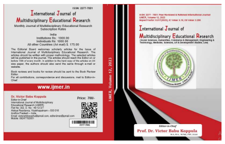
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Studies of the Intermolecular Interactions in Pyrrolidinium Based Ionic Liquid with 2-methoxy aniline at Different Temperatures

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ABSTRACT

In this experimental investigation, the thermo physical features of density (ρ), and speed of sound(U) were calibrated for aromatic aniline group of 2-methoxy aniline with solution of pyrrolidinium based ionic liquid such as 1-Butyl-1-methylpyrrolidinium tetra fluoroborate ([BMPyy]BF_4) in various concentrations within temperatures from 303.15 to 313.15K. From these experimental determined values, various thermodynamic acoustic parameters of excess isentropic compressibility(K^E_s) and excess molar volume (V^E_m) are expressed in specific and non-specific molecular interactions. Moreover, calibration of the partial molar volume's and partial isentropic compressibility's of both components shows strong interactions in the combination [BMPyy]BF_4 + 2-methoxy aniline at 303.15K temperature than any other higher temperatures in that combination.

Key words: FTIR analysis, Ionic Liquid, Partial isentropic compressibilites, Partial molar volumes.

1. INTRODUCTION

The specific features of Ionic Liquids (ILs) have great plausible utilization in contemporary epoch[1]. Remarkably, the thermo physical distinctive features of ILs at ambient temperatures play a juggernaut role in many sectors such as industrial engineering, pharmaceutical manufacturing and waste management treatment[2]. With inventiveness, the binary fluids which contain IL accompanied by any organic solvent portrays versatile thermo physical features of these fluids. Noteworthy, the molecular interactions at particular point of concentration of binary fluids are worthwhile and that leads to designer solvents of ILs at that point[3]. In this ambience, several peer groups of brainchildren uplift the binary fluid combination of ILs into high-yield ionic liquid applications in foregoing sectors[4]-[7]. In this investigation research, the authors appraised the binary fluid which includes IL of 1-butyl, 1-methylpyrrolidinium tetrafluoroborate ([BMPyrr]BF_4) with 2-methoxy aniline at subsequent interval of temperature ranges from of temperature ranges from 303.15K to 313.15K throughout an ambient atmospheric pressure. This protic IL commonly engineered in electrochemical energy applications for lithium batteries and super capacitors[8]-[11]. Due to the aniline group attachment in the aromatic benzene of 2-methoxy aniline, they are greatly used as solvents in many physical and chemical fields. Moreover, 2-methoxy aniline is used to prevent the corrosion in oil refinery rigs. Consequently, the study of the binary combinations of pyrrolidinium group with $-NH_2$ group provides much more attention. The thermodynamic excess features such as excess molar volume(V^E_m) and excess isentropic compressibility($K^E_{s,m}$) of binary fluids are extracted from fundamental features of density(ρ) and speed of sound(U)[12]-[13]. These fundamental features help to understand the geometrical configurations and that develops the structure property correlations.

2. MATERIAL AND METHODS

Table 1: Specification of Source, CAS Number, Mass Fraction Purity and Further Purification

Name of the chemical	Source	CAS Number	Mass fraction purity	Further purification methods
2-methoxy aniline(2MA)	HiMedia Laboratories, India	90-04-0	>98.00%	*GLPC
[BMPyrr]BF_4 (IL)	HiMedia Laboratories, India	345984-11-4	>99.70%	**Millipore

*Gas-Liquid partition chromatography carried through inert gas Ar.
**Impurities separation by filtration.

2.1 Specimen information

The 2-methoxy aniline (2MA) have been subjected to glass chromatography. 2MA and ([BMPyrr]BF_4)(IL) have been catalogued in Table 1. Additionally, the Table 2 illustrated the density and speed of sound of pure fluids of IL and 2MA. The specimens were correlated with standard articles[12],[14]-[15].

