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# Study of structure and optical properties of $Fe_2O_3{\cdot}CaO{\cdot}Bi_2O_3$ glasses

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ABSTRACT

Glasses with compositions $0.05Fe_2O_3{\cdot}0.95\{xCaO{\cdot}(100-x)Bi_2O_3\}$ ($20 \le x \le 40$ mol.%) have been prepared using the normal melt quench technique. The density and molar volume have been determined. Infrared (IR) spectroscopy is used to investigate the structure of the glass matrix. The optical studies in the UV–VIS–NIR region for all these glasses show a sharp cutoff and a large transmitting window. The values of both of the optical band gap ($E_g$) and width tails ($\Delta E$) are determined. It is observed that $E_g$ is decreased and $\Delta E$ increased with the increase of CaO in the glass matrix. The metallization criterion ($M$), interaction parameter ($A_{th}$), average electronic polarizability of the oxide ion ($\alpha_{O^{2-}}$) and optical basicity ($\Lambda$) of these glasses are determined from the values of optical band gap. Small value of $M$ makes them appealing candidates for non-linear optical materials. Both $\alpha_{O^{2-}}$ and $\Lambda$ increase with increase in CaO content. The compositional dependence of the above properties are discussed and correlated to the structure of the glass.

## 1. Introduction

Unconventional novel oxide glasses formed with the network former $Bi_2O_3$ have recently been attractive materials of research due to their interesting physical properties leading to many applications [1]. It has been reported that the pure bismuthate glasses can be obtained at high cooling rates [2]. These glasses find wide applications in the field of glass ceramics, layers for optoelectronic devices, thermal and mechanical sensors, reflecting windows, etc. [3–9]. Heavy metal oxide (HMO) glasses based on $Bi_2O_3$, PbO and $Ga_2O_3$ are known to be very good candidates for producing highest refractive index among oxide glasses and an excellent infrared (IR) transmission to the longest possible wavelength. As an example, some $Bi_2O_3{\cdot}PbO{\cdot}Ga_2O_3$ glasses and $Bi_2O_3{\cdot}Fe_2O_3{\cdot}CdO{\cdot}PbO$ glasses were reported to have IR transmission down to 8 and 9 μm for samples of thickness 0.2 and 0.6 mm, respectively [10]. The third order non-linear susceptibility, which is a scale of the possible non-linear effects, was found to be high for HMO glasses and increases with the increase in HMO concentration [11]. The unique properties of these glasses arise due to their high density, low bond strength, high polarizability, long infrared cutoff and high non-linear optical susceptibility as compared to the conventional glass formers such as $SiO_2$ and $GeO_2$. However, the high polarizability and low field strength result in a limited glass formation range (GFR) for these glasses [12]. These glasses are also found to be efficient for γ-ray absorbers and also considered for use in scintillation detectors for high energy physics [13]. The attractive reason in studying these glasses is due to the fact that they do not contain any conventional glass formers such as $SiO_2$, $GeO_2$, $P_2O_5$. The aim of the present study is to obtain by means of Infrared spectroscopy specific data regarding the local structure of $Fe_2O_3{\cdot}CaO{\cdot}Bi_2O_3$ glasses as well as to study the changes caused by CaO content on optical and physical properties of these glasses. The interest for investigated glass system is determined by the presence of unconventional glass former, $Bi_2O_3$ that behaves as a network former in the present glass system.

## 2. Experimental details

Glasses with compositions $0.05Fe_2O_3{\cdot}0.95\{xCaO{\cdot}(100-x)Bi_2O_3\}$ with $20 \le x \le 40$ mol.% were prepared using the normal melt quench technique from AR grade chemicals of $Bi_2O_3$, CaO and $Fe_2O_3$. Appropriate amounts of reagents were mixed and melted in a porcelain crucible for 0.5 h at 1150 °C. The samples were obtained by quenching the melt between two stainless steel plates held at room temperature (*RT*). The density (*D*) of all the samples was calculated by Archimedes's method with xylene as a buoyant liquid. The density was calculated according to the formula:

$$D = \frac{W_{air}}{W_{air} - W_{xylene}} \times \rho_{xylene} \quad (1)$$

The molar volume was calculated using the relation $V_M = M_T/D$, where $M_T$ is the total molecular weight.

