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RESEARCH ARTICLE

DC Conductivity of Lithium-Zinc-Boro-Phosphate Glasses

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Abstract

Objectives: Investigation of electrical transport properties of glass system in the composition $(P_2O_5)_{0.65-x}(B_2O_3)_{0.1}$ $(ZnO)_{0.25}$ $(Li_2O)_x$ where x=0.10, 0.15, 0.20, 0.25, 0.30, 0.35 and 0.40 and establish the conduction mechanisms. **Methods**: Glasses were synthesized using the standard melt quenching technique at 1473K. The samples were confirmed to be amorphous through XRD studies. An independent measurement of dc conductivity was carried out for temperature range 300-620K, by following two probe method. **Findings**: Conductivity of the present glasses is found to be of the order of 10^{-3} $(\Omega\ m)^{-1}$ which is higher than the reported values for lithium-ion solid-state electrolytes. Conductivity variation with temperature obeyed Mott's Small Polaron Hopping at high temperatures and Variable Range Hopping at low temperatures. SPH model fits to the conductivity gave activation energies in the range 58-409 meV. VRH model fits gave density of states at Fermi level of the order of 10^{23} $eV^{-1}m^{-3}$. The nano-crystallite phases identified and determined size found to be in the range 5-37 nm and considered to be a glass-nanocomposites. **Novelty/Applications:** For the first time, zinc-lithium-borophosphate glasses are investigated for conduction mechanisms. Activation energy and density of states at the Fermi level are determined. At high temperature Mott's SPH model and at low temperature Mott's VRH model are found to be suitable to explain conductivity of these glasses.

Keywords: Boro Phosphate glasses; Small Polaron Hopping; activation energy; Variable Range Hopping; the density of states; conduction mechanism

1 Introduction

The phosphate network gets de-polymerized due to the addition of Li_2O and consequently glassy network re-polymerized due to the cross-linking of alkali ion polyhedral connecting neighboring phosphate groups [(1)].

Zinc borophosphate glasses showed high physical and chemical durability when the two-dimensional phosphate networks were converted into highly cross-linked structures by the addition of B_2O_3 [(2)]. The glass system, $0.45Li_2O$ - (0.55-x) P_2O_5 - xB_2O_3 with mixed glass formers, exhibited higher ionic conductivity than pure borate

and phosphate systems[3].

Zinc borophosphate glasses were investigated for thermal, chemical, and structural properties and results showed that they have good thermal stability and chemical durability. Zinc Oxide belongs to intermediate oxides, and therefore it enters the structural network and forms covalent Zn-O-P bonds[4,5].

Analysis of dc conductivity of ZnO-B_2O_5-P_2O_5 revealed that electrical conductivity is affected by structural change. The electronic conduction was reported to be dominant in the higher concentration range[6].

Centikaya Colak et al. (2016) have investigated Zinc-borate glasses and reported that ZnO plays a dual role as a modifier and a network former. If ZnO behaves as a modifier, the number of NBOs will increase, and this will cause the expansion of the glass network[7].

For the $0.45Li_2O$ - $0.05ZnO$ - $0.20B_2O_3$ - $0.30P_2O_5$ glass composition, the dynamics of lithium ions in glass has been investigated by S Kabi[8] using the proposed model of time and temperature dependence of the mean square displacement of ion and as highlighted that dc activation energy plays an important role to govern the ion conduction process.

Dielectric properties, AC conductivity and activation energies were investigated for Vanado- Zinc- Boro-Phosphate glasses at different frequencies[9].

The previous researchers Koudelka et al.[5], investigated the lithium zinc borophosphate glasses by varying lithium at the expense of ZnO up to 50 mole % Li_2O with the objective of structural analysis. Electrical properties have not been investigated. Since zinc-lithium-borophosphate glasses have not been studied for conduction mechanisms so for by any research group. It was therefore planned to prepare zinc borophosphate glasses with varied content of Li_2O at the cost of P_2O_5 and study the transport properties by measuring dc conductivity independently by two probe method. The results obtained are presented in this paper.

