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ORIGINAL PAPER

Dielectric and AC conductivity studies in Li_2O-CoO-B_2O_3-TeO_2 glasses

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Abstract CoO and Li_2O mixed with borotellurite glasses in the compositions, $(B_2O_3)_{0.2}$-$(TeO_2)_{0.3}$-$(CoO)_x$-$(Li_2O)_{0.5-x}$, where x = 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, 0.45, and 0.50 were synthesized by fast cooling the melt to room temperature. Absence of crystalline phases in the samples was confirmed by X-ray diffraction studies. Changes in dielectric properties with frequency and temperature over wide ranges have been measured. Dielectric constant and loss increased with increase in CoO content. AC conductivity has been analyzed using Mott's small polaron model and activation energy was determined. Activation energy decreased and conductivity increased with increase in CoO content up to 0.3 mole fractions, and they behaved oppositely for higher concentration of CoO. This observed change of trend in activation energy and conductivity at 0.3 mole fraction of CoO ascribed to switch over of conduction mechanism occurring from predominantly ionic to electronic regime. For the first time, a transition of conduction mechanism is observed in borotellurite glasses. Temperature and composition independent relaxation mechanism in these glasses has been confirmed by plotting the scaled conductivity master curves. Hunt's model has been invoked to understand the frequency dispersion of conductivity.

Keywords Borotellurite glasses · Dielectric constant · Conductivity · Hunt's model

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Introduction

Tellurium oxide (TeO_2)-based glasses are of technical and scientific interest because of their unique properties. It was reported that tellurite glasses improved their quality in terms of transparency and refractive index when some amount of B_2O_3 is added to their network [1]. They are highly stable against divitrification, non-toxic, and resistant to moisture for long periods as compared to other glasses [2]. These glasses measured electrical conductivity of several orders of magnitude higher than silicate, borate, and phosphate glasses containing same amount of modifiers [3]. The addition of TeO_2 to any other glass former, such as B_2O_3, is of scientific and practical interest and this leads to the formation of interesting structural units that affect their overall physical properties [4]. Ionic conductivity studies in TeO_2 glasses have been pursued largely because of their utility in solid state devices. For example, ionic conductivity was found to increase steeply in Li_2O-TeO_2 glasses and that was interpreted to be due to increase in the number of non-bridging oxygen with increase in Li_2O concentration [5]. Tellurite glasses containing transition metal ions were used as elements in memory switching devices, cathode materials for batteries, and as optical CD memory devices [6].

AC conductivity was found to be increased with frequency and decreased with alkali content in single alkali-doped vanadotellurite glasses [7] and mixed alkali-doped vanadotellurite glasses [8]. Tellurite glasses doped with single and mixed transition metal ions [TMI], $(V_2O_5)_x$-$(TeO_2)_{1-x}$ and $(V_2O_5)_{0.4}$-$(CoO)_x$-$(TeO_2)_{0.6-x}$, were studied for dielectric properties with frequency and temperature as input variables over wide ranges [9]. Conductivity in a set of two transition metal ion-doped tellurite glasses has been studied and the results suggested that multiphonon tunneling of large polarons between the micro clusters that existed in them was

responsible for conduction [10]. Mixed transition effect was detected in Fe_2O_3-MnO-TeO_2 glasses, where activation energy and resistivity both passed through maximum of 0.4 transition ion ratio, Mn/(Mn + Fe) [11].

Mixed alkali effect was observed in borotellurite glasses in terms of AC conductivity [12]. Ag_2O-doped borotellurite glasses showed decrease in dielectric parameters and increase in conductivity increased with increase in frequency. Activation energy for conduction decreased with increase in mole fractions of Ag_2O [13, 14]. LiCl-doped borotellurite glasses were measured for both DC and AC conductivity, and conduction mechanism in these glasses was proposed to be due to switching of alkali ions between bridging and non-bridging oxygen in the network. Dielectric and AC conductivity studies were carried out on bismuth oxide incorporated borotellurite glasses [15]. Frequency exponent of conductivity has been viewed.

Theoretical models such as polaron hopping [16, 17], quantum mechanical tunneling [18], correlated barrier hopping, and Hunt's model [19] have always been used to interpret conductivity behavior with temperature in most of the above cited oxide glasses.

No research papers were found in literature on dielectric and AC conductivity studies of CoO- and Li_2O-doped borotellurite glasses though it is known that these glasses are chemically more durable than borate and tellurite glasses [20]. In the non-crystalline solids, cobalt ions exist both in Co^{2+} and Co^{3+} valence states. Hopping of electrons (polarons) between these ions adds electronic conduction [21]. The transition metal ion-doped glasses not only are interesting for structure and luminescence like properties, they do help in understanding insulating nature, dielectric properties, and electrical conduction mechanisms operated in the systems in different regimes of frequencies [22]. Further, mixed conducting glasses where both polarons and ions are participating in the conduction process are of great importance as they can be useful in solid state battery applications. Keeping in view of these aspects, dielectric and conductivity studies on a set of CoO- and Li_2O-doped borotellurite glasses have been carried out. The results were thoroughly analyzed and presented in this article.

Experimental

Borotellurite glasses mixed with CoO and Li_2O in the compositions, $(B_2O_3)_{0.2}$-$(TeO_2)_{0.3}$-$(CoO)_x$-$(Li_2O)_{0.5-x}$, where x = 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, 0.45, and 0.50 were synthesized by melt quenching method at 1300 K in a molybdenum-made electric furnace using AR grade chemicals, H_3BO_3, CoO and Li_2CO_3, and Sigma-Aldrich-made TeO_2. The samples thus formed were collected, annealed at 575 K, and labeled as BTCL1, BTCL2, BTCL3, BTCL4, BTCL5, BTCL6, BTCL7, BTCL8, BTCL9, and BTCL10, respectively. They were then subjected to powder X-ray diffraction (XRD) studies in a Bruker D8 Advance X-ray diffractometer. The XRD scans were carried out for the Bragg's angle, 2θ in the range from 0 to 80 °.

