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## Vanadium Doped Sodium Disilicate Glass and Glass Ceramics: Structural Characterization

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**Abstract** Glass samples of compositions  $x\text{Na}_2\text{O}-2x\text{SiO}_2-z\text{V}_2\text{O}_5$  with  $z$  varying from 0 to 15% mole fraction are prepared by melt quench technique. The structural analysis of glasses is carried out by FTIR and XRD. The spectra of high  $\text{V}_2\text{O}_5$  (1-15 mol%) containing glasses show an increased number of distinct peaks in the low frequency region with a convolution of several broad Gaussians in the high frequency region. The distinct peaks and peak positions of the deconvoluted Gaussians are assigned to Si-O-Si, V-O-V, Si-O, V=O etc bonds. A variation in the band positions and the relative amounts of bonds with  $\text{V}_2\text{O}_5$  is also shown. The results showed that, with increasing vanadium oxide content, light transmission through sodium silicate glasses was reduced due to legand field and charge transfer mechanisms. The presence of vanadium oxide in sodium silicate glasses also leads to formation of crystalline phases. X-ray diffraction pattern as well as optical inspection of the base samples did not show any evidence of devitrification.

**Keywords** XRD, IR Spectroscopy, Devitrification, Deconvolution,  $\text{V}_2\text{O}_5$

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### 1. Introduction

Due to the rapid progress made in the glass industry in recent times, glass has come out as the most versatile engineering materials of the modern times. Chemically glass may be defined as a fused mixture of silicates alkali and alkaline earth compounds and other glass constituents such as calcium oxide Barium oxide [1]. In familiar usage, the term 'glass' refers to a class of materials to great practical usefulness, with a number of characteristics properties such as transparency, brittleness, high resistance to chemical attack, effectiveness as an electrical insulator; ability to contain vacuum and the property of softening progressively and continuously when heated [2]. In this work, alkali metal oxide (i.e.  $\text{Na}_2\text{O}$ ) silicate glasses with different amount of vanadium pentoxide have been chosen to interpret the framework structure mainly by means to infrared spectral analysis.

The term glass is derived from a latin term "Glaseum" which means lustrous and transparent materials. It may have appeared first in the Middle Eastern regions such as Egypt and Mesopotamia around 3000 to 2000 B.C. Egyptian craftsmen developed a method for producing glass vessels around 1500 B.C. Egyptian made first synthetic glass in the form of beads sand called it "faience". About 2000 years ago, Syrian craftsmen invented glassblowing, a skill adopted by the Romans, who carried it with them as they swept through Western Europe on their conquest. The rise of Venice to prominence in the 13<sup>th</sup> century; Venice becomes the centre of glassmaking in the western world. As the industrial revolution gathered momentum, new manufacturing



technologies enabled the production of scientific glass instruments, bottles, windowpanes, and many other items. In modern age, glass plays an important and essential role in social life, science and technology. The physical, optical and other properties of glass make it suitable for different applications such as utensils, optoelectronic materials, laboratory equipments, thermal insulator (glass wool), nuclear and solar energy technologies etc. In addition to other applications, it is also being used as a decorative material. Now a day, it features in almost every aspect of our lives. The aim of the present study was to observe the IR spectra of silicovanaded ( $\text{Na}_2\text{O-SiO}_2\text{-V}_2\text{O}_5$ ) glass with varying amount of  $\text{V}_2\text{O}_5$  with the aim of understanding bonding mechanism and to observe the effect of vanadium pentoxide ( $\text{V}_2\text{O}_5$ ) in sodium silicate ( $\text{Na}_2\text{O-SiO}_2$ ) glass and its devitrification form.

