

Structural and Physical Properties Study of Some Sodium-Strontium-Vanadium Unconventional Oxide Glasses

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Abstract: Glasses with the composition $\{(100-x) (0.75 V_2O_5+0.25 SrO) + x Na_2O\}$, $x = 0, 4, 8, 12$ and 16 , have been prepared by the melt-quenching method. The as measured IR spectra were analyzed where it appeared that VO_5 , VO_4 and VO_3 are the major structural groups in the glass network and the increase of Na_2O increases the force constant of V-O bonds. The difference between the experiment and empirical values of both density and molar volume values confirm the amorphous structural nature of the studied samples. The dc electrical conductivity has been analyzed according to Mott's small polaron hopping model, where it was found generally that, the conductivity decreased and the activation energy increased by increasing Na_2O content. The calculated total mass attenuation coefficient values show approximate stability with the variation of Na_2O and it appeared that all glasses can be used as good shielding materials for the 356 keV γ -ray energy.

Keywords: Glass, IR Spectroscopy, Density, DC Conductivity and Mass Attenuation Coefficient.

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1. Introduction

Most glasses are generally insulators in nature, while some glasses exhibit semi-conducting properties. It is well known that, glasses containing transition metal ions (TMIs) are classified as the best amorphous semi-conductors due to the usual existence of these ions in more than single valence states. The unpaired $3d^1$ electron of vanadium induces polarization for the surrounding lattice and the interaction between electrons and lattice is strong enough to form small polaron [1, 2]. Therefore semiconducting TMIs glasses are of high interest for their important applications as switching and memory storage devices [3]. Therefore, many reports have been published concerning with glasses containing TMIs, where the authors have considered that these ions may act as glass network formers [4-6].

In case of pure V_2O_5 glass, V^{5+} ions exhibit both 4- and 5-fold coordination states depending on the sample preparation conditions [7]. A detailed analysis of this problem has been found in ref. [8]. It was observed also that the structure of vanadate glasses depends mainly on the nature of both the network former and modifier cations [4-6].

The structural changes of a glass network as a function of composition are directly acted to vary most of its physical properties [9, 10]. The electrical conduction in vanadate glasses (containing V^{3+} , V^{4+} and V^{5+} ions) proceeds through the hopping of electrons from one site to another [11]. But more detailed information about the conduction process is limited, since many factors affect such process, such as the type and concentration of TMI, the number of valence states associated with such TMIs, the glass preparation conditions and the microstructures of the prepared glass matrix. Thus, information about the structure of a glass is imperative for further understanding of the glass properties.

From another point of view, glasses containing heavy metal ions (HMIs) are also of special interest due to their important applications, especially in the field of ionizing radiation protection [10 - 15].

However, this article aims to study the structure and physical properties of some sodium-strontium-vanadium unconventional oxide glasses. IR spectroscopic analysis will be applied for structural investigation. Density and molar volume, dc electrical conductivity and γ -ray attenuation parameters will be also investigated as physical properties.

2. Experimental Work and Calculations

Glass samples obeying the following molecular formula, $\{(100-x) (0.75 V_2O_5+0.25 SrO) + x Na_2O\}$, ($x = 0, 4, 8, 12$ and 16), have been prepared by the melt quenching technique. Vanadium pent-oxide and strontium oxide were introduced as such, while Na_2O was interested as sodium carbonate. Appropriate amounts of the starting materials were weighted such that when melted give the required composition of samples. The batches were mixed well in an agate mortar to obtain batches of fine powders. The mixtures were then melted in porcelain crucibles in an electrically heated furnace at 1200 K for 2hs and melts were stirred several times

during melting in order to homogenize the prepared samples. Melts were then poured on a pre-heated stainless steel plate at room temperature to obtain solid glass samples. The visual examination showed that the obtained glasses are free of air bubbles, cracks and inhomogeneity.

Infrared (IR) transmission spectra of the studied glasses are obtained in the wave number range of 400–4000 cm^{-1} , by using KBr disk method and a Fourier transform infrared (FTIR) Berken-Elmer spectrometer model (RTX) at room temperature. The values of the vibrational energies of the structural groups in the glass networks were supplied directly by the apparatus software.

The experimental density (ρ_{exp}) of all samples was measured at RT using the suspension weight method based on Archimedes principle with toluene as the buoyant liquid, applying equation (1),

$$\rho_{\text{exp}} = \left(\frac{w_a}{w_a - w_t} \right) \times \rho_t \quad (1)$$

The empirical density (ρ_{emp}) values of the corresponding crystalline component were also calculated by using equation (2),

$$\rho_{\text{emp}} = \sum X_i \rho_i \quad (2)$$

where ρ_i are the densities of the oxides forming a glass sample, X_i is the mole fraction of the corresponding oxides.

