Thermophysical properties of the binary mixtures of 2-methyl-tetrahydrofuran with benzene and halobenzenes

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Abstract

Densities, speeds of sound, heats of mixing, refractive indices and surface tensions for the binary mixtures of 2-methyl-tetrahydrofuran with benzene, fluorobenzene and chlorobenzene have been determined at atmospheric pressure at the temperature of 298.15 K. Excess molar volumes, excess isentropic compressibilities, excess molar enthalpies, refractive index deviations and surface tension deviations have been calculated from the experimental data and fitted to a Redlich–Kister polynomial function. The Prigogine–Flory–Patterson theory has been used to predict the excess thermophysical properties of the binary mixtures.
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1. Introduction

The interaction between cyclic ethers and different kind of compounds, such as alkanes, aromatic hydrocarbons, alcohols or halogenated compounds have been subject of many research efforts which are still on [1–4]. The analysis of the thermodynamic properties of these kind of liquid mixtures provides useful information about both the molecular interactions and structure and also allows to elaborate models to correlate and predict the behaviour of the mixtures.

Following our systematic study on the thermodynamic properties of binary mixtures containing 2-methyl-tetrahydrofuran [5–13] we present here densities, speeds of sound, isentropic compressibilities, heats of mixing, refractive indices and surface tensions and related mixing thermodynamic properties for binary mixtures of 2-methyl-tetrahydrofuran with benzene, fluorobenzene and chlorobenzene at 298.15 K.

The Prigogine–Flory–Patterson theory [14,15] has been used to analyse the V^E and H^E results. Furthermore, we have also applied this theory to predict the isentropic compressibilities, κ_S, of the liquid mixtures.

As far as we know mixing thermodynamic properties for these systems have not been reported in the literature.

2. Experimental

The compounds used were: 2-methyl-tetrahydrofuran (>99%), benzene (>99.9%), fluorobenzene (>99%) obtained from Aldrich and chlorobenzene (>99.5%) provided by Fluka. The purity of the chemicals was checked by comparing the measured densities and refractive indices with those reported in the literature. No further purification was considered necessary.

The pure compound properties at 298.15 K are collected in Table 1, together with literature values [16–20].

Densities, ρ, of the pure compounds and their mixtures were determined with an Anton Paar DMA-58 vibrating tube densimeter automatically thermostated within ±0.01 K. The accuracy of the densimeter after proper calibration with deionized doubly distilled water and dry air is $\pm 10^{-5}$ g cm^{-3} and the precision of the density measurements was $\pm 5 \times 10^{-6}$ g cm^{-3}.

Speeds of sound, u, were obtained with an Anton Paar DSA-48 vibrating tube densimeter and sound analyzer. The temperature was automatically kept constant within ±0.01 K. The precision of the speed of sound measurements is ± 0.1 m s^{-1}. The accuracy of the speed of sound is ± 1 m s^{-1}. Calibration of the apparatus was carried out with air and deionized double-distilled water.

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Table 1
Physical properties of the pure compounds at 298.15 K and comparison with literature data

Property	2-Methyl-tetrahydrofuran	Benzene	Fluorobenzene	Chlorobenzene
ρ (g cm^{-3})				
Experimental	0.84920	0.87339	1.01879	1.10094
Literature	0.84882[a]	0.87360[b]	1.01872[c]	1.10099[c]
u (m s^{-1})				
Experimental	1202.8	1299.2	1166.0	1267.9
n_D				
Experimental	1.402870	1.497740	1.462902	1.521758
Literature	1.40508[b]	1.49792[b]	–	1.52185[b]
σ (mN m^{-1})				
Experimental	24.68	28.57	27.43	33.00
Literature	–	28.17[d]	26.65[d]	32.90[d]
κ_S (TPa^{-1})				
Experimental	814.0	678.3	722.0	565.0
α (kK^{-1})				
Experimental	1.239	1.213	1.136	0.991
C_P (J mol^{-1} K^{-1})	156.89[e]	135.76[b]	146.36[b]	151.04[f]
κ_T (TPa^{-1})	1109.9	967.3	970.0	762.8
$\tilde{V}$	1.295	1.290	1.275	1.246
P^* (J cm^{-3})	558	622	568	601
s (Å^{-1})	1.25	1.24	1.24	1.23

[a] Ref. [16].
[b] Ref. [17].
[c] Ref. [18].
[d] Interpolated from Ref. [19].
[e] Ref. [11].
[f] Ref. [20].

