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Molecular Interactions Study of Binary Mixtures of Furfural and N,N-Dimethylacetamide at Varying Temperatures

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Abstract:

The density (ρ) and viscosity (η) of binary liquid mixtures of Furfural and N,N-dimethylacetamide (DMA) have been measured over the entire composition range at temperatures of 298.15 K, 303.15 K, 308.15 K, and 313.15 K under atmospheric pressure. From the experimental density and viscosity data, excess molar volumes (V^E) and deviations in viscosity ($\Delta\eta$) were evaluated. The composition dependence of these excess properties was correlated using the Redlich–Kister polynomial equation and is found to be satisfactory. The experimental observations together with the calculated thermodynamic properties were discussed to elucidate the intermolecular interactions existing between the components of the investigated binary mixtures.

Key Words: Density, Viscosity, Molecular Interactions, Excess properties.

1. Introduction:

The investigation of thermodynamic and transport properties of liquid mixtures provides valuable insight into the intermolecular interactions, molecular associations, and structural modifications that accompany the mixing process. Among various solvent systems, binary mixtures containing polar aprotic solvents have been extensively studied because of their importance in numerous industrial applications such as polymer processing, extraction techniques, pharmaceutical manufacturing, coating formulations, and electrochemical technologies.

Furfural and N,N-dimethylacetamide (DMA) are industrially important polar organic solvents whose binary mixtures provide valuable insight into intermolecular interactions and liquid-state structure. Furfural, a biomass-derived heterocyclic aldehyde obtained from agricultural residues and lignocellulosic biomass, contains both a π-electron-rich furan ring and a polar aldehyde group, making it useful in petroleum refining, resin production, pharmaceuticals, agrochemicals, and extraction processes. These structural features enable furfural molecules to participate in various intermolecular interactions, including dipole–dipole and donor–acceptor interactions. DMA, on the other hand, is a highly polar aprotic solvent characterized by a strong carbonyl group, high dielectric constant, and excellent solvating ability. Owing to these properties, DMA is widely employed in polymer processing, fibre manufacturing, pharmaceutical synthesis, coatings, and electrochemical applications.

The carbonyl oxygen atom of DMA acts as an effective electron donor, promoting strong interactions with a variety of organic molecules.

The binary mixture of furfural and DMA is particularly interesting from a physicochemical standpoint because both molecules possess polar carbonyl functionalities capable of engaging in significant dipole–dipole interactions. Furthermore, differences in molecular size, shape, and structural characteristics between the two components may influence molecular packing, free volume, and local arrangement within the liquid mixture. The balance between unlike molecular interactions and the disruption of the pure-component structures can lead to non-ideal mixing behavior. Such effects are commonly reflected in excess and deviation thermodynamic and transport properties, which serve as sensitive indicators of the strength and nature of intermolecular interactions as well as the structural modifications occurring upon mixing. Therefore, the study of volumetric and viscometric, properties of the furfural + DMA system over a range of temperatures provides valuable information regarding molecular association and the resulting liquid-state behaviour of the mixture.

The present work aims to investigate the volumetric and viscometric behaviour of binary mixtures of Furfural and N,N-dimethylacetamide (DMA) over the entire composition range at temperatures of 298.15 K, 303.15 K, 308.15 K, and 313.15 K. Experimental measurements of density and viscosity were employed to evaluate excess molar volume (V^E) and viscosity deviation ($\Delta\eta$). The study seeks to elucidate the nature and strength of intermolecular interactions, molecular packing effects, and structural modifications occurring upon mixing, as well as the influence of temperature on these interactions.

2. Experimental:

2.1 Chemicals

Chemicals used in this study were of analytical grade, N,N-Dimethylacetamide (SRL, mass fraction purity > .995), Furfural (SRL, mass fraction purity > .99).

2.2 Apparatus & Method

The binary mixtures were prepare by mixing known masses of pure liquids. All the mass measurements were performed on an electronic balance (MTL224, HSCo, India) accurate to ±0.1 mg. The samples for measurements were immediately use after preparation & the mixtures were stored in amber glass bottles to avoid contamination and evaporation. Density measurements were made using a single-capillary pycnometer made of borosil glass having a bulb capacity of 10 cm^3. The precision of density measurements was 0.2 Kg/m^3. The measurements were replicate at least three times for each measurement and the results reported are the average value. The viscosities of pure liquids & their mixtures were measured by using Ubbelohde type suspended level viscometer. The viscometer was calibrated with triple distilled water. The viscometer containing the test liquid was allowed to stand for about 15 min. in a constant water bath so that the thermal fluctuations in viscometer were minimized. The time of flow was recorded in triplicate with a digital stopwatch with an accuracy of ±0.01 s. The temperature was maintained by an electronically controlled constant water bath supplied by Vidya Udyog to an accuracy of $\pm 1^0$C.

