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Excess Free Volumes, Internal Pressures and Molar Available Volumes in the Binary Liquid Mixtures of N-Methyl-2-Pyrrolidone at Different Temperatures

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Abstract: *Available volume, molar free volume, internal pressure and their excess values are determined from the measured data of ultrasonic speeds, densities and viscosities of binary fluid mixtures of N-methyl-2-pyrrolidone (NMP) and Propyl amine (PA) or Butylamine (BA) or Dipropylamine (DPA) at different temperatures with an interval of 5 K from 303.15 to 318.15 K. Excess properties are fitted to a Redlich–Kister type equation. The excess available volume and excess molar free volumes are found to be negative where as the excess internal pressures are found to be positive. The results observed from these studies suggest dipole-dipole and Hydrogen-bonded interactions between unlike molecules in all the three systems investigated.*

Keywords: *Density, viscosity, speeds of sound, Thermo acoustic properties, Relich-Kister polynomial, molecular interactions*.

1. INTRODUCTION

The knowledge on variation of thermodynamic properties with temperature involving non electrolyte solutions is of substantial significance in the chemical industry connecting chemical separations, mass transfer, fluid flow and heat transfer. When two or more solvent molecules are associated with one another to form a liquid mixture, it brings about a marked effect on the intermolecular interactions, due to changes in free volume, energy and molecular orientations leading to corresponding variations in the thermodynamic properties. In recent years, ultrasonic technique has become a powerful tool in providing information regarding the molecular behavior of liquids and solids owing to its ability of characterizing physiochemical behavior of the medium. The present work is a continuation of our research programme on thermodynamic, thermo-acoustic and transport behaviour of binary liquid mixtures of industrially important components [1-6].

Lactams are significant class of amides not extensively studied despite their use as solvents and biological applications. Hence, one of the lactams, N-methyl-2-Pyrrolidone (NMP) is chosen as a common solvent for the present study. NMP known for its solvent power is rapidly becoming the product of choice for paint strippers, agricultural chemicals, and process solvent applications [7-10]. In this paper we report the internal pressures, free volumes and molar available volumes of the binary systems N-methyl-2-Pyrrolidone (NMP) + propylamine (PA) or Butylamine (BA) or Dipropylamine (DPA) at temperatures 303.15 - 318.15 K.

2. EXPERIMENTAL SECTION

The chemicals, NMP (>99%) obtained from Merck and Propylamine (>99%), Butylamine (>99%), Dipropylamine (>99%) procured from S.D. Fine chemicals (India) are further purified by using standard methods [9]. The purity of the chemicals was assessed by comparing their

measured densities (ρ), ultrasonic velocities (U) and viscosities (η), which are in good agreement with literature [11-15] values as can be seen in Table 1. The binary mixtures have been prepared gravimetrically using an electronic balance (Shimadzu AY 120, Japan) with an uncertainty of ± 1x 10^{-7} Kg and are stored in air-tight glass bottles. The densities, ρ, of pure liquids and their mixtures are determined using a 10^{-5} m^3 double-arm pycnometer as described by Nikam et al [16]. The density values from triplicate replication at each temperature are accurate unto 4 parts in 10^5 parts. The ultrasonic velocity of pure components and their mixtures have been measured by single crystal variable path fixed frequency interferometer provided by Mittal Enterprises, New Delhi (Model-F 05). The measured speeds of sound are found to be accurate up to 0.1 $m \cdot s^{-1}$. Temperature control for the measurement of viscosity, density and ultrasonic velocity is achieved by using a microprocessor assisted circulating water bath, (supplied by Mac, New Delhi) regulated to ±0.01 K, using a proportional temperature controller.

Table1. *Comparison of experimental values of density (ρ), and ultrasonic speed (U) of pure liquids with the corresponding literature values at 303.15 K*

	10^{-3} x ρ (kg m^{-3})		U (m s^{-1})	
Liquid	Expt	Lit.	Expt	Lit.
N-methyl-2-pyrrolidone	1.0238	1.02376 [11]	1552.8	1552 [12]
Propylamine	0.7093	0.70927 [13]	1193.1	1193.96 [13]
Butylamine	0.7289	0.72842 [14]	1226.0	1227 [14]
Dipropylamine	0.7316	0.73133 [13]	1175.0	1174 [15]

3. RESULTS AND DISCUSSION

Assuming that ultrasonic absorption is negligible, using experimental results of density, viscosity and ultrasonic velocity, the thermodynamic and thermoacoustic parameters such as free volume (V_f), internal pressure (π_i), molar available volume (V_a) and their excess parameters have been calculated using the following standard expressions and are presented in Tables 2-4.

