COMPARITIVE STUDY OF BINARY LIQUID MIXTURES CONTAINING P-METHOXY BENZOATE ACID AND AMIDES

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COMPARITIVE STUDY OF BINARY LIQUID MIXTURES CONTAINING P-METHOXY BENZOATE ACID AND AMIDES

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ABSTRACT

The Speed of sound, density and viscosity values of binary mixtures containing P-methoxy benzoic acid (P-MBA) with Amides -Formamide, Di methyl formamide(DMF) and N-Methyl formamide (NMF) are measured from 303.15 K to 313.15K (5K interval) temperatures using standard techniques. Several thermo acoustic parameters like molar volume (V_m), isentropic compressibility (K_s), intermolecular free length (L_f), acoustic impedance (Z), surface tension (χ) and their excess parameters have been calculated from the measured values. These excess parameters have been fitted to Redlich–Kister type polynomial equation using the least square method. The determined values of partial and excess partial molar volumes ($\bar{v}_{m,1}$, $\bar{v}_{m,2}$) and ($\bar{v}_{m,1}^{E}$, $\bar{v}_{m,2}^{E}$) are drawn from V_m^{E} . Finally, FT-IR spectra of these mixtures at different mole fractions were carried out to analyze the hydrogen bonding formation between the molecules in terms of intra or intermolecular relations.

Keywords: Binary liquid mixtures, redlich-kister polynomial, FTIR spectroscopy, excess parameters, measured values

INTRODUCTION

Pure fluids and fluid blends used in this study are effective preservatives in many varieties like antimicrobial food preservative, antifungal as well as antiseptic agents, solvents as cosmetic and perfumery industries and also acts as metabolites in pharmaceutical industries. P-Methoxy benzoic acid ($C_8H_8O_3$) has antiseptic property and it is soluble in water, alcohols, and ethyl acetate because it is available in powder form. It is also known as P-anisic acid and draconic acid. P-Methoxy benzoic acid has a boiling of about 280 °C and a melting of 184°C,

it is a metabolite of aniracetam. Measurement of density and ultrasonic velocity in binary liquid mixtures and its observed results of excess values reflects the molecular interaction in liquid mixtures, which is very useful to build comprehensive calculations like RK polynomials/partial molar volumes for the considered liquids. Similar kinds of results were obtained by the earlier workers [1-3] carried out variety of research investigations on the liquid mixtures. The effects shown by the molecules with other functional groups on these molecules plays a vital role in understanding the behavior of hydrogen bonding in the considered mixture. *EXPERIMENTAL*

P-methoxy benzoic acid (Hi Media Laboratories Pvt. Ltd. Mumbai, India) is available in powder form. It was soluble in water with the mixture of 530 mg/1Lr at room temperature.

The binary liquid mixtures were prepared just before start the experiment for different samples with the help of mass variation. The uncertainty in the final mole fraction was estimated to be less than ± 0.0001 . The speed of sound was measured at 303.15K to 313.15K using ultrasonic interferometer (M/s Mittal Enterprises, India) operating at a fixed frequency of 2 MHz with an accuracy of ± 0.1 m/s. for the binary systems of P-MBA with formamide, DMF and NMF.

The densities of the pure components and their multi components were measured with 10 mL of specific gravity bottle in high accurate digital electronic balance (BaijnathPremnath SF 400A, Kanpur, U.P) with an accuracy of ± 0.02 mg.

