



## Research Article

# Structural, elastic and thermo-physical properties of $\text{Er}_2\text{O}_3$ nanoparticles doped bio-silicate borotellurite glasses

S. A. Umar<sup>1,2</sup> · M. K. Halimah<sup>2</sup> · M. N. Azlan<sup>3</sup> · L. U. Grema<sup>4</sup> · G. G. Ibrahim<sup>2,6</sup> · A. F. Ahmad<sup>2</sup> · A. M. Hamza<sup>2,5</sup> · M. M. Dihom<sup>2,7</sup>

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### Abstract

This paper presents a study on the structural, morphological and elastic properties of erbium oxide nanoparticles doped bio-silicate borotellurite glass system fabricated using the technique of melt-quenching with chemical compositional formula  $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.8}(\text{SiO}_2)_{0.2}\}_{1-y}(\text{Er}_2\text{O}_3 \text{ NPs})_y$ , where  $0.01 \leq y \leq 0.05$ . The objective of the work was to study the some elastic and thermal parameters associated with non-destructive ultrasonic technique, with the aim of utilizing the glasses for erbium doped fiber amplifier application. Various measurements and Characterizations techniques which includes density and molar volume measurements, energy dispersive X-ray fluorescence, X-ray diffraction (XRD), Fourier transform infrared (FTIR), Transmission electron microscopy and Pulse-echo technique were carried out in this study. The density values for the glasses increased from 4.1900 to 4.6003  $\text{g cm}^{-3}$  with the addition of 1–5% of  $\text{Er}_2\text{O}_3$  NPs in the glass structure. The increase can be ascribed to increase in the overall molar weight of the glass due to incorporation of heavy  $\text{Er}^{3+}$  ions. The XRD patterns revealed no presence of sharp peaks with the observed broad hump around  $2\theta = 20\text{--}30^\circ$  indicating the glassy and amorphous nature of the studied glasses. The FTIR spectra showed absorption with peaks around 1362–1385  $\text{cm}^{-1}$ , 1221–1250  $\text{cm}^{-1}$ , 655–689  $\text{cm}^{-1}$  and 602–630  $\text{cm}^{-1}$  wave number ranges, indicating the presences of  $\text{H}_3\text{BO}_3$ ,  $\text{SiO}_4$ ,  $\text{BO}_3$ ,  $\text{TeO}_3$  and  $\text{TeO}_4$  structural units in the glass system. The microstructural nature observed in the glass morphology showed nanoparticle agglomerations in the glasses. The ultrasonic velocities, elastic moduli, micro-hardness, Poisson ratio, softening and Debye's temperatures, thermal expansion coefficient as well as other important parameters were calculated from the density and ultrasonic data. The values of the ultrasonic velocities, elastic moduli, Debye's temperature, softening temperature, thermal expansion coefficient and the acoustic impedance increased with increase in the  $\text{Er}_2\text{O}_3$  NPs concentration.

**Keywords** Rice husk silica ·  $\text{Er}_2\text{O}_3$  NPs · XRD · FTIR · Ultrasonic technique · Elastic moduli

✉ S. A. Umar, usaltilde@yahoo.com; M. K. Halimah, halimahmk@upm.edu.my; M. N. Azlan, azlanmn@fsm.ups.edu.my; L. U. Grema, lawangrema@yahoo.com; G. G. Ibrahim, ibgana55@gmail.com; A. F. Ahmad, ahmad\_al67@yahoo.com; A. M. Hamza, amhamza419@gmail.com; M. M. Dihom, mdihom71@gmail.com | <sup>1</sup>Department of Physics, Faculty of Science, Federal University Lafia, Lafia, Nasarawa State, Nigeria. <sup>2</sup>Department of Physics, Faculty of Science, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia. <sup>3</sup>Physics Department, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris, 35900 Tanjung Malim, Perak, Malaysia. <sup>4</sup>Department of Mechanical Engineering, Ramat Polytechnic Maiduguri, Maiduguri, Borno State, Nigeria. <sup>5</sup>National Agency for Science and Engineering Infrastructure, Abuja, Nigeria. <sup>6</sup>Department of Physics, Yobe State University, Damaturu, Nigeria. <sup>7</sup>Department of Physics, Al Asmarya Islamic University, Zliten, Libya.



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## 1 Introduction

In the last few decades, the need for an increased rice husk commercialization is ever increasing with continued increase in the global rice production above 645 million tonnes annually [1]. The commercialization of the rice milling waste beyond the present 30% becomes necessary so as to solve the environmental pollution problems caused by their continued disposal and also help in solving some of the mankind's problems [1, 2]. One of the areas considered these days for the rice husk is in glass and ceramics fabrications for optical fibre, optoelectronics and other glass and ceramics technological applications [3–5]. Many different researchers have worked on the silicate extraction (from the rice husk) for fabrication of different glass compositions for various technological and industrial applications [6, 7].

Glass science and technology research in the present days has been giving great attention to the fabrications and utilizations of  $\text{TeO}_2$ -based glasses [8]. Tellurium oxide based glasses have demonstrated more advantages over other silicate and phosphate glasses due to their low melting point, high rare RE ions solubility, high effect of Stark splitting, high refractive index and high optical transparency in wide spectral region [9–11]. Tellurium oxide alone does not form glass on its own, but rather requires combination with other glass formers or modifiers.  $\text{TeO}_2$ - $\text{B}_2\text{O}_3$  combination was found to be one of the most favourable combinations for the glass utilization in the area of optical fibre amplifiers, optoelectronics and laser applications [5, 9]. Borotellurite glasses present a compromising quality against the individual disadvantages of  $\text{B}_2\text{O}_3$  and  $\text{TeO}_2$  by having high chemical durability, high refractive index, high thermal stability, low melting point, and high refractive index [12].

