

Research Article

Effect of Zinc oxide on the structure-properties of $(35-x)\text{Li}_2\text{O}-x\text{ZnO}-15\text{PbO}-50\text{P}_2\text{O}_5$ glassesMohammed Abid^{1,2*}, Khadija Bougrine², Mustapha Hafid² and M'hamed Taibi³¹Regional Center for Education and Training Trades (CRMEF), Laboratoire de Physique-Chimie, Région de Rabat-Salé- Kénitra, Annexe-Kénitra, Morocco²Ibn Tofail University in Kenitra, Sciences Faculty, Laboratoire d'Optoélectronique, Physico-Chimie des Matériaux et Environnement, B.P., 133, 14000 Kénitra, Morocco³Mohammed V University in Rabat, Centre des Sciences des Matériaux, Laboratoire de Physico-Chimie des Matériaux Inorganiques et Organiques(LPCMIO), ENS, B.P. 5118, Rabat 10000, Morocco**Abstract**

Quaternary lithium zinc lead phosphate glass system with composition of $(35-y)\text{Li}_2\text{O}-y\text{ZnO}-15\text{PbO}-50\text{P}_2\text{O}_5$ ($y=0; 5; 15; 25; \text{ and } 35$ mol%) was successfully prepared using a conventional melt-quenching method. Their composition dependence of physical and spectroscopic properties such as density, molar volume, glass transition temperature and FT-IR spectra have been discussed in association with the effect of substitution of zinc oxide (ZnO) for lithium oxide (Li_2O). Thus, ZnO is assumed to play an intermediate role in a way that Zn^{2+} cation tends to break P-O-P bonds forming ZnO_4 units with covalent Zn-O bonds. The predominant structural units in all these glasses are metaphosphate (P_2O_6)²⁻ groups. An incorporation model of ZnO into the glassy network is proposed.

Keywords: Phosphate glasses, Zinc oxide, DSC, density, Molar volume, Infrared spectroscopy

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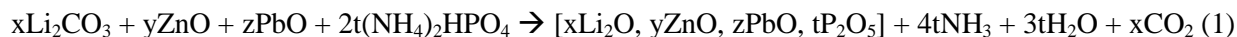
Introduction

The basic structural phosphate network is made of corner-sharing PO_4 tetrahedral units that can be classified according to their connectivity, Q_n terminology, where n is the number of bridging oxygen (BOs) per tetrahedron. In this formalism, the pure phosphate glasses are formed by Q_3 tetrahedra with three BOs and one double bonded oxygen $\text{P}=\text{O}$, forming a three-dimensional cross-linked network. In ultra-phosphate glasses both Q_2 and Q_3 structures are present, while in metaphosphate glasses prevailed Q_2 tetrahedra which form long chains or rings. Beside these, small phosphate groups like: pyro Q_1 and orthophosphate Q_0 could also be present in the glass structure [1]. Pure phosphate glass (P_2O_5) have a poor chemical durability, a high hygroscopic and a volatile nature with regard to hydrolysis of the P-O-P bonding by atmospheric moisture which restricted their use in replacing silicate glasses for an enhanced range of technological applications [2,3]. Hence, a mixture of oxides including network modifiers and network intermediates are added to the phosphate glass system to stabilize the glass network because P-O-M (where M = metal cation) bond is generally more stable toward atmospheric hydrolysis or solution attack [4-6]. This has led to the incorporation of oxide like ZnO [7-9], PbO [10, 11], CaO [12], TiO_2 [6, 13], Fe_2O_3 [14-16], etc. into phosphate glassy network. The Zn^{2+} ion is known to adopt four-coordinated sites in oxide glasses [17, 18]. X-ray diffraction profiles and pair function distribution calculations of zinc phosphate glasses indicate that Zn^{2+} cation is tetracoordinated and surrounded by four oxygens [19]. Musinu et al. [20] recently investigated the coordination of Zn^{2+} ion in semiconducting phosphate glasses by EXAFS spectroscopy. They found that the Zn-O bond distance is intermediate between tetra- and hexa-coordination in a zinc metaphosphate glass. They also observed that Zn coordination changes when the glass composition deviates from the metaphosphate composition. Le Saout et al. characterized the structure of $x\text{PbO}-(0,6-x)\text{ZnO}-0,4\text{P}_2\text{O}_5$ system using ³¹P NMR, Raman, and infrared spectroscopies. The investigations revealed no significant change in the average chain length composed of PO_4 tetrahedral units with the substitution of Zn for Pb cation [21]. Hence, it is of interest to investigate the FT-IR spectroscopy and properties of the simultaneous admixture of PbO and ZnO into the phosphate compositions since there is a few literatures reported on this glass system. The purpose of the present work was to investigate the role undertaken by zinc ions on the physical properties and structure of nominal composition $(35-y)\text{Li}_2\text{O}-y\text{ZnO}-15\text{PbO}-50\text{P}_2\text{O}_5$ ($0 \leq y \leq 35$ mol%), called LiZnPbP glasses in subsequent text.