The viscosities of pure and compounds for the binary systems of P-MBA with formamide, DMF and NMF were measured with an Ostwald viscometer with an accuracy of 0.1%. The precision of experimentally measured values of density, viscosity, and speed of sound data was compared with their literature values are shown in Table 01

Liquid	Temp	Densit	$y(\rho) \text{ kg m}^3$	Viscosity	γ(η) mPa s	Speed of	f sound(U)	C _P	à
	(K)			-		n	$1.s^{-1}$	JK mol	$k K^{-1}$
		lit	Expt	lit	Expt	lit	Expt	_	
P-Methoxy	303.15	1.1896	1.1886	1.585	1.581	1559.6	1557.2	204.07	1.0004
benzoic acid	308.15	1.1226	1.1246	1.428	1.422	1539.6	1532.6	205.03	1.0013
(a)	313.15	1.0956	1.0966	1.341	1.346	1520.6	1524.4	205.00	1.0028
Formamide	303.15	1.1257	1.1255	2.877	2.784	1588.0	1584.6	107.11	0.9962
(b)	308.15	1.1198	1.1197	2.500	2.461	1577.2	1581.4	105.20	0.9966
	313.15	1.1183	1.1186	2.273	2.251	1568.0	1564.0	108.56	0.9992
NMF	303.15	0.9946	0.9944	1.585	1.566	1408.5	1408.4	124.95	0.9988
(b)	308.15	0.9903	0.9902	1.462	1.474	1400.6	1401.6	126.05	0.9964
	313.15	0.9861	0.9858	1.352	1.361	1382.2	1393.2	129.47	1.0042
	303.15	0.9476	0.9468	0.767	0.770	1459.6	1454.2	152.00	1.0034
DMF	308.15	0.9421	0.9419	0.726	0.732	1434.7	1431.8	153.02	1.0011
(b)	313.15	0.9351	0.9343	0.679	0.681	1420.8	1417.6	157.14	1.0022

Table 1: Comparison of measured to literature values of pure liquids

(a) Reference [4] (b) Reference [5]

RESULTS AND DISCUSSIONS

The excess thermo-acoustical parameters measure the system's non-ideality as a consequence of associative/other interactions between the molecules. Excess properties of studied binary systems at different mole fractions have been calculated from the experimental values of densities (ρ), viscosities (η) and speed of sounds (U) of P-methoxy benzoic acid with formamide, DMF and NMF at temperatures from T = 303.15 K, 308.15 K and 313.15K determined [6] by the equations from (1) to (4)

$$V_{m}^{E} = X_{1}M_{1} + X_{2}M_{2}/\rho - (X_{1}M_{1}/\rho_{1} + X_{2}M_{2}/\rho_{2})$$
(1)

$$K_{s}^{E} = K_{s} - K_{s}^{id}$$
(2)

Benson & Kiyohara [7] suggested a relation for calculating the ideal mixture K_s^{id}

$$K_{s}^{id} = \Sigma \phi_{i} \{K_{s}, i^{0} + T V^{i0(\alpha i0)2} / C_{p}, i^{0}\} - T(\Sigma X_{i} V i^{0}) (\Sigma \phi_{i} \alpha_{i}^{0,2} / \Sigma X_{i} C_{p}, i^{0})$$
(3)

$$L_{f}^{E} = L_{f} - K_{T} (K_{s}^{id})^{1/2}$$
(4)

 K_T be a temperature dependent constant whose value is 91.368 + 0.3565 T x 10⁻⁸

$$Z = U^* \rho$$
(5)
(χ) = 6.3X10⁻⁴. ρ . $U^{3/2}$ (6)

Where ρ is the density ,U be the Speed of sound of the mixture and X_1 , X_2 , M_1 , M_2 , ρ_1 , ρ_2 , Z_1 , Z_2 and χ_1 , χ_2 are the mole fractions, molar mass ,densities, acoustic impedances and surface tensions of actual compounds . In equation (3) T, α_i^{o} , $C_{p,j}^{o}$, and ϕ_i be the Temperature, thermal expansion coefficient, isobaric molar heat capacity and volume fraction of i^{th} component.