Table 2: Physical properties of pure component 2-methoxy aniline and IL with literature at specific temperatures

Sample	Parameter	Expt. /Lit.	Temperatures		
			303.15K	308.15K	313.15K
2MA	ρ (kg.m^{-3})	Expt.	1091.80	1087.40	1083.80
	ρ (kg.m^{-3})	Lit.	1091.80^a	1087.40^a	1083.80^a
	ρ (kg.m^{-3})	Lit.	1091.75^b	1087.35^b	1083.78^b

	U (m.s^{-1})	Expt.	1595.44	1579.44	1466.32
	U (m.s^{-1})	Lit.	1595.44^{a}	1579.44^{a}	1466.32^{a}
	U (m.s^{-1})	Lit.	1595.40^{b}	1579.20^{b}	1466.30^{b}
IL	ρ (kg.m^{-3})	Expt.	1389.02	1364.53	1352.18
	ρ (kg.m^{-3})	Expt.	1389.02^{c}	1364.53^{c}	1352.18^{c}
	U (m.s^{-1})	Expt.	1584.34	1552.56	1535.44
	U (m.s^{-1})	Lit.	1584.34^{c}	1552.56^{c}	1535.44^{c}

Here [a][12], [b][14], [c][15]

2.2 Evaluation Aproach

The prepared binary fluids and pure fluids were fountained in to glass vials with the help of mass analytical balance (Mettler Toledo) which having an accuracy $\pm 10^{-11}$ kg. These vials were covered with air tight lids to prevent evaporation and adsorption of atmospheric moisture. The measured final molefraction for binary fluids are having uncertainty less than ± 0.0001. The binary fluids of system such as IL+2MA were prepared at specified concentrations, i.e., molefractions of these systems carried out 12 subsequent values from 0 to 1. The temperature dependent thermophysical features alike density (ρ) and speed of sound (U) of IL binary fluids were simultaneously calibrated through an instrument vibrating-tube digital density and speed of sound analyser in the temperatures 303.15K, 308.15K and 313.15K. The entire instrument contains temperature bath which has been controlled through incorporated Peltier thermostat with an accuracy of ±0.01K. So the uncertainty values of experimental density (ρ) and speed of sound (U) were below of 0.01kg.m^{-3} and 0.5m.s^{-1} respectively.

3. DATA VALUE AND VALIDATION

The thermo physical fundamental features ρ and U were experimentally calibrated for binary fluid of IL+2MA with temperature ranges 303.15K, 308.15K, 313.15K. These were graphically portrayed in Figure 1 and Figure 2. The observed features of non-linear increasing trend suggest that fluids have molecular interactions exists between them[15].

This gives a first observable fact that as the temperature increases the interactions of molecules decreases. The eventual general expression was

$$Y^{E} = Y^{r} - Y^{id} \qquad (1)$$

Here $Y^{E} = V_{m}^{E}$, $K^{E}_{s,}$ and Y {$=V_{\mathrm{m}}$(molar volume), K_{s} (isentropic compressibility)} are the real value of fluids. And, the ideal component of thermodynamic acoustic parameters for molar volume and isentropic compressibility stands for

$$Y^{id} = x_{1}Y_{1} + (1 - x_{1})Y_{2} \qquad (2)$$

Here, x_1 is the molefraction of component IL (1) with respect to 2MA(2). Y_1 and Y_2 are the pure acoustic molar values of IL(1) and 2MA(2) respectively. The isentropic compressibility K_s was calculated from the equation

$$K_{s} = \frac{1}{\rho U^{2}} \qquad (3)$$

3.1 Redlich-Kister Polynomial Analysis

The conventional non-lineal curve fitting strategy for attributes of binary fluids pertains Redlich-Kister polynomial regression[16]-[18] which contains legendre coefficients.

$$Y_{RK}^{E} = x_{1}(1 - x_{1})\Sigma_{p=0}^{p=N} A_{p,T} L_{p}(2x_{i} - 1) \qquad (4)$$

Here, Y^{E}_{RK} means excess thermo dynamic feature which contain any value (i.e., V_{m}^{E}, K_{s}^{E}) has been taken. The standard deviation was also calibrated for the consequence.

$$\sigma(Y_{RK}^{E}) = \sqrt{\Sigma_{i=1}^{i=N} \frac{\left(Y_{i,exp} - Y_{i,cal}\right)^{2}}{M - N}} \qquad (5)$$