The ultrasonic velocity in pure liquids and their binary mixtures were measured using a single-crystal variable path multi-frequency ultrasonic interferometer (ASICO, Haryana, India) operating at 2 MHz & calibrated with triple distilled water, benzene & toluene. The ultrasonic data were reproducible within ±0.1 m/s. The reliability of experimental measurements of ultrasonic velocity data ascertained by comparing the data of pure liquids with the corresponding values, which were available in the literature [1-4].

3. Results and Discussion:

The experimentally determined values of density(ρ) and viscosity (η) at 298.15 K to 308.15 K of all pure liquids have been compared with the literature values [5-14] in Table 1. The experimentally measured values of density, viscosity and evaluated values of excess molar volume and deviation in viscosity for the system studied have been presented in Table 2. This non-linear variation is a

deviation from ideal behaviour that suggests interactions between molecules of component liquids of the mixtures.

Excess molar volumes, V^E, are calculated from observed densities of pure components and binary mixtures using, [15]

$$V^E = (x_1M_1+x_2M_2)/\rho_{12} - x_1 M_1/\rho_1 - x_2M_2/\rho_2 \quad (1)$$

Where M_1, x_1, M_2 and x_2 are molecular weight, mole fraction and densities of component 1 and 2 respectively ρ_{12} is mixture density.

The sign and magnitude of V^E varies with physical, chemical and structural characteristics of the liquid components on mixing. The negative V^E arises due to dominance of the following factors: [16]

(a) Chemical interaction between constituent molecules such as hetero-molecular association through the formation of H-bond, often termed as specific interaction.

(b) Association through weaker physical forces such as dipolar force or any other force of this kind.

(c) Accommodation of molecules of one component into the interstitial positions of the structural network of molecules of the other component.

(d) Geometry of the molecular structure that favors fitting of the component molecules with each other.

It is expected that the dispersion forces should make positive contributions to excess values while dipole-dipole, dipole induced dipole, charge-transfer interaction and hydrogen bonding between unlike molecules should make negative contribution [17].

The excess molar volume (V^E) values for the furfural + DMA binary mixture shown graphically in Fig. 1. were negative throughout the entire composition range, with minimum values of about −0.127, −0.138, −0.155 and −0.166 $cm^3 \cdot mol^{-1}$ occurring near the equimolar composition ($x_1 \approx 0.5$) at 298.15, 303.15, 308.15, and 313.15 K, respectively. The negative V^E values reveal that the mixture undergoes the mixture undergoes volume contraction upon mixing, suggesting the presence of significant attractive interactions between furfural and DMA molecules. These interactions are primarily due to to dipole–dipole attractions between the polar carbonyl groups of the two components.

As the temperature increases, the magnitude of the negative V^E values becomes larger, suggesting that the molecules pack more efficiently at higher temperatures, resulting in a greater reduction in free volume. This may be due to temperature-assisted molecular rearrangement, which allows unlike molecules to approach each other more closely and form a more compact liquid structure. The pronounced minimum in V^E observed near the equimolar composition indicates that heteromolecular interactions are strongest in this region, leading to maximum association between furfural and DMA molecules and consequently the greatest degree of volume contraction.

For the investigated system, over the whole composition range a decrease in V^E with increasing temperature from 298.15 K to 313.15 K is observed, same temperature dependence is detected for other mixtures too [9,20-22].

The deviation in viscosity, $\Delta\eta$, was calculated by using the relation, [15]

$$\Delta\eta = \eta - (x_1\eta_1 + x_2\eta_2) \quad (2)$$

Where η is the viscosity of the binary mixtures. x_1, x_2 and η_1, η_2 are the mole fractions and viscosities of pure components 1 and 2 of the binary liquid mixtures, respectively.

