

Influence of Sm_2O_3 Ion Concentration on Structural and Thermal Modification of TeO_2 - Na_2O Glasses

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Abstract

The effect of Sm^{3+} ions concentration doped TeO_2 - Na_2O glasses on structural and thermal parameters have been discussed. Glass samples with molar composition $(80-x)\text{TeO}_2$ - $20\text{Na}_2\text{O}$ - $x\text{Sm}_2\text{O}_3$ glasses ($x=0, 0.3, 0.6, 1, 1.2, 1.5$) are prepared by melt quenching technique. Crystallization temperature (T_c), melting temperature (T_m) and glass transition temperature (T_g) are measured by using differential thermal analysis (DTA), it is found that the stability factor (ΔT) increases from $(58.5-97.8)^\circ\text{C}$ with the increasing of Sm_2O_3 . The amorphous phase nature of the glass samples are observed by using X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive x-ray (EDX) spectrometer are applied to study the structural properties of the glass samples. Values of density (ρ), molar volume (V_M), and ionic packing density (V_i) were calculated for each of the glass compositions. The effect of the Sm_2O_3 on the glass structure have been investigated by using FTIR and Raman spectroscopies, the FTIR spectra are characterized by a band of 637 cm^{-1} for the telluride glass, high frequency peak at 668 cm^{-1} presented by Raman spectra which indicates that these glasses network are basically consists of TeO_4 and $\text{TeO}_3/\text{TeO}_{3+1}$ structural units. The spectra of Raman shows the presence of Sm-O bond, Na-O bond, Te-O-Te bridging configurations, vibrations of Te-O-Te bonds and stretching modes of non-bonding oxygen found on the $\text{TeO}_3/\text{TeO}_{3+1}$ structural unit.

Keywords: Telluride glass; Rare Earth; Glass transition; FTIR; Raman spectra

Introduction

Under normal quenching conditions tellurium dioxide (TeO_2) does not have the ability of forming a glassy network without the assistant of the secondary material component which is called a network modifier. This specific feature makes TeO_2 a conditional glass former [1,2]. Recent studies indicate that heavy metal oxide (HMO) glasses have been found to be more affirmative convenient glassy materials especially for photonic applications with acceptable low phonon energies [3,4], their wide advantages of tellurite glasses makes them to be represented as a more attractive structure among the HMO glasses such as low temperature of melting, good corrosion resistance, excellent chemical durability, low glass transition temperature T_g , better thermal stability, high thermal expansion coefficient, low phonon energy, wide optical transmission region (especially in the infrared region) ($0.36\text{ }\mu\text{m}$ - $6.3\text{ }\mu\text{m}$), high refractive index and required rare earth concentration in the matrices [5]. Basically, the structure of TeO_2 glasses contains of three dimensional network of TeO_4 trigonal bipyramids (tpb) units having two each of oxygen at two equatorial and axial sites with one other equatorial site being occupied by a lone pair of electrons, once the modifier is introduced to the structure, the three dimensional network will break down with the conversion of TeO_4 units into TeO_{3+1} and TeO_3 units [6,7]. In addition to the glass former conditionality and fast quenching requirements properties of tellurite glasses, they are also counted as a good host for the formation of rare earth doped glasses. The reason behind this characteristic is the Te-O bonds are weak and can easily be broken by heavy metal and the rare earth atoms can enter in the glass networks [8]. In the literature there are relatively few reports on structural of glasses doped with Sm_2O_3 ions [9,10]. Because of that reason the present work is to investigate the influence of Sm^{3+} ion on the structural and thermal properties of TeO_2 - Na_2O glasses, the thermal modification of the glass system were analyzed by DTA technique and the structure of the Sm_2O_3 doped glasses has been studied through Raman and FTIR Spectra.

