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Physical Properties of Aqueous Mixtures of Acetamide-LiCl Eutectic Ionic Liquids as a Function of Temperature and Composition

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The density, conductivity and viscosity of aqueous solutions of acetamide-LiCl eutectic ionic liquid were investigated at atmospheric pressure and temperatures from (303.15 to 343.15) K and within the whole composition range. The density values decrease linearly with the increase of the mass content of water and the data decrease with increasing temperature. The mole volume (V_m) values were calculated from the experimental results. The perfect linear relationship between the V_m values and the mole fraction of water indicates that the studied mixtures are ideal solution. The viscosity values decrease sharply with the increase of the mass fraction of water in ionic liquid rich region and then tend to decrease gently in water rich region. The relationship between reciprocal of viscosity ($1/\eta$) and the mass fraction of water can be well fitted with a second-order polynomial equation. With the increase mole fraction of water, the conductivity values of the solution increase gradually first and then decrease dramatically and the highest conductivity values appear at about 0.940-0.958 mole fraction of water. The relationship between the conductivity and the mole fraction of water can be well described by a Castell-Amis equation.

Keywords: Acetamide-LiCl, Eutectic ionic liquid, Water, property.

INTRODUCTION

Eutectic ionic liquid (EIL), also called deep eutectic solvent (DES) or ionic liquid analogue (ILA), is a type of ionic solvent prepared from salts and donor molecules[1]. In 2003, the deep eutectic phenomenon was first described for a mixture of an organic salt-choline chloride (2-hydroxyethyltrimethyl-ammonium chloride) and a hydrogen bond donor compound-urea (CHCl:Urea) in a 1:2 mole ratio by Abbott *et al.*[2], the melting point of the eutectic mixture is 12 °C, which is much lower than either of the individual components. Compared with traditional ionic liquids, this eutectic ionic liquid is low-toxic, easily prepared from industrial bulk materials and sometimes biodegradable, which make it more and more attractive in the area of electrochemistry, material preparation, organic synthesis and isolation process. Except for organic salts, inorganic metal salts (or their hydrate) were confirmed to be effective components of eutectic ionic liquid[3] and the hydrogen bond donors can also be selected from amines[4], carboxylic acids[5], amides[6], alcohol[3] or phenol[3]. Among which, the acetamide based eutectic ionic liquids have received especial attention. Acetamide forms eutectic mixture with various compounds for its high solubility with a large number of organic or inorganic compounds[7] and the physicochemical properties of acetamide based eutectics resemble that of the properties of water solution for the "water-like" acid-base properties of acetamide. Table-1 showed the acetamide-inorganic salts based binary or ternary melts which are liquid at room temperature, the high density than water, the high conductivity, the moderate viscosity and tunable liquid range provide these systems kinship to the traditional room temperature ionic liquids.

It is firmly believed that the exquisite acetamide based eutectic ionic liquids, with the mutual properties of water and ionic liquids, must have broad application value as green solvents in reaction and separation engineering other than electrochemistry field[16]. Our recent research interest has been focused on the high efficiency absorption of sulfur dioxide (SO_2) in ionic liquids[8]. In our study, several acetamide based eutectic ionic liquids, such as acetamide-KSCN, Acetamide-NH_4SCN, acetamide-NaSCN, acetamide-$ZnCl_2$ and acetamide-LiCl were prepared and firstly used as absorbents in SO_2 absorption. They were confirmed to be promising alternative SO_2 absorbent, with the advantages of high efficiency, low cost and ease of recycle[15,17]. The indispensable disadvantage of pure eutectic ionic liquids is their high viscosity, which can be overcome by the adoption of binary mixtures with water[18]. Due to the promising application prospect, it was necessary to characterize them, measure their physical

