

A Comparative Study of 1,3 Diones and Related Oxygen, Nitrogen Heterocycles in Predicting the Acoustical and its Allied Properties in Co-solvents at 293k.

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Abstract: Densities, speed of sound and viscosity were measured for four 1,3 diones and related O,N heterocycles in different cosolvents at 293 K. A quantitative relationship has been established among these thermodynamic properties. The results so obtained are very much comparable and are in good agreement. A molecular interaction study has also been made successfully in the light of these acoustical properties.

Key words: Acoustical impedance; Sound velocity; internal pressure; 1,3 diones and related O,N heterocycles

INTRODUCTION

Excess thermo-acoustical and its allied properties are very helpful in predicting the physico-chemical behaviors and molecular interactions occurring in a variety of liquid mixtures. Successful attempts have been made by several workers (Pal, A., 2003; Bhattacharya, 1985; Pandey, 1998; Hildebrand, 1964; Knopoff, 1970; Aminabhavi, 1987; Aminabhavi, 2006; Ohnishi, 1989) on the measurements and theoretical prediction of various thermo-acoustical properties of liquid binary and ternary mixtures over a wide range of compositions and still in progress (Wankhede, 1995; Treszczanowicz, 1990; Aminabhavi, 1994; Canosa, 2003; Pandey, 2005; Redlich, 1948). In a chemical industry, these properties are very significant in design calculations, heat transfer and mass transfer etc. There has been an increasing interest in the study of molecular interactions and a number of experimental techniques have been used to investigate the interactions in liquid mixtures. Extensive work has been done earlier by many workers (Pandey, 1999; Pal, 1998; Tamura, 2000; Acree, 1995; Beg, 1995; Croucher, 1979) to estimate the acoustical properties of liquid mixtures which are helpful in assessing the molecular size and shape (Pandey, 1999; Pal, 1998), contribution to steric hindrance (Pandey, 1999; Tamura, 2000) coupling of torsional oscillations (Acree, 1985) and extent of non-ideal behaviors (Beg, 1995). The latter, in fact depends on the molecular structure (Croucher, 1979) and intermolecular interactions in different combinations. The role of internal pressure in liquid-solution thermodynamics was recognized by Hildebrand (1964). The use of this property for a long time was qualitative but recently its usefulness has been explored for quantitative study of intermolecular forces. Pioneer attempts have been made by several workers (Dack, M.R.J., 1975; Heric, 1971; Flory, P.J., 1965; Pandey, 1993) to show the significance and its correlation with other properties. The internal pressure has been computed from the knowledge of viscosity (η), density (ρ), and ultrasonic velocity (U) using an indirect method suggested by Pandey (1993). In the present investigation speed of sound, Viscosity and density for 1,3 diones and related O,N heterocycles have been measured over a wide range of compositions. As far as our knowledge is concerned, no one has attempted in predicting the various thermo-acoustical properties for 1,3 diones and related O,N heterocycles so far. Due to scarcity of experimental data, its theoretical applicability could not be done. Also, prediction of internal pressure and its correlation with solubility parameter for mixtures over a wide range of composition at 293 K using experimentally determined value of viscosity, density and ultrasonic velocity.

2. Experimental/materials and Methods

Component liquid dimethyl formamide obtained from the BDH Chemicals Ltd., Poole, England of Anal R grade, were purified and dried in accordance with the usual procedure (Vogel, A.I., 1989). Selected 1,3 diones and related O,N heterocycles were-

A] 1-(2-hydroxy-5-methyl phenyl)-3-(4'-methoxy phenyl) 1,3 propanedione

D] 1-(2-hydroxy-3-bromo-5-methyl phenyl)-3-(4'-methoxy phenyl) 1,3 propanedione

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- E] 3-(2--hydroxy-4-methyl phenyl)-5- styryl isoxazole
 H] 3-(2-hydroxy-5-methyl phenyl)-4- benzoyl-5-(p- methoxy phenyl) isoxazole.