2 Materials and Methods

The composition of glasses is defined as $(P_2O_5)_{0.65-x}$ $(B_2O_3)_{0.1}$ $(ZnO)_{0.25}$ $(Li_2O)_x$ where x=0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40 was prepared by standard melt-quenching technique and labeled as BPZL1, BPZL2, BPZL3, BPZL4, BPZL5, BPZL6, BPZL7 respectively.

The AR grade chemicals, ammonium dehydrogenate phosphate ($NH_4H_2PO_4$), Boric Acid (H_3BO_3), Lithium Oxide (Li_2CO_3), and Zinc Oxide (ZnO) were used as starting materials. The appropriate molar ratio of chemicals was weighed in precision weighing balance sensitive to accuracy of 0.1 mg and mixture was grounded into fine powder in an agate mortar, taken in silica crucible and melted in a muffle furnace at 1473K.

The melt was quenched to room temperature between stainless steel plates. To relieve the mechanical stresses, the samples were annealed at 450K.

The details of the preparation of glasses have been described in references[10].

Powder XRD studies were carried out for finding structural phase confirmation. This experiment was carried out in a Rigaku make diffractometer with Cu-Kα radiation in the Bragg's angle range 10°-80°.

The samples of 3.5 mm thickness and cross-sectional area in the range 65 mm^2 to 100 mm^2 size were shaped, silver coated on both major surfaces to form electrode contact, and measured conductivity using two probe methods by using Chromel (Cr)-Alumel(Al) type- K thermocouple for temperature measurement in the range 300-620K and applying a constant voltage across the sample and measuring the current through the sample. Conductivity was determined as $\sigma = 1/\rho$, $\rho = RA/t$ with R being resistance, A cross-sectional area, and t the thickness of the glass.

3 Results and Discussion

3.1 XRD

The glass samples BPZL1, BPZL2 & BPZL3 shows no sharp peaks but a broad hump appears between 2θ angle from 20° to 30°.Samples BPZL4 & BPZL7 shows no bumps and peaks. XRD pattern showing no sharp peaks and bumps like peak are the indication of amorphous nature. Samples BPZL5, BPZL6 and BPZL8 shows few sharp peaks. So, they may be treated as semi-crystalline in nature. The semi-crystallinity can be due to any or all the constituent oxides. All samples show small peak centered at 43.33° is observed in all samples and crystallite size is found to be 27.47 nm determined by Debye-Scherrer's formulae. In sample BPZL5 and BPZL7 peaks centered at 2θ values 28.26° and 28.41° and determined crystallite size of 37.17 nm and 35.31 nm respectively. Sample BPPZ4, BPZL5, BPZL6 and BPZL7 possess average crystallite size of the order of 76.77 nm, 93.34 nm, 25.02 nm and 21.94 respectively and dominant peak centered at 2θ angle 28.10° for samples BPZL4, BPZL5 and BPZL6 shows average crystallite size of 21.98 nm. Sample BPZL8 shows several sharp peaks considered to be crystalline in nature. These glasses containing nano-crystallites can be considered as glass- nanocomposites.