Among the number of rare earth (RE) ions used in the areas of optical fibres, optoelectronic and lasers,  $\text{Er}^{3+}$  ions are found to be more suitable for optical signal amplifiers and sometimes laser applications and white-light emitting devices [13]. Since  $\text{Er}^{3+}$  ions in glasses possess two useful emission bands around 1500 nm and 2700 nm, the former is utilized in the EDFA and the 2700 nm band is utilized in medicine and optical sources for sensors [14]. The area of optical transmission in the communication system requires gain bandwidth widening of the optical amplifiers that is utilized in the wavelength division multiplexing (WDM) systems [15].

Non-destructive ultrasonic techniques have been used by various researchers in the study of elastic properties of non-crystalline and crystalline materials alike [16]. The ultrasonic velocities were used together with density and their molecular/structural data for the calculation

of the elastic moduli, microhardness, acoustic impedance, softening temperature, Debye temperature, fractal bond connectivity and the Poisson ratio [16, 17]. In another study, the elastic properties of erbium oxide doped  $\text{TeO}_2$ - $\text{ZnO}$ - $\text{P}_2\text{O}_5$ - $\text{LiNbO}_3$  were reported. The elastic moduli investigated were found to have increased with  $\text{Er}^{3+}$  ions concentration [18].

In this study a choice was made for  $\text{SiO}_2$ - $\text{TeO}_2$ - $\text{B}_2\text{O}_3$  composition for erbium oxide nanoparticles' hosting with the aim of increasing the rice husk commercialization (through  $\text{SiO}_2$  extraction) in the area of lasers and EDFA applications. The choice of  $\text{TeO}_2$  was to improve the refractive index and the optical transparency in the IR region.  $\text{B}_2\text{O}_3$  was selected to compromise the high phonon energy of the  $\text{TeO}_2$ , lower the glass forming temperature, improve the RE solubility and hardness [19].  $\text{SiO}_2$  composition aimed at improving both the thermal and chemical stability as well as the mechanical strength of the glasses [2].

Non-destructive ultrasonic technique was employed in this work for the collection of ultrasonic velocity data. The elastic moduli, Poisson ratio, micro-hardness, Debye temperature, softening temperature, acoustic impedance, thermal expansion coefficient and fractal bond connectivity as well as the morphological and structural properties were studied. The ultrasonic studies provide information on glass structure as well as some thermal and mechanical features of studied glasses [16, 20]. For better utilization of glass material in the EDFA and laser applications, studying the elastic properties of such material is very important especially as some glasses' elastic properties might not always favour fibre drawing.

### 1.1 Experiments

From a rice milling factory in Malaysia, rice husk (rice milling waste) was obtained. Using the simple cold acid (HCl) leaching technique, 98.548% of  $\text{SiO}_2$  was extracted. The extracted  $\text{SiO}_2$  was used with  $\text{Er}_2\text{O}_3$  NPs (Alfar Aeser, 99.9%),  $\text{B}_2\text{O}_3$  (Alfar Aeser, 99.9%) and  $\text{TeO}_2$  (Alfar Aeser, 99.9%) for the fabrication of  $\{[(\text{TeO}_2)_{0.7} (\text{B}_2\text{O}_3)_{0.3}]_{0.8} (\text{SiO}_2)_{0.2}\}_{1-y} (\text{Er}_2\text{O}_3 \text{ NPs})_y$  using melt-quenching technique.

For the glass fabrication, powdered chemicals were weighed using an electronic weighing balance in accordance with the compositional chemical equation. The weighed chemicals were made homogeneous by stirring for ½ hour in an alumina crucible. The crucible containing the homogeneous mixture is then put into a 400 °C electric furnace for 1 h preheating before transferring to another furnace at around 900 °C for another 2 h for mixture melting. The preheating was necessary to enable the evaporation of any possible moisture in the mixture as  $\text{B}_2\text{O}_3$  is highly hygroscopic in nature [10]. The molten material was turned into a cylindrical melt and transferred to the 400 °C furnace for

1 h annealing to remove thermal stress and bubbles before being allowed to cool down to room temperature [9]. The cylindrical glasses were finally cut to about 4–5 mm thickness for non-destructive ultrasonic probing and the remaining portion grinded for XRD, TEM and FTIR analyses.

The XRF analysis of the extracted SiO<sub>2</sub> was performed using the Energy Dispersive X-ray Spectrometer (Shimadzu model EDX-720) to determine its purity. The density was measured using MD-300S, Alfa Mirage electronic densimeter and using the Archimedes principle. Using Archimedes principle, the glass density ( $\rho$ ) and molar volume ( $V_m$ ) were calculated as follows;

$$\rho = \frac{W_{Air}}{W_{water}} \tag{1}$$

$$V_m = \frac{M_w}{\rho} \tag{2}$$

where water density was taken to be 1 g/cm<sup>3</sup>,  $M_w$  = molecular weight of glass,  $W_{air}$  = weight of sample in air, and  $W_{water}$  = sample weight in water [9].

Other characterization performed includes the X-ray diffraction (XRD) using the XRD system {PANalytical (Philips) PW3050/60}, Fourier transform infrared (FTIR) using Perkin Elmer 1650 spectrometer and Transmission Electron Microscopy (TEM) using JOEL transmission electron microscope that operates at 200 kV. Using Ritec Ram-5000 Snap System, ultrasonic pulse-echo technique was performed to determine the ultrasonic (longitudinal and shear) velocities in the samples.

