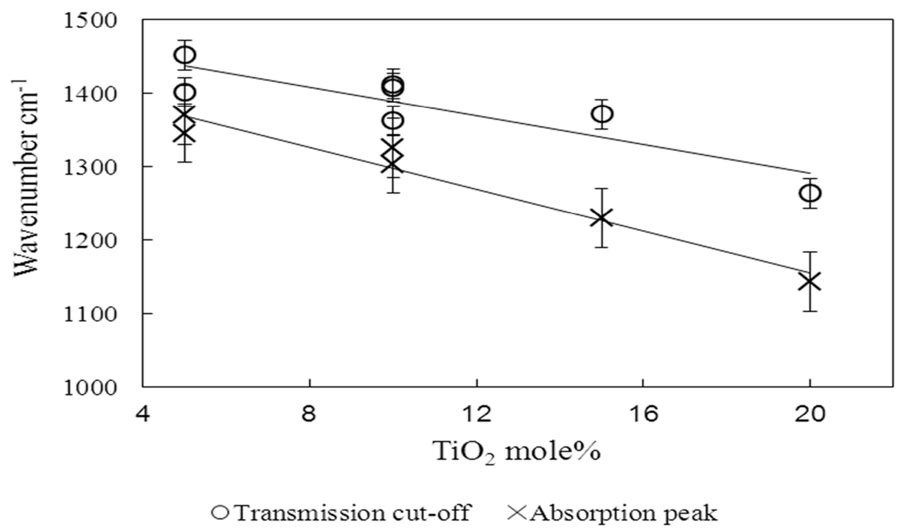

Figure 3: Transmission cut-off and absorption peak variation with TiO_2 content in TBP glasses.

In the plot of transmission cut-off and absorption peak wavenumber against the TiO_2 content in mole percent, (Figure 3), the linear descending variations observed in the both curves, may be related to the replacement of Pb and Bi as large atoms with smaller Ti atom in the glass structure. The second absorption peak [26, 30], in correspondence with Pb-O or Bi-O bonds, tends to shift to lower wavenumbers (i.e., low photon energy state), because of the occurrence of structural distortion in the glass network. Moreover, the introduction of higher amount of strong Ti-O bond could induce more distortion in the neighboring heavy oxides, because the overall oxygen population in the glass network increases. Consequently, both coordination number and non-bridging oxygen are expected to rise. In addition, the widening differences observed between the transmission cut-off and the absorption peak wavenumbers suggest broadening of the absorption curve, which endorses the above explanations.

The stable TBP glass samples in Table 1, were suitable enough to execute the ultra-violet/visible (UV/Vis) spectroscopy investigations [22, 26]. In order to study variation of absorption coefficient (attenuation) concerning the TBP glass system, the measured attenuations, η, are conventionally presented in decibel per kilometer, i.e., $10\log_{10}(I_o/I)/t$ dB/km in correlation with all-optical fiber and the related device applications [2, 3]. The plot of attenuation versus inverse of wavelength, 1/λ, as the proportionate to the incident light photon energy, i.e.E, (Figure 4), does not exhibit quite linear curves for the stable TBP glasses, as it is expected by the Urbach empirical equation [17, 18] particularly in lower energy levels.

Results from Figure 4 suggest that the linear least square regression values, R^2 [31], in the curves decreases with the increase of the polarizable ions constituted in the glass structure. Figure 5 indicates that higher linear curves are expected from glasses with more TiO_2 content, though it introduces more oxygen into the glass structure. Therefore, the real attenuations in low and high energy levels are in fact higher than that the Urbach empirical equation would predict. Generally, presence of the large atoms such as; Pb^{2+} and Bi^{3+} in glass network, causes the structural distortion, and thus polarizability and so refractivity of the glass increases. Hence, glass refractive index rises and the related absorption edge tends to higher wavelengths in which the optical non-linearity becomes eminent [32, 33].