Densities, viscosities and ultrasonic velocities were measured at 293 K over a wide range of compositions. Densities were determined with a bicapillary pycnometer with an estimated error of $\pm 0.012\%$. Ultrasonic velocity measurements were made using a crystal controlled variable path ultrasonic interferometer at a frequency of 2MHz with an accuracy of about $\pm 0.07\%$. The values of viscosity were obtained by using pre-calibrated Ostwald type viscometer with an accuracy of 1.7×10^{-3} cP. All the measurements were carried out using a waterbath thermostatically controlled to ± 0.01 K.

3. Theory and Calculation:

The relative viscosities have been analysed by Jones-Dole (1929) equation.

$$(\eta_r - 1) / \sqrt{c} = A + B \sqrt{c} \quad (1)$$

where, c is molar concentration of the ligand solution, A is the Falkenhagen coefficient which is the measure of solute – solute interactions and B is the Jones-Dole coefficient which is the measure of solute - solvent interaction.

The apparent molal volumes (ϕv) and apparent molal adiabatic compressibilities ($\phi k_{(s)}$) in solutions are determined respectively, from density (ds) and adiabatic compressibility (β_s) of solution using the equation,

$$\phi v = (M/ds) + [(do - ds)10^3] / m ds do \quad (2)$$

$$\text{and } \phi k_{(s)} = [1000 (\beta_s do - \beta_o ds) / m ds do] + (\beta_s M / ds) \quad (3)$$

where, do is the density of pure solvent, m is molality, M is the molecular weight of solute, β_o is adiabatic compressibility of pure solvent and β_s is adiabatic compressibility of solution.

β_s is calculated from ultrasonic velocity using the equation (Ishwara Bhat, 2004).

$$\beta_s = 100 / (Us^2 ds) \quad (3a)$$

and

$$\beta_o = 100 / Uo^2 do \quad (3b)$$

where, Us is the ultrasonic velocity in the solution in m/s. β_s is in bar^{-1} and $\phi k_{(s)}$ is in $\text{cm}^3 \text{mol}^{-1} \text{bar}^{-1}$. The values of $\phi k_{(s)}$ and ϕv were plotted versus $\sqrt{\text{molality}}$ of solutes. The curves represent the least square and ϕv can be given as–

$$\phi v = \phi^0 v + Sv \sqrt{m} \quad (4)$$

and

$$\phi k_{(s)} = \phi^0 k_{(s)} + S k_{(s)} \sqrt{m} \quad (5)$$

where, $\phi^0 v = v^0$ and $\phi^0 k_{(s)} = k^0$ are the infinite dilution apparent molal volumes and apparent molal adiabatic compressibilities respectively. Sv and $Sk_{(s)}$ are the experimental slopes representing ion-ion interactions.

The intermolecular free length (Lf), specific acoustic impedance (z) and relative association (RA) (Laux, D., 2009) are calculated by using the following equation-

$$Lf = K \times \sqrt{\beta_s} \quad (6)$$

$$Z = Us \times ds \quad (7)$$

$$R_A = \frac{ds}{do} \times \frac{Uo}{Us} \quad (8)$$

Table I: Variation of relative viscosity (η_r), adiabatic compressibility(Bs), apparent molal volumes (ϕ_v) and apparent molal adiabatic compressibilities ($\phi_{k(s)}$) with concentration