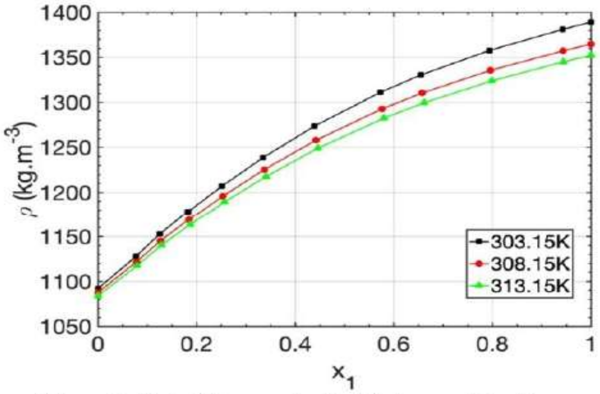


Figure 1: Plot of thermo physical features of density versus molefraction

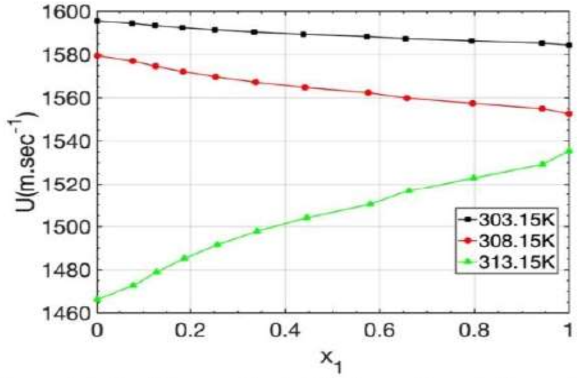
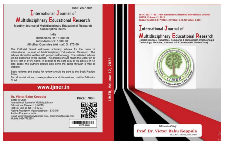


Figure 2: Plot of thermo physical features of speed of sound versus molefraction

Where M stands for the number of experimental values and N stands for the adjustable parameter. These excess values are examined with Redlich-Kister polynomial non-linear regression with legendre coefficients. And these values of $A_{i,T}$(i=0,1,2,3) are determined along with standard deviation for the experimental values. Table 3 catalogued the whole values of all composites. The abnormality of thermodynamic acoustic excess parameters of V_{m}^{E}, K_{s}^{E} are portrayed in Figure 3 & Figure 4 respectively. Due to thermal agitations of all fluids, temperature rise ushers to descend the excess parameters [19]. The values of V_{m}^{E} are attributes positive or small negative for high concentrations of 2MA and the trend turns to negative during the increasing concentration of [BMPyy]BF$_4$ in the respective composites.



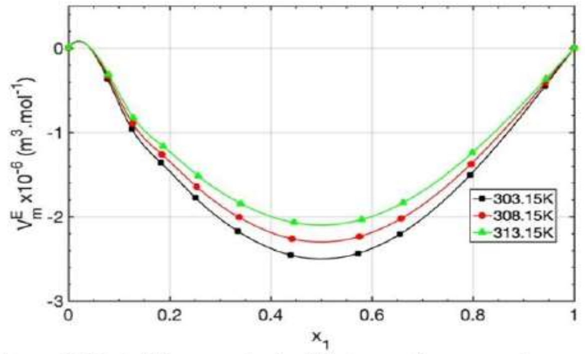


Figure 3: Plot of thermo physical features of excess volume versus molefraction

The flipping sharpness of $V_m{}^E$ is negative. This is clearly visible in Figure 3, for the replicated sequence $V_m{}^E$. This clearly distinguishes the formation of H-bond, which is stronger at higher concentration and weaker in lower concentrations of composites. In addition to that, formation of H-bond is very weak at high concentrations of higher temperatures[20]. The abnormality of excess isentropic compressibility at all temperatures has been illustrated in Figure 4 over an entire concentration for all composites. The $K^E{}_s$ values are negative at all investigated temperatures for all composites. The sign of $K^E{}_s$ plays a vital role in assessing the compactness due to molecular interactions in multi-component mixtures suggests interstitial accommodation and oriental ordering leading to more compact structure.

