Original Article

Spectroscopic studies of ZnO borate–tellurite glass doped with Eu$_2$O$_3$

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ABSTRACT

Optical, physical and thermal properties of glasses with composition 24B$_2$O$_3$ – (25 – x) ZnO – 51TeO$_3$, x Eu$_2$O$_3$, mol% (where x = 0.0, 0.5, 1 and 3%) were studied. The density, crystallization temperature, oxygen packing density and glassy temperature were found to increase with the addition of Eu$_2$O$_3$. The molar volume, optical band gap (E$_g$) and refractive index values were found to decrease with increasing Eu$_2$O$_3$ content. The FTIR measurements indicated that the continuous increase of europium concentration helped conversion of [BO$_3$] to [BO$_4$] groups in the base glass ZnO TeO$_3$-B$_2$O$_3$. Thermal analysis reveals that the glass transition temperature $T_g$, the glass thermal stability $\Delta T$ and the glass forming ability $K_3$ increased by the addition of Eu$_2$O$_3$. The transmission and absorption spectra of all glasses indicated that there is a decrease in the optical band gap $E_g$ values due to the enhancement of the number of non-bridging oxygen (NBO) atoms with the addition of Eu$_2$O$_3$. The studied glasses have high values of nonlinear refractive index $n_2$ and nonlinear optical susceptibility $\chi^{(3)}$ with a good thermal stability. Thus, such glasses have promised applications in nonlinear optical devices.

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1. Introduction

Glasses with tellurite offer distinct properties when compared with other glasses, such as good chemical durability and chemical resistance, high linear and non-linear refractive index, high transmittance especially in near infrared (NIR) to middle infrared (MIR) regions and high electrical conductivity [1,2]. They also are suitable candidates for use in fiber optics, optical amplifiers, and laser, in addition to being widely used as photonic crystal fibers (PCFs); therefore, these glasses are considered as potential nonlinear materials [3–5]. Extensive studies were performed on B$_2$O$_3$ glasses for the past few decades because of their promised properties, which can be summarized as follows: 1 – glasses with low melting point, which can help save energy, 2 – high transparency, which is useful in optics, 3 – glasses with high thermal stability, 4 – easy to prepare and can dissolve a high concentration of rare
earth ions, which means that these glasses are more suitable for optical device fabrication [6,7]. Formation of glass with two glass formers was known previously and is of scientific interest. The addition of TeO₂ into the borate network, which means the formation of glass with two glass formers, improves the transparency of the glass and its refractive index. Boro-tellurite glasses containing ZnO have a wide glass forming possibility and low ability to crystallize [8-10]. Doped glasses with rare earth ions have superior technological applications such as in fiber amplifiers, planar waveguides and display monitors of wavelength-converting devices [11-14]. It is known that europium is an element with optical active properties. The optical properties of trivalent europium ions are very sensitive to the surrounded atoms inside the glass, i.e., these properties are dependent on the glass composition. The selection of Eu³⁺ ions to add to the glass matrix is useful for the study of disordered materials, because europium has an energy level with simple structure and non-degenerate ground ⁵⁷⁷⁰ and emitting ⁷⁵⁰ states [15,16]. We think that there is insufficient information regarding the presence of Eu³⁺ in the glasses of borate–tellurite composition. Therefore, it is very interesting to discover the relation between the change in the glass structural units and the linear and nonlinear properties of Eu³⁺ ions in borate–tellurite glasses.

2. Experimental

2.1. Glass preparation

In the present study, glasses having the composition 24B₂O₃–(25–x) ZnO–51TeO₂, x Eu₂O₃, mol% (where x = 0.0, 0.5, 1 and 3%) were prepared using pure reagent grade H₂BO₃, ZnO, TeO₂ and Eu₂O₃ as starting materials. For each composition, the raw materials in the powder form were weighed accurately; then, they were mixed together using agate mortar and were melted in alumina crucibles at about 800 °C for 30 min in air. The glass melts were stirred occasionally with an alumina rod to achieve good homogeneity. The highly viscous melt was cast into a cylindrically shaped split mold of mild steel, and the produced glass was annealed at 450 °C in another furnace for 1 h, after which the furnace was switched off and the glass was allowed to cool gradually in situ for 24 h.

