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DIELECTRIC STUDIES IN Li_2O AND CoO DOPED BOROPHOSPHATE GLASSES

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ABSTRACT

Borophosphate glasses in the compositions, $(B_2O_3)_{0.2}$. $(P_2O_5)_{0.3}$. $(Li_2O)_{0.5-X}$. $(CoO)_X$, where x = 0.05, 0.1, 0.15, 0.25, 0.30, 0.35, 0.40 and 0.45 were prepared at 1400K by following melt quenching method. Their amorphous nature was confirmed by XRD studies and was investigated for dielectric properties in the frequency range from 100Hz to 1MHz and temperature range from 300K to 573K. The conductivity was derived from the dielectric spectrum. The frequency exponent, s, dc and ac components of the conductivity were determined. The temperature dependence of conductivity at different frequencies was analyzed using Mott's small polaron hopping model, and the high temperature activation energy has been estimated and discussed. The variation of conductivity and activation energy with composition revealed a changeover of conduction mechanism from predominantly ionic to electronic regime for mole fractions of CoO between 0.3 and 0.35. This is a new result. Hunt's model has been employed to analyze frequency dependence of conductivity. Relaxation features of the dielectric properties have been extracted from the analysis of electric moduli with frequency. Activation energy for relaxation mechanism has been determined. Frequency exponent was found to be temperature dependent. Quantum mechanical theory and correlated barrier hopping models were found to be inadequate to explain frequency exponent behavior with temperature.

For the first time that borophosphate glasses doped with Li_2O and CoO were studied for dielectric properties and ac conductivity as a function of temperature and frequency and the data has been analysed thoroughly.

Keywords: Borophosphate glasses, Dielectric constant and loss, Ac conductivity, Frequency exponent, Master curves, Relaxation process.

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INTRODUCTION

Phosphate, Borate and Tellurite glasses doped with alkali ions have been studied by many researchers due to their novel technological applications such as solid electrolytes for lithium secondary batteries [1, 2]. Electrical studies in oxide glasses containing transition metal (TMI) and alkali ions are reported owing to interest in their conduction mechanisms and their other properties. Such studies in pure borate and phosphate glasses have limitations due to their hygroscopic nature despite the fact they have low melting temperatures and can be easily prepared. However, addition of few boron atoms into phosphate glass network lead to enhanced chemical durability and improved thermal properties [3-6]. Ionic conduction in Ag_2S doped silver phosphate glasses have been studied for frequencies up to 60 GHz and temperature up to 413K. Conductivity obeyed power law type variation with progressive increase with frequency [7, 8]. The glasses in the compositions $50P_2O_5$. $25Li_2O$. $25Na_2O$ have been studied for dielectric constant and that was found to increase with temperature and frequency [9]. Replacement of P-O-P bonds by P-O-B bonds increased glass transition temperature and chemical durability in xB_2O_3 . $(60\text{-}x)P_2O_5$. $40Na_2O$ glasses [10]. The ionic conductivity showed strong and positive mixed glass former effect (MGFE) in $0.35Na_2O$. $0.65[xB_2O_3$. $(1\text{-}x)P_2O_5]$ glasses [11]. Conductivity exhibited two maxima with B_2O_3 content in $50Na_2O$. $50[xB_2O_3 – (1\text{-}x)P_2O_5]$ glasses [12]. Mixed network former effect has been observed in $0.50Ag_2O$. $0.50(xB_2O3$. $(1\text{-}x)P_2O_5)$ glasses and that were interpreted to be due to interaction between the mobile ions and the glass network [13]. The activation energy and power law exponent of conductivity showed composition dependent trends in xLi_2O . $10PbO$. $(90\text{-}x)(55B_2O_3+45P_2O_5)$ glasses[14]. The glasses of composition $50Li_2O$. xB_2O_3. $(50\text{-}x)P_2O_5$ have been measured for conductivity for the temperature range from 273K to 393K [15]. Conductivity increased up to x = 0.2 and decreased for higher values of x. This was attributed to the crystallization of lithium for x > 0.20. Mixed glass former effect has been observed in AgI doped silver borophosphate glasses [16]. Changeover of predominance of conduction mechanism from ionic to electronic has been observed in Dy_2O_3 doped borophosphate glasses $60V_2O_5$. $5P_2O_5$. $(35\text{-}x)B_2O_3$. xDy_2O_3 [17]. Transition of conduction mechanism from electronic to ionic or vice-versa has been observed in different oxide glasses [18-20].The glass transition temperature and chemical durability decrease with increasing potassium content in xK_2O . $(50\text{-}x)ZnO$. $10B_2O_3$. $40P_2O_5$ glasses [21]. The glasses, xLi_2O - $(1\text{-}x)[0.5B_2O_3$ - $0.5P_2O_5]$ and xAg_2O - $(1\text{-}x)[0.5B_2O_3$ - $0.5P_2O_5]$ have been measured conductivity in the temperature range from 298K to 600K and frequency in the range from 10Hz to 13MHz [22].