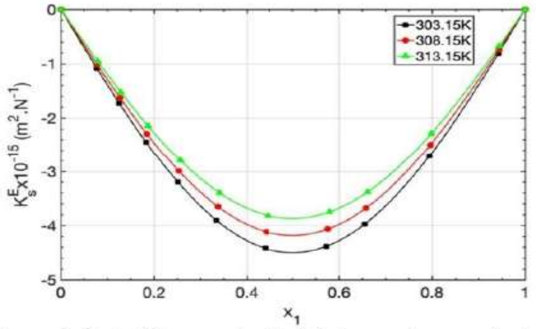


Figure 4: Plot of thermo physical features of excess isentropic compressibility versus molefraction

Fort and Moore [21] indicated that the binary fluids having distinct molecular sizes and shapes mix well there by reducing the volume which causes values of $K^E{}_s$ to be negative. The $K^E{}_s$ value of negative is clearly visible in Figure 4. This also clearly distinguishes a greater steric hindrance to the formation of hydrogen bonds in the respective composites.

3.2 Reduced Redlich-Kister Polynomial Analysis

The ineluctable Redlich Kister approach sporadically misguides the dissimilar composites. In conjunction to that it deceives interactions of molecules at low concentration regions in composites. Consequently, desnoyers[22] suggested a contemporary befitted Reduced Redlich-Kister (RRK)[23] polynomial analysis to address more specific features in composites. So the equation becomes

$$Q_{Y^E_{RRK}}(x_1) = \frac{Y^E_{RK}(x_1)}{x_1(1-x_1)} \qquad (6)$$

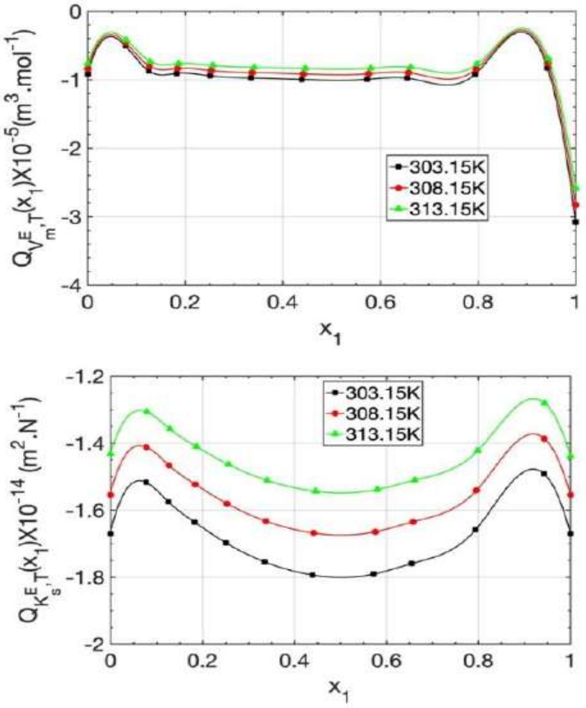
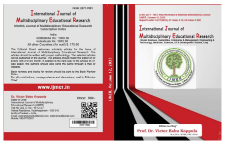


Figure 5: Plot of thermo physical features of reduced excess volume versus molefraction

Table 3: Coefficients of redlich-kister equation parameters and standard deviations at different temperatures.

Combination	T/K	Redlich-kister equation coefficients				error
		$A_{0,T}$	$A_{1,T}$	$A_{2,T}$	$A_{3,T}$	$\sigma(Y^E_{RK})$
$V_m{}^E$ (10^{-6} m^3.mol^{-1})						
IL(1) + 2MA (2)	303.15	-21.92	-5.22	-3.18	-4.52	0.012
	308.15	-19.99	-4.75	-2.95	-4.07	0.015
	313.15	-18.23	-4.33	2.71	-3.70	0.013
$K_s{}^E$ (10^{-15} m^2.N^{-1})						
IL(1) + 2MA (2)	303.15	-0.019	0.000	0.002	0.000	4.44E-07
	308.15	-0.018	0.000	0.002	0.000	4.095E-08
	313.15	-0.016	0.000	0.002	0.000	3.784E-07

