Measurements of the Properties of Binary Mixtures of Dimethylsulphoxide (DMSO) with 1-Alkanols (C$_4$, C$_6$, C$_7$) at 303.15 K

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This paper presents experimental data for densities, ρ, ultrasonic velocities, u, and refractive indices, n, of pure dimethylsulphoxide (DMSO), 1-butanol, 1-hexanol, 1-heptanol, and their binary mixtures, with DMSO as a common component, over the whole composition range at 303.15 K. The molar refraction, R_m, molecular association, M_A, excess molar volume, V^E, and deviation in isentropic compressibility, ΔK_s, were calculated from the experimental data. The apparent molar volume, $V_{\phi,2}$, and apparent molar isentropic compressibility, $K_{\phi,2}$, of alkanols in DMSO were also calculated. The values of $V_{\phi,2}$ and $K_{\phi,2}$ were used to estimate the partial molar volume, $\bar{V}_{\phi,2}^0$, and partial molar isentropic compressibility, $\bar{K}_{\phi,2}^0$, of alkanols in DMSO at infinite dilution. The changes in these parameters with composition and the size of the alkyl chain length in the alkanol molecule are discussed with reference to the nature of interactions between component molecules. Excess molar volumes have also been estimated from measurements of refractive indices.

KEY WORDS: 1-butanol; density; dimethylsulphoxide; 1-hexanol; 1-heptanol; refractive index; ultrasonic velocity.

1. INTRODUCTION

The present work is a continuation of our research program [1–3] on the thermodynamic properties of binary liquid mixtures of industrially important components. Both the solvents, viz., *n*-alkanols and dimethylsulphoxide (DMSO), are considered as "supersolvents" resulting from their wide

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applications in industrial and medicinal fields. Alkanols are considered as amphiphilic materials of biological and industrial importance [4]. DMSO is a "high energy reaction solvent" having a wide range of applications [5, 6]. Its cryoprotective effects on biological systems [7–9] are well known. The highly polar S=O group and two hydrophobic CH_3 groups make it a polyfunctional molecule. Alkanols are self-associated through hydrogen bonding. Therefore, interesting results may be obtained regarding molecular interactions between these component molecules in the binary mixtures.

In this paper we report measured densities, ρ, ultrasonic velocities, u, and refractive indices, n, of the binary mixtures of DMSO with 1-butanol, 1-hexanol, and 1-heptanol, along with those of the pure liquids at 303.15 K, covering the entire composition range expressed by the mole fraction, x_1, of DMSO. From the experimental values of ρ, u, and n, the molar refraction, R_m, molecular association, M_A, and excess molar volume, V^E; the deviation in the isentropic compressibility, ΔK_s, the apparent molar volume, $V_{\phi,2}$, and the apparent molar isentropic compressibility, $K_{\phi,2}$, of alkanols in DMSO; and the partial molar volume, $\bar{V}_{\phi,2}^0$, and partial molar isentropic compressibility, $\bar{K}_{\phi,2}^0$, of alkanols in DMSO at infinite dilution have been calculated. These functions offer a convenient, model-free approach for the study of thermodynamic and transport properties of liquids and liquid mixtures [10], not easily obtained by other means. Furthermore, a relatively new method [11] has been used for the estimation of excess molar volumes of binary mixtures from the known experimental values of refractive indices and densities of pure liquids and refractive indices of their mixtures. To the best of our knowledge, no one has reported the estimation of V^E from refractive index measurement, except for the work of Fucaloro [11] who has reported partial molar volumes using this method.

2. EXPERIMENTAL

The chemicals (AR grade) employed were supplied by S.D. Fine-Chem Ltd., India. Their purities (in mass percent) were DMSO 99%, 1-butanol 99.5%, 1-hexanol 98%, and 1-heptanol 99%. All the chemicals, except 1-hexanol, which was purified by a method given in the literature [12], were used as received. But before use, these chemicals were degassed and dried over molecular sieves (Sigma Union Carbide Type 0.4 nm). The solutions were prepared by mass using a precise XB 220A (Swiss make) electronic balance with a precision of ± 0.1 mg. The precision of the mole fraction is estimated to be better than 10^{-4}. Care was taken to obtain

dust-free samples. They were stored in special airtight bottles to prevent contamination and evaporation.

