Viscosity and Excess Molar Volume of Binary Ionic Liquid Mixture at Temperature 298.15 K

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Abstract- Densities and viscosities of 1-butyl-3-methylimidazolium tetrafluoroborate [Bmim][BF4] ionic liquid with methyl formate, methyl acetate, ethyl formate and acetone were measured over the whole concentration range at 298.15 K and atmospheric pressure. Viscosity were also computed from Grunberg-Nissan and Kendall and Monroe models and compared with the experimental results. The excess molar volume V^E and the viscosity deviations $\Delta \eta$ for the binary mixture have been calculated and were fitted to the Redlich-Kister polynomials. A fair agreement was achieved when compared the experimental viscosities with the theoretical viscosities and they are within the experimental limits. Interactions involved in the liquid mixture were studied from the values of V^E and $\Delta \eta$ which are negative over the whole composition range. The minimum deviations for excess molar volume and viscosity are observed at about mole fraction of the ionic liquid, $x \approx 0.2$ -0.3 and $x \approx 0.55$ -0.65

Index Terms - Ionic liquid, Grunberg-Nissan, Kendall-Monroe, viscosity and excess molar volume

1. INTRODUCTION

Ionic liquids (ILs) are entirely constituted by ions with melting points below 100°C [1]. Due to the importance and interest of Ionic liquids, they are now being used in all areas of chemistry, as solvents for organic and inorganic synthesis, as electrolytes in batteries and solar cells, as new types of energetic materials, as stationary phases in chromatography, and in a variety of other analytical applications, as well as being the subject of fundamental study in physical chemistry [2]. Physical properties including viscosity and density[4], are essential for designing any process involving ionic liquids on an industrial scale. Moreover, the magnitude and extent of interactions involved in the process can be studied by understanding the excess properties. Ionic liquids study has sufficient scope for further research modelling and correlations [5]. In this work, we measured, density and viscosity of mixtures consisting of methyl formate, methyl acetate, ethyl formate, and acetone with 1-butyl-3methylimidazolium tetrafluoroborate ([Bmim] [BF4]) ionic liquid over the entire composition range at 298.15 K and atmospheric pressure. The studies of excess properties such as deviation in viscosity, excess molar volume, excess Gibbs free energy of activation of viscous flow and Grunberg-Nissan interaction constant of binary mixtures are useful in understanding the nature and extent of intermolecular interactions between two liquids [6-10 8-12]. Deviation in viscosity $(\Delta \eta)$, excess molar volume (V^E) and excess Gibbs free energy of activation of viscous flow (ΔG^{*E})

have been calculated from the density (ρ) , and viscosity (η) , data and the excess molar volume V^E and the viscosity deviations $\Delta \eta$ for the binary mixture have been calculated and were fitted to the Redlich-Kister polynomials. Grunberg-Nissan (G-N) and Kendall-Monroe(K-M) equations with no parameters, have been used in correlating viscosity data of the binary mixtures.

2. EXPERIMENTAL

2.1 Materials

High purity and AR grade samples of methyl formate, methyl acetate, ethyl formate, and acetone used in this experiment were obtained from Merck Co. Inc., Germany and purified by distillation in which the middle fraction was collected. The liquids were stored in dark bottles over 0.4nm molecular sieves to reduce water content and were partially degassed with a vacuum pump. The purity of each compound was checked by gas chromatography and the results indicated that the mole fraction purity was higher than 0.99. All the materials were used without further 1-butyl-3-methylimidazolium purification. tetrafluoroborate, [Bmim][BF4] was purchased from Merck Co. Inc., Germany (CAS No 174501-65-6). Ionic liquid was prepared by mass and mole fraction was calculated. Every precaution was taken to minimize water contamination because trace amounts of water in ILs can affect the physical properties and phase behavior. the ILs were dried under vacuum for (2 to 3) days before use. The purity of chemicals used was confirmed

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by comparing the densities and viscosities with those reported in the literature as shown in table 1.