Present investigations have been motivated by the fact that borophosphate glasses doped with alkali cum transition metal ions have not been investigated for dielectric and ac conductivity by many researchers despite the fact that they are known to be less hygroscopic and more chemically durable than pure phosphate and borated glasses. Further, changeover of conduction mechanisms which lead to battery applications of glasses as electrodes have not been probed in borophosphate glasses so for. That is why, we investigated dielectric properties as a function of temperature in borophosphate glasses of composition, $(B_2O_3)_{0.2}$. $(P_2O_5)_{0.3}$. $(Li_2O)_{0.5\text{-}X}$. $(CoO)_x$, where x=0.05, 0.10, 0.15, 0.25, 0.30, 0.35, 0.40 and 0.45 labeled as BPCL1, BPCL2, BPCL3, BPCL4, BPCL5, BPCL6, BPCL7 and BPCL8 respectively.

EXPERIMENTAL

Glasses were synthesized using AR grade Himedia make chemicals, H_3BO_3, $NH_4H_2PO_4$, CoO and Li_2Co_3, by following melt quenching method. The chemicals in required weight ratios were melted at 1400K in a SiC make electric furnace. After an hour the melt was quenched to room temperature and the randomly sized glasses thus formed were collected. To remove thermal strains in the glasses, they were annealed at 450K and were XRD studied for the confirmation of their non-crystallinity.

The glasses were cut to the required size (3mm x 2mm x 2mm) and coated with silver paint on either surfaces. The measurements of capacitance, C and dissipation factor, tan δ, were carried out in a precision impedance analyzer (Wayne Kerr, 6520B) for the frequency range from 100 Hz to 1 MHz and temperature from 300K to 600K. The dielectric constant (ε′), dielectric loss factor (ε″) and ac conductivity (σ_{ac}) were determined as per the expressions [19].

$$\varepsilon' = \frac{Cd}{\varepsilon_0 A} \quad (1)$$

$$\varepsilon'' = \varepsilon' \tan\delta \quad (2)$$

$$\sigma = \omega \varepsilon_0 \varepsilon'' \quad (3)$$

Where ε_0 is the permittivity of free space, ω the frequency of the input signal, d the thickness and A the cross sectional area of the glass.

RESULTS AND DISCUSSION

Dielectric constant and loss: The measured dielectric constant, ε′, and dielectric loss, ε″, were found to lie in the range of 10^2 to 10^4 and 10^1 to 10^5 respectively. The plots of ε′ and ε″ versus ln(F) for four different temperatures for BPCL3 glass are shown in Figs 1(a) & 1(b) respectively. It may be noted that only to avoid overlapping and to make it easy to read the variation of the parameters, the data for only four different temperatures is displayed in Figs 1(a) & (b). More explicitly the temperature behavior of ε′ and ε″ for BPCL3 glass is shown in Fig.2. Similar nature of variation of ε′ and ε″ with temperature and frequency has been observed for the remaining glasses of the present series.

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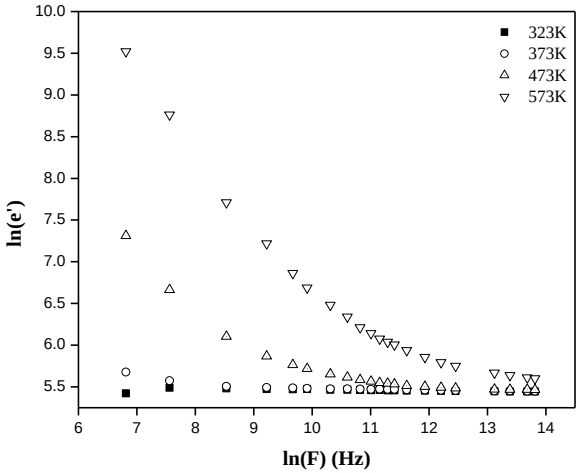

Fig 1 (a): Plots of ln (ε′) versus ln (F) for BPCL3 glass at different temperatures.