## 2. Experimental Details

In order to structural analysis of vanadium doped sodium silicate glasses and the effect of vanadium pentoxide in it, simple  $x\text{Na}_2\text{O-}2x\text{SiO}_2\text{-}2z\text{V}_2\text{O}_5$  base glass samples were prepared and characterized. In this work, mainly IR spectroscopy and XRD of the base and heat treated sample are used to characterize the materials. The infrared spectroscopy provides absorption spectra, which gives information about the strength and stiffness of chemical bonds; binding of atoms in compound and characterize the vibrational motion of atom-atom bonds. These absorption bands are much like fingerprints of particular structural arrangement of compound. The qualitative and quantitative analysis of the infrared absorption spectra is employed with the deconvolution to several Gaussians to characterize the materials. According to the National Research Council, "characterization means the description of those feature of the composition and structure of a material which are significant for a particular preparation, study of various properties and suffice for the reproduction of the materials" [3]. In X-ray diffraction techniques the measured pair distribution functions are compared to the distribution functions that are calculated from various models of glass structures and the best fit is taken. A brief description of the methods used for the characterization of materials can be obtained from Hench and Gould [4]. The glass compositions chosen for present study are listed in Table 1.

**Table 1:** Nominal composition, melting temperature and optical quality of glasses of various compositions

Title of the samples	Nominal composition in mol %			Melting temperature in °C	Optical quality	XRD
	$\text{Na}_2\text{CO}_3$	$\text{SiO}_2$	$\text{V}_2\text{O}_5$			
NaSV0	33.33	66.67	0	1370	Transparent, White	Amorphous
NaSV1	33	66	1	1300	Transparent, White	Amorphous
NaSV2	33.67	65.33	2	1350	Transparent, Milky	Amorphous
NaSV5	31.67	63.33	5	1350	Radish (light)	Amorphous
NaSV10	30	60	10	1350	Radish (dark)	Amorphous
NaSV15	28.33	56.67	15	1250	opaque	NI**

Na for  $\text{Na}_2\text{CO}_3$ , S for  $\text{SiO}_2$ , V for  $\text{V}_2\text{O}_5$ ;

## 3. Results

### 3.1. The results of IR spectra

The infrared spectra recorded for binary  $x\text{Na}_2\text{O-}2x\text{SiO}_2$  and ternary  $x\text{Na}_2\text{O-}2x\text{SiO}_2\text{-}z\text{V}_2\text{O}_5$  ( $z=1, 2, 5, 10, 15$  mol %) of base and heat-treated glass system are illustrated in Fig. 1 and Fig. 2 respectively. The spectrum of the binary  $x\text{Na}_2\text{O-}2x\text{SiO}_2$  glass system shows two very distinct peaks at  $465$  and  $1065\text{ cm}^{-1}$  in the lower wavenumber region. The absorption intensity at  $465\text{ cm}^{-1}$  is higher than as it is at  $1065\text{ cm}^{-1}$ . Also, the band at the wavenumber  $465\text{ cm}^{-1}$  is broader the band at the wavenumber at  $1065\text{ cm}^{-1}$ . With the addition of small amounts of  $\text{V}_2\text{O}_5$  changes the shape of this spectrum and the peaks become sharper. Glass samples NaSV1-NaSV15 gave strong absorption bands within a broad Gaussian in the lower frequency region and the sharpness of the peaks becomes more distinct with the increase of  $\text{V}_2\text{O}_5$  concentration (up to 5 mol %) and then disappears slowly with the increase of  $\text{V}_2\text{O}_5$  (up to 15 mol %). The weak band appears at  $788\text{ cm}^{-1}$  in NaSV0 glass is disappeared in NaSV1-NaSV15 samples. Apparently all the spectra in the low  $\text{V}_2\text{O}_5$  containing glass, NaSV1-NaSV2 are similar to look at but the close inspection shows that



their relative intensities are different. In the high V<sub>2</sub>O<sub>5</sub> containing glasses (NaSV5-NaSV15) the band appears at 1062 cm<sup>-1</sup> becomes broader and the sharpness is increased in NaSV5 but after that the sharpness decreases in NaSV10-NaSV15. A very weak band appears at 1400 cm<sup>-1</sup> in NaSV0 glass. This band becomes sharper with the increase of V<sub>2</sub>O<sub>5</sub> concentration. In the region 1500-4000 cm<sup>-1</sup> there are only two peaks at 1650 cm<sup>-1</sup> and 3467 cm<sup>-1</sup>. The very strong band appears at 3467 cm<sup>-1</sup> becomes sharper and broader with the increase of V<sub>2</sub>O<sub>5</sub> concentration. The absorption intensity of the band at 3467 cm<sup>-1</sup> is stronger than the band at 1650 cm<sup>-1</sup>.