Both the experimental and the empirical molar volume values were calculated using equation (3 and 4) respectively,

$$(V_m)_{\text{exp}} = \frac{M_M}{\rho_{\text{exp}}} = \frac{\sum n_i M_w}{\rho_{\text{exp}}} \quad (3)$$

$$(V_m)_{\text{emp}} = \frac{M_M}{\rho_{\text{emp}}} = \frac{\sum n_i M_w}{\rho_{\text{emp}}} \quad (4)$$

where M_M is the mean molecular weight of a sample, M_w is the molecular weight of the used oxides, n_i is the mole fraction of such oxides, ρ_{exp} is the experimental density and ρ_{emp} is the empirical density values.

The average inter-site separation between any adjacent vanadium ions was obtained from the glass composition and the measured experimental density.

For measuring the dc conductivity of the studied glasses, the as-quenched glass samples were polished to obtained samples of about 1mm thickness and 8 mm diameter. The obtained disks were then coated with an air drying silver paste to achieve good electrical contact. The measurements were done by applying the two probe method in the temperature range from 300 to 500 K. The samples were inserted inside a holder in such a way that, they kept in contact with two polished, cleaned and spring-loaded copper electrodes. A Keithley 6485 Picoammeter were used to measure the dc conductivity.

The total mass attenuation coefficients $(\mu_l/\rho)_{m(\text{total})}$ of a glass sample was calculated by applying WIN-XCOM program based on the mixture rule (equation 5) [10, 12],

$$\left(\frac{\mu_l}{\rho} \right)_{m(\text{total})} = \sum_{i=0}^n w_i \left(\frac{\mu_l}{\rho} \right)_{m(i)} \quad (5)$$

where (μ_l) is the linear attenuation coefficient (that can be calculated by multiplying the total mass attenuation coefficient of a sample by its experimental density value), w_i is the fractional weight of the oxides in each sample and $(\mu_l/\rho)_{m(i)}$ is the mass attenuation coefficients of a component constituting a glass sample.

The half value layers (HVL) of the studied glass were also calculated by applying equation (6), [10, 12]

$$\text{HVL} = 0.693 / \mu_l \quad (6)$$

Also, the Mean free path (MFP) (λ) of γ -ray photon was then calculated according to equation (7), [10, 12],

$$\lambda (\text{MFP}) = 1/\mu_l \quad (7)$$

3. Results and Discussion

3.1. Structure- Infrared Spectroscopy:

The recorded IR spectra for the studied glasses in the range from 400 to 4000 cm^{-1} are exhibited in Fig. (1). It is easy to observe the broadening of the obtained vibrational bands that usually observed for amorphous solids and glasses [16]. Inspecting these spectra, it is hard to extract really the bands corresponding to various structural groups. Therefore, the obtained spectra were then subjected to the de-convolution program in between about 400 and 1100 cm^{-1} , to obtain the vibrational bands as really as possible [17]. The selected de-convoluted range of the IR spectra is due to the fact that, there are no vibrational bonds due to vanadium structural groups after about 1100 cm^{-1} , but all the observed bands after that is due to water content, OH- groups and/or hydrogen bonds [16].

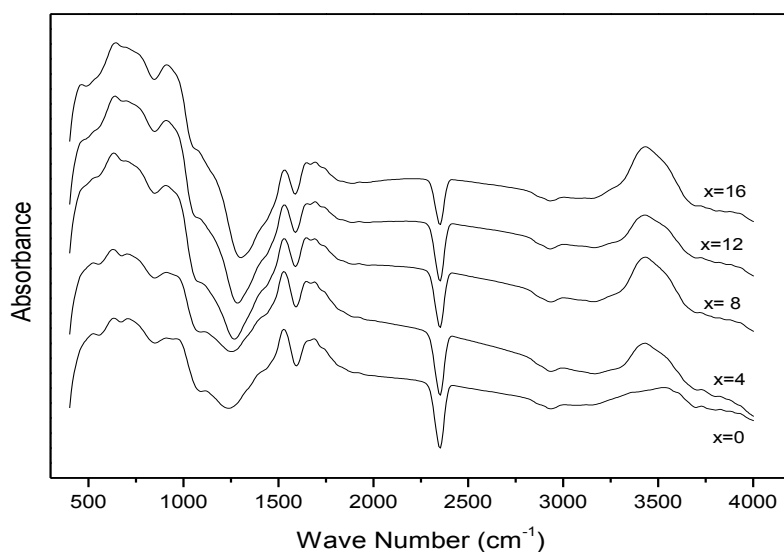


Fig. (1) The IR spectra of the studied glasses.