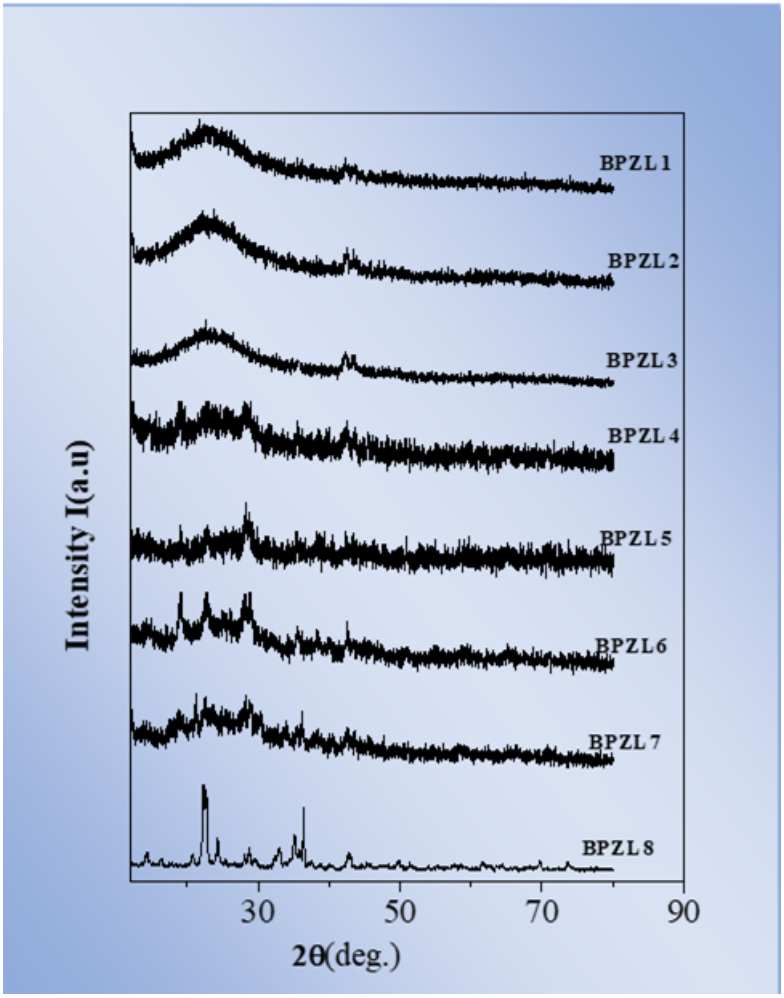

Fig 1. XRD patterns of BPZL glasses

3.1 Conductivity

The conductivity measured in the temperature range from 320 K to 610K varied in the range from $0.5x10^{-4}(\Omega m)^{-1}$ to $7.64x10^{-3}(\Omega m)^{-1}$as shown in Figure 2 (a) for BPZL2 glass. The samples BPZL1, BPZL3, BPZL4, BPZL5 and BPZL6 showed increase in dc conductivity as temperature increased and termed it as semiconducting behavior of sample. The plot of conductivity, Log(σ_{dc}) at different temperatures 473K,503K, 553K, and 608 K versus Li_2O mole fractions, are shown in Figure 2(b) are exhibiting the temperature and composition dependent conductivity.

Figure 2(b) suggests that conductivity is dependent on Li_2O content. The conductivity drastically increased at Li_2O mole fraction 0.25. Conductivity decreased at 0.35mole fractions of Li_2O.

As per the glass compositional formulae at a 0.25-mole fraction, the glass forming and modifiers are in the ratio of P_2O_5 (0.40), B_2O_3(0.1) and ZnO (0.25) and Li_2O (0.25) mole fractions, ZnO acts as a modifier and former role ascertained on the basis of conductivity and activation energy variations with composition and its effect on structural reorganization of the glass system.

At 0.35 mole fraction of Li_2O, ZnO acts as an intermediate network former along with other two glass former P_2O_5 and B_2O_3 resulting more BO's and is predicted to cause a decrease in the conductivity. This trend clearly reflects that ZnO's role in structural reorganization is due to its properties as an intermediate network former. From 0.25 to 0.35-mole fraction of Li_2O, the conductivity and activation energy behaves perfectly monotonically at temperature 553K as compared to variation at 503K. The electrical properties show that maximum appears at the mole% ratio of Li_2O/ZnO equal to unity and at the mole% ratio of $(P_2O_5+B_2O_3)/(Li_2O+ZnO)$ equal to unity.

