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In the name of God, the Compassionate, the Merciful

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Optical and Thermal Properties of Mixed Alkali Phosphate Based Glasses

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ABSTRACT— Glassy samples with a composition of $40\text{P}_2\text{O}_5-30\text{V}_2\text{O}_5-(30-x)\text{Li}_2\text{O}-x\text{K}_2\text{O}$, $0 < x < 30$ (mol %) were prepared by the conventional melt quenching technique in two forms of bulk and blown film. X-ray diffraction patterns corroborated the amorphous feature of the samples. Density of samples was investigated by using Archimedes principle. Based on absorption and reflection spectra, indirect allowed optical gaps, Urbach energies, refractive index and dielectric coefficients were calculated. Study of FTIR spectra revealed that all of the samples mainly contain metaphosphate and pyrophosphate units. Glass transition temperatures were also evaluated using DSC curves. Non-linear variation of many physical properties by gradually substitution of Li ions with K, confirmed the existence of mixed alkali effect in glassy system.

KEYWORDS: Phosphate Glasses, Mixed alkali effect, Refractive index, UV, FTIR, DSC

I. INTRODUCTION

Many glassy networks containing alkali elements have exhibited a non-linear behavior of physical properties, including electrical conductivity, glass transition temperature, density, viscosity and spectroscopic parameters by progressive substitution of one alkali element with another one. This fact is well-known as mixed alkali effect (MAE) [1-8]. Order of departure from linearity depends on total concentration of alkali elements, size and mass difference of alkali elements and temperature of observation [1-3]. It should be mentioned that the MAE is just detected, when

the total alkali concentration is higher than ~10mol% [1]. Glasses with non-linear behavior can have fundamental role in tuning favorable properties in technology and industry [6, 9]. Schott and co-workers developed phosphate based glasses, with comparatively higher refractive indices than silicate optical glasses about 100 years ago [10]. P_2O_5 glasses form tetrahedral PO_4^{3-} units with three bridging oxygen (BO) and one non-bridging oxygen (NBO) in which, PO_4^{3-} units are connected to each other via bridging oxygen atoms [10, 12]. Phosphate based glasses have attracted considerable attention, due to their technological and biological applications and some unique physical properties, compared with borate and silicate glasses, such as high thermal expansion coefficients, lower transition temperatures (T_g), UV transmission and optical features [12-16]. Several investigations have shown that, in spite of low chemical durability of pure phosphate glasses, the addition of some transition metal ions (TMIs), alkali and alkaline earth oxides into glass networks can remarkably increase their durability. It is shown that the chemical durability of these glasses increase by displacement of P–O–P bonds with more stable P–O–M (M= V, W, Fe, Mo, ...) bonds [13-15, 17-19]. Structural units of phosphate glasses are classified in the Q^n groups, where n represents the number of BOs per unit. As the modifying oxides are added to P_2O_5 , structural groups pass from Q^3 (Ultraposphate) to Q^2 (Metaphosphate), Q^1 (Pyrophosphate), Q^0 (Orthophosphate) and

creating NBOs [10, 20]. Furthermore, spectroscopic studies have shown that TMIs in alkali phosphate glasses mostly exist in more favorable low valance state or octahedral coordination than in high valance state or tetrahedral coordination [15, 21].

Previously, mixed alkali effect was studied mostly in borate systems. The aim of the present paper is to investigate the MAE for its optical, thermal and also other physical properties of new quaternary $40\text{P}_2\text{O}_5-30\text{V}_2\text{O}_5-(30-x)\text{Li}_2\text{O}-x\text{K}_2\text{O}$, with $0 < x < 30$ (mol %) phosphate based glassy samples.

II. EXPERIMENTAL TECHNIQUE

New glassy samples with general formula $40\text{P}_2\text{O}_5-30\text{V}_2\text{O}_5-(30-x)\text{Li}_2\text{O}-x\text{K}_2\text{O}$ where $x=1, 5, 15, 25, 29$ mol%, were prepared by the conventional melt quenching technique. Stoichiometric amount of initial materials were weighed and the well – ground mixtures were heated in alumina crucibles up to their melting temperature, typically 850°C in an electric furnace. After one hour, the melt was poured on a preheated stainless steel plate into annealing furnace. The obtained samples were then annealed for 3h near the glass transition temperature to relieve the mechanical and thermal stresses resulting from the quenching. In order to prepare blown films, alumina tube was used so that, end of the alumina tube was dipped into a molten material in crucible. Finally, by steady blowing, thin blown films were prepared with constant micro thickness from each composition.