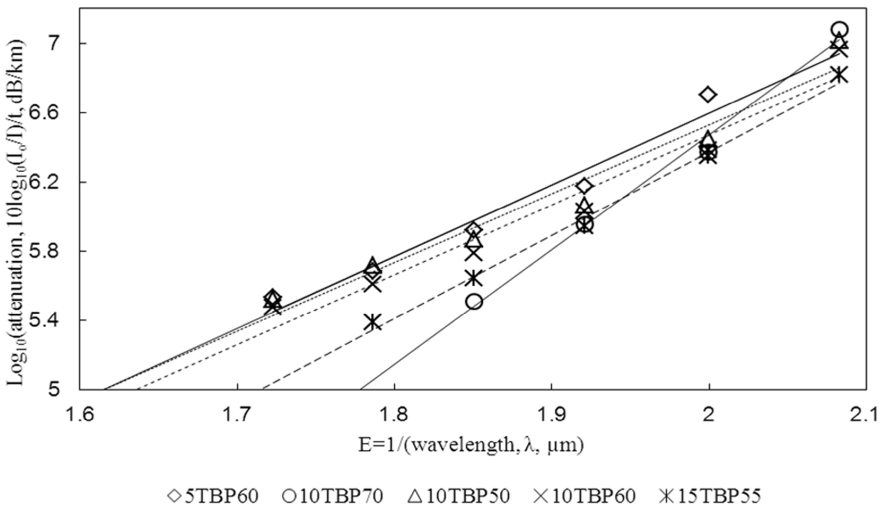

Figure 4: Plot of attenuation against photonic energy in TBP glasses.

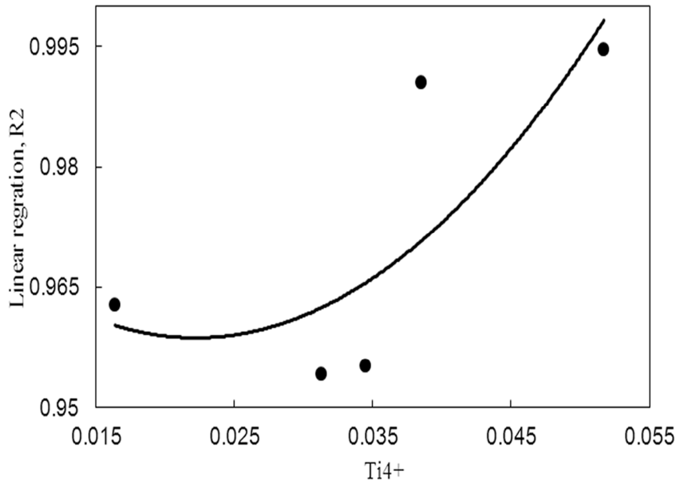

Figure 5: Increase of linearity in the curve deduced from the Urbach empirical equation with Ti^{4+} in the TBP glasses.

Therefore, influence of large cations; Bi^{3+} and Pb^{2+}, constituting TBP glasses, would shift the light absorption to lower energy levels by distorting the glass network, [34, 35]. There are many other factors could also enhance the above curves` non-linearity, which could be pertained to: `the samples thicknesses and their uniformity`, `poor sample surface smoothness`, glass inhomogeneity`, and `multiple reflections or light scattering due to the presence of different inclusions [36]. Attempts have been made to minimize the above effects by controlled melting period and stirring of the molten samples. Importantly, the preparations of moldings by consecutive grounding and polishing are vital, in order to obtain relatively uniform thin slabs (of about 1mm thickness). Moreover, in order to relief any possible stress relaxation, glass annealing process may be applied. Lastly, to minimize glass inhomogeneity and internal reflections effects, only narrow incident light is allowed to pass through the samples by covering the samples with aluminum foil pieces having a slit in the middle.

Besides to, the above analogies concerning reasons for the non-linearity of curves in Figure 4, the effects of ultra-refractive index and dispersion properties of TBP glass samples in promoting interfacial reflection have been considered, (refer to the introduction). Rough estimations of absorption coefficient (attenuation) at long wavelengths have been measured by curves fitting to $1/\lambda \rightarrow 0$, (Table 2). In the introduction section, constant, A and attenuation, η, are estimated as λ in the equation (3) tends to infinity, i.e., E=0, and ΔE can be measured from the extrapolation of the curves in Figure 4. In order to obtain the photon energy edge constant Eg, which is required in absorption measurements, Tauc plot method was executed [19], (Figure 6 and Table 2).

Table 2: Prediction of attenuation tails at the minimum photon energy (as the wavelength approaches infinity) by uv/visible absorption traces and executing of Tauc plot method for TBP glasses.