Experimental

Preparation of glasses

Glasses with composition $(35-y)\text{Li}_2\text{O}-y\text{ZnO}-15\text{PbO}-50\text{P}_2\text{O}_5$ ($y=0; 5; 15; 25; \text{ and } 35$ mol%) were prepared by mixing the appropriate amounts of lithium carbonate (Li_2CO_3), zinc oxide (ZnO), lead oxide (PbO), and diammonium hydrogen phosphate ($(\text{NH}_4)_2\text{HPO}_4$) according to the reaction scheme (1):



Mixtures of the starting materials were firstly ground in an agate mortar and preheated in alumina crucible at 120°C overnight. They were then annealed at the temperatures varying between 300 and 400°C for 12 h to remove NH_3 , H_2O , and CO_2 . The temperature was then progressively increased to 1050°C and held constant at this value for 15 min. The batch was finally quenched to room temperature under air atmosphere to produce vitreous samples.

X-ray diffraction studies

X-ray diffractograms of the powdered samples were recorded at room temperature using a Siemens D5000 diffractometer with CuK radiation ($\lambda = 1,5418 \text{ \AA}$) in the 2θ ranges of 10° - 60° at a scanning rate of 2° per minute. The XRD analysis was used to confirm the amorphous nature of the glasses.

Thermal studies

Differential scanning calorimetry of powder samples was made at a heating rate of $10 \text{ K}\cdot\text{min}^{-1}$ using the DSC-SETRAM type apparatus 121 to determine the glass transition temperature (T_g); the estimated error is $\pm 3^\circ\text{C}$.

Density and molar volume measurements

Density measurements were carried out at room temperature, using Archimedes method with distilled water as the immersion medium. The relative error in these measurements was about $\pm 0.03 \text{ g}\cdot\text{cm}^{-3}$. The molar volume (V_m) was calculated from the experimentally determined density (ρ) according to the relation: $V_m = M/\rho$, where M is the molar weight of the glass.

Infrared spectroscopy study

The Fourier transform infrared (FTIR) spectroscopy measurements were made on glass powders dispersed in KBr pellets ($3 \text{ wt.}\%$). The infrared spectra of the powder glass samples were recorded at room temperature in the range between 400 - 1400 cm^{-1} using a FT-IR Perkin-Elmer spectrometer.

Results and Discussion

The obtained results concerning nominal compositions, density, molar volume and glass transition temperature for all studied samples are listed in **Table 1**.

Table 1 Composition, density ρ , molar volume V_m and glass transition temperature T_g of $(35-y)\text{Li}_2\text{O}-y\text{ZnO}-15\text{PbO}-50\text{P}_2\text{O}_5$ ($0 \leq y \leq 35$) glasses

Composition (mol%)				ρ ($\text{g}\cdot\text{cm}^{-3}$)	V_m (cm^3/mol)	T_g ($^\circ\text{C}$)
Li ₂ O	ZnO	PbO	P ₂ O ₅			
35	0	15	50	2.89	39.76	330
30	5	15	50	3.07	38.27	332
20	15	15	50	3.35	36.60	334
10	25	15	50	3.65	35.01	337
0	35	15	50	3.93	33.82	367

X-ray diffraction

Powder X-ray diffraction patterns demonstrates typical amorphous scattering characteristic for all the samples prepared in this work (**Figure 1**).