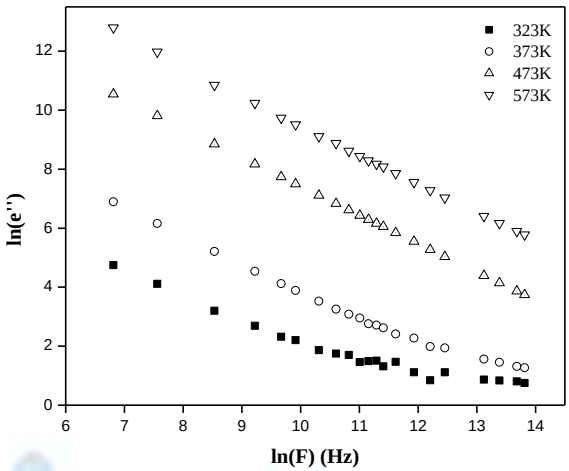

Fig 1 (b): Plots of ln (ε″) versus ln (F) for BPCL3 glass at different temperatures.

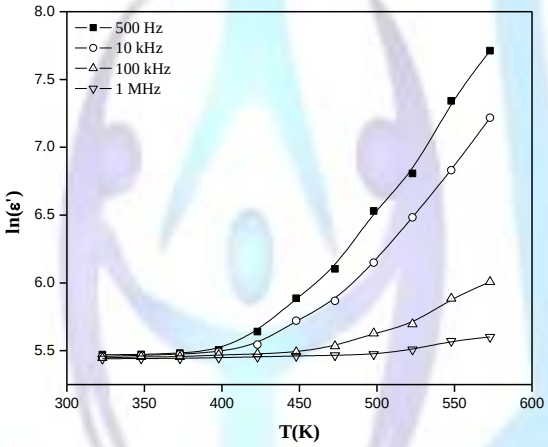

Fig 2: Dispersion of dielectric constant, ε′ versus temperature, T at different frequencies for the glass BPCL3.

Decrease of ε′ and ε″ with frequency can be understood by the fact that the ionic contribution to the total polarization in the glass (dielectric) decreases as the frequency is increased. Increase of dielectric parameters as can be seen for BPCL3 in Fig. (2) may be due to permanent dipoles which enhance amplitude of the thermal vibration of the charges as the temperature is increased [9]. These results are consistent with the reported literature for similar glass systems [23 and 24].

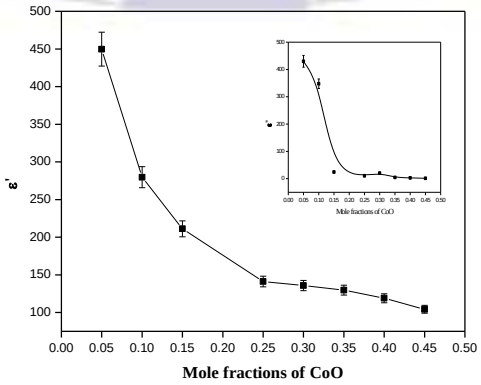

Fig 3: Compositional dependence of dielectric constant, ε′, and dielectric loss, ε″ (inset), at a frequency of 500 kHz and temperature 573K.

The variation of ε′ with mole fractions of CoO is shown in Fig. 3. The change in ε″ with CoO is shown as an inset to the Fig.3. Both ε′ & ε″ decreased with increase in CoO content. Similar kind of behavior is observed for remaining glasses of the present series.

Electrical Conductivity: The measured total conductivity can be expressed in the form of Almond-West single exponent power law [22].

$$\sigma_{Total} = \sigma_0 + A\omega^s \quad (4)$$

Where σ_0 is zero frequency conductivity, A the temperature dependent constant, s is the frequency exponent and $A\omega^s$ represents ac component of conductivity which depends on frequency. Plots of $\ln(\sigma_{Total})$ versus ln(F) at different temperatures for the glass BPCL3 is shown in Fig.4.

