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## **SPECTROSCOPIC INVESTIGATION ON LITHIUM YTTRIUM SILICATE GLASSES DOPED WITH $V_2O_5$**

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### **ABSTRACT**

In this work, glass systems of the composition  $(40-x) Li_2O - 10Y_2O_3 - 50SiO_2: xV_2O_5$  where ( $x = 0.2, 0.4, 0.6, 0.8, 1.0$  mol%) have been prepared by the conventional melt quenching technique. The samples were characterized by the XRD. Optical absorption and IR of these glass samples have been investigated. Optical absorption spectra exhibits two broad absorption bands at about 640 and 1020 nm due to  $^2B_2 \rightarrow ^2B_1$  and  $^2B_2 \rightarrow ^2E$  transitions of  $VO^{2+}$  ions. With increase in the concentration of  $V_2O_5$ , the intensity of these peaks is observed to increase with a red shift. The IR spectral studies indicated that the glass samples contains various structural units with the linkages of the type  $Si-O-Si$ ,  $Si-O-V$ ,  $Y-O$ ,  $V-O-V$ ; the increasing content of  $V_2O_5$  in the glass samples seemed to have weakened such linkages. Finally, the analysis of the results of OA spectra of the studied glass have indicated that a considerable proportion of vanadium ions do exist in  $V^{4+}$  state in addition to  $V^{5+}$  state, and the redox ratio increases with increase in the concentration of crystallizing agent  $V_2O_5$ .

**Keywords:** *Glasses, Optical Absorption, IR*

### **INTRODUCTION**

Lithium silicate glasses with high thermal stability, chemical durability and good optical transparency over a wide range of wavelengths are particularly useful in data busses which cover short distances. When alkali silicate glasses are mixed with some sesquioxides (e.g.,  $Sb_2O_3$ ,  $Al_2O_3$ ,  $Y_2O_3$ ,  $La_2O_3$ ,  $Sc_2O_3$  etc.,), their thermo-physical, chemical and mechanical stability will be further improved. Such glasses were proved to be high efficient luminescence materials (Dikovska *et al.*, 2006; Alekseeva *et al.*, 2011; Hughes *et al.*, 2009; Kong *et al.*, 2005; Tokurakawa *et al.*, 2007; Guo *et al.*, 2004; Korzenksi *et al.*, 2001; SrinivasaRao *et al.*, 2011; Marasinghe *et al.*, 1998; Venkateswara Rao *et al.*, 2008; Moguš-Milanković *et al.*, 2012; Salem *et al.*, 2012). Among various sesquioxides, the addition of  $Y_2O_3$  to silicate glass systems widens the spectral range of transparency, enhances the refractive index and lowers phonon energies (SrinivasaRao *et al.*, 2011; Marasinghe *et al.*, 1998; Venkateswara Rao *et al.*, 2008).

In view of these qualities,  $Y_2O_3$  mixed lithium silicate glasses were considered as excellent host materials for rare earth ion doping and proved to be efficient both in continuous wave operation as well as in pulsed regimes.  $Yb^{3+}$  doped laser materials are of interest for the next generation nuclear fusion (Graça *et al.*, 2003; Shapaan *et al.*, 2012) and are being used as gain media in the microchip laser at sufficiently high doping levels (Salman and Mekki, 2011). Energy level structure of  $Yb^{3+}$  ion is exceptionally simple, consisting of a ground state manifold,  $^2F_{7/2}$ , Stark-split into four sublevels and an excited-state manifold,  $^2F_{5/2}$ , Stark-split into three sublevels. Thus, excited state absorption (ESA) for both the pump and signal wavelengths is absent for this ion. The upper manifold lies approximately  $10500\text{ cm}^{-1}$  above the ground level (Satyanarayana *et al.*, 2009). This large energy gap favors significant reduction of multi-phonon non radiative decay (Rao, 2002). The broad absorption spectrum due to Stark-splitting of this ion provides a wide choice of pump wavelengths. Further, the broad emission spectrum of  $Yb^{3+}$  ion and its large saturation fluencies enable to achieve lasing inverse level occupation and corresponding laser generation over a wide range of lasing region ( $\sim 1-1.2\text{ }\mu\text{m}$ ). Such interesting features of this rare earth ion make its host as an attractive medium for the generation and amplification of ultra-short pico-and femto second laser pulses (Ahmed *et al.*, 2012).