Erying et al [17] obtained the relationship between the internal pressure P_i, the external pressure P and the free volume V_f of a liquid as follows:

$$V_f = [KRT/(P_i+P)\ (\rho+V_f^{1/3}/K)^2]^3$$

The above relationship shows that free volume of a molecule at a particular temperature and pressure depends only on the internal pressure of the liquid in which it is immersed. The free volumes of the binary mixtures have been computed using its relationship with the ultrasonic velocity and viscosity as follows:

$$V_f = \left[\frac{M_{eff}U}{K\eta}\right]^{3/2} \tag{1}$$

where 'K' (= 4.28 x 10^9 in S. I system) is a temperature independent proportionality constant and the other symbols have their usual meaning.

Suryanarayana and Kuppuswami [18] used an indirect alternative method for computing the internal pressure of a liquid from its viscosity, density and ultrasonic velocity as shown below

$$\pi_i = \text{bRT}\left[\frac{K\eta}{U}\right]^{1/2}\left[\frac{\rho^{2/3}}{M^{7/6}}\right] \tag{2}$$

where '*b*' is a packing factor, 'R' is Universal gas constant, '*T*' is the absolute temperature and other symbols have their usual meaning.

The molar available volume is the parameter related molecular interactions of the medium and calculated by using the expression

$$V_a = V_m\left(1-\frac{U}{U_\infty}\right) \tag{3}$$

The excess properties such as V_f^E, V_a^E and π_i^E have been calculated using the equation

$$Y^E = Y_{mix} - (x_1 Y_1 + x_2 Y_2) \quad (4)$$

where Y^E is V_f^E or V_a^E or π_i^E and x_1 and x_2 are mole fractions of liquid 1 and 2 respectively.

These excess functions are fitted to Redlich – Kister [19] type polynomial equation.

$$Y_{cal}^E = x_1 x_2 \sum a_{j-1} (x_2 - x_1)^{j-1} \quad (5)$$

The values of coefficient a_{j-1} are evaluated by the method of least squares with all points weighed equally and the standard deviations are calculated using

$$\sigma_{YE} = \left[\frac{\Sigma (Y_{obs}^E - Y_{cal}^E)^2}{(m-n)} \right]^{1/2} \quad (6)$$

where 'm' is the number of experimental data points and 'n' is the number of coefficients considered (n = 5 in the present calculation).

It is evident from Tables 2-4, that all the three binary systems exhibit non-linear increase/decrease in V_f, V_a and π_i values with composition of NMP. This indicates the presence of intermolecular interactions between the component molecules of the mixture. In order to substantiate the presence of interactions (either adhesive or cohesive forces) between the molecules, it is essential to study excess parameters like excess free volume (V_f^E), excess internal pressure (π_i^E), excess molar available volume (V_a^E) etc., as these parameters are found to be more sensitive towards intermolecular interactions in the liquid mixtures [20].

In pure state, alkyl amines are weakly self-associated [8]. Although, the addition of a strongly polar solvent (NMP) disrupts the association of the amine, the increase in ultrasonic speed with NMP addition indicates strong interactions between the component molecules [16]. However, the excess functions are very constructive in understanding molecular interactions between components of liquid mixtures. By definition, the excess function Y^E represents the excess of a given quantity Y of a real mixture over its value for an ideal mixture Y^{id} at the same conditions of pressure, temperature, and composition [17]. When alkyl amines are added to NMP it results in considerable decrease in intermolecular spaces between molecules leading to decrease in free volume and hence increase in internal pressure. So negative values of V_f^E and positive π_i^E values are observed in the present case. Our finding is in good agreement with the views proposed by Jacobson [21].

The V_f^E values are found to decrease (Table 2) with increase in the concentration of NMP, attaining a minimum value between 0.4-0.5 mole fraction of NMP. This indicates that the associated structure of the polar component (NMP) due to dipolar association has been broken by solvent molecules. The negative V_f^E values indicate the contributions made by the strong dipole-dipole interactions between the unlike molecules of the components [22].