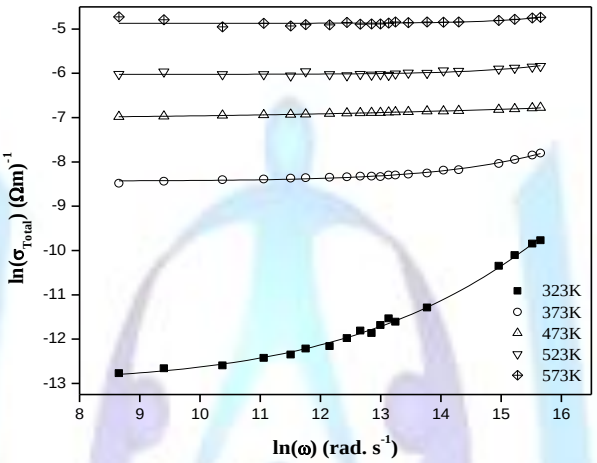

Fig 4: The plots of total conductivity, $\ln(\sigma_{Total})$ versus ln(F) for BPCL3 glass. Solid lines are the best fits to Eq. (4).

The best fits of Eqn. (4) to the data gave σ_{dc}, A and s. Similar regressional analysis has been performed on conductivity of the remaining glasses of the series. Pure ac component of the conductivity was extracted by subtracting zero frequency conductivity from the total conductivity.

The temperature dependence of ac conductivity has been considered in the light of Mott's small polaron hopping model. According to Mott's SPH model the electrical conductivity in the non-adiabatic regime is expressed as [27],

$$\sigma = (\sigma_0/T) \exp(-W/k_BT) \quad (5)$$

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

As per Eq. (5), the plots of $\ln(\sigma_{ac}T)$ versus reciprocal temperature at different frequencies for the glass BPCL7 is depicted in Fig. 5. The ac conductivity increased with increase in frequency. The least square linear lines were fit to the data and activation energy, W_{ac}, was determined from the slopes of the linear fits. The W_{ac} values thus obtained for four different frequencies of ac conductivity are recorded in Table.1.

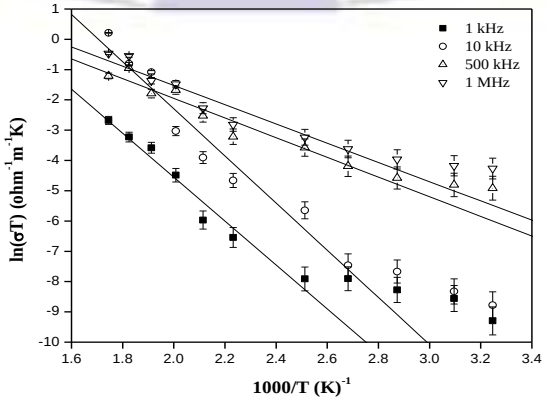

Fig 5: The plots of $\ln(\sigma_{ac}T)$ versus (1/T) for BPCL3 glass. Solid lines are the least square linear fits to the data in the high temperature region.

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Table. 1. Variation of ac activation energy, W_{ac}, at different frequencies for BPCL glasses.

Compositions of CoO	Glass	W_{ac} (eV)			
		1 kHz	10 kHz	500 kHz	1 MHz
0.05	BPCL1	0.465	0.402	0.277	0.262
0.10	BPCL2	0.493	0.438	0.325	0.298
0.15	BPCL3	0.553	0.457	0.352	0.339
0.25	BPCL4	0.571	0.499	0.383	0.350
0.30	BPCL5	0.608	0.549	0.429	0.354
0.35	BPCL6	0.521	0.378	0.419	0.289
0.40	BPCL7	0.496	0.308	0.370	0.247
0.45	BPCL8	0.346	0.237	0.327	0.186