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A vanadate-based glass exhibits good semiconducting properties, relatively high electrical conductivity and chemical durability and also posses low crystallization tendency, melting point (Montani and Frechero, 2006; Prashant Kumar and Sankarappa, 2008; Reddy *et al.*, 2008; Saddeek *et al.*, 2009; Khattak *et al.*, 2009; Murawski, 1984; Shaaban *et al.*, 2009; Narayana Reddy C and Anavekar, 2008; El-Desoky, 2005).

These properties make vanadate glasses potential candidates for technological applications such as in electrical threshold, threshold switching, memory switching, optical switching devices as well as cathode materials for solid state devices and optical fiber (Ovshinsky, 1968; Drake *et al.*, 1969; Livage *et al.*, 1990; BalajiRao *et al.*, 2004; Pascuta *et al.*, 2008; Montani *et al.*, 1992; Ghosh and Chaudhuri, 1988).  $V_2O_5$  is known as a conditional glass former which does not form glass on their own, but readily forms glass only with a modifier such as alkali, alkaline earth and transition metal oxides (TMO) or other glass formers (Saddeek *et al.*, 2009; Singh *et al.*, 1988; Chiodelli *et al.*, 1982; VeerannaGowda VC and Anavekar, 2004).

Vanadate glasses also belong to the class of amorphous oxide semiconductors (Maniu *et al.*, 2004) where in this system the vanadium ions can adopt two different valence states,  $V^{4+}$  and  $V^{5+}$ . The electrical conduction is attributed to the hopping of  $3d^1$  unpaired electron from a  $V^{4+}$  site to a  $V^{5+}$  site which induces polarization of the lattice forming a polaron (Sayer and Mansingh, 1972; Mott, 1968; Austin and Mott, 1969; Chung and Mackenzie, 1980; Owen, 1977; Ghosh, 1990).

In the present paper we have synthesized  $Li_2O-Y_2O_3-SiO_2$  glasses doped with different concentrations of  $V_2O_5$  and investigated the structural changes that take place due to the varied oxidation states of vanadium ions using optical absorption and IR spectral studies.

### MATERIALS AND METHODS

For the present study, a particular composition viz., (40-x)  $Li_2O-10Y_2O_3-50SiO_2$ : x  $V_2O_5$  (with x ranging from 0 to 1.0) is chosen.

The details of composition are:

