J. Chem. Eng. Data **2004,** *49,* 1003−1007 **1003**

Concentration Dependence of Surface Tension for Very Dilute Aqueous Solutions of Organic Nonelectrolytes

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Surface tensions of pure organic compounds and their dilute aqueous solutions (molar fraction typically $x_2 < 0.015$) at 298.15 K have been measured. The Szyszkowski equation has been used for correlation of the experimental surface tension data, and the ability of the equation to describe the concentration dependence of surface tension has been tested. When accurate values of surface tension were known, the Szyszkowski equation has been found to not be suitable for correlation, especially in the case when values of the first derivative, obtained from the parameters of a correlation equation, have been wanted. Therefore values of limiting slopes of surface pressure concentration dependence have been evaluated by a linear regression of the most dilute results (typically $x_2 < 0.001$).

Introduction

In recent years, there has been an increasing effort to study the relationship between the properties of the vapor−liquid interface and the activity of a solute in the bulk liquid solution. This effort is motivated, among other things, by the aim to determine values of activity coefficients from surface tension data for systems where classical methods of their determination cannot be used. Studies are focused, especially, on very dilute solutions, because limiting activity coefficients are of great practical importance in separation technology, environmental pollution control, chemical technology, food technology, and biotechnology applications as well as of theoretical interest (for example, in solution chemistry and biothermodynamics). Therefore, a new method to obtain values of limiting activity coefficients suitable for systems, where known methods cannot be used, would have extensive utilization.

Another reason to study the relation between surface tension and activity coefficient in bulk liquid solutions is that the Gibbs isotherm, often used for describing adsorption phenomena, cannot be applied over wide concentration intervals without the knowledge of activity coefficients. Information about concentration dependence of activity coefficients is needed. If the limiting activity coefficients are known, it is possible to obtain the concentration dependence of activity coefficients by application of a suitable model (van Laar equation, Margules equation, models based on lattice theory of solution etc.; a survey is given, e.g., in Prausnitz et al.).[1]

Several papers dealing with the topic of evaluation of limiting activity coefficients from surface tension data have appeared recently; the authors, however, have drawn general conclusions on the basis of a very narrow set of experimental data, where only some types of compounds were included.[2−4]

More experimental information about limiting activity coefficients and surface tension is clearly needed to explore mutual relations between properties in the bulk and surface phase of liquid solutions. Since accurate values of limiting activity coefficients for the 17 solutes with different functional groups in water have been reported recently,[5] the main objective of this work has been to provide accurate surface tension data for the most of these systems to obtain data for simultaneous treatment of a surface tension and limiting coefficient data.

Table 1. Organic Solutes Used. Specification of Purity and Values of Density ρ Used for Correction of the Reading on the Display of Ring Tensiometer According to Eq 1

solute	CAS RN[a]	producer/purity	ρ/g·cm^{-3} [b]
1-butanol	[71-36-3]	Aldrich/99.9%[c]	0.80559
2-butanol	[78-92-2]	Penta/>99.5% GC[d]	0.80240
cyclopentanol	[96-41-3]	Fluka/>99%[c]	0.94241
ethylene glycol	[107-21-1]	Riedel de Haen/>99.8%[c]	1.10980
2-butanone	[78-93-3]	Fluka/>99.5% GC[c]	0.79953
cyclopentanone	[120-92-3]	Fluka/>99%[c]	0.94379
2,4-pentadione	[123-54-6]	Lachema/p.a. 99.5%[c]	0.96813
ethyl acetate	[141-78-6]	Lachema/p.a. 99.7%[c]	0.89449
methyl acetoacetate	[105-45-3]	Fluka/99+% GC[c]	1.07075
2-ethoxyethyl acetate	[111-15-9]	Aldrich/99+%[c]	0.96748
propionitrile	[107-12-0]	Fluka/99% GC[c]	0.77654

[a] Chemical Abstracts Service Registry Number. [b] Sources of densities defined in the text. [c] Used directly without any purification or drying. [d] Distilled and dried by molecular sieves.