Infrared transmission spectra were recorded at RT using FTIR spectrometer (NEXUS-670) in the wavelength range of 300–1500 cm$^{-1}$. The powdered samples were thoroughly mixed with dry KBr in the ratio of 1:20 in order to obtain thin pellets.

The samples were polished for optical measurements and then optical absorption spectra of finely polished samples were recorded at RT using Solid-Spec 3700 UV–VIS–NIR Spectrophotometer (Shimadzu) in the wavelength range of 500–900 cm$^{-1}$.

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**Table 1**

Density ($D$), molar volume ($V_M$), cutoff wavelength ($\lambda_{cutoff}$), optical band gap ($E_g$), band tailing parameter ($B$), Urbach energy ($\Delta E$), metalization criterion ($M$), interaction parameter ($A_{th}$), oxide ion polarizability ($\alpha_{O^{2-}}(E_g)$) and optical basicity ($\Lambda$) in $0.05Fe_2O_3{\cdot}0.95\{xCaO{\cdot}(100-x)Bi_2O_3\}$ glass system.

| Glass no. | $x$ (mol.%) | $D$ (gm/cm$^3$) | $V_M$ (cm$^3$/mol) | $\lambda_{cutoff}$ (nm) | $E_g$ (eV) | $B$ (cm eV)$^{-1/2}$ | $E_g$ (eV) | $B$ (cm$^{-1/3}$ eV$^{-2/3}$) | $\Delta E$ (eV) | $M$ | $A_{th}$ | $\alpha_{O^{2-}}(E_g)$ (Å$^3$) | $\Lambda$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | $n = 2$ | | $n = 3$ | | | | | | |
| FCB25 | 25 | 7.10 | 49.79 | 500 | 1.96 | 6.60 | 1.75 | 3.11 | 0.22 | 0.312 | 0.015 | 4.173 | 1.260 |
| FCB30 | 30 | 6.91 | 48.29 | 522 | 1.79 | 4.57 | 1.55 | 2.33 | 0.25 | 0.299 | 0.017 | 4.692 | 1.314 |
| FCB35 | 35 | 6.57 | 47.85 | 533 | 1.76 | 4.35 | 1.52 | 2.23 | 0.26 | 0.297 | 0.018 | 4.957 | 1.330 |
| FCB40 | 40 | 6.47 | 45.50 | 543 | 1.63 | 3.69 | 1.36 | 1.93 | 0.31 | 0.285 | 0.020 | 5.260 | 1.350 |

Metallization criterion, interaction parameter, average electronic oxide polarizability and optical basicity were also calculated.

## 3. Results and discussion

### 3.1. Density and molar volume

The density is a powerful tool, capable of exploring the changes in the structure of glasses, and is affected by structural softening/compactness, change in geometrical configurations, coordination numbers, cross link densities and dimensions of interstitial spaces of the glass. Thus decrease of a heavy metal ion leads to a decrease in the density that has a linear dependence on the composition. It is remarkable to note (Table 1) that in the present glass samples density decreases linearly from 7.10 to 6.47 gm/cm$^3$, congruent with a linear decrease in the molar volume from 49.79 to 45.50 cm$^3$/mol as the alkaline earth modifier is added at the expense of $Bi_2O_3$ content. The decrease in molar volume can be explained that molar volume depends on both, i.e. density and molecular weight and in the present glass system both decreases with the increase in CaO content at the expense of $Bi_2O_3$ content.

### 3.2. FTIR spectra

The infrared spectra of glassy samples have been investigated to obtain the information on the structure and arrangement of building structural groups with respect to each other and type of bonds present in the glass. Fig. 1 shows the FTIR spectra of $Fe_2O_3{\cdot}CaO{\cdot}Bi_2O_3$ glasses. In the figure two types of bands can be seen in the wavenumber regions 400–500 and 800–1000 cm$^{-1}$. The intensity of these bands varies with the addition of CaO content, which may be due to change in the disorder of the glass network. These results are important in order to recognize the ionic transport phenomena in the glass samples, which is related to the structure of the glass samples. The IR spectra (Fig. 1) are dominated by the bands associated to the structural vibrations of the highest cation, $Bi^{3+}$. The dominating bands around 900 and 485 cm$^{-1}$ are an evidence for existence of [$BiO_3$] pyramidal and [$BiO_6$] octahedral units in the entire composition range. The broadening of band (near 485 cm$^{-1}$) for $x = 20$ mol.% is a strong evidence of [$BiO_6$] distorted polyhedral. The IR bands around 900 cm$^{-1}$ represent the convolution of the absorption bands reported for different bismuthate glasses at 840 and 860 cm$^{-1}$ assigned to the total symmetric stretching vibrations of the [$BiO_3$] and [$BiO_6$] polyhedral units, respectively [14,15].