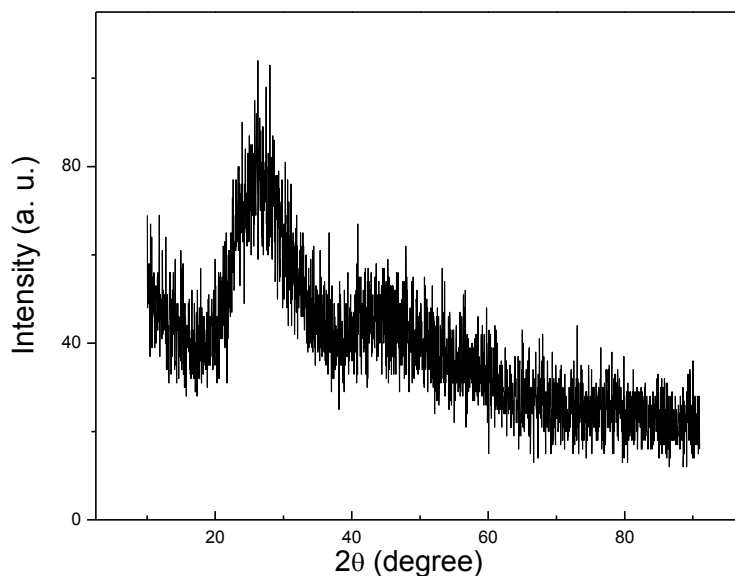


Figure 1 X-ray diffraction patterns for typical sample 20Li₂O–15ZnO–15PbO–50P₂O₅

Density and molar volume

Table 1 presents the value of density (ρ), molar volume (V_m), of each sample of LiZnPbP glasses. From the **Figure 2**, the density of the studied glasses increases with increasing content of zinc oxide (ZnO) from 2.89 g/cm³ for y=0 to 3.93 g/cm³ for y=35 mol% ZnO. The observed increase is almost linear. This variation can be explained by the fact that the low-density lithium element (0.543 g/cm³) is replaced by the high-density zinc element (7.133 g/cm³).

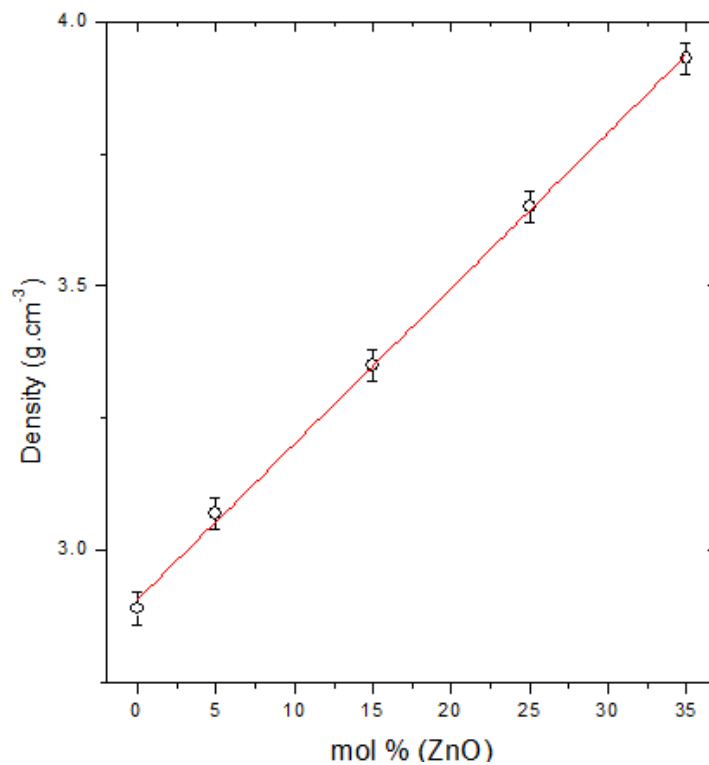


Figure 2 Composition dependence of density

The molar volume of LiZnPbP glasses, V_m , was calculated from the molecular weight, M and density ρ using the equation (2):