However, all the resulting spectra were de-convoluted to enable us to shed further light on the structural changes of VO_3 , VO_4 and VO_5 , since these groups represent the most abundant units in these glasses. Fig. (2) shows the de-convoluted spectrum of the sample containing 12 mol% Na_2O , as a representative figure. All the obtained major absorption bands in the spectra of all the studied glasses are summarized in Table (1).

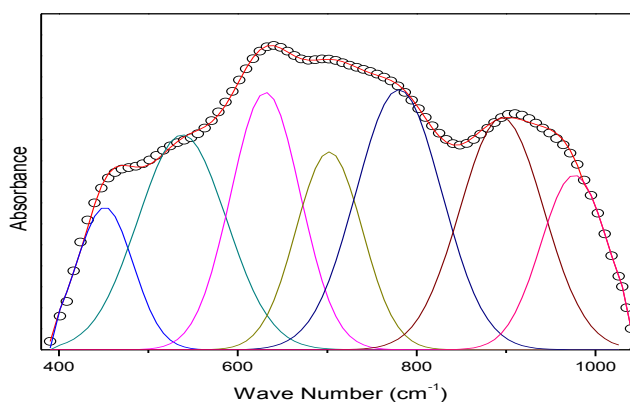


Fig. (2). The de-convoluted spectrum of the glass sample containing 12 mol% Na_2O , as representative figure.

Table (1). The IR vibrational bands of all the studied glasses.

Na_2O mol %	IR vibration bands (cm^{-1})						
0	467	520	625	710	764	899	971
4	465	521	628	710	765	900	975
8	452	523	632	707	766	905	976
12	463	530	637	706	768	910	978
16	458	535	639	705	770	912	981

These bands can be attributed as follows:

- * The band appeared around 460 cm^{-1} can be attributed to the bending vibrations of V–O–V bridging oxygen bonds [18].
- * The band appeared in all samples between 520 and 535 cm^{-1} can be attributed to the presence of V-O-V stretching vibration [19].
- * The strong bands appeared around 630 cm^{-1} may be due to the vibration of VO_3 groups [3].

- * The band appeared around 705 cm⁻¹ can be assigned to the stretching vibration of the doubly bonded oxygen atom (V=O) [3].
- * The bands that appeared between 764 and 770 cm⁻¹ can be attributed to V-O-V bending vibration in VO₄ polyhedra [18].
- * The band appeared at 900 cm⁻¹ may be due to the symmetric stretching vibration of [VO₄] and the high-frequency bands at 971–981cm⁻¹ can be attributed not only to the VO₅ groups but also to the branched VO₄ polyhedra groups having one V=O double bond [20].

Inspecting these bands, it is seen that the bands appeared around 530, 630, 765, 900 and 975 shifts gradually to the higher wave number with the gradual increase of Na₂O. Therefore the force constant of V–O bonds in these glasses are then calculated by applying equation (8) [21],

$$F_{V-O} = 4\pi^2 c^2 \mu v_{eff}^2 \quad (8)$$

where *c* is the speed of light, *v_{eff}* is the effective cation site vibrational frequency and *μ* is the reduced mass of both vanadium and oxygen cations. The reduce mass *μ* of the V–O bond can be obtained by applying equation (9) [21],

$$\mu = \frac{m_v m_o}{m_v + m_o} \quad (9)$$

where *m_v* and *m_o* are the masses of vanadium and oxygen respectively.

The obtained values of the force constant are presented in Table (2), and exhibited graphically in Fig. (3) for the band appeared at 630 cm⁻¹ as representative figure. Inspecting the obtained force constant values, it can be seen that, it increased with increasing Na₂O content for most energy bands of the studied glass samples. This may be the reason of the observed shift for most IR absorption bands. However, it can be supposed that the introduced Na⁺ cation act to increase the force constant of V-O band in the glass network. That is sodium cation act to increase the connectivity of the glass matrix.

