Molecular Interaction Studies of H-Bonded Complexes of Benzamide in 1,4-Dioxan with Alcohols From Acoustic and Thermodynamic Parameters

[1]AN. Kannappan, [2]R. Kesavasamy and [3]V. Ponnuswamy
[1]Department of Physics, Annamalai University,
Annamalainagar-608002, Tamilnadu, India
[2]Department of Physics, Sri Ramakrishna Engineering College,
Coimbatore-641022, TamilNadu, India
[3]Department of Physics, Sri Ramakrishna Mission Vidhyalaya College of Arts and
Science, Coimbatore-641020, Tamil Nadu, India

Abstract: The ultrasonic velocity, density and viscosity measurements were carried out for the mixtures of benzamide with Propan-2-ol, butan-1-ol, 1-pentanol and n-hexanal in 1,4-dioxan at 303, 308 and 313K. The derived acoustic and thermodynamic parameters namely adiabatic compressibility, free length, free volume, internal pressure, viscous relaxation time and Gibbs free energy were evaluated with a view to investigate the nature of molecular interaction.The obtained results support the occurance of complex formation through intermolecular hydrogen bonding in these ternary liquid mixtures.

Key words: Gibbs free energy, internal pressure, ultrasonic velocity, viscous relaxation time

INTRODUCTION

In recent years, many attempts have been made to study the nature of inter molecular interaction in liquid mixtures namely dipole-dipole[1] ,polar-polar[2], polar-non polar[3] and H-bonding[4]. Several researches[5-9] have studied the complexes of alcohols with ketones, amides, esters, nitriles and amines using ultrasonic velocity measurements. The investigation regarding the molecular association in organic ternary mixtures having alcohol as one of the components is of particular interest, since alcohols are highly polar and self associated through hydrogen bonding in pure state[10]. Amides can be regarded as the templates for protein backbones. Understanding the mutual interaction of amides with hydroxyl groups is important in relation to the conformational stability of proteins. Benzamide is the simplest aromatic carboxylic amide used in the synthesis of various organic compounds and 1,4- dioxan is a heterocyclic organic compound used as an aprotic solvent. Sankar *et al*.[11] have studied H-bonded complexes of benzamide and nicotinamide with alcohols. In this work we have undertaken the investigation of complex formation involving benzamide-alcohol in non-polar solvent.

MATERIALS AND METHODS

The samples of 1,4-dioxan (BDH), Benzamide (NICE) were used without purification. Propan -2-ol ,1-pentanol, butan -1-ol and n-hexanol used were of analar grade. The mixtures of benzamide with propan -2-ol butan -1-ol,1-pentanol and n-hexanol in 1,4-dioxan of different concentration were prepared by molality and converted into mole fraction. In all the mixtures the mole fraction of second component, benzamide was kept fixed while the remaining two (X_1, X_3) were varied so as to have the mixtures of different composition. The ultrasonic velocity was measured by a single crystal interferometer with a high degree of accuracy operating at a frequency of 3MHz (model F-05, with digital micrometer) at 303, 308 and 313K.The viscosity was measured by Ostwald viscometer. An electronically digital operated constant temperature water bath is used to circulate water through the double walled measuring cell made up of steel containing the experimental solution at the desired temperature. Densities of the mixtures have been found by relative measurement method.

Theory and calculation: Intermolecular free length (L_f), is calculated using the standard expression:

$$L_f = K\, \beta^{1/2} \qquad (1)$$

where, K is a temperature dependent constant known as Jacobson constant and β is the adiabatic compressibility that can be calculated from the speed of sound (U) and the density of the medium(ρ) using the equation as:

$$\beta = (u^2\rho)^{-1} \qquad (2)$$

Suryanarayana obtained a relation for free volume in terms of ultrasonic velocity and the viscosity (η) of liquid as:

$$V_f = (M_{eff}U / k\, \eta)^{1/2} \qquad (3)$$

On the basis of statistical thermodynamics Suryanarayana derived an expression for the determination of internal pressure π_i by the use of free volume as:

$$\pi_i = b\, R\, T(K\eta/U)^{1/2} (\rho^{2/3}/M_{eff}^{7/6}) \qquad (4)$$

where, b stands for cubic packing which is assumed to be 2 for liquids and K is a dimensionless constant independent of temperature and nature of liquids and its

value is 4.281×10^9, T is the absolute temperature and M_{eff} is the effective molecular weight.