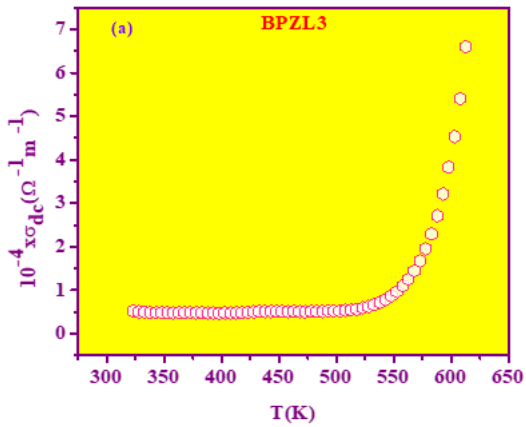

Figure 2(a) Plot of σ_{dc} versus T(K) at 608K

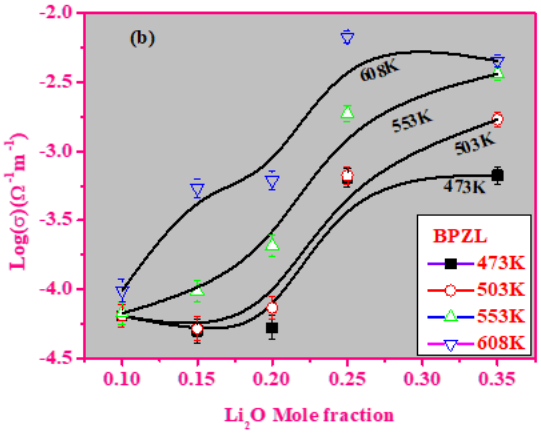

Fig 2. (a) Plot of σdc versus T(K) at 608K (b) Plots of Log(σ_{dc}) versus Li_2O fraction at different temperatures

For the Non-Crystalline or amorphous solids or glass systems which are amorphous in nature they can exhibit temperature dependent conductivity and show thermally activated process of conductivity. To investigate the conduction mechanism in the present samples, we employed Mott's Small Polaron Hopping (SPH) model and variable range hopping (VRH) model[10–14].

Present sample was investigated in the temperature range 300K to 620K, the temperature where the data deviated from Mott's SPH model is termed as lower temperature region in comparison with higher temperature region of our investigating temperature range.

The conductivity in the non-adiabatic regime as per the SPH model is given as

$$\sigma = (\sigma 0/T)\ exp\ (-W/kBT) \tag{1}$$

Where σ_0 is the pre-exponential factor and W is the activation energy.

The data is best linear fits to Mott's (SPH) model expression as per equation (1), and the plots drawn are shown in Figure 3.

It is observed that the activation energy for Mott's (SPH) model lies in the range of 58 – 409 meV[14,15].

The SPH model showed activation energy in the high-temperature regime, which is found to decrease with Li_2O gradually. Activation energy values suggest that it suits the high-temperature region with improved lower activation energy values.

The data that deviated from Mott's (SPH) fit has been considered for analysis using Mott's variable range hopping (VRH) model. In 1968, Mott put forward the idea that $T^{-1/4}$ hopping was due to variable range. The low-temperature mode of transport occurs in the vicinity of the Fermi energy. At very low temperatures, Mott has proposed that carriers having sufficient energy to hop to nearest neighbors will hop further to find sites of comparable energy. As per the model, the conductivity is given by,