Another aim has been to test the correlation of experimental data by the Szyszkowski equation for dilute aqueous solutions. The Szyszkowski equation has been chosen because it is used widely and it is not an empirical equation as it can be derived using a simple model of vapor−liquid interface and certain simplifications.[6]

Experimental Section

Materials. Organic compounds used as solutes including their source and purity are specified in Table 1. Water used as the solvent was distilled and subsequently treated by Milli-Q Water Purification System (Millipore, USA).

Apparatus and Procedure. A commercial ring tensiometer, Lauda TD1, was used to measure the surface

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10.1021/je049955d CCC: $27.50

tension σ of pure compounds as well as their aqueous solutions. The value of surface tension was obtained by multiplying the reading shown on the display of the device, σ_n, by a correction factor specified by the manufacturer

$$\sigma = \left(0.8759 + 0.0009188\,\frac{\sigma_n}{\rho}\right)\sigma_n \qquad (1)$$

where ρ is the density of the measured liquid. The densities of the pure compounds were taken from Hovorka et al.[7] (for most of the solutes), from CDATA database[8] (2-butanol, ethyl acetate), and from Douhéret and Pal[9] (ethylene glycol). Since very dilute aqueous solutions were studied, densities of these solutions were considered to be equal to the value of density of pure water ($\rho = 0.99703$ g·cm^{-3} at 298.15 K), except for more concentrated aqueous solutions of ethylene glycol. Densities of this system were taken from Ray and Némethy.[10]

Concentration of solutions, prepared by mass, ranged generally from molar fraction $x_2 = 0.015$ to $x_2 = 0.0002$ (except water (1) + ethylene glycol (2)). All the solutions were kept in closed bottles at (25.0 ± 0.1) °C for 2 h, and then approximately 25 mL of solution was added into the measuring glass vessel that was placed in the glass thermojacket of the Lauda tensiometer. A Medingen U10 thermostat equipped with an electronic unit from Labsystem, Prague, Czech Republic was used to regulate temperature. The temperature of sample was kept at (25.0 ± 0.1) °C and measured by Platinum Thermometer Greisinger GMH2000.

The platinum ring was washed between single measurements in deionized water and methanol and then annealed in a flame of an alcohol-fueled burner. Since the relative volatility of components of solutions was sufficiently low, the change of concentration due to vaporization from solution during the measurement can be neglected. Typical reproducibility of replicated measurements was ±0.1 mN·m^{-1}.

Results and Discussion

Experimental data for pure compounds and solutions are given in Tables 2 and 3, respectively. Each reported value of surface tension represents an average of eight measurements. Table 2 also shows literature values for comparison. The differences between our values and literature values in the case of pure water are within the experimental uncertainty (i.e., 0.1 mN/m). The differences in the case of single pure organic compounds are often higher than uncertainty estimated from the experimental uncertainty of our value (i.e., 0.1 mN/m) and the literature value (typically 0.1 mN/m). It is surprising that these differences are great even for such compounds with a simple structure as 1-butanol and 2-butanone. To test if low purity could explain this fact, we measured surface tension of three samples of pure 1-butanol with different purities (98.89, 99.91, and 99.995% determined by GC). The results of these test measurements agreed within the experimental error, and therefore purity is not possibly such an important problem for studied systems. To test the possible effect of impurities in the case of aqueous solutions, we still prepared aqueous solutions of 1-butanol with the same concentration from the mentioned three samples of 1-butanol with different purity. Values of surface tension of all solutions were the same within experimental uncertainty ±0.1 mN·m^{-1}. The results of these tests, together with the result for pure water, suggest that our measurements are accurate within an experimental uncertainty of ±0.1 mN·m^{-1}.