A. X-ray diffraction pattern

X-ray diffractometry (XRD) was used to confirm the amorphous nature of the samples with an equipment, manufactured by Bruker , model D8 Advance X-ray diffractometer and the measurements were carried out in the range of $10^\circ < 2\theta < 120^\circ$ angles.

B. Density

The density of the glassy samples was determined by using the Archimedes principle

with ethyl-methyl-ketone as the immersion liquid at room temperature.

C. Ultraviolet and visible spectra

The optical absorption and reflectance spectra of the blown glassy films were recorded with an UV-Vis spectrophotometer, manufactured by Scinco, model s4100 in the wavelength region of 200-900nm at room temperature.

D. Infrared spectra

The FTIR spectra of glassy blown films were recorded using KBr pellet technique with a spectrometer, manufactured by Unicam, model 4600 in the frequency range of $4000-400\text{ cm}^{-1}$ at room temperature.

E. Differential scanning calorimetry

Differential scanning calorimetry (DSC) was used to investigate the thermal behavior of the glass samples and to determine the glass transition temperature with a DSC, manufactured by Netzsch, model 400. For this purpose 6-9 (mg) of the powdered glass samples were sealed in alumina pans and heated with a rate of $10^\circ\text{C}/\text{min}$ using Ar as purge gas in the temperature range of $100-550^\circ\text{C}$.

III. RESULTS AND DISCUSSION

A. Amorphous structure

XRD patterns confirmed the amorphous nature of the samples. As is shown in Fig. 1 the patterns do not manifest any distinct or sharp peak. A broad halo in patterns is displaying an amorphous phase of samples [14, 22].

B. Density

The density of samples, D , was determined by using Archimedes principle. From the density values, the molar volume, V_m of the samples was calculated by using the equation:

$$V_m = M/D \quad (1)$$

where $M = \sum x_i M_i$, x_i is the mole fraction of oxide (i), M_i is the molecular weight of oxide (i). Fig. 2 depicts the compositional

dependence of density and molar volume. As is shown in this figure, their non-linear variation proves the existence of MAE.

Table 1. Physical properties of PVLK glassy system

PVLK (mol%)	D (g/cm^3)	V_m (cm^3)	OPD ($atom/l$)	N_{Li} ($10^{22} ion/cm^3$)	N_V ($10^{22} ion/cm^3$)	E_{opt} (eV)	E_U (eV)	β ($cm.eV$) ^{-1/2}	T_g ($^{\circ}C$)
40-30-29-1	2.828	42.80	88.78	0.82	0.84	2.06	0.38	18.85	365
40-30-25-5	2.901	42.60	89.19	0.71	0.85	2.10	0.43	17.30	348
40-30-15-15	2.723	47.74	79.60	0.38	0.75	2.20	0.34	23.16	345
40-30-5-25	2.523	54.06	70.29	0.11	0.67	2.29	0.29	25.27	341
40-30-1-29	2.711	51.26	74.14	0.02	0.70	2.07	0.37	21.53	343

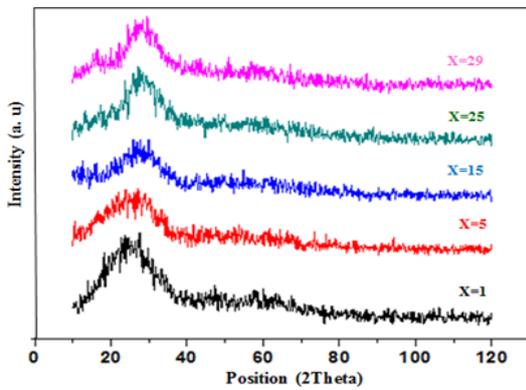


Fig. 1. XRD patterns of samples.