The densities of the pure liquids and their binary mixtures were measured using a single-capillary pycnometer, made of Borosil glass, having a bulb capacity of 8×10^{-6} m^3. The capillary, with graduated marks, had a uniform bore and could be closed by a well-fitted glass cap. The marks on the capillary were calibrated by using triple-distilled water at 303.15 K. The pycnometer was kept for about 30 min in an electronically controlled thermostated water bath (303.15 $\pm$ 0.02 K), and the position of the liquid level on the capillary was noted. The volume of the pycnometer at each mark was calculated by using the literature [13] value of the density of pure water at 303.15 K. The volume thus obtained is used to determine the density of the unknown liquid. The observed values of densities of pure DMSO, 1-butanol, 1-hexanol, and 1-heptanol at 303.15 K were 1094.9, 802.0, 811.8, and 815.7 kg·m^{-3} which compare well with corresponding literature values of, respectively, 1091.0 [6], 801.8, 811.4, and 815.7 kg·m^{-3} [14]. The ultrasonic velocities were measured using a single-crystal variable-path ultrasonic interferometer operating at 3 MHz as described in the literature [1–3]. The measured values of ultrasonic velocities of pure DMSO, 1-butanol, 1-hexanol, and 1-heptanol at 303.15 K were 1490.9, 1231.0, 1282.0, and 1306.0 m·s^{-1}, respectively, which compare well with the corresponding literature values of, respectively, 1490.8 [15]/1490.9 [16], 1223.0 [17], 1287.0 [17], and 1313.0 [17] m·s^{-1}. The observed refractive indices of pure DMSO, 1-butanol, 1-hexanol, and 1-heptanol at 303.15 K are 1.4751, 1.3946, 1.4134, and 1.4201, which are in good agreement with the literature values of, respectively, 1.4742 [18], 1.3947 [14], 1.4137 [14], and 1.4200 [14]. The refractive indices were measured with a thermostated Abbe refractometer. The uncertainties in the measured values of ρ, u, and n were not more than $\pm 1.5 \times 10^{-1}$ kg·m^{-3}, $\pm$ 2 m·s^{-1}, and ± 0.0002 unit, respectively. The temperature of the samples was maintained at 303.15 $\pm$ 0.02 K in an electronically controlled thermostatic water bath (Julabo, Germany).

3. RESULTS AND DISCUSSION

Table I shows the experimental values of ρ, u, and n of pure DMSO, 1-butanol, 1-hexanol, 1-heptanol, and 27 binary mixtures of DMSO with alkanols over the whole composition range at 303.15 K. The derived parameters R_m (using Lorentz–Lorenz equation) and M_A [19] were calculated from the following relations:

$$R_\mathrm{m} = [(n^2 - 1)/(n^2 + 2)]V \qquad (1)$$

$$M_A = [(u^2/u_{im}^2) - 1] \qquad (2)$$

where $V(=(x_1 M_1 + x_2 M_2)/\rho)$, is the molar volume of the mixture; x_1 and x_2 are the mole fractions of component 1 (DMSO) and component 2 (alkanols); and M_1 and M_2 are their molar masses, and u_{im} is the ultrasonic velocity of the ideal mixture calculated using the relation suggested by Van Dael and Vangeel [20]. The dependence of R_m and M_A on the composition of the mixtures is also included in Table I. The molar refraction, R_m, increases with the chain length in the homologous series from 1-butanol to 1-heptanol while it decreases with an increase in DMSO concentration (Table I). The average value of R_m for the –CH_2 group estimated from the data for 1-butanol, 1-hexanol, and 1-heptanol comes out to be constant, equal to $4.6410 \times 10^{-6}\, m^3 \cdot mol^{-1}$. This is in good agreement with the value of $R_m (= 4.6380 \times 10^{-6}\, m^3 \cdot mol^{-1})$ calculated from known literature [21] values of R_m of C and H. Moreover, the value of R_m obtained for the $-CH_2$ group of the present alkanols is in close agreement with the value $4.6440 \times 10^{-6}\, m^3 \cdot mol^{-1}$ for the alkanols series reported by Sjoblom et al. [22].