Table 2. Experimental Values of Surface Tension at 298.15 K Obtained in This Work for Water and 11 Pure Organic Solutes Studied Compared with Literature Data

	σ/mN·m^{-1}		
compound	this work	literature	Δ/mN·m^{-1} [a]
water	71.95[b]	72.1[8,c]	−0.15
		71.8[11,c]	+0.15
		71.99[12]	−0.04
		72.13[13]	−0.18
		72.04[14]	−0.09
		71.98[14]	−0.03
		72.14[15]	−0.19
		72.01[16]	−0.06
		71.99[17]	−0.04
		72.01[18]	−0.06
		71.98[19]	−0.03
1-butanol	24.3	24.9[20,c]	−0.6
		24.02[21]	+0.28
		24.2[22]	+0.1
		24.2[23]	+0.1
		23.7[24]	+0.6
		24.1[25]	+0.2
2-butanol	23.3	23.0[26]	+0.3
		23.0[24]	+0.3
		21.5[25]	+1.8
cyclopentanol	32.8	32.5[20,c]	+0.3
ethylene glycol	47.8	48.9[27,d]	−1.1
		48.0[28,e]	−0.2
		48.6[29]	−0.8
		48.0[30]	−0.2
2-butanone	24.2	23.96[31,f]	+0.24
		23.2[32]	+1.0
		23.84[33]	+0.36
cyclopentanone	33.0	32.8[20,c]	+0.2
		33.2[11,c,g]	−0.2
acetyl acetone	30.9	30.4[20,c]	+0.5
		29.5[34]	+1.4
ethyl acetate	23.7	23.4[20,c]	+0.3
		23.82[35]	−0.12
2-ethoxyethyl acetate	28.7	31.8[11,c]	−3.1
methyl acetoacetate	35.7		
propionitrile	26.9	26.7[20,c]	+0.2
		26.7[28,e]	+0.2

[a] Difference between the value of σ obtained in this work and the literature value of σ. [b] Average calculated from results of 30 replicated measurements (measurement of pure water was done daily to check apparatus). [c] Secondary reference citing an original unavailable source. [d] Value of surface tension obtained by linear interpolation from published values of surface tension at temperatures (283.15, 293.15, 303.15, and 313.15) K, respectively. [e] Value of surface tension obtained by linear interpolation from published values of surface tension at temperatures (293.15 and 353.15) K, respectively. [f] Value of surface tension obtained by linear interpolation from published values of surface tension at temperatures (297.95, 308.00, and 323.30) K, respectively. [g] Value of surface tension obtained by linear interpolation from published values of surface tension at temperatures (293.15 and 303.15) K, respectively.

For the sake of comparison with the literature data for aqueous solutions, which are usually published as surface pressures rather than surface tension, we calculated the surface pressure values as the differences between our value of the surface tension of water ($\sigma_1^\bullet = 71.95$ mN·m^{-1}) and our surface tension data for solutions (σ, Table 3) as

$$\pi = \sigma_1^\bullet - \sigma \qquad (2)$$

The data on concentration dependence of surface tension/pressure for 1-butanol in water were found in papers of Dunning and Washburn,[24] Bennes and Bou Karam,[25] Posner et al.,[36] Clint et al.,[37] Glinski et al.,[38] and Donaldson and Anderson.[39] The last two of the cited papers[38,39] give the results in graphic form only, and therefore the com-

Table 3. Experimental Values of Surface Tension at 298.15 K as a Function of Solute Mol Fraction x_2 for Aqueous Solutions of 11 Organic Solutes[a]