Oxygen packing density (OPD), the number of oxygen atoms per formula unit of samples was evaluated by using the following equation:

$$OPD = \frac{D}{M} \quad (2)$$

Ion concentration of samples, N was also determined by using the following expression:

$$N = \frac{DN_{avo}P}{M} \quad (3)$$

where N_{avo} is the Avogadro number, $P = nx$ and defines as mole fraction of oxide in glass composition multiplied number of ions atom in an oxide [23-26]. The calculated values of above parameters are given in Table 1. As is seen, ion concentration of Lithium decreases with increasing of Potassium content in glass network.

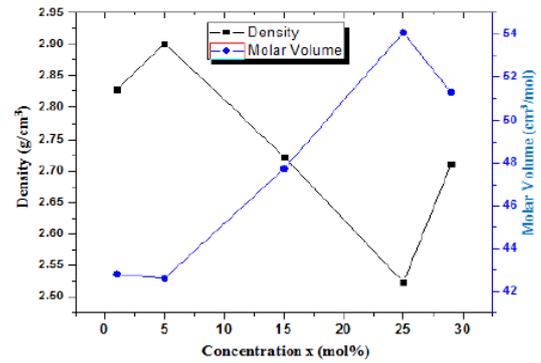


Fig. 2. Compositional dependence of density and molar volume.

C. Optical band gap and Urbach energy

The study of optical absorption in the ultraviolet and visible regions is a useful technique for investigation of band structure and band gap energy in crystalline and non-crystalline systems. Fig. 3 represents the optical absorption spectra of all samples. It is clear that, the absence of sharp absorption edge in spectra, exhibits the amorphous nature of samples [9, 28].

The optical absorption coefficient, $\alpha(\nu)$, can be calculated from the absorption spectra data using the equation:

$$\alpha(\nu) = \frac{1}{l} \left(\frac{I_0}{I_t} \right) \quad (4)$$

where I_0 and I_t are the intensities of the incident and transmitted UV-Vis ray and l is the thickness of the blown films.

Fig. 4 represents the $Ln\alpha$ versus $h\nu$. As is seen, optical absorption coefficient $\alpha(\nu)$ of glassy semiconductors increases with photon energy and divided into three regions: A, B, and C.

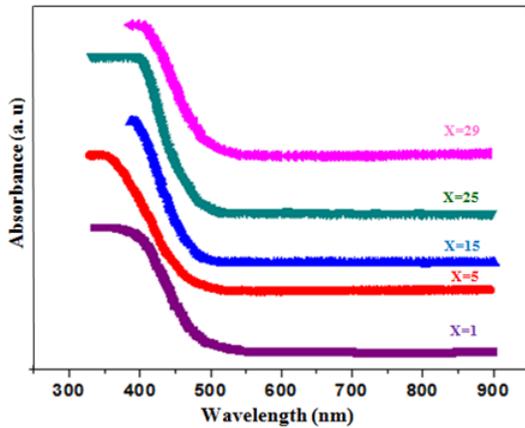


Fig. 3. Absorbance spectra of all samples.

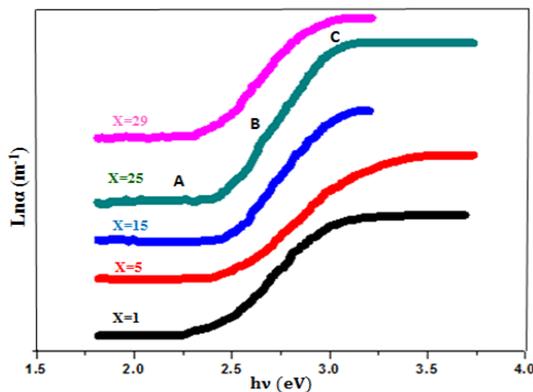


Fig. 4. $Ln\alpha$ versus $h\nu$ for all samples.

The first absorption region, A, is shown in low energies, which there is no excited states and the order of the absorption coefficient depends only on the material and its purity [12, 29]. In the second absorption region (B) or Urbach absorption range, the absorption coefficient $\alpha(\nu)$ is an exponential function of the photon energy $h\nu$ and follows the Urbach rule:

$$\alpha(\nu) = \alpha_0 \exp\left(\frac{h\nu}{E_U}\right) \quad (5)$$

where α_0 is a constant, $h\nu$ is the incident photon energy, and E_U is the Urbach energy, which determines the extent of localized states

tailing in the band gap. Sumi and Toyozawa attributed the exponential behavior of absorption coefficient to the interaction of excitons and electrons with phonons.