Experimental Procedure

TeO_2 - Na_2O - Sm_2O_3 glass samples having compositions $(80-x)\text{TeO}_2$ - $20\text{Na}_2\text{O}$ - $x\text{Sm}_2\text{O}_3$ with ($x=0,0.3,0.6,1,1.2,1.5$) are prepared using melt quenching technique, glasses code of the sample and their molar ratio of the compositions are listed in Table 1. High purity 99.9% of raw materials from Sigma Aldrich with an appropriate amounts of chemicals for 15 gm batch were weighed by using electronic balance Precisa 205A SCS model with accuracy $\pm 0.001\text{ gm}$, the chemical compositions were mixed well by using a milling machine. Platinum crucible of about 30 ml capacity containing the batch was used and preheated at 250°C for about 20 min in order to reduce the batch blanket coverage on the top of the glass and enlarges the free non coverage glass melt surface [11], then the batch is melted at 900°C for 40 min by a controlled electric furnace. The melts are poured on to a stainless steel mold and annealed at 250°C for 3 hours, then the sample cooled down until its temperature reaches the room temperature, sample powder of the glass prepared and used for characterizing each of T_g , T_c and T_m by Pyris Dymond TG/DTA technique at a heating rate 50°C to 1000°C at $10^\circ\text{C}/\text{min}$. To analysis the amorphous state of the samples an advanced powder XRD Bruker D8 were used, the CuK α radiation specification was (1.54 \AA) at 40 kV, 100 mA and scanning angle 2θ ranges between 10° - 80° . A Perkin-Elmer FTIR double beam spectrometer over the range 400 - 4000 cm^{-1} are used for studying the transmission measurements, the spectra resolution of 4 cm^{-1} at room temperature for samples made by

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using KBr pellets were investigated. Raman measurement is performed using a confocal Horbia Jobin Yvon (Model HR800 UV) with Argon ion laser (excitation wavelength 514.55 nm) operates at 20 mW in the range of 100-900 cm^{-1} . Relatively fine glass powders are used for Raman and FTIR measurements.

Traditional Archimedes method is used for determining the density of the glass sample after measuring the weight of the sample by using a sensitive analytical balance Precisa XT220A, toluene ($\rho_t=0.8669 \text{ g cm}^{-3}$) is used as an immersed liquid. The density ρ is calculated using the expression [12]:

$$\rho = \left[\left(\frac{W_a}{W_a - W_t} \right) * \rho_t + \rho_a \right] \quad (1)$$

Where W_a and W_t are weight of the glass sample in air and in toluene respectively, ρ_a is the density of air and ρ_t is the density of the toluene. The molar volume (V_M) of the glasses was calculated from density values according to [13]:

$$V_M = \frac{M}{\rho} \quad (2)$$

where M is the molar weight and ρ is the density of the glass. The ionic packing density (V_i) is calculated using Makishima and Mackenzie approach [13,14],

$$V_i = \left(\frac{1}{V_M} \right) * \sum (V_i * x_i) \quad (3)$$

Where x_i is the mole fraction (mol%) and V_i is packing density parameter (m^3/mol). For an oxide glass of the form M_xO_y , the value of V_i yields [13],

$$V_i = \left(\frac{4\pi N_A}{3} \right) \left[Xr_M^3 + Yr_o^3 \right] \quad (4)$$

where N_A is Avogadro's number (mol^{-1}), r_M and r_o are the Shannon's ionic radius of metal and oxygen, respectively. Table 1 represents the glass sample code and their molar ratio effect on each of the density, molar volume and ionic packing density. The effect of Sm_2O_3 % on both of the density and molar volume for each composition of the glass samples is shown in Figure 1, from Figure 1 it can be seen that the density of TNS glass system increases from 4.903 g cm^{-3} for $x=0$ mol% to 5.019 g cm^{-3} for $x=1.2$ mol% of Sm^{3+} ion. Higher molecular mass of Sm_2O_3 (348.74 g mol^{-1}) and those for TeO_2 (159.60 g mol^{-1}) might be responsible for increase its density [15]. The decrease in density at $x=1.5$ mol% might be caused by Sm^{3+} ions which take a part in the structure of glass and make the density to be decreased [16]. A molar volume of the TNS glass system which is plotted against Sm_2O_3 % in the Figure 1 reveals that the molar volume increased from TNS1 to TNS3 glasses as the Sm^{3+} content increased from 0 mol% to 0.6 mol%, while the molar volume of glass TNS4 and TNS5 decreased with increasing of Sm^{3+} content, this effect can be explained as the Sm^{3+} mole friction content more increased the structure of glasses will get more compact due to increase in packing density of oxygen [17] and this indicates that the structure of TNS glasses changed, this behavior of molar volume is in consistent with the increasing behavior of rigidity and compactness of glass samples [18] (Table 1).