The glasses were shaped to required size (3 mm × 2 mm × 2 mm), and their two large surfaces were silver painted. The measurements of capacitance, C and dissipation factor, tanδ, were carried out in a high precision impedance analyzer (Wayne Kerr, 6520B) for the frequency range from 100 Hz to 3 MHz and the temperature from 300 to 575 K. Temperature was measured using Chromel-Alumel thermocouple with an accuracy of ±1 K. The dielectric constant (ε'), dielectric loss (ε'') and AC conductivity (σ_{AC}) were determined as per the expressions [8, 12],

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

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

$$\sigma_{AC} = \omega \varepsilon_0 \varepsilon'' \quad (3)$$

where, ε_0 is the permittivity of free space, $\omega = 2\pi f$ (f is the frequency) d the thickness, and A the cross sectional area of the glass.

Results and discussion

XRD

The X-ray powder diffraction patterns of all the ten BTCL glasses are shown in Fig. 1. No sharp peaks are observed in the spectra of any of the patterns which suggest absence of long range atomic arrangement in the samples. This result confirms amorphous nature of the present sample. Similar XRD patterns were obtained for the remaining glasses.

Dielectric properties

The measured dielectric constant, ε', and dielectric loss, ε'', were found to lie in the range of 10^2 to 10^3 and 10^1 to 10^6, respectively. The plots of ε' and ε'' versus ln(F) for different temperatures for the glass BTCL2 are shown in Fig. 2a, b, respectively. These values of ε' and ε'' are comparable with those reported in reference [8].

It can be noticed that ε' and ε'' decrease with increase in frequency and increase with increase in temperature. Similar nature of variation of ε' and ε'' has been observed for other glasses in the present series. The increase in dielectric constant with increase in temperature may be due to the fact that increase of temperature weakens the intermolecular forces and hence enhances the orientational vibration [20, 21]. Further, addition of cobalt ions may be creating more

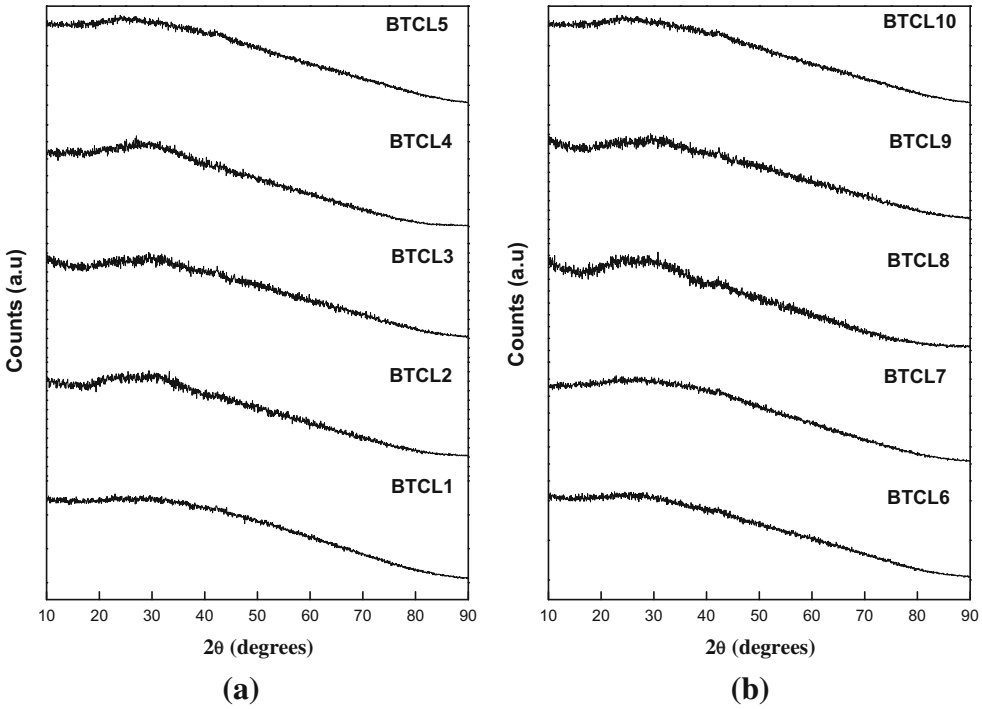


Fig. 1 X-ray diffraction patterns of **a** BTCL1 to BTCL5 glasses **b** BTCL6 to BTCL10 glasses

and more non-bridging oxygen ions. These non-bridging oxygen ions may create migration paths for the charge carriers which in turn increases space charge polarization and hence increases dielectric constant with temperature. The dielectric constant is large at lower frequencies which indicates rapid polarization processes occurring in the glasses. This may be due to that as the frequency is increased, the polarizability contribution from ionic and orientation sources decreases and finally disappears due to the inertia of the ions. At low temperatures, the contribution of electronic and ionic components to the total polarizability will be small. As the temperature is increased the electronic and ionic polarizability contributions start to increase.

The compositional dependence of ε' at 573 K for a frequency of 1 MHz is shown in Fig. 3. Inset shows variation of ε'' with x. It can be noted that both ε' and ε'' decrease with increase in CoO content. This clearly infers that space charge contribution to the dielectric constant is more for lower concentrations than at higher concentrations of CoO. The number of non-bridging oxygen ions (defects) produced in the network with increasing content of CoO increases which in turn decreases dielectric constant with CoO content. Similar type