2.2 Apparatus and Procedure

Before each series of experiments, we calibrated the instrument at atmospheric pressure with doubly distilled water. The calibration was accepted if the measurements were within $\pm 3 \times 10^{-10}$ 4 g.cm⁻³ and 6×10^{-3} m Pa.s of the published values. The densities of the pure components and their mixtures were measured with the bicapillary pyknometer The uncertainty in the density measurements was better than $\pm 3 \times 10^{-4} \text{ g.cm}^{-3}$ and reproducible to \pm 1.31×10⁻² g.cm⁻³ The liquid mixtures were prepared by mass in a air tight stopped bottle using a electronic balance model Shimadzuax-200 accurate to within ±0.1mg.The average uncertainty in the composition of the mixtures was estimated to be less than ± 0.0001 . The viscosities of pure liquids and the mixtures were measured at atmosphere pressure and at different temperatures using Canon Ubbelohde suspended-level viscometer. The viscometer was immersed in a well stirred water bath (Raga Industries) with temperature control to ± 0.01 K. The flow time was recorded with a digital stopwatch capable of measuring time to within ± 0.01 s.Each experiment was repeated four times at each temperature for all composition and results were averaged. The viscosity η of the liquid was then calculated from the following relationship $v = \eta / \rho = k (t-\theta)$ (1)where η is the absolute viscosity is the density is the flow time v is the kinematic viscosity, and k and θ are the viscometer constant and the Hagen Bach correction factor respectively. The calibration of the viscometer was carried out with doubly distilled water and doubly distilled benzene .Care

was taken to reduce evaporation during the measurements. The estimated uncertainty in viscosity measurements was within \pm 0.006 m Pa.s and reproducible to \pm 3.3 m Pa.s.

3. TABLES AND FIGURES

Components	Molar mass, M/ g.mole ⁻¹	Density, ρ/g.cm ⁻³		Viscosity,	, η/mPa.s
		exp		exp	
[Bmim][BF4]	226.02	1.2211	1.208	117.3	114 ²
methyl formate	60.05	0.9657	0.9663 ¹²	0.320	0.328 ¹²
methyl acetate	74.08	0.9162	0.9159 ¹³	0.371	0.379 ¹³
ethyl formate	74.08	0.9294	0.9285 ¹⁴	0.354	0.368 ¹⁴
acetone	58.08	0.7846	0.7850 ¹³	0.310	$0.316^{10^{13}}$

Table 1. Comparison of experimental densities (ρ) and viscosities (η) with literature values

Table 2.	Redlich-Kister coefficients and their Standard Deviations (SD) for the Excess Molar Volumes
	and Viscosity Deviations at 298.15 K

	B ₀	B ₁	B ₂	B ₃	SD					
$V^{E}/cm^{3}mole^{-1}$	-4.53	6.58	-3.18	4.63	0.08					
$\Delta \eta$ /mPa.s	-178.84	106.68	-73.27	44.98	0.30					
	[Bmim][BF4] + Methyl Acetate									
$V^{E}/\mathrm{cm}^{3}\mathrm{mole}^{-1}$	-6.23	7.31	4.78	11.18	0.04					
$\Delta \eta$ mPa.s	-178.81	112.46	-107.94	81.77	0.55					
[Bmim][BF4]+EthylFormate										
V ^E /cm ³ mole ⁻¹	3.74	5.55	3.09	6.46	0.04					
$\Delta \eta$ mPa.s	-179.72	113.80	-68.82	29.68	0.24					
	[Bmim][BF4] + Acetone									
V ^E /cm ³ mole ⁻¹	-6.11	7.11	-3.29	5.19	0.05					

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$\Delta \eta$ /mPa.s	-186.03	127.42	-84.78	51.19	0.16

Table 3. Mole fraction(x), mass fraction(w), experimental density (ρ), experimental and theoretical viscosity ($\eta_{exp}, \eta_{G-N}, \eta_{K-M}$), Excess Molar Volume (V^E), and Viscosity Deviations ($\Delta \eta$) for binary systems at 298.15 K

[Bmim][BF4] + Methyl Formate

x	w/	$\rho/\mathrm{g.cm}^{-3}$	η_{exp}	η _{G-N} /	η _{K-M} /	$V^{E}/$	Δη/
	g		mPa.s	mPa.s	mPa.s	cm ³ mole ⁻¹	mPa.s
0.0521	0.1721	1.1117	0.501	0.712	0.827	-0.905	-5.3
0.1017	0.3001	1.1155	0.912	0.823	1.012	-1.335	-10.5
0.2039	0.4907	1.1214	1.904	2.012	2.216	-1.602	-20.7
0.3001	0.6013	1.1314	3.507	3.715	3.925	-1.632	-29.7
0.3404	0.6594	1.1352	4.416	4.819	5.127	-1.576	-33.2
0.4012	0.7096	1.1486	6.325	6.631	6.934	-1.453	-38.0
0.4502	0.7586	1.1578	8.213	10.134	10.413	-1.337	-41.6
0.5021	0.7902	1.1669	10.614	10.712	11.416	-1.183	-44.7
0.5517	0.8166	1.1722	13.736	13.921	14.213	-1.032	-46.9
0.6012	0.8512	1.1834	17.121	20.512	22.242	-0.841	-49.0
0.7123	0.9013	1.1923	28.302	31.613	34.521	-0.265	-49.0
0.8012	0.9414	1.2036	44.202	46.712	48.413	-0.099	-44.1
0.9017	0.9613	1.2214	68.414	70.431	72.127	-0.019	-30.8

