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Volumetric, Viscometric and Speed of Sound Studies of Binary Mixtures of *Tert*-butyl Acetate with Fluorobenzene, Chlorobenzene and Bromobenzene at (298.15 and 308.15) K and at Atmospheric Pressure 0.087 MPa

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Abstract Volumetric, viscometric and speed of sound studies of binary mixtures of *tert*-butyl acetate with fluorobenzene, chlorobenzene and bromobenzene have been made over the entire range of composition, at (298.15 and 308.15) K and at atmospheric pressure (p = 0.089 MPa). From the experimental values of density, viscosity, and speed of sound, the excess molar volumes V^{E}, deviations in viscosity $\Delta\eta$ and deviation in isentropic compressibility ΔK_S have been calculated. The V^{E} and ΔK_S values are negative while the $\Delta\eta$ the values are positive over the entire composition range for the binary mixtures. The derived parameters have been fitted with the Redlich–Kister polynomial equation. The interaction parameters of McAllister model are used to correlate the experimental values of density, viscosity and speed of sound.

Keywords Excess molar volumes · Deviations in viscosity · Deviations in isentropic compressibility · *tert*-butyl acetate · Benzenes

1 Introduction

The thermodynamic and transport properties of binary liquid mixtures help to identify the nature of interactions in the constituent binaries. The compound *tert*-butyl acetate (*t*-BAc) is used as a solvent in the manufacture of many industrially many important chemicals such as adhesives and thinners. It is a very effective degreasing agent. Fluorobenzene (FB) is useful solvent for highly reactive species and chlorobenzene (CB) is mainly used as an

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intermediate in the production of commodities such as herbicides. bromobenzene (BB) is used to introduce a phenyl group via palladium-catalyzed coupling reactions such as the Suzuki reaction. Also it can be used to prepare the Grignard reagent, phenylmagnesium bromide, and as an ingredient in the manufacture of phencyclidine. A literature survey showed that very little work has been done on the mentioned properties for binary mixtures of halogen substituted benzene with branched esters. The interactions between esters and hydrocarbons were reported [1] for the binary mixtures of butyl acetate with some aromatic hydrocarbons. We have previously reported [2, 3] density, viscosity and speed of sound studies of binary mixtures of *t*-BAc with methanol and hydrocarbons. Herein I present the density, viscosity and speed of sound of *t*-BAc with FB, CB and BB at 298.15 and 308.15 K.

2 Experimental

2.1 Materials

The FB, CB (Sisco Research Lab Pvt. Ltd., purity > 99.5%), BB, acetone (S.D. Fine Chemicals, purity > 99%) and *t*-BAc (Spectrochem Pvt. Ltd., purity > 99%), were used after a single distillation. The purity of the solvents, after purification, was ascertained by comparing their densities, viscosities and speed of sound with the corresponding literature values at 298.15 and 308.15 K (Table 1). Binary mixtures were prepared by mass in air-tight stoppered glass bottles. The masses were recorded on an Adairdutt balance to an accuracy of $\pm$ 1 $\times$ 10^{-4} kg. Care was taken to avoid evaporation and contamination during mixing. The estimated uncertainty in mole fractions are $< 1 \times 10^{-4}$.

2.2 Apparatus

Densities were determined by using a bicapillary pycnometer as described earlier [4]. The calibration of the pycnometer was done by using conductivity water with 0.99705 kg·m^{-3} as its density [5] at 298.15 K. The estimated uncertainty of density measurements of solvent and binary mixtures is 0.1 kg·m^{-3}. At least four measurements were made which had an average deviation of $\pm$ 0.1 kg·m^{-3}.

The dynamic viscosities were measured using an Ubbelohde suspended level viscometer [4], having a flow time of approximately 300 s at 298.15 K. It was first thoroughly cleaned with warm chromic acid so that there were no obstructions in the capillary and the liquid ran cleanly without leaving drops behind. It was then thoroughly washed by drawing distilled water through it followed by distilled acetone and finally dried by aspirating clean hot air through it. The temperature of the thermostat was controlled within 0.02 K. A mechanical stirrer was used to maintain a uniform temperature. A suitable quantity of the liquid under investigation, usually measured exactly, was introduced into the viscometer with a pipette and allowed 10–15 min to reach the temperature of the thermostat. The liquid was then sucked up and released and the time of out flow between the two marks was determined with an electronic stopwatch reading to 0.01 s. The flow time measurement was repeated a number of times (usually 5 or 6). The different readings did not deviate from the mean by more than 0.2 s. To determine the influence of temperature on viscosity, the time of out flow was measured at 298.15 and 308.15 K. The viscometer was calibrated separately at each temperature with exactly the same volume of a liquid of known viscosity