### 1.2 Results and discussions

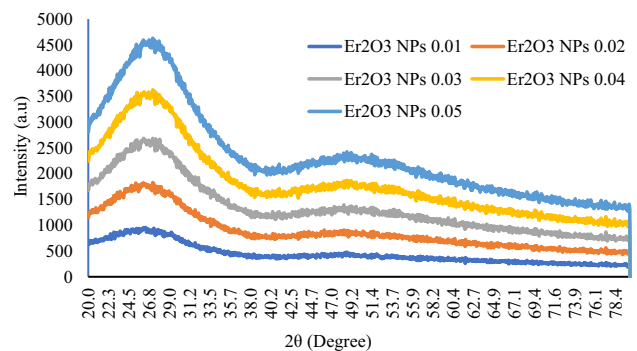
Table 1 presents the XRF analysis result of the SiO<sub>2</sub> extracted from rice husk using cold HCl leaching method. Unlike the hot leaching technique presented by Mustapha et al. [21] which requires heating, the present SiO<sub>2</sub> extraction require no heating during leaching and is cost effective as well. About 98.548% pure SiO<sub>2</sub> was achieved as shown in Table 1.

**Table 1** Result of the XRF analysis of rice husk extracted SiO<sub>2</sub>

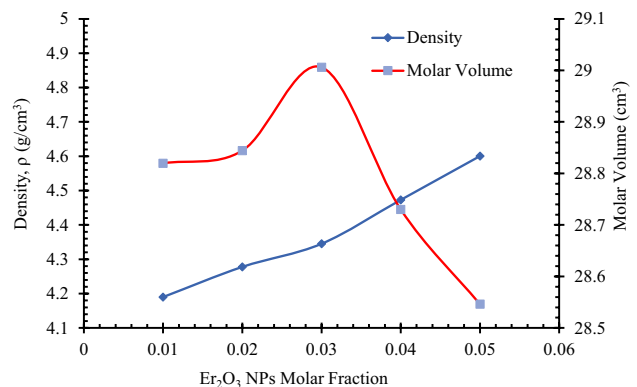
Element (Oxide)	Amount/Percentage (%)
SiO <sub>2</sub>	98.548
SO <sub>3</sub>	0.793
CaO	0.407
Fe <sub>2</sub> O <sub>3</sub>	0.129
K <sub>2</sub> O	0.079
MnO	0.035
ZnO	0.009

The XRD patterns for the studied glasses are presented in Fig. 1. XRD result study is used for identification of amorphous nature and crystalline phase presences in samples [22]. The patterns in Fig. 1 showed the spectra recorded between 20° ≤ 2θ ≤ 80° observed at lower scattering angles is a broad diffusion indicating the presence of long range structural disorder in the glasses [23]. The presence of a broad hump around 2θ = 27° indicates the glassy/amorphous nature of the studied glasses. As observed in the Figure, as the Er<sub>2</sub>O<sub>3</sub> NPs are increased in concentration, the broad hump continue to narrow, suggesting the tendency of crystallization with continued increase in the Er<sub>2</sub>O<sub>3</sub> NPs concentration [24].

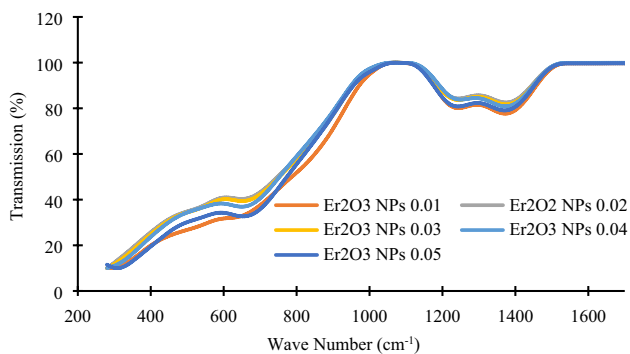
The density and molar volume variation with Er<sub>2</sub>O<sub>3</sub> NPs concentration is presented in Fig. 2. The density value increased from 4.1900 to 4.6004 gcm<sup>-3</sup> as the concentration of Er<sub>2</sub>O<sub>3</sub> NPs increased from 0.01 to 0.05 Molar. The high density value observed might be due to the high material compactness resulting from to the presence of nanoparticle of Er<sub>2</sub>O<sub>3</sub>. The increase in the density value with dopant increase might be associated to decrease in the non-bridging oxygen number which can be confirmed



**Fig. 1** XRD patterns of the  $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.8} (SiO_2)_{0.2}\}_{1-y} (Er_2O_3 NPs)_y$  glass system



**Fig. 2** Density and molar volume  $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.8} (SiO_2)_{0.2}\}_{1-y} (Er_2O_3 NPs)_y$  glass system



**Fig. 3** FTIR Spectra of  $\{[(\text{TeO}_2)_{0.7} (\text{B}_2\text{O}_3)_{0.3}]_{0.8} (\text{SiO}_2)_{0.2}\}_{1-y} (\text{Er}_2\text{O}_3 \text{ NPs})_y$  glass system

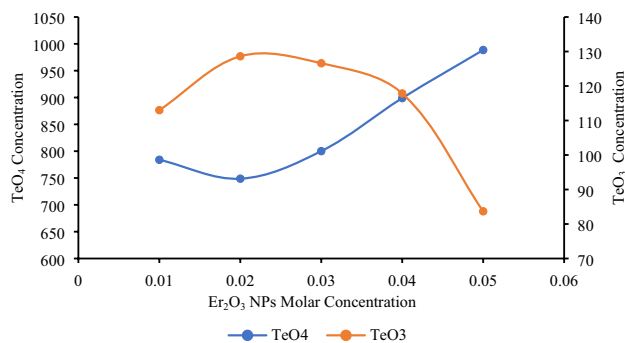
by the  $\text{TeO}_4/\text{TeO}_3$  concentration variations in Fig. 4. The increase in the density might also be due to increased substitution of lighter  $\text{B}^{3+}$ ,  $\text{Si}^{4+}$ , and  $\text{Te}^{4+}$  ions with comparatively much heavier ions of  $\text{Er}^{3+}$ , resulting in the overall glass molecular weight increase [19]. The molar volume increased from 28.8196 to 29.0061  $\text{cm}^3$  and then decreased down to 28.6456  $\text{cm}^3$  as the dopant's concentration raised from 0.01 to 0.03 and down to 0.05 M respectively. The initial value increase can be attributed to a decrease in atomic compactness in the glasses network due to breakage one of the double bonds of oxygen atoms in the network (decreased connectivity) resulting from the formation of  $\text{TeO}_3$  structural units [9]. The decrease in the glasses' molar volume with addition of more dopant can be due the increase in the concentration  $\text{TeO}_4$  units suggesting more cross-linkage between the oxygen atoms and the cations in the glass network [17].