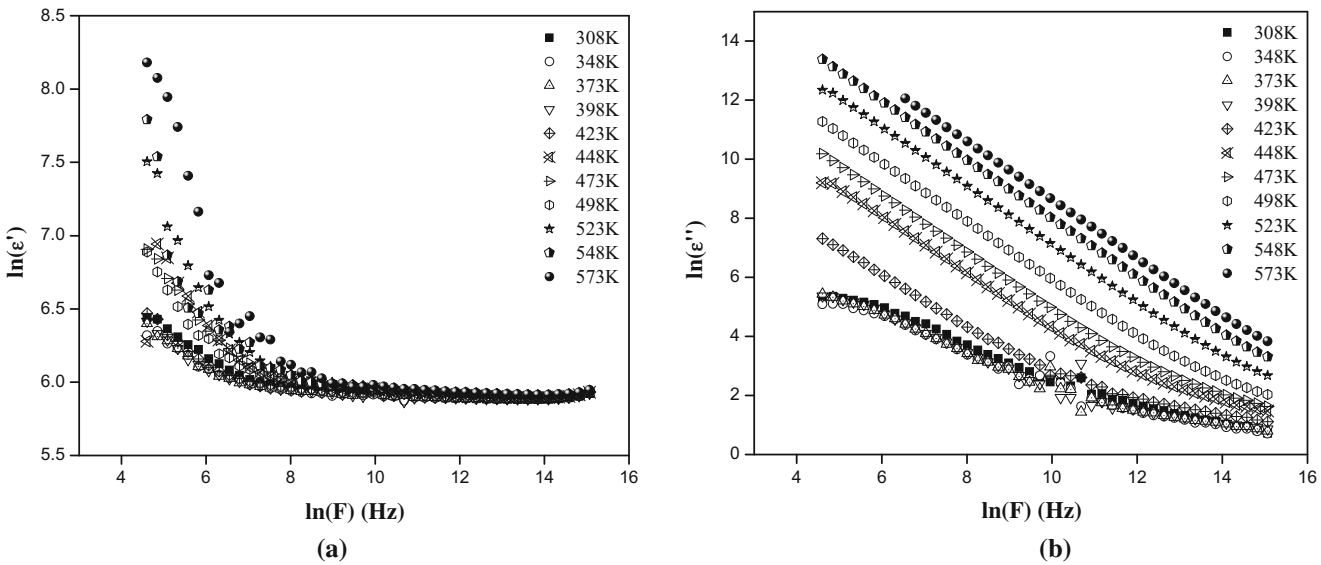


Fig. 2 Plots of **a** ln(ε') versus ln(F) and **b** ln(ε'') versus ln(F) for BTCL2 glass at different temperatures

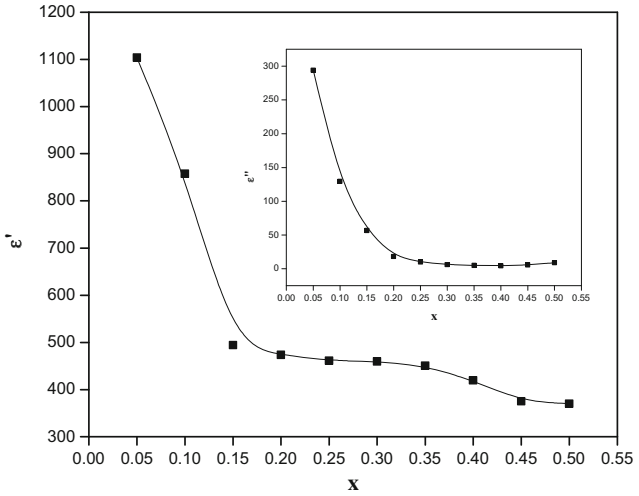


Fig. 3 Compositional dependence of dielectric constant, ε', and dielectric loss, ε'' (*inset*), at a frequency of 1 MHz and temperature of 573 K

of variation of dielectric parameters with x has been observed for all the experimental range of frequency and temperature.

AC conductivity

The frequency dependence of conductivity for the BTCL3 glass is depicted in Fig. 4. It can be observed that the conductivity increases with increase in frequency and temperature. Similar kinds of results were noted for the remaining BTCL glasses. The conductivity is generally expressed as follows:

$$\sigma_{\text{Total}} = \sigma_0 + A\omega^s \tag{4}$$

Where σ_0 (=σ_{DC}) is the frequency-independent component, A the temperature dependent constant, s is the frequency exponent, and σ_{AC} (=$A\omega^s$) represents the dissipative contribution to the total conductivity [23].

The best fits of Eq. (4) to the data which gave σ_{DC}, A, and s. Similar regressional analysis has been performed on conductivity data of the remaining glasses of the series. Pure AC component of the conductivity, σ_{AC}, was extracted by subtracting σ_{DC} from σ_{Total}.

The temperature dependence of σ_{AC} has been analyzed with the help of Mott's small polaron hopping model [8, 16]. As per Mott's small polaron hopping theory, the plots of $\ln(\sigma_{AC}T)$ versus $(1/T)$ at different frequencies were made for the glass BTCL9 and shown in Fig. 5. The σ_{AC} increases with increase in frequency. The least square linear lines were fit and activation energy, W_{AC}, was determined from the slopes. The W_{AC} values thus obtained for three different frequencies are recorded in Table 1. Activation energy increases with CoO content up to 0.3 mol fractions and decreases for higher amounts of CoO. In order to highlight that σ_{DC} lies below $\sigma_{AC,}$ a linear fit to the DC data is also shown in Fig. 5. Similar analysis has been carried out on the conductivity data of all the glasses of the present series.

DC activation energy, W_{DC} values are found to be less than AC activation energy, W_{AC} for all the compositions. This result is consistent with the observation made in other oxide glasses [6]. From Table 1, it can be noticed that W_{DC} increased with CoO concentration up to 0.3 mol fractions and decreased for $x > 0.3$. This behavior activation energy is in agreement with AC result.

Variation of W_{AC} for the frequencies of 1 MHz and 10 kHz and conductivity at the temperature of 573 K with CoO concentration is sketched in Fig. 6. From the figure, it can be noted that conductivity decreases with CoO content up to 0.3 mol fractions and increases for higher concentrations of CoO. Activation energy, $W_{AC,}$ behavior with CoO is observed

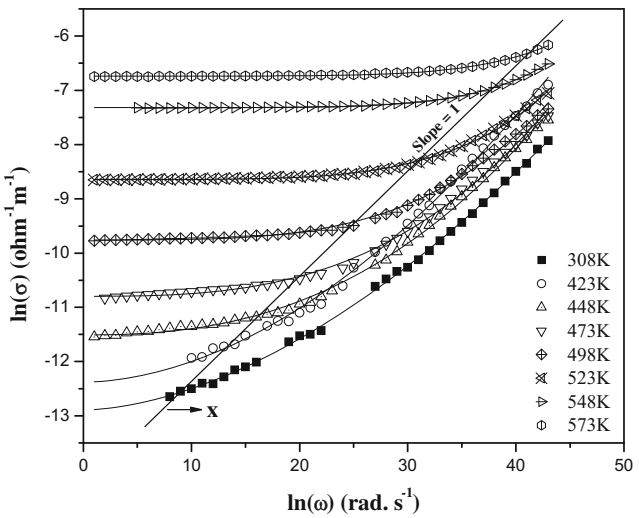


Fig. 4 The plots of $\ln(\sigma_{Total})$ versus $\ln(\omega)$ for BTCL3 glass. *Solid lines* are the best fits to Eq. (4). The linear line, x, with slope unity connects the cross over frequency, F_0, of each isotherm