2.2. Density

The densities of these glasses (ρ) were determined by using Automatic Gas Pycnometers for True Density, Ultrapyc 1200e, and apparatus with helium gas.

2.3. FTIR

FTIR spectra of the glass samples were recorded at room temperature in the range of 400–4000 cm⁻¹ using Shimadzu FTIR 8400S spectrophotometer [resolution of 0.85 cm⁻¹ by KBr pellet technique]. The powdered samples were thoroughly mixed with dry KBr in the ratio of 1:20 by weight, and then the pellets were formed under a pressure of 9–10 tons.

2.4. DTA measurement

The thermal behaviors (differential thermal analysis DTA) of the finely powdered quenched samples were examined using SEATRAM Instrumentation Regulation, Labsys TM TG-DSC16 (Setaram, Caluire, France) under inert gas. The powder was heated in Pt-holder with another Pt-holder containing Al₂O₃ as a reference material. The results obtained were used as a guide for determining the required heat-treatment temperatures needed to induce crystallization in the samples, as will be shown later.

2.5. Optical absorption

The absorption and transmission spectra of the polished samples of 2-mm thickness at room temperature were measured in the range of 200–1800 nm using a recording spectrophotometer (Jasco Corp., V-570, Rev. 1.00).

3. Results and discussion

3.1. Density and molar volume

Although the measurements of the density (ρ) and the molar volume (Vₘ) are so simple tools, they are enough to tell us about the structural changes that occur in the studied glasses. Both of them depend on softening or compactness of structure of the studied sample. The molar volume Vₘ can give us a good idea about spatial distribution of the oxygen in the glass sample. It is known that the molar volume is a very perfect parameter to determine the compaction or expansion occurring in the glass structure. Molar volume is a bit better in the investigation of the structure and structural information than density. Therefore, we calculate the molar volume, Vₘ, for all studied glasses as follows:

\[ Vₘ = \frac{\sum Xₖ M_k}{\rho} \] (1)

where Xₖ is molar fraction of oxides used in the glass, Mₖ is molecular weight of oxides used and ρ is the density. As shown in Table 1, there is a decrease in the values of calculated molar volume with increase the amount of Eu ions added. The observed increase in ρ of the glass with increase in the contents of Eu₂O₃ may be attributed to the change in the coordination of boron atom in the glass sample. Replacing ZnO of less molar mass [ZnO = 81.39 amu] by Eu₂O₃ of larger molar mass content [Eu₂O₃ = 351.93 amu] leads to formation of a large number of oxygen ions, which are available in glass structure. An observed decrease in the molar volume may be explained by the decrease in the bond length or the decrease in inter-atomic spacing among the atoms of glass network, which leads to a compact structure of the glass. The average distance between two boron atoms \( d_{BO} \) is calculated to estimate the change in the glass structure by addition of Eu₂O₃. The volume containing one mole of boron can be calculated with the given formula \( (Vₘ') \):

\[ Vₘ' = \frac{Vₘ}{2(1 - X₂)} \] (2)

[Table 1 is not provided in the text.]
where $V_m$ is molar volume and $X_2$ is the mole fraction of $\text{B}_2\text{O}_3$

$$\langle d_{c-z} \rangle = \sqrt[3]{\frac{V_m^2}{N_A}}$$  \hspace{1cm} (3)

where $N_A$ is the Avogadro number.

Crystalline volume $V_c$ for all studied glass samples is calculated using the relation

$$V_c = \sum X_i V_i$$  \hspace{1cm} (4)

where $V_i$ is the molar volume of crystalline form of each component [17] (i.e., $V_i = 28.148, 28.300, 14.5$ and $47.557 \text{ cm}^3/\text{mol}$ for $\text{TeO}_2, \text{B}_2\text{O}_3, \text{ZnO}$ and $\text{Eu}_2\text{O}_3$ by taking crystalline density $5.67, 2.46, 5.05$ and $7.40 \text{ g/cm}^3$ for $\text{TeO}_2, \text{B}_2\text{O}_3, \text{ZnO}$ and $\text{Eu}_2\text{O}_3$, respectively). The volume deviation $V_o = (V_m - V_c)$ is listed in Table 1, and we can notice that from this table that the $V_m$ of the glasses is usually much greater than the corresponding values of $V_c$, which indicate the presence of excess structural volume in these samples; this is characteristic of their glassy nature.