[Bmim][BF4] + Methyl Acetate

0.0521	0.1721	0.9645	0.602	0.727	0.923	-1.477	-5.3
0.1017	0.3001	1.0134	0.912	0.864	1.086	-1.804	-10.5
0.2039	0.4907	1.0481	2.160	2.131	2.492	-2.155	-20.4
0.3001	0.6013	1.0891	3.610	4.013	4.523	-2.166	-30.1
0.3404	0.6594	1.1023	4.719	5.121	6.525	-2.072	-34.2
0.4012	0.7096	1.1413	6.223	6.817	7.625	-1.947	-38.1
0.4502	0.7586	1.1421	8.233	9.234	10.146	-1.786	-41.8
0.5021	0.7902	1.1427	10.227	11.012	11.783	-1.689	-45.1
0.5517	0.8166	1.1489	13.131	14.156	14.989	-1.394	-47.6
0.6012	0.8512	1.1572	16.349	19.645	21.895	-1.144	-50
0.7123	0.9013	1.1724	25.822	27.231	29.134	-0.635	-51.5
0.8012	0.9414	1.1872	40.889	43.141	45.616	-0.194	-47.5
0.9017	0.9613	1.2146	63.764	64.413	65.134	-0.033	-35.6

[Bmim][BF4] + Ethyl Formate

0.0521	0.1721	0.9614	0.613	0.722	0.917	-0.156	-5.3
0.1017	0.3001	0.9722	1.006	0.867	1.064	-0.203	-10.4
0.2039	0.4907	0.9924	2.014	2.232	2.398	-0.839	-20.2
0.3001	0.6013	1.0846	3.823	4.113	4.586	-1.106	-30.1
0.3404	0.6594	1.0996	5.119	5.121	6.723	-1.120	-34.4
0.4012	0.7096	1.1123	6.526	6.847	7.712	-1.091	-38.1
0.4502	0.7586	1.1294	8.507	9.012	10.132	-1.029	-41.6
0.5021	0.7902	1.1386	10.659	10.934	11.384	-0.937	-44.4
0.5517	0.8166	1.1501	13.312	14.101	14.301	-0.817	-46.9
0.6012	0.8512	1.1592	16.927	20.023	23.144	-0.649	-49.3
0.7123	0.9013	1.1732	26.651	27.817	28.012	-0.303	-50.5
0.8012	0.9414	1.1941	43.743	44.521	44.721	-0.057	-44.3
0.9017	0.9613	1.2089	69.079	70.423	71.525	-0.018	-30.3

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0.0521	0.1721	0.8460	0.551	0.601	0.667	-0.958	-5.3
0.1017	0.3001	0.8923	0.716	0.791	0.805	-1.461	-10.6
0.2039	0.4907	0.9737	1.529	1.579	1.601	-1.942	-20.6
0.3001	0.6013	1.0293	3.004	3.132	3.467	-2.033	-30.8
0.3404	0.6594	1.0572	4.072	4.186	4.230	-1.976	-35.5
0.4012	0.7096	1.0741	5.224	5.444	5.526	-1.887	-39.4
0.4502	0.7586	1.0942	6.927	7.103	7.411	-1.616	-43.1
0.5021	0.7902	1.1151	9.035	10.112	11.413	-1.584	-46.0
0.5517	0.8166	1.1267	12.067	13.131	13.567	-1.384	-48.1
0.6012	0.8512	1.1413	15.149	19.899	21.123	-1.169	-51.1
0.7123	0.9013	1.1629	25.517	26.413	26.894	-0.679	-51.5
0.8012	0.9414	1.1852	40.189	41.012	42.613	-0.284	-47.9
0.9017	0.9613	1.2054	64.791	65.147	65.618	-0.131	-34.6

[Bmim][BF4] + Acetone





Fig. 1 Excess molar volumes, V^E for the mixtures of x[Bmim][BF4]+(1-x) organic solutes at 298.15 K. \diamond ; [Bmim][BF4] + MethylFormate, \blacksquare ; [Bmim][BF4] + Methyl Acetate, \blacktriangle ; [Bmim][BF4] + Ethyl Formate, \times ;[Bmim][BF4] + Acetone