The corresponding refractive indices at 589.3 nm sodium D wavelength were measured using a high precision automatic refractometer Abbemat-HP DR. Kernchen whose temperature was internally controlled within ±0.01 K. The apparatus was calibrated with deionized double-distilled water. The reproducibility of the measurements is $\pm 1 \times 10^{-6}$ and the corresponding accuracy is $\pm 2 \times 10^{-5}$.

The surface tensions, σ, were determined using a drop volume tensiometer Lauda TVT-2 [21]. The temperature was kept constant within ±0.01 K by means of an external Lauda E-200 thermostat. Details of the experimental procedure can be found in a previous paper [22]. The accuracy of the surface tension measurement is ±0.5% of the final value of surface tension and the corresponding reproducibility is ±0.01 mN m^{-1}.

For all these measurements mixtures were prepared by mass using a Sartorius semi-micro balance with a precision of $\pm 10^{-5}$ g. The possible error in the mole fractions is estimated to be less than 10^{-4}.

Heats of mixing were determined using a Thermometric 2277 thermal activity monitor with a combination measuring cylinder (LKB 2277-204) running under constant flow conditions, total flow rate $= 5 \times 10^{-3}$ mL s^{-1}. During the experiments the temperature is kept constant within $\pm 2 \times 10^{-4}$ K. Two Shimadzu LC-10ADVP HPLC pumps were used to drive the liquids. The pumps were calibrated for each liquid before the start of calorimetric measurements. The uncertainty in the mole fractions of the binary mixtures, calculated from the uncertainty in the flow delivered by the pumps, is ±0.001. The calibration of the calorimeter was achieved with reference to the very accurate H^E of the mixture n-hexane with cyclohexane [23]. This calibration was checked by determining the excess molar enthalpies of the endothermic system benzene + cyclohexane [24] and of the exothermic system 1,4-dioxane + tetrachloromethane [25] and comparing the obtained results with well-established literature data, in both cases the deviations lie within ±1% over the whole composition range. The accuracy in the determination of the heats of mixing could be expected to be ±1%. More details of procedure and calibration can be found in a previous paper [26].

3. Results and discussion

Excess molar volumes, V^E, were calculated from the density of the mixture, ρ, densities, ρ_i and molar masses, M_i, of the pure compounds, and the corresponding molar fractions, x_i, by means of the equation:

$$V^E = x_1 \left(\frac{M_1}{\rho} - \frac{M_1}{\rho_1} \right) + x_2 \left(\frac{M_2}{\rho} - \frac{M_2}{\rho_2} \right) \tag{1}$$

Isentropic and excess isentropic compressibilities were estimated from densities, ρ, and speeds of sound, u, by using the following equations:
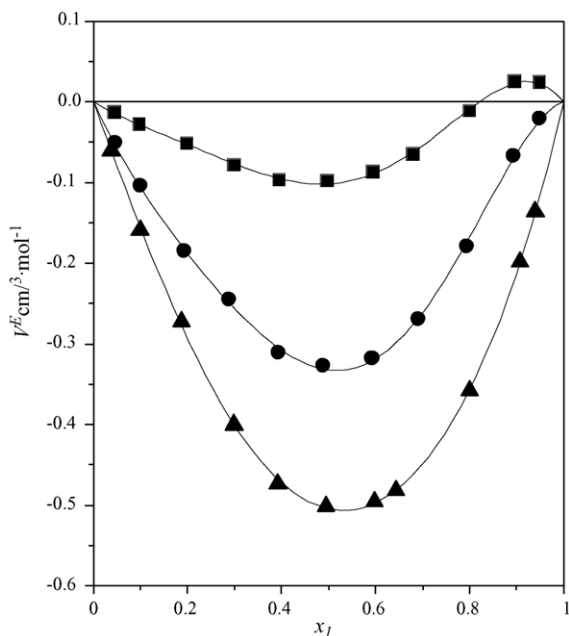
$$\kappa_S = \frac{1}{\rho u^2} \tag{2}$$

