Electrical Conductivity, Viscosity, and Sound Velocity of Lithium Bromide + Lithium Nitrate + Acetamide Melt Systems

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Densities, electrical conductivities, viscosities, and sound velocities of $0.22[x\text{LiBr} + (1 - x)\text{LiNO}_3] + 0.78\text{CH}_3\text{CONH}_2$ were measured as functions of temperature ($296.75 \leq T/\text{K} \leq 343.15$) and composition ($x = 0.0-1.0$ mole fraction). Electrical conductivity, viscosity, and structural relaxation time were found to have non-Arrhenius temperature dependence and were analyzed by using the Vogel–Tamman–Fulcher equation. A significant mixed anion effect has been found in the electrical conductivity isotherms. Viscosity and structural relaxation time vary linearly with composition. The variation of electrical conductivity is governed by the mobility of Br^- and NO_3^- ions and the competitive polarization exerted by anions on Li^+, whereas the polymeric and rigid structure formed between Li^+ and acetamide primarily govern the viscosity process.

Introduction

The most common deviation from the linearity in the electrical conductivity and viscosity isotherms has been reported for the mixed alkali systems (Day, 1976; Dietzel, 1983; Ingram, 1987) when one alkali ion is progressively replaced by another alkali ion in ternary systems. The magnitude of deviation from the additivity in viscosity and conductivity isotherms in oxide melt media (Day, 1976) is manyfold in comparison to mixed alkali systems in ionic (Moynihan, 1979; Sangma et al., 1984) or molecular liquid (Gupta and Ismail, 1990, 1991; Mahiuddin, 1994) media. The same phenomenon has also been observed for an alkaline earth metal cation pair (Dietzel, 1983) and for the alkali metal with different anion pair (Chandra and Chandra, 1994; Choi and Kim, 1995). Such deviation may be referred to, with a more general term, as the mixed ion effect.

Unlike the mixed alkali ion effect in conductivity and viscosity isotherms, studies on the mixed anion effect in similar properties are limited (Chandra and Chandra, 1994; Choi and Kim, 1995). Molten acetamide is a good nonaqueous solvent for many inorganic salts due to its dipolar nature (Kerridge, 1988), and molten acetamide–electrolyte binary systems have a tendency to form supercooled liquid (Berchiesi et al., 1983). On the other hand, alkali metal cation forms a polymeric type solvated ion with acetamide (Plowiec et al., 1985; Amico et al., 1987) with lower ionic mobility and high relaxation time. Accordingly we report here the density, electrical conductivity, viscosity, and sound velocity of $0.22[x\text{LiBr} + (1 - x)\text{LiNO}_3] + 0.78\text{CH}_3\text{CONH}_2$ melt systems as functions of temperature and composition.

Experimental Section

Material and Equipment. Both LiNO_3 and LiBr (E. Merck, >99%) were recrystallized twice from the conduc-

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Table 1. Values of Density of the $0.22[x\text{LiBr} + (1 - x)\text{LiNO}_3] + 0.78\text{CH}_3\text{CONH}_2$ Melt as Functions of Temperature and Composition