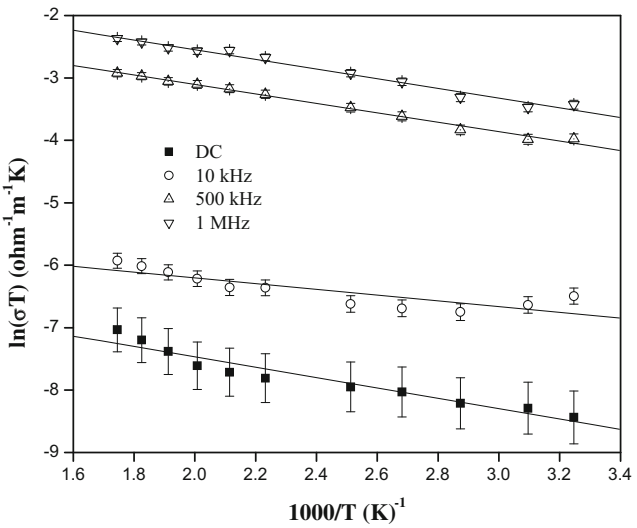


Fig. 5 The plots of $\ln(\sigma T)$ versus $(1/T)$ for BTCL9 glass. *Solid lines* are the least square fits to the data in the high-temperature region

Table 1 Variation of AC activation energy, W_{AC}, at different frequencies for BTCL glasses

Glass	Compositions of CoO	W_{DC} (eV)	Activation energy, W_{AC} (eV) ± 0.001 eV		
			10 kHz	500 kHz	1 MHz
BTCL1	0.05	0.873	0.458	0.156	0.101
BTCL2	0.10	0.880	0.467	0.177	0.109
BTCL3	0.15	0.888	0.472	0.181	0.134
BTCL4	0.20	0.893	0.485	0.190	0.152
BTCL5	0.25	0.906	0.492	0.200	0.172
BTCL6	0.30	0.977	0.508	0.216	0.285
BTCL7	0.35	0.279	0.458	0.156	0.154
BTCL8	0.40	0.130	0.312	0.067	0.071
BTCL9	0.45	0.092	0.321	0.069	0.063
BTCL10	0.50	0.062	0.309	0.051	0.057

to be opposite to that of σ_{AC}. Increase of W_{AC} and decreases of σ up to 0.3 mol fractions of CoO and their reverse behavior for higher concentrations of CoO may be that there is a transition occurring in conduction mechanism from predominantly ionic regime to polaronic regime for CoO mole fractions around 0.3. Transition of predominant conduction mechanism from one regime to another has been observed in lithium vanadium tellurite [24, 25], sodium vanadophosphate [26], and several glass systems which were listed in references [7, 8, 24, 27, 28]. However, it can be specifically noted that transition of conduction mechanism is observed for the first time in mixed conducting borotellurite glasses. Few theoretical ideas such as ion-polaron interaction, existence of two independent paths for ions and polarons wherein one is blocking the path of the other when the content of one is progressively added at the cost of the other, dynamic structure model etc., have been proposed to explain such transitions of conduction mechanism from one regime to another [25]. Of these, blocking of conducting paths of polarons by the ions or vice-versa may be responsible for the observed transition of conduction mechanism in the present glasses. The mutual blocking of path ways of polarons and lithium ions may reach maximum giving rise to minimum of conductivity and maximum of activation energy at around 0.3 mol fractions of CoO.

It may be noted that the number of non-bridging oxygen (NBOs) in the glass structure was found changing with the substitution of one oxide with another oxide in reference [15]. The observed changes in conductivity were discussed in terms of NBOs. The information about NBOs can be obtained through FTIR, XPS like studies. Due to non-availability of facilities, the present glasses have not been subjected to these studies. Hence, no support for increase of NBOs with increasing CoO substitution can be provided for the present glasses.

Frequency exponent, *s*

Temperature dependence of frequency exponent, *s*, is shown in Fig. 7. The *s* values lie between 0.65 to 0.95 and decreases

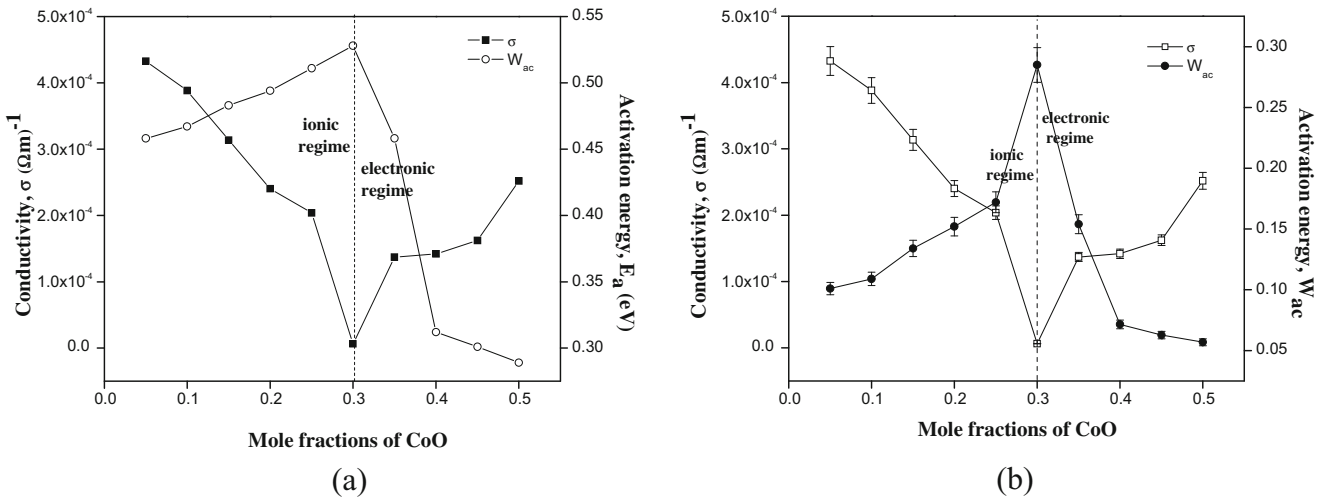


Fig. 6 Variation of σ_{AC} at 573 K and W_{AC} at frequency **a** 10 kHz **b** 1 MHz with mole fractions of CoO for BTCL glasses. *Solids lines* drawn are the guides to the eye