The FTIR spectra for the studied  $\text{Er}_2\text{O}_3$  NPs doped silica borotellurite glasses are presented in Fig. 3. Various IR peak positions observed and recorded include the absorption peaks around wave number range of 602–630  $\text{cm}^{-1}$ , 655–689  $\text{cm}^{-1}$ , 950–1150  $\text{cm}^{-1}$ , 1221–1250  $\text{cm}^{-1}$  and 1362–1385  $\text{cm}^{-1}$ . The assignment of each absorption peak is presented in Table 2.

Table 3 and Fig. 4 present the variation of the trigonal pyramidal ( $\text{TeO}_3$ ) and trigonal bipyramidal ( $\text{TeO}_4$ ) structural units against the  $\text{Er}_2\text{O}_3$  NPs molar concentration.

**Table 3** Relative areas of the peaks deconvoluted for the study of FTIR spectra of the  $\{[(\text{TeO}_2)_{0.7} (\text{B}_2\text{O}_3)_{0.3}]_{0.8} (\text{SiO}_2)_{0.2}\}_{1-y} (\text{Er}_2\text{O}_3 \text{ NPs})_y$  glasses

Y	$\text{TeO}_4$	$\text{TeO}_3$	$\text{BO}_3$	$\text{BO}_3/\text{BO}_2\text{O}$
0.01	784	113	9.5	17.458
0.02	748.75	128.62	7.9421	12.851
0.03	800.12	126.6	8.422	13.573
0.04	898.9	117.84	8.67	13.879
0.05	988.5	83.68	10.277	15.084



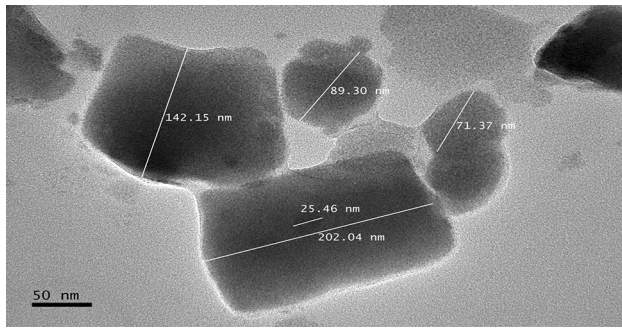
**Fig. 4** Variation of concentration of  $\text{TeO}_3$  and  $\text{TeO}_4$  structural units with molar fraction  $\text{Er}_2\text{O}_3$  NPs in  $\{[(\text{TeO}_2)_{0.7} (\text{B}_2\text{O}_3)_{0.3}]_{0.8} (\text{SiO}_2)_{0.2}\}_{1-y} (\text{Er}_2\text{O}_3 \text{ NPs})_y$  glass system

Increase in the  $\text{TeO}_4$  concentration is associated with increase in oxygen atoms bonding in the glass network while the  $\text{TeO}_3$  concentration increase relates to the increase in the oxygen atoms bond breakage.  $\text{BO}_3$  concentration presented in Table 2 is from the area under the curve around 1221  $\text{cm}^{-1}$  and 12,250  $\text{cm}^{-1}$  [26]. While the increase in the  $\text{BO}_3$  concentration represents the area under the curve around 1362–1385  $\text{cm}^{-1}$  [27]. Thus, with  $\text{TeO}_4$  concentration increase we have more bridging oxygen atoms created and  $\text{TeO}_3$  creates more non-bridging oxygen atoms in the glass network [28].

The TEM microstructure of the studies  $\text{Er}_2\text{O}_3$  NPs doped rice husk silicate borotellurite glass system is presented in Fig. 5.  $\text{Er}_2\text{O}_3$  NPs of various sizes can be observed in the

**Table 2** FTIR spectral peak assignment for  $\{[(\text{TeO}_2)_{0.7} (\text{B}_2\text{O}_3)_{0.3}]_{0.8} (\text{SiO}_2)_{0.2}\}_{1-y} (\text{Er}_2\text{O}_3 \text{ NPs})_y$  glass system

Position of FTIR peak ( $\text{cm}^{-1}$ )	Assignment of IR
602–630	Te–O bond vibrations between the trigonal bipyramid unit ( $\text{TeO}_4$ ) and the bridging oxygen atom [9]
655–689	Te–O bond asymmetric vibrations in the $\text{TeO}_3$ units [5]
950–1150	Si–O–Si asymmetric stretching of the bridging oxygen atoms of silica [2]. The peak position might also be attributed to B–O stretching vibration in $\text{BO}_4$ structural unit [8]
1221–1250	Stretching vibrations of B–O in the $\text{BO}_3$ units in boroxol rings [19]
1362–1385	Asymmetric stretching modes of borate triangles $\text{BO}_3$ , $\text{BO}_2\text{O}$ with NBO [25]



**Fig. 5** TEM micrograph of  $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.8}(\text{SiO}_2)_{0.2}\}_{1-y}(\text{Er}_2\text{O}_3)_y$  glass with 3%  $\text{Er}_2\text{O}_3$  NPs

diagram with larger particles of micro-sizes. The larger particles might have formed from the fusion of small nanoparticles through Oswald ripening effect in the glass network, which happened after the formation of glass [28].