	C molal	η_r centipoise	Bs $\text{cm}^2\text{dyne}^{-1}$	$\phi_{k(s)}$ $\text{cm}^2\text{dyne}^{-1}\text{mole}^{-1}$	ϕ_v $\text{cm}^3\text{mole}^{-1}$
A- 70% DMF-Water	0.01	2.99657	4.49326E-07	0.323002867	567337.5538
	0.009	3.35	4.69099E-07	0.378231833	644628.8916
	0.008	3.68417	4.78903E-07	0.436084272	731639.6012
	0.006	3.726458	4.85955E-07	0.591459612	980795.6351
	0.004	3.89652	4.91046E-07	0.898182984	1477957.815
	0.002	3.99596	4.97816E-07	1.825084882	2970264.298
	0.001	4.15462	5.10504E-07	3.758114542	5996315.363
D-70% DMF-Water	0.01	2.96867	3.47731E-07	0.23592006	518039.464
	0.009	3.7845	3.80451E-07	0.291842798	582970.1657
	0.008	4.94004	4.08945E-07	0.357194132	661406.2437
	0.006	5.12465	4.25368E-07	0.49840183	885833.3614
	0.004	5.86445	4.34364E-07	0.766508821	1337032.302
	0.002	6.12675	4.45613E-07	1.578749693	2683847.731
	0.001	6.98645	4.68936E-07	3.346848335	5406148.575
E-70% DMF-Water	0.01	5.3	3.9928E-07	0.279533291	539010.2992
	0.009	5.374	3.83936E-07	0.295908861	590129.0449
	0.008	5.379	3.68414E-07	0.316451551	655787.6908
	0.006	5.394	3.65104E-07	0.41745718	873480.2166
	0.004	5.516	2.97466E-07	0.490439015	1293152.78
	0.002	5.67854	2.57164E-07	0.813667201	2527505.442
	0.001	5.764	2.26178E-07	1.351061117	4829208.238
H-70% DMF-Water	0.01	3.25891	3.8948E-07	0.269492641	522658.6733
	0.009	5.78	3.5994E-07	0.27111751	563498.0377
	0.008	6.77	3.42696E-07	0.286336252	622097.3337
	0.006	7.69	3.05223E-07	0.324655506	773774.2442
	0.004	8.2359	2.93154E-07	0.459154732	1132123.333
	0.002	8.35987	2.75757E-07	0.835738072	2165415.192
	0.001	9.33	2.54693E-07	1.468065459	4067055.228

Table II: Values of A, B, $\phi_{k(s)}$, Sk, ϕ_v , Sv for different solutes.

70%	A	B	$\phi_{k(s)}$ $\text{cm}^2\text{dyne}^{-1}\text{mole}^{-1}$	Sk $\text{cm}^2\text{dyne}^{-1}$ $\text{mole}^{-3/2}\text{dm}^{1/2}$	ϕ_v $\text{cm}^3\text{mole}^{-1}$	Sv $\text{cm}^3\text{mole}^{-3/2}\text{dm}^{1/2}$
DMF-Water						
A-	121.2379237	-1049.489913	2.752413888	-43.45180493	6851949.332	-69001671.88
D-	232.2447958	-2191.070873	2.416288372	-39.11255814	6179494.967	-62193158.06
E-	178.5393819	-1452.675921	1.56694609	-13.97019856	5575518.536	-55290277.73
H-	327.5404571	-3044.778407	1.171212705	-15.66934032	4678500.51	-45681472.43

Table III: Variation of Molar compressibility (W), relaxation strength (r), solvation number (Sn), specific volume (V) and the internal pressure (Π) with concentration