Fig. 1. Excess molar volumes, V^E, at 298.15 K for 2-methyl-tetrahydrofuran (1) + aromatic compounds (2): benzene (■); fluorobenzene (●) and chlorobenzene (▲).
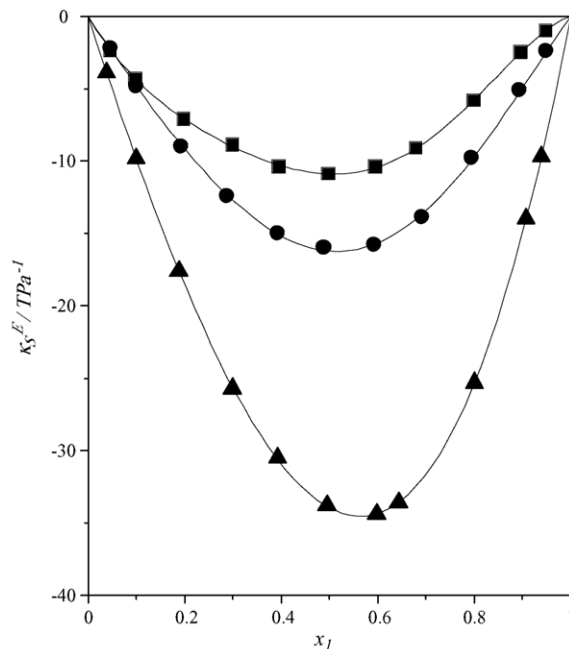

Fig. 2. Excess isentropic compressibilities, κ_S^E, at 298.15 K for 2-methyl-tetrahydrofuran (1) + aromatic compounds (2): benzene (■); fluorobenzene (●) and chlorobenzene (▲).

$$\kappa_S^E = \kappa_S - \kappa_S^{id} \qquad (3)$$

where κ_S^{id} was obtained according to Benson and Kiyohara [27]:

$$\kappa_S^{id} = \sum_i \phi_i \left[\kappa_{S,i} + \frac{TV_i\alpha_i^2}{C_{P,i}} \right] - T \left(\sum_i x_i V_i \right) \frac{\left(\sum_i \phi_i \alpha_i\right)^2}{\left(\sum_i x_i C_{P,i}\right)} \qquad (4)$$

where ϕ_i is the volume fraction of component i in the mixture referred to the unmixed state, x_i the corresponding mole fraction, T the absolute temperature and $\kappa_{S,i}$, V_i, α_i and $C_{P,i}$ are the isentropic compressibility, the molar volume, the thermal expansion coefficient and the molar heat capacity of the pure component i, respectively. All these properties of the pure substances are listed in Table 1.

Excess molar volumes and excess isentropic compressibilities are graphically represented in Figs. 1 and 2.

Excess molar enthalpies, H^E, are given in Table 2 and represented in Fig. 3.

Refractive index deviations, Δn_D, were calculated following the suggestions of Fialkov and Fenerly [28] and Fialkov [29] by means of the equation:

$$\Delta n_D = n_D - \phi_1 n_{D,1} - \phi_2 n_{D,2} \qquad (5)$$

where n_D is the refractive index of the mixture and $n_{D,i}$ is the refractive index of component i.

Finally, surface tension deviations, $\Delta \sigma$, were calculated from surface tension measurements according to the following equation:

$$\Delta \sigma = \sigma - x_1 \sigma_1 - x_2 \sigma_2 \qquad (6)$$

where σ is the surface tension of the mixture and σ_i is the surface tension of the component i.

Refractive index and surface tension deviations are represented in Figs. 4 and 5. The measured thermophysical properties (densities, speeds of sound, isentropic compressibilities, refractive indices and surface tensions) together with the corresponding derived excess or deviation properties can be found in the supplementary material.

