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EFFECT OF DIFFERENT CONCENTRATIONS OF DOPED RARE EARTH ELEMENT ON BORATE–SILICA OXIDE GLASS STRUCTURE

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A new series of doped rare earth element erbium oxide (Er_2O_3)-based zinc borate silica glasses was prepared. These compounds exhibited the chemical formula [(X Er_2O_3)(50–X)% ZnO)][(20%B_2O_3)(30%SiO_2)], where X = 0.5%, 1%, 2%, 3%, 4%, and 5%mol%. The SiO_2 used was obtained from a rice husk. The glass samples were subjected to different measurements that include X-ray diffraction (XRD), differential scanning calorimetry (DSC), density, and molar volume. The amorphous phase of the glass was confirmed using XRD. Results of the DSC analysis indicated that the *Tg* of the glass samples decreased from 391.18 °C to 382.03 °C with gradual increase of the concentrations of Er_2O_3 from 0.5 mol% to 5 mol%. The density of the glasses and their molar volume increasedwith increased doped erbium oxide in the glass.

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1. Introduction

Soda lime, which is the most common type of glass, is composed of about 75% silica (SiO_2) and sodium oxide (Na_2O) from soda ash **[1].** Production of glasses includes two kinds of glass systems, namely, artificial and natural glasses. Oxide glasses are the best-known class of non-crystalline materials, which include a large number of glass families. The network of oxide glasses is composed of network formers and network modifiers **[2].** Boron is used in preparing glasses for both scientific and technological applications **[3].** Continued efforts to expand the number of new glassy resources, either by doping or adding transition metal ions to oxide glasses, are performed; the study of novel properties is highly related due to their potential applications in various technological fields **[4].** Rare earth element (REE) ions actively function in generating and applying guided radiations **[5].**REEs possess variedphysical and optical properties. Only tiny quantities of REEs are used in most applications. [6] **, [7].** In this work, we report our findings on the analysis of samples of silica–borate glasses that contain various concentrations of the doped rare earth element Er^{+3}.This work is divided into two parts. The first part focuses on obtaining silica oxide from rice husk ash and workability of the silica oxide to behave as a network former in the glass structure. The second part involves fabricating and characterizing the glass sample using the formulation **[(x Er_2O_3) (50–X%ZnO)][(20% B_2O_3)(30% SiO_2)].**The glass samples are characterized via X-ray diffraction, and the glass transition temperature *Tg* is determined by differential scanning calorimetry (DSC). The density and molar volume are also determined in this study.

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2. Experimental Procedure

2.1 Preparation of SiO_2 using rice husk (RH)

The RH is obtained from a Malaysian domestic procurement and processing of paddy and rice (BERNAS). Exactly 50 g of RH is washed with distilled H_2O, which completely removes dust and clay. The RH is then treated using a mixture of 17 mL hydrochloric acid and 500 mL distilled H_2O at 110 °C. A hot plate magnetic machine is used to heat the sample for 3 h. The RH is rinsed with distilled H_2O and dried at 110 °C in a calibrated electric oven for 3 h. After drying, the RH is then placed in an alumina crucible and heated in a high- temperature electrical furnace, as shown in Fig. 1and 2. The setting temperatures employed are 550 °C for 6 h, 550 °C for 8 h, and 750 °C for another 6 h. The highest percentage of the SiO_2, which is determined using the fused glass–beads technique, is 99.5 wt% at 550 °C and 8 h.

Fig. 1 RH before treated in electrical furnace

Fig. 2 RH after treated in electrical furnace

2.2 Glass fabrication

The glass samples are prepared using B_2O_3 and SiO_2 (99.5% purity), and the dopant used is Er_2O_3 (99.8% purity). Around7 g of the samples are heated in alumina crucibles. The glass samples are prepared using melt quenching to obtain homogeneous and transparent glass.The samples used **[(XEr_2O_3)(50–X% ZnO)][(20%B_2O_3)(30%SiO_2)]**, where X = 0.5%, 1%, 2%, 3%, 4%, and 5%mol%**.** All of the reagents are precisely weighed and mixed using an agate mortar and pestle. The mixtures are then placed in an alumina crucible and heated in an electric furnace at 1200 °C for 1 h. The molten glass samples are placed in molds to produce cylindrical glass blocks to prevent breaks and crack. The glass blocks are removed from the mold and polished to obtain parallel and specific thickness and shiny surfaces. The amorphous nature of the samples is confirmed by XRD. The thermal stability of the samples is measured using a Mettler Toledo DSC 822e. The scanning temperature ranges from 30 °C to 440 °C at 20 °C/min **[7]**.The density of the solid glasses is first measured via Archimedes technique, wherein a sample is weighed in air (w_a) and in H_2O (w_L). The density of the sampleis then calculated using the formula

$$\rho = \frac{A}{/B/}\rho^{\circ}$$

ρ is the density of the sample in g/cm^3.
Aisthe weight of sample in air in (g).