$V_0$ : 40.0  $Li_2O-10Y_2O_3-50SiO_2$

$V_2$ : 39.8  $Li_2O-10Y_2O_3-50SiO_2$ : 0.2  $V_2O_5$

$V_4$ : 39.6  $Li_2O-10Y_2O_3-50SiO_2$ : 0.4  $V_2O_5$

$V_6$ : 39.4  $Li_2O-10Y_2O_3-50SiO_2$ : 0.6  $V_2O_5$

$V_8$ : 39.2  $Li_2O-10Y_2O_3-50SiO_2$ : 0.8  $V_2O_5$

$V_{10}$ : 39.0  $Li_2O-10Y_2O_3-50SiO_2$ : 1.0  $V_2O_5$

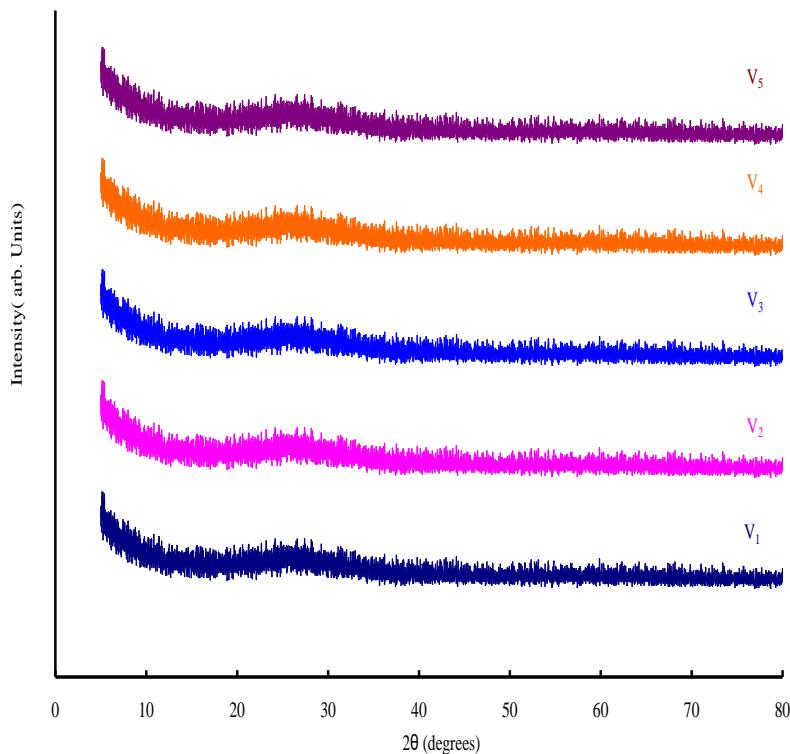
Among various compositions, this range of concentration seems to have formed a relatively clear and transparent glass. The starting materials used for the preparation of the glasses were Analytical grade reagents (99.9% pure) of  $LiCo_3$ ,  $Y_2O_3$ ,  $SiO_2$  and  $V_2O_5$ . Powders of these compounds in appropriate amounts (all in mol%) were thoroughly mixed in an agate mortar and melted in a platinum crucible in the temperature range of 1400–1450 °C in a PID temperature controlled furnace for about 1/2 h till a bubble free liquid was formed. It may be noted here that by trial and error we have found this particular range (1400–1450 °C) of temperature is the lowest possible temperature at which the samples could clearly be melted. The resultant bubble free melt was then poured on rectangular brass mold (containing smooth polished inner surface) kept at room temperature. The samples were subsequently annealed at 400°C in another furnace and cooled to room temperature at the rate of about 1 °C/min. The amorphous state of the prepared glasses was checked by X-ray diffraction spectra recorded on Xpert PRO, pan alytical X-ray diffractometer. Optical absorption spectra of  $V_2O_5$  doped glasses were recorded to a spectral resolution of 0.1 nm at room temperature in the spectral wavelength range covering 300–2100 nm using JASCO Model V-670 UV–vis–NIR spectrophotometer. IR transmission spectra of these glasses were recorded in KBr matrices in the range 400–2000  $cm^{-1}$  using potassium bromide pellets (300 mg) containing pulverized sample. These pellets were pressed in a vacuum die at -680 MPa.

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In the present paper we have synthesized  $\text{Li}_2\text{O}-\text{Y}_2\text{O}_3-\text{SiO}_2$  glasses doped with different concentrations of  $\text{V}_2\text{O}_5$  and investigated the structural changes that take place due to the varied oxidation states of vanadium ions using optical absorption, IR spectral studies.

**RESULTS AND DISCUSSION**

Figure 1 shows the XRD patterns of in  $\text{Li}_2\text{O}-\text{Y}_2\text{O}_3-\text{SiO}_2$  doped with different concentrations of  $\text{V}_2\text{O}_5$ . These X-ray diffraction patterns confirm the amorphous nature of the samples.



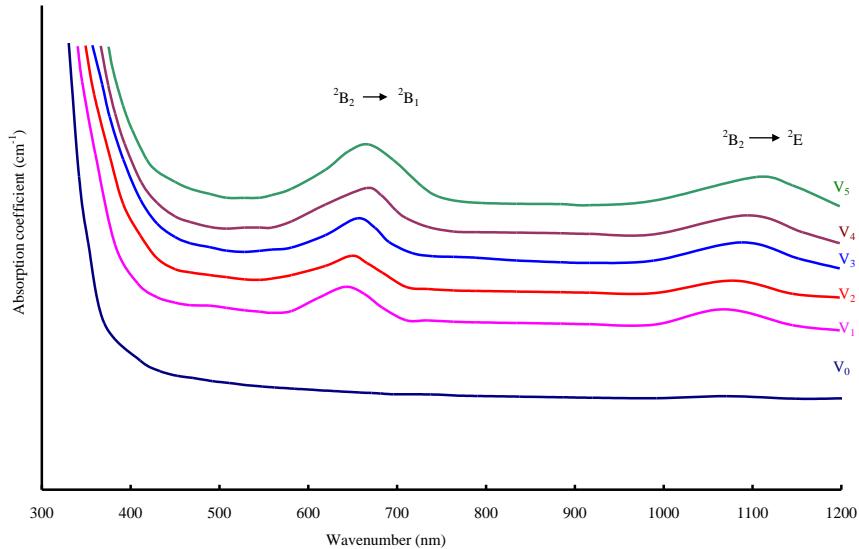
**Figure 1: XRD Patterns of  $\text{Li}_2\text{O}-\text{Y}_2\text{O}_3-\text{SiO}_2$  Doped with Different Concentrations of  $\text{V}_2\text{O}_5$**