The values of each property were correlated with a Redlich–Kister polynomial equation, in this equation $Q = V^E$, κ_S^E, H^E, Δn_D or $\Delta \sigma$, A_i are adjustable parameters and y is the mole fraction for excess molar volumes, excess isentropic compressibilities, excess molar enthalpies and surface tension

Table 2
Excess molar enthalpies, H^E, of the binary mixtures at 298.15 K

| x_1	H^E (J mol^{-1})	x_1	H^E (J mol^{-1})	x_1	H^E (J mol^{-1})
2-Methyl-tetrahydrofuran + benzene					
0.054	−22	0.377	−171	0.783	−139
0.097	−46	0.494	−188	0.890	−82
0.188	−97	0.575	−194	0.944	−45
0.279	−138	0.679	−177		
2-Methyl-tetrahydrofuran + fluorobenzene					
0.056	−80	0.388	−521	0.786	−404
0.101	−156	0.474	−578	0.889	−240
0.195	−312	0.583	−568	0.940	−135
0.288	−431	0.685	−517		
2-Methyl-tetrahydrofuran + chlorobenzene					
0.058	−85	0.391	−572	0.784	−448
0.105	−168	0.488	−628	0.883	−262
0.198	−332	0.585	−623	0.932	−165
0.294	−477	0.683	−575		
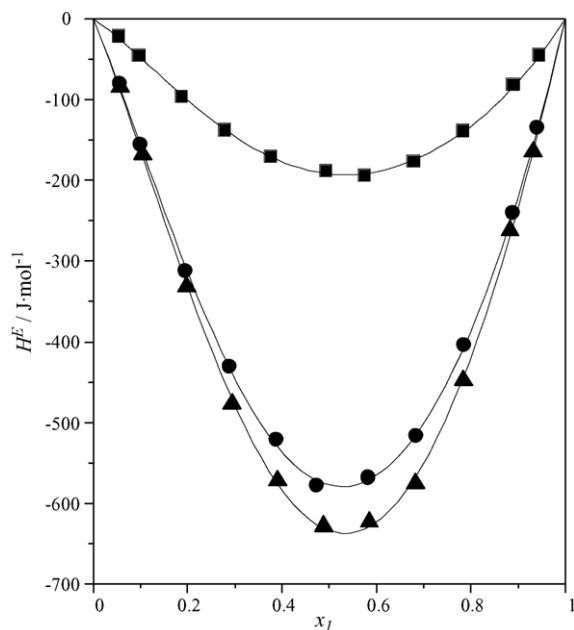
Fig. 3. Excess molar enthalpies, H^E, at 298.15 K for 2-methyl-tetrahydrofuran (1) + aromatic compounds (2): benzene (■); fluorobenzene (●) and chlorobenzene (▲).

deviations or the volume fraction for refractive index deviations.

$$Q = y_1 y_2 \sum_{i=0}^{r} A_i (y_1 - y_2)^i \qquad (7)$$

The values of the parameters A_i, together with the standard deviations $s(Q)$ are given in Table 3.

V^E, κ_S^E and H^E values are negative for the three binary mixtures, except small positive V^E values for the system containing benzene at mole fractions of 2-methyl-tetrahydrofuran bigger
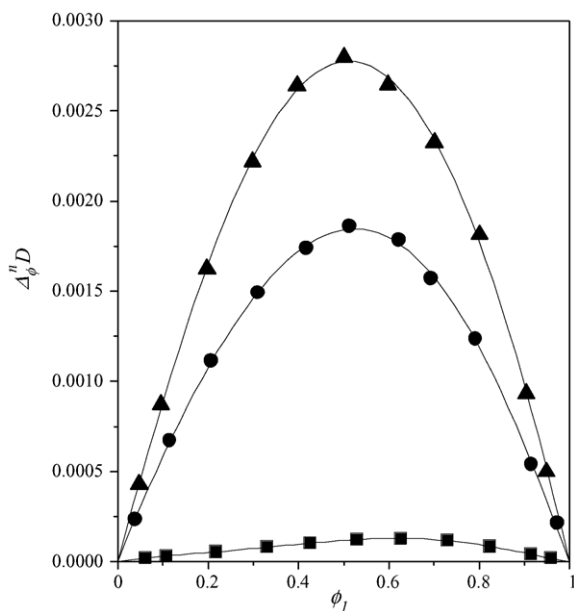