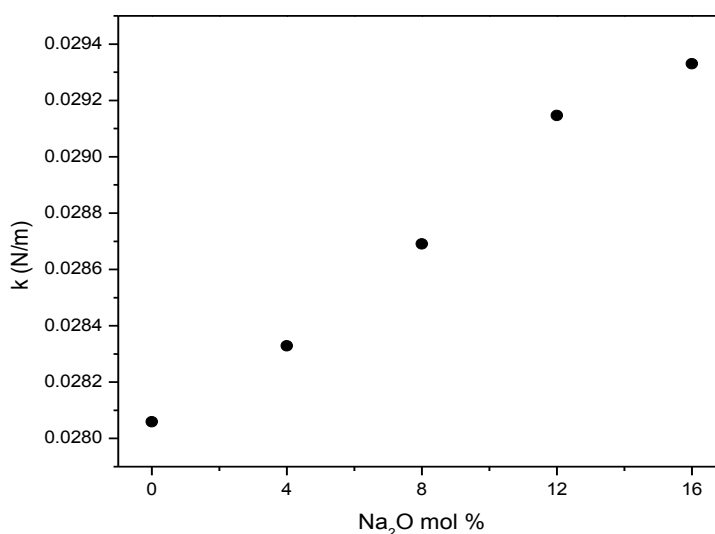


Fig. (3). The variation of the force constant of V-O bond versus Na₂O content. for the band appeared at 630 cm⁻¹, as a representative figure.

Table (2). The force constant of all the studied glasses.

Na ₂ O mol %	F _{v,o} m(N/m)						
	0	1.57	1.94	2.81	3.62	4.19	5.81
4	1.55	1.95	2.83	3.62	4.20	5.82	6.83
8	1.47	1.96	2.87	3.59	4.21	5.88	6.84
12	1.54	2.02	2.91	3.58	4.24	5.95	6.87
16	1.51	2.06	2.93	3.57	4.26	5.97	6.91

However, it can be supposed that, the introduced Na⁺ ions act to increase the force constant of V-O bonds in the glass network. That is sodium ions act to increase the connectivity of the glass network.

3.2. Physical Properties:

3.2.1. Density & Molar Volume:

Density of solids is an essential property and the comparison between the experimental and empirical densities is important for confirming the amorphous nature of the studied glass samples. It is also of interest since the measured experimental value is usually used for calculating many physical constants of a solid.

The obtained ρ_{exp} & ρ_{emp} values of all the studied glasses as well as $V_{m,exp}$ & $V_{m,emp}$ values are presented in Table (3),

Table (3). Density (exp. & emp.) and molar volume (exp. & emp.) values as well as the oxygen ion density of all glasses versus Na₂O content.

Na ₂ O mol %	ρ_{exp} (g/cm ³)	ρ_{emp} (g/cm ³)	$V_{m,exp}$ (cm ³ /mole)	$V_{m,emp}$ (cm ³ /mole)	N(O) 10 ⁺²² cm ⁻³
0	3.14	4.8	51.6	33.6	4.67
4	3.11	4.7	50.8	33.5	4.59
8	3.05	4.6	50.5	33.4	4.48
12	3.01	4.5	49.8	33.2	4.40
16	2.98	4.4	48.9	33.1	4.33

The variation of both density values (ρ_{exp} & ρ_{emp}) can be shown in Fig.(4) as a function of Na₂O.

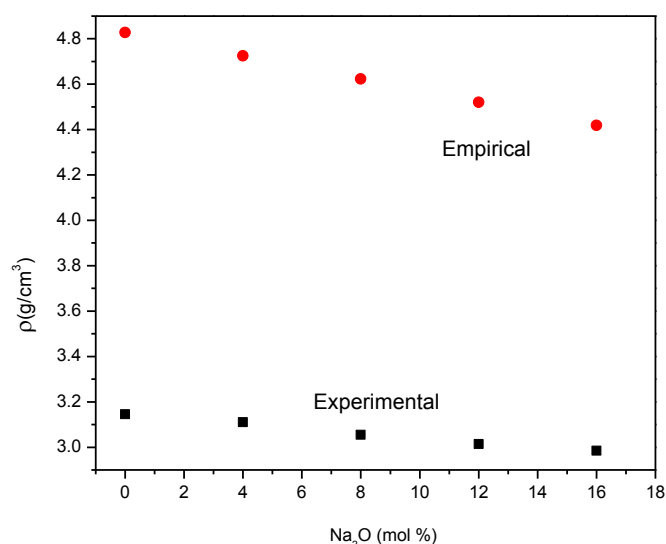


Fig. (4). The variation of both ρ_{exp} & ρ_{emp} versus Na₂O content.

It is observed that, ρ_{emp} is usually higher than the corresponding ρ_{exp} , while $V_{m,emp}$ is lower than $V_{m,exp}$; which refers to the amorphous nature and the short range order character of the studied glass samples. It is observed also that the density decreased gradually with increasing Na₂O content. Which may be due to the difference between the molecular weights of Na₂O, SrO and V₂O₅ moles, where their molecular wights are 62.0, 104.0 and 182.0 g/mol respectively. However, the change in density can be explained rather simply as due to the replacement of molecules (V₂O₅ and SrO) by lighter (Na₂O) molecules. On the other hand, the variation of both V_m values (exp. & emp.) can be shown in Fig. (5), with the gradual increase of Na₂O content.

