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Densities, isobaric thermal compressibilities and derived thermodynamic properties of the binary systems of cyclohexane with allyl methacrylate, butyl methacrylate, methacrylic acid, and vinyl acetate at $t = (298.15 \text{ and } 308.15)$ K

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Abstract

Densities of the binary systems of cyclohexane with allyl methacrylate, butyl methacrylate, methacrylic acid, and vinyl acetate have been measured as a function of the composition, at 298.15 and 308.15 K and atmospheric pressure, using an Anton Paar DMA 5000 oscillating U-tube densimeter. The calculated excess molar volumes were correlated with the Redlich–Kister equation and with a series of Legendre polynomials. The excess molar volumes are positive for the four binaries studied. Within the short temperature range considered here the coefficient of thermal expansion is positive for all the systems studied; it varies only slightly with the nature of the acrylate except for the system cyclohexane + vinyl acetate.

Keywords: Densities; Excess molar volumes; Monomers; Cyclohexane; Methacrylates; Vinyl acetate

1. Introduction

The mixing of different compounds gives rise to solutions that generally do not behave ideally. Excess or residual properties reflect the interactions that take place between solute–solute, solute–solvent, and solvent–solvent species. In general, positive excess molar volumes may be due to the compensation between strong like interactions (such as those present in alcohols) and equally strong unlike H-bond interactions (such as those present between alcohols and ether). Other possible interactions are dipole–dipole and dipole–induced dipole. Size effects, such as free volume and interstitial accommodations reflect not only on excess molar volumes but also on excess enthalpies. Negative excess molar volumes will occur when the unlike interactions prevail over self-association. Binary mixtures are an important class of solvents and solutions and the behavior of some of their physical properties is still not clear. For example, when a solution presents positive excess molar volumes the excess dynamic viscosity is negative, and vice versa. The effect of temperature on the molar volume and excess molar volume can be expressed by the coefficient of excess isobaric thermal expansion, $\alpha = (1/V_\mathrm{m})(\partial V_\mathrm{m}/\partial T)_P$.

This work is part of our program to provide data for the characterization of the molecular interactions between solvents and commercially important monomers, in particular the influence of the chemical structure of the solute in the systems under consideration. So far we have studied the volumetric behavior of the monomers with cyclic hydrocarbons [1], aromatic solvents [2–4] and aliphatic and cyclic ethers [5–7]. Cyclohexane is an aprotic and very stable solvent having zero dipole moment; it may be useful in polymerization and other chemical reactions, in the cleaning of polymer surfaces, electronic materials, etc. The monomers considered in this study are important industrial chemicals used as precur-

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sors in the large-scale preparation of useful polymers and latex systems, and are also interesting because they contain both a double bond and an ester group. Here we report experimental values for the excess molar volumes for the binary systems of cyclohexane (1) with allyl methacrylate (2), butyl methacrylate (3), methacrylic acid (4), and vinyl acetate (5).

González et al. [8] measured the excess volumes of binary mixtures of vinyl acetate with cyclohexane and *n*-alkanes (hexane, heptane, octane, and nonane) at 298.15 K and found all to be positive and to increase slightly with the chain length of the *n*-alkane. Sastry and Dave [9,10] measured the excess volumes, isentropic compressibility, and dielectric behavior of 15 binary mixtures of alkyl (methyl, ethyl, and butyl) methacrylate with hexane, heptane, carbon tetrachloride, chlorobenzene, and *o*-dichlorobenzene at 308.15 K and found that with aliphatic hydrocarbons, the results were controlled by dispersing interactions while with chlorinated solvents the controlling factors were specific interactions (O–Cl and n–π types). Sastry and Valand [11] measured the excess volumes of alkyl (methyl, ethyl, and butyl) acrylates in several alkanols at 298.15 and 308.15 K, and found that they were always positive. These results were explained on the basis of non-specific interactions between the components. Luo et al. [12] measured the excess volumes of methyl methacrylate with cyclohexane at 298.15 K and found them to be positive, Sastry et al. [13] measured the excess volumes of methyl methacrylate with cyclohexane and aromatic hydrocarbons, such as benzene, toluene, ethylbenzene, and (*o*, *m*, *p*)-xylene at 298.15 and 303.15 K and found that except for toluene all presented positive excess volumes. The excess volumes for the system methyl methacrylate + cyclohexane showed a non-symmetric behavior with a maximum at about $V^E \approx 1.05\ \mathrm{cm^3 \cdot mol^{-1}}$ at $x \approx 0.45$. Oswal et al. [14] measured the excess volumes of methyl methacrylate with hydrocarbons, alkyl chlorides, aromatic hydrocarbons, alkyl chlorides, and alkyl amines at 303.5 K and explained the results on the varying extent of molecular interactions and interstitial accommodation between unlike components.