B istheweight of sample in air (w_a) - weight of sample in liquid (w_L) in (g).

ρ^o is the density of liquid (for water about 1 g/cm^3).

After determining the density of all the glasses, the specific volume Vs is then calculated using the equation

$$Vs = \frac{1}{\rho}$$

The molar volume values are calculated using the measured densities and molecular weight of 1 molw$_m$ of glass sample using the equation

$$Vm = \frac{1}{\rho} \mathrm{Wm}$$

3. Result and Discussion

Fig 3 shows the XRD of the glass samples. Sharp Bragg peaks are not observed; however, this result only confirms the amorphous nature of the material, and all glasses studied are purely amorphous and non-crystalline **[8]**, [7].

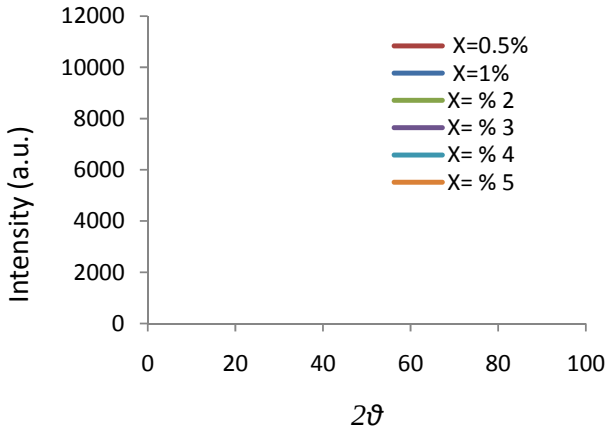


Fig.3. XRD pattern of the glass samples

The glass samples are ground in a porcelain mortar to measure the *Tg*. DSC is used to study the thermal stability of the prepared samples using a Mettler Toledo DSC 822e. The scanning temperature employed is in the range of 30 °C to 440 °C at 20 °C/min. Fig. 4 shows the range of transition temperatures of samples 3, 4, and 6

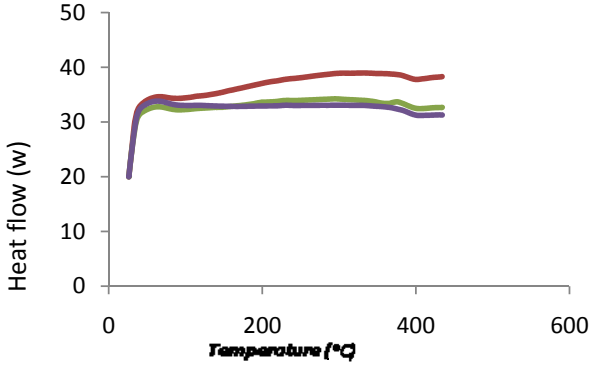


Fig. 4The DSC curves characteristictemperature such as glass transition temperature (Tg)

Table 1 shows the erbium oxide percentage and the transition temperature of the glass samples. The DSC results reveal that *Tg* decreased monotonically with increased erbium oxide

content, as shown in Fig. 5. The *Tg* value depends on the strength of chemical bonds in a structure. The electron bond generated between the silicon and oxygen atoms provides silica glass with its imposing mechanical strength and thermal properties. Rare earth ions require the coordination of a sufficiently high number of non-bridging oxygen ions. Therefore, rare earth ions are inclined to share non-bridging oxygen ions. Network modifiers are required to increase the amount of obtainable non-bridging oxygen ions in a silica glass network, and consequently, raise the amount of rare earth ions. Thus, ZnO was used as network modifiers to facilitate the incorporation of rare earth ions because of their larger size compared with that of a basic network. These modifiers break bridging oxygen ions to form non-bridging oxygen ions and can be used to coordinate rare earth ions [9]. Increasing the amount of non-bridging oxygen ions indicates the breaking of chemical bonds, which decreases the *Tg* value [4].

Table 1.Glass transition temperature obtained by DSC.