The values of the ultrasonic velocities of the system of Erbium NPs doped RHSBT glasses were presented in Table 4. The ultrasonic wave velocities were calculated as;

$$v = \frac{2x}{t} \tag{3}$$

$$v_m = \left( \frac{v_L^3 + v_s^3}{3} \right)^{\frac{-1}{3}} \tag{4}$$

where  $v$  is the longitudinal ( $V_L$ ) or shear ( $V_s$ ) velocity,  $t$  is time of travel of the ultrasonic wave to and fro inside the glass sample and  $x$  is the sample thickness and  $v_m$  is the mean ultrasonic velocity [29]. The longitudinal, shear and the mean ultrasonic velocities for the studied glasses increased from 3722.15 to 4087.26 m/s, 2214.38 to 2345.19 m/s and 2994.21 to 3190.21 m/s respectively as  $\text{Er}_2\text{O}_3$  NPs concentration was increased 1–5% molar composition. The velocities are directly related to the material density and hence increase in material density may cause an increase in the values of the ultrasonic velocities [30]. The increase in the values may also be associated with the increased compactness in the glass network which

resulted from increase in the number of bridging oxygen (BO) in the network [31].

Table 4 and Fig. 6 present the elastic moduli of the Erbium NPs doped RHSBT glasses. The elastic [Longitudinal (L), Shear (G), Young (E) and Bulk (K)] moduli were calculated using the data of density ( $\rho$ ), longitudinal ( $V_L$ ) and Shear ( $V_s$ ) velocities as reported by [17] as;

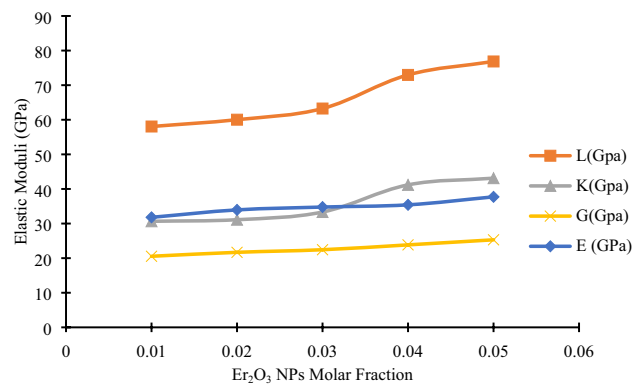
$$G = V_s^2 \rho \tag{5}$$

$$L = V_L^2 \rho \tag{6}$$

$$K = L - \frac{4}{3}G \tag{7}$$

$$E = (1 - \sigma)2G \tag{8}$$

The longitudinal, shear, young and bulk moduli increased from 58.050 to 76.853 GPa, 20.546 to 25.302 GPa, 31.801 to 37.720 GPa and 30.656 to 43.117 GPa respectively. The increase in the elastic moduli has a connection to the fact that the material connectivity in the glass system increased with the increase in  $\text{Er}_2\text{O}_3$  NPs content. This is due to the formation of more  $\text{TeO}_4$  units in the glass network which suggests a decrease in the formation of NBO



**Fig. 6** Elastic moduli variation with molar fraction of  $\text{Er}_2\text{O}_3$  NPs in  $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.8}(\text{SiO}_2)_{0.2}\}_{1-y}(\text{Er}_2\text{O}_3)_y$  glass system

**Table 4** Longitudinal Velocity ( $V_L$ ,  $\text{ms}^{-1}$ ), Shear Velocity ( $V_s$ ,  $\text{ms}^{-1}$ ), Mean Velocity ( $V_m$ ,  $\text{ms}^{-1}$ ), Longitudinal Modulus (L, GPa), Shear Modulus (G, GPa), Bulk Modulus (K, GPa) and Young Modulus (E, GPa) of  $\text{Er}_2\text{O}_3$  NPs in  $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.8}(\text{SiO}_2)_{0.2}\}_{1-y}(\text{Er}_2\text{O}_3)_y$  Glass System

Y	$V_L$ (m/s)	$V_s$ (m/s)	$V_m$ (m/s)	L (GPa)	G (GPa)	K (GPa)	E (GPa)
0.01	3722.15	2214.38	2994.21	58.050	20.546	30.656	31.801
0.02	3745.94	2251.51	3038.92	60.026	21.685	31.112	33.951
0.03	3815.30	2272.34	3071.98	63.249	22.436	33.335	34.770
0.04	4038.99	2308.72	3142.49	72.969	23.842	41.180	35.412
0.05	4087.26	2345.19	3190.21	76.853	25.302	43.117	37.720

and hence more rigid network formation [32, 33]. The elastic moduli increase might also result from the increase in the overall stretching force constant of the glasses. This might be attributed to the modifier role of the Er<sup>3+</sup> ions in the glass network. The Er<sup>3+</sup> ions increase can cause an increase in the coulomb contribution to the lattice energy in the glass system [34].

The micro-hardness and fractal bond connectivity values for different Nano Er<sub>2</sub>O<sub>3</sub> concentrations in the Nano Erbium Doped RHSBT Glass System are presented in Fig. 7 and Table 5. The micro-hardness (*H*), Poisson ( $\sigma$ ) and the fractal bond connectivity (*d*) were calculated as follows;

$$d = 4G/K \tag{9}$$

$$\sigma = \frac{(L - 2G)}{2(L - G)} \tag{10}$$

$$H = \frac{(1 - 2\sigma)E}{6(1 + \sigma)} \tag{11}$$

The variation in the micro-hardness as can be observed shows direct proportionality with the fractal bond connectivity in Fig. 7 and inverse proportionality with the Poisson ration in Fig. 8. By definition, micro-hardness refers to material’s resistance to penetration or indentation [29]. The increase observed in the micro-hardness value is an indication of an increased rigidity and network connectivity in the glasses and the decrease indicates the opposite [17]. The fractal bond connectivity value changed from 2.6808 to 2.7880 and then to 2.3472 as the Nano Er<sub>2</sub>O<sub>3</sub> concentrations increased from 1 to 5% in the glass network. This is an indication of the structural change from tetrahedral structure (3D) to a 2D layer or structure [29].