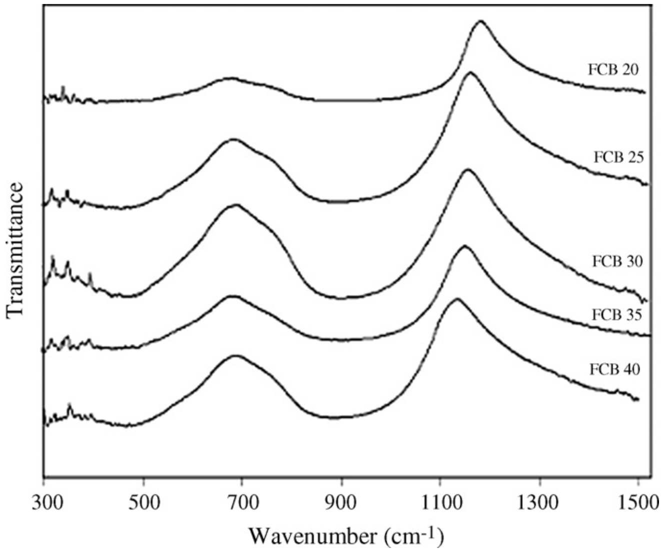

**Fig. 1.** Infrared transmission spectra for $Fe_2O_3{\cdot}CaO{\cdot}Bi_2O_3$ glasses.

### 3.3. Optical properties

#### 3.3.1. Optical band gap

Fig. 2 shows the optical absorption spectrum of the glasses under study. The measurement of optical absorption and the absorption edge is important especially in connection with the theory of electronic structure of amorphous materials. The non-sharp edges in the optical absorption spectra (Fig. 2) of the present glass sys-

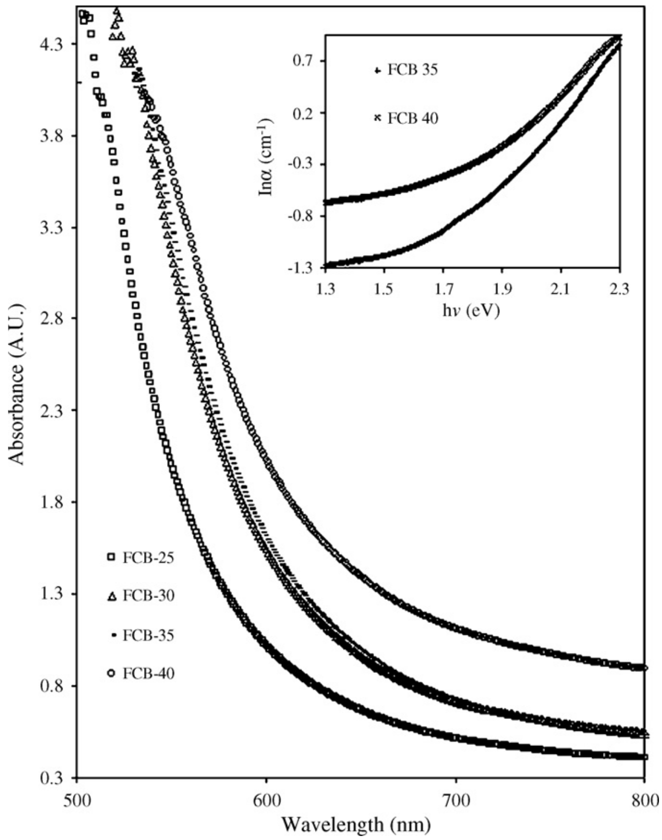

**Fig. 2.** Optical absorption spectra for $Fe_2O_3{\cdot}CaO{\cdot}Bi_2O_3$ glasses. Insert: Urbach's plot for $Fe_2O_3{\cdot}CaO{\cdot}Bi_2O_3$ glasses.