E_U values of all samples have been calculated by using the above equation and slopes of the Fig. 4 in the B region, which are listed in Table 1. According to Table 1 the Urbach energy vary between 0.29-0.43 eV, which is in accordant with reported data for inorganic glassy systems [9, 23, 28, and 30].

In the third absorption region (C) or high absorption region, the photons have enough energy to excite electrons from valance to conduction band and the absorption coefficient $\alpha(\nu)$ can be written as follow:

$$\alpha(\nu) = \beta \frac{(h\nu - E_{opt})^m}{h\nu} \quad (6)$$

where β is a constant, E_{opt} is an optical band gap and the index m depends on type of electron transitions in k -space and takes the values 2, 1/2 for indirect allowed and direct allowed transitions, respectively. Fig. 5 represents the plots of $(\alpha h\nu)^{1/2}$ versus $h\nu$, for $m = 2$ and the values of optical band gap have been calculated by extrapolation linear region of the $(\alpha h\nu)^{1/2}$ versus $h\nu$ curve to $(\alpha h\nu)^{1/2} = 0$. The values of E_{opt} and constant parameter β are listed in Table 1. According to Table 1, values of optical band gap and Urbach energy vary non-linearly with concentration of x, which manifest the existence of mixed alkali effect. Mixed alkali effect also observed in physical properties of borate glasses and in the absence of acceptable universal theory to explain MAE in optical properties of glasses, the non-linear variation of optical gap is attributed to the formation of large number of non-bridging oxygens (NBO) [9, 12, 14, 23, 28 and 31].

The refractive index plays a crucial role in the optical materials and application of glasses.

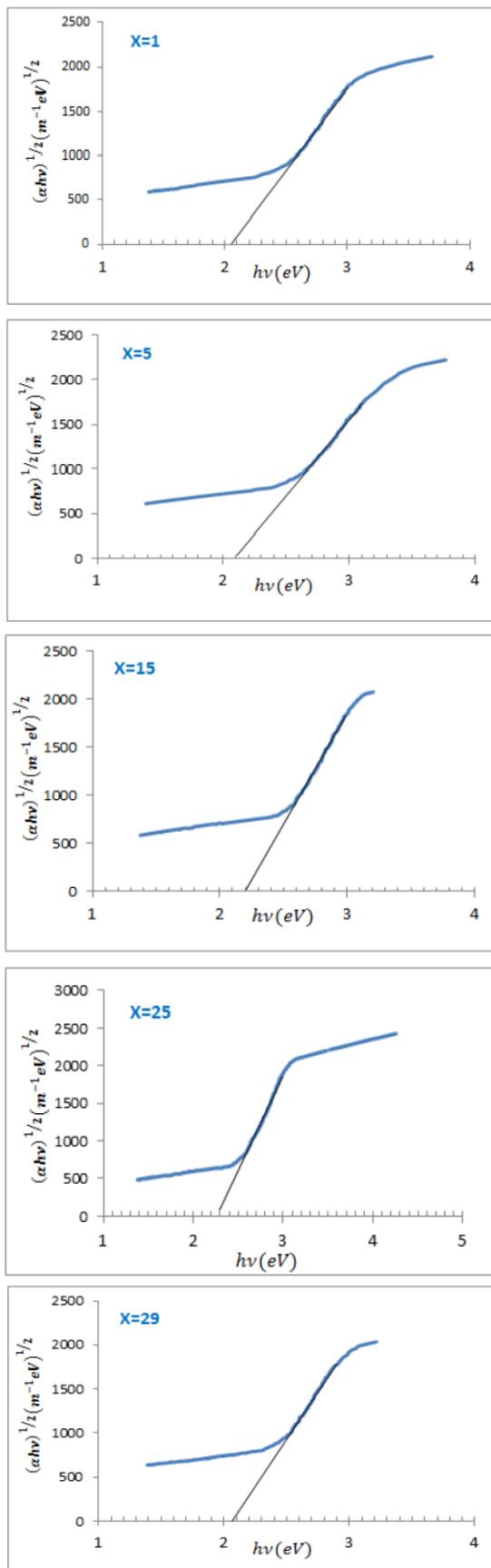


Fig. 5. $(\alpha hv)^{1/2}(m^{-1}.eV)^{1/2}$ versus $hv(eV)$ refractive index

Fig. 6 represents the reflectance spectra of samples in the visible range. The refractive index of blown films was calculated by using the following equation (for normal incident angle):