Sample	Property energy constant, $\Delta E(=1/\lambda)\times10^4$ cm^{-1}	Photon energy gap or edge $E_g \times10^4$ cm^{-1}	Attenuation, as $\lambda\rightarrow\infty$ $\eta[=10\log_{10}(I_o/I)/t]\times10^{-2}$ dB/km	Wavelength, λ, at attenuation, $\eta=10^5$ dB/km, Nm
5TBP60	0.1048	1.758	2.0479	539.0
10TBP50	0.1094	1.928	3.8388	535.5
10TBP60	0.1075	1.807	2.4734	531.0
10TBP70	0.0656	1.896	$1.6904\text{x}10^{-5}$	518.4
15TBP55	0.0903	1.823	$5.6663\text{x}10^{-2}$	520.3

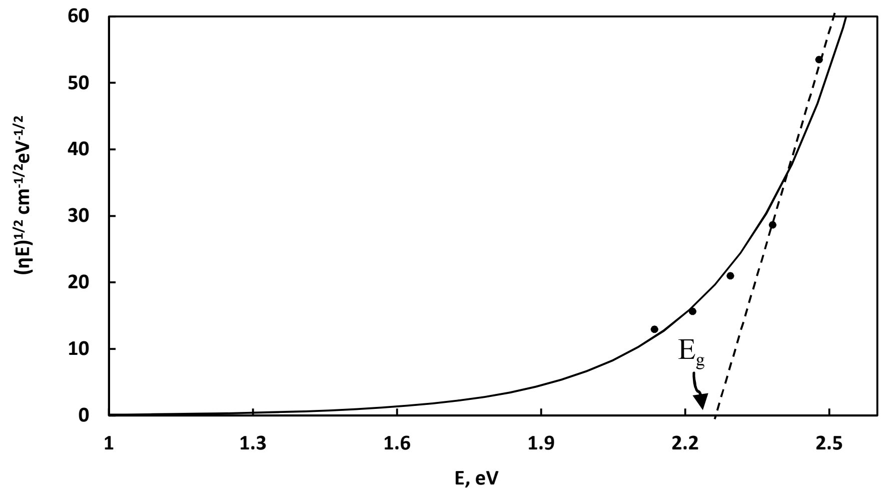

Figure 6: Estimating photon energy edge constant, E_g, for TBP glasses by employing Tauc plot (method).

In Table 2, results which are corresponded to different wavelength, λ, are presented at the particular attenuation, i.e., $10\log_{10}(I_o/I)/x=10^5$ dB/km (number of decibels per length), in relation to the interest of absorption region concerning the all-optical communication devices, [37].

In an overall view, in an analogy concerning effects of the above heavy oxides and TiO_2 on the TBP glass system, the results indicate that on the contrary to Bi_2O_3, as the PbO content relatively increases in total, the attenuations of TBP glasses becomes lower at shorter wavelengths, i.e., higher energy level, (Figure 7). The low smoothness observed in the resulting plots are due to the existing differences in PbO and Bi_2O_3 polarizations and their inconsistence substitutions in the glass formula, since, limited stable glass sample were available.

In Table 2 and Figure 4, 10TBP70 glass in comparison with 10TBP60 and 10TBP50, exhibits a steeper curve, which suggests the appreciable attenuation in relatively shorter wavelengths in the visible regions around 550 nm. Correspondingly, in longer wavelengths i.e. lower photon energies, E, of the curve, the attenuation tends to lowest. Thus, 10TBP70 glass with respect to its relative ultra-index of refraction at Na spectrum (n_D~2.4) and high dispersion properties (Abbe Number~10), [32], can be a considerable candidate for non-linear optical applications, e.g., all-optical switching devices, [22]. These observations, in Figure 7, could be in contradictory to the effects of Pb^{2+}, which claims larger cation radius and higher refractivity in the oxide form, in comparison with the effects of Bi^{3+}, [32, 38]. However, it may be explained that in substituting Bi_2O_3 for PbO, the population of a strong polarizing anion such as, O^{2-}, which generally improves optical properties, reduces. On the other hand, addition of oxygen in the glass structure increases the cations coordination numbers and so, the population of non-bridging oxygen. The both mentioned phenomena are demanded to stimulate the overall optical properties, [39, 40]. Therefore, the absorption band is expected to move to longer wavelengths in uv/visible regions. Moreover, when TiO_2 is added to TBP glasses, along with O^{2-} anions, the population of Ti^{4+}, as a relatively lower polarizable cation, increases in the glass structure too. It can have adverse effects in relation to other cations on the polarizability of the glass structure. Figure 8 depicts the reduction of the attenuation at infinite wavelengths with Ti^{4+} content in the TBP glass.