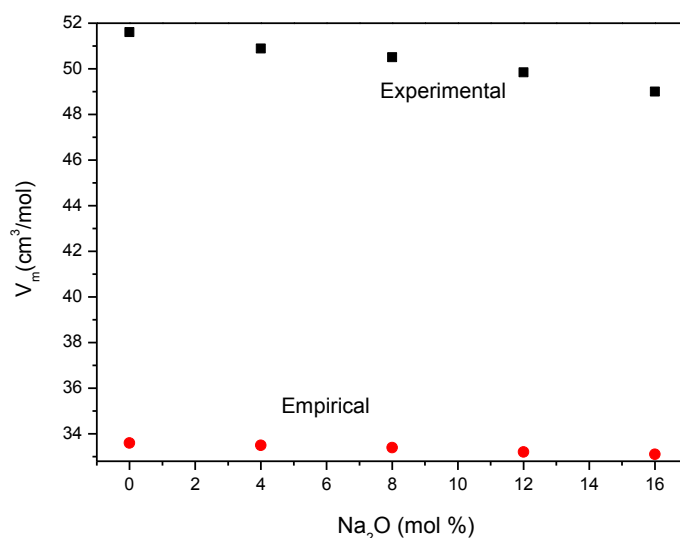


Fig. (5). The variation of the molar volume versus Na₂O content.

The decrease in V_M with Na₂O can be understood by regarding the covalent radii of Na, V & Sr (154, 125 & 192 pm respectively), where, it is seen that, the covalent radius of Na is higher than that of V, and lower than that of Sr. But remembering that five oxygen anions (from V₂O₅) and one oxygen (from SrO) are removed versus only single oxygen anion is introduced, which may shrink, largely the oxygen network. Therefore, the number of oxygen ion density are then calculated and the obtained values are represented also in Table (3). It is appeared that, the calculated number of oxygen density decreased gradually as Na₂O was gradually increased. However, the decrease of V_m can be understood to be due to the following two factors:

- The force constant of vanadium oxygen bands increases by increasing Na₂O content and hence the interatomic spacing distance decreased.
- The decrease of the oxygen ion density of the glass networks.

3.2.2. DC Conductivity:

The dc conductivity of the studied glasses were measured in the temperature range from 300 to 500 K, and Fig. (6), exhibits the variation of $\ln(\sigma T)$ versus $(1000/T)$ for all the studied glasses.

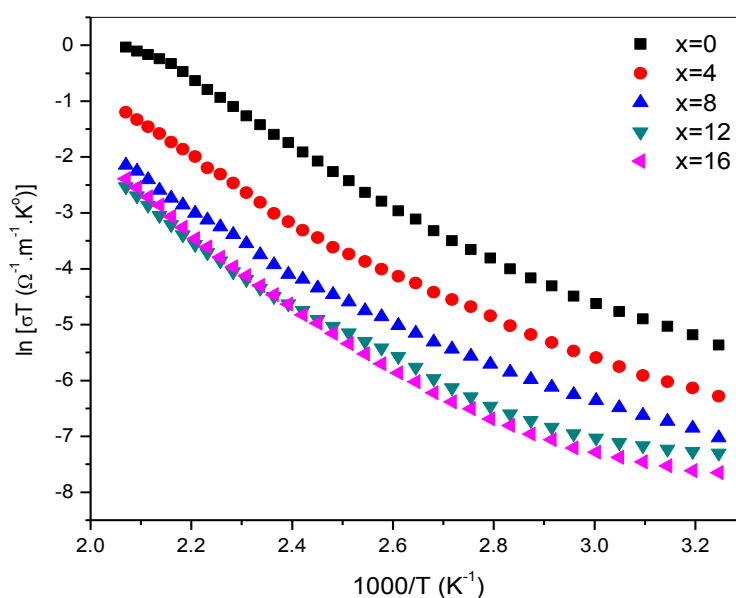


Fig. (6). The variation of $\ln(\sigma T)$ versus $1000/T$.

It is observed that, this figure shows an Arrhenius behavior type of equation (10) [1, 22],

$$\sigma = \sigma_0 \frac{\exp(-W/kT)}{T} \quad (10)$$

where σ_0 is pre-exponential factor, k is Boltzmann constant, T is the absolute temperature and W is the activation energy. The high temperature activation energy of all glasses, were computed from the slopes of the obtained $\ln(\sigma T)$ versus $(1000/T)$ curves in the high temperature range. Fig. (7) shows the variation of the dc conductivity with the gradual increase of Na_2O content at a various fixed temperatures, while Fig. (8) shows the variation of electrical activation energy with the gradual increase of Na_2O content also.