Surface tension forces are due to intermolecular forces between the liquid's molecules at the liquid's outer boundaries. Surface tension is a principal factor in capillary action. The higher values over the whole composition for these systems can be suggested that strong molecular interactions are present in these mixtures because of density and acoustic impedance values are varies with mole fraction this results increase the speed of sound. Similar studies were observed for excess surface tension in the considered binary liquids. surface tension (χ) and acoustic impedance (Z) were determined with the help of equation (5,6) reported in Table 2

Mole	PMB	A+ FA	Mole	PMBA	+ DMF	Mole	PMB	A + NMF
fraction			fraction			fraction		
	χ x10 ⁻⁹	$Zx10^3$		χ x10 ⁻⁹	$Zx10^3$		χ x10 ⁻⁹	$Zx10^3$
	$N m^{-1}$	$kg m^{-2} s^{-1}$		$N m^{-1}$	$kg m^{-2} s^{-1}$		$N m^{-1}$	$kg m^{-2} s^{-1}$
				303.15K				
0.0000	8.1948	1.0504	0.0000	8.2228	1.0604	0.0000	8.8460	1.1287
0.2322	8.8022	1.1884	0.2240	9.6492	1.2028	0.2523	9.1522	1.1523
0.4701	9.4501	1.2866	0.4761	10.4616	1.2921	0.4819	10.1538	1.2617
0.6342	10.1328	1.4270	0.6789	11.3744	1.3266	0.6524	11.1603	1.3295
0.8246	11.8998	1.5192	0.8332	13.2249	1.3667	0.8286	12.1517	1.3743
1.0000	12.2796	1.5358	1.0000	14.2151	1.3790	1.0000	13.2651	1.3840
				308.15K				
0.0000	7.9459	1.0549	0.0000	7.9801	1.0842	0.0000	8.1746	1.1359
0.2322	8.9133	1.1984	0.2240	9.2562	1.2129	0.2523	8.7450	1.1644

Table 2 Calculated values of surface tension (χ) and acoustic impedance (Z)

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0.4701	9.1142	1.2499	0.4761	10.0908	1.3013	0.4819	9.7632	1.2735	
0.6342	9.9833	1.3676	0.6789	10.9623	1.3222	0.6524	10.7272	1.3160	
0.8246	11.6549	1.5975	0.8332	12.5275	1.3499	0.8286	11.5844	1.3317	
1.0000	11.9075	1.6099	1.0000	12.9050	1.3851	1.0000	12.4350	1.3553	
				313.15K					
0.0000	7.8777	1.0595	0.0000	7.7763	1.0618	0.0000	8.1729	1.1458	
0.2322	9.3680	1.2672	0.2240	8.9450	1.2217	0.2523	8.4470	1.1705	
0.4701	10.1396	1.3934	0.4761	9.7417	1.3128	0.4819	9.4347	1.2806	
0.6342	10.8890	1.5667	0.6789	10.4494	1.3422	0.6524	10.2812	1.3085	
0.8246	11.7179	1.6647	0.8332	11.7952	1.3775	0.8286	10.9259	1.3295	
1.0000	12.1724	1.6724	1.0000	12.1341	1.3887	1.0000	12.6481	1.3967	

The excess values for the above parameters were fitted to a Redlich- Kister type polynomial by the nonlinear Least – squares method [8]

$$Y^{E} = X_{1} (1-X_{1}) \Sigma^{n}_{i=1} A_{i} (2 X_{1} - 1)^{i-1}$$
(7)

Where $Y^E = Ks^E$, V^E , L_f^E . The values of coefficient A_i were determined by a regression analysis based on the least- squares method.

Standard deviation (σ) = $[\Sigma_{i=1}^{n}(Y_{exp}^{E} - Y_{cal}^{E})/D-N]^{1/2}$ (8)

Where D is number of experimental data points and N is number of parameters





Fig. 1(a-c) Variation of **Excess isentropic compressibility** with respect to mole fraction of PMBA + FA/DMF /NMF at three different temperatures.





Fig. 2(a-c) Variation of **Excess molar volume** with respect to mole fraction of PMBA + FA/DMF /NMF at three different temperatures.





Fig 3(a-c) Variation of **Excess free length** with respect to mole fraction of PMBA +FA/ DMF /NMF at three different temperatures.