Thus, it could be concluded that in correspondence with the increase of TiO_2 in the glass composition, oxygen ions are increased in the network structure and subsequently, the reduction of attenuation, in longer wavelengths in the uv/visible region, is expected. In Figure 9, the curve deduced from the plot of the attenuation against the ratio of oxygen to cations, endorses this, i.e., oxygen as a strong polarizer normally increases the attenuation.

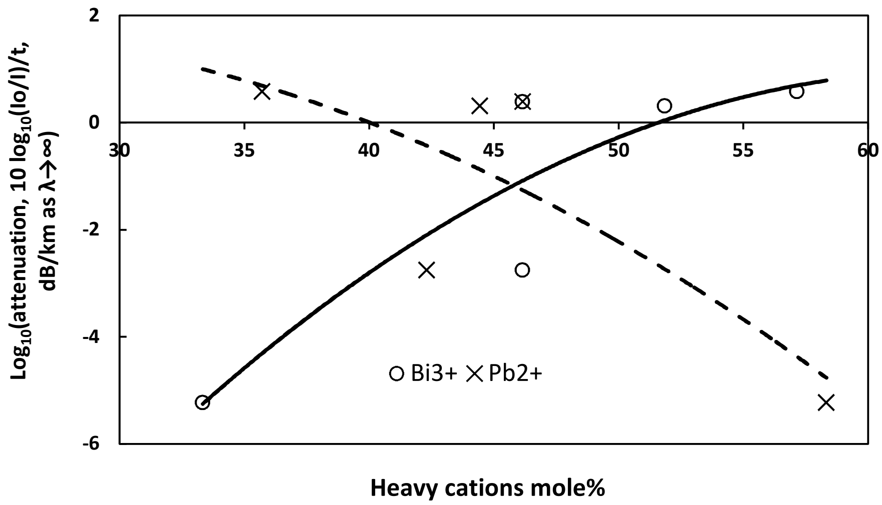

Figure 7: Effects of Bi^{3+} ions on the attenuation in long waves in comparison with Pb^{2+}ions in TBP glasses.

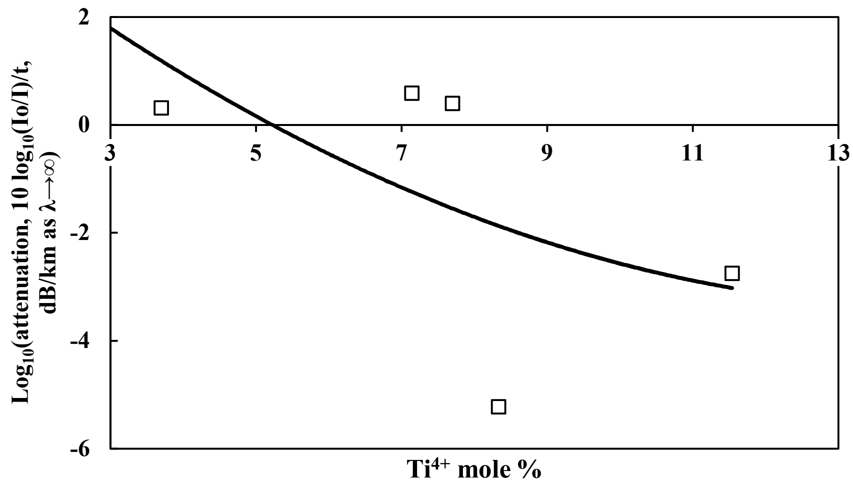

Figure 8: Overall decrease of attenuation in long wavelength with the Ti^{4+} content in TBP glasses.