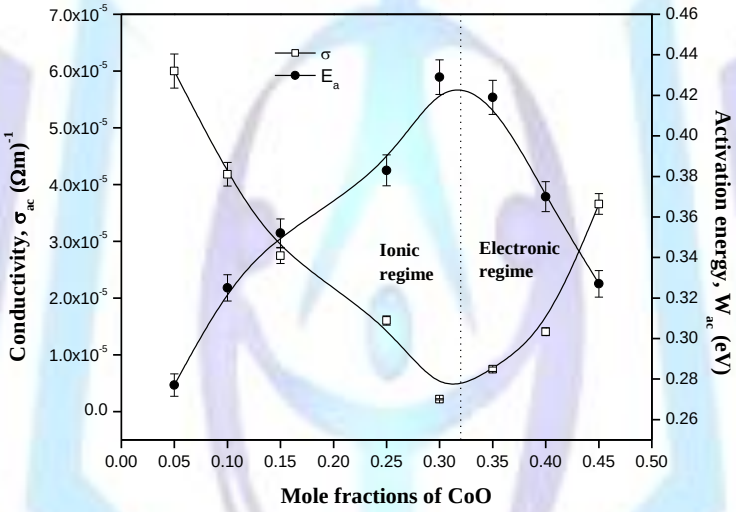

Fig 6: Variation of ac conductivity, σ_{ac}, at 523K and activation energy, W_{ac} at frequency 500kHz with mole fractions of CoO for BPCL glasses. Solid lines drawn are the guides to the eye.

From Table.1, it can be observed that W_{ac} increases with increase in frequency and increases with CoO content up to 0.3 mole fractions and decreases for higher concentrations of CoO. Variation of W_{ac} at frequency of 500 kHz and conductivity at temperature 523K of with CoO content is sketched in Fig. 6. From the figure, one can note that conductivity decreases with CoO content up to 0.3 mole fractions and increases for higher concentrations of CoO. Increase of E_a and decrease of σ, up to 0.3 mole fractions of CoO and their reverse behavior for higher concentrations of CoO may be that there is a transition of conduction mechanism from predominantly ionic regime to polaronic regime for mole fractions between 0.3 and 0.35. Transition of predominant conduction mechanism from one regime to another has been observed in several glass systems [18]. However, it can be remarked here that transition of conduction mechanism is observed for the first time in borophosphate glasses doped with CoO and Li_2O. Few theoretical ideas have been proposed [18, 28] to explain such transitions of conduction mechanisms from one regime to other.

Frequency exponent, s: Temperature dependence of frequency exponent, s, is shown in Fig.7. The measured s, values lie between 0.1 and 0.98 and decreases with increase in temperature. This kind of variation of s with temperature has been observed in different TMI/ alkali doped borate [29], phosphate [8] and borophosphate [31] glasses. The presently observed nature of change in s with temperature completely disagree with quantum mechanical theories (QMT) [30] and qualitatively agree with correlated barrier hopping (CBH) model [32] to the extent that s decreases with increase in temperature. However, CBH model derived expression [31] did not fit well with s of any of the present glasses. CBH model fit to the data of BPCL3 for ω = 1MHz is shown in Fig. 7. It can be observed that model fit line do not pass through the data points. Similar result was obtained for other glasses also. So, it can be said that CBH model also cannot explain present data satisfactorily.

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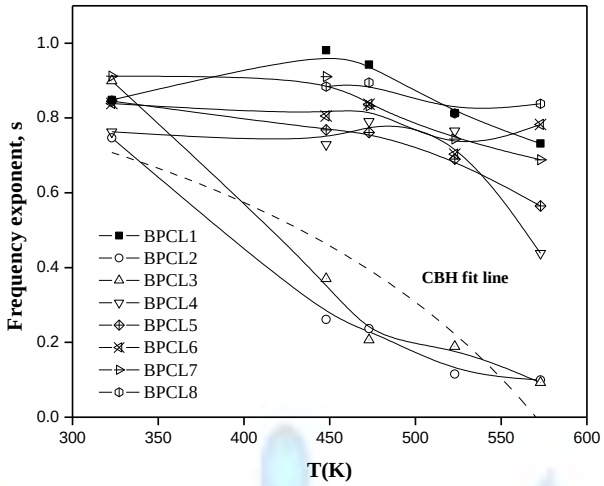
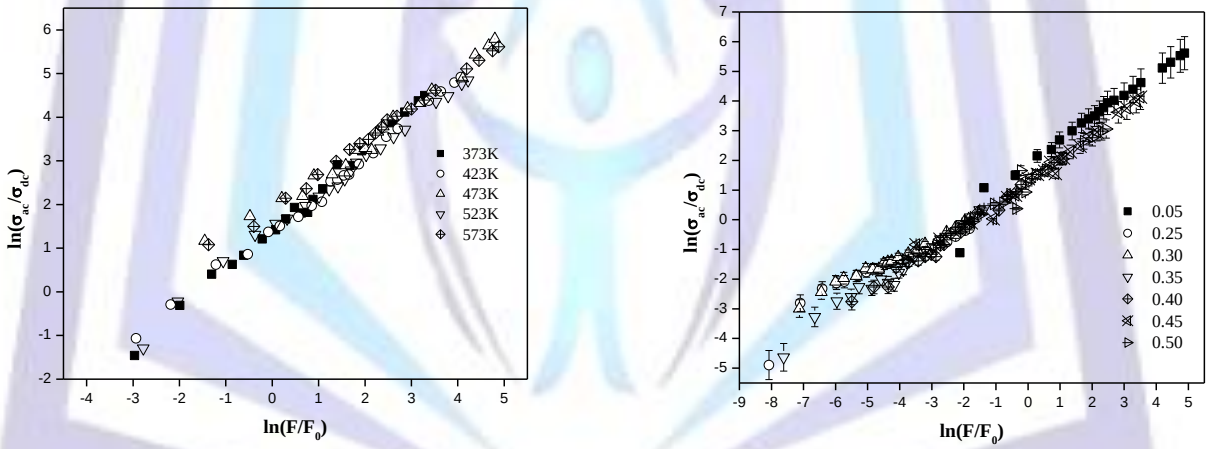