Results and Discussion

XRD and SEM-EDX spectral analysis

In order to check the non-crystalline nature of the glass samples, the XRD measurement is performed for all samples and the result can be seen in Figure 2. From Figure 2 it can be seen that the results did not reveal any kinds of sharp peaks, therefore, proving the amorphous

Sample Code	TeO_2 %	Na_2O %	Sm_2O_3 %	M g mol^{-1}	V_t	V_M $\text{cm}^3 \text{mol}^{-1}$	ρ g cm^{-3}
TNS1	80	20	0	140.07	0.4012	28.571	4.903
TNS2	79.7	20	0.3	140.64	0.4017	28.623	4.914
TNS3	79.4	20	0.6	141.21	0.4019	28.700	4.920
TNS4	79	20	1	141.97	0.4051	28.593	4.965
TNS5	78.8	20	1.2	142.34	0.4092	28.359	5.019
TNS6	78.5	20	1.5	142.91	0.4025	28.919	4.942

Table 1: Glasses sample code and calculated density, molar volume and ionic packing density.

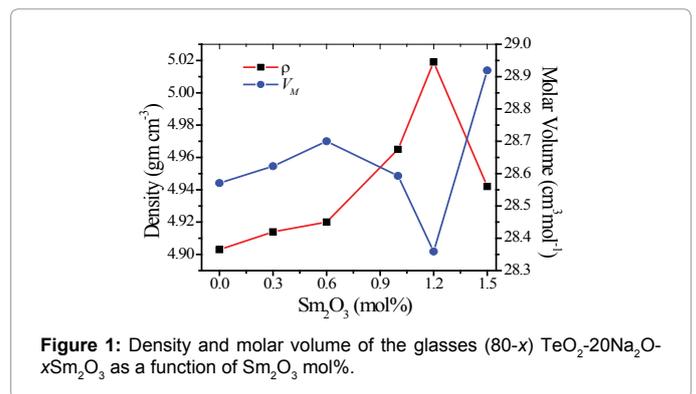


Figure 1: Density and molar volume of the glasses (80-x) $\text{TeO}_2\text{-}20\text{Na}_2\text{O-xSm}_2\text{O}_3$ as a function of Sm_2O_3 mol%.

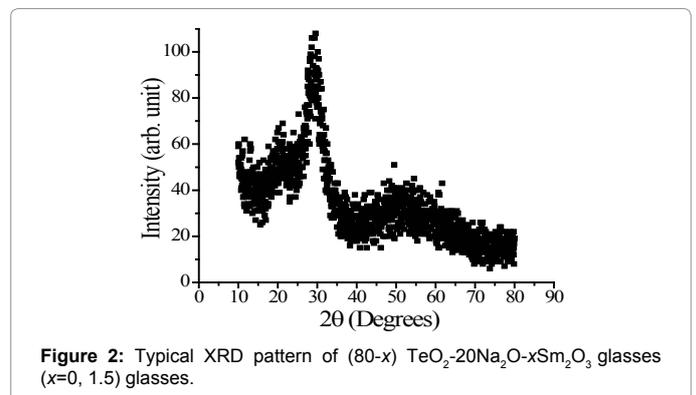


Figure 2: Typical XRD pattern of (80-x) $\text{TeO}_2\text{-}20\text{Na}_2\text{O-xSm}_2\text{O}_3$ glasses ($x=0, 1.5$) glasses.

nature structure of the present glass.

SEM-EDX investigations were performed on the (80-x) $\text{TeO}_2\text{-}20\text{Na}_2\text{O-xSm}_2\text{O}_3$ with ($x=1$) % mole fraction glasses in order to identify changes in morphology and chemical composition. No crystals were detected by SEM in any of the investigated glasses. The SEM micrograph in Figure 3a for the glass containing 1% mole fraction of Sm_2O_3 showing homogeneous glassy phase is typical for these glasses. The EDX analysis are shown in Figure 3b on several different spots on glass sample gives almost identical spectra confirming homogeneous character of the sample. The chemical composition calculated from EDX spectra is in good agreement with nominal composition of the glass. Similar results were obtained for all investigated $\text{TeO}_2\text{-Na}_2\text{O-Sm}_2\text{O}_3$ glasses.