$$\sigma = A\ exp(-B/T1/4) or\ ln(\sigma) = lnA - (B/T1/4) \tag{2}$$

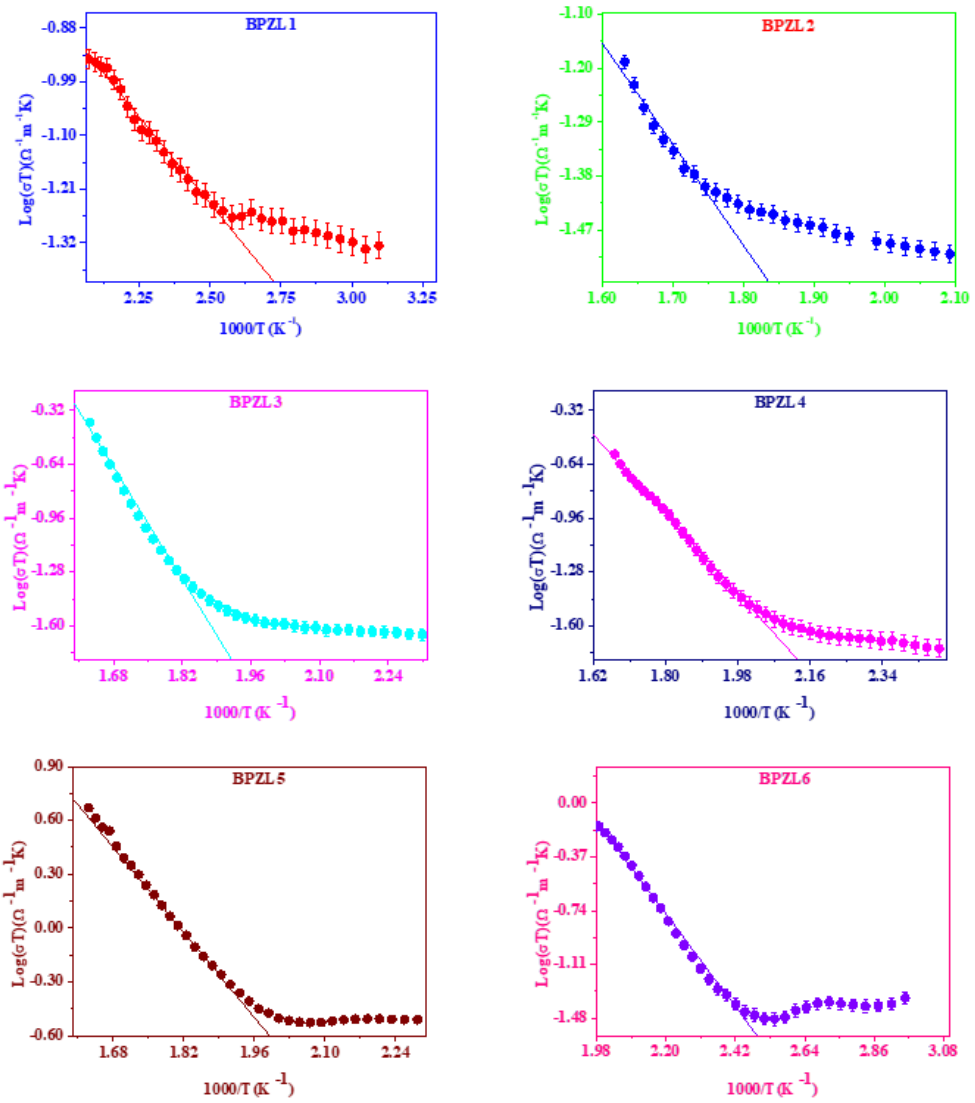

Fig 3. Plots of Log(σT) versus (1000/T). Lines are the fits to Mott's SPH model

where $A=[e^2/2(8\pi)^{1/2}]\ {}_0[N(E_F)/\alpha\ k_BT]^{1/2}$, $B=4[2\alpha^3/9\pi k_B N(E_F)]^{1/4}$

$N(E_F)$ is the density of states at the Fermi level.

In the low-temperature regime, the plots of Log(σ) versus $T^{-1/4}$ fits to straight lines.

As per the Eqn. (2) the VRH model plots have been drawn and best linear fits within the limit of ± (0.2-0.5%) of error to slopes are shown in Figure 5.

From the slopes of VRH plots, the density of states at Fermi level $N(E_F)$ has been determined and tabulated in Table 1. Obtained $N(E_F)$ values suggests that the density of states is varying with Li_2O content. Similar results were obtained in ref[12].

Chatterjee and Ghosh (2018) have studied ion transport of xLi_2O - (1-x) P_2O_5 glasses, where $0.30 \leq x \leq 0.55$, on increasing Li_2O content, the dc conductivity increases, and the activation decreases along with the increase of the concentration of NBOs in the glassy network. Present results agree with these[1]. By using IR and Raman spectroscopy and solid-state NMR spectroscopy data, Young-Seok Kim et al. (2014) investigated $xZnO\text{-}10B_2O_3\text{-}(100\text{-}x)\ P_2O_5$, where x from 30 to 60 mol% glasses that density and covalency of the bridging oxygen increase when the ZnO content was increased. NMR spectra inferred that excess Zn ions act as network formers, contributing to the formation of the structure and increasing the bond strength. The mole fraction 0.25 of ZnO is kept constant and is found to enhance the chemical, thermal and structural stability in the BPZL glasses[2]. T.D Tho et al. (2012) for lithium borophosphate glasses reported that activation energy values 0.600 - 0.655 (eV) agreed with, and