All the bands appeared at the IR spectra of base and heat-treated samples are defined as very weak, weak, medium, strong, very strong, broad and shoulder according to their intensities. A comparison of the IR spectra of base and heat-treated glasses shows that the band positions of the base glasses are shifted towards high frequency due to heat treatment. The color of all the samples is also changed due to heat-treatment.

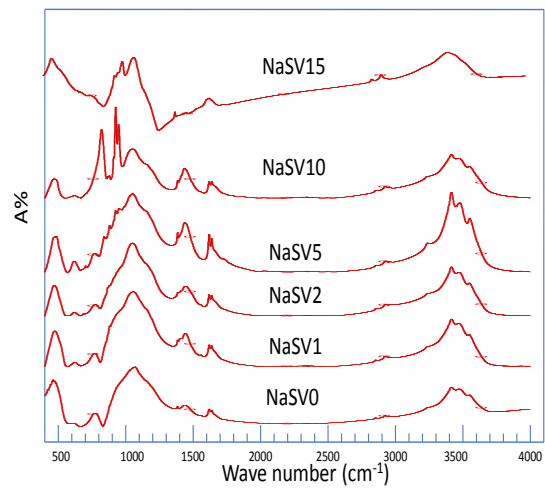
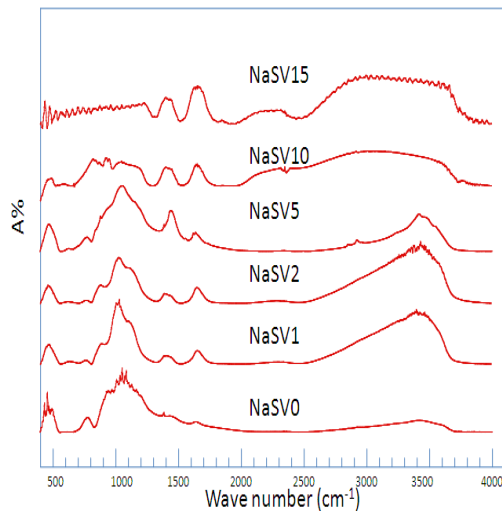


Figure 1: Infrared Spectra of  $x\text{Na}_2\text{O}2x\text{SiO}_2z\text{V}_2\text{O}_5$  Base Glasses ( $z=0-15$  mol %)

Figure 2: Infrared Spectra of  $x\text{Na}_2\text{O}2x\text{SiO}_2z\text{V}_2\text{O}_5$  Heat-treated Glasses ( $z=0-15$  mol %)

**Table 2:** The position and nature of characteristics IR bands of the  $x\text{Na}_2\text{O}-2x\text{SiO}_2-z\text{V}_2\text{O}_5$  base glasses

Specimens	Position of prominent absorption maxima (cm <sup>-1</sup> )									
NaSV0	460m	-	792w	996sh	1052s	1100sh	1463sh	1654sh	-	3467b
NaSV1	472m	633vw	761vw	892sh	1012vs	1109sh	1408w	1654m	2342vw	3400s&b
NaSV2	472m	623vw	765vw	879sh	1023vs	1140sh	1400w	1648m	2295vw	3452s&b
NaSV5	470m	629vw	776vw	881sh	1053vs	-	1440m	1638w	-	3470s
NaSV10	485m	582vw	667sh	821sh	1045s	-	1470s	1652s	2351sh	3450vs&b
NaSV15	434m	-	-	-	-	-	1403m	1651s	2352sh	3487vs&b

Where s=strong, m=medium, b=broad, sh=shoulder, w=weak, vw=very weak, vs=very strong