To the best of our knowledge no other literature data are available for the excess molar volumes of the systems reported here.

2. Experimental

Cyclohexane, mass fraction 0.9994; allyl methacrylate (AMA), mass fraction 0.995 (stabilized with 50–185 ppm of monomethyl ether of hydroquinone); butyl methacrylate (BM), mass fraction 0.995 (stabilized with 10 ppm of monomethyl ether of hydroquinone); methacrylic acid (MA), mass fraction 0.9999 (stabilized with 100–250 ppm of hydroquinone); vinyl acetate (VA), mass fraction 0.998 (stabilized with 3–5 ppm of hydroquinone) were purchased from Aldrich. The supplier certified the purity of all the reagents. AMA, BM, MA, and VA were vacuum distilled previous to use, to eliminate the stabilizer. After purification all reagents were stored under molecular sieves. The purity of the solvents was further ascertained by comparing their densities at 298.15 and 308.15 K with the values reported in the literature (Table 1).

The density of the samples was measured with an Anton Paar model DMA 5000 oscillating U-tube densimeter, provided with automatic viscosity correction, and two integrated Pt 100 platinum thermometers (DKD traceable) with a stated accuracy of $5 \times 10^{-6}\ \mathrm{g\,cm^{-3}}$. The temperature of the densimeter was regulated to ±0.001 K with a solid-state thermostat. The densimeter was calibrated daily with both dry air and bi-distilled degassed water.

All liquids were boiled or heated to remove dissolved air. Solutions of different compositions were prepared by mass in a $10\ \mathrm{cm^3}$ rubber-stoppered vial to prevent evaporation, using a Mettler AG 204 balance accurate to $\pm 10^{-4}$ g. To minimize the errors in composition, the heavier component was charged first and the sample kept in ice water. Total uncertainty (ISO 9001) in the mole fraction is 9.95×10^{-5}; precision of the density (duplicate) measurement $\pm 2 \times 10^{-6}\ \mathrm{g\,cm^{-3}}$, and of the temperature ±0.002 K. Total uncertainty in the density

Table 1
Purity and densities of pure components at 298.15 and 308.15 K

Component	Purity (mass %)	Density (g cm^{-3})			
		298.15 K		308.15 K	
		Measured	Literature	Measured	Literature
Cyclohexane (1)	99.94	0.773780	0.7737 [13] 0.77368 [20]	0.764303	0.7645 [13] 0.7646 [21] 0.76424 [20]
Allyl methacrylate (2)	99.5	0.927997	0.9287 [24]	0.917423	
Butyl methacrylate (3)	99.9	0.890438	0.891 [22] 0.8909 [23]	0.880857	0.88149 [22]
Methacrylic acid (4)	99.99	1.009127	1.012 [23]	0.998641	1.00161 [22]
Vinyl acetate (5)	99.8	0.925775	0.92567 [20]	0.913134	0.91384 [19]

measurement, as reported by the equipment manufacturer was 5×10^{-6} g cm^{-3}.

Proper safety measures were taken when handling all the materials.