The departure of the ratio of u^2/u_{im}^2 from unity represented as M_A, is a measure of the nonideality of the system as a result of association/dissociation between the component molecules [23]. Table I shows that M_A has negative deviations for all three alkanols over the whole composition range and decreases as the number of $-CH_2$ groups in the molecules increases from 1-butanol to 1-heptanol. Thus, we may conclude that the nonideality of the systems varies in the order: 1-butanol < 1-hexanol < 1-heptanol. Similar trends in the variation of M_A with composition in binary mixtures have also been reported by Ali et al. [3].

The functions, V^E and ΔK_s, are highly sensitive to intermolecular interactions between the component molecules of the mixture. The values of the functions, V^E and ΔK_s, were computed using the following relations:

$$V^E = \sum_{i=1}^{2} x_i M_i (1/\rho_1 - 1/\rho_2) \qquad (3)$$

$$\Delta K_s = K_s - \sum_{i=1}^{2} \phi_i K_{si} \qquad (4)$$

where M_i and ϕ_i are the molar mass and volume fraction of the ith component, respectively. The values of V^E and ΔK_s were fitted with a

Table I. Experimental Values of Densities, ρ, Ultrasonic Velocities, u, Refractive Indices, n, Molar Refraction, R_m, and Molecular Association, M_A, as a Function of Mole Fraction, x_1, of DMSO for the Binary Mixtures at 303.15 K

x_1	ρ $(kg \cdot m^{-3})$	u $(m \cdot s^{-1})$	n	R_m $(10^{-5} m^3 \cdot mol^{-1})$	M_A
DMSO + 1-Butanol					
0.0000	802.0	1231.0	1.3946	2.2137	
0.1034	825.7	1247.5	1.4001	2.1888	−0.0050
0.2122	851.8	1267.5	1.4064	2.1638	−0.0080
0.3156	877.9	1288.0	1.4127	2.1399	−0.0107
0.4009	900.5	1305.5	1.4185	2.1215	−0.0136
0.5073	930.1	1327.3	1.4263	2.0992	−0.0196
0.6002	957.2	1347.5	1.4335	2.0799	−0.0252
0.6957	986.6	1371.0	1.4417	2.0614	−0.0292
0.8112	1024.6	1406.6	1.4527	2.0399	−0.0276
0.9051	1058.4	1443.0	1.4633	2.0244	−0.0195
1.0000	1094.9	1490.9	1.4751	2.0094	
DMSO + 1-Hexanol					
0.0000	811.8	1282.0	1.4134	3.1413	
0.1336	832.6	1300.8	1.4149	2.9760	−0.0072
0.2712	857.6	1311.6	1.4185	2.8138	−0.0289
0.3808	880.7	1325.3	1.4216	2.6818	−0.0393
0.4814	904.6	1336.5	1.4259	2.5657	−0.0516
0.5762	929.7	1349.5	1.4311	2.4596	−0.0604
0.6660	956.3	1368.0	1.4372	2.3614	−0.0609
0.7650	989.2	1395.0	1.4448	2.2533	−0.0536
0.8480	1021.1	1420.5	1.4535	2.1669	−0.0448
0.9246	1054.7	1448.5	1.4629	2.0873	−0.0317
1.0000	1094.9	1490.9	1.4751	2.0094	
DMSO + 1-Heptanol					
0.0000	815.7	1306.0	1.4201	3.6057	
0.1096	825.7	1312.3	1.4209	3.4399	−0.0115
0.2535	846.0	1319.5	1.4242	3.2151	−0.0305
0.3668	866.8	1329.9	1.4277	3.0327	−0.0404
0.4740	889.4	1338.0	1.4312	2.8578	−0.0540
0.5788	915.8	1351.5	1.4355	2.6857	−0.0612
0.6708	943.2	1369.0	1.4402	2.5343	−0.0615
0.7598	973.2	1384.6	1.4458	2.3905	−0.0652
0.8376	1003.5	1409.3	1.4528	2.2700	−0.0552
0.9031	1034.5	1428.6	1.4615	2.1723	−0.0500
1.0000	1094.9	1490.9	1.4751	2.0094	