**Table 3:** The position and nature of characteristics IR bands of the  $x\text{Na}_2\text{O}-2x\text{SiO}_2-z\text{V}_2\text{O}_5$  heat-treated glasses

Specimens	Position of prominent absorption maxima (cm-1)									
NaSV0	478m	637vw	793w	-	1084vs &b	1150sh	1465vw	1643vw	-	3485s
NaSV1	487m	651vw	789vw	-	1073vs &b	1167sh	1461w	1650vw	2362vw	3487s
NaSV2	486m	648vw	792vw	980sh	1067vs &b	1150sh	1465w	1665vw	2321vw	3482s
NaSV5	495m	635vw	787vw	930m	1075vs &b	1185sh	1459m	1645vw	-	3492s
NaSV10	493m	-	-	963w	1075s	1170sh	1457m	1644m	2367vw	3487vs&b
NaSV15	481m	674sh	781w	-	1095s	1172w	1472w	1644w	2359vw	3492vs&b

Where s=strong, m=medium, b=broad, sh=shoulder, w=weak, vw=very weak, vs=very strong

The spectra are deconvoluted to several Gaussians depending on their shape using a computer program to estimate the band positions and relative amounts of various bonding mechanism. The deconvolution of NaSV0-NaSV15 glasses are shown in Fig. 3. This process makes it easy to calculate the relative area of each component bands and band positions. Each component band is related to some types of vibration in a specific structural group. The concentration of the structural group is proportional to the relative area of its component bands.

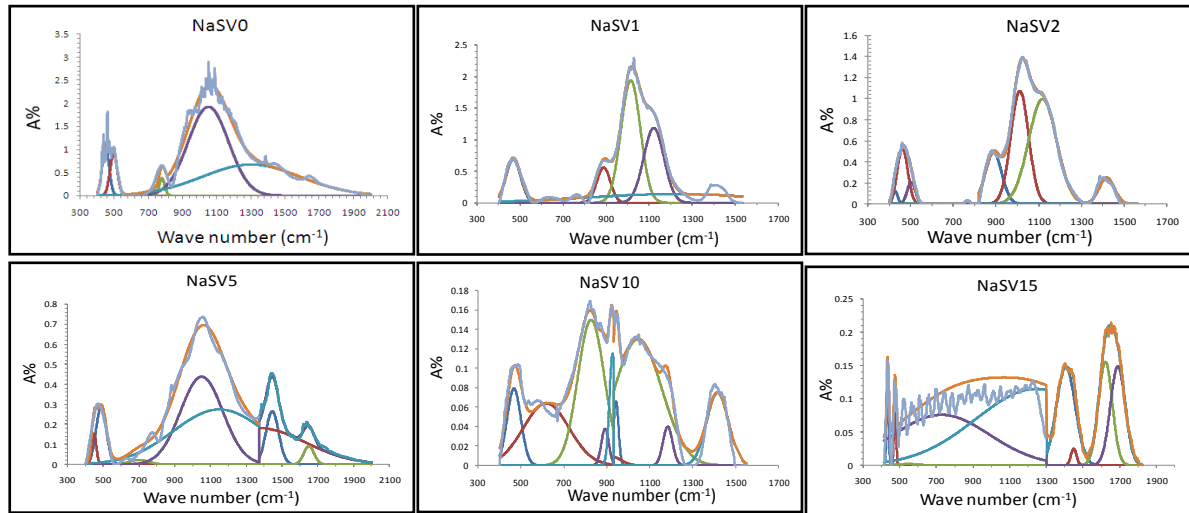


Figure 3: Deconvoluted spectra of  $x\text{Na}_2\text{O}-2x\text{SiO}_2-z\text{V}_2\text{O}_5$  ( $z = 0, 1, 2, 5, 10, 15$  mol %)

### 3.2. X-ray Diffraction of Base and Heat-treated Samples

The X-ray diffraction for NaSV0-NaSV10 base and heat-treated samples are shown in Fig.4. The X-ray diffraction patterns did not show any peaks and there was a broad haloes between  $2\theta=12$  to  $33$  (deg.). These patterns indicate the amorphous nature of the studied samples.