3. Results and discussion

For every binary system 21 density measurements were performed (with repetition) at each temperature in the full mole fraction range ($0 \le x \le 1$). The excess molar volumes V_m^E of the solutions of molar composition x were calculated from the densities of the pure liquids and their mixtures according to the following equation [15]:

$$V_m^E = \frac{x_1 M_1 + x_2 M_2}{\rho} - \left[\frac{x_1 M_1}{\rho_1} + \frac{x_2 M_2}{\rho_2}\right] \qquad (1)$$

where ρ and ρ_i are the densities of the solution and pure component 'i' in g cm^{-3}, and M_i the molar mass of pure component i in g mol^{-1}. The corresponding values of ρ and V_m^E are reported in the Supplementary Tables 1–4 and in Figs. 1 and 2.

The first term in Eq. (1) represents the actual molar volume of the solution and the second, the molar volume it would occupy if the mixture behaved ideally. In general, while these two molar volumes are similar in size (usually larger than 100 cm^3 mol^{-1}) their difference is usually smaller by two to three orders of magnitude and thus may carry a significantly larger error.

Partial molar volumes for the binary system were calculated using the relations [15]:

$$\bar{V}_{m1} = V_m + x_2 \left(\frac{\partial V_m}{\partial x_1}\right)_{P,T} \qquad (2)$$

$$\bar{V}_{m2} = V_m - x_1 \left(\frac{\partial V_m}{\partial x_1}\right)_{P,T} \qquad (3)$$

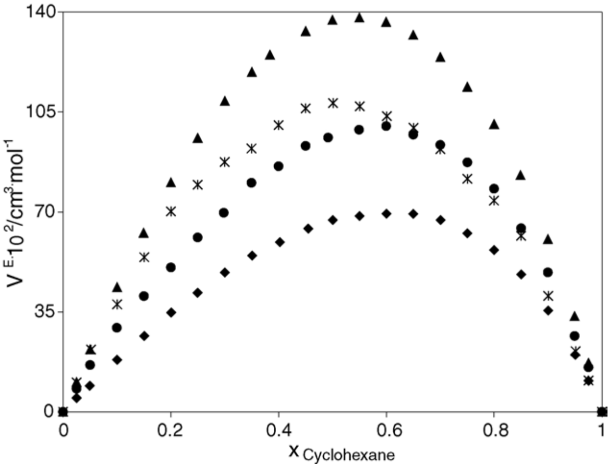

Fig. 1. Excess molar volumes at 298.15 K: (●) cyclohexane + allyl methacrylate; (◆) cyclohexane + butyl methacrylate; (*) cyclohexane + methacrylic acid; (▲) cyclohexane + vinyl acetate.

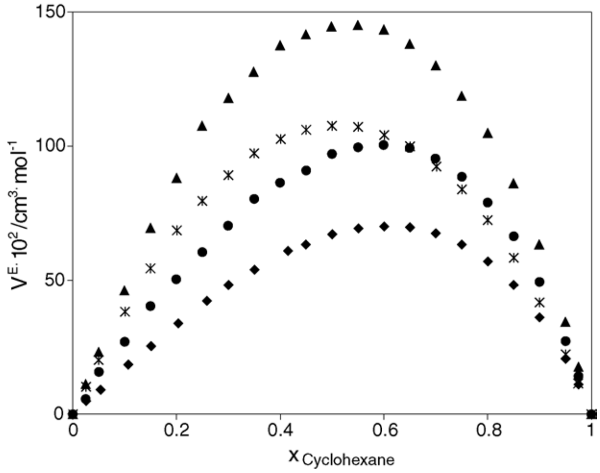

Fig. 2. Excess molar volumes at 308.15 K: (●) cyclohexane + allyl methacrylate; (◆) cyclohexane + butyl methacrylate; (*) cyclohexane + methacrylic acid; (▲) cyclohexane + vinyl acetate.

The pertinent values are reported in Supplementary Table 5 and are necessarily consistent.