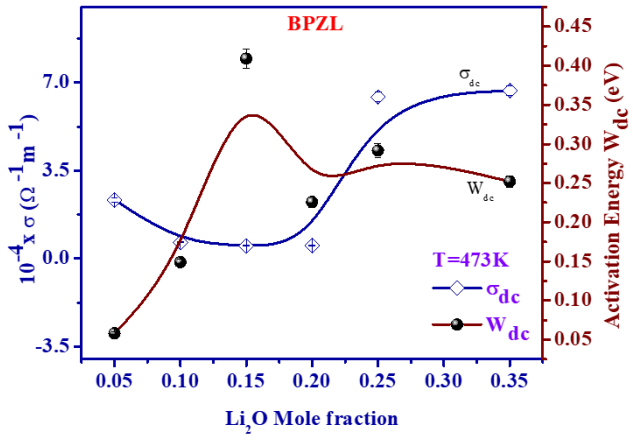

Fig 4. Plot of conductivity at 473K and activation energy versus Li20

Table 1. Density of states N(E_F) at Fermi level

Glass	Li_2O (Mole fraction)	Density of states (10^{23}) x N(E_F) ($eV^{-1}m^{-3}$)
BPZL1	0.05	128.86
BPZL2	0.10	32.27
BPZL3	0.15	4.45
BPZL4	0.20	9.54
BPZL5	0.25	5.10
BPZL7	0.35	8.65

conductivity values lie in the range $3.16x10^{-10}$ - $3.16x10^{-8}$ $(\Omega\ m)^{-1}$are found lower than BPZL glasses[3]. S. Kabi (2019) has been reported for $0.45Li_2O$ - $0.05ZnO$ - $0.20B_2O_3$ - $0.30P_2O_5$ glass at temperature range 293K - 413K that dc conductivity lies in the range 1.77x10-7 (Ω m)-1 - 5.623x10-5 (Ω m)-1 and activation energy Edc was 0.68 (± 0.01) eV is found to be comparable with our glasses[8].

J.S. Ashwajeet et al. (2015) reported conductivity values in the range1.088 x10-3-3.378x10-3 and activation energy in the range of 0.502 eV - 0.741 eV for borophosphate glasses doped with CoO and Li_2O. The conductivity values agreed with present glass at 0.25,0.35 mole fraction of Li_2O doped glasses, whereas activation energy values are well agreed with obtained values for all glass samples[12]. Thipperudra et al. (2021) have been investigated the Borophosphate glasses doped with SrO and Li_2O reported the dc conductivity in the range $4.24x10^{-8}$ $(\Omega\ m)^{-1}$ - $6.92x10^{-5}$ $(\Omega\ m)^{-1}$ is comparable to our glasses at lower concentrations of Li_2O[13].

The conductivity for semiconducting cobalt-phosphate glasses has been measured at temperature range 213-530 K conductivity values reported were lower compared to our room temperature conductivity at 303K. From Mott's VRH conduction determined density of states N(E_F) at Fermi level is comparable to our results[14]. M. A. Salorkar et al. (2021) have been reported $1.23x10^{-5}$ $(\Omega\ m)^{-1}$ at x=0 mol% of Li_2SO_4 composition, is agreed with our glasses[15].

V. K. Deshpande et al. (2020)have been reported conductivity was in the range 10^{-2} $(\Omega\ m)^{-1}$ - 10^{-1} $(\Omega\ m)^{-1}$ is comparable to present glasses at a higher temperature, concentrations of Li_2O[16]. For Borophosphate glasses as highlighted by Abd El Keriem (2015) confirmed variation in relative NBO density with an increase in Li_2O content at the expense of Cao. With the addition of modifier oxide Li_2O, the extra oxygen atoms are introduced to form negative non-bridging oxygen (NBO) sites, whose charge is compensated by the modifier cations' positive charge. For BPZL glasses, NBO density was found to increase as Li_2O increased at the expense of P_2O_5 [17]. Raguenet et al. (2011) were reported conductivity in the range $3.0x\ 10^{-10}$ $(\Omega m)^{-1}$ - $1.3x10^{-7}$ $(\Omega m)^{-1}$and values are close to present values[18].