The samples were heated at  $475^\circ\text{C}$  for 6 hours. The diffraction patterns were showed that the samples NaSV2 and NaSV5 were vitrified at this temperature and time. The other samples NaSV0, NaSV1 and NaSV10 were not formed crystallization at this temperature and time.

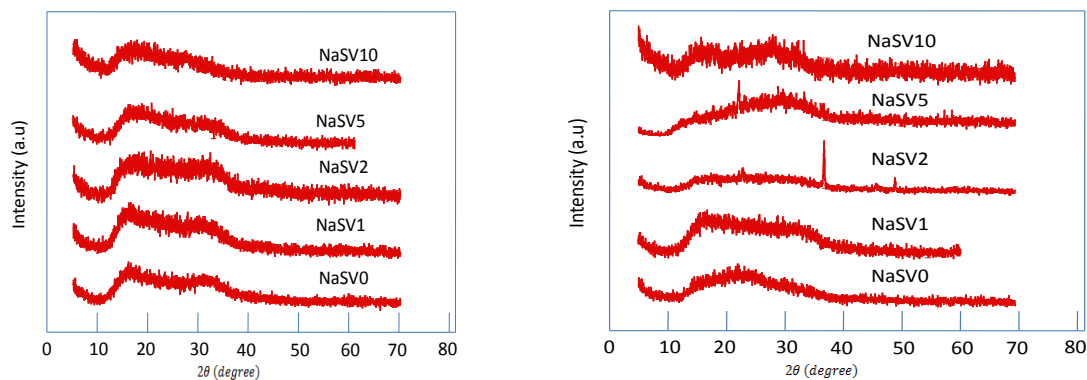


Figure 4: XRD Pattern of  $x\text{Na}_2\text{O}-2\text{SiO}_2-z\text{V}_2\text{O}_5$  bases and heat-treated glasses

### 4. Discussion

Careful inspection of the IR spectra shows that  $400-1500\text{ cm}^{-1}$  consists of distinct peaks and shoulders, while in the region  $1600-4000\text{ cm}^{-1}$  shows only two sharp bands with maxima at  $1650\text{ cm}^{-1}$  and  $3467\text{ cm}^{-1}$ . Due to the differences in the pattern only the region  $400-2000\text{ cm}^{-1}$  is deconvoluted to several Gaussians. The infrared spectroscopic analysis reveals that the bonds obtained at  $465, 477, 480$  and  $610\text{ cm}^{-1}$  may be due to Si-O-Si and V-O-V bending vibration and the bands in the region  $400-500\text{ cm}^{-1}$  may be attributed to the bending vibrations of oxygen such as Si-O-Si and V-O-V. The weak band observed at  $477\text{ cm}^{-1}$  is attributed to bending vibration of Si-O-Si [5-10]. The very weak band at  $748$  and  $788\text{ cm}^{-1}$  in the spectrum can be resulted from the Si-O-Si symmetric bending mode. In vitreous silica and titanium alkali silicate glasses, Bell et al. and Kyoshi Kusabiraki