TABLE-1
TYPICAL ACETAMIDE BASED EUTECTIC IONIC LIQUIDS AND THEIR PROPERTIES

EILs	Composition	ρ (g/cm³), 25 °C	mp (°C)	Viscosity cp 25 °C	Conductivity 25 °C (ms/cm)	[Ref.]
Acetamide:$ZnCl_2$	4:1 mol %	1.36	-16	~500	0.18	3
Acetamide:Urea:ammounium nitrate	27.4:28:34.6 wt %	1.18	7.5	78	6.3	8
Acetamide:$LiPF_6$	5.5:1 mol %	-	-52	~70[c]	~1.2	9
Acetamide:$Ca(NO_3)_2 \cdot 4.37H_2O$	0.7409:0.2591 mol %	1.35[b]	<-20	98.74	4.091	10
Acetamide:LiBETI	4:1 mol %	-	-57	222.4	1.27 (6:1 mol %, 30 °C)	11
Acetamide: $Mg(ClO_4)_2$	0.83:0.17 mol %	1.36	-75[a]	53	~6	12
Acetamide: $Zn(ClO_4)_2$	0.8:0.2 mol %	-	-78[a]	54	6.3	13
Acetamide: $LiTf_2N$	0.8:0.2 mol %		-67	99.56	1.07	14
Acetamide:KSCN	0.75:0.25 mol %	1.207	5	84.2[c]	3.11[c]	15

[a]glass transition temperature, [b]at 27.5 °C, c at 30 °C

properties and summarize the changing rule for designing new eutectic ionic liquids and developing their application in the actual processes. Unfortunately, the properties of deep eutectic solvent and their aqueous solutions are rather scarce[19-21], related work is still subject to a wide study. In this paper, the density, conductivity and viscosity of acetamide- LiCl itself and its aqueous solutions were systematically discussed, at atmospheric pressure and temperatures from (303.15 to 343.15) K.

EXPERIMENTAL

Acetamide/LiCl eutectic ionic liquids were prepared according to literature method[15]. Acetamide and LiCl with the minimum content of 98.5 %, 99.0 %, respectively, were purchased from Modern Instruments Chemical Reagents co., LTD, Shijiazhuang, China. Ultrapure water (with a conductivity ≤ 0.1 us/cm at 298.15 K) was used and prepared in our lab using UPH-IV-10T ultrapure water Purifier System (Chengdu Ultrapure Science and Tecnnology co., LTD). The water content of pure eutectic ionic liquid was less than 0.1 % (determined by Karl Fischer titration within ± 3 % accuracy). The eutectic ionic liquid solutions were prepared by mixing accurately weighed eutectic ionic liquid and water at room temperature. The density, viscosity and conductivity of the eutectic ionic liquids were measured in a water bath with a temperature control accuracy of ± 0.01 K. The density was determined by pycnometer method and the pycnometer was calibrated by anhydrous ethanol or water. The weight was measured by an analysis balance with ± 0.0001 g accuracy (Shanghai Precision and Scientific Sky Beautiful Instrument Co. LTD). The conductivity was measured by DDS-11A conductivity detector (Leici Xinjing instrument limited company, Shanghai, China). The standard KCl solution with c = 0.01 mol L^{-1} was used for calibrating the conductivity detector. The viscosity was detected by Pinkevitch method according to GB/T10247-2008. Each measurement was done in at least 3 runs and the average value was calculated for the further study. The density and viscosity of pure water used in this study were taken from literature[15]. Uncertainties of density, viscosity and conductivity were ± 0.001, ± 0.2 and ± 0.2 %, respectively.

RESULTS AND DISCUSSION

Density of acetamide- LiCl (3:1) aqueous solution: The densities were determined at atmospheric pressure and over the temperature range from 303.15 to 343.15 K. The results were listed in Table-2 and more visually presented in Fig. 1. It can be seen from Fig. 1 that at the same water proportion, the density values always decrease with an increase of temperature. While at the same temperature, the density values decrease with the increase of the mass content of water. This means the physical properties of eutectic ionic liquids can be adjusted by adding a certain amount of water to meet the special application for the hydrophilic eutectic ionic liquids. From the experimental density data, the molecular volume V_m of acetamide-LiCl (3:1) aqueous solutions at several temperatures were calculated from the density data by eqn. 1; where M_1 and M_2 are the molar mass of water and the eutectic ionic liquid, X_1 is the molar fraction of water in the mixture. ρ is the density of the binary mixtures. As is shown in Table-3 and Fig. 2, molar volume V_m depends linearly with the molar fraction of water in the mixtures (eqn. 2), the fitted parameters of eqn. 2 and the correlation coefficients were listed in Table-4. The gradients increase with the increase of temperature, which reflected that V_m is more sensitive at elevated temperature. The perfect linear relationship between V_m and the mole fraction of water indicates the studied mixtures are ideal solution; similar results have been described for imidazolium ionic liquids by Wang *et al.*[22].