The estimations of these abundance parameters K_s^E , V_m^E and L_f^E are fitted to the R-K type polynomial condition and the coefficients A_i are dictated by the strategy for least squares, these qualities alongside the estimations of (σ) are appeared in table 3

By watching the deviations in the estimations of abundance parameters for the blend of double fluid blends under scrutiny, we can foresee the quality of associations between the particles of fluid mixture [9]. The deviations in the estimations of these abundance parameters are because of a few contributing variables, they are (1) The various powers of collaboration between the atoms, similar to the charge move powers and development of hydrogen bonds between particles prompts negative/positive abundance values (2) Geometrical fitting of one atom in to the next because of the varieties fit as a fiddle and size of the particles of segment fluids, it is called structural contribution. (3) Physical inter atomic forces.

	1				
Parameter	Temperature	\mathbf{A}_{0}	$\mathbf{A_1}$	$\mathbf{A_2}$	σ
	(K)				
		P-MBA + Forn	namide		
	303.15	-2.6218	0.2923	1.8532	0.0431
$10^{12} K_{\rm S}^{\rm E} / (m^2 . N^{-1})$	308.15	-3.0613	0.3064	0.9877	0.0498
	313.15	-3.4820	0.4105	0.0953	0.0610
	303.15	2.3742	-0.1227	-1.0137	0.6402
$10^{6} V_{m}^{E} / (m^{3}.mol^{-1})$	308.15	2.7109	-0.0328	0.0471	0.8217
	313.15	3.0296	0.1104	1.0045	0.9956
	303.15	-0.2921	0.1350	0.0668	0.0029
$10^{13} L_{f}^{E} / (m)$	308.15	-0.2131	0.0945	0.0949	0.0028
	313.15	-0.1426	0.0605	0.0782	0.0020

Table 3 Parameters of Redlich-Kister equation (A_k) and Standard Deviation (σ) for P-MBA with FA/DMF/ NMF at different temperatures

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		P-MBA + D	MF			
	303.15	-2.0012	-0.8225	3.1475	0.0409	
$10^{12} K_{\rm S}^{\rm E} / (m^2 . N^{-1})$	308.15	-2.6173	-0.5763	3.1322	0.0516	
	313.15	-3.1030	-0.2168	2.3809	0.0532	
	303.15	3.4078	0.4973	-3.2442	0.8963	
10 ⁶ V _m ^E /(m ³ .mol ⁻¹)	308.15	4.0893	0.5705	-2.1330	1.1268	
	313.15	4.5663	1.5875	-1.2388	1.3810	
	303.15	-0.0215	-0.0047	0.0284	0.2708	
$10^{13} L_f^E / (m)$	308.15	-0.0181	-0.0039	0.0276	0.3779	
	313.15	-0.0276	-0.0064	0.0318	0.4595	
		P-MBA + N	MF			
	303.15	-0.0153	0.0002	0.0052	0.3962	
$10^{12}K_{\rm S}^{\rm E}/({\rm m}^2.{\rm N}^{-1})$	308.15	-0.0242	0.0040	0.0041	0.4864	
	313.15	-0.0273	0.0074	-0.0032	0.5778	
	303.15	0.5758	0.1866	-0.3220	0.1384	
10 ⁶ V _m ^E /(m ³ .mol ⁻¹)	308.15	0.6614	-0.0137	0.0292	0.1456	
	313.15	0.2921	0.0766	-0.2655	0.0668	
	303.15	-0.0143	-0.0019	0.0049	0.0478	
$10^{13} L_{f}^{E} / (m)$	308.15	-0.0197	-0.0042	0.0053	0.0310	
	313.15	-0.0219	-0.0053	0.0082	0.0188	

From the treatment of excess thermo acoustic quantities for liquid mixtures proposed by Desnoyers and Perron [10] in agreement with the original statements of Redlich and Kister from equation (7), table 3 concludes that the excess quantity $(K_s^E, L_f^E \text{ and } V_m^E)$ gives the spring of upshots in the systems it shows the non-ideality in the mixtures but the values of (σ) are less than one that indicates strong interactions at huge suffusion.