T/K	$\rho/(\text{kg m}^{-3})$	T/K	$\rho/(\text{kg m}^{-3})$	T/K	$\rho/(\text{kg m}^{-3})$
	$x = 0.0$		$x = 0.2$		$x = 0.4$
324.40	1165.8	325.90	1171.8	330.25	1176.0
322.10	1167.3	323.75	1173.3	328.05	1177.6
320.30	1168.7	321.85	1174.7	326.05	1179.1
318.15	1170.2	319.95	1176.2	323.85	1180.4
315.95	1171.7	318.05	1177.7	321.75	1181.8
313.80	1173.5	315.95	1179.5	319.95	1183.5
311.65	1175.2	313.80	1181.2	317.75	1185.1
309.70	1176.9	311.50	1182.9	315.70	1186.7
307.65	1178.5	309.35	1184.6	313.30	1188.2
305.55	1179.7	307.80	1185.8	311.00	1190.5
303.50	1180.9	306.30	1187.0	308.85	1192.2
301.35	1182.5	304.25	1188.6	306.50	1194.0
299.30	1184.1	302.30	1190.1	304.20	1195.6
				302.00	1197.2
	$x = 0.6$		$x = 0.8$		$x = 1.0$
335.65	1177.9	333.55	1186.5	338.00	1189.9
333.65	1179.5	331.50	1188.1	335.85	1191.4
331.55	1181.0	329.55	1189.6	333.85	1193.1
329.35	1182.3	327.40	1190.9	331.60	1194.6
327.15	1183.7	325.15	1192.3	329.55	1196.2
324.85	1185.4	322.95	1194.0	327.25	1198.3
322.15	1187.0	320.65	1195.6	325.20	1199.9
320.15	1188.6	318.65	1197.3	323.05	1201.5
318.15	1190.2	316.25	1198.8	320.85	1203.0
315.85	1192.4	311.55	1202.8	318.75	1204.4
313.65	1194.1	309.35	1204.6	316.50	1205.8
311.20	1195.9	307.15	1206.2	314.35	1207.5
309.10	1197.5	304.95	1207.9	312.15	1209.1
306.95	1199.2			309.90	1210.8
				307.15	1212.4

tivity water and dried over P_2O_5 in a vacuum desiccator. Details of the purification of acetamide (Loba, >99%), preparation of the samples, and the temperature control unit to maintain the temperature of the measurements to ± 0.02 K are given elsewhere (Mahiuddin, 1996).

Density Measurements. Densities ($\rho/(\text{kg m}^{-3})$) of all the samples were measured as a function of temperature with the help of a calibrated single-stem pycnometer. The

Table 2. Least-Squares-Fitted Values of the Density Equation, $\rho = a - b((T/K) - 273.15)$ for the $0.22[xLiBr + (1 - x)LiNO_3] + 0.78CH_3CONH_2$ Melt

x	a/(kg m^{-3})	b/(kg m^{-3} K^{-1})	std dev in ρ/(kg m^{-3})
0.0	1203.5 ± 0.3	0.7371 ± 0.0078	0.21
0.2	1212.9 ± 0.1	0.7827 ± 0.0030	0.08
0.4	1219.1 ± 0.3	0.7600 ± 0.0071	0.22
0.6	1223.9 ± 0.5	0.7401 ± 0.0103	0.33
0.8	1231.4 ± 0.4	0.7472 ± 0.0080	0.26
1.0	1237.9 ± 0.3	0.7371 ± 0.0068	0.24

Table 3. Electrical Conductivities (κ) Data for the $0.22[xLiBr + (1 - x)LiNO_3] + 0.78CH_3CONH_2$ Melt

T/K	κ/(S m^{-1})	T/K	κ/(S m^{-1})	T/K	κ/(S m^{-1})
$x = 0.0$		$x = 0.2$		$x = 0.4$	
343.15	0.6724	343.15	0.6698	343.15	0.7250
337.85	0.5535	337.85	0.5618	337.85	0.6308
333.05	0.4686	333.05	0.4746	333.05	0.5321
327.85	0.3859	327.85	0.3872	327.85	0.4327
323.15	0.3198	323.15	0.3157	323.15	0.3554
317.85	0.2559	317.85	0.2493	317.85	0.2801
313.15	0.2057	313.15	0.1978	313.15	0.2218
307.95	0.1564	308.05	0.1499	307.95	0.1674
303.15	0.1102	303.25	0.1111	303.25	0.1424
298.15	0.0759	298.15	0.0794	298.15	0.1004
296.75	0.0683				
$x = 0.6$		$x = 0.8$		$x = 1.0$	
343.15	0.9112	343.15	0.9718	343.15	1.1342
337.85	0.7656	337.75	0.8195	337.85	0.9660
333.05	0.6442	333.05	0.6960	333.05	0.8300
327.85	0.5312	327.85	0.5731	327.85	0.6747
323.15	0.4407	323.15	0.4739	323.15	0.5667
317.85	0.3438	317.85	0.3767	317.85	0.4607
313.15	0.2736	313.15	0.3013	313.15	0.3679
307.95	0.2074	307.95	0.2304	307.95	0.2867
303.25	0.1577	303.25	0.1768	303.15	0.2203
298.15	0.1132	298.15	0.1293	298.15	0.1633
297.35	0.1080				