Fig. 4. Refractive index deviations, Δn_D, at 298.15 K for 2-methyl-tetrahydrofuran (1) + aromatic compounds (2): benzene (■); fluorobenzene (●) and chlorobenzene (▲).
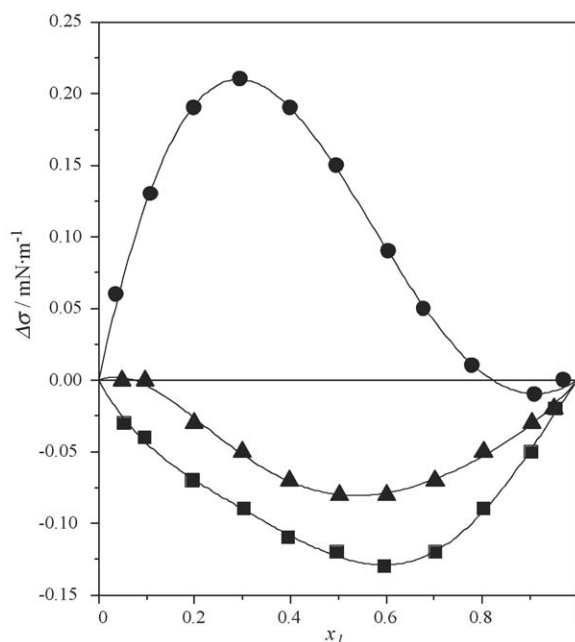

Fig. 5. Surface tension deviations, $\Delta \sigma$, at 298.15 K for 2-methyl-tetrahydrofuran (1) + aromatic compounds (2): benzene (■); fluorobenzene (●) and chlorobenzene (▲).

than 0.8. V^E, κ_S^E and H^E values for the systems containing the halogenated compounds are markedly more negative than H^E values for the system with benzene, being for the system containing chlorobenzene bigger in absolute value than those for the system with fluorobenzene. Minimum values of these properties for all the systems are located at a mole fraction close to the equimolecular composition.

Refractive index deviations are positive in the whole composition range, as shown in Fig. 4. Δn_D values are very small in the system containing benzene. Δn_D values for the systems containing halogenated derivatives, specially chlorobenzene, are bigger than Δn_D values in the system with benzene, and maximum values in these systems appear at a volume fraction close to 0.5. It is remarkable that refractive index deviations show a similar trend, although with opposite sign, that the excess molar volumes.

Finally, as shown in Fig. 5, surface tension deviations are negative for the binary systems containing benzene and chlorobenzene, while the curve is s-shaped for the binary mixture containing flurobenzene, with positive $\Delta \sigma$ values in almost the whole composition range and slightly negative values at mole fractions of the cyclic ether bigger than 0.8.

The observed behaviour for the thermodynamic properties obtained can be analysed in terms of molecular interactions, structural and surface effects. Negative V^E, κ_S^E and H^E values for the system containing benzene indicate the existence of a specific interaction among the mixed components, this interaction takes place between the lone pair of electrons of the oxygen atom of the cyclic ether and the benzene which is a weak electron acceptor. Although the 2-methyl-tetrahydrofuran is a better Lewis base than the tetrahydrofuran [30] the H^E values for the mixture tetrahydrofuran with benzene [31] are more negative,

Table 3
Parameters, A_i, of Eq. (6) and standard deviations, s, for the binary mixtures at 298.15 K

Property	A_0	A_1	A_2	A_3	$s(Q)$
2-Methyl-tetrahydrofuran + benzene					
V^E (cm^3 mol^{-1})	−0.406	0.060	0.592	0.474	0.002
κ_S^E (TPa^{-1})	−43.6	−1.8	9.2	24.8	0.1
H^E (J mol^{-1})	−768	−120	105	−134	3
Δn_D	0.000473	0.000354	−0.000044	−0.000362	0.000007
$\Delta\sigma$ (mN m^{-1})	−0.49	−0.23	−0.04	0.30	0.004
2-Methyl-tetrahydrofuran + fluorobenzene					
V^E (cm^3 mol^{-1})	−1.328	−0.140	0.604	0.682	0.004
κ_S^E (TPa^{-1})	−64.9	−5.3	16.6	7.2	0.1
H^E (J mol^{-1})	−2309	−290	343	−257	5
Δn_D	0.007370	0.000786	−0.000805	−0.000593	0.000022
$\Delta\sigma$ (mN m^{-1})	0.58	−1.03	0.09	0.15	0.002
2-Methyl-tetrahydrofuran + chlorobenzene					
V^E (cm^3 mol^{-1})	−2.017	−0.250	−0.036	−0.216	0.004
κ_S^E (TPa^{-1})	−135.8	−35.5	−4.1	0.4	0.1
H^E (J mol^{-1})	−2543	−405	523	−152	6
Δn_D	0.011010	0.000587	−0.001107	0.000155	0.000031
$\Delta\sigma$ (mN m^{-1})	−0.32	−0.07	0.19	−0.18	0.004

probably due to the steric hindrance of the methyl group that hinders the interaction with the aromatic ring.