Redlich–Kister [24] type polynomial equation:

$$Y^E = x_1 x_2 \sum_{i=1}^{5} A_i (1 - 2x_1)^{i-1} \tag{5}$$

Table II. Coefficients A_i of Eq. (5) along with Standard Deviations, $\sigma(Y^{\mathrm{E}})$ for the Binary Mixtures at 303.15 K

Properties	A_1	A_2	A_3	A_4	A_5	$\sigma(Y^{\mathrm{E}})$
DMSO + 1-Butanol						
$V^{\mathrm{E}}(10^{-7}\,\mathrm{m}^3 \cdot \mathrm{mol}^{-1})$	5.6895	−6.7051	10.6294	1.6493	−12.3552	0.0168
$\Delta K_{\mathrm{s}}(10^{-11}\,\mathrm{m}^2 \cdot N^{-1})$	−2.4970	−0.8630	4.5614	−5.0711	1.1616	0.1151
DMSO +1-Hexanol						
$V^{\mathrm{E}}(10^{-7}\,\mathrm{m}^3 \cdot \mathrm{mol}^{-1})$	21.5263	−1.5637	12.3142	−15.5951	10.9971	0.0482
$\Delta K_{\mathrm{s}}(10^{-11}\,\mathrm{m}^2 \cdot N^{-1})$	13.8740	−8.4853	−15.3771	−1.8739	21.1951	0.0210
DMSO +1-Heptanol						
$V^{\mathrm{E}}(10^{-7}\,\mathrm{m}^3 \cdot \mathrm{mol}^{-1})$	63.8196	11.3495	12.3229	−6.1783	71.6531	0.1137
$\Delta K_{\mathrm{s}}(10^{-11}\,\mathrm{m}^2 \cdot N^{-1})$	22.5137	−6.3488	−5.4593	−7.6695	21.9166	0.1494

where Y^{E} is either V^{E} or ΔK_{s}.

The values of coefficient A_i of Eq. (5) were calculated by the least-squares method, with all points being given equal weight, and are given in Table II along with the corresponding standard deviations, σ, calculated by using the relation,

$$\sigma(Y^{\mathrm{E}}) = \left[\sum \left(Y_{\mathrm{exp}}^{\mathrm{E}} - Y_{\mathrm{cal}}^{\mathrm{E}} \right)^2 / (m - l) \right]^{1/2} \tag{6}$$

where m is the number of experimental data points and l is the number of coefficients considered ($l = 5$ in the present work). Graphical presentations of smoothing curves of V^{E} and ΔK_{s} are shown in Figs. 1 and 2.

The curves in Fig. 1 reveal that values of V^{E} are positive over the entire mole fraction of DMSO for all three systems investigated. Mixing of DMSO with alkanols will first lead to the mutual dissociation of DMSO – DMSO and alkanol – alkanol associates (both are strongly associated due to highly polar S=O group in the former and H-bonding in the latter molecules), with subsequent formation of DMSO – alkanol associates. The interactions, which are expected to operate between DMSO and alkanols, are

(a) hydrogen bonding between oxygen atom of highly polar S=O group of DMSO and hydrogen atom of −OH group of alkanol molecules (>S=O......H−O−),