$$V_m = M_{\text{glass}}/\rho_{\text{glass}} \quad (2)$$

The molecular weight, M_{Glass} was calculated using the relation (3):

$$M_{\text{Glass}} = (35-y) \times M(\text{Li}_2\text{O}) + y \times M(\text{ZnO}) + 15 \times M(\text{PbO}) + 50 \times M(\text{P}_2\text{O}_5) \quad (3)$$

where $(35-z)$, y , 15 and 50 are the mole percent of Li_2O , ZnO , PbO and P_2O_5 respectively: $M(\text{Li}_2\text{O})$, $M(\text{ZnO})$, $M(\text{PbO})$ and $M(\text{P}_2\text{O}_5)$ are their respective molecular weights.

As can be seen from the Table 1 and **Figure 3**, the molar volume of LiZnPbP glasses, V_m , decreases with increasing ZnO content within the concentration region of $y = 0-35$ mol% ZnO. Obviously, an opposite trend for the density and molar volume of the investigated samples were observed in this work, which was a good agreement with previously reported results [22, 23]. A decrease in molar volume of the LiZnPbP glasses from $39.76 \text{ cm}^3/\text{mol}$ (for $y=0$) to $33.82 \text{ cm}^3/\text{mol}$ (for $y=35$) was observed due to the substitution of two lithium by zinc. Since the radius of Zn^{2+} (0.074 nm) was lower than of the sum of two Li^+ ion radii ($2 \times 0.068 \text{ nm}$), the addition of ZnO into the glass system has caused a decrease in the bond length or inter-atomic spacing between the atoms (since Li^+-O^- are ionic bonds while $\text{Zn}-\text{O}$ are covalent bonds), whereby the glass network will expand and form less pores. Therefore, the compactness of the glass will increase and reduce non-bridging oxygen NBO's, which increased the rigidity and reticulation of the glasses [24].

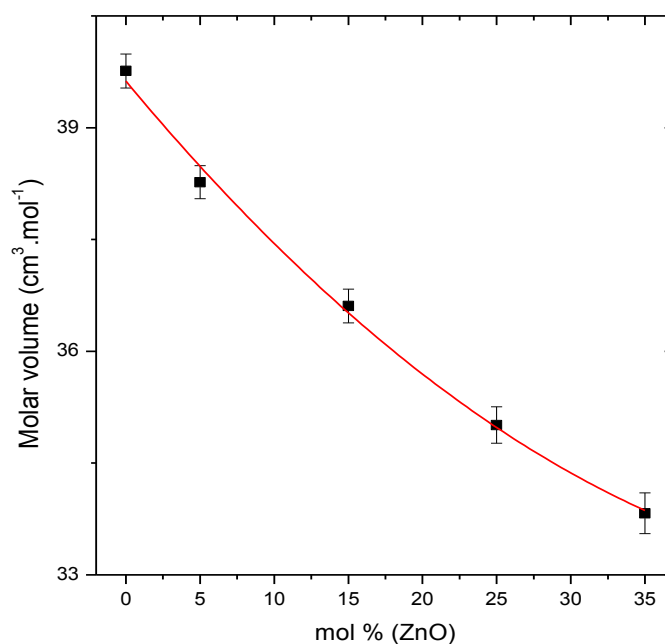


Figure 3 Composition dependence of molar volume

DSC study

The values of glass transition temperature T_g of the LiZnPbP phosphate glasses are given in Table 1.