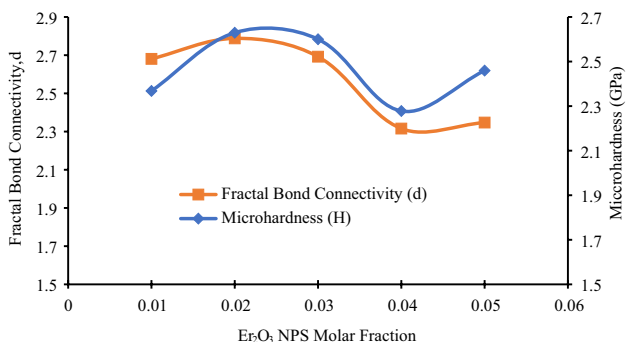
The Poisson’s ratio of a material gives a measure of the resistance of the material to volume change as well as to shape change. It can also be defined as the negative of

**Table 5** Micro-hardness (*H*), Poisson Ratio ( $\sigma$ ), Softening Temperature (*T<sub>s</sub>*), Fractal Bond Connectivity (*d*) Values for Er<sub>2</sub>O<sub>3</sub> NPs in  $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.8} (SiO_2)_{0.2} \}_{1-y} (Er_2O_3 \text{ NPs})_y$  Glass System

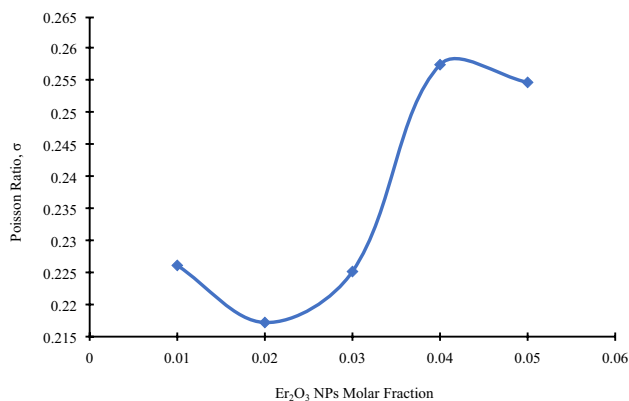
Y	H (GPa)	$\sigma$	T <sub>s</sub> (K)	$\theta_D$ (K)	D
0.01	2.3681	0.2261	1046.78	536.984	2.6808
0.02	2.6293	0.2172	1083.11	545.957	2.7880
0.03	2.6002	0.2251	1109.43	552.973	2.6922
0.04	2.2780	0.2573	1135.87	566.605	2.3158
0.05	2.4594	0.2546	1162.97	576.050	2.3473

the ratio of contraction of transverse strain to extension of longitudinal strain in the applied force direction [35, 36]. Poisson’s ratio is small for shear resistant but compressible materials such as cellular solids and can reach 0.5 for incompressible bodies such as rubber. Glasses fall in between these two and have values from 0.1 to 0.4 and specifically for oxide glasses mainly in the 0.16–0.3 with highly polymerized silica rich glass having the lowest value of theoretical volume occupied (*V*) and the highest for glass networks consisting of chains and cluster units [37].

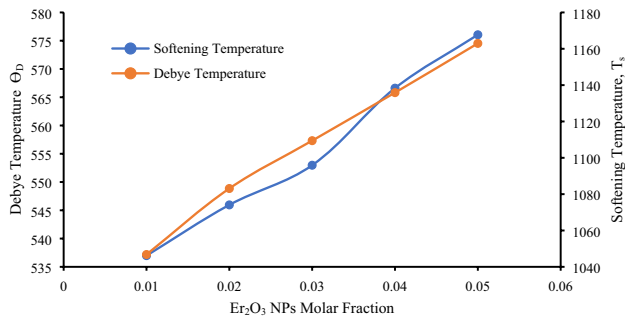
The Poisson ratio relation with the concentration of Er<sub>2</sub>O<sub>3</sub> NPs in an erbium NPs doped RHBT glass system is presented in Fig. 8. The relation showed a decrease in the value from 0.22609 to 0.217202 as the Er<sub>2</sub>O<sub>3</sub> NPs molar concentration increased from 1 to 2%. The value then increased to 0.257384 when the Er<sub>2</sub>O<sub>3</sub> NPs molar concentration was increased to 4% which the decreased to 0.25459 when the Er<sub>2</sub>O<sub>3</sub> NPs molar concentration reached 5%. The decrease in the Poisson’s ratio value with increase in Er<sub>2</sub>O<sub>3</sub> NPs molar concentration may be due to an increase in the glass ionicity which is caused by more polar bonding presence in the modifiers [34]. The increase in the Poisson ratio may be associated to increase in the crosslink density in the glass network [38].



**Fig. 7** Micro-hardness and fractal bond connectivity variation with molar fraction of Er<sub>2</sub>O<sub>3</sub> NPs in  $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.8} (SiO_2)_{0.2} \}_{1-y} (Er_2O_3 \text{ NPs})_y$  glass system



**Fig. 8** Poisson ratio variation with molar fraction of Er<sub>2</sub>O<sub>3</sub> NPs in  $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.8} (SiO_2)_{0.2} \}_{1-y} (Er_2O_3 \text{ NPs})_y$  GLASS system



**Fig. 9** Softening temperature and Debye temperature variation with Er<sub>2</sub>O<sub>3</sub> NPs molar fraction in  $\{[(\text{TeO}_2)_{0.7} (\text{B}_2\text{O}_3)_{0.3}]_{0.8} (\text{SiO}_2)_{0.2}\}_{1-y} (\text{Er}_2\text{O}_3 \text{ NPs})_y$  glass system