Table 4. Values of the Viscosity (η) for the $0.22[xLiBr + (1 - x)LiNO_3] + 0.78CH_3CONH_2$ Melt

T/K	η/(mPa s)	T/K	η/(mPa s)	T/K	η/(mPa s)
$x = 0.0$		$x = 0.2$		$x = 0.4$	
343.15	26.54	343.15	24.07	343.15	21.38
337.75	32.84	337.85	29.73	337.75	26.42
333.05	40.18	333.05	36.37	333.05	32.22
327.85	51.06	327.85	46.19	327.85	40.80
323.15	64.32	323.15	58.28	323.15	51.40
317.85	85.27	317.85	77.05	317.85	67.62
313.15	111.6	313.15	101.3	313.15	88.67
307.95	155.7	307.95	140.8	307.95	122.5
303.15	215.3	303.25	191.6	303.15	168.6
298.15	311.8	298.15	283.4	298.15	243.4
$x = 0.6$		$x = 0.8$		$x = 1.0$	
343.15	19.00	343.15	15.47	343.15	15.04
337.85	23.35	337.85	18.89	337.75	18.34
333.05	28.41	333.05	22.79	333.05	22.16
327.85	35.84	327.85	28.50	327.85	27.72
323.15	44.93	323.15	35.41	323.15	34.51
317.85	58.95	317.85	45.90	317.85	44.90
313.15	76.89	313.15	59.18	313.15	57.83
307.95	105.7	307.95	80.12	307.95	78.63
303.25	144.4	303.25	108.0	303.25	106.1
298.15	207.3	298.15	152.4	298.15	150.4

Table 5. Values of Ultrasonic Velocity (u) for the $0.22[xLiBr + (1 - x)LiNO_3] + 0.78CH_3CONH_2$ Melt

T/K	u/(m s^{-1})	T/K	u/(m s^{-1})	T/K	u/(m s^{-1})
$x = 0.0$		$x = 0.2$		$x = 0.4$	
333.15	1578.1	333.15	1572.5	333.05	1579.3
327.95	1589.2	327.85	1583.9	327.95	1590.4
323.15	1600.2	323.15	1593.5	323.15	1600.8
317.95	1611.2	317.95	1605.2	317.95	1611.3
313.25	1621.2	313.15	1616.5	313.15	1621.8
308.05	1633.2	307.95	1627.5	307.95	1632.7
303.25	1644.0	303.25	1637.0	303.25	1643.2
298.05	1654.8	298.25	1648.1	298.15	1653.3
$x = 0.6$		$x = 0.8$		$x = 1.0$	
333.15	1570.0	333.15	1568.3	333.15	1564.1
327.95	1583.2	327.95	1579.2	327.85	1574.7
323.15	1590.4	323.15	1589.1	323.15	1584.2
317.95	1602.4	317.95	1599.9	317.95	1595.3
313.15	1611.6	313.25	1609.9	313.25	1605.1
307.95	1623.0	308.05	1620.9	307.95	1615.0
303.25	1631.6	303.25	1631.1	303.25	1625.0
298.15	1644.4	298.05	1642.1	298.05	1635.7

1991) and the cell constant was found to be 100.3 m^{-1} with a negligible temperature coefficient.

Ultrasonic Velocity. Measurements of ultrasonic velocity (u/(m s^{-1})) in the solutions were made as functions of temperature with an accuracy of ±0.1 m s^{-1} by using a multifrequency ultrasonic interferometer M83 (Mittal Enterprises).

Viscosity Measurements. Viscosities (η/(mPa s)) were measured by using a Schott-Geräte viscometer AVS310 equipped with a Ubbelhode viscometer with a cell constant of 0.3127 mm^2 s^{-2}. The experimental reproducibility between the duplicate viscosity values of all samples was within ±0.4%.