[6,11], obtained the frequency of Si-O-Si bending motion at  $730\text{ cm}^{-1}$  and  $800\text{-}750\text{ cm}^{-1}$  respectively, where oxygen atom moves approximately at right angles to the Si-O-Si planes. Again, the oxygen rocking Si-O-Si bending vibration is observed in the frequency region  $470\text{-}410\text{ cm}^{-1}$  [6, 12]. The bands at  $873$ ,  $896$  and  $898\text{ cm}^{-1}$  can be assigned to V-O stretching vibration. These frequencies are higher than the frequency of V-O stretching bond as observed by several authors within  $820\text{-}871\text{ cm}^{-1}$  [13]. The strong bands at  $1062$ ,  $1065$  and  $1134\text{ cm}^{-1}$  can be assigned to Si-O-Si asymmetric stretching vibration. This frequency of Si-O-Si asymmetric stretching band is observed by several researchers [6, 14, 15]. The bands at  $1161$  and  $1191\text{ cm}^{-1}$  may be assigned to Si-O asymmetric stretching vibration. This frequency of Si-O band is observed by [16] within the range  $1100\text{-}1210\text{ cm}^{-1}$ . For the O-H stretching and bending vibration of water of crystallization all the hydrate salts absorb near  $1640\text{ cm}^{-1}$  [17] and therefore the broad Gaussian having maxima at  $1453$  and  $1650\text{ cm}^{-1}$  may be due to absorption of moisture. This atmospheric moisture can be easily absorbed by the pellet in recording IR spectra causing the appearance of IR bands belonging to O-H bending mode. A very strong and broad peak at  $3400\text{-}3487\text{ cm}^{-1}$  can be assigned to H-O-H stretching vibration. This frequency is observed by researchers [18, 19] within the range  $3200\text{-}3600\text{ cm}^{-1}$ . For the glasses in the ternary system the molar ratio  $\text{SiO}_2/\text{Na}_2\text{O}$  were kept 2 while  $\text{V}_2\text{O}_5$  substituted systematically into the glass system. The effect of  $\text{V}_2\text{O}_5$  is obvious from the change in spectral shape due to the addition of small amount of  $\text{V}_2\text{O}_5$ . The deconvoluted band positions the samples are slightly shifted towards high frequency and abrupt change in the relative height of the peak (Fig.v3) indicates the structural change. The region between  $400$  and  $550\text{ cm}^{-1}$  was attributed to the overlapping of the bending vibrations of Si-O-Si linkages from  $[\text{SiO}_4]$  structural units with the angular deformation vibrations of the V-O-V chains [20-22]. In the  $550\text{-}900\text{ cm}^{-1}$  region of the IR spectra, there are higher absorption band characteristic of motions of the vanadate network, more precisely of stretching vibrations of V-O bonds inside pyrovanadate and orthovanadate structural units. These bands can be decomposed in their components. The bands at about  $761\text{-}792\text{ cm}^{-1}$  are related to V-O stretching vibrations in the pyrovanadate structural units. The band at  $835\text{ cm}^{-1}$  is assigned to the asymmetric stretching vibration of the V-O-V bridges from  $[\text{V}_2\text{O}_7]^{4-}$  pyrovanadate structural units. With the increase of  $\text{V}_2\text{O}_5$  content up to 15 mol%, a sharp increasing trend was observed for IR absorption band. The structural changes occur with increase of  $\text{V}_2\text{O}_5$  content. The compositional evolution suggests the gradual conversion of pyrovanadate into orthovanadate structural units [23]. The bands at about  $997\text{-}1030\text{ cm}^{-1}$  can be assigned to stretching vibration of V=O. This frequency observed by several researchers [24-26]. With the increase of  $\text{V}_2\text{O}_5$  concentration V=O shifted towards high wavenumber [24]. The shifting of band of the V=O bond towards higher wavenumber indicates the presence of metavanadate phase containing  $\text{Na}_2\text{O}$  in the usual glass network. The comparison of the deconvoluted and observed chemical bonds with related glasses and crystalline phases are given in Table 4.

The influence of  $\text{V}_2\text{O}_5$  is discussed on the basis of change of spectral shape due to the systematic addition of  $\text{V}_2\text{O}_5$ . The variation in band positions and intensity of bands in NaSV1-NaSV5 glasses are different from that of NaSV10-NaSV15 glasses. This variation justifies the transformation of vanadate species, i.e. ortho and pyrovanadate are formed of 1-15 mol% containing glasses. Figure 5 (a&b) and Figure 6 (a&b) describe the variation of band positions and intensity i.e. relative area of the assigned bands with  $\text{V}_2\text{O}_5$  concentration without & with percentage error bars (5%). The band positions of Si-O-Si, V-O and Si-O are almost unchanged. The relative intensity i.e. relative area of V=O asymmetric stretching vibration decreased with  $\text{V}_2\text{O}_5$  concentration up to 5 mol% of  $\text{V}_2\text{O}_5$  concentration and then increased with the increase of  $\text{V}_2\text{O}_5$  concentration. This abrupt variation in relative area of V=O bond indicates the transformation of pyrovanadate phase into metavanadate phase as observed in IR analysis. The relative area of V=O bond show a positive correlation with  $\text{V}_2\text{O}_5$  content. This behavior is a clear indication that the band is formed due to vibrational motions involving  $\text{V}^{5+}$  ion.