The Debye and softening temperatures' variation with concentration of Er<sub>2</sub>O<sub>3</sub> NPs are presented in Fig. 9 and Table 5. The softening temperature ( $T_s$ ) and the Debye temperature ( $\theta_D$ ) were calculated using the following standard relations as follows;

$$T_s = \frac{M_w}{c\rho} V_s^2 \quad (12)$$

$$\theta_D = \frac{h}{k} \left[ \frac{9PNa}{4\pi} \right]^{\frac{1}{3}} v_m \quad (13)$$

where  $M_w$  is the glass molecular weight,  $v_m$  is the mean ultrasonic velocity,  $h$  is the Plank's constant,  $P$  is the number of atoms in the chemical formula,  $k$  is the Boltzmann constant,  $c = 1.35 \times 10^9 \text{ cm}^5 \text{ K}^{-1} \text{ s}$  is constant,  $N_a$  is the Avogadro's number,  $\rho$  is the density of the given sample and  $V_s$  is the ultrasonic shear velocity inside the glass [17, 18].

As the concentration of Er<sub>2</sub>O<sub>3</sub> NPs increased from 1 to 5% in the glass system, the values of the softening temperature and Debye temperature increased from 1046.78 to 1162.97 K and 536.984 to 576.050 K respectively. Debye temperature provides the description properties related to atomic vibrations in a solid material; it represents the excitation temperature of high frequency lattice in any material under study. In glassy materials, network vibrational spectrum are considered as a diffuse version of the lattice spectrum of the equivalent crystal nearest to it [39]. The increase in the value of the Debye temperature recorded can be ascribed to stronger packing and increase connectivity in the glass system arising from more bonding of single bonded oxygen atoms as well as increased lattice vibrations in the glass network [40]. Whereas the softening temperature represents the temperature at which viscous flow translates to plastic flow in a material. The increase in  $T_s$  value with Er<sub>2</sub>O<sub>3</sub> NPs composition increase might be associated with increase in the rigidity and connectivity because of increased connectivity. The

increased connectivity might as well be attributed to the observed TeO<sub>4</sub> concentration increase and TeO<sub>3</sub> concentration decrease in the glass structure, which signifies more bonding of oxygen atoms in the glasses [17].

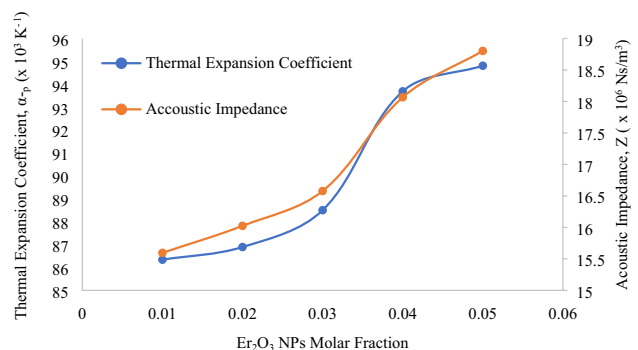
The variation of thermal expansion coefficient ( $\alpha_p$ ) and acoustic impedance ( $Z$ ) with Er<sub>2</sub>O<sub>3</sub> NPs concentration is presented in Fig. 10 and Table 6. The acoustic impedance and the thermal expansion coefficient were determined from the longitudinal ultrasonic data using the following expressions as reported by Marzouk and Gafaar [31] as follows;

$$Z = V_L \rho \quad (14)$$

$$\alpha_p = 23.2(V_L - 0.57457) \quad (15)$$

In glasses, the thermal expansion coefficient value and its transition temperature are interdependent. The value is also dependent on other parameters which includes the composition and nature of glass modifier, temperature region as well as thermal history of the glasses [41]. The increase in the linear thermal expansion coefficient represents a decrease in the thermal stability of the glasses with increase in Er<sub>2</sub>O<sub>3</sub> NPs composition [42]. Acoustic impedance is a parameter responsible for both reflection and transmission of sound energy inside a material [31]. Increase in the value recorded for the studied glass system can be ascribed to atomic compactness and glass network rigidity [43].

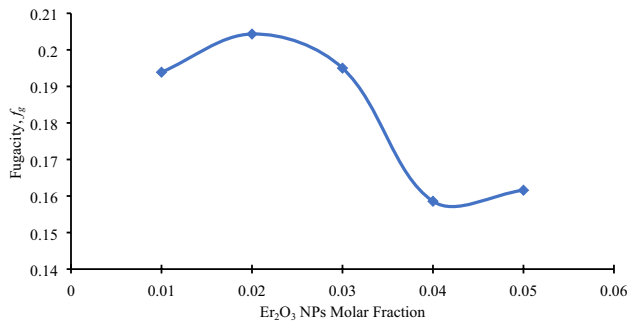
The variation of the fugacity value with Er<sub>2</sub>O<sub>3</sub> NPs molar concentration is presented in Fig. 11. This is the proportion of free volume at the temperature of glass transition. The variation in the fugacity as shown in the figure indicates increase and then a decrease in the value. This is because of an interstitial space increase in the beginning with increase in the Er<sub>2</sub>O<sub>3</sub> NPs molar concentration resulting from the formation of more NBOs. The decrease in the



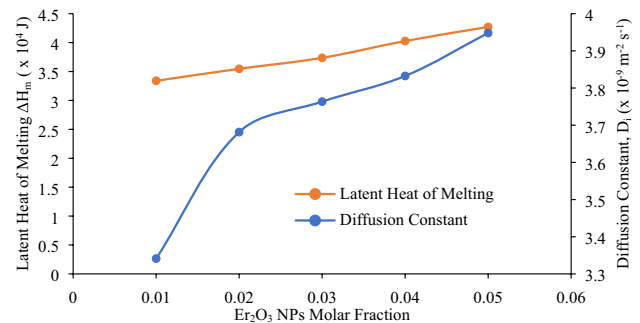
**Fig. 10** Acoustic impedance and thermal expansion coefficient variations with Er<sub>2</sub>O<sub>3</sub> NPs molar fraction in  $\{[(\text{TeO}_2)_{0.7} (\text{B}_2\text{O}_3)_{0.3}]_{0.8} (\text{SiO}_2)_{0.2}\}_{1-y} (\text{Er}_2\text{O}_3 \text{ NPs})_y$  glass system