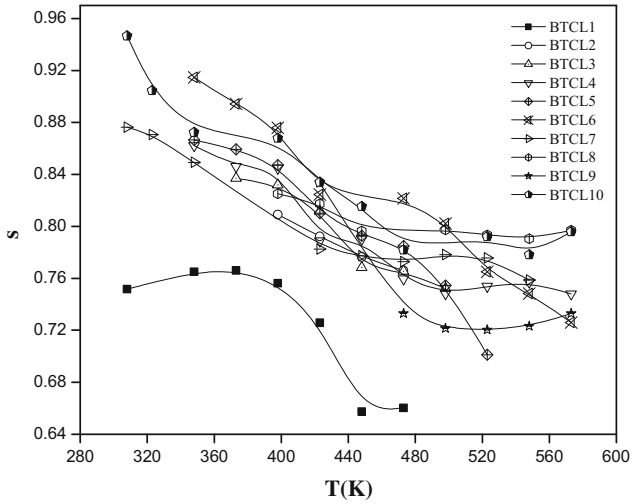


Fig. 7 Plots of temperature dependence of frequency exponent, *s*, for BTCL glasses

with increase in temperature. This kind of variation of *s* with temperature has been observed in different TMI/ alkali-doped borate [29], phosphate [30], and borophosphate [31] glasses. The presently observed nature of change in *s* with temperature completely disagrees with quantum mechanical theories (QMT) [18] as they predict temperature independent *s*. However, it agrees qualitatively with correlated barrier hoping (CBH) model [31] to the extent that *s* decreases with increase in temperature. CBH model expression fits to the *s* of all the samples that are shown in Fig. 8. In the figure, it can be observed that CBH fit lines and the experimental *s* values do not agree with each other completely. So, it can be stated that CBH model explains s qualitatively and fails to explain quantitative variation of *s* with temperature. It may be noted that the frequency exponent was found to be varied according to CBH model in silver ferro-phosphate glasses [32]. To compare, there are no evidences in the literature for temperature dependence of frequency exponent in mixed conduction borotellurite glasses obeying CBH model.

Conductivity master curves

Frequency, F_0, at which frequency independent conductivity starts becoming frequency dependent is defined through $\sigma'(F_0) = 2\sigma_{DC}$ [33]. It is known that F_0 is thermally activated. Using F_0, conductivity master curves for the glass BTCL2 for different temperatures are drawn and shown in Fig. 9a. Also, conductivity master curves for all the glasses at 573 K are drawn and shown in Fig. 9b.

From Fig. 9a, it can be noted that the time-temperature superposition principle is fulfilled. This means that a temperature independent relaxation mechanism prevails in the present glasses. Similar result has been obtained for other glasses of the present series [33]. It may be noted that scaling law has been found to be valid for TMI-doped glasses [34]. From

Fig. 8 Plots of *s* versus *T* for BTCL glasses. *Solid line* is CBH model fit to the data

Fig. 9b, it can be seen that at 573 K, conductivity isotherms of all glasses fall onto one master curve. This result can be taken as further evidence for the existence of common relaxation mechanism in these glasses at all measured temperatures.

Temperature independence of relaxation in the present glasses has been further tested by normalizing AC conductivity according to scaling formula [33].

$$\frac{\sigma_{(AC)}}{\sigma_{(DC)}} = F\left(\frac{\omega}{\sigma_{DC}T}\right) \tag{5}$$

As per Eq. (5), the plots of $(\sigma_{AC}/\sigma_{DC})$ versus $(\omega/\sigma_{DC}T)$ for the BTCL1 glass for different temperatures are plotted and shown in Fig. 10. Here, we note that individual curves are shifted to higher values of *x*-axis as temperature increases. Similar results were found in borate [34] and borophosphate glasses [35, 36].

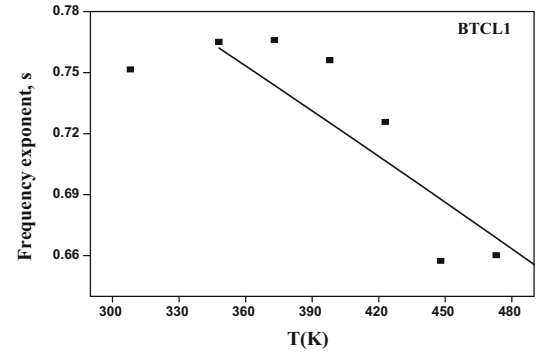
Relaxation processes

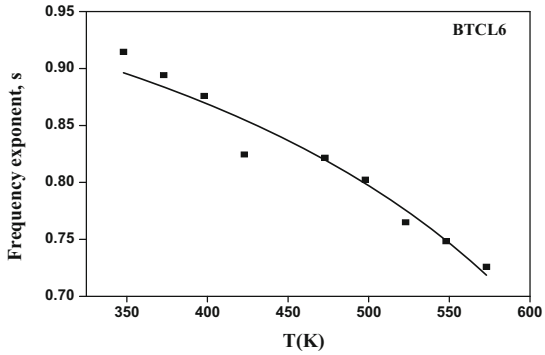
Hunt's model has been invoked to understand relaxation process in the present series of glasses. Hunt's model considered two distinct charge migration processes depending on frequency, i.e., $\omega < \omega_m$ and $\omega > \omega_m$ where ω_m is the cross over frequency F_0 [36]. In these two frequency domains, the total conductivity was expresses as [8, 36]

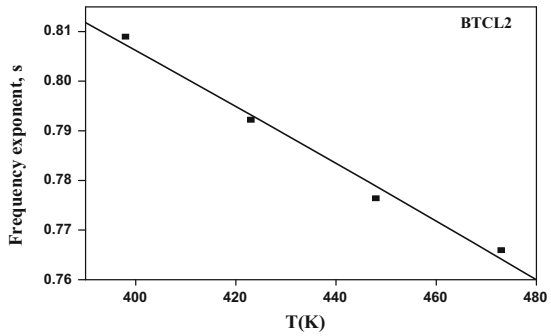
$$\sigma = \sigma_{DC}\left(1 + A\left(\frac{\omega}{\omega_m}\right)^s\right) \quad \text{for} \quad \omega > \omega_m \tag{6}$$