$$V_m = \frac{x_1M_1 + (1-x_1)M_2}{\rho} \quad (1)$$

$$V_m = a + bX \quad (2)$$

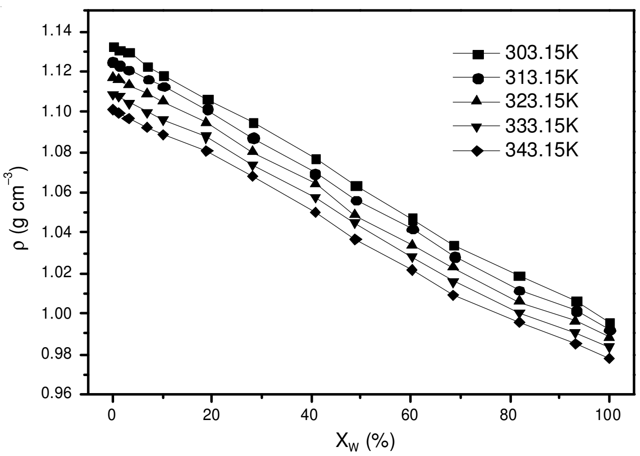

Fig. 1. Density of [water (1) + [acetamide-LiCl] (2)] mixtures at different mass fraction of water

TABLE-2
DENSITY (ρ) OF [WATER (1) + [ACETAMIDE-LiCl] (2)] MIXTURES AT SEVERAL TEMPERATURES

Mass fraction of water in mixture	Mole fraction of water in mixture	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
0.00	0.000	1.1321	1.1248	1.1171	1.1089	1.1010
1.33	0.122	1.1306	1.1233	1.1158	1.1076	1.0997
3.20	0.255	1.1294	1.1208	1.1131	1.1044	1.0968
7.00	0.438	1.1225	1.1163	1.1092	1.1001	1.0926
10.17	0.539	1.1184	1.1127	1.1050	1.0962	1.0883
18.98	0.708	1.1068	1.1012	1.0945	1.0876	1.0803
28.24	0.803	1.0949	1.0871	1.0801	1.0736	1.0677
40.80	0.877	1.0770	1.0698	1.0643	1.0578	1.0504
48.90	0.908	1.0636	1.0564	1.0488	1.0450	1.0366
60.35	0.940	1.0470	1.0422	1.0337	1.0281	1.0216
68.70	0.958	1.0339	1.0286	1.0228	1.0160	1.0089
81.90	0.979	1.0191	1.0118	1.0059	1.0006	0.9957
93.30	0.993	1.0066	1.0017	0.9962	0.9908	0.9850
100.00	1.000	0.9957	0.9922	0.9881	0.9832	0.9778

TABLE-3
MOLAR VOLUME (V_m) OF [WATER (1) + [ACETAMIDE-LiCl] (2)] MIXTURES AT SEVERAL TEMPERATURES

Mole fraction of water in mixture	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
0.000	164.508	165.576	166.717	167.950	169.155
0.122	146.528	147.481	148.472	149.571	150.646
0.255	126.963	127.941	128.821	129.837	130.731
0.438	100.332	100.890	101.539	102.374	103.075
0.539	85.412	85.852	86.443	87.137	87.777
0.708	60.698	61.002	61.379	61.765	62.183
0.803	46.773	47.110	47.417	47.700	47.964
0.877	35.953	36.193	36.379	36.603	36.862
0.908	31.458	31.672	31.902	32.018	32.277
0.940	26.806	26.930	27.151	27.299	27.475
0.958	24.293	24.416	24.557	24.719	24.895
0.979	21.133	21.286	21.409	21.522	21.629
0.993	19.049	19.142	19.249	19.354	19.468
1.000	18.094	18.158	18.233	18.324	18.425

TABLE-4
FITTED PARAMETERS OF EQUATION 2 AND R^2 FOR MOLAR VOLUME (V_m)
OF (WATER (1) + (ACETAMIDE-LiCl)(2)) MIXTURES AT SEVERAL TEMPERATURES

	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
a	164.39	165.46	166.59	167.87	169.07
b	-146.37	-147.36	-148.38	-149.58	-150.67
R^2	1	1	1	1	1