$$n = \left(\frac{1+R}{1-R} \right) + \sqrt{\frac{4R}{(1-R)^2} - k^2} \tag{7}$$

where R is the reflectance and $k = \frac{\alpha \lambda}{4\pi}$ is the extinction coefficient [1, 32]. Fig. 7 shows the wavelength dependence of the refractive index. Furthermore, other physical parameters such as dielectric coefficient (ϵ), molar refractivity (R_M) and electronic polarizability (α_e) were calculated in two wavelengths (400 and 700 nm) by using the following equations:

$$\epsilon = n^2 \tag{8}$$

$$R_M = \frac{M}{D} \left(\frac{n^2 - 1}{n^2 + 2} \right) \tag{9}$$

$$\alpha_e = \frac{3}{4\pi N_t} \left(\frac{n^2 - 1}{n^2 + 2} \right) \tag{10}$$

where N_t is the number of vanadium ions per unit volume [32-34]. The obtained values are given in Table 2. As is observed, due to MAE refractive index, dielectric coefficient, molar refractivity and electronic polarizability have non-linear trend by changing alkali ions concentration in glassy system.

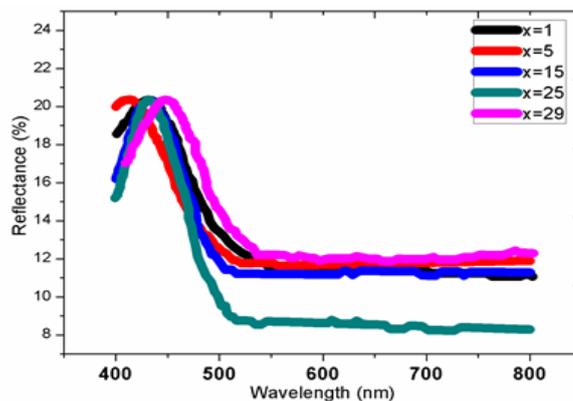


Fig. 6. Reflectance spectra of samples.

D. FTIR

Fourier transform infrared (FTIR) spectrum is a basic technique for investigation of structure and covalent bonds in chemical compositions, which each component absorb incident photon energy in specific wavenumber in IR region [12]. The FTIR spectra of all samples are shown in Fig. 8 in the wavenumber range of

400-4000 cm^{-1} and all detected bonds are summarized in Table 3. Glassy networks with structure of ultraphosphate, composed of one non-bridging and three bridging oxygens. The fundamental feature of ultraphosphate units is a P=O bond at about 1378 cm^{-1} [12, 14, 31, 35].

Table 2. Physical properties of PVLK glassy system

PVLK (mol%)	n_{400}	n_{700}	ε_{400}	ε_{700}	$R_M (\text{cm}^{-3})$		$\alpha_e (10^{-24} \text{cm}^3/\text{ion})$	
					400 (nm)	700 (nm)	400 (nm)	700 (nm)
40-30-29-1	2.513	2.019	6.315	4.076	27.385	21.668	18.09	14.33
40-30-25-5	2.617	2.045	6.849	4.182	28.162	21.930	18.62	14.50
40-30-15-15	2.346	2.000	5.504	4.000	28.655	23.870	18.94	15.78
40-30-5-25	2.276	1.807	5.180	3.265	31.474	23.258	20.82	15.38
40-30-1-29	2.370	2.058	5.617	4.235	31.070	26.595	20.54	17.58

Table 3. Bond assignments of samples in FTIR spectra.