The values of V_m^E of the binary systems were correlated with composition using two methods:

(a) The Redlich–Kister expression [16]

$$V_m^E = x_1 x_2 \sum_{k=0}^{n} A_k (x_1 - x_2)^k \qquad (4)$$

where the A_k's are the adjustable parameters of the model. The Redlich–Kister equation was originally developed to correlate the excess Gibbs function and calculate the values of the activity coefficients. It turned out to be such a powerful and versatile correlating tool that its use has been extended to other properties, particularly, excess molar volumes and excess enthalpies of mixing. Notwithstanding, it suffers from the important drawback that the values of its adjustable parameters change as the number of terms in the series is increased, so that no physical interpretation can be attached to them.

(b) A series of Legendre polynomials $L_k(x_1)$:

$$V_m^E = x_1 x_2 \sum_{k=0}^{n} a_k L_k(x_1) \qquad (5)$$

which for the four first terms ($k = 0, 1, 2, 3$) becomes

$$V_m^E = x_1 x_2 [a_0 + a_1(2x_1 - 1) + a_2(6x_1^2 - 6x_1 + 1) + a_3(20x_1^3 - 30x_1^2 + 12x_1 - 1)] \qquad (6)$$

Legendre polynomials belong to the category of orthogonal functions such as Fourier, Bessel, and Chebyshev, which have the valuable property that for a continuous series of observations (infinite) the values of the coefficients do not change as the number of terms in the series is increased. This is an important property because if a physical expla-

Table 2
Coefficients A_k, Eq (4), standard deviation s, Eq. (8), Durbin–Watson statistic d, $(V^{E}_{mi})_{x=0.5}$,and $\bar{V}^{E,\infty}_{mi}$

System	$A_0 \times 10^2$	$A_1 \times 10^2$	$A_2 \times 10^2$	$A_3 \times 10^2$	$A_4 \times 10^{2}$	$s \times 10^4$	d[a]	$(V^{E}_{mi})_{x=0.5} \times 10^2$ ($cm^3 mol^{-1}$)	$\bar{V}_m \times 10^2$	$\bar{V}_m \times 10^2$ ($cm^3 mol^{-1}$)
T = 298.15 K										
(1) + (2)	3.875	1.3734	0.0745	−0.0467	0.9182	65.09	2.66	96.87	354.09	619.41
(1) + (3)	2.672	1.0471	0.5015	0.2820		26.62	2.53	66.79	184.41	45.02
(1) + (4)	4.259	0.2669	0.3430			157.54	1.61	106.49	433.56	486.94
(1) + (5)	5.484	0.9322	0.4555	0.2664		31.20	2.51	137.11	474.11	713.84
T = 308.15 K										
(1) + (2)	3.860	1.487	0.603			76.75	2.52	96.50	297.65	594.98
(1) + (3)	2.683	1.110	0.346	0.3315	0.1717	22.18	2.84	67.07	175.88	464.30
(1) + (4)	4.303	0.219	0.223			24.05	1.99	107.59	430.75	474.49
(1) + (5)	5.808	0.577	0.607	0.689		104.20	2.45	145.19	514.86	768.11

[a] $d = \frac{\sum_{u=2}^{N}(e_u - e_{u-1})}{\sum_{u=1}^{N} e_u^2}$; $e_u = V^{E}_{m,u,calc} - V^{E}_{m,u,exptl}$.

nation can be assigned to one of its coefficients, its value remains constant. For the case of discrete measurements, such as determination of molar volumes of mixing, the values of the coefficients will vary, but slightly. In addition, the series of Legendre polynomials have the important characteristic that the structure of its first four terms is the same as that of the first four terms of the Redlich–Kister expression. The mathematical procedure to transform a power expansion such as that of Redlich–Kister, into an orthogonal series has been described in detail by Tomiska [17,18]. Tomiska provides the iteration formulas for Legendre or Chebyshev's series of any order as well as the proof that the procedure is independent of the conversion coefficients from the actual excess property.