Table 1 Comparison of experimental density (ρ), viscosity (η) and speed of sound (u) of pure liquids with literature values at 298.15 and 308.15 K

Liquids	T (K)	$\rho \times 10^{-3}$ (kg·m^{-3})		η (mPa·s)		u (m·s^{-1})	
		Expt.	Lit.	Expt.	Lit.	Expt.	Lit.
t-BAc	298.15	0.8611	0.86057[a]	0.683		1092	1092.76[b]
	308.15	0.8494	0.84938[a]	0.596		1055	1049.71[b]
FB	298.15	1.0178	1.0188[c]	0.569	0.5821[d]	1168	1166[e]
			1.0187[d]		0.5509[c]		
	308.15	1.0058	1.0062[d]	0.516	0.5193[d]	1136	
CB	298.15	1.1007	1.1008[f]	0.758	0.758[g]	1268	1267.5[h]
			1.1006[i]		0.7562[j]		
	308.15	1.0895	1.0897[j]	0.680	0.679[k]	1236	1237[l]
			1.0896[i]				1233[i]
BB	298.15	1.4877	1.4891[m]	1.076	1.0810[o]	1152	1153.4[n]
			1.48796[f]		1.0698[m]		
			1.4888[n]				
	308.15	1.4741	1.4750[p]	0.921	0.920[q]	1124	1124.1[n]

[a]Ref. [14]
[b]Ref. [15]
[c]Ref. [16]
[d]Ref. [17]
[e]Ref. [18]
[f]Ref. [19]
[g]Ref. [21]
[h]Ref. [22]
[i]Ref. [20]
[j]Ref. [23]
[k]Ref. [24]
[l]Ref. [25]
[m]Ref. [26]
[n]Ref. [27]
[o]Ref. [28]
[p]Ref. [29]
[q]Ref. [30]

and density, usually water, the density and viscosity of which were taken from the literature [5]. From the densities and times of flow, absolute viscosities of the liquid mixtures were calculated with the help of the equation

$$\eta = \rho\left(\frac{at - b}{t}\right),$$

where η is the viscosity, ρ is density of the liquid, t is flow time, and a and b are the constants for a given viscometer.

To evaluate the viscometer's constants, the length of the capillary of the viscometer (ι) term is to be corrected as $\iota' = \iota + 0.05\ r$, where r being the radius of the viscometer capillary. Since ι is much larger (150–160 mm) as compared to r (0.5 mm), i.e., $\iota = \iota'$ and hence the end effect on the viscometer was negligible. Accuracy of the viscosity measurements was ± 0.001 mPa·s.

The speeds of sound (u) in the solutions were measured at a frequency of 2 MHz through the interferometric method (using Mittal's F-81 model) at (298.15 and 308.15) K (± 0.02 K). The performance of the interferometer was checked by measurements of the speed of sound of pure liquids such as water, benzene, methylbenzene, isopropylbenzene, and FB and comparing the results with the precise literature values. The uncertainty in speed of sound measurements is ± 0.1%.

2.3 Results and Discussion

The experimental values of ρ, η and u of mixtures at (298.15 and 308.15) K as a function of mole fraction are listed in supplemental material. The density values have been used to calculate excess molar volumes V^{E} using the equation

$$V^{\mathrm{E}} = \sum_i \left(\frac{1}{\rho} - \frac{1}{\rho_1}\right) x_i M_i, \tag{1}$$

where ρ denotes the density of solution and x_i, ρ_i and M_i are the mole fraction, density and molar mass of component i, respectively. The viscosity deviations $\Delta\eta$ were calculated using the equation

$$\Delta\eta\ (\mathrm{mPa{\cdot}s}) = \eta_{12} - x_1\eta_1 - x_2\eta_2, \tag{2}$$

where η_{12} is the viscosity of the mixture and x_1, x_2 and η_1, η_2 are the mole fraction and the viscosity of pure components 1 and 2, respectively.