Wavenumber (cm^{-1})	Bond assignment	x=1	x=5	x=15	x=25	x=29
~1624	$\text{P}_2\text{O}_7^{4-}$	1625	1636	1633	1629	1629
~930	Asymmetric P-O-P	932	932	909	917	917
~760	Symmetric P-O-P	767	764	770	762	762
~630-760	V-O-V V-O-P	767	767	770	762	762
~330-520	O-P-O	462	462	462	437	530
~1100	V=O	1094	1094	1094	1101	1101
~430-680	Lattice vibration of V_2O_5	462 616	462 585	462 608	437 585	531 ????
~3436	O-H	3432	3455	3455	3478	3470

However, when P_2O_5 mixed with other oxides, such as transition metal oxides, some changes occur in position and extent of the bonds [31]. In glass networks, V_2O_5 has a dual role, partly acts as a glass modifier and partly as a glass former [14, 20]. With addition of V_2O_5 modifier oxide into glass network, it causes a position shift of P=O bond to lower frequency of $\sim 1260 \text{cm}^{-1}$ and gradually conversion of ultraphosphate units to metaphosphate and pyrophosphate units [12]. Hence, we can attribute the broad bond around 1260cm^{-1} to formation of metaphosphate and pyrophosphate units. Moreover, V_2O_5 modifier causes a depolymerization of the glass network and shortens the long chains by converting P-O-P to P-O-V bonds [14, 20].

The broad bonds around 1160cm^{-1} are related to asymmetric stretching of PO_2 units [14].

Also the peaks around 1624cm^{-1} are due to stretching vibration of $\text{P}_2\text{O}_7^{4-}$ (pyrophosphate)

units [36]. Absorption bonds around 1260cm^{-1} , 1160cm^{-1} and 1624cm^{-1} manifest that all samples under investigation contain mainly metaphosphate and pyrophosphate units. The peaks around 930cm^{-1} and 760cm^{-1} are related to the asymmetric and symmetric P-O-P bonds, respectively [14, 35, 37].

The bonds observed in $\sim 330-520 \text{cm}^{-1}$ range belong to the bending vibration of O-P-O phosphate chains [20, 37]. The peaks obtained in the region $\sim 630-760 \text{cm}^{-1}$ are due to V-O-V and V-O-P bonds [20]. The bonds situated in the range $\sim 430-680 \text{cm}^{-1}$ are attributed to the lattice vibration of V_2O_5 [37]. The bonds around 1100cm^{-1} pertain to V=O vanadyl groups in VO_5 trigonal bipyramids and a stretching vibration of V=O [14, 37]. It is worth to note that, the vibration modes of mobile ions occur in low frequencies (a few tens in units of cm^{-1}) and far-infrared spectroscopy is suitable in detecting such

peaks [12]. Furthermore, the absorption peaks at $\sim 3436\text{ cm}^{-1}$ are assigned to stretching vibration of O–H bonds, due to hygroscopic nature of P_2O_5 [12, 36, 38]. Finally non-linear variation of peaks position with concentration of x , exhibits the existence of MAE.

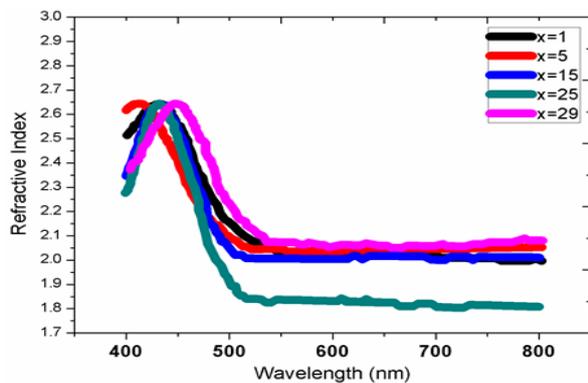


Fig. 7. Refractive index spectra.