tem are clear indication of amorphous nature of the samples. Also Fig. 2 shows that absorption has been increased in the visible region of spectrum as CaO content is increased. The cutoff wavelength ($\lambda_{\text{cutoff}}$) also shifts towards red as CaO content increases in $Fe_2O_3{\cdot}CaO{\cdot}Bi_2O_3$ glasses. This shift may be due to the increase in non-bridging oxygen ions which binds an excited electron less tightly than a bridging oxygen in the present glass system. The optical absorption method being the most direct and simplest method was used to determine the optical band gap of the samples. The fundamental absorption refers to band to band transitions and it manifests itself by a rapid rising in the absorption used to determine the optical band gap. The absorption coefficient, $\alpha(\nu)$, below and near the edge of each curve was determined, using the relation [16]:

$$\alpha(\nu) = \left(\frac{1}{d}\right) \ln\left(\frac{I_0}{I_t}\right) \tag{2}$$

where $d$ is the thickness of each sample and $\ln(I_0/I_t)$ corresponds to absorbance, '$A$'. The absorption edge as observed in the UV region (Fig. 2) can be divided into two regions, depending upon the value of the absorption coefficient, $\alpha$, for many amorphous materials. The first region, usually known as Urbach tail [17], which is characterised with $\alpha < 10^4\,\text{cm}^{-1}$ and depends exponentially on the photon energy ($h\nu$) as

$$\alpha(\nu) = \alpha_0 \exp\left(\frac{h\nu}{\Delta E}\right) \tag{3}$$

where $\alpha(\nu)$ is constant and $\Delta E$ is the width of band tail energy.

In the second region, where $10^4 \le \alpha \le 10^6\,\text{cm}^{-1}$, the following relation is obeyed [16,18]:

$$\alpha h\nu = B(h\nu - E_g)^n \tag{4}$$

where $B$ is a constant called band tailing parameter, $E_g$ is the optical band gap energy and n is the index, which takes different values depending on the mechanism of interband transitions. $n = 2, 3, 1/2, 1/3$ corresponds to indirect allowed, indirect forbidden, direct allowed and direct forbidden transitions respectively. In amorphous materials, at higher levels of $\alpha$ ($\ge 10^4\,\text{cm}^{-1}$) the absorption edge is usually interpreted in terms of indirect transitions across an optical gap [18]. The variation of $(\alpha h\nu)^{1/2}$ vs. $h\nu$ (Tauc's plot) is shown in Fig. 3 for FCB35 and FCB40 glasses. The value of $E_g$ was determined from the above relation (Eq. (4)) by extrapolating the linear region of the curves for $(\alpha h\nu)^{1/2} = 0$ as shown in Fig. 3. The values of $E_g$ thus obtained for all the glass samples are listed in Table 1. The same data was fitted in Eq. (4) for $n = 3$ (inset of Fig. 3). The calculated values of $E_g$ (for $n = 3$) for all the glass samples are also listed in Table 1. The optical band gap in amorphous materials is closely related to the energy gap between the valence band and the conduction band. The variation in the values of $E_g$ on increasing the CaO content can be understood in terms of structural changes that are taking place in the studied glass system. It is remarkable to note from Table 1 that $E_g$ becomes smaller with increasing amount of CaO content which also supports the increase in NBO's. The observed blue shift in the optical band gap can be explained on the basis of variation in non-bridging oxygen ion concentration. In metal oxides (M–O), the valence band maximum (VBM) mainly consists of O(2p) orbital and the conduction band minimum (CBM) consists of M(nS) orbital. When a metal oxygen bond is broken, the bond energy is released and the nonbonding orbitals have higher energies than the bonding orbitals [19]. This might have introduced certain energy states in forbidden gap causing the decrease in optical band gap with increasing proportion of CaO. The width of the band tails (Urbach's Energy, $\Delta E$) associated with valence band and conduction bands was believed to be originated from the same physical origin. This origin is attributed to phonon-assisted indirect electronic transitions between localized states, where the density of these states is exponentially dependent on energy [20]. The Urbach's energy arises from the random potential fluctuations in the material into the band gap [21,22]. The Urbach plot for FCB35 and FCB40 glass sample is shown in the inset of Fig. 2. The values of Urbach's energy ($\Delta E$) were calculated by determining slopes of linear regions of the curves and are presented in Table 1 for all the glass samples. It is remarkable to note from Table 1 that $\Delta E$ behaves inversely to that of $E_g$. It can be explained on the basis that Urbach's energy was generally used to characterize the degree of disorder in amorphous and crystalline solids. Materials with larger values of $\Delta E$ would have a great tendency to convert weak bonds into defects. Therefore, the value of Urbach's energy is considered as a measure of defects concentration. Referring to Table 1 the obtained increase in the value of $\Delta E$ with increasing CaO content suggests that there is a tendency for an increase in the concentration of defects in the studied glasses.