The emergence of new peaks in the range  $960\text{-}1030\text{ cm}^{-1}$ , which can be assigned to the characteristic vibrations of V=O bond, indicates the formation of several crystalline phases in the region between  $\text{V}_2\text{O}_5$  and Sodium metavanadate ( $\text{NaVO}_3$ ). The successive increase of  $\text{V}_2\text{O}_5$  in these glasses makes it possible to form a series of complexes in the glasses. It can be concluded from the present IR studies of this system that the shifting of the V=O band towards the higher wavenumber with the addition of  $\text{V}_2\text{O}_5$  indicates that  $\text{VO}_5$  polyhedra are being formed with the addition of oxygen with  $\text{VO}_4$  polyhedra. This results the formation of a series of complexes with several V=O bonds.



The X-ray diffraction patterns of the base samples shown in Fig. 4 reveals that all the samples NaSV0-NaSV10 formed glass. The XRD pattern of non-crystalline materials i.e. glass was observed by many researchers [27]. The patterns show the amorphous nature of the samples within the diffraction limit of X-ray diffractometer. But the patterns of the heat-treated samples shown in Fig. 5 reveals that only the samples NaSV2 and NaSV5 formed crystallization and the other samples did not form crystallization. The reason of this fact may be the insufficiency in time of heat-treatment. If the samples were kept more time at required temperature, the samples would become crystallized.

**Table 4:** Comparison of the main IR absorption bands observed in base glasses with related glasses and crystalline phases

Remarks	Structural groups	Absorption band ( $\text{cm}^{-1}$ )	Corresponding references
Bending vibration	Si-O-Si	440	[6]
		465	[7]
		470	[8]
		[430-480]	Present work
Asymmetric stretching vibration	V-O-V	850	[28]
		820	[29]
		[835]	Present work
Stretching vibration	V-O	854 -871	[30]
		820	[13]
		[873-898]	Present work
Stretching vibration	V=O	966-1005	[24]
		1020	[25,26]
		[997-1030]	Present work
Asymmetric stretching vibration	Si-O-Si	1074	[14]
		1040	[6]
		1060	[15]
		[1062-1134]	Present work
Asymmetric stretching vibration	Si-O	1100-1210	[16]
		[1130-1191]	Present work
Vibration	O-H	1450-2090	[9]
		[1423-1456]	Present work
Bending vibration	H-O-H	1615-1649	[31]
		1629	[32]
		1674	[19]
		[1628-1697]	Present work
Stretching vibration	H-O-H	[3200-3600]	[18]
		3393	[32]
		[3400-3487]	Present work