The viscous relaxation time was obtained using the relation:

$$\tau = (4/3)\, \beta\, \eta \qquad (5)$$

Gibbs free energy is calculated from the relation:

$$\Delta G = K\, T\, \ln(K\, T\, \tau/h) \qquad (6)$$

where, τ is the viscous relaxation time, K the Boltzman constant, T, the absolute temperature and h is the Planck's constant.

RESULTS AND DISCUSSION

The values of density, viscosity and ultrasonic velocity of H-bonded complexes of benzamide with, propan -2-ol, butan -1-ol, 1-pentanol and n-hexanol in 1,4-dioxan of various mole fraction at 303,308and 313K are presented in Table 1.The acoustical parameter such as adiabatic compressibility, free length, free volume, internal pressure, Gibbs free energy and viscous relaxation time are presented in Table 2 and 3.

Table 1: Values of density (ρ), viscosity (η) and ultrasonic velocity (U) at 303,308 and 313K of benzamide with formal concentration of different alcohols

Mole fraction		Density (p)/Kgm^{-3}			Viscosity $\eta/(\times 10^{-3}\text{Nsm}^{-2})$			Velocity (U/ms^{-1})		
X_1	X_3	303K	308K	313K	303K	308K	313K	303K	308K	313K
1,4-dioxan+benzamide+propan-2-ol										
0.9190	0.0000	1037.7	1035.3	1030.9	1.4748	1.3436	1.2355	1361.4	1344.3	1324.9
0.9027	0.0178	1030.7	1030.0	1024.7	1.3991	1.3008	1.2043	1354.2	1336.0	1315.2
0.8869	0.0350	1026.1	1023.8	1019.5	1.3835	1.2913	1.1922	1350.3	1334.2	1313.2
0.8716	0.0516	1021.9	1018.6	1015.9	1.3661	1.2675	1.1809	1347.1	1331.7	1310.1
0.8550	0.0687	1017.6	1015.1	1011.5	1.3396	1.2399	1.1609	1342.2	1328.4	1307.2
0.8423	0.0835	1013.2	1012.4	1007.2	1.3247	1.2137	1.1118	1339.0	1325.7	1303.3
1,4-dioxan+benzamide+butan-1-ol										
0.9190	0.0000	1037.7	1035.3	1030.9	1.4748	1.3436	1.2355	1361.4	1344.3	1324.9
0.9025	0.0178	1031.6	1030.9	1025.6	1.4013	1.3099	1.2239	1356.6	1339.2	1317.9
0.8869	0.0349	1028.9	1024.7	1020.3	1.3883	1.2988	1.1989	1352.1	1338.0	1316.4
0.8704	0.0529	1022.8	1019.5	1016.8	1.3631	1.2748	1.1788	1350.9	1335.0	1315.8
0.8567	0.0678	1018.4	1015.9	1012.4	1.3531	1.2554	1.1669	1346.4	1332.1	1309.8
0.8422	0.0835	1014.1	1013.3	1008.0	1.3398	1.2253	1.1278	1343.4	1329.7	1309.5
1,4-dioxan+benzamide+1-pentanol										
0.9190	0.0000	1037.7	1035.3	1030.9	1.4748	1.3436	1.2355	1361.4	1344.3	1324.9
0.9026	0.0179	1033.3	1031.7	1026.5	1.4353	1.3148	1.2262	1358.8	1341.9	1322.7
0.8867	0.0352	1029.8	1025.6	1021.2	1.4209	1.3073	1.2050	1356.7	1338.9	1317.9
0.8715	0.0516	1023.7	1020.3	1017.3	1.4079	1.2899	1.1947	1353.3	1335.6	1316.4
0.8568	0.0678	1019.3	1016.3	1013.3	1.3907	1.2770	1.1771	1350.9	1334.4	1314.3
0.8425	0.0833	1014.9	1013.3	1008.9	1.3703	1.2699	1.1601	1348.8	1331.4	1310.5
1,4-dioxan+benzamide+n-hexanol										
0.9190	0.0000	1037.7	1035.3	1030.9	1.4748	1.3436	1.2355	1361.4	1344.3	1324.9
0.9027	0.0179	1034.2	1032.6	1028.2	1.4570	1.3302	1.2285	1359.9	1342.2	1323.0
0.8868	0.0350	1030.7	1027.4	1022.1	1.4443	1.3141	1.2131	1358.1	1339.2	1319.7
0.8716	0.0517	1027.2	1021.2	1017.7	1.4202	1.2978	1.2015	1354.8	1336.6	1318.8
0.8568	0.0677	1020.2	1016.8	1014.2	1.3992	1.2916	1.1823	1353.3	1335.0	1315.3
0.8421	0.0837	1015.4	1014.2	1009.8	1.3860	1.2861	1.1688	1350.1	1334.4	1313.0