Figure 2 shows the optical absorption spectra of  $\text{Li}_2\text{O}-\text{Y}_2\text{O}_3-\text{SiO}_2:\text{V}_2\text{O}_5$  glasses. The absorption edge observed at 340 nm for pure glass and it is found to be shifted gradually towards higher wavelength with an increase of the concentration of  $\text{V}_2\text{O}_5$ . Additionally, the spectra of glasses doped with 0.2 mol% of  $\text{V}_2\text{O}_5$  have exhibited two broad absorption bands at 650 and 1080 nm corresponding to  $^2\text{B}_2 \rightarrow ^2\text{B}_1$  and  $^2\text{B}_2 \rightarrow ^2\text{E}$  transitions of  $\text{VO}^{2+}$  ions (Ballhausen and Gray, 1962); with increase in the concentration of  $\text{V}_2\text{O}_5$  up to 1.0 mol%, the half width and peak height of these bands are observed to increase and shifted slightly towards higher wavelength.

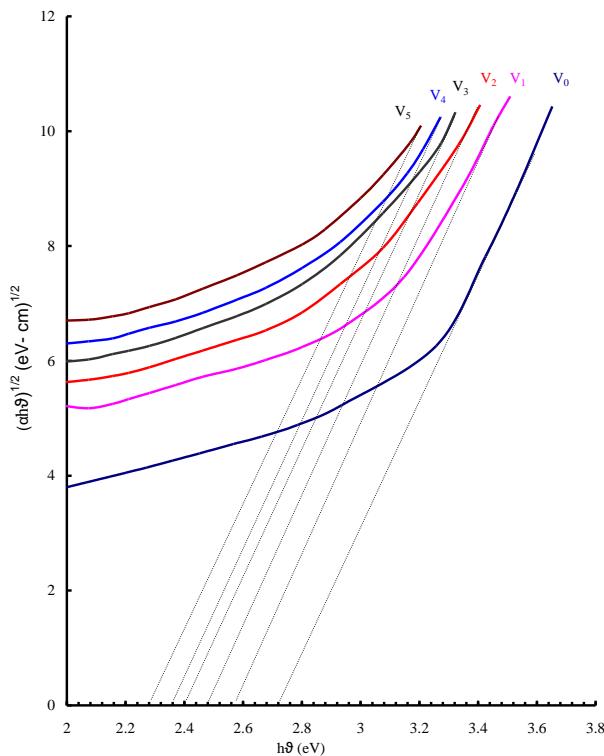
From the observed absorption edges, we have evaluated the optical band gaps ( $E_g$ ) of these glasses by drawing Urbach plots (Figure 3) of all glasses. From the extrapolation of the linear portion of these curves, the values of optical band gap ( $E_g$ ) obtained for  $\text{Li}_2\text{O}-\text{Y}_2\text{O}_3-\text{SiO}_2:\text{V}_2\text{O}_5$  glasses along with cut-off wavelengths and band positions are presented in Table 1. The value of  $E_g$  is found to decrease with the increase in concentration of  $\text{V}_2\text{O}_5$ .  $\text{V}^{4+}$  ion belongs to  $d^1$  configuration with  $^2\text{D}$  as the ground state. In the presence of pure octahedral crystal field, the  $^2\text{D}$  state splits into  $^2\text{T}_2$  and  $^2\text{E}$ , while an octahedral field with tetragonal distortion further splits the  $^2\text{T}_2$  level into  $^2\text{E}$  and  $^2\text{B}_2$ ; among these, the  $^2\text{B}_2$  will be the ground state. Further  $^2\text{E}$  level splits into  $^2\text{A}_1|3z^2-r^2\rangle$  and  $^2\text{B}_1|x^2-y^2\rangle$  where as  $^2\text{B}_2$  splits into three  $|xy\rangle$ ,  $|yz\rangle$  and  $|zx\rangle$  states. Thus, for the vanadyl ions we can expect 3 bands corresponding to the transitions  $^2\text{B}_2 \rightarrow ^2\text{B}_1$  ( $d_{xy} \rightarrow d_x^2 - d_y^2$ ),  $^2\text{B}_2 \rightarrow ^2\text{E}$  ( $d_{xy} \rightarrow d_{zx,yz}$ ) and  $^2\text{B}_2 \rightarrow ^2\text{A}_1$  ( $d_{xy} \rightarrow d_z^2$ ). However, in the spectra of the present glasses, only the first two bands are observed.