All measurements were made as functions of composition ($x = 0.0-1.0$ mole fraction) and temperature (296.75 ≤ T/K ≤ 343.15).

Results and Discussion

Measured densities (ρ/(kg m^{-3})) for $0.22[xLiBr + (1 - x)LiNO_3] + 0.78CH_3CONH_2$ melt systems as functions of temperature and composition are presented in Table 1 and are found to be a linear function of temperature (Table 2).

Electrical conductivities (κ), viscosities (η), and ultrasonic velocities (u) of the present systems are presented in Tables 3–5, respectively.

The temperature dependence of conductivity and viscosity was observed to be non-Arrhenius ($\sim$7–14% and $\sim$3% deviation from the linearity in Arrhenius plot, respectively). For such a case, the Vogel–Tamman–Fulcher (VTF) equation of the form

$$Y = A_y \exp[\pm B_y/(T - T_{0y})] \qquad (1)$$

is generally used to explain the temperature dependence of the transport properties, and this is again with the present system. In eq 1, Y refers to either conductivity or viscosity, A_y and B_y are constants, and T_{0y} is the ideal glass transition temperature. The B_y parameter is negative for the electrical conductivity. The computed values of the parameters in eq 1 are listed in Table 6. Values of T_0 for electrical conductivity are higher than for viscosity at all compositions. For the $0.22LiNO_3 + 0.78CH_3CONH_2$ binary melt system at $x = 0.0$ the computed T_{0y} values are reasonably comparable within $\sim$20–35 K with the reported T_g value for the $0.2LiNO_3 + 0.8CH_3CONH_2$ melt system (McManis et al., 1985) and justifies our data fitting.

Variations of T_{0y} with mole faction, x, are illustrated in Figure 1. It is apparent that T_{0y} for the present systems

reproducibility of the densities of individual samples was within ±0.01%.

Electrical Conductivity. Electrical conductivities (κ/(S m^{-1})) of the samples were measured by using a precision component analyzer 6425 (Wayne Kerr) operating in the (2.5×10^{-9} to 10) S range with a sensitivity of ±0.01 nS and a platinized platinum electrode. The conductivity cell was calibrated with 0.1 mol kg^{-1} KCl solution (Wu et al.,

Table 6. Least-Squares-Fitted Values of the Parameters of Equation 1 for the 0.22[xLiBr + (1 − x)LiNO$_3$] + 0.78CH$_3$CONH$_2$ Melt^a

x	A_κ/(S m^{-1}) (A_η/(mPa s))	B_y/K	T_{0y}/K	std dev in ln Y
0.0	50.82 ± 8.60 (0.0819 ± 0.0106)	586.7 ± 25.9 (870.6 ± 30.8)	207.5 ± 2.1 (192.5 ± 2.3)	0.033 (0.003)
0.2	52.80 ± 6.17 (0.0817 ± 0.0095)	599.0 ± 27.7 (845.7 ± 27.1)	206.0 ± 2.6 (194.4 ± 2.0)	0.004 (0.004)
0.4	43.41 ± 2.40 (0.0674 ± 0.0078)	565.4 ± 12.2 (872.8 ± 27.5)	205.0 ± 1.2 (191.6 ± 2.0)	0.045 (0.002)
0.6	74.96 ± 6.90 (0.0664 ± 0.0052)	618.1 ± 21.9 (856.1 ± 19.0)	203.0 ± 2.0 (191.8 ± 1.4)	0.005 (0.002)
0.8	83.86 ± 9.21 (0.0634 ± 0.0062)	643.9 ± 25.3 (842.1 ± 23.7)	198.7 ± 2.3 (190.0 ± 1.8)	0.003 (0.003)
1.0	84.68 ± 9.40 (0.0625 ± 0.0090)	626.5 ± 28.1 (834.3 ± 34.0)	197.9 ± 2.7 (191.0 ± 2.6)	0.007 (0.004)