Eqs. (4) and (5) were fitted using a least-squares optimization procedure, with all points weighted equally and minimizing the following objective function OF defined by

$$\mathrm{OF} = \sum_{1}^{N} (V^{E}_{mi,expt} - V^{E}_{mi,calc})^2 \qquad (7)$$

where N is the number of observations. The values of the different adjustable parameters, A_k of Eq. (4) and a_k of Eq. (5) are reported in Tables 2 and 3 for different values of k, together with the pertinent statistics. The standard deviation s was calculated from

$$s = \left[\sum_{1}^{N} \frac{(V^{E}_{mi,expt} - V^{E}_{mi,calc})^2}{N - k} \right]^{1/2} \qquad (8)$$

where N is the number of observations and k the number of adjustable parameters. The statistical significance of adding one or more terms after the third was examined using a χ^2-based test, with the simultaneous requirement that the residues (given by the difference between the calculated and experimental value of the molar excess volume) be randomly distributed, as suggested by Wisniak and Polishuk [19]. Randomness of the residues was tested using the Durbin–Watson statistic. It was not deemed necessary to perform a stepwise regression. Fig. 3 shows the residuals distribution of the Redlich–Kister fit for the binary system, which is random as shown by the Durbin–Watson statistic.

The variation of $V^{E}_{m}/x_1 x_i \quad (i = 2, 3, 4, 5)$ with composition was used to test the quality of the binary data; this function is extremely sensitive to experimental errors, particularly in the dilute ranges and helps detecting outliers. In addition, its values at infinite dilution represent the values of

Table 3
Coefficients a_k, Eq. (5), standard deviation s, Eq. (8), Durbin–Watson statistic d, $(V^{E}_{mi})_{x=0.5}$,and $\bar{V}^{E,\infty}_{mi}$

System	$a_0 \times 10^2$	$a_1 \times 10^2$	$a_2 \times 10^2$	$a_3 \times 10^2$	$a_4 \times 10^2$	$s \times 10^4$	d[a]	$(V^{E}_{mi})_{x=0.5} \times 10^2$ ($cm^3 mol^{-1}$)	$\bar{V}_m \times 10^2$ ($cm^3 mol^{-1}$)	$\bar{V}_m \times 10^2$ ($cm^3 mol^{-1}$)
T = 298.15 K										
(1) + (2)	4.083	1.345	0.574	−0.187	0.210	67.09	2.66	96.26	354.09	619.41
(1) + (3)	2.839	1.216	0.334	0.113		27.39	2.53	66.82	184.41	450.24
(1) + (4)	4.374	0.267	0.229			161.86	1.61	106.49	433.56	486.94
(1) + (5)	5.636	1.092	0.304	0.106		32.10	2.51	137.10	474.11	713.84
T = 308.15 K										
(1) + (2)	4.061	1.487	0.402			78.85	2.52	96.49	297.65	594.98
(1) + (3)	2.832	1.309	0.329	0.133	0.039	22.86	2.84	67.09	175.88	464.29
(1) + (4)	4.378	0.242	0.148	0.035		21.33	2.69	107.58	424.90	480.33
(1) + (5)	6.010	0.990	0.405	0.276		107.28	2.47	145.15	514.86	768.11

[a] $d = \frac{\sum_{u=2}^{N}(e_u - e_{u-1})}{\sum_{u=1}^{N} e_u^2}$; $e_u = V^{E}_{m,u,calc} - V^{E}_{m,u,exptl}$.

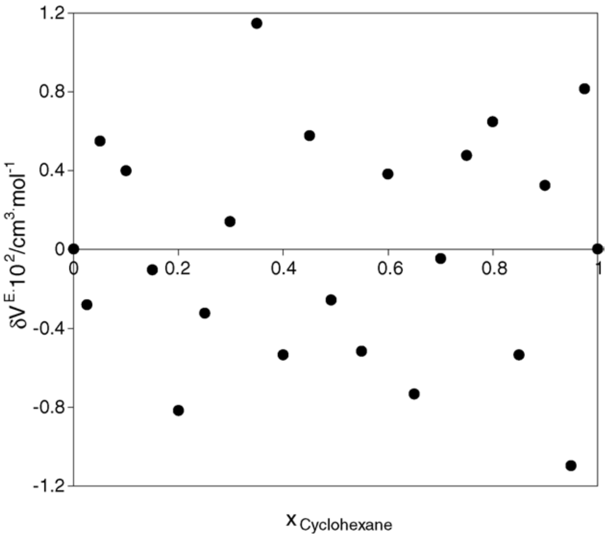

Fig. 3. Residual distribution plot for the system cyclohexane + allyl methacrylate at 298.15 K, according to the fit given in Supplementary Table 3.