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We can understand the observed decrease in the optical band gap with increase in the concentration of  $\text{V}_2\text{O}_5$  is as follows: The gradual increase in the concentration of vanadyl ions, causes a creation of large number of donor centers; subsequently, the excited states of localized electrons originally trapped on  $\text{VO}^{2+}$  sites begin to overlap with the empty 3d states on the neighboring  $\text{V}^{5+}$  sites. As a result, the impurity band becomes more extended into the main band gap. This development might have shifted the absorption edge to the lower energy which leads up to a significant contraction in the band gap.



**Figure 2: Optical Absorption Spectrum of  $\text{Li}_2\text{O}-\text{Y}_2\text{O}_3-\text{SiO}_2$  Doped with Different Concentrations of  $\text{V}_2\text{O}_5$**

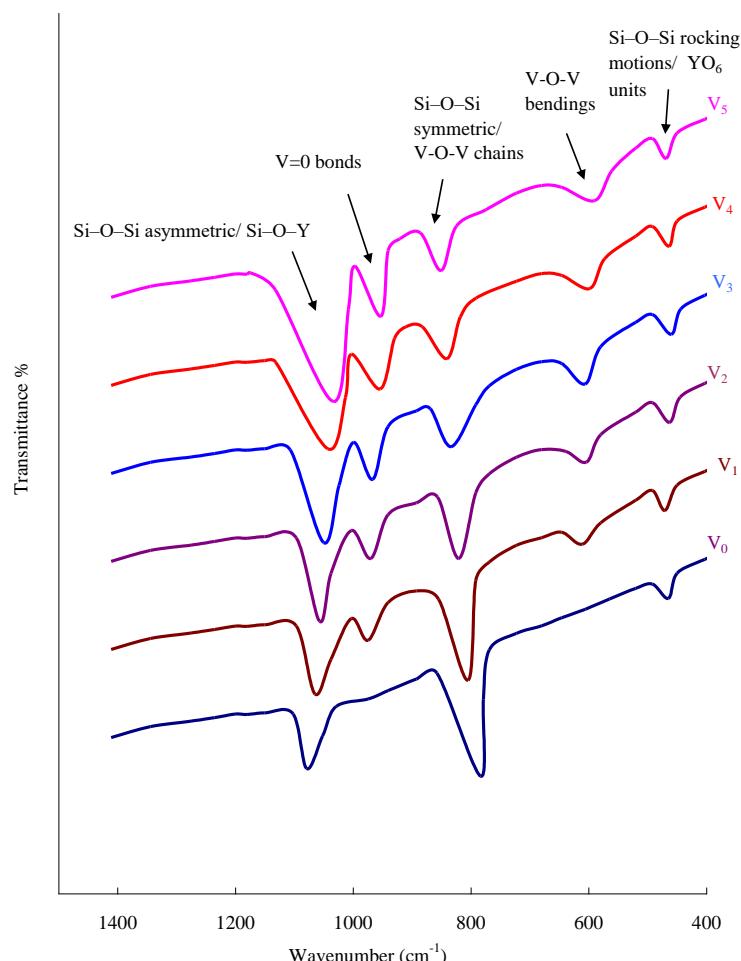


**Figure 3: Tauc Plots of  $\text{Li}_2\text{O}-\text{Y}_2\text{O}_3-\text{SiO}_2$  Doped with Different Concentrations of  $\text{V}_2\text{O}_5$**

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**Table 1: Data on Optical Absorption Spectra of  $\text{Li}_2\text{O}-\text{Y}_2\text{O}_3-\text{SiO}_2$ :  $\text{V}_2\text{O}_5$  Glasses**