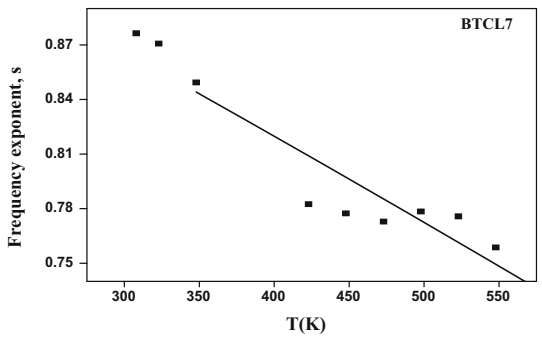
$$\sigma = \sigma_{DC}\left(1 + K(d)\left(\frac{\omega}{\omega_m}\right)^r\right) \quad \text{for} \quad \omega < \omega_m \tag{7}$$

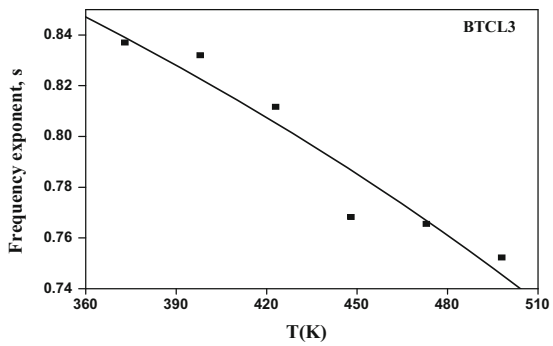
By taking $\omega_m = F_0$, Eq.(6) has been fit to the conductivity of all the glasses. Here, F_0 is the characteristic frequency for the onset of frequency dependent conductivity. To save space, typical fits only for four different temperatures for the present glasses are shown in Fig. 11, on ln-ln scale. The best fits gave σ_{DC}, *A*, and *s* which are numerically found to be almost same as that obtained from the fits of Eq. (4) to the total conductivity of the glasses as shown in Fig. 4 for BTCL3 glass. This result clearly indicates that AC conductivity in the present glasses can be understood by the Hunt's model in the frequency domain, $\omega > \omega_m$, and the relaxation process can be understood to be thermally activated. Hunt's model has been reported to be adequate in understanding relaxation process in single and mixed TMI-doped tellurite glasses [8] and in borate, phosphate and tellurite glasses [37], and silver vanadate glasses [38].

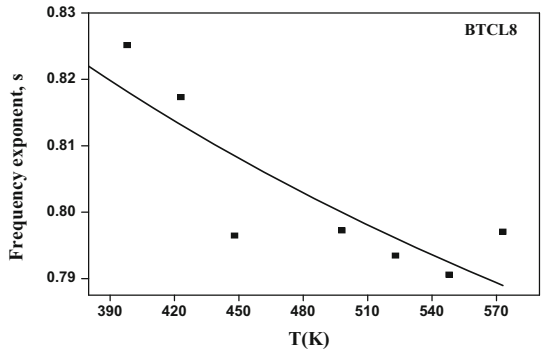

BTCL1
Frequency exponent, s
T(K)

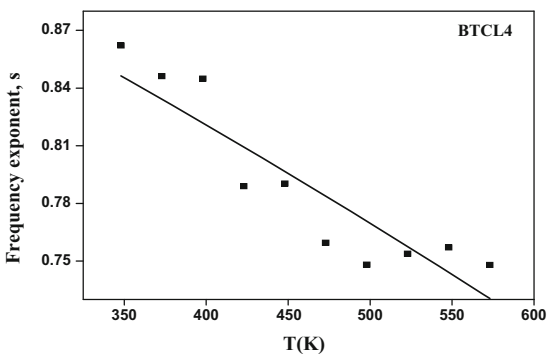

BTCL6
Frequency exponent, s
T(K)

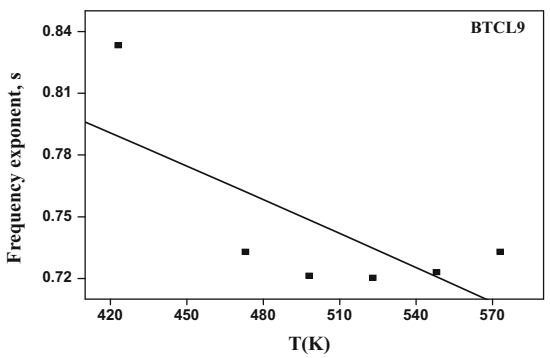

BTCL2
Frequency exponent, s
T(K)

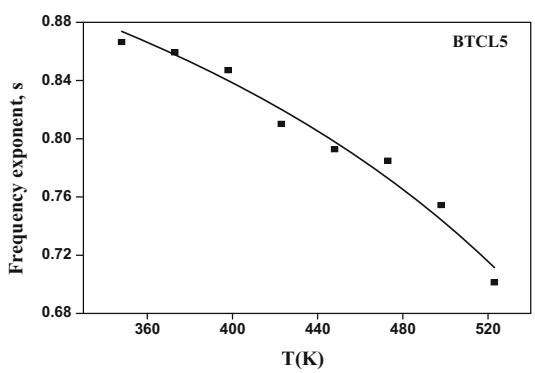

BTCL7
Frequency exponent, s
T(K)

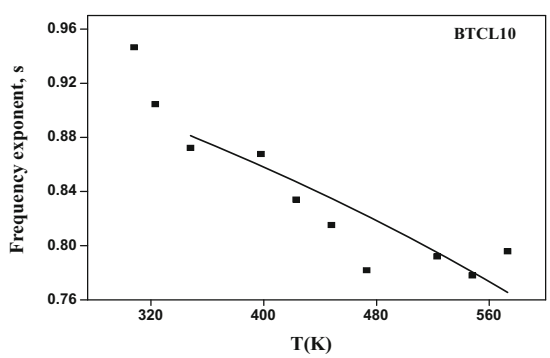

BTCL3
Frequency exponent, s
T(K)


BTCL8
Frequency exponent, s
T(K)


BTCL4
Frequency exponent, s
T(K)


BTCL9
Frequency exponent, s
T(K)


BTCL5
Frequency exponent, s
T(K)


BTCL10
Frequency exponent, s
T(K)