When an halogen atom is attached to benzene V^E, κ_S^E and H^E values decrease, that is, the specific interactions between the components of the mixture increase. This is due to two effects: first, the halogen atom can interact directly with the oxygen atom, as shown by the negative values for the mixture chloro-cyclohexane with 2-methyl-tetrahydrofuran [9] although this interaction is not too strong. On the other hand, the presence of the halogen atom enhances the electron acceptor character of the fluorobenzene and chlorobenzene with respect to the acceptor properties of the benzene and therefore the $n(O)–\pi$ interaction is higher for the mixtures 2-methyl-tetrahydrofuran with haloben-zene than for the mixture with benzene and this fact leads to more negative V^E, κ_S^E and H^E values.

It has been reported before [32] that, for a system, values of V^E and Δn_D have an opposite sign and similar trend when Δn_D are calculated in a volume fraction basis. It is confirmed in the three binary mixtures analysed here. Consequently, we can assume that Δn_D values are the result of the above discussed factors.

In a general way, $\Delta\sigma$ values should be negative for any mix-ture, because the compound which is more surface active should be displaced to the surface while the other compound should stay in the bulk. The more difference between surface tension of the pure components, the more negative surface tension devia-tions should be [33]. Although, during the mixing process, other effects such as interactions between unlike molecules may take place. In our study, the three mixtures show small absolute values of $\Delta\sigma$, which indicate that unlike interactions produce a lower adsorption on the surface of the more surface active molecules (aromatic compounds in our case). As discussed above, specific interactions are less important in the system containing benzene, which leads to the more negative $\Delta\sigma$ values for the system 2-methyl-tetrahydrofuran with benzene. Following this reasoning

one could expect that $\Delta\sigma$ should be positive for the systems containing the halogenated benzenes, this occurs for the mix-ture containing fluorobenzene that shows positive $\Delta\sigma$ values in almost the whole composition range, however, the great differ-ence between surface tensions of 2-methyl-tetrahydrofuran and chlorobenzene leads to the observed slightly negative $\Delta\sigma$ values for this system.

4. Prigogine–Flory–Patterson (PFP) theory

The Prigogine–Flory–Patterson theory considers excess ther-modynamic properties of binary mixtures to be the sum of dif-ferent contributions. The excess molar volume can be expressed as: an interactional term which is proportional to the interaction parameter, χ_{12}, a free volume contribution which arises from the dependence of the reduced volume upon the reduced tempera-ture as a result of the differences between the degrees of thermal expansion of the components and an internal pressure contribu-tion which depends both in the difference of internal pressures and of the reduced volumes of the components:

$$\frac{V^E}{x_1 V_1^* + x_2 V_2^*} = \frac{(\tilde{V}^{1/3} - 1)\tilde{V}^{2/3}\psi_1\theta_2\chi_{12}}{[(4/3)\tilde{V}^{-1/3} - 1]P_1^*}$$
$$- \frac{(\tilde{V}_1 - \tilde{V}_2)^2[(14/9)\tilde{V}^{1/3} - 1]\psi_1\psi_2}{[(4/3)\tilde{V}^{-1/3} - 1]\tilde{V}}$$
$$+ \frac{(\tilde{V}_1 - \tilde{V}_2)(P_1^* - P_2^*)\psi_1\psi_2}{P_1^*\psi_1 + P_2^*\psi_2} \tag{8}$$

with respect to the excess molar enthalpy, it can be divided into an interactional contribution and a free volume contribution:

$$\frac{H^E}{x_1 U_1^* + x_2 U_2^*} = \frac{[-\tilde{U} + \tilde{T}\tilde{C}_P]\psi_1\theta_2\chi_{12}}{P_1^*}$$
$$+ \tilde{C}_P[\psi_1\tilde{T}_1 + \psi_2\tilde{T}_2 - \tilde{T}] \tag{9}$$