**Table 6** Fugacity ( $f_g$ ), Acoustic Impedance ( $Z$ ), Thermal Expansion Coefficient ( $\alpha_p$ ), Diffusion Constant ( $D_i$ ), Latent Heat of Melting ( $\Delta H_m$ ) values for different  $Er_2O_3$  NPs Molar Fraction in  $\{(TeO_2)_{0.7}(B_2O_3)_{0.3}\}_{0.8}(SiO_2)_{0.2}\}_{1-y}(Er_2O_3\ NPs)_y$  Glass System

$y$	$f_g$	$Z (\times 10^6\ Ns/m^3)$	$\alpha_p (\times 10^3\ K^{-1})$	$D_i (\times 10^{-9}\ m^{-2}\ s^{-1})$	$\Delta H_m (J)$
0.01	0.1939	15.5958	86.3406	3.3412	33,395.71516
0.02	0.2043	16.0244	86.8925	3.6816	35,466.82031
0.03	0.1950	16.5779	88.5016	3.7635	37,366.15985
0.04	0.1586	18.0660	93.6913	3.8325	40,271.45791
0.05	0.1616	18.8030	94.8111	3.9482	42,710.3687



**Fig. 11** Variation of fugacity with molar fraction of  $Er_2O_3$  NPs in  $\{(TeO_2)_{0.7}(B_2O_3)_{0.3}\}_{0.8}(SiO_2)_{0.2}\}_{1-y}(Er_2O_3\ NPs)_y$  glass system



**Fig. 12** Latent heat of melting and diffusion constant variation with molar fraction of  $Er_2O_3$  NPs in  $\{(TeO_2)_{0.7}(B_2O_3)_{0.3}\}_{0.8}(SiO_2)_{0.2}\}_{1-y}(Er_2O_3\ NPs)_y$  glass system

value afterwards indicates an increase in material compactness as the  $Er_2O_3$  NPs molar concentration is increase. The decrease in the fugacity may as well be considered to be due to an increase in the concentration of bridging oxygen (BO) in the glass network [44]. This parameter also related to the Poisson ratio and is a factor of material permeability in the glass network [45].

The calculations of the latent heat of melting ( $\Delta H_m$ ) and the diffusion constant ( $D_i$ ) were carried out using the expressions as reported by Chinnamat et al. [29], as presented in Eqs. (16) and (17) as follows;

$$\Delta H_m = \frac{9M\theta_D^2 r_i^2 k_B^2}{128h^2} \tag{16}$$

$$D_i = \frac{k_B\theta_D r_i^2}{96h} \tag{17}$$

The Variation of diffusion constant and latent heat of melting with concentration of  $Er_2O_3$  NPs in the studied glass system is presented in Fig. 12. The diffusion constant value for the studied glasses increased from  $3.3412 \times 10^{-9}$  to  $3.9482 \times 10^{-9}\ m^{-2}\ s^{-1}$  when the concentration of  $Er_2O_3$  NPs was increased from 1 to 5 mol%. The increase in the value might be connected to increase in the ionic bond length resulting from the substitution of other oxides with shorter bond length with longer bonds of  $Er_2O_3$  NPs, making the overall bond length of the glasses longer [29].

The increase might also be connected to increase in the number of vibrating atoms in the glass network [31]. The latent heat of melting increased in value from 33.397 to 42.7104 kJ as the  $Er_2O_3$  NPs composition increased from 1 to 5 mol%. Increase in the value together with increase in the diffusion constant confirmed the increase in the Debye's and softening temperatures [31, 46].

## 2 Conclusion

Using the agricultural waste (rice husk), silica was extracted with 98.584% purity and was used in the fabrication of a system of erbium doped silica borotellurite glasses with chemical composition  $\{(TeO_2)_{0.7}(B_2O_3)_{0.3}\}_{0.8}(SiO_2)_{0.2}\}_{1-y}(Er_2O_3\ NPs)_y$  using the method of melt-quenching. The aim was to improve the rice husk commercialization from its current 30% in the area of Erbium doped fiber amplifier technology. The glasses were subjected to various measurements and characterizations which include density and molar volume, EDX, XRD, TEM, FTIR and the pulse-echo non-destructive ultrasonic technique for the study of the glasses' structural and elastic properties. The measured density values increased from 4.1900 to 4.6004  $gcm^{-3}$  and the XRD patterns revealed the amorphous nature of the glasses. FTIR spectral study showed the presence of  $TeO_4$ ,  $TeO_3$ ,  $SiO_4$ ,  $H_2BO_3$ , and two different structural units of



$\text{BO}_3$ , with the TEM revealing the presence of  $\text{Er}_2\text{O}_3$  NPs and larger particles of agglomerated NPs of  $\text{Er}_2\text{O}_3$  were formed through Oswald ripening effect. The ultrasonic velocities as well as elastic moduli increased with increased  $\text{Er}_2\text{O}_3$  NPs concentration. The microhardness, softening temperature, Debye temperature, thermal expansion coefficient, acoustic impedance, diffusion constant, fractal bond connectivity, fugacity as well as the latent heat of melting were determined for the studied glasses. Based on the studied parameters, the glasses proved to be strong enough to stand fiber drawing and thermal stress.

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### Compliance with ethical standards

**Conflict of interest** The authors wish to state that there is no conflict of interest whatsoever in this work and all authors who contributed to the development of this article have been listed and financial contributors duly acknowledged.

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