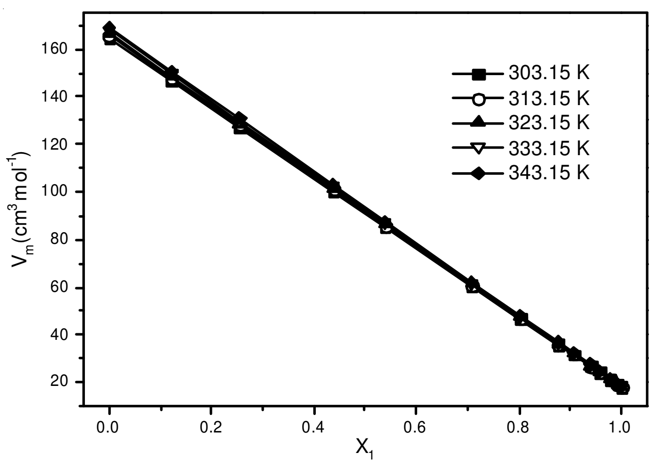

Fig. 2. Plot of molar volume (V_m) obtained from experimental density (ρ) against mole fraction of water for (water (1) + (acetamide-LiCl) (2)) mixtures at several temperatures

Viscosity of acetamide-LiCl aqueous solution: The viscosity of binary mixtures of water (1) + [acetamide-LiCl] (2)] at several temperatures were detected and the results were given in Table-5. It can be seen that the viscosity of pure acetamide-LiCl is rather high, the data is about dozens of times of those data for pure $BMImBF_4$ (cp) or $BMImPF_6$ (cp), at 303.15 K the viscosity values of $BMImBF_4$ and $BMImPF_6$ are 65.2cp and 172.8cp separately[23]. With the addition of water, whose viscosity is 0.80cp at 303.15 K, the viscosity values decrease sharply with the increase of the mass fraction of water in ionic liquid-rich region and then tend to decrease gently in water rich region. The viscosity of an eutectic ionic liquid can be correlated with the molecular-ion interaction. The experimental results indicate that a small addition of water is enough to drastically change the strong intermolecular interactions and to affect the dependence between viscosity and the mass fraction of water. It can also be seen from Table-5 that the viscosity

TABLE-5
VISCOSITY (η) OF THE BINARY MIXTURE OF [WATER (1) + [ACETAMIDE-LiCl] (2)] AT DIFFERENT TEMPERATURES

Mass fraction of water in mixture	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
0.00	7692.31	2166.67	885.55	410.17	240.12
1.33	2726.60	1040.03	420.34	254.19	138.02
3.20	1202.06	535.01	239.16	130.55	73.17
7.00	534.21	260.44	146.95	83.37	49.75
10.17	297.25	132.27	75.32	39.93	26.72
18.98	61.14	27.30	17.46	11.70	8.27
28.24	12.79	8.89	6.53	4.80	3.64
40.80	6.03	4.44	3.30	2.86	2.53
48.90	3.81	2.85	2.21	1.81	1.62
60.35	2.55	2.00	1.36	1.11	1.10
68.70	1.79	1.42	1.11	0.86	0.87
81.90	1.11	0.89	1.10	0.63	0.65
93.30	0.89	0.72	0.62	0.52	0.55
100.00	0.80	0.65	0.55	0.47	0.40

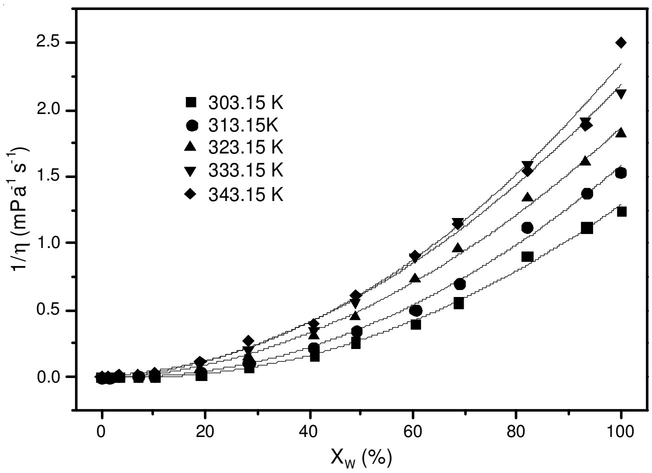

Fig. 3. Plot of reciprocal viscosity 1/η against mass fraction of water for (water (1) + (acetamide-LiCl) (2)) mixtures at different temperatures