(b) dipole–dipole interaction between unlike molecules, and

(c) dispersion forces which are likely to operate in every system [25].
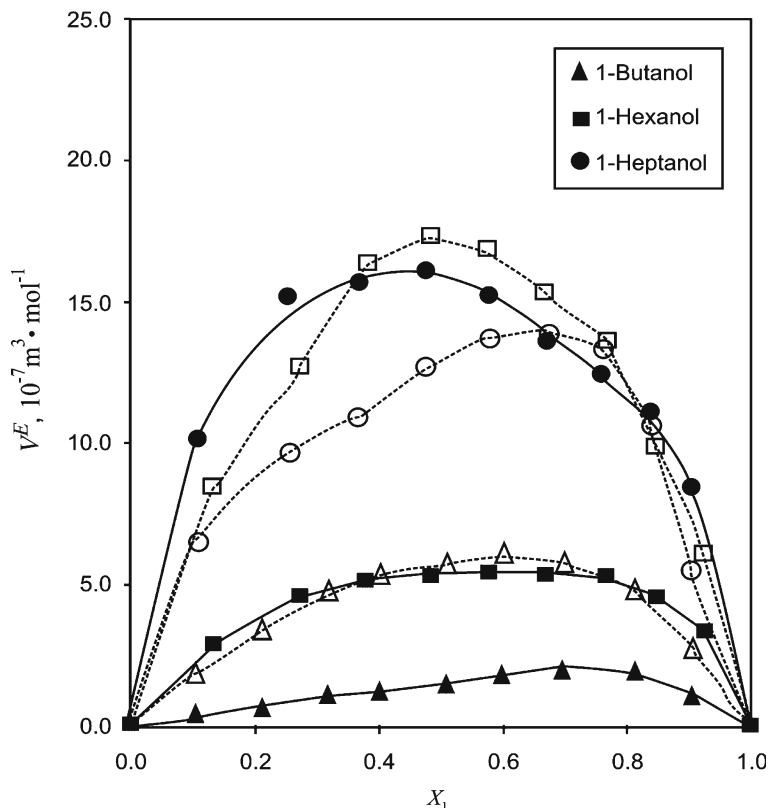
Fig. 1. Variation of excess molar volume, V^E, (calculated from density (—) and from refractive index (----)) with mole fraction, x_1, of DMSO for the binary mixtures at 303.15 K. Points show experimental data and curves show smoothed values.

The observed positive trends in V^E values indicate that the effect due to the breaking up of self-associated structures of the components of the mixtures is dominant over the effect of H-bonding and dipole–dipole interaction between unlike molecules. The V^E values increase in the sequence: 1-butanol < 1-hexanol < 1-heptanol which also reflects the decreasing strength of interaction between unlike molecules in the mixture.

As the size of the alkyl group increases from 1-butanol to 1-heptanol, the steric hindrance also increases, resulting in decreased interaction between DMSO and alkanol molecules, and, hence, the strength of interaction should follow the sequence: 1-butanol > 1-hexanol > 1-heptanol. Thus, the extent of positive deviation (Fig. 1) supports our view.
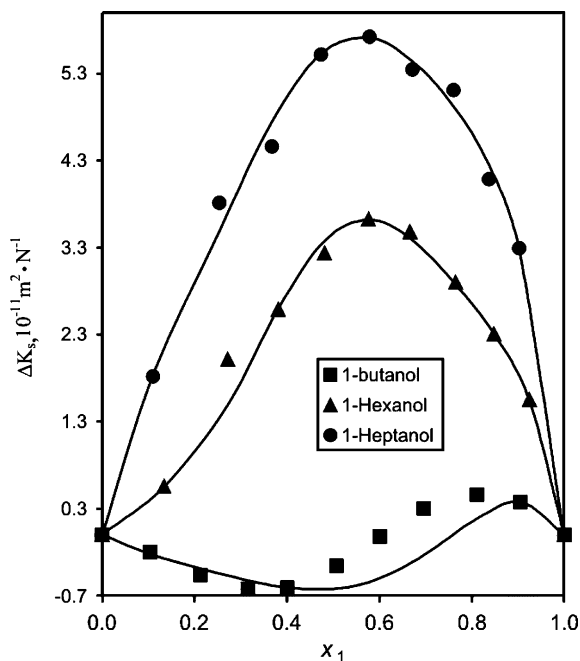
Fig. 2. Variation of deviations in isentropic compressibility, ΔK_s, with mole fraction x_1 of DMSO for the binary mixtures at 303.15 K. Points show experimental data, and curves show smoothed values.