the partial excess molar volume at infinite dilution, $\bar{V}_{mi}^{E,\infty}$ [15] which can be also calculated from the adjustable parameters using

$$\bar{V}_{m1}^{E,\infty} = A_0 - A_1 + A_2 - \cdots = \bar{V}_{m1}^{\infty} - V_{m1}^{0} \qquad (9)$$

$$\bar{V}_{m2}^{E,\infty} = A_0 + A_1 + A_2 + \cdots = \bar{V}_{m2}^{\infty} - V_{m2}^{0} \qquad (10)$$

for the Redlich–Kister expression and

$$\bar{V}_{m1}^{E,\infty} = a_0 - a_1 + a_2 - \cdots = \bar{V}_{m1}^{\infty} - V_{m1}^{0} \qquad (11)$$

$$\bar{V}_{m2}^{E,\infty} = a_0 + a_1 + a_2 + \cdots = \bar{V}_{m2}^{\infty} - V_{m2}^{0} \qquad (12)$$

for the Legendre polynomial. In Eqs. (9)–(12) V_{mi}^{0} is the molar volume of pure component *i*. In addition, it should be realized that in the absence of homo-association, the value of the partial excess molar volume at infinite dilution reflects the true solute–solvent interaction. Eqs. (9) and (11) or (10) and (12) yield the same values of $\bar{V}_{mi}^{E,\infty}$.

Inspection of the results of Supplementary Tables 1–4 and Figs. 1 and 2 indicates that the excess molar volumes are positive for the four binaries studied here. These results are consistent with those reported in the literature for similar systems [8–13]. In addition, the values of the partial excess molar volume at infinite dilution are very similar. The magnitude and sign of $V_{m}^{E}$is a reflection of the type of interactions taking place in the mixture. This is well exhibited by the mixtures studied here, where the acrylate solutes are characterized by the simultaneous presence of a double bond and an ester group. In addition, methacrylic acid has a free –COOH group, which can lead to H bonding. The relative magnitude of V_{m}^{E} is a result of the effect of breaking the ester's dipole–dipole association and H bonding. These results are similar to those obtained by González et al. [8] for the excess molar volumes of cyclohexane + vinyl acetate and by Sastry et al. [13] for cyclohexane + acrylates and by Peralta et al. [1] for cyclohexane + acrylates. The maximum value of the excess molar volume for the system cyclohexane + vinyl acetate is more than that for the binary cyclohexane + butyl methacrylate indicating a larger dominant contribution from the structure-breaking effects in the acetate by the globular cyclohexane molecule.

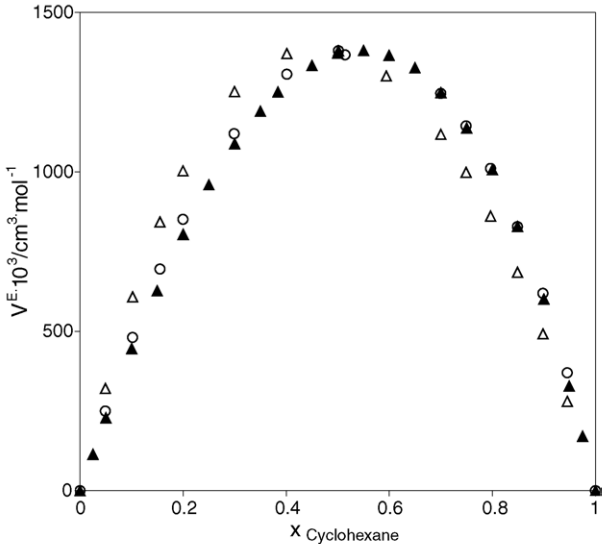

Fig. 4. Comparison of experimental results of the system cyclohexane (1) + vinyl acetate (2) at 298.15 K; (▲) this work; (△) González et al. [8]; (○) calculated from the Redlich–Kister expression given by González et al. [8].