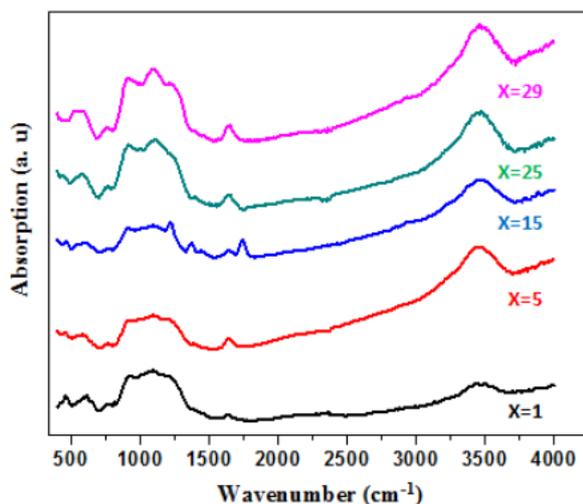


Fig. 8. FTIR spectra of samples

E. DSC

Glass transition temperature, T_g is a significant feature, which is exhibited by the supercooled liquids and play important role in material science [39]. Fig. 9 shows the DSC curves of all samples in the temperature range of 100–550°C. As is shown, all samples manifest the endothermic peak as a feature of glass transition temperature. The obtained T_g values are listed in Table 1. Fig. 10 shows the compositional dependence of glass transition temperature. T_g decreases with increasing of

concentration x and reaches a minima in composition $x=25$ (mol%) and then increases, which non-linear variation of T_g reveals the existences of MAE. The decrease in T_g can be attributed to small increase in formation of non-bridging oxygen [40].

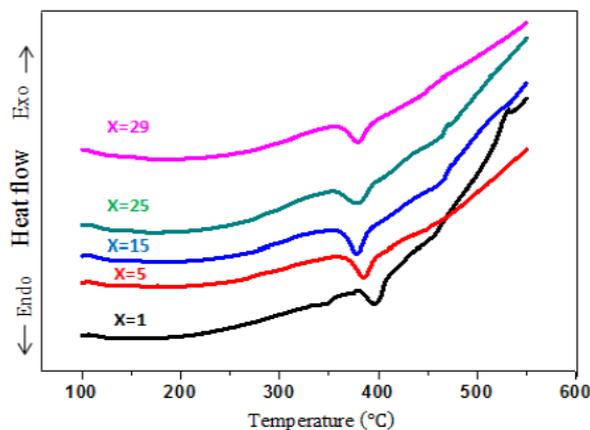


Fig. 9. DSC curve of all samples

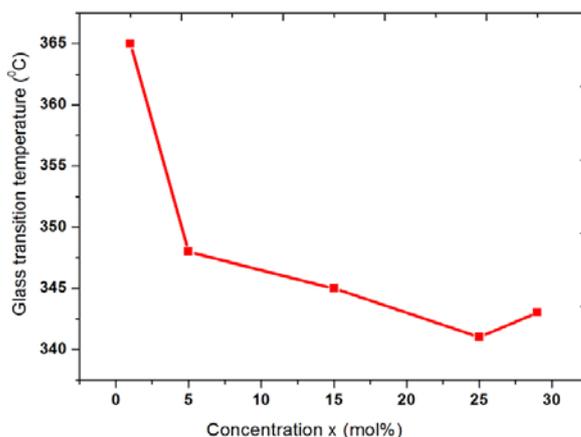


Fig. 10. Compositional dependence of T_g

IV. CONCLUSION

Glassy samples with general formula $40\text{P}_2\text{O}_5-30\text{V}_2\text{O}_5-(30-x)\text{Li}_2\text{O}-x\text{K}_2\text{O}$, $0 < x < 30$ (mol %) were prepared by standard melt quenching method and following results achieved from physical analyses:

- a. Absence of distinct and sharp peaks in XRD patterns, confirmed the amorphous nature of samples.

- b.** Values of density, molar volume and oxygen packing density were evaluated and confirmed mixed alkali effect.
- c.** Optical gaps and Urbach energies were calculated by using ultraviolet and visible spectra. Results of Urbach energies varied between 0.29-0.43 eV, which are in accordant with inorganic glass systems and also non-linear variation of E_{opt} and E_U proved the existence of mixed alkali effect.
- d.** Refractive index, dielectric coefficient, molar refractivity and electronic polarizability changed non-linearly as a consequence of mixed alkali effect phenomena.
- e.** Study of FTIR spectra, showed the basic bonds of phosphate and vanadium units, also most of the bond positions varied non-linearly by replacing Li ions with K ions.
- f.** Thermal behavior of samples were investigated and glass transition temperature (T_g) varied non-linearly with concentration x . The decreasing of T_g , is attributed to small increase in number of non-bridging oxygens.
- g.** Finally, mixed alkali effect phenomenon was strongly revealed in several physical properties of present prepared glassy samples, which can be useful in designing suitable compound for particular applications in technology and industry.

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