	C molal	W	r	Sn	V	Π atm
A- 70% DMF-Water	0.01	2907.940871	-0.103287641	-1820574086	1.269083848	2.03069E+20
	0.009	3068.126469	-0.12187816	-2131866756	1.347254968	1.96142E+20
	0.008	3136.660784	-0.12678225	-2457946372	1.381425355	1.92546E+20
	0.006	3179.950136	-0.128109516	-3333704287	1.403418165	1.89863E+20
	0.004	3219.067657	-0.131830016	-5062520591	1.42279893	1.88204E+20
	0.002	3260.513347	-0.133027191	-10286912536	1.443939066	1.85743E+20
	0.001	3344.93506	-0.137555566	-21182245258	1.486661671	1.81488E+20
D-70% DMF-Water	0.01	3152.944492	-0.165860063	-1699632925	1.037839633	2.69737E+20
	0.009	3191.055292	-0.09241691	-2102515691	1.063965613	2.38647E+20
	0.008	3212.615314	-0.033780563	-2573324650	1.082262795	2.15979E+20
	0.006	3224.361633	-0.003127441	-3590623697	1.092347017	2.04538E+20
	0.004	3256.965912	0.004744359	-5522140109	1.106696621	1.99515E+20
	0.002	3270.573233	0.02224984	-11373746483	1.11538676	1.9276E+20
	0.001	3297.319486	0.056430609	-24111614803	1.13273374	1.79943E+20
E-70% DMF-Water	0.01	2557.141689	-0.100663266	-1536727794	1.125049906	2.2825E+20
	0.009	2492.753568	-0.109598891	-1626752109	1.090599206	2.38334E+20
	0.008	2445.970978	-0.127976754	-1739685071	1.063841105	2.50423E+20
	0.006	2444.29032	-0.135956285	-2294961180	1.061740192	2.53586E+20
	0.004	2453.732746	-0.359264516	-2696177125	1.035097867	3.40468E+20
	0.002	2401.375167	-0.507063141	-4473116597	0.99216001	4.14684E+20
	0.001	2265.183104	-0.586970063	-7427427205	0.91888117	4.83833E+20
H-70% DMF-Water	0.01	3243.116434	-0.058969629	-1994983840	1.055865863	2.29519E+20
	0.009	3099.231115	-0.082770316	-2007012319	0.997715232	2.5113E+20
	0.008	3019.547225	-0.100269879	-2119672704	0.9652696	2.6589E+20
	0.006	2753.806941	-0.108150973	-2403340164	0.865875833	2.99602E+20
	0.004	2675.707172	-0.114608063	-3399002908	0.83648409	3.12843E+20
	0.002	2549.24409	-0.119099516	-6186751301	0.79001422	3.3325E+20
	0.001	2400.287901	-0.127976754	-10867706274	0.735456351	3.62239E+20

Table IV: Variation of Viscous relaxation time(τ), specific impedance(Z), Intermolecular free length(Lf) and relative association (RA)with concentration

	C molal	T s	Zg/s.cm ²	Lf A ⁰	RA
A-70% DMF-Water	0.01	1.79525E-06	1324.262382	40.34375149	0.275274836
	0.009	2.09531E-06	1257.891075	41.2218566	0.258581556
	0.008	2.35248E-06	1229.454776	41.6504033	0.252002123
	0.006	2.41452E-06	1210.900672	41.95593594	0.248004361
	0.004	2.55116E-06	1196.374248	42.1751523	0.244491946
	0.002	2.65234E-06	1179.481905	42.46486669	0.240869993
	0.001	2.82794E-06	1147.873812	43.00265407	0.233792571
D-70% DMF-Water	0.01	1.3764E-06	1664.611704	35.49093257	0.333529024
	0.009	1.91976E-06	1571.761324	37.1231728	0.328886434
	0.008	2.6936E-06	1503.146932	38.48820717	0.326312842
	0.006	2.90648E-06	1467.02465	39.25344304	0.324926383
	0.004	3.39641E-06	1442.310358	39.66637071	0.32113472
	0.002	3.64021E-06	1418.431755	40.17670284	0.319576488
	0.001	4.36827E-06	1372.078844	41.21473336	0.316554247
E-70% DMF-Water	0.01	2.82158E-06	1492.022701	38.0306857	0.310640031
	0.009	2.75103E-06	1545.388985	37.29280754	0.320021219
	0.008	2.64227E-06	1597.325007	36.53118892	0.327173532
	0.006	2.62583E-06	1606.136805	36.36669361	0.327436
	0.004	2.18776E-06	1802.148433	32.82571657	0.325966431
	0.002	1.94709E-06	1979.720993	30.52113173	0.334273022
	0.001	1.73825E-06	2193.537168	28.62337281	0.357836096
H-70% DMF-Water	0.01	1.69237E-06	1559.383685	37.56108425	0.333131144
	0.009	2.77394E-06	1668.712621	36.10859821	0.351244038
	0.008	3.09341E-06	1738.685234	35.23304679	0.3620816
	0.006	3.12955E-06	1945.19807	33.25093343	0.403165023
	0.004	3.21918E-06	2019.404816	32.58691521	0.41692721
	0.002	3.07372E-06	2142.49308	31.60520405	0.441155734
	0.001	3.16838E-06	2310.53821	30.3741319	0.473258069