Fig. 2 Viscosity deviation, $\Delta \eta$ for the mixtures of x[Bmim][BF4]+(1-x) organic solutes at 298.15 K. \diamond ; [Bmim] [BF4] + MethylFormate, \blacksquare ; [Bmim][BF4] + Methyl Acetate, \blacktriangle ; [Bmim] [BF4] + Ethyl Formate, \times ; [Bmim][BF4] + Acetone

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Fig. 3 Plot of experimental and theoretical viscosities of binary liquid mixture with mole fraction of ionic liquid. ◆;Experimental viscosity, ■;viscosity calculated from Grunberg-Nissan equation and ▲; viscosity calculated from Kendall and Monroe

3. RESULTS AND DISCUSSION

A comparison is made for the pure components ([Bmim][BF4], methyl formate, methyl acetate, ethyl formate and acetone) in Table 1 between the

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experimental density and viscosity determined in this work and those reported in the literature. It can be seen that the values for [Bmim][BF4] and organic solvents agree well with those reported in the literature. The excess molar volumes, V^E and the viscosity deviations, $\Delta\eta$ of the mixture showed that the values are negative over the whole concentration range. To investigate the molecular interaction between [Bmim][BF4] and organic solvents, viscosity deviation, $\Delta\eta$, excess molar volumes V^E and excess Gibbs free energy of activation of viscous flow, ΔG^*E , have been evaluated from experimental density and viscosity using the following equations as below;

$$V^{E} = x_{1}M_{1} + x_{2}M_{2} / \rho - (x_{1}M_{1} / \rho_{1} + x_{2}M_{2} / \rho_{2})$$
(2)

$$\Delta \eta = \eta - (x_{1}\eta_{1} + x_{2}\eta_{2})$$
(3)

where x_1 and x_2 are the mole fractions calculated from mass fractions. M_1 and M_2 are molar masses, ρ_1 and ρ_2 are densities, η_1 and η_2 are the viscosities of pure components 1 and 2 respectively. The excess Gibbs free energy of activation of viscous flow was obtained from Eq.(3).

$$\Delta G^{*E} = RT[\ln \eta V - (x_1 \ln \eta_1 V_1 + x_2 \ln \eta_2 V_2)]$$
(4)

where *R* is the universal constant of gases, T is the absolute temperature, V_1 and V_2 are the molar volumes of component 1 and 2, x_1 and x_2 represents the mole fraction of component 1 and 2. η_1 , η_2 and η are the viscosity of component 1 and 2 and mixture respectively.

Molar volume is obtained from the relation as; $V = x_1 M_1 + x_2 M_2 / \rho$ (5)

The values of V^E and $\Delta \eta$ were correlated by a Redlich-Kister [15] type polynomial as;

$$\Delta Y^{E} = x_{1}x_{2}[A_{0} + A_{1}(2x_{1} - 1)^{1} + A_{2}(2x_{1} - 1)^{2} + A_{3}(2x_{1} - 1)^{3} + A_{4}(2x_{1} - 1)^{4}]$$
(6)

$$\Delta Y^{E} = x_{1} x_{2} \sum_{k=1}^{n} A_{K} (2x_{1} - 1)^{k-1}$$
(7)

The values of the parameters A_k , are obtained by fitting the equation to the experimental values with the least-squares method. The correlated results for excess molar volume, and viscosity deviation are presented in table 2. The standard deviation $\sigma(\Delta Y)$ is calculated from equation as;

$$\sigma(\Delta Y) = \left[\sum (Y_{\text{exp}} - Y_{cal})^2 / N - n\right]^{1/2}$$
(8)

where ΔY is the excess volume, V^E and deviation in viscosity $\Delta \eta$, The subscript exp and calc represents the experimental and calculated values respectively. N and n are the number of experimental data points and the number of coefficients in the Redlich-Kister polynomial equation.

Kendall and Monroe [16] derived the equation for analyzing the viscosity of binary mixtures based on zero adjustable parameter as;

$$\eta = (x_1 \eta_1^{1/3} + x_2 \eta_2^{1/3})^3 \tag{9}$$

$$E\eta = x_1 x_2 (x_1 \eta_1^{1/3} + x_2 \eta_2^{1/3})^3$$
(10)

where $E\eta$ is a modified Kendall-Monroe equation.