The compositional dependences of the glass transition temperature (Figure 3) show an unmonotonous behaviour of T_g , which is a slight increase within the content region of 0–25 mol% ZnO ($\Delta T=7^\circ\text{C}$), from 300°C for $35\text{Li}_2\text{O}-15\text{PbO}-50\text{P}_2\text{O}_5$ glass ($y=0$) to 337°C for the glass of composition $10\text{Li}_2\text{O}-25\text{ZnO}-15\text{PbO}-50\text{P}_2\text{O}_5$ ($y=25$) (Table 1), while we can see a significant increase of T_g in the concentration region of 25–35 mol% ZnO ($\Delta T=30^\circ\text{C}$), from 337°C for $10\text{Li}_2\text{O}-25\text{ZnO}-15\text{PbO}-50\text{P}_2\text{O}_5$ glass ($y=25$) to 367°C for the glass of composition $35\text{ZnO}-15\text{PbO}-50\text{P}_2\text{O}_5$ ($y=35$) (Table 1).

We suppose that in these glasses, when zinc (Zn^{2+}) and lead (Pb^{2+}) cations substitute for lithium (2Li^+) ions (in $50\text{Li}_2\text{O}-50\text{P}_2\text{O}_5$ glass for Pb^{2+} , and in $35\text{Li}_2\text{O}-15\text{PbO}-50\text{P}_2\text{O}_5$ glass for Zn^{2+}), $-\text{P}-\text{O}^{\delta-} \dots \text{Zn}^{\delta+}$ and $-\text{P}-\text{O}^{\delta-} \dots \text{Pb}^{\delta+}$ bonds are formed with a strong covalent Zn–O and Pb–O bonds than Li–O (because the Zn and Pb electronegativities being

larger than that of Li). This behavior means that the transition temperature increases in ZnO-rich phosphate glasses. We assume that in this case lead and zinc can be incorporated into the glass network resulting in the formation of P–O–Pb and P–O–Zn linkages respectively.

FT-IR spectra analysis

The results of FTIR spectra of LiZnPbP metaphosphate glasses with various contents of zinc oxide (ZnO) region of 0–35 mol%, in the frequency between 400 and 1400 cm^{-1} , are shown in **Figure 4**. The IR bands assignments are given in **Table 2**.

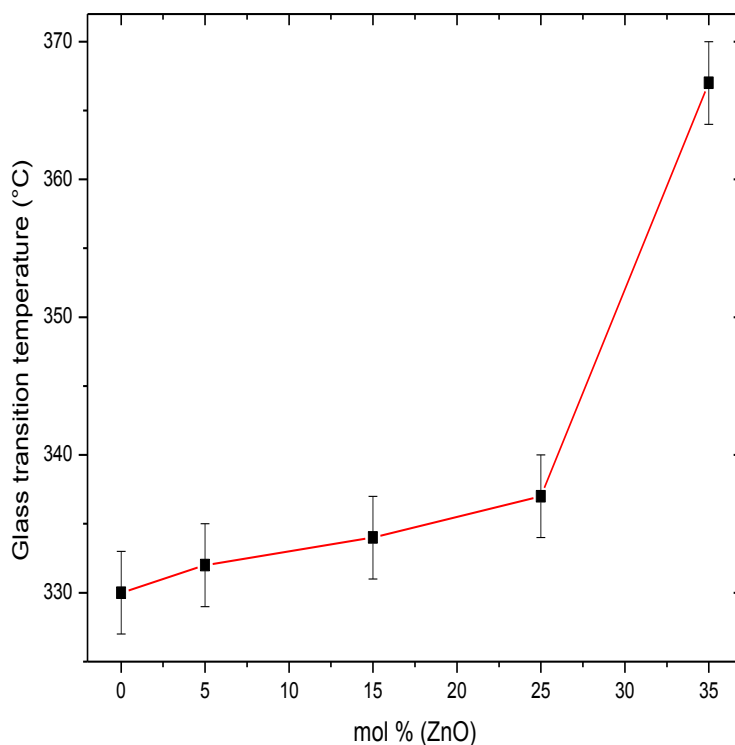


Figure 4 Composition dependence of glass transition temperature for $(35-y)\text{Li}_2\text{O}-y\text{ZnO}-15\text{PbO}-50\text{P}_2\text{O}_5$ glasses

Table 2 Various bands positions (cm^{-1}) in the IR spectra of $(35-y)\text{Li}_2\text{O}-y\text{ZnO}-15\text{PbO}-50\text{P}_2\text{O}_5$ glasses