The isentropic compressibility κ_S was calculated using the Laplace relation,

$$\kappa_S = \left(\frac{1}{u^2\rho}\right), \tag{3}$$

and the deviation from isentropic compressibility, $\Delta\kappa_S$, was obtained by the relation,

$$\Delta\kappa_S = \kappa_{S12} - \phi_1\kappa_{S1} - \phi_2\kappa_{S2}, \tag{4}$$

where κ_{S12} is the experimental isentropic compressibility of the mixture, ϕ_1, ϕ_2 and κ_{S1}, κ_{S2} are the volume fraction and isentropic compressibility of the pure components. The volume fraction ϕ_i, i = 1, 2, was calculated using the equation

$$\phi_i = \frac{x_i v_i^0}{x_1 v_1^0 + x_2 v_2^0}, \quad i = 1,\ 2, \tag{5}$$

where v_i^0 are the molar volumes of pure components, 1 and 2.

The excess molar volumes and deviations in viscosity and isentropic compressibility were fitted with the Redlich–Kister [6] equation of the type

$$Y = f_1 f_2 \sum_{i=0}^{n} a_i (f_1 - f_2)^i, \tag{6}$$

where Y is either V^E, or $\Delta\eta$, or $\Delta\kappa_S$, and n is the degree of polynomial, the f_1 and f_2 are mole fractions for V^E and $\Delta\eta$ and volume fraction for $\Delta\kappa_S$. Coefficients a_i were obtained by fitting Eq. 6 to the experimental results using a least-squares regression method. In each case, the optimum number of coefficients was ascertained from an examination of the variation in standard deviation σ.

σ was calculated using the relation

$$\sigma(Y) = \left[\frac{\sum (Y_{\text{expt}} - Y_{\text{calc}})^2}{N - n}\right]^{1/2}, \tag{7}$$

where 'N' is the number of data points and 'n' is the number of coefficients. The calculated values of the coefficients a_i along with the standard deviations σ are given in Table 2.

The variation of V^E with the mole fraction x_1 of t-BAc for FB, CB and BB at 298.15 K is represented in Fig. 1. The values of V^E are negative for all the three binary mixtures studied. The negative values of V^E fall in the order BB > CB > FB. The negative values of V^E indicate that there is a contraction in volume during the mixing process. This signifies the presence of weak specific interactions and geometrical fitting of one component into

Table 2 Parameters and standard deviations (σ) of Eq. 6 for t-BAc + FB, + CB, and + BB, at 298.15 and 308.15 K and p = 0.089 MPa

Properties	T (K)	a_0	a_1	a_2	σ
t-BAc + FB					
V^E ($m^3 \cdot mol^{-1}$)	298.15	− 0.6582	0.3217	0.0701	0.0045
	308.15	− 0.9173	0.5404	− 0.1278	0.0058
$\Delta\eta$ (mPa·s)	298.15	0.0956	− 0.0315	0.0043	0.0005
	308.15	0.0771	− 0.0315	0.0043	0.0005
$\Delta\kappa_S$ (TPa^{-1})	298.15	− 93.9577	48.7942	− 3.4167	0.5024
	308.15	− 129.3123	58.8293	− 5.6416	0.4415
t-BAc + CB					
V^E ($m^3 \cdot mol^{-1}$)	298.15	− 0.8458	0.5787	− 0.0327	0.0041
	308.15	− 1.0440	0.7052	0.0062	0.0062
$\Delta\eta$ (mPa·s)	298.15	0.1077	− 0.0425	0.0070	0.0003
	308.15	0.0918	− 0.0387	0.0047	0.0003
$\Delta\kappa_S$ (TPa^{-1})	298.15	− 110.1293	36.3799	− 6.9881	0.4634
	308.15	− 150.6315	21.9409	1.8609	0.445
t-BAc + BB					
V^E ($m^3 \cdot mol^{-1}$)	298.15	− 1.1279	1.0263	0.0590	0.0061
	308.15	− 1.3765	1.0582	0.0482	0.0040
$\Delta\eta$ (mPa·s)	298.15	0.0457	− 0.0253	0.0039	0.0006
	308.15	0.0601	− 0.0370	− 0.0497	0.0065
$\Delta\kappa_S$ (TPa^{-1})	298.15	− 234.805	46.4123	5.8414	0.4907
	308.15	− 281.282	49.1071	11.9851	1.2113