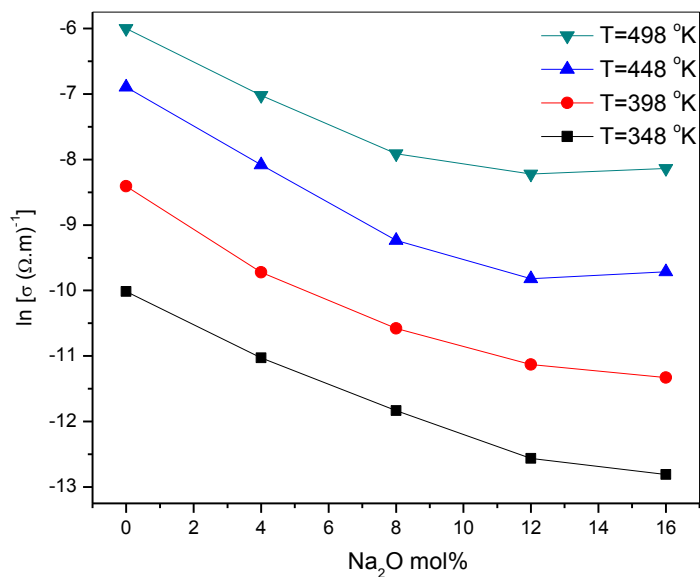


Fig. (7). The variation of the dc conductivity as a function of Na_2O content at different fixed temperatures.

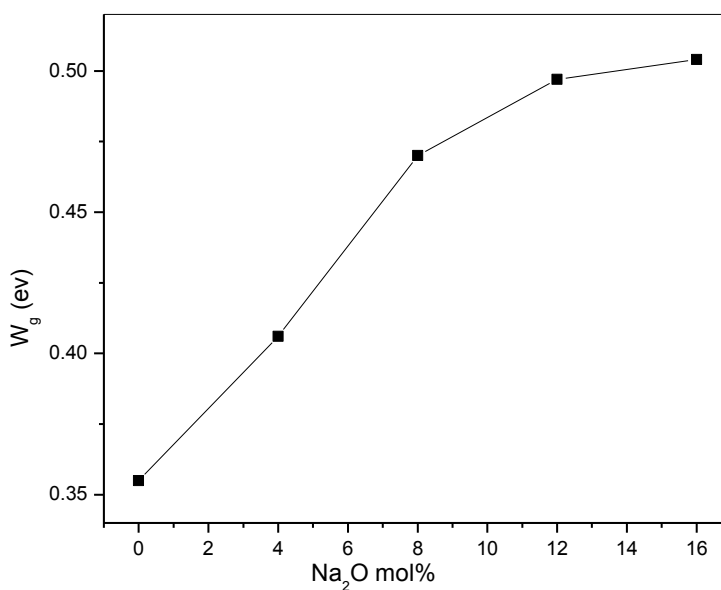


Fig. (8). High temperature activation energy as a function of Na_2O content.

The observed slight deviation from linearity in $\ln(\sigma T)$ versus $(1000/T)$ curves indicated that, the activation energy is temperature dependent and it is a typical characteristic of semi-conducting oxide glasses. This means that the thermally activated hopping conductivity is a characteristic feature for the electrical conduction in these glasses and the small polaron hopping models is the predominant conduction mechanism. Also, the conductivity of these glasses consists of both ionic and electronic conduction. Generally ionic conduction depends mainly on the concentration and mobility of the present alkali sodium ions.

In vanadate glasses the dc conductivity is mainly electronic and depends strongly upon the average distance (R) between any two adjacent vanadium ions. The average distance was calculated by using the following relation

$$R = (1/N)^{1/3} \quad (11)$$

where N is the concentration of vanadium ions per unit volume, and it can be calculated by using equation (12),

$$N = 2 \left[\left(\frac{\rho_{\text{exp}} F_w}{M_w} \right) N_A \right] \quad (12)$$

where ρ_{exp} is the experimental density of a glass sample, F_w is the weight fraction of V_2O_5 in such sample, M_w is the molecular weight of V_2O_5 and N_A is Avogadro number. The calculated values of R and N are summarized in Table (4). Sodium ion concentration $N(\text{Na})$ can be calculated by using equation (12) also.

It is shown theoretically that, the polaron radius can be calculated by using equation (13) [23]

$$r_p = (\pi/6)^{1/3} R/2 \quad (13)$$

and the obtained values are given in Table (4), also.

Table (4): High temperature activation energy, sodium ion density, vanadium ion density, vanadium ion spacing distance, small polaron radii and density of states at Fermi level for the studied glasses.