From the graph of $\phi k(s)$ versus \sqrt{m} , $\phi^0 k(s)$ (thermodynamic parameter i.e., limiting molal adiabatic compressibility) at zero mole fraction have been evaluated, as given in table 2. Viscous relaxation time τ was calculated using the equation;

$$T = 4\eta / 3 ds \times Us^2 \tag{9}$$

Molar compressibility W was calculated using the equation;

$$W = M \times (\beta s)^{-1/7} / ds \tag{10}$$

The relaxation strength (r) was calculated using the equation;

$$r = 1 - (Us/U\alpha)^2 \tag{11}$$

where, $U\alpha = 1600 \text{ ms}^{-1}$

The solvation number (Sn) (Ishwara Bhat, 2004) was calculated using the equation;

$$Sn = - (\phi k(s) / \beta_0 \times M \text{ solute} \times ds) \tag{12}$$

Values of specific volume ($V = 1/ ds$) was also reported in the table and the internal pressure (Π) was calculated by the equation given by Pandey (1993),

$$\Pi = 44.2 \times T^{4/3} \times Us^{3/2} \times ds \tag{13}$$

RESULTS AND DISCUSSION

The value of $\phi k(s)$ are also evaluated at different concentrations of solute in 70% DMF-water medium as shown in Table I. The variation between $(\eta_r - 1) / \sqrt{c}$ versus \sqrt{c} for each system is showing validity of Jones-Dole equation from which B-coefficient can be calculated. The large and small values of 'A' show the stronger and weaker solute – solute interactions respectively. When the solute is introduced into solvent of organic-water mixture, it will interfere with the ordered structure of water in the solute's co sphere. It is evident from Table II that the B-coefficient is an adjustable parameter, which may be either positive or negative and it is a measure of the effective hydrodynamic volume of solute which accounts for the solute-

solvent interactions. It is known as a measure of order or disorder introduced by the solute into the solvent. Solutes with negative viscosity B-coefficient is characterized as "Structure breakers" indicating weak solute-solvent interactions. Such type of results is also shown by Pandey (1993).

It could be observed that the values of $\phi k(s)$ are found to be decreasing with increase in the concentration of solute in case of A and D. The positive values of $\phi k(s)$ show the electrostatic force in the vicinity of ions, causing electrostatic solvation of ions. The apparent molal volume (ϕv) are found to be decreased with increase in the concentration of solute.

Decrease in internal pressure with concentration in case of E and H as shown in table-3 again proves presence of some sort of solute-solvent interactions. It could also be seen from table 4 that intermolecular free length (Lf) decreases linearly on increasing the concentration of solute in 70% DMF-water mixture. As concentration increases, number of ions or particles increases in a given volume leading to the decrease in the gap between two species (Ishwara Bhat, 2004). This indicates that there is a strong interaction between ion and solvent molecules, suggesting a structure promoting behavior of the added solute. This may also imply the decrease in number of free ions, showing the occurrence of ionic association due to strong ion-ion interactions. This fact is again supported from the decrease of β_s with the concentration which may be due to aggregation of solvent molecules around the ions (Kaulgud, M.V., 1961) in case of A and D, while the reverse trend is shown by the solutes E and H.

Relative association (R_A) is influenced by two factors: - (i) the breaking up of the solvent molecules on addition of electrolyte to it, and (ii) the solvation of ions that is simultaneously present, the former resulting in decrease and later increase of relative association. The increase of R_A with concentration suggests that solvation of ions predominates over the breaking up of the solvent aggregate on addition of solutes in case of A and D. But in case of E and H, reverse trend is observed which may be due to the fact that in this case; it might be possible that, the breaking up of the solvent molecules predominates over the solvation of ions. Solvation number is the number of solvent molecules attached to the central ion by surrendering their translational degree of freedom. Positive value of S_n (table III) indicates structure forming tendency of solute i.e. association of solute molecules with the solvent molecules whereas negative value indicates structure breaking tendency of solute.

It is also observed that (table IV) there is a linear variation of R_A with concentration. Z increases linearly with concentration.

Solute E and H shows all together opposite behavior which may due to presence of Nitrogen and Oxygen in a ring and the lone pair of these heteroatom might be interacting with the solvent molecules resulting in altogether different behavior.

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