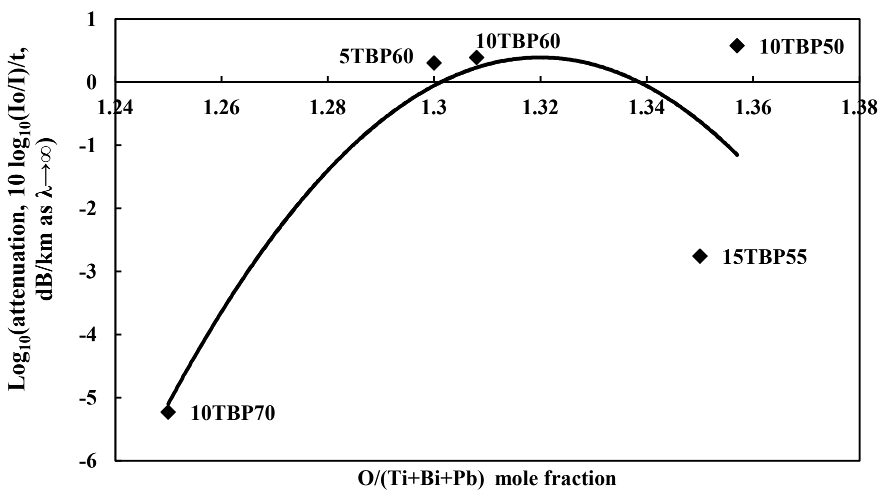

Figure 9: Increase of attenuation to a maximum in long wavelengths as oxygen ion fraction rises in the TBP glass structure.

However, the curve in Figure 9, suggets that the attenuation in long wavelength increases with oxygen fraction in the glass network, provided the relative amount of polarizable ions, such as, Bi^{3+} and $Pb^{2+}$are not reduced in total. The observed maximum in the corresponding curve, due to the presence of sample 15TBP55, in comparison with 10TBP60 and 10TBP50, may explain the compensating effect of Ti^{4+}, which is substituting the highly polarizable cations. Therefore, the attenuation declines or shifts to shorter wavelengths (higher energy levels).

In order to investigate the effective absorbed energy levels in related optical devices, the inverse of wavelengths at a particular attenuation in correspondence with the interest of the communication devices (all-optical switches), i.e., 10^5 dB/km, are plotted against the content of polarizable oxides in the glass composition, (Figures 10, 11 and 12), [37]. The results indicate that at a particular attenuation of the interest, in the presence of relatively strong polarizable ions, addition of Ti^{4+} to the TBP glass network increases the required energy level, (Figure 10). Similarly, in Figures 11 and 12, Pb^{2+} tends to increase the required energy level, when comparing with the higher polarizable Bi^{3+} and O^{2-} ions. In Figure 12, the observed minimum region in the curve is attributed to the increase of O^{2-} in the glass structure along with Ti^{4+}, which has the lowest polarizability in comparison with the other constituents in TBP glasses [22, 32].

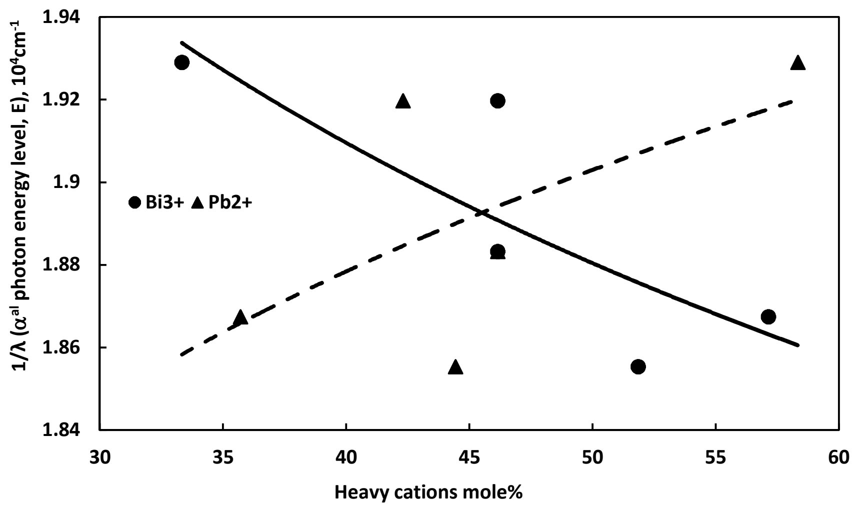

Figure 10: The effect of heavy cations on photon energy level at particular attenuation, 10^5dB/km in the TBP glass system.