Similar behavior in V^E with composition has also been reported for ethanol +1-hexanol [17], N,N-dimethylacetamide + 1-hexanol, and DMSO + N,N-dimehylformamide [26] binary mixtures.

Recently, Fucaloro [11] estimated the partial molar volumes of components of binary mixtures (water + ethanol) by using the experimental values of refractive indices and densities of the pure components and refractive indices of the mixtures. Following the procedure adopted by Fucaloro, details of which can be found in the literature [11], we estimated the excess molar volumes of the present binary mixtures from refractive index measurements. The estimated values of V^E, together with the values of V^E obtained by using experimental densities of pure liquids and their mixtures, are graphically shown in Fig. 1. It is clear (Fig. 1) that like V^E (calculated from density), the values of V^E (calculated from refractive index) exhibit positive deviations from the linear dependence on composition for all the mixtures studied. Thus, the procedure of calculating V^E (using n) seems to be qualitatively correct.

The observed positive values of ΔK_s (Fig. 2), except for small negative values of ΔK_s for DMSO + 1-butanol mixture in the DMSO poor region, over the entire composition range indicate the presence of weak interactions between the component molecules in all the three systems investigated. This again reinforces our view that the dissociation of structure in the mixtures dominates over the combined effect due to H-bonding and dipole–dipole interactions between the component molecules. According to Fort and Moore [25], positive deviations in ΔK_s, from the linear dependence on composition suggest the presence of weak interactions between the component molecules in the mixture.

The apparent molar volumes were evaluated using the relations [27],

$$V_{\phi,1} = (V_m - n_2 V_2^*)/n_1 \tag{7}$$

and

$$V_{\phi,2} = (V_m - n_1 V_1^*)/n_2 \tag{8}$$

where $V_{\phi,1}$ and $V_{\phi,2}$ are the apparent molar volumes of the DMSO in alkanol and of the alkanol in DMSO, V_1^* and V_2^* are the molar volumes of DMSO and alkanol, respectively, and V_m is the volume of the mixture containing one mole of (alkanol + DMSO). It can be expressed as

$$V_m = V_m^E + x_1 V_1^* + x_2 V_2^* \tag{9}$$

Combining Eqs. (7) and (9), and Eqs. (8) and (9), we get

$$V_{\phi,1} = V_1^* + (V_m^E/x_1) \tag{10}$$

and

$$V_{\phi,2} = V_2^* + (V_m^E/x_2) \tag{11}$$

Graphical extrapolation of $V_{\phi,1}$ and $V_{\phi,2}$ vs. $\sqrt{C_1}$ and $\sqrt{C_2}$, where C_i is the molar concentration of component i, gives values of the partial molar volumes at infinite dilution, $\bar{V}_{\phi,1}^0$ and $\bar{V}_{\phi,2}^0$, respectively.

The deviations in $\bar{V}_{\phi,1}^0$ and $\bar{V}_{\phi,2}^0$ at infinite dilution, ΔV_1 and ΔV_2 were calculated using the following equations [28]:

$$\Delta V_1 = \bar{V}_{\phi,1}^0 - V_1^* \tag{12}$$

and

$$\Delta V_2 = \bar{V}_{\phi,2}^0 - V_2^* \tag{13}$$

The values of $\bar{V}_{\phi,1}^0$, $\bar{V}_{\phi,2}^0$, V_1^*, V_2^*, ΔV_1, and ΔV_2 are included in Table III. It is clear from Table III that $\bar{V}_{\phi,1}^0$ and $\bar{V}_{\phi,2}^0$ values are larger than the corresponding molar volumes V_1^* and V_2^*, leading to positive ΔV_1 and ΔV_2, thereby, suggesting the presence of weak molecular interactions between the components in the systems under study. It is interesting to note that ΔV_1 and ΔV_2 values increase as we move from 1-butanol to 1-heptanol. Thus, as also suggested by V^E values, the strength of interaction between DMSO and alkanol molecules follows the order: 1-heptanol < 1-hexanol < 1-butanol.