Samp. No	3	4	6
Er_2O_3(%)	2	3	5
Tg (***℃***)	391.18	387.87	382.03

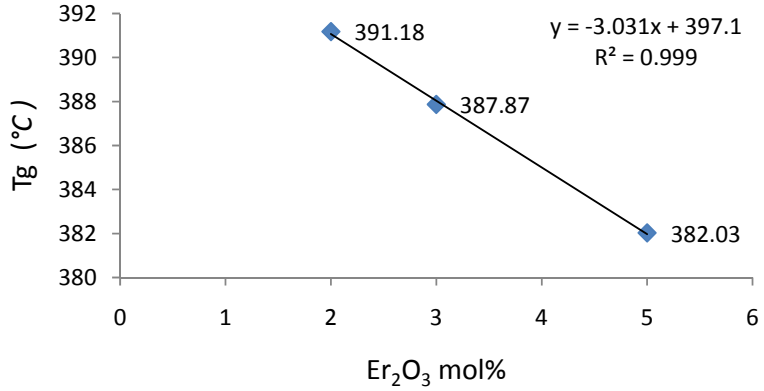


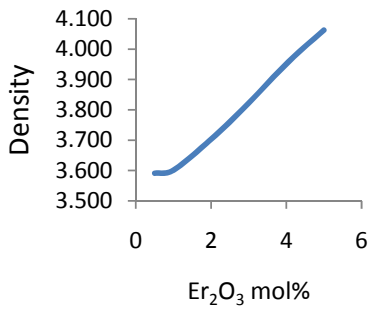
Fig. 5 Transition temperature againstEr_2O_3 mol%

The densities in the structures of glasses and non-crystalline solidsshould be studied because glass density changes abruptly when glass structure slightly changes [3]. Thus, the densities of the glass samples were measured to determine any structural change in the glass as erbium oxide gradually increased at the expense of zinc oxide. Table 2 presents the results of the densities, specific volumes, and molar volumes of the samples.

Table 2.contain Density, Specific volume and molar volume values of glass samples.

Samp. No	1	2	3	4	5	6
Er_2O_3 %	0.5	1	2	3	4	5
Density	3.591	3.601	3.703	3.822	3.952	4.063
Sp. Vol.	0.278	0.278	0.270	0.262	0.253	0.246
Mol. Vol	20.646	21.005	21.240	21.371	21.429	21.585

Physical properties provide an insight into the atomic structures of a glass network [10]. Glass is a mixture of element oxides. Glass density functions as the volume of element ions; it depends on ion number and the manner in which ions can enter the glass structure [11]. In fig 6, the density change as a function of doped erbium oxide. Density gradually increased from 3.591 gm/cm^3 to 4.063 gm/cm^3 because the amount of erbium oxide gradually increased at the expense of zinc oxide. Only a slight change in density was observed. This finding is attributed to the role played by erbium oxide, which contributes to glass homogeneity and the provision of excess oxygen in the network. Specific volume directly represents the inverse of density. The interesting parameter is molar volume because it relates to direct oxygen distribution in the glass network [3]. Fig. 7 shows the change in molar volume with an increase in erbium oxide content; such increase is slight and gradual. The existence of ZnO in borosilicate glasses exhibits an increase in oxygen packing density, which squeezes the structure of the samples. The structure becomes more compact as oxygen packing density increases. Thus, as density increases, molar volume also rises.



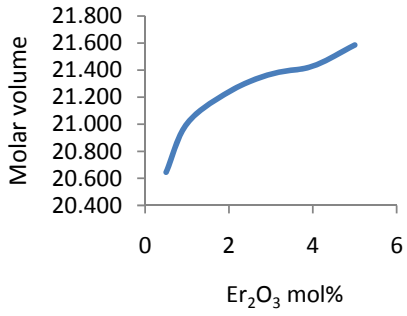


Fig 6. Density against Er_2O_3 content *Fig 7. Molar volume against Er_2O_3 content*

4. Conclusion

The transparent pink samples are investigated by XRD. The obtained patterns indicate that all the glass samples are pure and non-crystalline. Sharp peaks that result from crystalline phases of a material are not present in the structure. Thus, the result confirms the amorphous nature of the material. The results of DSC indicate that the *Tg* of the glass samples decreases with the gradual increase of erbium oxide from 0.5 mol% to 5 mol%. Increased non-bridging oxygen ions indicates the breaking of chemical bonds, which in turn decreases *Tg*. The density of all the samples and the molar volume increases with increased erbium oxide content in the borate silica. All the above conclusions agree with the experimental results.

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