Table 2: Values of Adiabatic compressibility (β), Free length (L_f) and Free volume (V_f) at 303K, 3078K and 313K of benzamide with formal concentration of different alcohols

Mole fraction		Adiabatic compressibility $(\beta)/(\times 10^{-10}\,m^2\,N^{-1})$			Free length $L_f/(\times 10^{-10}\,m)$			Free volume $V_f/(\times 10^{-7}\,m^3\,Mol^{-1})$		
X_1	X_3	303K	308K	313K	303K	308K	313K	303K	308K	313K
1,4-dioxan+benzamide+propan-2-ol										
0.9190	0.0000	5.1993	5.3451	5.5257	0.4550	0.4650	0.4772	0.8661	0.9773	1.0845
0.9027	0.0178	5.2905	5.4394	5.6416	0.4589	0.4691	0.4822	0.9215	1.0073	1.1044
0.8869	0.0350	5.3429	5.4864	5.6881	0.4612	0.4711	0.4842	0.9249	1.0076	1.1091
0.8716	0.0516	5.3919	5.5359	5.7348	0.4633	0.4732	0.4862	0.9314	1.0244	1.1114
0.8550	0.0687	5.4551	5.5827	5.7849	0.4660	0.4752	0.4883	0.9442	1.0181	1.1250
0.8423	0.0835	5.5045	5.6200	5.8449	0.4681	0.4768	0.4908	0.9507	1.0681	1.1876
1,4-dioxan+benzamide+butan-1-ol										
0.9190	0 .0000	5.1993	5.3451	5.5257	0.4550	0.4650	0.4772	0.8661	0.9773	1.0845
0.9027	0.0178	5.2674	5.4088	5.6137	0.4579	0.4677	0.4810	0.9256	1.0045	1.0858
0.8869	0.0349	5.3160	5.4510	5.6556	0.4600	0.4696	0.4828	0.9435	1.0263	1.1293
0.8704	0.0529	5.3574	5.5038	5.6804	0.4618	0.4718	0.4839	0.9496	1.0314	1.1350
0.8567	0.0678	5.4165	5.5465	5.7574	0.4644	0.4737	0.4871	0.9513	1.0476	1.1399
0.8422	0.0835	5.4642	5.5810	5.7851	0.4664	0.4751	0.4883	0.9579	1.0786	1.1940
1,4-dioxan+benzamide+1-pentanol										
0.9190	0.0000	5.1993	5.3451	5.5257	0.4550	0.4650	0.4772	0.8661	0.9773	1.0845
0.9026	0.0179	5.2410	5.3825	5.5683	0.4567	0.4666	0.4791	0.8989	1.0062	1.0932
0.8867	0.0352	5.2755	5.4390	5.6379	0.4583	0.4690	0 .4820	0.9099	1.0108	1.1153
0.8716	0.0516	5.3339	5.4942	5.6727	0.4608	0.4714	0.4835	0.9183	1.0266	1.1270
0.8568	0.0678	5.3758	5.5254	5.7131	0.4626	0.4728	0.4853	0.9323	1.0402	1.1489
0.8425	0.0833	5.4159	5.5672	5.7709	0.4643	0.4745	0.4877	0.9502	1.0447	1.1683
1,4-dioxan+benzamide+n-hexanol										
0.9190	0.0000	5.1993	5.3451	5.5257	0.4550	0.4650	0.4772	0.8661	0.9773	1.0845
0.9025	0.0179	5.2285	5.3755	5.5562	0.4562	0.4663	0.4785	0.8835	0.9930	1.0950
0.8869	0.0350	5.2602	5.4273	5.6177	0.4576	0.4685	0.4812	0.8963	1.0111	1.1152
0.8715	0.0517	5.3039	5.4808	5.6497	0.4595	0.4708	0.4825	0.9188	1.0307	1.1340
0.8568	0.0677	5.3522	5.5181	5.6990	0.4646	0.4724	0.4846	0.9409	1.0393	1.1606
0.8421	0.0837	5.4027	5.5374	5.7434	0.4638	0.4733	0.4865	0.9539	1.0485	1.1813