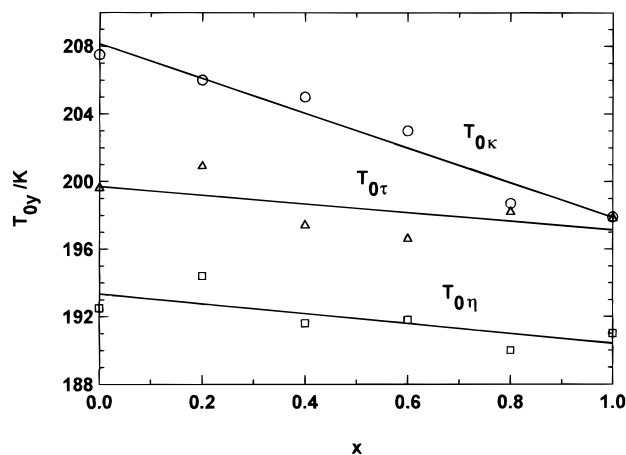^{a} Values for the viscosity are given in parentheses.

Figure 1. Plots of $T_{0\kappa}$ (◯), $T_{0\tau}$ (△), and $T_{0\eta}$ (□) versus mole fraction, x, for 0.22[xLiBr + (1 − x)LiNO$_3$] + 0.78CH$_3$CONH$_2$ melt systems. Symbols and solid lines represent observed and calculated (from linear equation) values, respectively.
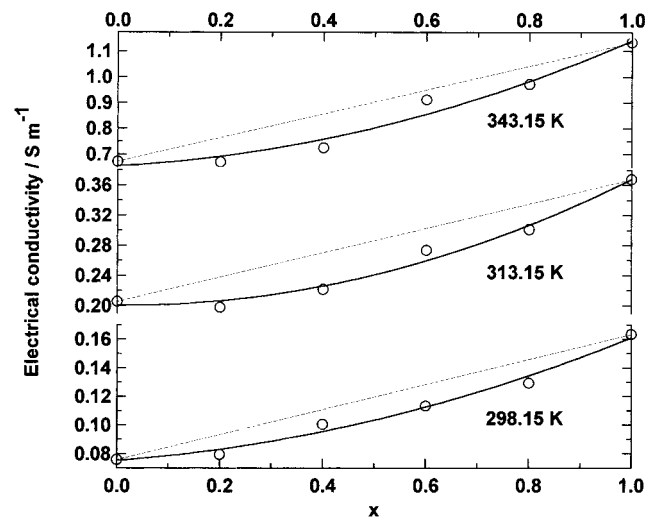

Figure 2. Variation of electrical conductivity, κ, isotherms with mole fraction, x, at different temperatures for the 0.22[xLiBr + (1 − x)LiNO$_3$] + 0.78CH$_3$CONH$_2$ melt systems. Open circles and solid curves are observed and calculated (from the polynomial equation up to the second degree) values and broken lines represent the additive property, respectively.

Figure 3. Variation of viscosity, η, with mole fraction, x, at different temperatures for 0.22[xLiBr + (1 − x)LiNO$_3$] + 0.78CH$_3$CONH$_2$ melt systems. Open circles and solid curves are observed and calculated (from linear equation) values, respectively.

Figure 4. Variation of structural relaxation time, τ, with mole fraction, x, at different temperatures for 0.22[xLiBr + (1 − x)LiNO$_3$] + 0.78CH$_3$CONH$_2$ melt systems. Open circles and solid lines represent observed and calculated (from linear equation) values, respectively.

decreases linearly as LiNO$_3$ is progressively replaced by LiBr. Similar variation in T_{0y} vs x has been reported for the mixed alkali ion and mixed anion systems in ionic melt and/or in aqueous media (Sangma et al., 1984; Kalita et al., 1998a, 1998b).