Fig 7: Temperature dependence of frequency exponent, s for BPCL glasses.

Conductivity master curves: The cross over frequency, F_0 from dc to dispersive like behavior has been found to be increasing with temperature. The frequency F_0 is defined through $\sigma'(F_0) = 2\sigma_{dc}$ [33]. It has been observed that the frequency F_0 is a thermally activated one with the same energy as the dc conductivity. Using F_0 value, the conductivity master curves are drawn and shown in Fig. 8(a) for the glass BPCL1 for different temperatures. Similarly, conductivity master curves for all the glasses at 573K are drawn and shown in Fig. 8(b).



(a) (b)

Fig 8: (a) The ac conductivity master curves for the BPCL1 glasses for different temperatures.

(b) The ac conductivity master curves of all the BPCL glasses at 573K.

From Fig. 8(a), it can be noted that the time-temperature superposition principle is fulfilled. That is, shape of σ_{ac} in log-log plot is temperature independent. This means that a temperature independent relaxation mechanism is prevalent in these glasses. Similar result has been obtained for other glasses of the present series [33]. The validity of the scaling law has been found to be valid for TMI doped glasses also [34]. From Fig. 8(b) we see that at 573K conductivity isotherms of all the glasses fall onto one master curve. This result is an indication for the existence of common ac conduction or relaxation mechanism in all the glasses at all measured temperatures. Temperature independence of relaxation in the present glasses has been further checked by normalizing ac conductivity according to scaling formula [33],

$$\frac{\sigma_{(ac)}}{\sigma_{(dc)}} = F\left(\frac{\omega}{\sigma_{dc}T}\right) \quad (6)$$

As per Eqn. (6) $(\sigma_{ac}/\sigma_{dc})$ versus $(\omega/\sigma_{dc}T)$ for BPCL1 glass for different temperature are plotted in Fig. 9. Here, we noted that individual curves are, shifted to higher values of x-axis as temperature increases. Similar results were found in sodium doped borate glasses [34].

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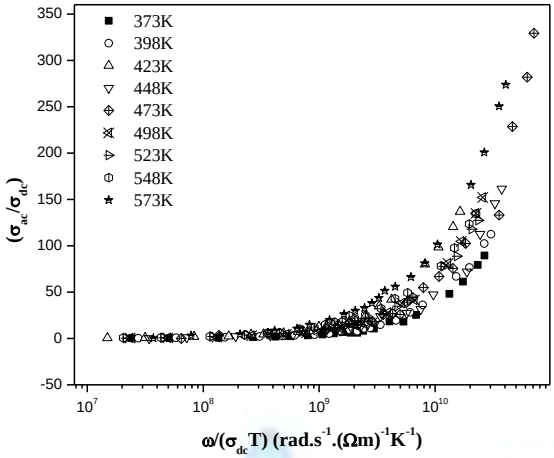


Fig 9: Conductivity master curves of BPCL1 glass.