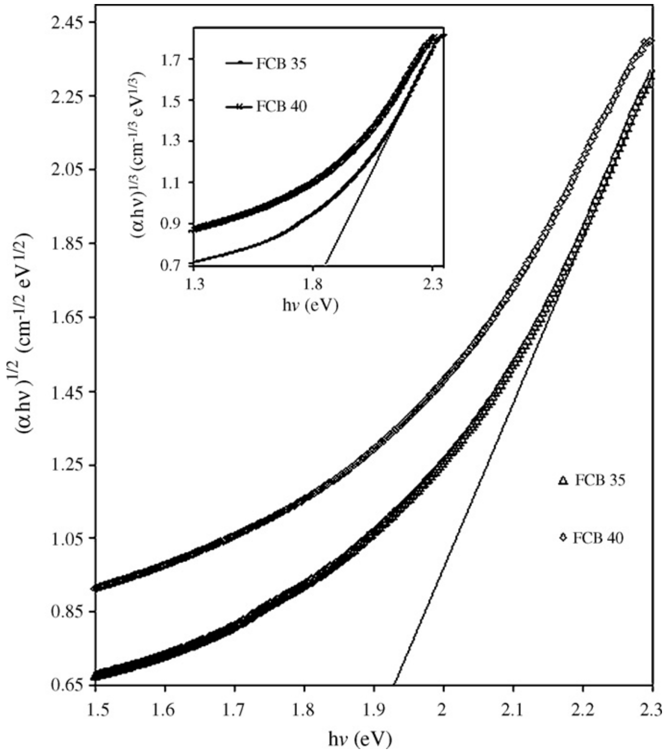

**Fig. 3.** Tauc's plots for FCB35 and FCB40 glasses ($n = 2$). Insert: Tauc's plots for $Fe_2O_3{\cdot}CaO{\cdot}Bi_2O_3$ glasses ($n = 3$).

### 3.3.2. *Tendency for metallization of the glasses*

A photon with a certain range of energy can be absorbed by modifying ions in an oxide glass via either of two processes: (i) internal transitions between the $d$-shell electrons or (ii) transfer of an electron from a neighbouring atom to the modifier ion and vice versa. The absorption edge occurs at $h\nu = E_g$, the energy gap. Duffy [23] has suggested that a good correlation exists between the energy gap of oxides and their molar refraction, as follows:

$$E_g = 20\left(1 - \frac{R_m}{V_m}\right)^2 \tag{5}$$

Metallization criterion of an oxide can be calculated using the relation:

$$M = \sqrt{\frac{E_g}{20}} = \left(1 - \frac{R_m}{V_m}\right) \tag{6}$$

For the condition $R_m/V_m = 1$ in a Lorenz–Lorenz equation ($R_m$, molar refraction; $V_m$, molar volume), the linear refractive index becomes infinite, which corresponds to the metallization of covalent solid materials. Therefore, the necessary and sufficient conditions for predicting the non-metallic or metallic character of solids are: $R_m/V_m < 1$ (non-metal) and $R_m/V_m \geq 1$ (metal). The Values of $M$ ($E_g$) are presented in Table 1 and ranges from 0.312 to 0.285. The increasing incorporation of CaO is mainly responsible for the $E_g$ diminishing. The lowest $E_g$ value (1.63 eV for FCB40), possess the lowest metallization criterion value (0.285). The small metallization criterion mean that the width of both valence and conduction bands become large, resulting in a narrow band gap (1.63 eV). The high non-linear optical susceptibly of HMO glasses as reported in literature [24] can also be explained on the basis of theory for metal–non-metal transitions in condensed matter proposed by Herzfeld [25–28]. The glasses containing large amount of $Bi_2O_3$ having large amount of third order non-linear susceptibility ($\chi^3$) possess a small metallization criterion in comparison to the glasses containing large amount of conventional glass formers. Hence these glasses are a good basis of non-linear optical materials.