x_2	σ/mN·m^{-1}	x_2	σ/mN·m^{-1}	x_2	σ/mN·m^{-1}
		1-Butanol			
0.014110	29.1	0.006003	40.7	0.000778	62.5
0.012883	29.9	0.003931	45.8	0.000596	64.4
0.012038	31.1	0.002872	49.2	0.000392	66.9
0.010125	33.7	0.001997	53.9	0.000292	68.1
0.008156	36.4	0.001457	56.5	0.000235	68.8
0.006847	38.3	0.001004	60.4	0.000138	69.4
		2-Butanol			
0.013940	36.8	0.005882	46.4	0.000718	64.8
0.011986	38.5	0.003914	50.9	0.000443	67.3
0.009938	40.6	0.001994	57.5	0.000211	69.7
0.007990	43.1	0.001000	62.6	0.000154	70.2
		Cyclopentanol			
0.014477	38.4	0.003934	52.6	0.000713	67.3
0.011894	40.6	0.002728	56.3	0.000574	68.0
0.010253	42.4	0.002519	57.5	0.000525	68.5
0.008154	44.8	0.001816	60.2	0.000369	69.6
0.005987	48.3	0.000986	65.1	0.000201	70.8
0.005255	49.6				
		Ethylene Glycol			
0.199955	57.9	0.029922	68.2	0.015846	69.7
0.100976	62.5	0.024884	68.8	0.014233	70.2
0.045192	66.6	0.021976	69.2	0.011850	70.4
0.040102	67.3	0.020003	69.4	0.010123	70.7
0.035421	67.7	0.018225	69.6	0.008251	70.8
		2-Butanone			
0.014135	47.4	0.002056	63.1	0.000296	70.0
0.012048	48.8	0.000997	66.8	0.000112	71.3
0.010219	50.4	0.000855	67.3	$8.96 \cdot 10^{-5}$	71.5
0.008019	52.7	0.000741	67.8	$7.54 \cdot 10^{-5}$	71.7
0.006112	55.1	0.000522	68.9	$5.71 \cdot 10^{-5}$	71.8
0.004050	58.6				
		Cyclopentanone			
0.014276	50.3	0.003863	61.1	0.000768	68.9
0.012150	52.0	0.002445	63.6	0.000559	69.6
0.009964	53.7	0.001362	67.0	0.000367	70.3
0.007908	55.5	0.000966	68.3	0.000186	71.1
0.006022	57.8				
		Acetyl Acetone			
0.010300	53.3	0.004045	61.4	0.000578	69.5
0.008089	55.6	0.001976	65.5	0.000490	69.9
0.006054	58.3	0.001016	68.3	0.000412	70.1
0.004973	59.8	0.000783	69.1	0.000205	70.7
		Ethyl Acetate			
0.014011	35.2	0.006035	46.7	0.000819	63.7
0.011950	37.8	0.003938	51.2	0.000617	65.2
0.009946	40.5	0.002074	57.3	0.000351	67.7
0.008035	43.4	0.000997	62.3	0.000189	69.6
		2-Ethoxyethyl Acetate			
0.014967	41.4	0.001352	60.3	0.000407	66.0
0.013003	43.2	0.000958	62.4	0.000221	67.9
0.010908	44.7	0.000777	63.5	0.000190	68.4
0.007004	48.6	0.000582	64.5	0.000178	68.9
0.005086	51.2	0.000460	65.6	0.000101	70.0
0.003046	55.2				
		Methyl Acetoacetate			
0.014040	53.2	0.005842	60.2	0.000793	69.3
0.012061	54.5	0.003971	62.4	0.000609	69.9
0.009960	56.1	0.001822	66.6	0.000411	70.4
0.008086	57.8	0.001003	68.6	0.000186	71.1
		Propionitrile			
0.014338	49.9	0.005968	59.5	0.000784	69.5
0.012002	52.4	0.003895	62.9	0.000563	70.3
0.010153	54.2	0.001988	66.6	0.000368	71.0
0.008178	56.6	0.000963	69.2	0.000183	71.6

[a] Uncertainty of σ is ±0.1 mN·m^{-1} (see the text). Uncertainty of x_2 was estimated using the error propagation law from equation $s(x_2) = \{[m_1{}^2 s^2(m_1) + m_2{}^2 s^2(m_2)]M_1{}^2 M_2{}^2/(m_1 M_2 + m_2 M_1)^4\}^{0.5}$, where M_i are molar masses of solute and solvent, m_i are weights of compounds used at preparation of solution, and $s(m_i)$ are uncertainties of m_i estimated as 3×10^{-4} g. Uncertainty of x_2 ranged from (6−7) × 10^{-7} in all cases.