The graphical representation of the deviation in viscosity ($\Delta\eta$) observed for the Furfural + DMA mixture as in Fig. 2 are negative across the entire composition range which indicates that, the viscosity of the mixture is lower than that predicted for an ideal mixture. This suggests that, upon mixing, the original molecular structures of the pure liquids are partially disrupted, leading to a decrease in resistance to flow. The negative $\Delta\eta$ values can be attributed to differences in the size and shape of the constituent molecules, as well as to structural rearrangements that promote greater molecular mobility within the mixture. The largest negative deviations are observed near the equimolar composition, where the concentration of unlike molecular pairs is highest, indicating that these effects are most significant in this region. As the temperature increases, the magnitude of $\Delta\eta$ decreases,

implying that thermal motion gradually reduces the influence of intermolecular interactions and structural ordering. Consequently, the mixture exhibits behavior that is closer to ideality at higher temperatures, as molecular mobility becomes increasingly governed by thermal agitation rather than specific interaction effects.

For the investigated system, over the whole composition range gradual decrease in the magnitude of $\Delta\eta$ with increasing temperature from 298.15 K to 313.15 K is observed, same temperature dependence is detected for other mixtures too [23-25].

The excess or deviation properties were fitted to the Redlich-Kister polynomial equation of the type [18, 19],

$$X^E = x_1 x_2 \sum_{i=0}^{n} A_i (2x_1 - 1)^i \qquad (5)$$

Where X^E represents an excess or deviation property. The coefficients A_i were evaluated using a least-squares method and have been presented in Table 3 along with their standard deviations. The standard deviation (σ) was evaluated by using the following relation [15],

$$\sigma\,(X^E) = [(X^E_{(obse.)} - X^E_{(Calc.)})^2 / (n - p)]^{0.5} \qquad (6)$$

Where n is the total number of experimental points and p is the number of A_i coefficients considered. The values of coefficients A_i evaluated by using least-square method with all points weighted equally and the corresponding values and standard deviation (σ) are listed in Table 3.

The maximum deviations for all investigated properties occur near the equimolar composition ($x_1 \approx 0.5$), where interactions between furfural and DMA molecules are strongest. At this composition, unlike molecular contacts are maximized, resulting in the greatest volume contraction. The negative values of V^E & $\Delta\eta$ throughout the composition range confirm the non-ideal nature of the furfural + DMA mixture. These results indicate attractive intermolecular interactions and compact packing and reduced resistance to flow between the unlike molecules.

Table 1. Densities (ρ) and Viscosities (η) of the Pure Liquids at Different Temperatures.

		ρ / g.cm^{-3}		η / mPa.s	
Pure Liquids	T / K	Expt.	Lit.	Expt.	Lit.
N,N-Dimethylacetamide	298.15	0.9366	0.9365[6] 0.9366[5] 0.93652[7]	0.944	0.9443[6]
	303.15	0.93203	0.932[5,6]	0.879	0.8792[6]
	308.15	0.92952	0.9295[8]	0.806	0.8056[8]
	313.15	0.92691	0.9268[8]	0.769	0.7696[6]
Furfural	298.15	1.15469	1.15467[9] 1.154314[11]	1.484	1.48[10]
	303.15	1.14935	1.149[10] 1.14935[9]	1.366	1.36[10]
	308.15	1.14465	1.1447[13,14] 1.1442[12]	1.271	1.2716[13]
	313.15	1.1368	1.13866[9] 1.1389[12] 1.13829[11]	1.171	1.17[10]

Table 2. Values of Densities (ρ), Viscosities (η), Excess molar volume (V^E) and Deviation in Viscosity (Δη) for Furfural + N,N-Dimethylacetamide at 298.15, 303.15, 308.15 and 313.15 K as a function of mole fraction of x_1.