The above specification is equivalent to apparent molar quantity of respective excess parameters in entire concentration range. Excess thermodynamic quantities have the convenience of showing the flipping sign, sharpness and magnitude of the dissimilarity composites, but the RRK polynomial specification elucidates promising features to lift the origin of the dissimilarity solutions. The abnormality of



$Q_{V_m^E,T}(x_1)$ at all temperatures has been illustrated in Figure 5 over an entire concentration for all composites. The values $Q_{V_m^E,T}(x_1)$ are attributes higher values for low temperatures. This evidently distinguishes the hydrophobic interactions are present and stronger in the region of higher concentrations of IL. In addition to that, Hydrophobic interactions are very weak at high concentrations of 2MA. For the present scenario, the reduced functions values $Q_{K_S^E,T}(x_1)$ are higher value on 2MA side and lower value on IL side. This clearly visible in Figure 6, for the replicated sequence of $Q_{K_S^E,T}(x_1)$

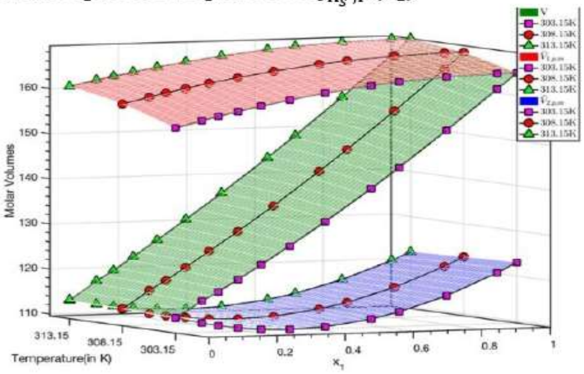


Figure 7: Plot of partial molar volumes/10^{-6} against molefraction and temperature for the solution [BMPyy]BF$_4$(1)+2MA(2).

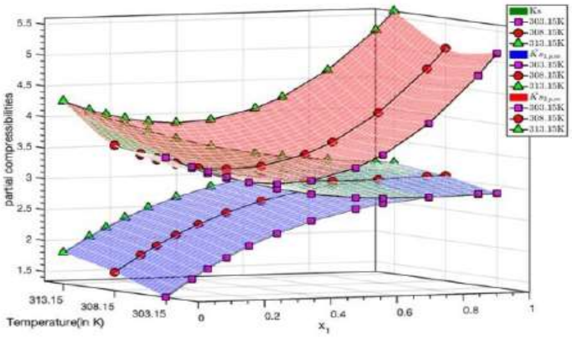


Figure 8: Plot of partial molar isentropic compressibilities/10^{-15} against molefraction and temperature for the solution BMPyy]BF$_4$(1)+2MA(2)

This result indicates composites are less compressible than the ideal mixtures. This strongly reveals strong interactions occur in these composites. It also clearly indicates that the 2MA molecules are more sterically hindered in IL molecules at 303.15K.

3.3 Partial molar volumes and partial molar isentropic compressibilities

The RRK functions of $Q_{V_m^E,T}(x_1)$ and $Q_{K_S^E,T}(x_1)$ at infinite dilution over a constant temperature and pressure was an addition tool to represents partial molar volumes[24] and partial isentropic compressibilities. The above extrapolation expression (6) has modified as

$$Q_{V_m^E}(x_1=0) = A_{0,T} - A_{1,T} + A_{2,T} - A_{3,T} = \bar{V}^{E,\infty}_{1,p,m} = \bar{V}^{\infty}_{1,p,m} - V_{1,m} \qquad (7)$$

$$Q_{V_m^E}(x_1=1) = A_{0,T} + A_{1,T} + A_{2,T} + A_{3,T} = \bar{V}^{E,\infty}_{2,p,m} = \bar{V}^{\infty}_{2,p,m} - V_{2,m} \qquad (8)$$