From the Table 1, it is found that the ultrasonic velocity, density and viscosity decreases with increase in mole fraction of alcohol for all the four systems. The variation of ultrasonic velocity in solution depends upon the increase or decrease of intermolecular free length after mixing the components, based on a model for sound propagation proposed by Eyring and Kincaud[12]. The decrease in velocity is perhaps due to structural changes occurring in the mixtures resulting in weakening of intermolecular forces[13]. Further the sound speed decreases with increase in temperature at any concentration as rise in temperature leads to less disordered structure and more spacing between the molecules. The decrease in density and viscosity with temperature indicates that decrease in intermolecular forces due to increase in thermal energy of the system, which causes increase in volume expansion and hence increase in free path length.

It is found that from the Table 2, the adiabatic compressibility and free length increase with increasing concentration of alcohol in all the systems. The adiabatic compressibility shows an inverse behavior as compared to ultrasonic velocity. This indicates that there is a significant interaction between solute and solvent molecules. It can be taken as an indication for the formation of complexes. The addition of interacting molecules break up the molecular clustering of the other, releasing several dipoles for interaction, which suggest that hydrogen bonded association, breaks up progressively. In view of greater force of interaction between solute and solvent forming hydrogen Bonding, there will be a increase in free length in the mixture[14].The increase in free length after mixing results in a decrease in ultrasonic velocity .This also in accordance with the expected increase in compressibility following a decrease in ultrasonic velocity showing there by weakening intermolecular interactions. Similar results were also reported in some liquids mixtures[14].

A close look at the values of free volume reveals that presence of solute -solvent interaction. The value of internal pressure decreases with increasing mole fraction as shown in Table 3. Alcohol is a polar, having profuse hydrogen bonding and hence the structural property like water may be there. Because of hydrogen bonding, the molecules must be arranged such that the void spaces may be available due to which the solute is more compressible[15]. On the other hand 1,4 -dioxan is

Table 3: Values of internal pressure (π_i), viscous relaxation time (τ) and Gibbs free energy (ΔG) at 303, 308 and 313K of Benzamide with formal concentration of different alcohols