Relaxation processes: Here, Hunt's model has been invoked to understand relaxation process which has been used earlier for many glasses [35-37]. Hunt's model considers two distinct charge migration processes depending on the frequency domains i.e., $\omega < \omega_m$ and $\omega > \omega_m$ where ω_m is generally taken as equivalent to the cross over frequency F_0, which is nothing but the characteristic frequency for the onset of ac conduction [25]. In these two domains, the total conductivity can be expressed as [38].

$$\sigma_t(\omega) = \sigma_{dc}\left(1 + A\left(\frac{\omega}{\omega_m}\right)^s\right) \quad \text{for} \quad \omega > \omega_m \tag{7}$$

$$\sigma_t(\omega) = \sigma_{dc}\left(1 + K(d)\left(\frac{\omega}{\omega_m}\right)^\gamma\right) \quad \text{for} \quad \omega < \omega_m \tag{8}$$

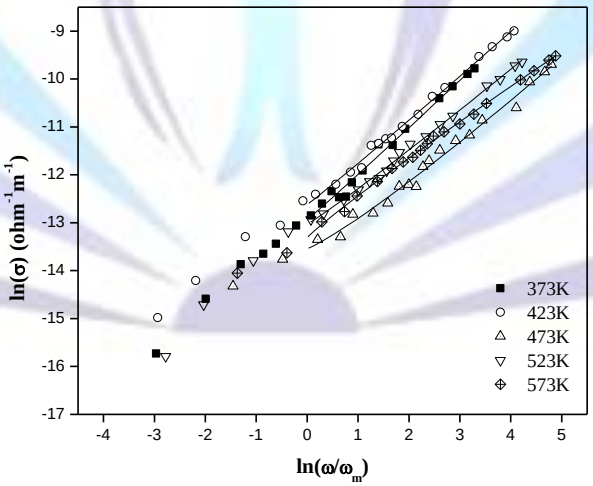


Fig 10: The plots of ac conductivity, ln(σ_{ac}) versus ln(ω/ω_m) for BPCL1 glass. The Solid lines are the best fits to Hunt's Eqn. (7).

Considering ω_m to be equivalent to F_0, the Eq. (7) has been fit to conductivity data of BPCL1 glass for different temperatures and shown in Fig. 10, on ln-ln scale [38]. The best fits gave σ_{dc}, *A* and exponent, s, which are of the same order as that obtained from the fits made to Eq. (4) (Fig. 4). Therefore, it can be said that the ac conductivity in present glasses can be understood by Hunt's model in the frequency domain $\omega > \omega_m$.

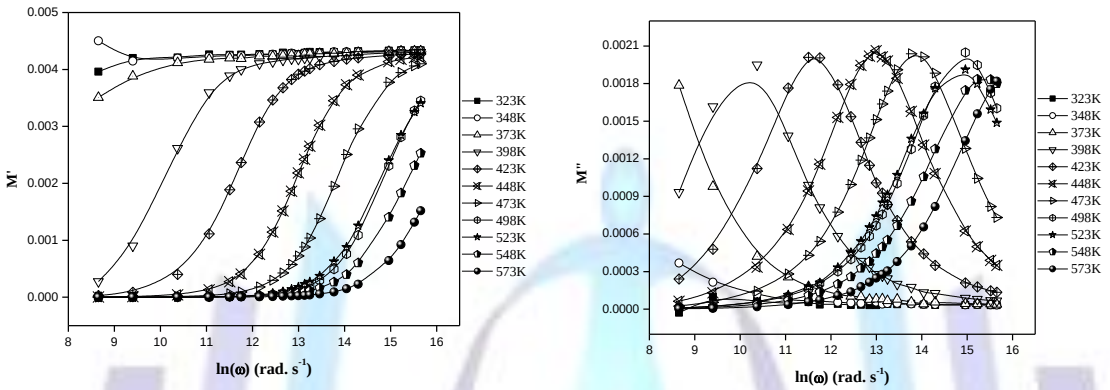
In order to understand electrical relaxation better, the real and imaginary parts of electric moduli, M′ and M″ were determined as per the following expressions [22],

$$M' = \frac{\varepsilon'}{(\varepsilon')^2 + (\varepsilon'')^2} \quad (9)$$

and

$$M'' = \varepsilon''/([(\varepsilon')]^{\wedge}2 + [(\varepsilon'')]^{\wedge}2) \quad (10)$$

Frequency dispersion of these two electrical moduli are plotted in Figs. 11(a) & (b) for BPCL3 glass for different temperatures.