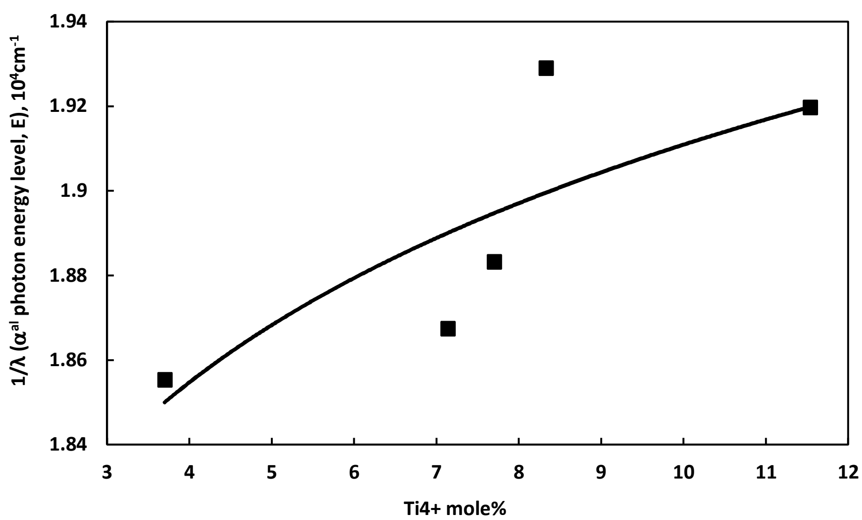

Figure 11: The effect of Ti^{4+} cation on photonic energy level at particular attenuation, 10^5dB/km in the TBP glass system.

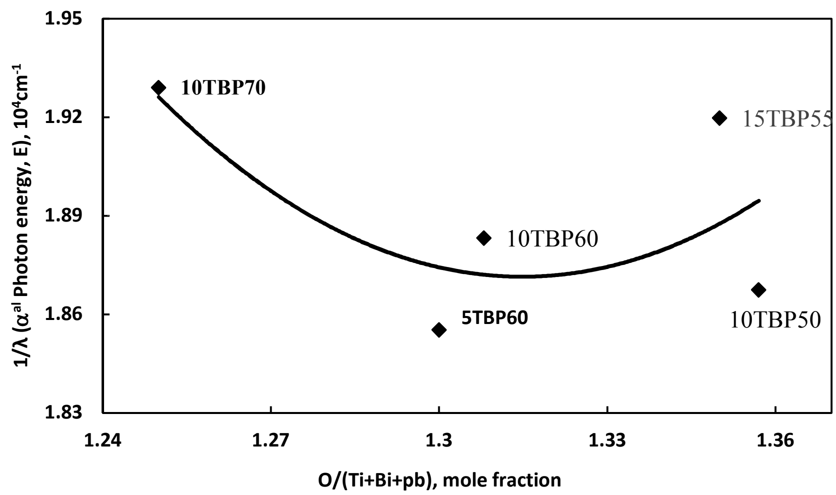

Figure 12: The effect of heavy cations on photonic energy level at particular attenuation, 10^5dB/km in the TBP glass system.

It should bear in mind that the observed scattered data`s in the above plots were partly due to the low consistancy in the stable glass formula, which were available.

4. Conclusions

The stable TBP glasses with the limited preparation, exhibited transmission between 0.55 to 7 µm, with absorption tails extended to below 0.55 µm. Steeper and linear absorption tails, in respect to the Urbach empirical equation, were observed in TBP glasses, which comprised relatively higher fraction of Ti^{4+} cation in their compositions. Thus, increase of TiO_2 content in the TBP glasses widened the transmission window. In highly polarizable TBP glasses, the real attenuations, in very low and high energy levels, were higher than those predicted by the Urbach empirical equation, for which non-linear curves were observed. From the photon energy level point of view in all-optical devices, concerning non-linear optics, e.g. switching, addition of Ti^{4+} increases the energy level in correspondence with attenuation in the uv/visible region, whereas, effects of O^{2-} on the energy level, despite its relative high polarity, was depended on the polarizability of the cation in the corresponding oxide, e.g. TiO_2. In the substitution of Bi_2O_3 with PbO, in TBP glasses, when the population of both highly polarizable ions; O^{2-} and Bi^{3+} declined comparatively in total, the addition of PbO in relation to Bi_2O_3 had similar effects as TiO_2 on the glass. From optimal point of view concerning low attenuation or low absorption in high energy states, sample 10TBP70 in comparison with other stable samples in TBP glasses, claimed the lowest structural polarisability, because in all, it comprised relatively lower O^{2-} and Bi^{3+}. Consequently, it would relatively acquire lower optical properties, which could be a drawback to applications concerning optical non-linearity. Lastly, the investigations may be extended by introducing suitable modifying oxides, while maintaining the desired stable ultra-optical properties. .

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