As shown by Supplementary Tables 1–4 and Figs. 1 and 2, within the short temperature range the V_{m}^{E}curves for the systems cyclohexane + acrylate are essentially not affected by increased temperature. This is most likely due to the fact that the small increase in kinetic energy is not enough to change in appreciable manner the self-association of the acrylate molecules. For the system with vinyl acetate the excess molar volume becomes more positive over the entire mole fraction range as the temperature increases from 298.15 to 308.15 K, probably due to an increase of kinetic energy of the molecules of the solutes, which decreases self-association and facilitates intercalation of one species into the other.

Fig. 4 gives a comparison between the experimental data measured here and those reported by González et al. [8]. It is seen that although the agreement between the two sets of data is very good, the data recovered by using the Redlich–Kister expansion suggested by González et al. does not represent a good fit.

In general, for small changes of V_{m}^{E} and small temperature intervals it is possible to calculate the excess isobaric thermal coefficient by the finite difference approximation:

$$\alpha^{E} = \left(\frac{1}{V_{m}^{E}}\right)\left(\frac{\partial V_{m}^{E}}{\partial T}\right)_{P} = 2\left(\frac{V_{m2}^{E} - V_{m1}^{E}}{(V_{m2}^{E} + V_{m1}^{E})(T_2 - T_1)}\right) = \frac{1}{5}\left(\frac{V_{m2}^{E} - V_{m1}^{E}}{V_{m2}^{E} + V_{m1}^{E}}\right) \qquad (13)$$

For both the Redlich–Kister and the Legendre expansion the value of V_{m}^{E} at $x = 0.5$ is equal to A_0, and a_0, respectively, so that Eq. (13) becomes:

$$\alpha^{\mathrm{E}}(x=0.5)=\frac{1}{5}\left(\frac{A_{02}-A_{01}}{A_{01}+A_{02}}\right) \tag{14}$$

In Eqs. (13) and (14) the indexes 1 and 2 represent the value of the parameter at the corresponding temperature (T_1 and T_2). Inspection of the values of the molar excess volume given in Supplementary Table 5 indicates that the effect of temperature is very small so that use of Eq. (4) and the values of A_0 given in Table 2 or 3 may lead to numerical errors of similar magnitude as the calculated value. For this reason, the value of the thermal expansion coefficient has been calculated as

$$\alpha(x=0.5)=-\frac{1}{\rho}\left(\frac{\mathrm{d}\rho}{\mathrm{d}T}\right)_P\approx\frac{1}{5}\left(\frac{\rho_1-\rho_2}{\rho_2+\rho_1}\right) \tag{15}$$

The values of $\alpha(x=0.5)$ are 1.18×10^{-3}, 1.14×10^{-3}, 1.13×10^{-3}, and $1.59\times10^{-3}\,\mathrm{K}^{-1}$ for the systems of cyclohexane + allyl methacrylate, + butyl methacrylate, + methacrylic acid, and + vinyl acetate, respectively. It is seen that the expansion coefficient is essentially the same for all the acrylates but for the system cyclohexane + vinyl acetate it is about 50% larger than that for the other binaries, reflecting the experimental fact that the change in $V_{\mathrm{m}}^{\mathrm{E}}$ with temperature for this system is also the largest one. The presence of two double bonds in allyl methacrylate or a –COOH group in methacrylic acid does not seem to influence the temperature effect.

A final observation relates the influence of the length of the alkyl chain in the alkyl methacrylate. According to the data of Peralta et al. [1] at 298.15 K the maximum excess volume of the system cyclohexane + methyl methacrylate is about 0.11 cm^3 mol^{-1} at $x_1 = 0.60$. Supplementary Table 2 shows that for cyclohexane + butyl methacrylate the maximum is 0.69 cm^3 mol^{-1} at $x_1 = 0.65$, indicating a substantial increase with increase in chain length.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tca.2005.06.007.

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