Sample	Cut-off Wavelength (nm)	Optical Band Gap $E_g$ (eV)	Position of ${}^2\text{B}_2 \rightarrow {}^2\text{B}_1$ Band (nm)	Position of ${}^2\text{B}_2 \rightarrow {}^2\text{E}$ Band (nm)
$\text{V}_0$	330	2.72	--	--
$\text{V}_2$	340	2.58	642	1068
$\text{V}_4$	351	2.48	649	1077
$\text{V}_6$	357	2.41	655	1081
$\text{V}_8$	366	2.36	665	1099
$\text{V}_{10}$	375	2.28	668	1104



**Figure 4: IR Spectrum of  $\text{Li}_2\text{O}-\text{Y}_2\text{O}_3-\text{SiO}_2$  Doped with Different Concentrations of  $\text{V}_2\text{O}_5$**

**Table 2: Data on Infrared Spectra  $\text{Li}_2\text{O}-\text{Y}_2\text{O}_3-\text{SiO}_2$ :  $\text{V}_2\text{O}_5$  Glasses Recorded at Room Temperature (Assignment of Band Positions in  $\text{cm}^{-1}$ )**

Assignment	$\text{V}_0$	$\text{V}_2$	$\text{V}_4$	$\text{V}_6$	$\text{V}_8$	$\text{V}_{10}$
Si-O-Si asymmetric/ Si-O-Y	1079	1065	1057	1050	1043	1034
$\text{V}=\text{O}$	--	975	970	967	953	950
Si-O-Si symmetric/V-O-V chains	784	805	821	834	841	851
V-O-V bendings	--	611	604	606	598	590
Si-O-Si rocking motions/ $\text{YO}_6$ units	465	471	463	460	467	469

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In Figure 4, the IR spectra of  $V_2O_5$  doped  $Li_2O-Y_2O_3-SiO_2$  glasses are presented. The spectra have exhibited conventional vibrational band due to  $Si-O-Si$  asymmetric vibrations at about  $1083\text{ cm}^{-1}$  and another band at about  $787\text{ cm}^{-1}$  due to  $Si-O-Si$  symmetric vibrations or due to bending mode of bridging oxygen situated perpendicularly to  $Si-Si$  axis within the  $Si-O-Si$  plane (Nakamura *et al.*, 1984; Srikumar *et al.*, 2011). The octahedral band of yttrium ions ( $YO_6$ ) is also located in these spectra at about  $482\text{ cm}^{-1}$  (Yanmin Q and Hai, 2009; Lianga *et al.*, 2011). The band due to  $Si-O-Si$  rocking motion is also predicted in this region (Rao, 2002; Nakamura *et al.*, 1984). With the introduction of  $V_2O_5$ , an additional band at  $970\text{ cm}^{-1}$  due to  $V-O$  stretching of  $V=O$  groups, band at  $815\text{ cm}^{-1}$  due to  $V-O-V$  stretchings and band at  $600\text{ cm}^{-1}$  due to  $V-O-V$  bending vibrations (Tanaka *et al.*, 1989). The bands observed in the spectra of glass  $V_1$  at  $980, 800\text{ cm}^{-1}$  can there be considered as common vibrational modes due to  $Si-O-V$  stretchings. As the concentration of  $V_2O_5$  is increased in the glass samples gradually, the bands due to asymmetrical vibrations of silicate and other structural units are observed to grow at the expense of symmetrical bands. The relevant data related to IR spectra of these glasses are presented in Table 2.

### Conclusion

The glass samples  $Li_2O-Y_2O_3-SiO_2$  doped with different concentrations of  $V_2O_5$  are prepared. The characterization of the samples by SEM clearly indicates the amorphous nature of the samples. The IR spectral studies indicated that the glass samples contains various structural units with the linkages of the type  $Si-O-Si$ ,  $Si-O-Y$ ,  $Si-O-V$ ,  $V=O$  and  $V-O-V$ ; the increasing content of  $V_2O_5$  in the glasses seemed to have weakened such linkages. The analysis of the results of optical absorption spectra of the studied glass have indicated that a considerable proportion of vanadium ions do exist in  $V^{4+}$  state in addition to  $V^{5+}$  state, and the redox ratio increases with increase in the concentration of  $V_2O_5$ .

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