x_1	ρ ($g.cm^{-3}$)	η (mPa.s)	V^E ($cm^3.mol^{-1}$)	Δη (mPa.s)
298.15 K				
0.0000	0.93660	0.944000		
0.1051	0.95760	0.983000	-0.024	-0.018
0.2005	0.97720	1.018000	-0.055	-0.034
0.3018	0.99840	1.052000	-0.093	-0.055
0.4032	1.02000	1.085000	-0.112	-0.077
0.5029	1.04170	1.128000	-0.127	-0.088
0.6042	1.06390	1.189000	-0.116	-0.081
0.7009	1.08540	1.260000	-0.09	-0.062
0.8014	1.10810	1.346000	-0.055	-0.031
0.9024	1.13150	1.412000	-0.026	-0.019
1.0000	1.15469	1.484000		
303.15 K				
0.0000	0.93203	0.879000		
0.1051	0.95300	0.915000	-0.027	-0.015
0.2005	0.97240	0.939000	-0.057	-0.038
0.3018	0.99360	0.976000	-0.091	-0.05
0.4032	1.01530	1.008000	-0.122	-0.067
0.5029	1.03690	1.042000	-0.138	-0.082
0.6042	1.05900	1.097000	-0.127	-0.076
0.7009	1.08020	1.175000	-0.087	-0.045
0.8014	1.10290	1.235000	-0.051	-0.034
0.9024	1.12630	1.302000	-0.029	-0.016
1.0000	1.14935	1.366000		
308.15 K				
0.0000	0.92952	0.806000		
0.1051	0.95030	0.842000	-0.031	-0.013
0.2005	0.96970	0.867000	-0.064	-0.032
0.3018	0.99070	0.903000	-0.101	-0.043
0.4032	1.01220	0.936000	-0.137	-0.057
0.5029	1.03360	0.972000	-0.155	-0.068
0.6042	1.05550	1.024000	-0.143	-0.063
0.7009	1.07660	1.083000	-0.111	-0.049
0.8014	1.09890	1.150000	-0.068	-0.029
0.9024	1.12190	1.212000	-0.033	-0.014
1.0000	1.14465	1.271000		
313.15 K				
0.0000	0.92691	0.769000		
0.1051	0.94750	0.802000	-0.034	-0.009
0.2005	0.96660	0.833000	-0.069	-0.017
0.3018	0.98730	0.859000	-0.109	-0.031
0.4032	1.00850	0.887000	-0.147	-0.044
0.5029	1.02960	0.915000	-0.166	-0.056
0.6042	1.05120	0.967000	-0.153	-0.045
0.7009	1.07190	1.019000	-0.12	-0.032
0.8014	1.09380	1.074000	-0.074	-0.017
0.9024	1.11640	1.124000	-0.036	-0.008
1.0000	1.13868	1.171000		

Table 3. Coefficients of A_i of the Redlich-Kister Equation and the Standard Deviation (σ) for Furfural + N,N-Dimethylacetamide at 298.15, 303.15, 308.15 and 313.15 K.

Function	T(K)	A_0	A_1	A_2	A_3	A_4	σ
V^E	298.15	-0.5028	0.01042	0.51339	-0.0567	-0.2402	0.00303
	303.15	-0.5513	0.03011	0.89206	-0.0741	-0.8021	0.00396
	308.15	-0.6149	-0.0615	0.76735	0.05767	-0.5593	0.00178
	313.15	-0.6586	-0.069	0.79932	0.06656	-0.5829	0.00164
Δη	298.15	-0.3552	-0.0243	0.62536	0.0288	-0.6064	0.0003
	303.15	-0.3102	0.01613	0.39793	-0.0384	-0.2919	0.00686
	308.15	-0.2629	-0.022	0.26146	0.02362	-0.1325	0.00321
	313.15	-0.2104	-0.0131	0.44125	0.02806	-0.4065	0.00218