Glass composition	PO_2 asymmetrical stretching	P-O^- stretching	P-O-P asymmetrical stretching	P-O-P symmetrical stretching	δPO_4^{3-}	Pb-O and Zn-O
y=0	1247	1089	903	788/720	546	472
y=5	1244	1085	902	785/716	546	468
y=15	1241	1065	900	783/717	543	468
y=25	1240	1065	898	786/712	544	467
y=35	1239	1077	900	790/716	540	465

The FT-IR spectra of LiZnPbP glass ($0 \leq y \leq 35$), exhibited several band, which can be divided as medium and broad. Similar to other phosphate glasses, the present samples also exhibited the main characteristic active vibrational modes of phosphate network in the range 400–1400 cm^{-1} . The band at 1247–1239 cm^{-1} attributed to the asymmetric stretching vibration band of the non-bridging oxygen atoms (NBO) bonded to phosphorus atoms PO_2 (Q^2 structural units) [21, 25, 26]. The absorption band observed around 1089–1077 cm^{-1} is assigned to the asymmetric stretching of P-O^- groups characteristic of Q^1 structural units (chain-end groups) [21, 25, 26]. The fundamental band at 901 cm^{-1} is attributed to asymmetric stretching of P-O-P bridges [21, 25–27], whereas the absorption bands at 776 and 714 cm^{-1} are due to symmetric stretching of P-O-P bridges [21, 22]. However, the glass is found to exhibit two bands in the frequency range 776–714 cm^{-1} which are ascribed to the presence of two P-O-P units in metaphosphate chain based on $(\text{P}_2\text{O}_6^{2-})$ cyclic groups as shown in **Figure 5** [28, 29]. The band around 534 cm^{-1} is due to deformation modes of P-O bonds (νPO_4^{3-}) [21, 27].

The characteristic bands of PbO and ZnO appear at low frequencies at about 470 cm^{-1} . It suggests that the bands characteristics of Zn–O (or Pb–O) vibrations remain in the region below 600 cm^{-1} . In zinc silicate glasses, the Zn–O tetrahedral bond is in the range of $600\text{--}400\text{ cm}^{-1}$, while the Zn–O octahedral bonds lies at $300\text{--}100\text{ cm}^{-1}$ [30]. From FTIR results it could be noted that, the intensities of different vibration bands of phosphate structures are not affected by increase in the content of ZnO, excepted the band at 470 cm^{-1} . We presumed the band at about 470 cm^{-1} is assigned to the vibration of Zn–O in ZnO_4 and Pb–O in PbO_4 groups [31–33].