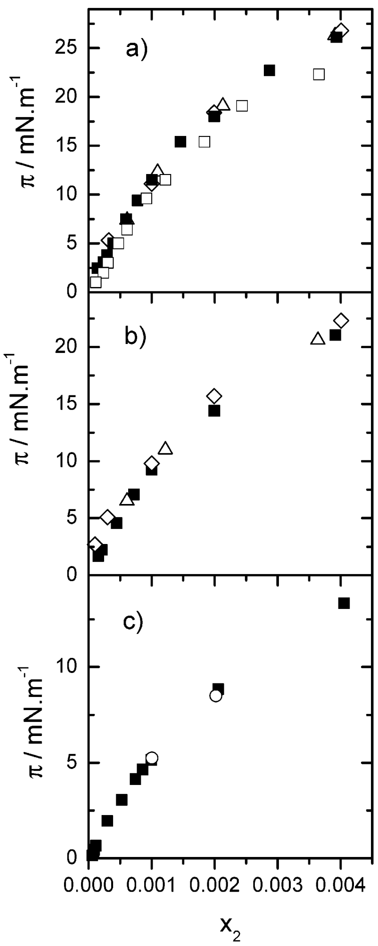

Figure 1. Comparison of surface pressure values from this work with literature data. (a) 1-Butanol: ■, this work; □, Posner et al.;[36] △, Dunning and Washburn;[24] ◇, Bennes and Bou Karam.[25] (b) 2-Butanol: ■, this work; △, Dunning and Washburn;[24] ◇, Bennes and Bou Karam.[25] (c) 2-Butanone: ■, this work; ○, Teitel'baum et al.[33]

parison with our data was not possible. Agreement of our results with data from refs 24 and 25 is very good, differences being lower than 0.2 mN·m^{-1} (see Figure 1). Clint et al.[37] published only value of limiting slope obtained by linear regression of their data for very dilute solutions: $(d\pi/dx_2)_{x_2 \to 0} = -12\,330$ mN·m^{-1}. The value of this slope is in very good agreement with the limiting slope obtained by the same way from our data (see discussion below and Table 5). The discrepancy between our data and Posner's data[36] is greater. Considering that our results agree well with data of three authors, we believe that the accuracy of Posner's values is questionable.

Other experimental data found in the literature concerned 2-butanol[24,25] at 298.15 K. The difference between our data and Dunning's data[24] does not exceed the experimental error of 0.1 mN·m^{-1}. Surprisingly, the agreement with Bennes' and Bou Karam's work[25] is not very good. It may be explained by the fact that the surface tension of pure 2-butanol published in ref 25 is rather different from our result as well as from the literature data (see Table 2). Therefore we think that there is a possibility of significant error in the cited paper[25] in the case of measurement with 2-butanol.

2-Butanone is the last substance for which the data on surface tension of aqueous solution were found in the literature.[33] The agreement (after correction for an error of conversion from volume fraction to molar fraction that

Table 4. Results of Weighted Correlation of Experimental Surface Pressure Data by Szyszkowski Equation ($\sigma_1^\bullet - \sigma = \pi = a \ln[1 + bx_2]$)

solute	a^a/mN·m^{-1}	$s(a)^a$/mN ·m^{-1}	b^a	$s(b)^a$	s^b	$s_{avg}{}^c$/mN·m^{-1}	$(\partial\pi/\partial x_2)_{x_2\to 0}{}^d$/mN·m^{-1}
1-butanol	15.23	0.24	1135	44	2.82	0.32 (0.67)	17290
2-butanol	12.94	0.16	1027	30	1.34	0.15 (0.30)	13290
cyclopentanol	14.51	0.63	658	60	4.73	0.57 (1.01)	9550
ethylene glycol	10.89	0.33	13.3	0.6	0.80	0.09 (0.23)	145
2-butanone	10.92	0.16	600	18	0.99	0.10 (0.30)	6550
cyclopentanone	10.90	0.23	435	18	1.15	0.12 (0.34)	4750
acetyl acetone	12.22	0.45	342	21	1.21	0.13 (0.37)	4180
ethyl acetate	13.28	0.39	988	69	3.40	0.41 (0.86)	13110
2-ethoxyethyl acetate	8.33	0.17	2302	130	2.88	0.33 (0.75)	19180
methyl acetoacetate	10.11	0.15	379	10	0.60	0.06 (0.18)	3830
propionitrile	17.82	0.52	168	8	0.91	0.09 (0.22)	3000