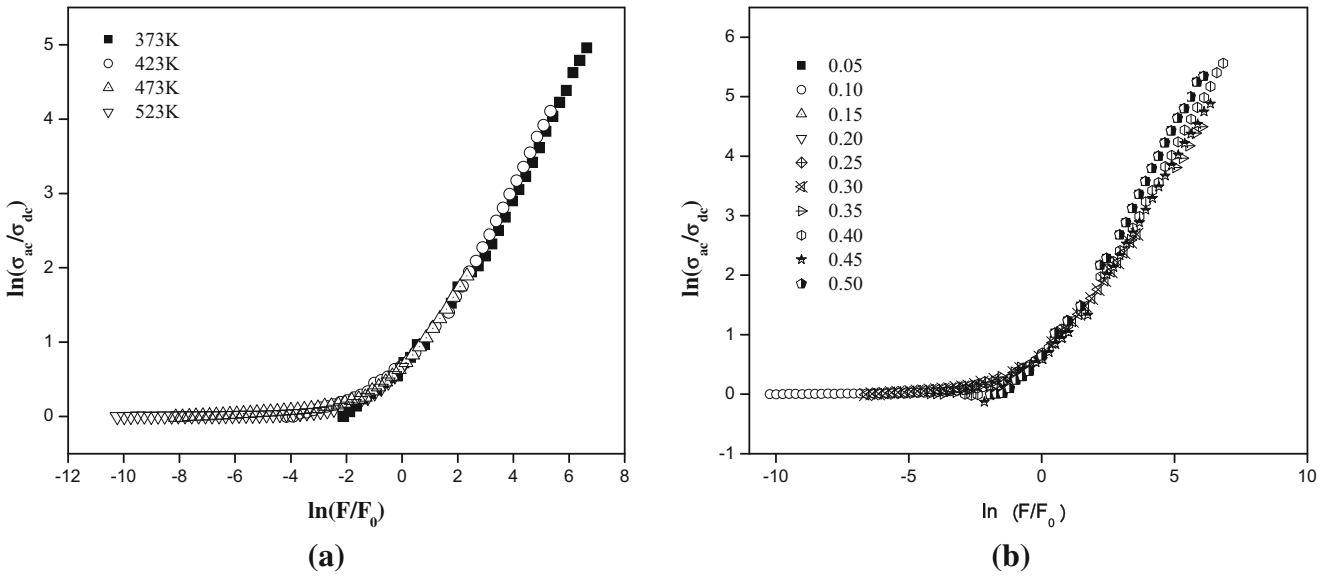


Fig. 9 **a** AC conductivity master curves for the BTCL2 glass for different temperatures. **b** AC conductivity master curves of all the BTCL glasses at 573 K temperature

Conclusions

Borotellurite glasses doped with CoO and Li_2O were synthesized and investigated for dielectric properties.

(i). Dielectric constant, ε', and loss, ε'', decreased with increase of frequency and CoO concentration and increases with increase in temperature.

(ii). Using dielectric data, AC conductivity has been determined. Conductivity variation with temperature has been analyzed in the light of the Mott's small polaron hopping model and high temperature activation energy was determined.

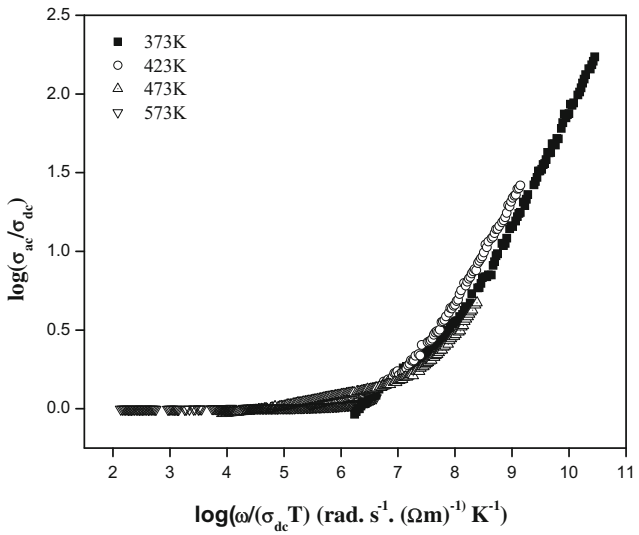


Fig. 10 Conductivity master curves of BTCL1 glass drawn as per scaling formula

(iii). AC conductivity decreased and activation energy increased with increasing CoO content up to 0.3 mol fractions, and they showed opposite behavior for higher concentration of CoO. This indicated a transition of conduction mechanism from predominantly ionic regime to electronic regime occurring in these glasses around 0.3 mol fractions of CoO. This is a chief result of this article.

(iv). Variation of frequency exponent of conductivity decreased with increase in temperature which negated the prediction of quantum mechanical tunneling theory. However, the decrease of frequency exponent with temperature is qualitatively in agreement with correlated barrier hopping (CBH) model but the exact nature of decrease is not in conformity with CBH model.

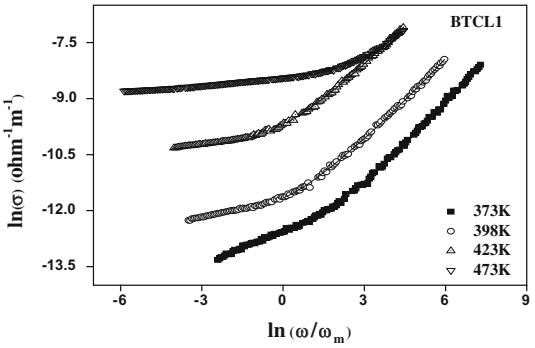
(v). The Hunt's model has been found to be satisfactorily explaining relaxation process in these glasses in the frequency domain, $\omega > \omega_m$.

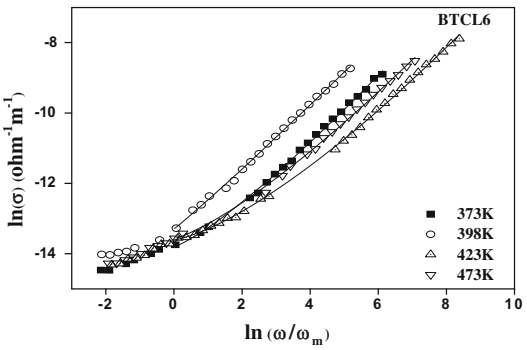
(vi). By drawing conductivity master curves, the time-temperature superposition principle has been verified and temperature-independent relaxation mechanism has been confirmed.

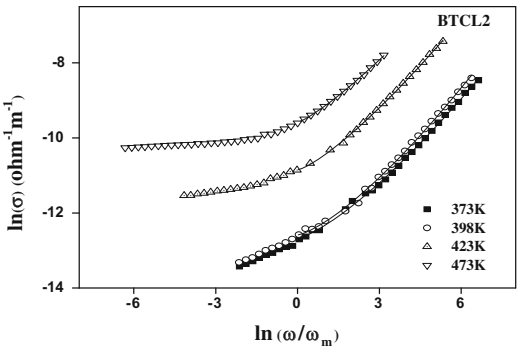
(vii). Relaxation process of AC conduction obeyed Hunt's model for frequencies above ω_m.