Na ₂ O mole %	W (ev)	N(Na) 10 ⁺²² (cm ⁻³)	N(V) 10 ⁺²² (cm ⁻³)	R(V) nm	r _p (nm)	N(E _F) 10 ⁺²²
0	0.355	0	1.75	0.385	0.137	2.09022
4	0.406	0.947	1.70	0.389	0.138	1.7803
8	0.470	1.91	1.65	0.393	0.139	1.48486
12	0.497	2.90	1.59	0.397	0.141	1.36095
16	0.504	3.93	1.55	0.401	0.142	1.30481

It is clear from Figs (7 & 8) that the conductivity decreased while the activation energy increased with the increase of Na₂O content up to 12 mol%, while in between 12 and 16 mol% Na₂O they tend to be approximately stable. The change in conductivity and activation energy may be due to the fact that, decreasing V₂O₅ content increases the distance between any two adjacent vanadium ions and this is shown in Fig.(9).

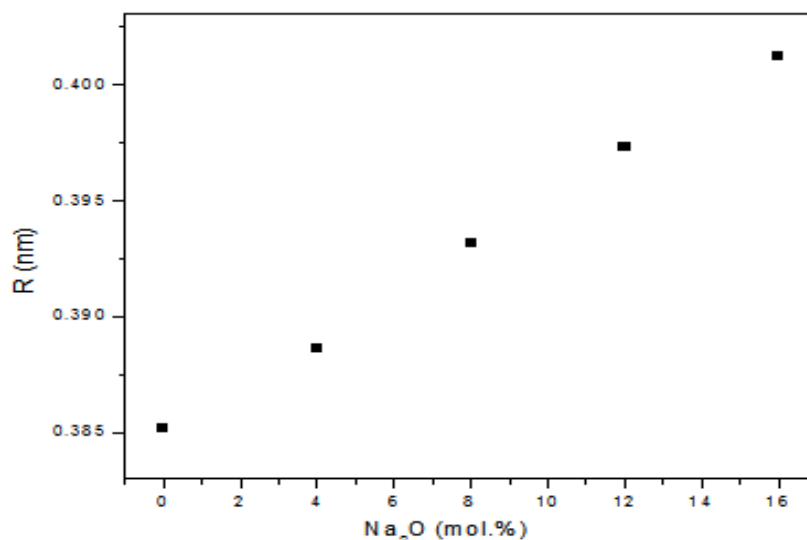


Fig. (9). The average distance (R (nm)), between any two adjacent vanadium ions versus Na₂O content.

The activation energy appears to increase with increasing the distance between vanadium ions. Clearly, the smaller value of activation energy (0.311 eV at Na₂O=0%) suggests that the dc conductivity is controlled by

electron hopping and depends upon the distance between vanadium ions (R). The obtained results are found to be in agreement with to the results obtained in other studies [3, 23].

At high Na_2O concentrations (12 and 16 mole %) and specially at high temperatures, the conductivity is almost constant which may be due to the following factors,

- The decrease of the electronic conductivity by decreasing vanadium ions content.
- The increase of the ionic conductivity which may be due to the increase of sodium ion density from (0 to $3.9 \times 10^{22} \text{ cm}^{-3}$) and the increase of the mobility of Na ions due to the increase of the vanadium ion spacing.

The density of states at Fermi level $N(E_F)$ is also estimated by using the following relation [23]

$$N(E_F) = \frac{4}{3\pi R^3 W} \quad (14)$$

The values of the density of states at Fermi level $N(E_F)$ decreased and hence the conductivity may be decrease.

3.2.3. Gamma-Ray Attenuation Parameters:

It was established that glasses containing HMIs can be used now for ionizing radiation protection, since glasses are transparent to visible light as well as they are easy manufactured [12]. Since the studied glasses here contain various amounts of Sr^{2+} ions, therefore, the attenuation parameters will be checked.

However, the total mass attenuation coefficients $(\mu/\rho)_{m(\text{total})}$ were calculated by applying Win X-COM program, based on the mixture rule (equation (5)) [10, 12]. The selected γ -ray energies are 356, 662, 1173 and 1332 keV, emitted from ^{133}Ba (for 356 keV), ^{137}Cs (for 662 keV) and ^{60}Co (for both 1173 & 1332 keV) as low γ -ray energies. The obtained values of $(\mu/\rho)_{m(\text{total})}$ are then exhibited in Table (5).

The linear attenuation coefficients (μ_l), the half value layer (HVL) and mean free path (λ) for the studied glasses were then calculated by applying equations (5, 6 and 7) respectively, for the same γ -ray energies and they are also listed in Table (5).