DTA spectral analysis

The DTA thermograms for (80-x) $\text{TeO}_2\text{-}20\text{Na}_2\text{O-xSm}_2\text{O}_3$ glasses sample are shown in Figure 4. Endothermic peaks due to the glass transition and melting point and exothermic peak due to the crystallization are clearly observed. The T_g value of 80 $\text{TeO}_2\text{-}20 \text{Na}_2\text{O}$

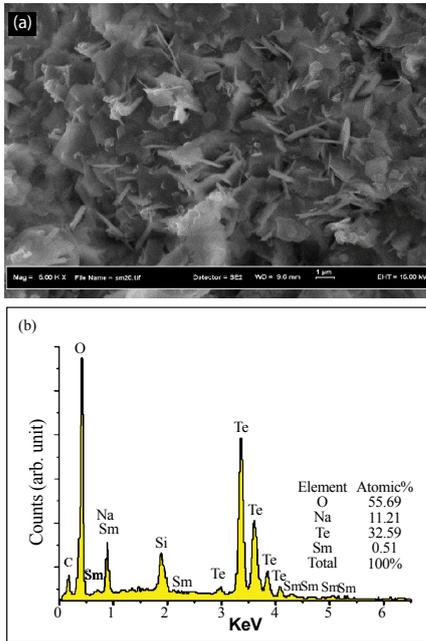


Figure 3: (a) SEM micrograph and (b) EDX spectra of TNS4 sample.

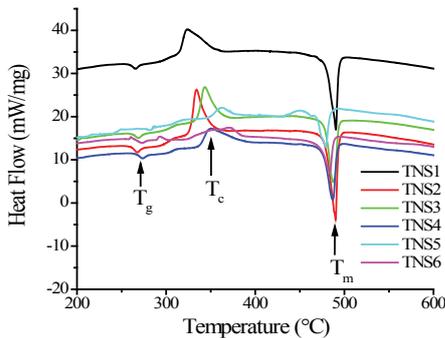


Figure 4: DTA traces of the (80-x) TeO₂-20Na₂O-xSm₂O₃ glasses at a heating rate is 10°C/min.

have been determined to be 265.8°C and by adding 0.3 mol% Sm₂O₃ into the composition it increased T_g to 267.6°C. Obtained results indicate that by increasing the amount of mol% Sm₂O₃, the T_g of the samples also increases, the small increase of T_g in these glasses shows that the structure is strongly and progressively modified.

The thermal stabilities ΔT of the TNS reference glass and Sm⁺³: TNS glass has been evaluated from their T_g , T_c and T_m values, the results are listed out in Table 2. ΔT is generally used as a rough measure of glass thermal stability and it is desirable for a glass host to have ΔT as large as possible [19]. The higher the ΔT is, the stronger the inhibition of nucleation and crystallizations will occur [20]. The ΔT of present glasses are calculated to be in the range of (58.5-97.8)°C, it is noted that the ΔT increases with increasing % Sm₂O₃ to the composition, results indicate that the present glass possesses good thermal stability and anti-crystallization ability. Hruby's parameter also calculated by using eq. (5), the greater values of the Hruby's parameter indicate higher glass forming tendency, the values of H in our glasses increased with the addition of the Sm₂O₃. Figure 5 shows the effect of increasing % Sm₂O₃ on ΔT , T_c , T_g , T_m and Hruby's parameter (Table 2).

$$H = \frac{T_c - T_g}{T_m - T_c} \quad (5)$$

FTIR spectral study

In order to understand the structural units of glasses studied, FTIR transmission spectra of TNS glasses and pure KBr have been recorded in the range 400-4000 cm⁻¹ as shown in Figure 6. From Figure 6 it can be seen that there are seven important absorption peaks appear in the FTIR transmission spectra. The identical peaks assignments are then listed in Table 3. For giving more detail about the FTIR spectra, the FTIR spectra are divided into three regions: 400-900 cm⁻¹, 1000-2500 cm⁻¹ and 2500-4000 cm⁻¹ as shown in Fig. (6a, 6b and 6c) respectively. The spectra in Figure 6a present three bands at optical visible range 759.6, 637.5 and 471.8 cm⁻¹ and are assigned to stretching vibrations of TeO₃ or TeO₃₊₁, TeO₄ structural units and bending vibrations of Te-O-Te or O-Te-O linkages with Na-O respectively. The change in sodium tellurite structure can be predicted as the shift in the position is attributed to the change in the bonding length predicting [21,22]. The shifts in the stretching vibration of TeO₄ and TeO₃ towards 637.5 and 759.6 cm⁻¹ are observed by increasing the Sm³⁺ ion concentration up to 1 mol%. This shift in the transmission bands is attributed to the deformation of TeO₄ group into TeO₃ through TeO₃₊₁ intermediate coordinate formation [23,24]. The ratio of 759.6/637.5 cm⁻¹ represents the relative concentrations of the TeO₃ and TeO₄ structural units, which is absolutely dependent on the glass composition.