Grunberg and Nissan [17] formulated equation to determine the molecular interactions leading to viscosity changes with one parameter to estimate the dynamic viscosity of binary liquid as;

$$\ln \eta = x_1 \ln \eta_1 + x_2 \ln \eta_2 + x_1 x_2 d (11)$$
(11)

where d' is an interaction which is a function of the composition and temperature of binary liquid mixture.

Mixture data was collected in Table 3. A careful perusal of the Table 3 clearly shows that values of experimental and computed viscosity (from two equations) increases with the increase of the amount of [Bmim][BF₄] in binary liquid systems. Fig. 1 shows the variation of V^E as a function of mole fraction of the ionic liquid. It can be seen that the values of V^E are negative in all the ranges of composition, indicating negative deviations from ideal behavior. Similar results have been reported for the mixtures of [Bmim] $[BF_4]$ acetonitrile, dichloromethane,2-+butanone, and N, N-dimethyl formamide [18] for 4methyl-N-butylpyridinium tetra fluoroborate + methanol[19] and for 1-butyl-3methylimidazolium hexafluoro phosphate $[Bmim][PF_6]$ + organic compounds. It is more interesting that all the V^E in every [Bmim][BF4] + organic solute system have minimum values at about $x \approx 0.2$ -0.3. The molecular dynamics simulations of the mixture also showed that the excess molar volumes are negative over the whole concentration range and present a minimum at [Bmim][BF₄]mole fraction of $x \approx$ The excess molar volume, V^E can be 0.2-0.3. interpreted in terms of molecular interactions, positive values are explained by the breaking of chemical or nonchemical interactions among molecules in the pure components during the mixing process, whereas a more efficient packing and attractive interaction in the mixtures than in the pure liquids is considered to be the major contribution to the negative values of

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 V^{E} [20]Methyl formate, methyl acetate, ethyl formate, and acetone as normal polar molecules are with dipole-dipole interaction. Ionic liquids [Bmim][BF₄] are with electrostatic attractions $[BF_4]$ anion and [Bmim]⁺ between the cation[]¹⁷. The positive contribution to V^E would be due to the breaking of two kinds of interactions::electrostatic attractions between the [BF₄]⁻anion and [Bmim]⁺ cation and dipoledipole interaction in the organic polar molecules in the mixing process. Wang and co-worker's studies the ionic of liquids [Bmim][BF₄] and [Bmim][PF₆] with organic polar compounds revealed that the filling effect of organic compounds in the interstices of ionic liquids and the ion-dipole interactions between organic polar compound and the imidazolium ring of the ionic liquids are the main contributitors to the negative values of the molar excess volumes. The actual value of V^E would depend on the balance of the two opposite contributions[18]. According to these investigations, it seems possible to suggest that the filling effect and the ion-dipole interactions predominate for the experimental V^E values when methyl formate, methyl acetate, ethyl formate, or acetone is mixed with $[Bmim][BF_4]$. The viscosity deviations $\Delta \eta$ for the mixtures in Fig. 2 show a clear trend: all values are negative and very large values due to the big differences between the ionic liquid's and the other compounds viscosity in the studied ranges of composition for every binary mixture. The minium values are observed at about $x \approx 0.55-0.65$.

As far as the viscosity of the mixtures is plotted against mole fraction of the organic solutes, it is found that the organic solutes appear to have a surprisingly similar effect on the viscosity of the ionic liquids, as shown in Fig. 3. Therefore, the viscosity of the mixture of a binary system can be predicted as a function of the composition of the organic solutes or the ionic liquid, at given temperature. As shown in Fig. 3, it is clear that the viscosities of the mixtures decrease rapidly when organic compounds are added to the ionic liquid. This decrease is particularly strong in dilute solutions of organic compounds in the ionic liquid. The strong coulomb interactions between the [BF₄]⁻ anion and [Bmim]⁺ cation are weakened with the neutral upon mixing organic compounds, which leads to a higher mobility of the ions and а lower viscosity of the mixtures.Simmilar results can be explained when we look

4. CONCLUSIONS

The values of V^E are negative in all the

ranges of compositions, and all the V^E values in every [Bmim][BF₄] + organic solute system have minimum values at about $x \approx 0.2$ -0.3. The filling effect and the ion-dipole interactions are suggested to predominate for the experimental V^E values when methyl formate, methyl acetate, ethyl formate, or acetone is mixed with [Bmim][BF₄]. The viscosity deviations $\Delta \eta$ are negative and very large values due to the big differences between ionic liquid's and other compounds viscosity. The minium values are observed at about mole fraction of the ionic liquid, $x \approx 0.55 - 0.65$.

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