Money & Hariharan (2008) have reported conductivity range $8.4x10^{-7}$ $(\Omega m)^{-1}$- $2.15x10^{-5}$ $(\Omega m)^{-1}$, activation values lie in the range 0.47 (eV) - 0.77 (eV). These results agree with the experimental results of our BPZL glasses[19]. The present investigated quaternary Li_2O doped glass containing ZnO yielded non-crystallinity up to 35% of Li_2O and produced higher conductivity[19]. Ravi Kumar et al. (2018) have been analyzed the conductivity and reported at 300 K was of the order 10^{-2} $(\Omega\ m)^{-1}$ found to

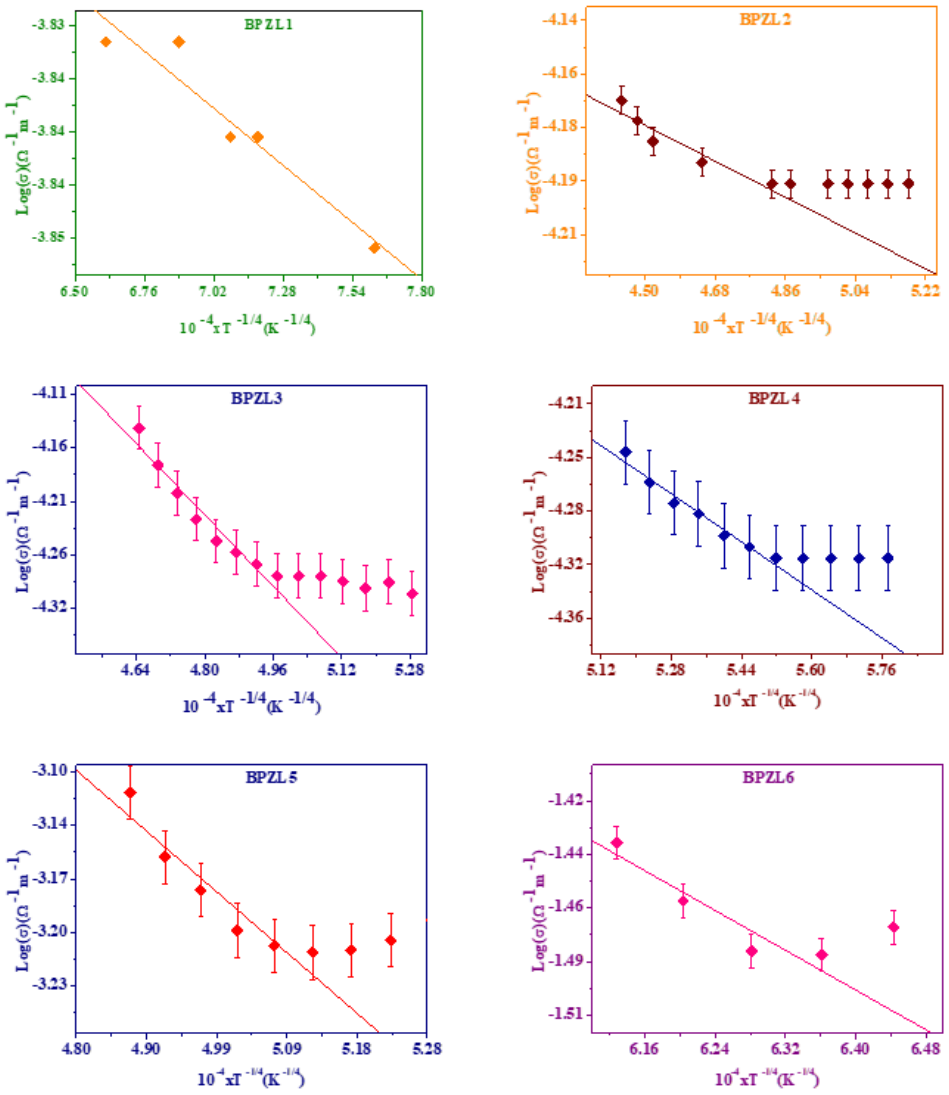

Fig 5. Plots of Log(σ) versus ($1/T^{1/4}$). Lines are fits to Mott's VRH model

be higher than our present glasses [20]. Taha et al. (2020) were reported in the range $4.57x10^{-9}$ $(\Omega\ m)^{-1}$ - $3.11x10^{-7}$ $(\Omega\ m)^{-1}$ and the activation energy was found 0.56 eV at 20 mol% of CuO higher content [21]. These results agree with activation energy, the conductivity of our glasses was found to be higher than these reported glasses [21]. Mohamed M. Ibrahim et al. (2021) have reported in the range $4.9x10^{-4}$ $(\Omega\ m)^{-1}$ - $9.9x10^{-7}$ $(\Omega\ m)^{-1}$; the lower concentration of Pb_3O_4 conductivity values is comparable to our glasses [22]. Hailemariam et al. (2021) have reported in the range $2.2x10^{-4}$ $(\Omega\ m)^{-1}$ - $6.28x10^{-5}$ $(\Omega\ m)^{-1}$ and $4.27x10^{-4}$ $(\Omega\ m)^{-1}$ - $3.37x\ 10^{-5}$ $(\Omega\ m)^{-1}$ for lithium doped and lithium with nickel ferrite doped glasses respectively, these results are comparable to our glasses [23]. Adhwaryu & Kanchan (2021) have reported in the range $2.42x10^{-5}$ $(\Omega\ m)^{-1}$ - $4.12x10^{-3}$ $(\Omega\ m)^{-1}$ comparable to present glasses [24]. Pagoti et al. (2021) investigated (100-x), $35B_2O_3$+ $10P_2O_5$+ $30Li_2O$+15ZnO+10SrO+ xNd_2O_3 where x= 0.0, 0.1, 0.5,1.0,1.5, and 2.0 in mole% glasses and was reported in this article that prepared glass showed superior stability against devitrification, high thermal, stability, and rigidity. Comparable to these reported glasses with specific Li_2O mole fractions in our present glasses showed enhanced conductivity and it attributes for presence of higher structural stability and rigidity in glass [25].

DC conductivity of the $50Li_2O$- $50P_2O_5$ glass with CuO it was reported that an increase in CuO contents and temperature increases the conductivity. For Li_2O contents and temperature increases, similar conductivity behaviour was observed in our glasses up to 25% mole fraction of Li_2O [26].