Mole fraction		Internal pressure $\pi/(\times 10^{-6}\,Nm^{-2})$			Viscous relaxation time $\tau/(\times 10^{-12}\,sec)$			Gibbs free energy $\Delta G/(\times 10^{-20}\,KJmol^{-1})$		
X_1	X_3	303K	308K	313K	303K	308K	313K	303K	308K	313K
1,4- dioxan+benzamide+propan-2-ol										
0.9190	0.0000	577.8	563.3	551.4	1.0224	0.9575	0.9103	0.7796	0.7716	0.7692
0.9027	0.0178	564.5	557.6	548.1	0.9869	0.9434	0.9059	0.7648	0.7652	0.7671
0.8869	0.0350	566.3	557.9	547.5	0.9856	0.9446	0.9041	0.7643	0.7656	0.7663
0.8716	0.0516	566.9	555.0	547.9	0.9821	0.9355	0.9029	0.7628	0.7617	0.7657
0.8550	0.0687	563.3	557.4	546.7	0.9743	0.9229	0.8954	0.7595	0.7550	0.7621
0.8423	0.0835	561.9	549.2	536.8	0.9722	0.9094	0.8664	0.7586	0.7497	0.7479
1,4- dioxan+benzamide+butan-1-ol										
0.9190	0 .0000	577.8	563.3	551.4	1.0224	0.9575	0.9103	0.7796	0.7716	0.7692
0.9025	0.0178	564.2	557.8	550.5	0.9842	0.9447	0.9161	0.7637	0.7658	0.7719
0.8869	0.0349	557.2	549.2	539.0	0.9840	0.9439	0.9040	0.7636	0.7655	0.7662
0.8704	0.0529	563.7	554.0	545.0	0.9737	0.9355	0.8928	0.7592	0.7617	0.7608
0.8567	0.0678	557.7	548.1	540.3	0.9772	0.9284	0.8928	0.7607	0.7584	0.7622
0.8422	0.0835	555.9	542.9	531.6	0.9762	0.9117	0.8699	0.7603	0.7507	0.7496
1,4- dioxan+benzamide+1-pentanol										
0.9190	0.0000	577.8	563.3	551.4	1.0224	0.9575	0.9103	0.7796	0.7716	0.7692
0.9026	0.0179	569.3	556.8	548.5	1.0030	0.9436	0.9104	0.7716	0.7653	0.7692
0.8867	0.0352	565.9	553.9	543.2	0.9994	0.9480	0.9058	0.0770	0.7673	0.7671
0.8716	0.0516	562.1	549.3	540.1	1.0012	0.9449	0.9036	0.7700	0.7659	0.7660
0.8568	0.0678	553.1	545.7	535.4	0.9968	0.9408	0.8966	0.7690	0.7640	0.7626
0.8425	0.0833	552.9	544.0	531.1	0.9895	0.9426	0.8926	0.7659	0.7649	0.7607
1,4- dioxan+benzamide+n-hexanol										
0.9190	0.0000	577.8	563.3	551.4	1.0224	0.9575	0.9103	0.7796	0.7716	0.7692
0.9027	0.0179	571.9	558.6	547.9	1.0156	0.9534	0.9101	0.7768	0.7697	0.7691
0.8869	0.0350	567.1	552.5	541.6	1.0129	0.9509	0.9087	0.7757	0.7686	0.7684
0.8715	0.0517	560.3	546.0	536.3	1.0043	0.9484	0.9051	0.7722	0.7675	0.7667
0.8568	0.0677	552.6	542.2	530.2	0.9984	0.9503	0.8985	0.7697	0.7683	0.7635
0.8421	0.0837	547.0	539.0	524.9	0.9983	0.9495	0.8950	0.7696	0.7680	0.7619

a non-polar having no hydrogen bonding. The reduction in internal pressure may be due to the loosening of cohesive forces leading to breaking the structure of the solute. When the temperature is increased, there is a tendency for the solute molecules to move away from each other, reducing the possibility for the interaction, which may further reduce the cohesive forces and ultimately leads to an increase in the free volume[16] as shown in Table 2.

The viscous relaxation time decreases with increase in mole fraction of alcohol and increase in temperature. The relaxation time that is in the order of 10^{-12} sec is due to the structural relaxation process[17] showing the presence of molecular interaction. Gibbs free energy confirms the same. The reduction of Gibbs free energy in all the systems indicates that the need for smaller time for the cooperative process or the rearrangement of the molecules in the mixtures decreases the energy that leads to dissociation[16].

CONCLUSION

It is concluded that ultrasonic studies provide a comprehensive investigation of molecular association between benzamide with, propan -2-ol, butan -1-ol,1-pentanol and n-hexanol in 1,4-dioxan arising from the hydrogen bonding between the solute and solvent molecules and the order of interaction is found to be n-hexanol> 1-pentanol> butan-1-ol> propan-2-ol.

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