### *3.3.3. Interaction parameter of glasses*

Interaction parameter $A$ is used to describe the polarizability state of an average oxide ion in numerous simple oxides and its ability to form an ionic covalent bond with the cation. The interaction parameter gives good information about the electronic polarizability of oxide glasses as well as optical basicity. The larger are the values of oxide ion polarizability and optical basicity, the smaller is the interaction parameter. We calculated the theoretical interaction parameter ($A_{th}$) for iron calcium bismuthate glasses on the basis of following equation [29]:

$$A_{th} = X_{Fe_2O_3}A_{Fe_2O_3} + X_{CaO}A_{CaO} + X_{Bi_2O_3}A_{Bi_2O_3} \qquad (7)$$

where $A_{Fe_2O_3}$, $A_{CaO}$ and $A_{Bi_2O_3}$ are the values of the interaction parameters of oxides $Fe_2O_3$, CaO and $Bi_2O_3$, respectively, and have been taken from literature [30]. $X_{Fe_2O_3}$, $X_{CaO}$ and $X_{Bi_2O_3}$ are the equivalent fractions of different oxides, i.e. the proportion of oxide atoms they contribute to the glass system. The calculated values of $A_{th}$ have been presented in Table 1.

### *3.3.4. Oxide ion polarizability ($\alpha_{O^{2-}}$) and optical basicity*

The estimation of average electronic oxide polarizability is particularly valuable with respect to both, i.e. crystals as well as glasses, which are of technological importance as optical and electronic materials. It is an expression of the degree of negative charge borne by the oxygen atoms. The values for average electronic oxide polarizability ($\alpha_{O^{2-}}$) of the glasses are calculated by using the relation [31–33]:

$$\alpha_{O^{2-}}(E_g) - \left[\left(\frac{V_m}{2.52}\right)\left(1 - \frac{(E_g)^{1/2} - 0.98}{1.23}\right) - \sum p\alpha_i\right] q^{-1} \qquad (8)$$

where $p$ and $q$ denote the number of cations and oxide ions, respectively in the chemical formula ApOq. The calculated values of $\alpha_{O^{2-}}$ are given in Table 1. The values of $\alpha_{O^{2-}}$ are greater for the present glass system as compared to the polarizability of conventional glasses reported in literature [24,34]. This is due to the high polarizability and low field strength of $Bi^{3+}$ ions. The addition of modifiers generally leads to an increase in the average oxide ion polarizability. The same trend is obtained in the present glass system (Table 1). The value of $\alpha_{O^{2-}}$ depends on the CaO content and increases with increase in CaO. The addition of iron in bismuthate glasses causes depolymerisation [35] and further addition of CaO content may create more open structure by creating non-bridging oxygens. The NBO's increases the amount of negative charge borne by the oxygen atoms and hence the $\alpha_{O^{2-}}$. Further, optical basicity which is also a measure of the negative charge borne by the oxygen atoms, is related to $\alpha_{O^{2-}}$ [36] as given below:

$$\Lambda = 1.67\left(1 - \frac{1}{\alpha_{O^{2-}}}\right) \qquad (9)$$

This relationship presents a general trend toward an increase in the optical basicity with increase in oxide ion polarizabilty ($\alpha_{O^{2-}}$) or vice versa. The calculated values of $\Lambda$ are given in Table 1 and are found to increase with increase in CaO content.

## 4. Conclusions

The density as well as molar volume of the iron calcium bismuthate glasses decreases with the increase in CaO content in the present glasses. The FTIR spectra of the glasses show that $Bi^{3+}$ cations are incorporated in the glass network as [$BiO_3$] pyramidal and [$BiO_6$] octahedral units. The band below 500 $cm^{-1}$ is due to vibrations of Bi–O bonds in the [$BiO_6$] polyhedral while the broad band (800–1000 $cm^{-1}$) represents the convolution of the absorption bands reported for different bismuthate glasses at 840 and 860 $cm^{-1}$ assigned to the total symmetric stretching vibrations of the [$BiO_3$] and [$BiO_6$] polyhedral, respectively. The decrease in $E_g$ and shifting of cutoff wavelength towards red in UV–VIS spectra with increase in CaO also supports the increase in NBO's in the present glasses. The values of $\Delta E$ suggest that the glasses with higher CaO content have greater tendency to convert weak bonds into defects. Small metallization criterion for these glasses makes them suitable candidates for non-linear optical materials. The decrease in optical band gap and interaction parameter ($A_{th}$) supports the increase in polarizability and hence the optical basicity of the glasses, which confirms the increase in concentration of non-bridging oxygens with increase in CaO content.

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