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Surface Thermodynamics of Aqueous Solutions of Morpholine and Methylmorpholine

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The surface tension of aqueous solutions of morpholine and methylmorpholine has been measured at temperatures from (298.15 to 328.15) K, covering the whole range of concentration. The surface tension of the solutions decreases with increasing morpholine concentrations at the water-rich side, and it remains practically constant for the rest of solutions. The surface enthalpy and surface entropy have been obtained from the temperature variation of the surface tension at constant mole fraction. The surface entropy of these aqueous solutions shows a nonrandom behavior at very low concentrations of morpholine and of methylmorpholine. There are two maxima (at $x_2 = 0.025$ and $x_2 = 0.085$) in the surface entropy of morpholine (2) + water (1) mixtures; methylmorpholine (3) + water (1) mixtures show one (at $x_3 = 0.048$). The surface enthalpies are similar to the surface entropies. The binding constants of morpholine and methylmorpholine on the water surface were calculated, and in both cases, the binding constants are temperature dependent. For morpholine, it increases with temperature; for methylmorpholine, it decreases.

1. Introduction

Amino alcohols, amino ethers, and their binary and ternary mixtures with water have been used for the sweetening of sour gases from natural gas streams and for many other applications.[1,2] The physical and chemical properties of these multifunctional (alkyl, amine, hydroxyl, and ether) compounds both as pure compounds and also mixtures with water are needed.[1,2] The physical and thermodynamic properties of pure linear amino alcohols such as density and viscosity,[3] thermal conductivity,[4] and heat capacity[5] have been reported earlier. The densities and excess molar volumes of aqueous solutions of amino alcohols,[6−11] viscosities,[12−14] and excess molar enthalpies of these binary mixtures[6,15−17] have been studied.

Densities of aqueous solutions of morpholine at low concentration have been studied by Kiyohara et al.[18] at 298.15 K and by Tremaine et al.[19] from (298.15 to 328.15) K. The density and excess molar volumes of (water + morpholine) and (water + methylmorpholine) mixtures at temperatures from (298.15 to 353.15) K have been reported from this laboratory.[20] The surface tensions of the aqueous solutions of the linear amino alcohols[21−26] were also studied. In this work, the surface properties of aqueous solutions of morpholine and methylmorpholine were investigated covering the full range of concentrations at temperatures from (298.15 to 328.15) K.

2. Experimental Section

2.1. Materials. Morpholine [$HN(CH_2CH_2)_2O$, 99%] and methylmorpholine [$CH_3N(CH_2CH_2)_2O$, 99%] were obtained from Aldrich Chemicals. These compounds were used as received, after confirmatory analysis by titration with standard hydrochloric acid. Nanopure water with a resistivity of 18.4 MΩ·cm was used. Mixtures of the morpholines with water were made by mass, with care being taken to minimize exposure to air (carbon dioxide). The estimated uncertainty in the mole fraction is about ±0.0001.

2.2. Surface-Tension Measurements. A Wilhelmy plate apparatus was used consisting of a 20 mm wide platinum plate suspended by a Krüss electrobalance connected to a Krüss processor tensiometer K-12 (Hamburg, Germany), which determines directly the surface-tension values at equilibrium. Solutions were measured in a 9 cm diameter vessel, and the temperature was controlled within ±0.5 K. Before beginning a series of experiments, the plate and vessel were rinsed with distilled water and the surface tension of water measured to ensure that the value was 72.01 ± 0.01 mJ/m^2 at 298.15 K. A solution was poured in the vessel and placed in the tensiometer. The surface-tension measurements were normally made in triplicate and the average taken. A Teflon sheet with a hole for the plate was used to cover the vessel during the measurements. This cover was useful for better temperature control and also minimized the absorption of the CO_2 from air by the morpholine solutions.