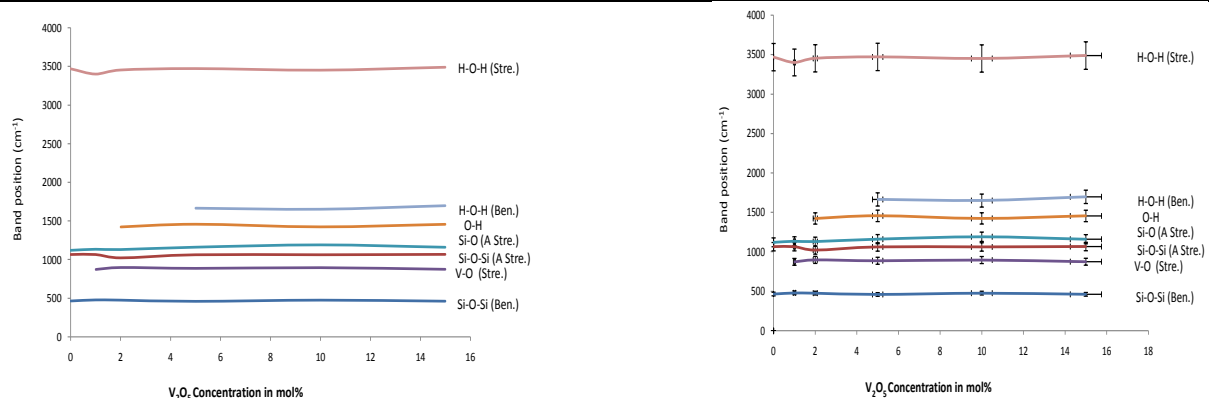


Figure 5: Variation of band position with  $\text{V}_2\text{O}_5$  concentration (a) without error bars (b) with error bars



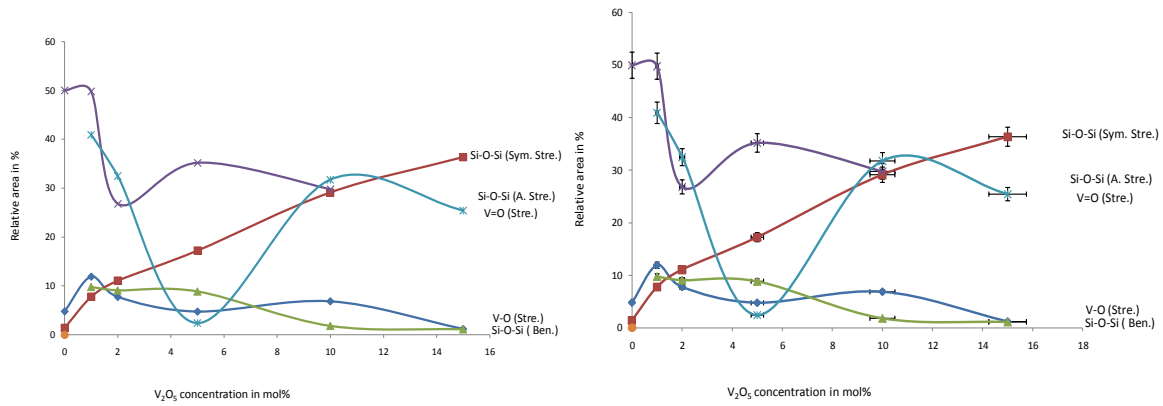


Figure 6: Relative areas of the chemical bonds as a function of  $V_2O_5$  concentration in mol% (a) without error bars (b) with error bars

## 5. Conclusion

The glass system  $xNa_2O \cdot 2xSiO_2 \cdot zV_2O_5$  were prepared by conventional method, where the ratio of  $SiO_2/Na_2O$  was 2 and the concentration of  $V_2O_5$  was  $z = 0, 1, 2, 5, 10$  and  $15$  mol%. The IR band positions in the glasses have a general tendency to shift towards high frequency region with the increase of  $V_2O_5$  concentration. The vanadate glasses exhibit well-defined bands characteristics of molecular groups identified as  $V=O$ ,  $V-O$ ,  $V-O-V$  and harmonics stretching  $V-O-V$  and  $V-O$  vibrations. The frequency of these groups is dependent on its bonding to other groups in the glass network. The variation in band positions and relative intensities with  $V_2O_5$  concentration suggests the glass system undergoes gradual structural changes i.e. pyro to ortho and metavanadate. The effect of  $V_2O_5$  is obvious for certain bonding mechanism where  $V^{5+}$  plays a significant role. The band positions of the  $Si-O-Si$  asymmetric stretching vibration shifted to the higher wavenumber with the increase of  $V_2O_5$ . The formation of  $O-H$  bond around  $1400, 1650$  and  $3400\text{ cm}^{-1}$  express the hygroscopic nature of the glass and provide wealth information about the structural units. The X-ray diffraction patterns of  $V_2O_5$  containing base samples  $NaSV0-NaSV15$  show the amorphous nature of the glass.

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