Shruthi and Madhu (2021)have reported that from plot log(σ_{ac})versus log(ω) showed that no dc plateau observed at low frequency region and conductivity was found to be completely frequency dependent as compared to our present glasses exhibited dc conductivity[27].

The conductivity of present glasses for a 0.25-mole fractions of Li_2O at room temperature is obtained to be $0.6x10^{-3}$ $((\Omega\ m)^{-1}$, and at 608K it is $6.74x10^{-3}$ $(\Omega\ m)^{-1}$. In our previous work dielectric properties and AC conductivity were reported for Vanado - Zinc - Boro - Phosphate glasses. These glasses were of different composition compared to present glasses[9].

For Li_3PO_4 single phase sample was investigated and reported by Norikazu et al. (2021) that particles of size 20-30 nm suitable for the fabrication of dense solid electrolyte in all-solid-state lithium battery applications[28]. Our present glass system BPZL6 and BPZL7 are containing the crystallite size of 21-25 nm is comparable to Li_3PO_4 nano-crystallite phase. All nanostructured materials have their common origin is glassy state and presence of nano crystallites of size range (5-100 nm) confined in a residual glassy matrix[29]. Our present glass system BPZL7 containing dominant peak crystallite phase at 2θ angle centered at 28.22° size of 2.57 nm and sample average crystallite size is 21.94 nm, sample BPZL4, BPZL5 and BPZL6 containing average crystallite size is also comparable to these results.

From Figure 2 (b), conductivity is continuously increasing with Li_2O content. This means that ions also contributing to total conductivity.

4 Conclusions

Glasses of composition $(P_2O_5)_{0.65-x}$ $(B_2O_3)_{0.1}$ $(ZnO)_{0.25}$ $(Li_2O)_x$ where x=0.10, 0.15, 0.20, 0.25, 0.35 and 0.40 were prepared by novel melt quenching method. Their amorphous nature was estimated from XRD studies. The electrical transport mechanism has been understood by analyzing conductivity variation with temperature using Mott's Polaron hopping mechanisms. SPH model fits gave activation energy for conduction, and VRH model fits gave density of states at Fermi level. Activation energy and density of states agree with literature values reported for other similar glasses. Changes in conductivity and activation energy with Li_2O content infer that Li_2O also contributes to total conductivity. Conductivity ranges obtained are better than earlier proposed glasses for battery application. So, present glasses can be considered for battery usage. Present glasses further need to be studied for structural, thermal stability and frequency dependent conductivity.

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