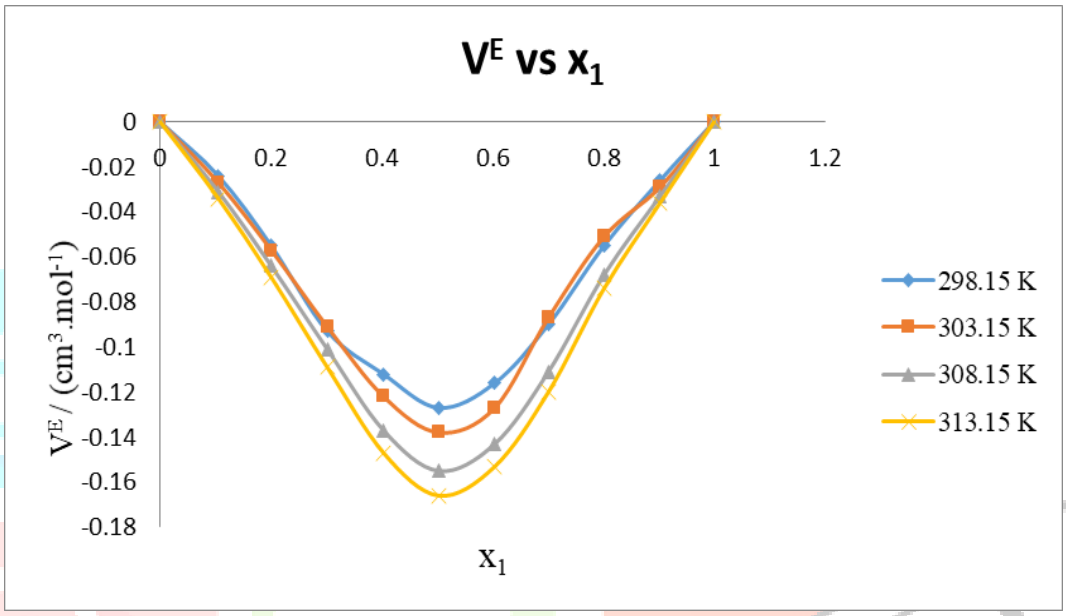

Fig. 1. Excess molar volume (V^E) for Furfural (1) + N,N-Dimethylacetamide(2) from 298.15 K to 313.15 K

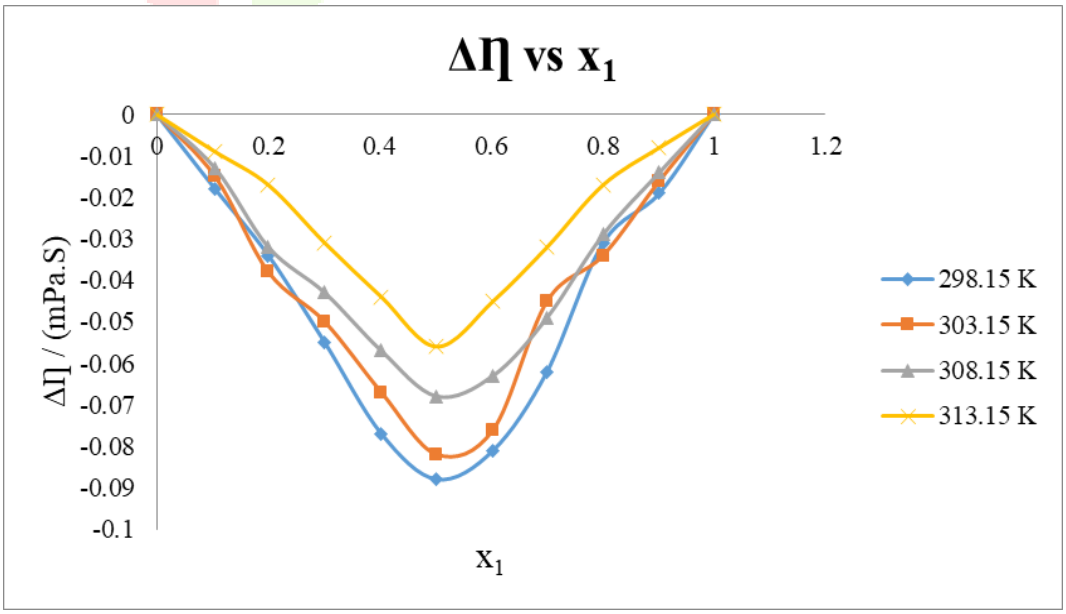

Fig. 2. Deviation in Viscosity (Δη) for Furfural (1) + N,N-Dimethylacetamide(2) from 298.15 K to 313.15 K

4. Conclusion:

In the present study, observed negative excess molar volume (V^E) and viscosity deviation ($\Delta\eta$) values for the furfural + DMA binary mixture reflect noticeable deviations from ideal behaviour due to attractive interactions between unlike molecules. The negative V^E values indicate volume contraction resulting from closer molecular packing, whereas the negative $\Delta\eta$ values suggest that mixing promotes structural rearrangement and greater molecular freedom of movement within the liquid phase. The maximum deviations occurring near the equimolar composition imply that heteromolecular interactions are strongest when the two components are present in nearly equal proportions. Moreover, the increasingly negative V^E values with rising temperature indicate improved packing efficiency, while the reduced magnitude of $\Delta\eta$ points to the gradual weakening of structural effects as thermal motion becomes more significant. Overall, the behaviour of the system is influenced by the combined effects of intermolecular attraction, compact molecular packing, and temperature-dependent structural modifications.

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