According to the electronegativity theory, the covalency of the bond will become stronger with the decrease of the difference of electronegativity between cation and anion ions. From the periodic table, since the values of electronegativity for Te, Na, Sm and O elements are 2.1, 0.9, 1.17 and 3.5, respectively, the covalency of

Sample Name	% Sm ₂ O ₃	T_g °C	T_c °C	T_m °C	$\Delta T = T_c - T_g$ °C	$H = \frac{T_c - T_g}{T_m - T_c}$
TNS1	0	265.8	323.6	489.5	57.8	0.349
TNS2	0.3	267.6	334.1	490.2	66.5	0.426
TNS3	0.6	269.1	343.5	486.9	74.3	0.518
TNS4	1	273.8	350.4	486.4	76.6	0.563
TNS5	1.2	281.7	362.2	478.6	80.6	0.692
TNS6	1.5	273.1	370.6	483.0	97.5	0.868

Table 2: Thermal parameters determined from the DTA traces of (80-x) TeO₂-20Na₂O-xSm₂O₃.

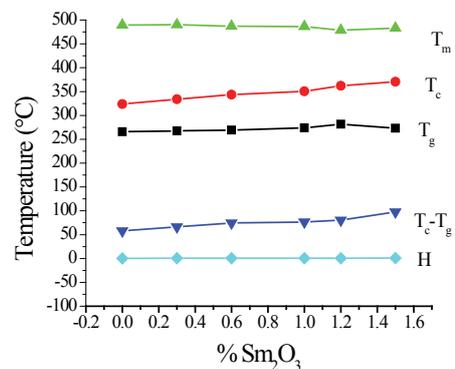
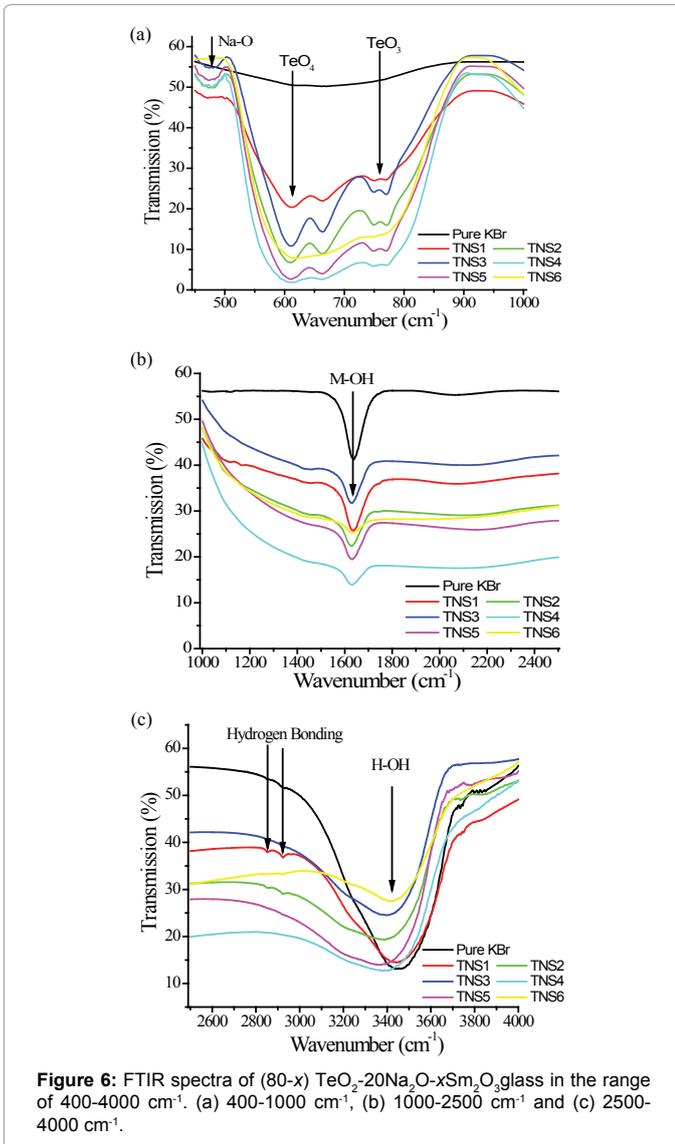


Figure 5: Effect of Sm₂O₃% on each of the on ΔT , T_c , T_g , T_m and Hruby's parameter.