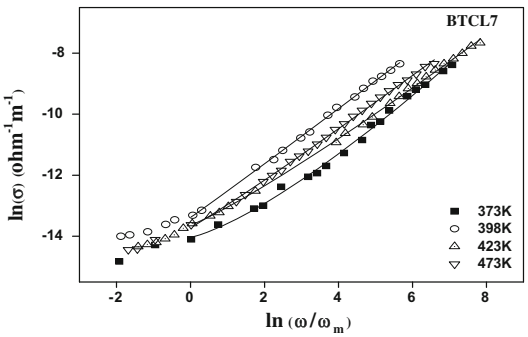
This is for the first time that borotellurite glasses doped with CoO and Li_2O have been exhaustively studied for AC conductivity and dielectric parameters.

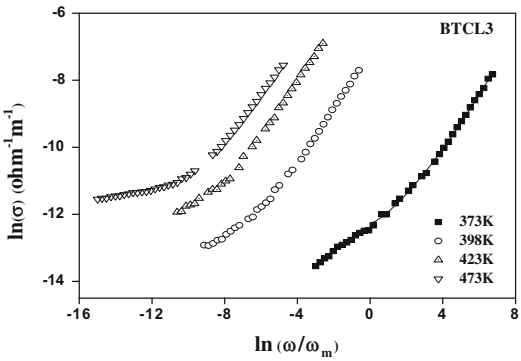
Fig. 11 The plots of AC conductivity, $\ln(\sigma_{AC})$ versus $\ln(\omega/\omega m)$ for ▶ BTCL glasses. The *solid lines* are best fits to the Hunt's Eq. (7)

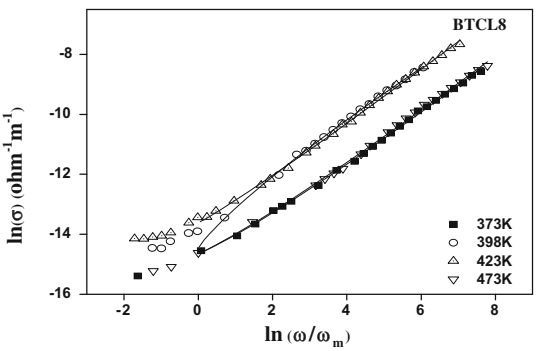

BTCL1
ln(σ) (ohm⁻¹m⁻¹)
ln (ω/ω_m)
373K
398K
423K
473K

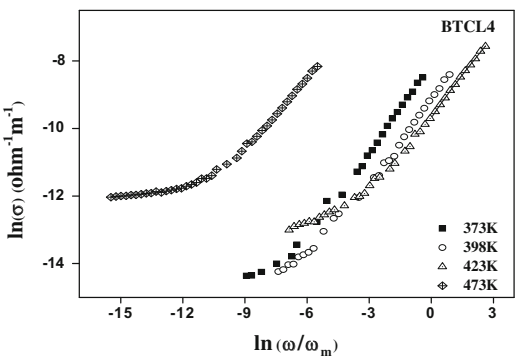

BTCL6
ln(σ) (ohm⁻¹m⁻¹)
ln (ω/ω_m)
373K
398K
423K
473K

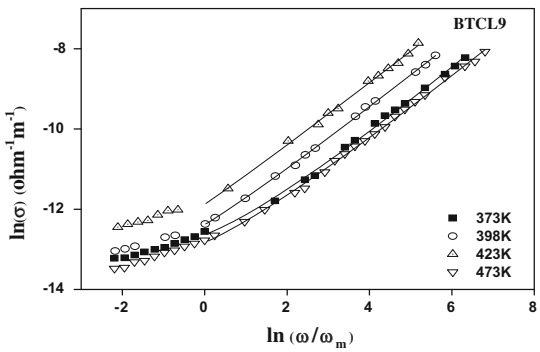

BTCL2
ln(σ) (ohm⁻¹m⁻¹)
ln (ω/ω_m)
373K
398K
423K
473K

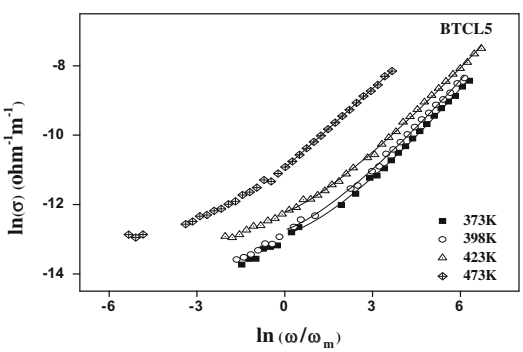

BTCL7
ln(σ) (ohm⁻¹m⁻¹)
ln (ω/ω_m)
373K
398K
423K
473K

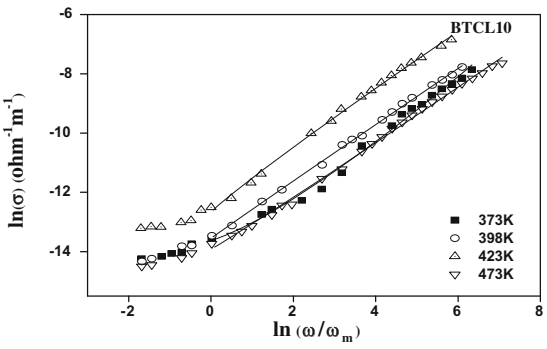

BTCL3
ln(σ) (ohm⁻¹m⁻¹)
ln (ω/ω_m)
373K
398K
423K
473K


BTCL8
ln(σ) (ohm⁻¹m⁻¹)
ln (ω/ω_m)
373K
398K
423K
473K


BTCL4
ln(σ) (ohm⁻¹m⁻¹)
ln (ω/ω_m)
373K
398K
423K
473K


BTCL9
ln(σ) (ohm⁻¹m⁻¹)
ln (ω/ω_m)
373K
398K
423K
473K


BTCL5
ln(σ) (ohm⁻¹m⁻¹)
ln (ω/ω_m)
373K
398K
423K
473K


BTCL10
ln(σ) (ohm⁻¹m⁻¹)
ln (ω/ω_m)
373K
398K
423K
473K

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