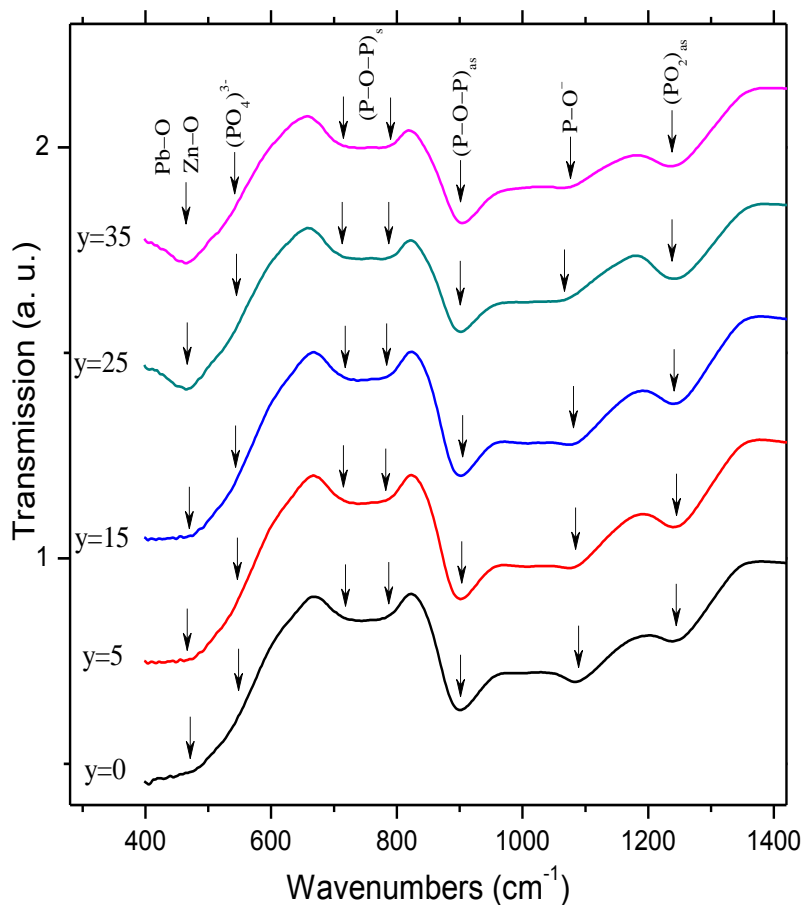


Figure 4 Infrared spectra of LiZnPbP glasses

The IR spectra of all the glasses of compositions LiZnPbP are identical to the spectrum of lithium–metaphosphate glass [35]. Therefore, the metaphosphate groups are predominant structural units in all these glasses. Thus, we observed that the structure of the phosphate chains is not affected by the substitution of Zn^{2+} for Li^+ in the glass, indicating that the fraction of bridging oxygens (P–O–P) is unaffected by glass compositions.

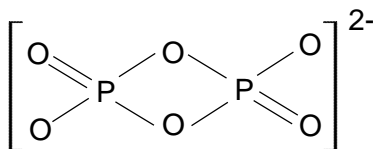


Figure 5 Metaphosphate $(\text{P}_2\text{O}_6)^{2-}$ contains two P–O–P units

The **Figure 6** show that the band frequencies of PO_2 asymmetric stretching mode shifts to lower frequencies as ZnO content increases from 1257 cm^{-1} for ($y=0$) to 1239 cm^{-1} for ($y=35$). This shift can be related to the larger field strength $F_{\text{M-O}}$ of Zn^{2+} ($=43\text{ nm}^{-2}$) $>$ Li^+ ($=23\text{ nm}^{-2}$), and to the metal atomic weight being larger in the order $M(\text{Zn})=65,4\text{ u} > M(\text{Li})=6,941\text{ u}$, that affects the metal to non-bridging oxygen bond along phosphate chains. The same behaviour was observed by Rouse et al [36] in alkali metal metaphosphate glasses, which proposed that the frequency of symmetric and asymmetric of PO_2 stretch will vary systematically with substituted cation (cation mass, M_c), force constant in the alkali–nonbridging oxygen bond ($F_{\text{m-o}}$) and O–P–O bond angle ($\nu(\text{PO}_2)$).

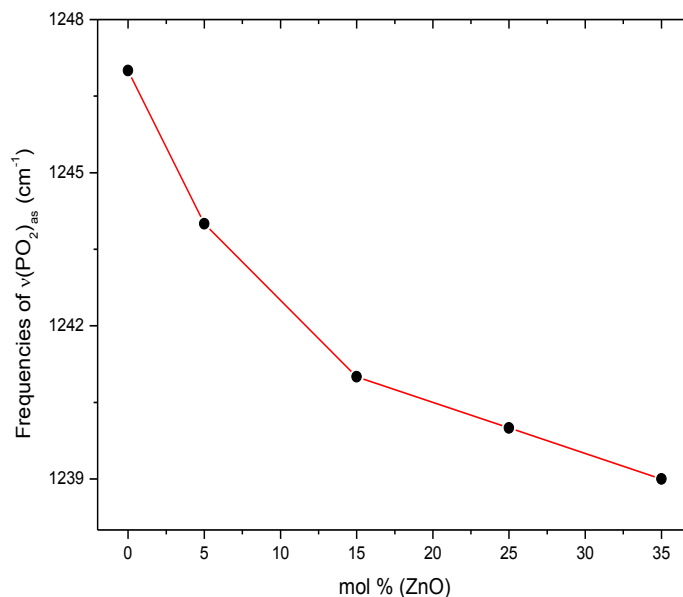
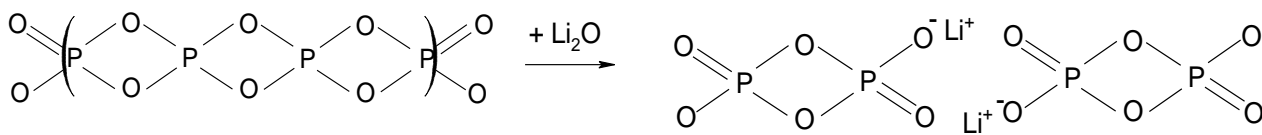
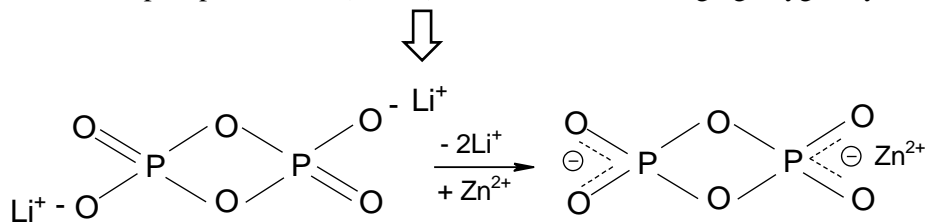