To examine the existence of the mixed anion effect in the present systems, we have illustrated the variation of electrical conductivity and viscosity with composition at different temperatures in Figures 2 and 3, respectively. From Figure 2 it is apparent that the conductivity iso-
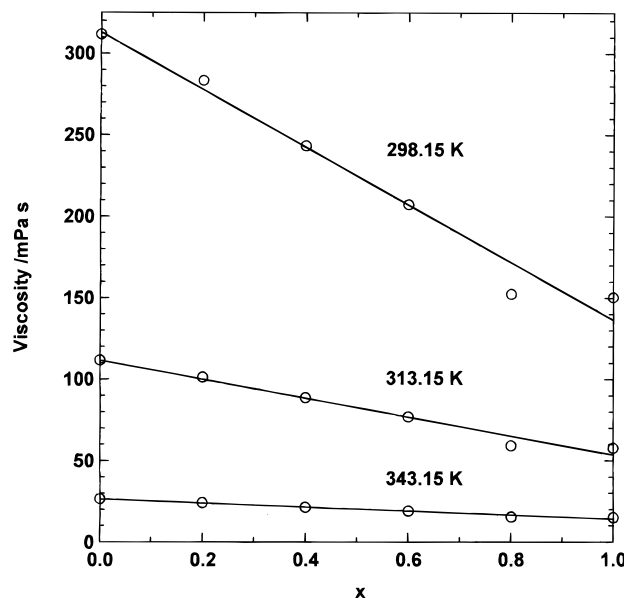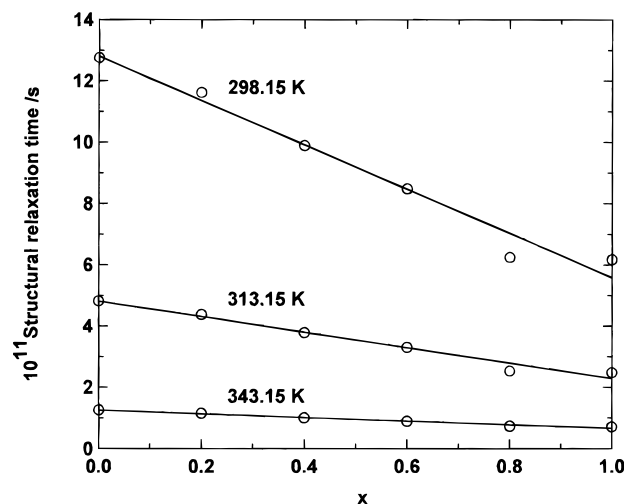
therms showed significant negative deviation (∼11−15%) and may be considered as the onset of the mixed anion effect, whereas viscosity isotherms (Figure 3) do not show any detectable mixed anion effect as lithium nitrate is progressively replaced by lithium bromide. In our earlier mixed Na/K thiocyanates in acetamide medium (Mahiud-din, 1996; Kalita et al., 1998c) we reported both positive

Table 7. Values of the Parameters in Equation 3 for the 0.22[xLiBr + (1 − x)LiNO$_3$] + 0.78CH$_3$CONH$_2$ Melt

x	$10^{14}A_\tau$/s	B_τ/K	$T_{0\tau}$/K	std dev in ln τ
0.0	7.976 ± 0.708	727.1 ± 18.6	199.6 ± 1.4	0.001
0.2	7.740 ± 1.462	711.3 ± 38.7	200.9 ± 3.1	0.004
0.4	5.944 ± 0.547	747.3 ± 20.5	197.4 ± 1.6	0.001
0.6	5.515 ± 0.766	745.5 ± 30.3	196.6 ± 2.4	0.003
0.8	6.218 ± 0.952	690.8 ± 39.0	198.2 ± 3.2	0.002
1.0	5.615 ± 0.763	702.6 ± 34.5	197.8 ± 2.8	0.002

(~4.6% in K$^+$ rich region) and negative (~27% in Na$^+$ rich region) deviations in electrical conductivity isotherms and ~27−55% negative deviation in the viscosity isotherms. Plowiec et al. (1985) reported that Na$^+$ in acetamide melt medium form a polymeric type solvated ion. On the other hand, K$^+$ has a structure breaking property and acetamide in the liquid state exhibits a water-like nature (Kerridge, 1988). As a result we have observed deviation in both conductivity and viscosity isotherms (Mahiuddin, 1996; Kalita et al., 1988c).