Table2. ***Excess molar volumes*** *($10^6 x V_m^E$ /$m^3 \cdot mol^{-1}$) for the binary mixtures of N-methyl-2-pyrrolidone (NMP) and alkyl amines (PA or BA or DPA) at temperatures of 303.15, 308.15, 313.15 and 318.15 K*

	NMP + PA					NMP + BA					NMP + DPA			
x_1	303.15 K	308.15 K	313.15 K	318.15 K	x_1	03.15 K	308.15 K	313.15 K	318.15 K	x_1	303.15 K	308.15 K	313.15 K	318.15 K
0.0000	0.000	0.000	0.000	0.000	0.0000	0.000	0.000	0.000	0.000	0.0000	0.000	0.000	0.000	0.000
0.0873	-8.838	-9.316	-9.308	-9.655	0.1033	-7.555	-7.953	-7.654	-7.945	0.1370	-9.616	-12.901	-12.970	-14.740
0.1771	-11.942	-12.999	-12.621	-13.339	0.2059	-10.111	-10.608	-10.497	-10.939	0.2631	-12.389	-16.053	-16.256	-18.365
0.2695	-12.701	-13.884	-13.581	-14.415	0.3077	-10.516	-11.264	-11.286	-11.765	0.3797	-12.625	-16.133	-16.501	-18.553
0.3646	-12.271	-13.360	-13.244	-14.018	0.4087	- 10.059	-10.882	-10.895	-11.263	0.4878	-11.728	-14.784	-15.222	-17.079

0.4626	-11.147	-12.298	-12.155	-12.890	0.5091	-9.049	-9.794	-9.816	-10.220	0.5882	-10.240	-12.795	-13.175	-14.775
0.5636	-9.613	-10.578	-10.462	-11.159	0.6087	-7.605	-8.205	-8.184	-8.516	0.6818	-8.349	-10.433	-10.747	-12.079
0.6676	-7.677	-8.443	-8.381	-8.939	0.7076	-5.806	-6.288	-6.321	-6.541	0.7692	-6.344	-7.928	-8.115	-9.195
0.7749	-5.404	-5.943	-5.886	-6.285	0.8057	-3.911	-4.244	-4.276	-4.427	0.8511	-4.305	-5.348	-5.441	-6.132
0.8857	-2.814	-3.142	-3.110	-3.354	0.9032	-2.033	-2.132	-2.169	-2.268	0.9278	-2.196	-2.701	-2.798	-3.109
1.0000	0.000	0.000	0.000	0.000	1.0000	0.000	0.000	0.000	0.000	1.0000	0.000	0.000	0.000	0.000

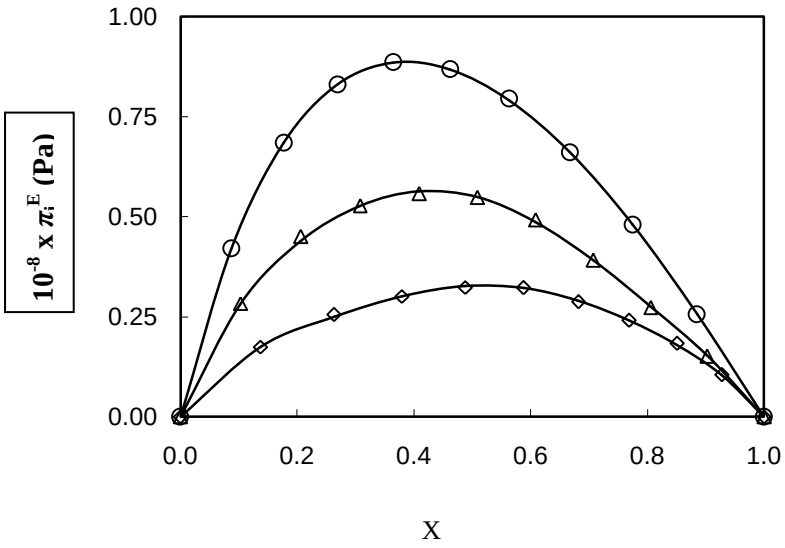

Fig1. *Variation of internal pressure, π_i^E, for the binary mixtures NMP + PA or BA or DPA at 303.15 K aginst mole fraction of NMP*

Excess values of π_i^E (Figure 1, Table 3) in the binary mixtures of NMP with PA or BA or DPA show a maximum value between 0.4-0.5 mole fraction of NMP, then decrease with further increase in its concentration. This indicates the weakening of intermolecular interaction between component molecules. Thus based on the magnitude of positive values in π_i^E, the order of the interaction, in the substituted benzenes under investigation at 308.15 and 318.15 K with NMP is as follows: PA > BA > DPA.