The oxygen packing density (OPD) values were calculated from the equation relation as follows:

$$\text{(OPD)} = 1000 \times C \left(\frac{\rho}{M_i}\right)$$  \hspace{1cm} (5)

where $\rho$, $M_i$, and $C$ are density, molar mass and number of oxygen ions present in the formula unit. The values of OPD increase with the increase in $\text{Eu}_2\text{O}_3$ content [18].

### 3.2. Thermal analysis

Fig. 1 represents the DTA measurements of all studied glass samples, from which it is observed that the glass transition temperature $T_g$ increases with the addition of $\text{Eu}_2\text{O}_3$. The increase in $T_g$ may be attributed to the increase in compactness as revealed by the decrease in the molar volume $V_m$ and the increase in oxygen packing density (OPD) (Table 2). The $T_g$ value increases by increasing the bond strength; thus, increase in $T_g$ may also be attributed to high bond strength of $\text{Eu}-\text{O}$ bond compared to $\text{Zn}-\text{O}$ bond [15, 19]. The $T_c$ is the crystallization temperature. The glass thermal stability $\Delta T = (T_g - T_c)$ increases with the increase in $\text{Eu}_2\text{O}_3$ content. The studied glasses have high thermal stability values where $\Delta T > 100 \degree \text{C}$. Thus, high values of $T_g$ and $\Delta T$ suggest that the addition of $\text{Eu}_2\text{O}_3$ to Zinc–boro tellurite glass increases its ability for fiber drawing. The glass forming ability $K_g$ (Hurby factor) was given by the relation:

$$K_g = \frac{T_c - T_g}{T_m - T_c}$$  \hspace{1cm} (6)

The $K_g$ value increased with the increase in $\text{Eu}_2\text{O}_3$ for sample 2 (Eu$_2$O$_3$ content = 0.5). However, with further addition of Eu$_2$O$_3$, there is a small decrease in the Hurby factor values. This means that there is a little effect on the nucleation and crystallization processes inside the glass, or the ability for glass formation is considerably constant [18].

### 3.3. FTIR measurements

Generally the presence of different structural groups in the different glass samples can be identified by using measured infrared spectra of these glasses. In our study, the infrared spectra of base glass (free of Eu ions) were characterized by presence of several peaks at 469, 630, 940, 1100, 1230, 1390 and two small peaks at 1610 and 1640 cm$^{-1}$ as revealed in Fig. 2. We can classify these peaks into 3 regions. The first region lying around 600–700 cm$^{-1}$ can be attributed to the presence of B–O–B bending vibration bond in borate network, second region from 800 to 1200 cm$^{-1}$ which may be attributed to presence of B–O stretching presence in BO$^-$ units, and the third region extended from 1200 to 1600 cm$^{-1}$ due to B–O stretching of BO$_3$ units. Besides, the band from 2400 to 3600 cm$^{-1}$ was due to vibration of O–H bond.
to O–H vibration of water group. There was a very weak band centered at 469 cm⁻¹, which may be attributed to the presence of the stretching vibration of equatorial and axial Te–O bonds in the TeO₄ trigonal bipyramids units, respectively [22–24].

Due to the absence of the band at about 806 cm⁻¹, we can say that the boroxol rings are absent in the network of our studied glass because it is well known that this band is assigned to the boroxol ring in borate glass network. Hence, the glass system contains [BO₃] and [BO₄] groups.

There are two broad bands, in all the samples, centered at 1360 cm⁻¹ and 940 cm⁻¹, respectively, and are attributed to B–O bonds found in both [BO₃] units and [BO₄], respectively. The bond at 1360 cm⁻¹ is attributed to symmetric stretching vibration of a B–O bond of orthoborate, pyroborate and metaphosphate groups in the form of [BO₃] units [22,23] and the other band at 940 cm⁻¹ is attributed to stretching vibration of a B–O bond in [BO₄] tetrahedral units of di-borate groups [22,24]. It was observed that the intensity of the first band decreased while the second increased progressively when europium oxide content increased by replacing an equal amount of ZnO from the glass system. Also, BO₃ band shifted toward lower wave number [1358–1352 cm⁻¹], whereas, BO₄ band shifted toward the longer wave number (961 cm⁻¹) with the rise in europium content. Hence, the continuous increase of europium concentration helped conversion of [BO₃] to [BO₄] groups in the base glass ZnO TeO₂–B₂O₃, which was already confirmed by the study of density and molar volumes.