Standard uncertainties u are u(p) = 4 kPa, u(T) = 0.02 K and combined expanded uncertainties Uc are Uc(ρ) = 1 × 10^{-3} g·cm^{-3} and Uc(V^E) = 0.04 $m^3 \cdot mol^{-1}$

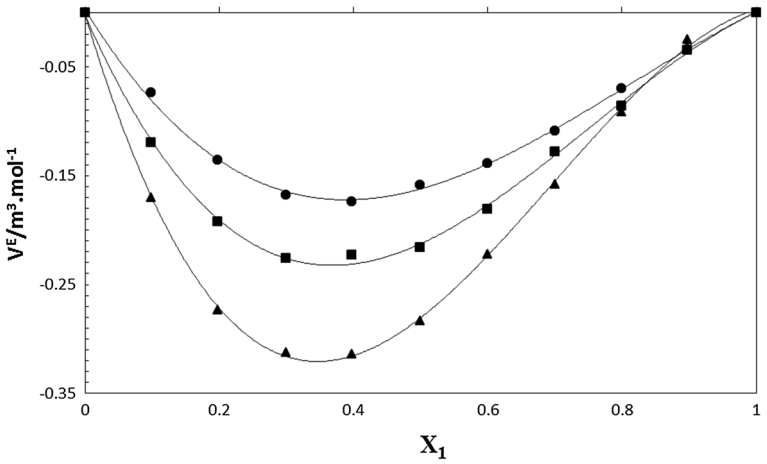

Fig. 1 Excess molar volumes V^E at 298.15 K for (x_1) *t*-BAc + (1 − x_1) benzenes: ● fluorobenzene, ■ chlorobenzene, ▲ bromobenzene

the empty space or voids of the other component. The excess molar volumes show dependences on the temperature.

Figure 2 depicts the variation of $\Delta\eta$ with the mole fraction x_1 of *t*-BAc. The $\Delta\eta$ values are very small and positive for all the three binary mixtures. The $\Delta\eta$ values of many organic liquids with aromatic hydrocarbons are characterised by very low (almost ideal nature) values [7–12]. The positive $\Delta\eta$ values show that there are only weak specific interactions present in the binary mixtures studied. The $\Delta\eta$ values show little or no effect of temperature.

The kinematic viscosities ($\nu = \eta/\rho$) of the binary mixtures of *t*-BAc with FB, CB and BB have been correlated with the help of McAllister's multi body interaction models [13]. The three-body McAllister's model is defined by

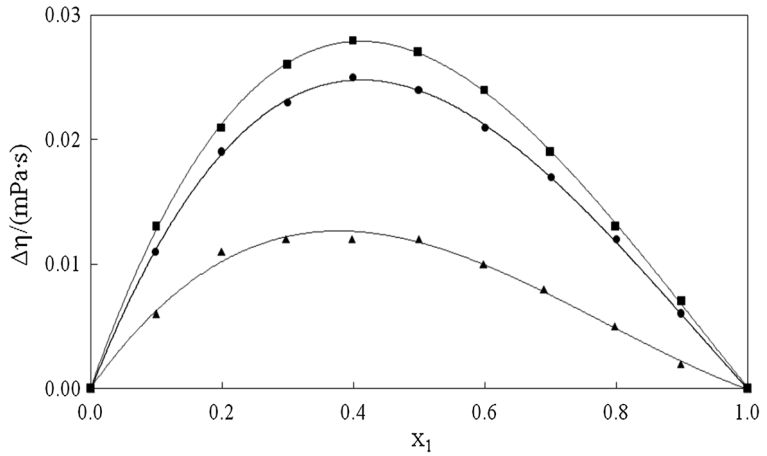

Fig. 2 Deviations in viscosity ($\Delta\eta$) at 298.15 K for (x_1) *t*-BAc + (1 − x_1) benzenes: ● fluorobenzene, ■ chlorobenzene, ▲ bromobenzene

$$\ln v = x_1^3 \ln v_1 + x_2^3 \ln v_2 + 3x_1^2 x_2 \ln v_{12} + 3x_1 x_2^2 \ln v_{21} - \ln\left[x_1 + \frac{x_2 M_2}{M_1}\right] + 3x_1^2 x_2 \ln\left[\frac{2}{3} + \frac{M_2}{3M_1}\right] + 3x_1 x_2^2 \ln\left[\frac{1}{3} + \frac{2M_2}{3M_1}\right] + x_2^3 \ln\left[\frac{M_2}{M_1}\right], \tag{9}$$

where v_{12} and v_{21} are interaction parameters and M_1 and M_2 are the molecular weights of components 1 and 2.