In the present systems (i.e., a common cation with different anions) unlike mixed alkali system Li$^+$ form a rigid polymeric type solvated ion with low mobility and NO$_3^-$ and Br$^-$ govern the conductivity process. Br$^-$ has higher ionic mobility in aqueous medium than NO$_3^-$ (Atkins, 1990). Similar inference may also be drawn in liquid acetamide medium. Accordingly we observed higher conductivity for 0.22LiBr + 0.78CH$_3$CONH$_2$ melt in the Br$^-$ rich region at $x = 1.0$ and lower conductivity for 0.22LiNO$_3$ + 0.78CH$_3$CONH$_2$ melt in the NO$_3^-$ rich region at $x = 0.0$. Br$^-$ has a slightly higher ionic radius than NO$_3^-$ (Marcus, 1985), and there will be competitive polarization between NO$_3^-$ and Br$^-$ on Li$^+$, resulting in the net deviation in the conductivity isotherms. In the case of viscosity it is the rigid polymeric aggregate between Li$^+$ and acetamide which predominantly governs the viscosity process.

To understand the structure of the system, we have calculated the structural relaxation time, τ, using the values of the ultrasonic velocity (Table 5) from the following relation (Bender and Pecora, 1986)

$$\tau = 4\eta\kappa_s/3 = 4\eta/3\rho u^2 \tag{2}$$

where η is the viscosity, κ_s is the isentropic compressibility, u is the ultrasonic velocity, and ρ is the density of the systems. The structural relaxation time, τ, at a particular temperature decreases linearly with mole fraction, x (Figure 4). The system possesses a higher τ value when the system is predominantly rich in NO$_3^-$ at $x = 0.0$ and it is lowest in the Br$^-$ rich region at $x = 1.0$.

The structural relaxation time, τ, is found to have a non-Arrhenius temperature dependence, and the following equation, similar to eq 1,

$$\tau = A_\tau \exp[B_\tau/(T - T_{0\tau})] \tag{3}$$

was used to analyze the temperature dependence of the relaxation time. In eq 3, A_τ and B_τ are the constant parameters and $T_{0\tau}$ is the ideal glass transition temperature. The computed values of the parameters in eq 3 are presented in Table 7. From Table 7 and Figure 1 it is apparent that the calculated $T_{0\tau}$ obtained by using eq 3 for the present systems are reasonably comparable with the ideal glass transition temperatures obtained for the electrical conductivity and viscosity (Table 6 and Figure 1).

From the variation of τ vs x (Figure 4) at different temperatures for the present systems, it is apparent that the structural relaxation time decreases linearly with the progressive replacement of LiNO$_3$ by LiBr. The higher structural relaxation time in the NO$_3^-$ rich region ($x = 0.0$) than in the Br$^-$ rich region ($x = 1.0$) may be due to the fact that NO$_3^-$ takes more time to hop or to rearrange in the less available free volume/hole and relax more in comparison to Br$^-$.

With the results in hand it may be concluded that the lower conductivity (Figure 2) and higher viscosity (Figure 3) at $x = 0.0$ for the 0.22LiNO$_3$ + 0.78CH$_3$CONH$_2$ binary melt may be due to the higher structural relaxation time and lower ionic mobility of NO$_3^-$ and the solvated polymeric type ion formed by Li$^+$. At the extreme end of the composition ($x = 1.0$) the higher conductivity and lower viscosity are governed by the higher ionic mobility of Br$^-$ and lower structural relaxation time (Figure 4) of the systems. At the intermediate composition ($0.0 < x < 1.0$) when both NO$_3^-$ and Br$^-$ are present in different proportions, there is a competition between NO$_3^-$ and Br$^-$ to polarize Li$^+$. Br$^-$ has slightly higher ionic radius than the NO$_3^-$ (Marcus, 1985) and will polarize Li$^+$ relatively more. As a result ionic mobility of Br$^-$ is affected, and on progressive replacement of NO$_3^-$ by Br$^-$ there is net negative deviation in the intermediate composition ($0.0 < x < 1.0$, Figure 2), whereas the viscosity of the system is governed by the structure of the system primarily by the solvated polymeric type structure formed between Li$^+$ and liquid acetamide. The structural relaxation time varies linearly (Figure 4), resulting in the linear variation of viscosity isotherms with composition (Figure 3).

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