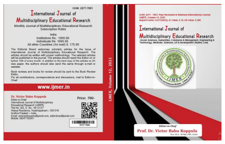
$\bar{V}^{E,\infty}_{1,p,m}$ and $\bar{V}^{E,\infty}_{2,p,m}$ are excess partial molar volumes two pure components at infinite dilutions. $\bar{V}^{\infty}_{1,p,m}$ and $\bar{V}^{\infty}_{2,p,m}$ are partial molar volumes at infinite dilutions. $V_{1,m}$ and $V_{2,m}$ are pure molar volumes of two components IL and 2MA. Similarly, the equation analogy is also true for partial isentropic compressibilities. But, the real partial molar volumes and partial isentropic compressibilities with respect to a molefraction at constant pressure and temperatures can be evaluated from the following differential equation

$$\bar{V}_{i,p,m} = V_m(x_i) - x_j\left(\frac{\partial V_m(x_j)}{\partial x_j}\right)_{T,P} \qquad (9)$$

$$\bar{K}_{i,p,s} = K_s(x_i) - x_j\left(\frac{\partial K_s(x_j)}{\partial x_j}\right)_{T,P} \qquad (10)$$

Here x_i and x_j are the mole fractions of two components in the composite ($i=1,2$ & $j=i-1$). The intermolecular interactions in the composites can be interpreted in terms of packing efficiency of molecules with the help of partial molar volumes and partial isentropic compressibilities. The partial molar volumes of two components $\bar{V}_{1,p,m}$ and $\bar{V}_{2,p,m}$ are play vital role in binary fluids. Because the domain influence of the components in the mixtures would changes with respect to the composition concentrations and as well as temperature. In this scenario, the partial molar volumes of all components has been shelled in Figure 7.

In this figure, the scaffold Z symbol graph contains three colored meshes, which are concerned to red ($\bar{V}_{1,p,m}$), green (total molar volume, V) and blue ($\bar{V}_{2,p,m}$). For all combinations, the partial molar volumes of both components $\bar{V}_{1,p,m}$ and $\bar{V}_{2,p,m}$are lower than of their individual values in the pure state, which reveals the domain influence of the individual components decreasing with their respective lower concentration regions. The abnormality is examined for all constant intervals of temperatures. This clearly suggests presence of solute-solvent interactions in between unlike molecules. Hence, from the representation of Figure 7. The effect of domain influence of volume is low at 303.15K. The partial isentropic compressibilities of two components $\bar{K}_{1,p,s}$ and $\bar{K}_{2,p,s}$ are also play crucial role in binary mixture. Because the geometrical influence of the components in the mixture changes with respect to the composition concentration and temperature. In this scenario, the partial isentropic compressibilities of components [BMPyy]BF$_4$+2MA have been shelled in Figure 8. In this figure, the mirror scaffolded Z symbol graph contains three colored meshes, which are concerned to red ($\bar{K}_{1,p,s}$), green (total isentropic compressibility, K_s) and blue ($\bar{K}_{2,p,s}$). The partial isentropic compressibilities $\bar{K}_{1,p,s}$ and $\bar{K}_{2,p,s}$ are more at 303.15K. This clearly suggests the breaking of dipole inclusions in



[BMPyy]BF_4+2MA at 303.15K has more than compared to the other higher temperatures in that combination.

4. CONCLUSIONS

In this frame work, the values of excess thermodynamic parameters have been calibrated for an entire composition of IL+2MA with accustomed levels of temperatures. This is clearly elucidating a strong hydrogen bonding, dipole-inclusion interactions present in the component molecules. Moreover, the reduced excess thermodynamic parameters have been executed by using contemporary RRK polynomial over an entire composition with accustomed levels of temperatures. This reveals more specific features about, smaller molar mass of 2MA molecules sterically hindered in larger molar mass of IL in their respective temperatures. The reduced excess parameters value decreases for increasing the temperatures in their compositions due to their thermal agitations. And the partial molar volumes, partial molar isentropic compressibilities of all components have been plotted in 3D format. This discloses the intermolecular interactions are strong in IL(1)+2MA(2) at 303.15K than other higher temperatures.

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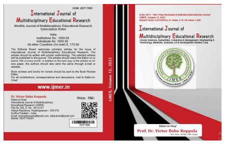

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