### 3.4. Optical properties

The transmittance T (λ) and the reflectance R (λ) spectra in the wavelength range of 200–2500 nm for ZnO boro-tellurite glass doped with Eu₂O₃ are illustrated in Fig. 3. It is noticed that the studied samples are in the absorbing region where R + T < 1.

For each of the studied samples, the reflectance values show a gradual increase from 430 nm up to 450 nm followed by constant values up to 1800 nm. A sharp increase in transmittance values is observed at 430 nm followed by a gradual increase up to 830 nm; thereafter, it became constant. It is noticed that T values decrease with the increase in Eu content, and an absorption region between 500 and 830 nm was observed for Eu-containing glass. Such absorption was attributed to the optical transitions from 7F₅ to 5D₄, 5G₄, 5L₆, 5D₃, 5D₂, 5D₁ and 5D₀ of Eu³⁺ ions [25].

The absolute values of T (λ) and R (λ) are used to calculate the optical constants, the optical absorption coefficient (α), the absorption index (k) and the refractive index (n) using the following relations [26]:

\[
\alpha = \frac{1}{t} \ln \left[ \frac{(1-R)^2}{2T} + \sqrt{\frac{(1-R)^2}{4T^2} + R^2} \right]
\]

\[
k = \frac{\alpha \lambda}{4\pi}
\]

\[
n = \frac{1 + R}{(1-R)} + \sqrt{\frac{4R}{(1-R)^2 - k^2}}
\]

Increase in (α) values was observed with the increase in Eu content as indicated in Fig. 4. The absorption peaks observed from 850 to 430 nm for Eu-containing samples are attributed to the presence of color centers induced by Eu ions.

<table>
<thead>
<tr>
<th>Glass sample</th>
<th>Tₙ (0°C)</th>
<th>Tᵣ (0°C)</th>
<th>Tₘ (0°C)</th>
<th>ΔT (0°C)</th>
<th>Kᵣ</th>
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<td>564</td>
<td>625</td>
<td>169</td>
<td>0.734</td>
</tr>
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<td>220</td>
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<tr>
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<td>652</td>
<td>755</td>
<td>242</td>
<td>0.701</td>
</tr>
<tr>
<td>3</td>
<td>431</td>
<td>660</td>
<td>747</td>
<td>239</td>
<td>0.724</td>
</tr>
</tbody>
</table>

**Fig. 2** – FTIR for base glass and glass doped with x mol% Eu₂O₃.

**Fig. 3** – The transmittance T (λ) and the reflectance R (λ) spectra for ZnO boro-tellurite glasses doped with x Eu₂O₃.
The relationship between the optical absorption coefficient (\(\alpha\)) and the photon energy was described by Tauc [27].

\[
(\alpha h \nu) = B(h \nu - E_g)^{r}
\]

where B is constant, \(h \nu\) is the incident photon energy, \(E_g\) is the optical band gap of the glass sample, and \(r\) depends upon the type of transition. The value of \(r\) is \(\frac{1}{2}\) for the allowed direct transition, whereas it is 2 for the indirect transition. The indirect band gap (E\(_g\)) was estimated by extrapolating the straight line part of the relation between \((\alpha h \nu)^{1/2}\) and photon energy at \((\alpha h \nu)^{1/2} = 0\), as shown in Fig. 5. It is clear that there is a decrease in the \(E_g\) values with the increase in Eu\(_2\)O\(_3\) content as shown in Table 2. This behavior is attributed to the structural changes in the glass network with the addition of Eu\(_2\)O\(_3\). The addition of Eu\(_2\)O\(_3\) enhances the number of non-bridging oxygen (NBO) atoms in the glass network. NBO’s ions shift the edge of the valence band toward the conduction band [28]. This leads to decreases in the optical band gap values.