values decrease with the increase of temperature but they also seem to be less affected by the presence of water due to the weakening of the H-bond interactions. The relationship between reciprocal of viscosity (1/η) (Table-6) and the mass fraction of water can be well fitted with a second-order polynomial equation (eqn. 3), the curve was shown in Fig. 3. Where η is the viscosity of the mixture and A, B, C were fitted parameters and x is the mass fraction of water in mixture. Fitted parameters of eqn. 3 and R^2 had been listed in Table-7. With the empirical equation, the viscosity value of a mixture can be accurately predicted and the water content can be calculated by the viscosity detection.

$$1/\eta = Ax^2 + Bx + C \quad (3)$$

Conductivity of acetamide-LiCl aqueous solution: The conductivity of the mixtures of [water (1) + [acetamide-LiCl] (2)] at several temperatures are given in Table-8 and more visually presented in Fig. 4. With the increase mole fraction of water, the conductivity of aqueous solutions of acetamide-LiCl (3:1) increase gradually first and then decrease dramatically, similar results had been observed by Vila *et al.*[24] and Zhu *et al.*[25]. They studied the typical imidazolium based ionic liquids. The highest conductivity values appear in the vicinity about 0.940-0.958 mole fraction of water. The maximum conductivity of 74.94 mS/cm at the 0.940 mole fraction of water were observed, which is higher than those of aqueous solution of $BMImBF_4$, whose highest conductivity is 37.81 mS/cm at the 0.9007 mole fraction of water. On the other hand, the increase in the conductivity from the value of the pure EIl to the value at the maximum is several hundred times for [water (1) + [acetamide-LiCl] (2)], while only 15 times for $BMImBF_4$[25]. The wide range of electrical conductivity means broad application prospects of these eutectic ionic liquids. The conductivity of eutectic ionic liquid aqueous solutions increases with

TABLE-6
RECIPROCAL VISCOSITY (1/η) OF THE BINARY MIXTURE OF
[WATER (1) + [ACETAMIDE-LiCl] (2)] AT DIFFERENT TEMPERATURES

Mass fraction of water in mixture	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
0.00	0.0001	0.0005	0.0011	0.0024	0.0042
1.33	0.0004	0.0010	0.0024	0.0039	0.0072
3.20	0.0008	0.0019	0.0042	0.0077	0.0137
7.00	0.0019	0.0038	0.0068	0.0120	0.0189
10.17	0.0034	0.0076	0.0133	0.0250	0.0374
18.98	0.0164	0.0366	0.0573	0.0854	0.1210
28.24	0.0782	0.1125	0.1532	0.2084	0.2745
40.80	0.1659	0.2253	0.3026	0.3499	0.3958
48.90	0.2623	0.3505	0.4525	0.5532	0.6158
60.35	0.3927	0.5010	0.7353	0.9009	0.9091
68.70	0.5589	0.7047	0.9638	1.1609	1.1508
81.90	0.9026	1.1211	1.3416	1.5813	1.5373
93.30	1.1251	1.3828	1.6132	1.9103	1.8198
100.00	1.2500	1.5385	1.8182	2.1277	2.5000

TABLE-7
FITTED PARAMETERS OF EQUATION 3 AND R^2 FOR RECIPROCAL VISCOSITY (1/η) OF (WATER (1) + (ACETAMIDE- LiCl) (2)) MIXTURES AT DIFFERENT TEMPERATURES

T (K)	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
A (10^{-3})	-1.85	-1.32	1.92	3.2	1.04
B (10^{-4})	1.47	1.71	1.69	1.89	2.23
C (10^{-3})	3.26	2.23	-15.8	-18.63	11.62
R^2	0.997	0.997	0.997	0.996	0.992