Sample Code	Sm_2O_3 (mol%)	Na-O	TeO_4	TeO_3	M-OH	Hydrogen Bonding	Hydrogen Bonding	OH-Group
Pure KBr	-	-	-	-	1637.53	2855.22	2933.75	3447.77
TNS1	0	471.80	637.51	759.61	1633.99	2851.32	2923.83	3443.73
TNS2	0.3	472.19	637.21	760.20	1627.15	2853.64	2923.07	3389.21
TNS3	0.6	473.30	635.9	765.61	1629.76	2855.22	2922.24	3395.65
TNS4	1	478.36	637.31	759.35	1630.26	2768.96	2927.44	3400.04
TNS5	1.2	475.12	619.19	759.95	1623.00	2848.98	2915.79	3382.01
TNS6	1.5	476.65	636.42	759.63	1632.26	2848.92	2921.24	3370.68

Table 3: Peaks position (in cm^{-1}) in the FTIR spectra of $(80-x)\text{TeO}_2\text{-}20\text{Na}_2\text{O-xSm}_2\text{O}_3$ glass.

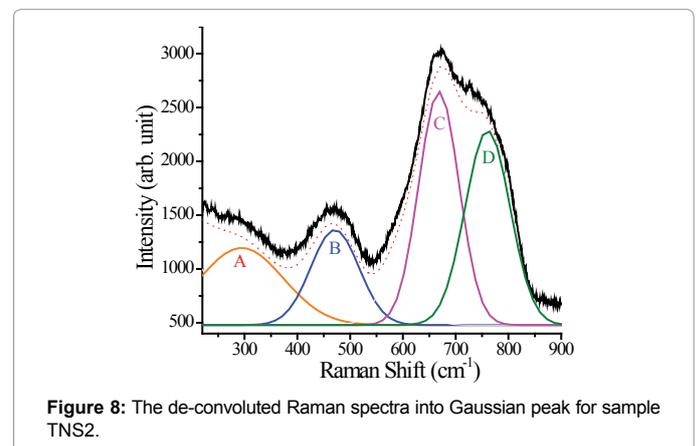
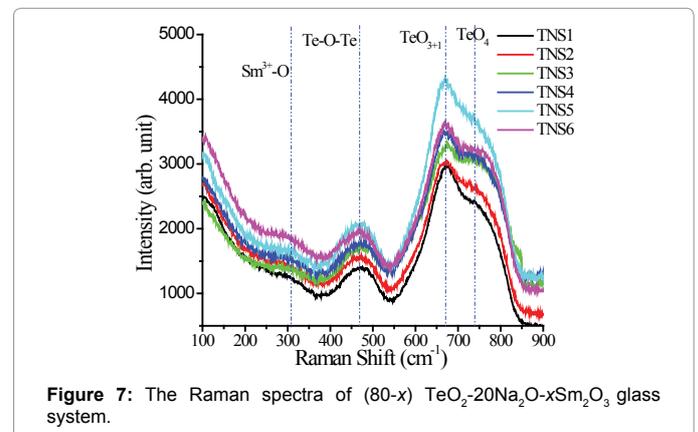
Te-O are stronger than Na-O and Sm-O, respectively. As a result, the higher affinity of the tellurium ions to attract oxygen atoms yields the apparition of TeO_4 structural units [25]. These three bands do not exist in the pure KBr spectra.

Figure 6b shows the hydroxyl-metal (M-OH) stretching vibrations bond which is observed at 1637.53 cm^{-1} . This band is shifted to 1632.26 cm^{-1} with increasing amount of Sm^{3+} concentration. This shift might

be due to the addition of rare earth in the host matrix of glasses that slightly increase the IR transmission and shift it to longer wavelength [26]. M-OH stretching vibration peaks also appeared for pure KBr sample spectra. The IR transmission bands occur around 2900 and 3400 cm^{-1} belong to stretching vibration of the hydroxyl group and hydrogen bond [27]. This is observed clearly in Figure 6c, the band ranges 2768.96-2927.44 cm^{-1} and 3400.04-3447.77 cm^{-1} which correspond to hydroxyl group and hydrogen bond, respectively (Table 3).