The refractive index (n) values (determined from Eq. (9)) at different wavelengths are plotted in Fig. 6. It is noticed from Fig. 6 that the refractive index values decrease with increasing Eu\(_2\)O\(_3\) content. Fig. 6 represents a peak with \(n_{\text{max}}\) value at \(\approx 840\) nm, which shifts to longer wavelength with increasing Eu\(_2\)O\(_3\) content. The values of n and \(n_{\text{max}}\) decreased with the addition of Eu\(_2\)O\(_3\). At \(\lambda > 1100\) nm, a normal dispersion is observed. Such a dispersion is analyzed by Wemple and DiDomenico (WDD) single oscillator model [29,30]. The (WDD) model suggests that the relation between n and \(h \nu\) is given in the following equation:

\[
(n^2 - 1)^{-1} = \frac{E_d}{E_\text{g}} - \frac{1}{E_\text{g}E_d(h \nu)^2}
\]

where \(E_d\) is the effective oscillator energy and \(E_\text{g}\) is the dispersion energy, which measures the strength of the inter-band optical transitions. \((n^2 - 1)^{-1}\) is plotted versus \((h \nu)^2\) as shown in Fig. 7. \(E_d\) and \(E_\text{g}\) values are determined from the slope and intercept of the extrapolated straight line and listed in Table 3. The effective oscillator energy, \(E_d\), is related to the bond energy of the chemical bonds existing in the glass matrix [31]. Thus, the formation of non-bridged oxygen ions decreases the

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**Fig. 4** – The absorption coefficient (\(\alpha\)) at different photon energies as function of Eu\(_2\)O\(_3\).

**Fig. 5** – The relation between \((\alpha h \nu)^{1/2}\) and photon energy as function of Eu\(_2\)O\(_3\).

**Fig. 6** – The relation between refractive index (n) and wavelength (\(\lambda\)) as function of Eu\(_2\)O\(_3\).

**Fig. 7** – The relation between \((n^2 - 1)^2\) and \((h \nu)^2\) as function of Eu\(_2\)O\(_3\).
fraction of covalent and increases the fraction of ionic bonds, which have lower bond energy than the covalent bonds. This behavior could explain the decrease of $E_d$ value with increase in Eu$_2$O$_3$ content.

The dielectric constant at infinite frequency $\varepsilon_\infty$ is determined from extrapolation of the straight line with the ordinate axis at $(h\nu)^2 = 0$. Petkov et al. [32] proposed the relations between the dispersion energy $E_d$ and other physical parameters as:

$$E_d = \beta N_c Z_g N_e$$

(12)

where $N_c$ is the coordination number of the cation, which is the nearest neighbor to the anion, $Z_g$ is the formula chemical valence of the anion, $N_e$ is the effective number of valence electrons per anion and $\beta$ is a parameter between 0.26 ± 0.04 eV for ionic materials and 0.37 ± 0.05 eV for covalent materials. Using the calculated $\beta$ values of Eu$_2$O$_3$-doped and -undoped Zn–boro-tellurite glass (Table 2) and the values of $N_c$ = 4, $Z_g$ = 2 and $N_e$ = 8 [26,29], the obtained $\beta$ values are around 0.261. These results confirmed that Eu$_2$O$_3$-doped glasses have more ionic character than the undoped glass, which could be attributed to the increase in the number of non-bridging oxygen (NBO) atoms in the glass network.

The refractive index, $n$, is related to the lattice dielectric constant ($\varepsilon_L$) by the following equation [33]:

$$n = \varepsilon_L - \left(\frac{\varepsilon_0}{4\pi^2\varepsilon_L}\right) \frac{N}{m'} \lambda$$

(13)

where $\varepsilon$ is the electronic charge, $\varepsilon_0$ is the permittivity of free space, $c$ is the speed of light and $N/m'$ is the ratio of the carrier concentration to the effective mass. Fig. 8 indicates a linear relation between $n^2$ and $\lambda^2$, for Eu$_2$O$_3$-doped and -undoped glasses, which is consistent with Eq. (13). The $\varepsilon_L$ values are determined from the extrapolation of the linear relation to $\lambda^2 = 0$. It is noticed that $\varepsilon_\infty < \varepsilon_L$, which was attributed to free charge carrier contribution [34].