Table3. *Excess internal pressures ($10^8 x\pi_i^E$ /Pa) for the binary mixtures of N-methyl-2-pyrrolidone (NMP) and alkyl amines (PA or BA or DPA) at temperatures of 303.15, 308.15, 313.15 and 318.15 K*

x_1	NMP + PA				x_1	NMP + BA				x_1	NMP + DPA			
	303.15 K	308.15 K	313.15 K	318.15 K		303.15 K	308.15 K	313.15 K	318.15 K		303.15 K	308.15 K	313.15 K	318.15 K
0.0000	0.000	0.000	0.000	0.000	0.0000	0.000	0.000	0.000	0.000	0.0000	0.000	0.000	0.000	0.000
0.0873	0.420	0.387	0.384	0.363	0.1033	0.283	0.269	0.249	0.242	0.1370	0.175	0.160	0.152	0.154
0.1771	0.684	0.655	0.618	0.596	0.2059	0.451	0.422	0.403	0.392	0.2631	0.255	0.245	0.233	0.235
0.2695	0.830	0.799	0.757	0.732	0.3077	0.527	0.509	0.495	0.479	0.3797	0.301	0.297	0.288	0.287
0.3646	0.886	0.844	0.815	0.783	0.4087	0.558	0.549	0.529	0.502	0.4878	0.323	0.316	0.313	0.309
0.4626	0.868	0.848	0.810	0.778	0.5091	0.548	0.539	0.519	0.497	0.5882	0.322	0.313	0.308	0.305
0.5636	0.794	0.773	0.734	0.711	0.6087	0.492	0.481	0.458	0.437	0.6818	0.288	0.286	0.281	0.281
0.6676	0.661	0.641	0.611	0.591	0.7076	0.391	0.385	0.371	0.351	0.7692	0.240	0.243	0.232	0.240
0.7749	0.480	0.466	0.441	0.428	0.8057	0.273	0.270	0.262	0.248	0.8511	0.183	0.183	0.172	0.174
0.8857	0.256	0.254	0.242	0.239	0.9032	0.152	0.141	0.140	0.136	0.9278	0.105	0.103	0.104	0.101
1.0000	0.000	0.000	0.000	0.000	1.0000	0.000	0.000	0.000	0.000	1.0000	0.000	0.000	0.000	0.000

The presence of stronger interactions, are further substantiated by the negative values of V_a^E for these binary liquid mixtures. Molar available volume is a quantity which depends on the molecular packing of the systems. In the present investigation V_a^E values (Table 4) show negative/positive deviations, confirming that strong magnitude of interactive forces, existing

between the unlike molecules of the component liquids in all the three binary systems, over the entire composition range of NMP.

Table4. *Excess molar available volumes ($10^6 x V_a^E$ /$m^3 \cdot mol^{-1}$) for the binary mixtures of N-methyl-2-pyrrolidone (NMP) and alkyl amines (PA or BA or DPA) at temperatures of 303.15, 308.15, 313.15 and 318.15 K*

x_1	NMP + PA				x_1	NMP + BA				x_1	NMP + DPA			
	303.15 K	308.15 K	313.15 K	318.15 K		303.15 K	308.15 K	313.15 K	318.15 K		303.15 K	308.15 K	313.15 K	318.15 K
0.0000	0.000	0.000	0.000	0.000	0.0000	0.000	0.000	0.000	0.000	0.0000	0.000	0.000	0.000	0.000
0.0873	0.119	0.050	-0.024	-0.108	0.1033	-0.199	-0.227	-0.258	-0.388	0.1370	-0.085	-0.027	-0.083	-0.151
0.1771	0.204	0.093	-0.043	-0.171	0.2059	-0.326	-0.378	-0.404	-0.584	0.2631	0.277	0.180	0.143	0.119
0.2695	0.261	0.106	-0.066	-0.220	0.3077	-0.395	-0.472	-0.528	-0.696	0.3797	0.593	0.543	0.529	0.508
0.3646	0.280	0.100	-0.104	-0.280	0.4087	-0.441	-0.519	-0.613	-0.824	0.4878	1.096	1.055	1.024	0.979
0.4626	0.276	0.071	-0.134	-0.308	0.5091	-0.448	-0.539	-0.622	-0.773	0.5882	1.675	1.553	1.578	1.519
0.5636	0.263	0.045	-0.128	-0.280	0.6087	-0.414	-0.504	-0.561	-0.722	0.6818	1.907	1.846	1.726	1.769
0.6676	0.256	0.070	-0.074	-0.207	0.7076	-0.348	-0.431	-0.488	-0.617	0.7692	1.881	1.846	1.746	1.736
0.7749	0.216	0.065	-0.050	-0.163	0.8057	-0.258	-0.326	-0.377	-0.449	0.8511	1.859	1.751	1.528	1.417
0.8857	0.133	0.050	-0.042	-0.108	0.9032	-0.133	-0.171	-0.217	-0.271	0.9278	1.275	1.189	1.110	0.928
1.0000	0.000	0.000	0.000	0.000	1.0000	0.000	0.000	0.000	0.000	1.0000	0.000	0.000	0.000	0.000

The values of the adjustable coefficients A_{j-1} of Redlich-Kister polynomial along with the standard deviations for excess free volume (V_f^E), excess internal pressure (π_i^E), and Molar available volume (V_a^E) at temperatures 308.15 and 318.15 K are presented in Table 5.