[a] Parameters of the Szyszkowski equation and uncertainty of parameters obtained by weighed statistical correlation. [b] Standard deviation of fit $s = (S_{\min}/(N - 2))^{1/2}$, where $S_{\min}$ is the objective function minimized $S_{\min} = \sum_{i=1}^{N}(\pi_{\exp,i} - \pi_{fit,i})^2/s^2(\pi_{\exp,i})$, N number of experimental points, $\pi_{\exp,i}$ is experimental value of surface presure, and $\pi_{\mathrm{fit},i}$ value calculated using parameters of Szyszkowski equation for the same composition as in the case of experimental point. $s(\pi_{\exp,i})$ is the estimate of standard deviation for experimental point. [c] Average absolute deviation $|\pi_{\mathrm{fit}} - \pi_{\exp}|_{\mathrm{average}} = \sum_{i=1}^{N}|\pi_{\mathrm{fit},i} - \pi_{\exp,i}|/N$. Maximum absolute value of difference $\pi_{\mathrm{fit},i} - \pi_{\exp,i}$ is given in parentheses. [d] Limiting value of first derivative of surface tension with respect to molar fraction of solute x_2 that was calculated from obtained parameters of the Szyszkowski equation.

Table 5. Values of the Limiting Slope of Surface Pressure Concentration Dependence Obtained by Linear Regression of the Most Diluted Concentration data (Typically $x_2 < 0.001$)

solute	$(\partial\pi/\partial x_2)_{x_2\to 0}$/mN·m^{-1}	$s[(\partial\pi/\partial x_2)_{x_2\to 0}]^a$/mN·m^{-1}
1-butanol	12010	320
2-butanol	10040	170
cyclopentanol	6640	140
ethylene glycol	126	3
2-butanone	5370	120
cyclopentanone	3890	90
acetyl acetone	4270	230
ethyl acetate	10520	420
2-ethoxyethyl acetate	17790	370
methyl acetoacetate	3330	72
propionitrile	2820	95

[a] Standard deviation estimate for limiting slope resulting from fitting.

was done in the Teitel'baum et al. article)[33] corresponds to experimental uncertainty of 0.1 mN/m of data from the both sources.

Our experimental data for all solutions were correlated by the Szyszkowski equation

$$\sigma_1^\bullet - \sigma = \pi = a \ln[1 + bx_2] \tag{3}$$

to decide whether this equation is suitable for description of surface pressure concentration dependence of very dilute aqueous solutions. Table 4 shows the parameters a and b of the Szyszkowski equation obtained by weighed regression together with the standard deviation of correlation s, the limiting value of the derivative $(\mathrm{d}\pi/\mathrm{d}x_2)_{x_2\to 0}$ (calculated from parameters of the Szyszkowski equation), and the average difference between experimental values and those calculated from correlation equation (maximal difference is given in parentheses). It can be seen that the Szyszkowski equation does not express our experimental data with an accuracy corresponding to the experimental uncertainty of ±0.1 mN·m^{-1}. Moreover, the scattering of experimental data around a correlation curve is not stochastic. There are parts of the concentration interval where residuals are positive and parts where residuals are negative. The values of the limiting slope derived from parameters of the Szyzkowski equation (see Table 4) are also obviously wrong; they are very different from the values obtained by linear regression of $\pi(x_2)$ data in the range of very dilute solutions, where the concentration dependence of surface pressure can be regarded as linear (Table 5). These values obtained by linear regression are probably more reliable; as mentioned above, the limiting slope value for 1-butanol evaluated in this manner is in good agreement with literature value of Clint et al.[37] (12330 mN/m).

Conclusions

This work has provided accurate surface tension data for dilute aqueous solutions of organic compounds with different function groups. These data together with limiting slope values, evaluated by linear regression of concentration dependence of surface pressure, will be used subsequently at description of mutual relations between bulk and surface phases. It has been found by statistical treatment that Szyszkowski equation is not suitable for the correlation of experimental surface pressure/tension data of aqueous solutions, especially when values of the first derivative ($\mathrm{d}\pi/\mathrm{d}x_2$) are wanted.

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Received for review January 26, 2004. Accepted April 28, 2004. This work was supported by the MSM 2234 00008 Grant from Ministry of Education of the Czech Republic and post-doc Grant 203/02/D117 from Grant Agency of the Czech Republic.

JE049955D