The apparent molar compressibility, $K_{\phi,1}$ of DMSO in alkanols and $K_{\phi,2}$ of alkanols in DMSO, were calculated using the relations [28],

$$K_{\phi,1} = (K_s V)^E / x_1 + K_{\phi,1}^* \tag{14}$$

and

$$K_{\phi,2} = (K_s V)^E / x_2 + K_{\phi,2}^*, \tag{15}$$

where $(K_s V)^E$ is the excess molar compressibility of the mixture and $K_{\phi,1}^*$ and $K_{\phi,2}^*$ are the molar isentropic compressibilities of DMSO and alkanols, respectively. The partial molar compressibilities, $\bar{K}_{\phi,1}^0$ and $\bar{K}_{\phi,2}^0$, of DMSO and alkanols at infinite dilution were obtained by graphical extrapolation of $K_{\phi,1}$ and $K_{\phi,2}$ vs. $\sqrt{C_1}$ and $\sqrt{C_2}$, respectively. The deviations in $K_{\phi,1}^0$ and $\bar{K}_{\phi,2}^0$ at infinite dilution, ΔK_1 and ΔK_2, were obtained using the following relations [28]:

$$\Delta K_1 = \bar{K}_{\phi,1}^0 - K_{\phi,1}^* \tag{16}$$

and

$$\Delta K_2 = \bar{K}_{\phi,2}^0 - K_{\phi,2}^* \tag{17}$$

The values of $\bar{K}_{\phi,1}^0$, $\bar{K}_{\phi,2}^0$, $\bar{K}_{\phi,1}^*$, $K_{\phi,2}^*$, ΔK_1, and ΔK_2 are listed in Table III. The $\bar{K}_{\phi}^0$ values characterize the compressibility of the component in the mixture, whereas the molar isentropic compressibility K_{ϕ}^*, of a pure component can be considered as its partial molar compressibility when dissolved in itself. Table III reveals that deviations in ΔK change from small negative for 1-butanol to positive for 1-heptanol, thereby, implying a decrease in molecular interactions with an increase in the carbon chain length of alkanols. Thus, we conclude that the behaviors of $V^E, \Delta\eta, \Delta K_s, \Delta V$, and ΔK support each other for the systems under study.

Table III. Values of $\bar{V}_{\phi,1}^0$, V_1^*, ΔV_1, $\bar{V}_{\phi,2}^0$, V_2^*, ΔV_2, $\bar{K}_{\phi,1}^0$, K_1^*, ΔK_1, $\bar{K}_{\phi,2}^0$, K_2^*, and ΔK_2, for the Binary Mixtures at 303.15 K

DMSO+	$\bar{V}_{\phi,1}^0$ (10^{-5} m³· mol⁻¹)	V_1^* (10^{-5} m³· mol⁻¹)	ΔV_1 (10^{-5} m³· mol⁻¹)	$\bar{V}_{\phi,2}^0$ (10^{-5} m³· mol⁻¹)	V_2^* (10^{-5} m³· mol⁻¹)	ΔV_2 (10^{-5} m³· mol⁻¹)	$\bar{K}_{\phi,1}^0$ (10^{-14} m⁵· N⁻¹ mol⁻¹)	K_1^* (10^{-14} m⁵· N⁻¹ mol⁻¹)	ΔK_1 (10^{-14} m⁵· N⁻¹ mol⁻¹)	$\bar{K}_{\phi,2}^0$ (10^{-14} m⁵· N⁻¹ mol⁻¹)	K_2^* (10^{-14} m⁵· N⁻¹ mol⁻¹)	ΔK_2 (10^{-14} m⁵· N⁻¹ mol⁻¹)
1-Butanol	7.1701	7.1358	0.0343	9.3297	9.2419	0.0878	2.0246	2.9321	-0.9075	6.6990	7.6045	-0.9055
1-Hexanol	7.3131	7.1358	0.1733	12.9081	12.5870	0.3212	2.3696	2.9321	-0.5625	9.2730	9.4339	-0.1609
1-Heptanol	7.8285	7.1358	0.6927	14.9320	14.2454	0.6866	3.6132	2.9321	0.6812	10.9290	10.2390	0.6900

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