The obtained values of partial molar volumes are reported in Table 4 by using the equations (9-12), also tabulated the infinite dilution values of partial molar volume and Excess partial molar volumes at infinite dilution in table 5.

The partial molar volumes for all the binary systems can be calculated using the formulae

$$\bar{V}_{m,1} = V_{m}^{E} + V_{1+X_{2}}^{*} (\delta V_{m}^{E} / \delta X)_{P,T}$$
(9)

$$\bar{V}_{m,2} = V_{m}^{E} + V_{2}^{*} + X_{1} (\delta V_{m}^{E} / \delta X)_{P,T}$$
(10)

Where $\bar{V}_{m,1}$ and $\bar{V}_{m,2}$ are the partial molar volumes and V_1^* and V_2^* molar volumes of pure compounds.

The values of infinite dilution of partial and excess partial molar volumes for the binary system $(V^{E,\infty}_{m,1}, V^{E,\infty}_{m,2})$ can be calculated by the following relations. $V^{E,\infty}_{m,1} = A_0 + A_1 + A_2 + A_2 + \dots = V^{\infty}_{m,1} - V^{*}_{1}$

$$V^{E,\infty}_{m,1} = A_0 + A_1 + A_2 + A_3 + \dots = V^{\infty}_{m,1} - V^{*}_1$$
(11)

$$V^{E,\infty}_{m,2} = A_0 - A_1 + A_2 - A_3 + \dots = V^{\infty}_{m,2} - V^{*}_{2}$$
(12)

Where A₀, A₁, A₂, A₃ are the coefficients obtained from R-K polynomial equation

Mole	PMBA	+ FA	Mole	PMBA	+ DMF	Mole	PMB	A + NMF
fraction			fracti			fraction		
			on					
	$\bar{V}_{m,1} \times 10^{-5}$	$\bar{V}_{m,2}$ x10 ⁻⁵		$\bar{V}_{m,1} \times 10^{-5}$	$\bar{V}_{m,2} x 10^{-5}$		$\bar{V}_{m,1} \times 10^{-5}$	$\bar{V}_{m,2}$ x10 ⁻⁵
				303.15K				
0.0000	3.28	20.14	0.0000	3.13	19.64	0.0000	3.14	18.79
0.2322	4.52	19.26	0.2240	4.24	18.53	0.2523	3.79	17.90
0.4701	4.77	18.14	0.4761	4.58	17.30	0.4819	4.06	16.84
0.6342	4.48	19.50	0.6789	4.78	19.66	0.6524	4.25	17.15
0.8246	5.48	20.76	0.8332	5.35	20.89	0.8286	5.39	18.57
1.0000	6.33	23.46	1.0000	5.84	22.27	1.0000	6.94	21.95
				308.15K				
0.0000	3.88	19.14	0.0000	3.24	16.17	0.0000	4.15	15.66
0.2322	4.15	18.62	0.2240	4.29	15.35	0.2523	4.40	13.64
0.4701	4.89	17.11	0.4761	4.72	14.48	0.4819	4.96	11.58
0.6342	5.05	18.98	0.6789	5.81	16.16	0.6524	5.15	14.77
0.8246	5.87	19.47	0.8332	6.34	18.85	0.8286	6.49	17.82
1.0000	6.37	22.03	1.0000	7.79	21.04	1.0000	7.24	18.88
				313.15K				
0.0000	4.36	19.27	0.0000	8.35	18.19	0.0000	10.63	15.82
0.2322	5.05	18.14	0.2240	9.85	17.44	0.2523	11.13	14.45
0.4701	5.74	17.07	0.4761	10.03	16.18	0.4819	11.63	13.80
0.6342	6.46	18.92	0.6789	10.93	18.76	0.6524	12.37	15.88
0.8246	7.51	19.67	0.8332	11.80	19.20	0.8286	13.39	18.08
1.0000	7.88	22.68	1.0000	11.91	21.44	1.0000	13.88	19.16