The correlating ability of Eq. 9 was tested by calculating the percentage standard deviation (σ %) between the experimental and calculated viscosity as

$$\sigma\ \% = \left[\frac{1}{(n-m)\sum\{(100(v_{\text{exptl}} - v_{\text{calcd}})/v_{\text{exptl}})^2\}^{1/2}}\right], \tag{10}$$

where 'n' represents the number of experimental points and m represents the number of coefficients. Table 3 lists the parameters for McAllister equation and percentage standard deviations.

The variation of $\Delta\kappa_S$ with mole fraction of t-BAc, x_1, is shown in Fig. 3. The values of $\Delta\kappa_S$ for mixtures of t-BAc with mole fraction, x_1, are also shown in Fig. 3. The values of $\Delta\kappa_S$ for mixtures of t-BAc with FB, CB and BB are negative which indicates that the mixtures are less compressible than the individual components, an observation which is very well supported by the sign of V^E.

The negative values are in the order FB > CB > BB. Both components of all the binary mixtures (t-BAc and halobenzenes) are polar in nature, therefore dipole–dipole interactions are present in the pure components. When a polar component t-BAc is added to another polar components (i.e., halobenzenes), then there is a possibility that the dipole–dipole interactions between the pure components (like molecules) may be disrupted and new dipole–dipole interactions may take place between unlike molecules. If this interaction is strong then the $\Delta\kappa_S$ values should be negative. The negative $\Delta\kappa_S$ values indicate that these dipole–dipole interactions between unlike molecules are stronger than those between the like molecules.

Table 3 Interaction parameters of McAllister model (v_{12}, v_{21}) and standard deviations (σ) at 298.15 and 308.15 K and p = 0.089 MPa

Systems	T (K)	McAllister		
		v_{12}	v_{21}	σ
t-BAc + FB	298.15	0.741	0.691	0.10
	308.15	0.657	0.622	0.04
t-BAc + CB	298.15	0.793	0.781	0.08
	308.15	0.705	0.701	0.03
t-BAc + BB	298.15	0.808	0.782	0.10
	308.15	0.706	0.687	0.58

Standard uncertainties u are u(p) = 4 kPa and u(T) = 0.02 K

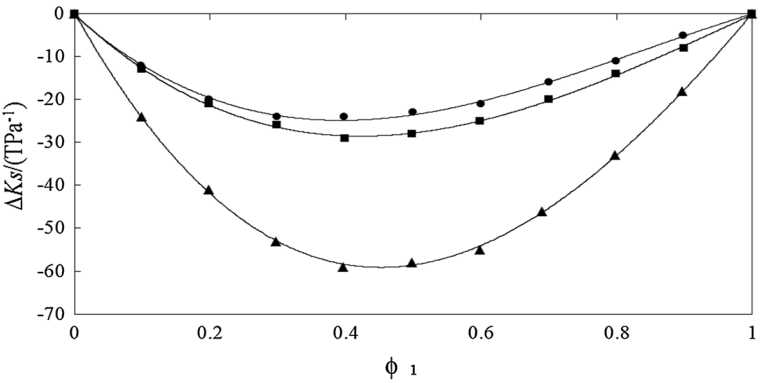

Fig. 3 Deviations in isentropic compressibility (ΔK_S) at 298.15 K for (ϕ_1) *t*-BAc + (1 − ϕ_1) benzenes: ● fluorobenzene, ■ chlorobenzene, ▲ bromobenzene

3 Conclusions

In this work I studied the volumetric properties of binary mixtures of *t*-BAc with FB, CB and BB. Densities were measured over the temperature range at (298.15 and 308.15) K and at pressure 0.087 MPa.

The values of V^E are negative for all the three binary mixtures, which indicates that there is a contraction in volume during the mixing process. The $\Delta\eta$ values are very small and positive for all the three binary mixtures. These values show little or no effect of temperature, which means there are only weak specific interactions present in the binary mixtures. The values of $\Delta\kappa_S$ are negative, which indicates that the mixtures are less compressible, an observation which is very well supported by the sign of the excess molar volumes. For binary mixtures parameters of the Redlich–Kister equation and McAllister's model were fitted to the experimental V^E, $\Delta\eta$ and $\Delta\kappa_S$ values.

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