Table 4
Comparison between experimental and theoretical V^E, κ_S and H^E at equimolecular composition at 298.15 K

System	χ_{12} (J cm^{-3})	V^E (cm^3 mol^{-1})		κ_S (TPa^{-1})		H^E (J mol^{-1})	
		Experimental	PFP	Experimental	PFP	Experimental	PFP
2-Methyl-tetrahydrofuran + benzene	−9.9	−0.101	−0.157	739.8	742.8	−192	−192
Fluorobenzene	−28.8	−0.332	−0.460	753.9	756.0	−577	−577
Chlorobenzene	−30.0	−0.504	−0.573	656.7	662.3	−636	−636

in these equations the reduced magnitudes for the mixture: $\tilde{V}$, $\tilde{T}$ and $\tilde{C}_P$ can be obtained from the reduced configurational energy, $\tilde{U}$, that was calculated, according to Barbe and Patterson [34], in terms of the reduced configurational energies of the pure components by:

$$\tilde{U} = \psi_1 \tilde{U}_1 + \psi_2 \tilde{U}_2 \tag{10}$$

where the contact energy fraction, ψ_i, is defined as:

$$\psi_1 = 1 - \psi_2 = \frac{x_1 P_1^* V_1^*}{x_1 P_1^* V_1^* + x_2 P_2^* V_2^*} \tag{11}$$

all other parameters in the above equations can be obtained using Flory's theory [35–38].

Oswal [39] extended the Prigogine–Flory–Patterson theory to estimate the isentropic compressibilities and speeds of sound of liquid mixtures. At a given temperature, T, the PFP theory can be used to calculate the molar volumes, V, the molar heat capacities, C_P of a liquid mixture if the interaction parameter, χ_{12}, is known. The terms $(\partial V/\partial T)_P$ and $(\partial V/\partial P)_T$ can be also calculated by means of the following equations:

$$\left(\frac{\partial V}{\partial P}\right)_T = \frac{-\tilde{V}^{7/3} + 2\tilde{V}^2 - 2\tilde{V}^{5/3}}{(4/3) - \tilde{V}^{1/3}} \frac{V^* T^*}{P^* T} \tag{12}$$

$$\left(\frac{\partial V}{\partial T}\right)_P = \frac{\tilde{V}(\tilde{V}^{1/3} - 1)}{(4/3) - \tilde{V}^{1/3}} \frac{V^*}{T} \tag{13}$$

where V^*, P^* and T^* are the characteristic volume, pressure and temperature of the mixture, respectively.

From all these quantities the isentropic compressibility, $\kappa_S = -V^{-1}(\partial V/\partial P)_S$, can be obtained using the following thermodynamic relation:

$$\left(\frac{\partial V}{\partial P}\right)_S = \left(\frac{\partial V}{\partial P}\right)_T + TC_P^{-1}\left(\frac{\partial V}{\partial T}\right)_P^2 \tag{14}$$

Flory parameters of the pure compounds along with their physical properties are gathered in Table 1. Thermal expansion coefficients, α, were derived from measured densities in this laboratory. Isothermal compressibilities, κ_T, were calculated from thermal expansion coefficients, experimental isentropic compressibilities and molar heat capacities. The number of contact sites per segment of a molecule, s, has been estimated using the Bondi's method [40].

In this work, the interaction parameter for each mixture was obtained by fitting the PFP theory to the corresponding experimental equimolecular H^E value. Once the interaction parameter is obtained, the thermodynamic properties of the mixture can be estimated.

Table 4 provides the comparison between experimental and theoretical V^E and κ_S value at equimolecular composition, the interaction parameter for each system are also shown. As one can see in Table 4, the estimation of excess molar volumes from H^E values are very satisfactory for the three mixtures. The better predictions for this property correspond to the system containing chlorobenzene, while predictions for the binary mixtures with benzene and fluorobenzene are less accurate. In any case the model is able to predict the trend of this property when changing the aromatic compound in the series.

With respect to isentropic compressibilities there is a excellent agreement between estimated and experimental values, for all the mixtures the deviation from experimental data are less than 0.9%. It can be pointed out that for these mixtures the theory slightly underestimate the isentropic compressibilities of the mixtures.

Acknowledgements

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tca.2005.08.034.

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