Table (5): The total mass attenuation coefficient $(\mu/\rho)_{m(\text{total})}$, linear attenuation coefficient (μ_l), half value layer (HVL) and mean free path (λ) values at different low γ -ray energies for the studied glasses.

SrO mole %	25	24	23	22	21	E
Na_2O mole %	0	4	8	12	16	(keV)
$(\mu/\rho)_{m(\text{total})}$ (g.cm^{-2})	0.097	0.097	0.097	0.097	0.097	356
	0.073	0.073	0.073	0.073	0.073	662
	0.055	0.055	0.055	0.055	0.055	1173
	0.052	0.052	0.052	0.052	0.052	1332
μ_l (cm^{-1})	0.305	0.301	0.296	0.292	0.289	356
	0.228	0.226	0.222	0.220	0.218	662
	0.173	0.171	0.168	0.166	0.165	1173
	0.162	0.161	0.158	0.156	0.155	1332
HVL (cm)	2.276	2.301	2.343	2.375	2.399	356
	3.036	3.066	3.119	3.157	3.185	662
	4.008	4.047	4.115	4.164	4.200	1173
	4.274	4.315	4.388	4.440	4.478	1332
λ (cm)	3.284	3.284	3.284	3.284	3.284	356
	4.382	4.382	4.382	4.382	4.382	662
	5.783	5.839	5.938	6.008	6.060	1173
	6.167	6.226	6.332	6.407	6.461	1332

In order to clarify the suitability of the studied glasses to act as shield for γ -ray energy, the obtained values of the $(\mu/\rho)_{m(\text{total})}$ are plotted in Fig. (10), as a function of the increase of Na_2O , for all the applied γ -ray energies. It is observed that the values of the obtained $(\mu/\rho)_{m(\text{total})}$ appeared to be stable with the increase of Na_2O but they varies considerably with the variation of the γ -ray photon energy. According to these result it can be approximately stated that, all the studied glasses can be used as shielding material for the 356 keV γ -ray energy.

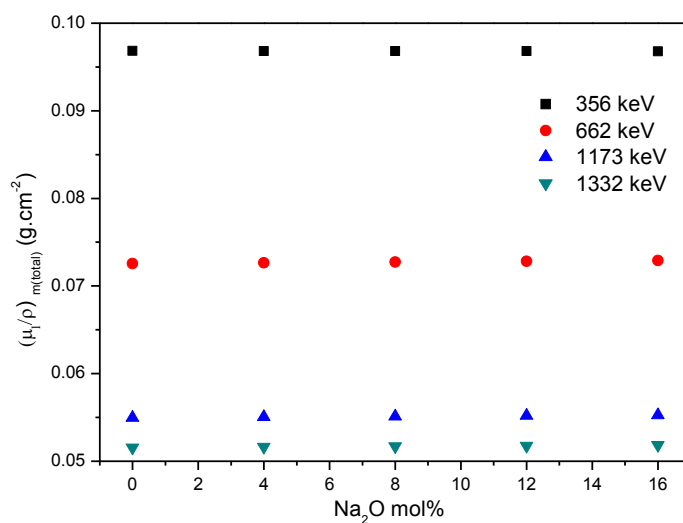


Fig. (10). The variation of the total mass attenuation coefficients $(\mu_t/\rho)_{m(\text{total})}$ as a function of Na_2O content at different γ -ray energies for the studied glasses.

From Table (5), the observed increase of the linear attenuation coefficient as well as the corresponding decrease in the *HVL* of the studied glasses are very limited and it may be due to the variation of the density of the glass samples, as well as the slight differences between the γ -ray mass attenuation coefficient of SrO , V_2O_5 and Na_2O molecules where they are 0.1060, 0.0983 and 0.0991 cm^2/g respectively at 356 keV γ -ray energy.

4. Conclusion

According to the obtained results and the supposed discussion, it can be concluded from the IR analysis that, the glass network is built up mainly of VO_3 , VO_4 and VO_5 . The force constant for most V-O bonds are increased by increasing Na_2O content. The density and molar volume are both decreased and the difference between both experimental and empirical values confirm the amorphous nature and the randomness character of the studied glasses. The electrical conductivity appeared to be mixed electronic and ionic, and it decreased with the increase of Na_2O content, but at high temperature and high Na_2O concentration ($\text{Na}_2\text{O} > 8\%$ mole) it tends to be stable. The conduction mechanism was concluded to be the small polaron hopping model. The calculated total mass attenuation coefficient of all glass showed approximate stability for the same γ -ray energy. Also it can be concluded that all the studied glasses act as good shield at 356 keV γ -ray energy only.

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