Table 4: Partial molar volumes for PMBA with FA/DMF /NMF at different temperat	ures
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Table 5 Values of partial molar volume of the components at infinite dilution $(V_{m,1}^{\infty}, V_{m,2}^{\infty})$ and excess partial molar volume at infinite dilution $(V_{m,1}^{E,\infty}, V_{m,2}^{\infty})$ for all the systems at three different temperatures

System	Temperature(K)	$V_{m,1}^{\infty}X10^{-5}$	$V^{\infty}_{m,2}X10^{-5}$	$\mathbf{V}^{\mathrm{E},\infty}_{~~m,1}\mathbf{X}10^{-5}$	${ m V}^{{ m E},\infty}_{~~m,2}{ m X}10^{-5}$
PMBA +FA	303.15	3.28	23.46	-3.05	-3.32
	308.15	3.88	22.03	-2.71	-2.63
	313.15	4.36	22.68	-3.80	-3.16
PMBA +DMF	303.15	3.18	22.27	-2.49	-2.89
	308.15	3.24	21.04	- 4.55	-4.87
	313.15	4.15	21.44	- 3.09	-3.22
PMBA +NMF	303.15	4.36	21.95	-3.55	-3.41
	308.15	8.35	18.88	-3.56	-3.25

313.15 10.63 19.16 - 3.25 -3.34						
	313.15	10.63	19.16	- 3.25	-3.34	

The molecular interactions present in the P-MBA + amides are also reflected in terms of partial molar volumes. The obtained values are shown in table 4 and table 5, from which it culminates that strong interactions exist throughout the taking liquid mixtures. From these results, it is also observe that the partial molar volumes is proportional with concentration of the composition. Harkot suggested [11,12]that the large negative excess volumes for liquid mixtures are due to geometrical factors, and hydrophilic interactions that give the higher degree of dissociation results were obtained by the increase of specific conductivity in a mixture.

FT-IR SPECTRA

The FT-IR spectra of pure solvents have been done in the wave number region 400-4000 cm⁻¹ and are presented in the (fig 4- fig 7). Partial band assignments of the solvent mixtures by comparing with the pure solvents have been made and compounds are discussed with the mole fractions at different cases considered in three combinations recorded at room temperature. Case (i)30% component (1) and 70% of the component (2),Case (ii)50% of component (1) and component(2) and case(iii) 70% of component(1) to 30% of component(2). For this figures the intensity of an absorption in the IR spectrum can be observed with variation in dipole moment that develops during the vibration. But, vibrations are develop a shift in dipole moment (C=O/C-O/C-H/N-H stretch) for P-MBA+ amides. From this outcome in a more intense absorption produce the analysis of hydrogen bonding formation in between the P-MBA+ amides mixture [13, 14].



Fig 4. FTIR analysis for Formamide



Fig 5 FTIR analysis for N methyl Formamide



Fig 6 FTIR analysis for Di Methyl Formamide



Fig 7 FTIR analysis of p-methoxy benzoic acid

CONCLUSIONS

The speed of sound, viscosity and densities of binary mixtures for P-Methoxy benzoic acid with amides were measured at temperature from 303.15K - 313.15K using standard techniques also compared using the standard relations obtained from the literature. Further various thermo - acoustic parameters were computed by the obtained data, the excess values of molar volume and excess acoustic impedance are found to be positive for all the binary systems over the entire range of composition. The values of excess properties of K_s^E and L_f^E are found to be negative for binary mixtures of P-Methoxy benzoic acid with formamide, DMF and NMF in the whole mole fraction range at the three different temperatures. The results can be explained in terms of the intra and inter molecular H-bonding of amide and the formation of the same between the polar and nonpolar molecules of the liquid mixtures. Further the values of the excess parameters are fitted to the Redlich-Kister polynomial equation and the results are also represented in table 3. Furthermore to showcase the molecular interactions in between methoxy molecules and amides with the upshots of partial molar volumes. Finally, FT-IR analysis of the pure solvent and their mixtures are also reported where the principal bands in the mixture.

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