(a) (b)

Fig 11: (a) Frequency dependence of M′ at different temperatures and (b) Frequency dependence of M″ at different temperatures for BPCL3 glass.

In these figures, relaxation features of dielectric properties of the studied glasses can be seen clearly. It should be noted that M′, increased with increasing frequency and at high frequencies it has reached saturation. The maximum of M″ (peak) is shifted to higher frequency with increasing temperature. Similar dispersion behavior has been exhibited by other glasses of the present series. This result is in agreement with observations made in [24].

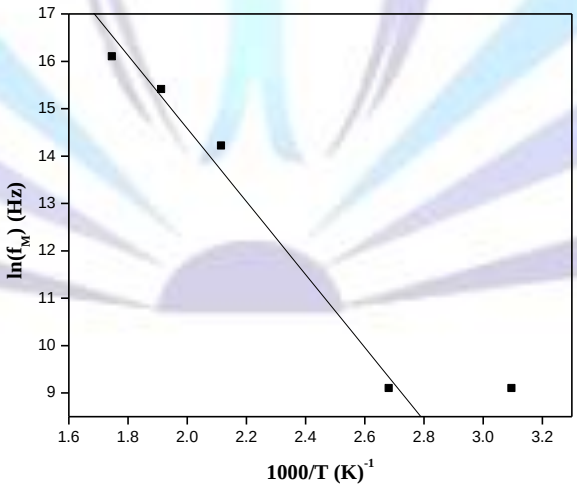


Fig 12: Variation of f_M with temperatures for BPCL3 glass.

The frequency corresponding to maximum M″ is known as relaxation frequency f_M. For the frequency range above f_M, the carriers are spatially confined to their potential wells [12]. Thus, frequency f_M indicates transition from long range to short range mobility and is defined by the condition, $2\pi f_M \tau_m = 1$ [12], where τ_m is the dielectric relaxation time. The τ_m values obtained at yhis different temperatures for the present glasses are tabulated in Table. 2. Shift in f_M towards higher frequency with increasing temperature reveals that relaxation mechanism is thermally activated. Therefore, Arrhenius relation, $f_M = f_0 \exp\left(\frac{-E_M}{k_T}\right)$ has been used to analyze f_M behavior with temperature. A plot of ln(f_M) versus (1/T) is

made and shown in Fig. 12. A linear line was fit to the data and activation energy E_M was determined. E_M value was found to be 0.664 eV. This value of E_M is in agreement with literature [12].

Table 2: Dielectric relaxation time, τ_m extracted from Fig 11 (b) of M″ curves.

Glass	T_0 (s)	
	373K	473K
BPCL1	4.261×10^{-6}	1.933×10^{-5}
BPCL2	9.759×10^{-7}	----
BPCL3	1.054×10^{-6}	2.508×10^{-8}
BPCL4	8.421×10^{-6}	5.881×10^{-8}
BPCL5	2.375×10^{-6}	7.823×10^{-8}
BPCL6	1.872×10^{-5}	1.632×10^{-6}
BPCL7	6.738×10^{-6}	7.029×10^{-6}
BPCL8	3.204×10^{-6}	2.092×10^{-5}

CONCLUSION

Transition metal ions (CoO) and alkali ions (Li_2O) doped borophosphate glasses were synthesized and investigated for dielectric properties over a wide range of frequency and temperature. The dielectric spectra in terms of frequency, temperature and composition were analyzed and discussed. The analysis of ac conductivity, frequency exponent and electric moduli lead to the following mentioned conclusions:

(i) The ac conductivity at high temperature has been found to vary as per Mott's small polaron hopping model. The activation energy for the conduction was determined.

(ii) Conductivity decreased and activation energy increased with increasing CoO content up to 0.30 mole fractions and behaved oppositely for higher concentration of CoO. This indicated transition of conduction mechanism from predominantly ionic regime to electronic regime. This is for the first time such a transition has been detected in borophosphate glasses.

(iii) The relaxation process of ac conduction obeyed Hunt's model for frequencies above ω_m.

(iv) Frequency exponent is found to be temperature dependent.

(v) Master curves drawn for conductivity indicated the existence of temperature and ion concentration independent relaxation mechanism in the present glasses.

(vi) Dispersion behavior of electric moduli revealed relaxation features of dielectric properties. Activation energy for relaxation mechanism has been determined.

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