Raman spectra

One of the most well-known types of vibrational spectroscopy is Raman spectroscopy. It is considered as a finger print about structural information of the glass. The Raman spectra of TNS glass series in the frequency range of 100-900 cm^{-1} is shown in Figure 7, and the deconvoluted of the Raman spectra for sample TNS2 is presented in Figure 8. The observed spectrum is then fitted to Gaussian peaks and to get four distinct solid line peaks named by (A,B,C and D). The sum of these picks are represented by a dotted line which is well coincide with the obtained solid line from the Raman spectra. These peaks are centered at 294.43 (A), 471.09 (B), 668.33 (C) and 760.52 (D) cm^{-1} . The corresponding absorption peak shifts are listed in Table 4. It can be observed that the shift is dominant by all band regions around 279.4-306.63 cm^{-1} , 465.11-473.45 cm^{-1} , 668.33-684.12 cm^{-1} and 759.06-773.3 cm^{-1} . The Raman band in the region around 279.4-306.63 cm^{-1} can be assigned to both $\text{Sm}^{3+}\text{-O}$ and TeO_3 tp which indicate that the presence of rare earth ions might significantly change the Te-O networking structure in glasses [27,28]. The Raman shift corresponding to the band in a range of 465.11-473.45 cm^{-1} is due to Te-O-Te linkages vibration. Meanwhile, the Raman peaks shift in the range of 668.33-684.12 cm^{-1}



Sample Code	% Sm ₂ O ₃	Raman Shifts/ cm ⁻¹			
		A	B	C	D
TNS1	0	284.34	473.17	671.8	759.06
TNS2	0.3	294.43	471.09	668.33	760.52
TNS3	0.6	289.74	473.45	671.01	767.81
TNS4	1	303.549	467.33	682.23	770.37
TNS5	1.2	306.63	465.11	684.12	773.3
TNS6	1.5	276.94	467.54	677.61	773.02

Table 4: Raman shift peaks position in cm⁻¹ for (80-x) TeO₂-20Na₂O-xSm₂O₃.

and 759.06-773.3 cm⁻¹ are corresponding to TeO₃ bp unit and TeO₄ tbp unit respectively. It can also be observed that increasing the Sm³⁺ ions from 0 to 1.5 mol % and adding into the TNS glass will turn the glass structure to transform slightly by the perturbation of TeO₄ tbp unit into TeO₃ tp unit through TeO₃₊₁ intermediate coordination, this has been satisfied by the Raman band shift at the certain region [29,30]. Moreover, it is noted that the intensity of Raman peaks around 668.33-684.12 cm⁻¹ and peaks around 759.06-773.3 cm⁻¹ are increased by the increasing of the concentration of Sm³⁺ ions as shown in Figure 7. The change in intensity might be due to the creation of TeO₃ tp unit by perturbation of TeO₄. It is asserted that the addition of Sm³⁺ ions in TNS glasses creates more number of TeO₃ tp units (Table 4).

Conclusion

In this study, glass formation range of (80-x) TeO₂-20Na₂O-xSm₂O₃ system is prepared by melt quenching method. The XRD results how all samples are amorphous in nature. The physical parameter such as glass density, molar volume and ionic packing density were found to be (4.903-5.019) g cm⁻³, (28.359-28.919) cm³mol⁻¹ and (0.4012-0.4092) respectively. Thermal and structural properties are investigated by DTA, FTIR and Raman spectroscopies. The thermal characteristics reveal that the glass transition temperature and stability factor increases with the increasing of Sm₂O₃ mol% content. This is due to the increase in bond number per unit volume which is an indication of change in packing density in the structure. The vibrational spectra of the glass system suggested that the glass network consists of TeO₄, TeO₃/TeO₃₊₁, Na₂O and Sm₂O₃ units. Raman spectra for the (80-x) TeO₂-20Na₂O-xSm₂O₃ glasses indicated that the TeO₄ tbps convert to TeO₃ tps with the increasing of Sm₂O₃ % mol fraction. The Raman shifts due to the structural units TeO₄ and TeO₃ have almost equal to the intensities in these glasses and overlapped to each other.

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