Figure 6 Frequencies variations of $\nu(\text{PO}_2)_{\text{as}}$ modes vs ZnO content in LiZnPbP glasses

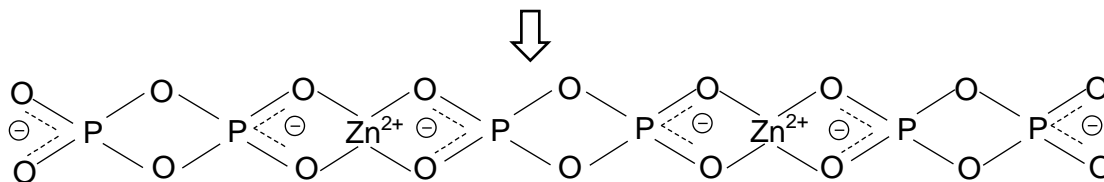
The low frequency band near 530 cm^{-1} (shoulder), which is attributed to deformation modes of P-O^- bonds (νPO_4^{3-}), is also present in all these glasses. Moreover, a narrow low frequency band appears at about 470 cm^{-1} probably due to Zn-O stretching in ZnO_4 units and Pb-O stretching in PbO_4 can clearly be observed in the spectra of the glasses composition $y \geq 25$. Thus, these results indicate that ZnO is assumed to play an intermediate role. Whereby the Zn^{2+} would break P-O-P bonds forming ZnO_4 tetrahedral units connect to the phosphate tetrahedra PO_4 by P-O-Zn covalent bonding. The process of incorporation of ZnO into the glassy network may therefore be represented as:



Linear metaphosphate chain $(\text{P}_2\text{O}_6^{2-})_{\infty}$ Creation of non-bridging oxygen by Li_2O



Reorganization of P-O bonds to include two P-O-P links in $\text{P}_2\text{O}_6^{2-}$ units with Zn^{2+} four-coordination



Linear metaphosphate chain $[\text{Zn}^{2+}(\text{P}_2\text{O}_6^{2-})]_{\infty}$ units

Conclusion

$(35-y)\text{Li}_2\text{O}-y\text{ZnO}-15\text{PbO}-50\text{P}_2\text{O}_5$ ($0 \leq y \leq 35$ mol%) metaphosphate glasses have been successfully prepared and characterized. The densities increased when ZnO content was added to the phosphate glasses, while the molar volume was decreased. The infrared spectra show that the structure of the phosphate chains is not affected by the substitution

of Zn^{2+} for Li^+ in the glass metaphosphate chain which are based on $(\text{P}_2\text{O}_6^{2-})$ groups. Whereby the Zn^{2+} would break P–O–P bonds forming ZnO_4 tetrahedral units connect to the phosphate tetrahedra PO_4 by P–O–Zn covalent bonding.

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