### 3.5. Nonlinear optical properties

Several theoretical models have been proposed for calculation of the third-order susceptibility $\chi^{(3)}$ and the non-linear index $n_2$ [35–37]. In this work, the model suggested by Tichy et al. [38] is applied. They combine the Miller’s generalized rule [39] with the static refractive index, which is evaluated from the WDD single oscillator model [29]. According to their proposed model, $\chi^{(3)}$ and $n_2$ are given by the following relations:

$$\chi^{(3)} = \frac{A}{(4\pi)^3} \left(\frac{E_d}{E_0}\right)^4 = \frac{A}{(4\pi)^3} (n_2^2 - 1)^4$$

Fig. 8 – The relation between ($n^2$) and ($\lambda^2$) as function of Eu$_2$O$_3$.

$$n_2 = \frac{12\pi \chi^{(3)}}{n_0}$$

where $A = 1.7 \times 10^{-11}$ and $n_0$ is the static refractive index. From Eq. (11), as $h\nu \rightarrow 0$, then $n = n_2$, i.e.,

$$n_0 = \sqrt{1 + \frac{E_d}{E_0}}$$

The third-order susceptibility $\chi^{(3)}$ and the non-linear index $n_2$ values for the studied glasses are listed in Table 3. It is observed from Table 3 that $\chi^{(3)}$ and $n_2$ values decreased with the addition of 0.5 mol Eu$_2$O$_3$. The observed decrease in Eu$_2$O$_3$-doped glass could be attributed to the replacement of a higher polarizable ZnO by a lower polarizable Eu$_2$O$_3$.

However, the $\chi^{(3)}$ values indicate a gradual increase with further increasing Eu$_2$O$_3$ concentration. This behavior could be explained by the enhancement of excitation electron with the increasing Eu$_2$O$_3$ concentration [40].

ZnO borate–tellurite glass doped with Eu$_2$O$_3$ showed higher values of nonlinear refractive index $n_2$ and nonlinear optical susceptibility $\chi^{(3)}$ than that reported for heavy elements, such as borate and silicate glasses [41,42]. These results suggested that Eu$_2$O$_3$-doped and -undoped ZnO borotellurite glasses have promising applications in nonlinear optical switching devices [40,43].

### 4. Conclusion

Glasses of composition 24B$_2$O$_3$ – (25 – x) ZnO – 51TeO$_2$, x Eu$_2$O$_3$, mol% (where $x = 0.0, 0.5, 1, \text{and} 3\%$) were prepared.
1. Density of studied glasses was measured and found to decrease with increasing the Eu$_2$O$_3$ content, whereas the molar volume of the same studied glasses were found to decrease.

2. Crystalline volume $V_c$ for all studied glass samples were calculated, and the volume deviation $V_o$ were estimated and found to decrease with increasing the Eu$_2$O$_3$ content.

3. FTIR spectra were characterized by the presence of several peaks at 469, 630, 940, 1100, 1230, 1390 and two small peaks at 1610 and 1640 cm$^{-1}$.

4. By addition of Eu$_2$O$_3$, there was a change in intensity and position of the peaks related to change from [BO$_3$] to [BO$_4$] groups.

5. The glass thermal stability $\Delta T$ ($T_g - T_L$) increases with the increase in Eu$_2$O$_3$ content.

6. The glass forming ability $K_f$ (Hruby factor) increased with the increase in Eu$_2$O$_3$.

7. The $E_g$ values were with increasing in Eu$_2$O$_3$ content, which attributed to the Enhancement of the number of non-bridging oxygen (NBO) atoms in the glass network.

8. The studied glasses have high values of nonlinear refractive index $n_2$ and nonlinear Optical susceptibility $\chi^{(3)}$.

The results obtained indicated that ZnO boro-tellurite glass doped with Eu$_2$O$_3$ has high nonlinear optical property values with good thermal stability, which make these glasses suitable candidates for their application in fiber optics and nonlinear devises and broadband optical amplifiers.

## Conflicts of interest

The authors declare no conflicts of interest.

## References