Table5. *Redlich-Kister Coefficients (A_{j-1}) and the Standard Deviations (σ) of Excess Parameters for the Binary Mixture of NMP and PA or BA or DPA at Different Temperatures*

	T/K	A_0	A_1	A_2	A_3	A_4	σ
N-methyl-2-pyrrolidone(1) + propylamine(2)							
V_f^E/10^{-6} m^3 mol^{-1}	303.15	-42.5886	-28.2808	-21.2032	-32.5488	-26.3877	0.086
	308.15	-46.9471	-31.8241	-22.9947	-29.7823	-24.3426	0.047
	313.15	-46.5280	-30.0274	-19.0381	-32.4394	-30.8042	0.103
	318.15	-49.3061	-32.0825	-22.9595	-31.2905	-26.1733	0.067
π_s^E / $10^{-8}$$Pa^{-1}$	303.15	3.3824	1.3885	0.7003	0.4533	0.0181	0.001
	308.15	3.3038	1.3984	0.4743	0.0584	0.0904	0.008
	313.15	3.1608	1.2666	0.1676	0.3131	0.7014	0.002
	318.15	3.0327	1.2414	0.4117	0.1463	0.2981	0.003
N-methyl-2-pyrrolidone(1) + butylamine(2)							
V_f^E/10^{-6} m^3 mol^{-1}	303.15	-36.6370	-23.9750	-13.1044	-20.2378	-19.0318	0.014
	308.15	-39.6450	-25.2772	-12.7740	-21.2788	-18.8752	0.054
	313.15	-39.7424	-25.9423	-13.6624	-16.5632	-13.6945	0.045
	318.15	-41.3874	-27.5044	-13.6661	-16.1186	-14.9078	0.052
π_s^E / 10^{-8} Pa^{-1}	303.15	2.2070	0.6954	-0.2213	0.2245	0.8269	0.006
	308.15	2.1667	0.6120	-0.3073	0.3147	0.7261	0.001
	313.15	2.0869	0.6490	-0.1895	0.0569	0.4437	0.002
	318.15	-3.1766	-0.9404	0.1029	0.5818	0.1890	0.010
N-methyl-2-pyrrolidone(1) + dipropylamine(2)							
V_f^E/10^{-6} m^3 mol^{-1}	303.15	-46.3749	-25.4772	-14.9055	-14.1529	-11.5086	0.017
	308.15	-58.2666	-34.8150	-26.0110	-22.0517	-12.3765	0.063
	313.15	-59.9688	-35.0538	-22.7284	-20.9459	-15.4622	0.068
	318.15	-67.1435	-39.4416	-30.2075	-24.9968	-12.2846	0.097
π_s^E / $10^{-8}$$Pa^{-1}$	303.15	1.3126	-0.1306	-0.1865	0.3043	0.8675	0.003
	308.15	1.2734	-0.0888	0.1007	0.0383	0.2706	0.001
	313.15	1.2598	-0.1125	-0.2198	0.0508	0.7190	0.002
	318.15	1.2346	-0.1188	0.1619	0.0603	0.1621	0.003

Increase in temperature causes variation in the excess thermodynamic properties as the local structure of the liquids are destroyed, thus affecting their intermolecular free length and kinetic

energy so the values of excess free volume (V_f^E), excess internal pressure (π_i^E), and excess molar available volume (V_a^E) varies with temperature (Table 2). Similar temperature dependence results are reported earlier by several researchers [23,24].

4. CONCLUSIONS

From the observed thermo-acoustic and thermodynamic studies of the binary liquid mixtures of N-methyl-2-Pyrrolidone + Propylamine, + Butylamine, + Dipropylamine at different temperatures and at atmospheric pressure, it is clear that all the three binary systems exhibited non-linear increase /decrease in V_f, and π_i values with the composition of NMP which indicate the presence of intermolecular interactions between the component molecules of the mixtures. The values of V_f^E for all the three binary mixtures are negative and the values of, π_i^E were positive over the entire range of composition of NMP and the temperatures under study. The observed thermoacoustic results suggest a strong interaction between the NMP and substituted benzene under study.

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