TABLE-8
CONDUCTIVITY OF THE BINARY MIXTURE OF [WATER (1) + [ACETAMIDE-LiCl] (2)] AT DIFFERENT TEMPERATURES

Mole fraction of water in mixture	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
0.000	0.09	0.20	0.46	0.82	1.26
0.122	0.13	0.31	0.65	1.17	1.84
0.255	0.21	0.48	0.92	1.69	2.87
0.438	0.57	1.16	2.05	3.33	4.96
0.539	1.17	2.18	3.51	5.33	7.73
0.708	4.92	7.42	10.55	12.74	16.05
0.803	13.45	18.55	24.87	31.39	38.25
0.877	25.25	32.48	40.42	48.60	56.88
0.908	32.34	41.84	50.16	59.15	68.04
0.940	39.15	47.66	56.88	65.72	74.94
0.958	40.33	48.18	56.41	64.87	73.38
0.979	32.43	36.31	39.50	42.90	46.20
0.993	22.88	27.47	31.68	35.55	39.13
1.000	0.00	0.00	0.00	0.00	0.00

TABLE-9
FITTED PARAMETERS OF EQUATION 4 AND σ FOR CONDUCTIVITY (κ) OF (WATER (1) + (ACETAMIDE- LiCl) (2)) MIXTURES AT DIFFERENT TEMPERATURES

Temperatures (k)	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K
k_{max}	40.33	48.18	56.88	65.72	74.94
x_{max}	0.964	0.964	0.949	0.949	0.949
a	0.509	0.444	0.642	0.643	0.631
b	8.239	6.764	5.270	6.851	1.279
σ	2.037	3.407	2.331	3.369	6.491

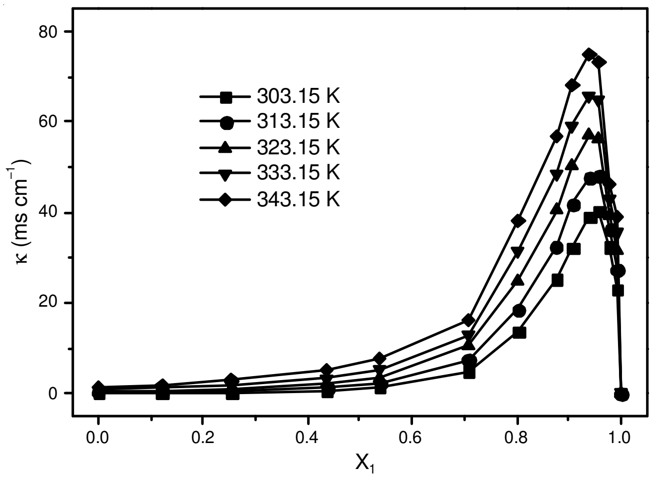

Fig. 4. Plot of conductivity (κ) against mole fraction for the system of (water (1) + (acetamide-LiCl) (2)) at different temperatures

an increase of temperature at whole concentration ranged, that is because the viscosity of the eutectic ionic liquid decreases sharply and the ions move faster with increasing temperature. The relationship between the conductivity and the mole fraction of water can be well described by a Castell-Amis equation (eqn. 4). Where κ is the electrical conductivity, κ_{max} is the maximum value of the electrical conductivity of the solution at the mole fraction scale x_{max}, x_1 is the mole fraction of water, a and b are fitting constants. The obtained parameters of κ_{max}, x_{max}, a and b are summarized in Table-9 together with the standard deviations (s, calculated according to eqn. 5).

$$\kappa = \kappa_{max}\left(\frac{1-x_1}{1-x_{max}}\right)^a \exp\left[b(x_{max}-x_1)^2 - \frac{a}{1-x_{max}}(x_{max}-x_1)\right] \quad (4)$$

$$\sigma = \left[\sum_{1}^{n_{dat}}\left(z_{exp}-z_{cal}\right)^2 / n_{dat}\right]^{1/2} \quad (5)$$

Here Z_{exp}, Z_{cal} and n_{dat} present the experimental value, calculated value by eqn. 4 and the number of experimental data, respectively.

Conclusion

The effects of water on the density, viscosity and conductivity of acetamide- LiCl eutectic ionic liquid were reported at temperature range from 303.15 to 343.15 K. The density and viscosity values decrease monotonically when the mole fraction of water increases in the whole composition range and the data decrease with increase of temperature. From the experimental values, the molecular volume V_m of acetamide-

LiCl (3:1) solution was calculated and the relationship between V_m and the mole fraction of water can be described by a linear equation. The dependence of reciprocal of viscosity (1/η) on the mass fraction of water has been fitted with a second-order polynomial equation. The relationship between the conductivity and the mole fraction of water can be described by a Castell-Amis equation. It is expected that the data and the obtained equation will be useful for the application of eutectic ionic liquid in the industrial process.

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