2.3. Results. The surface tensions of morpholine (2) + water (1) and methylmorpholine (3) + water (1) mixtures at temperatures from (298.15 to 328.15) K are given in Tables 1 and 2, respectively. Extrapolation of the results for pure morpholine to a temperature of 293.15 K allows a comparison with the value of Friedman et al.[27] The extrapolated value is 38.3 mJ/m^2 compared with the reported value of 38.7 mJ/m^2. The composition variation of the surface tension for the two mixtures at 298.15 K is shown in Figure 1. This plot shows that the surface tension of water is reduced much more sharply by adding methylmorpholine than morpholine. The reduction of the surface tension is in the low mole fraction region of ($x_2 = 0.0$ to $x_2 = 0.1$), and for the rest on the compositions ($x_2 = 0.1$ to $x_2 = 1.0$), the change in the surface tension is small. These plots are practically parallel for the mole fraction of $x_2 = 0.1$ to $x_2 = 1.0$. This shows the reducing effect of the methyl

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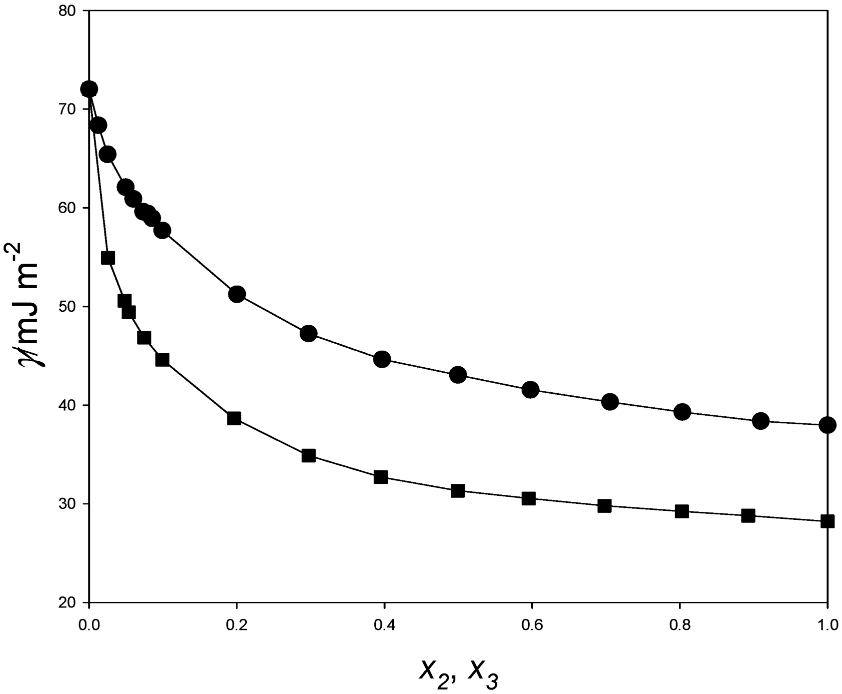

Figure 1. Surface tension of mixtures at 298.15 K: ●, water (1) + morpholine (2); ■, water (1) + methylmorpholine (3).

Table 1. Surface Tension γ (mJ·m^{-2}) of Morpholine (2) + Water (1) Mixtures at Temperatures from (298.15 to 328.15) K

	T/K			
x_2	298.15	308.15	318.15	328.15
0.0000	72.01	70.41	68.79	67.17
0.0125	68.36	66.65	64.55	62.68
0.0155	67.66	65.85	66.74	61.80
0.0251	65.41	63.27	61.14	59.00
0.0496	62.08	60.05	58.03	56.00
0.0600	60.89	58.94	56.98	55.03
0.0735	59.60	57.66	55.72	53.78
0.0793	59.43	57.69	55.63	53.65
0.0854	58.94	56.56	54.18	51.80
0.0991	57.71	55.42	53.12	50.83
0.2005	51.23	49.14	47.05	44.96
0.2973	47.24	45.52	43.80	42.09
0.3969	44.64	43.20	41.75	40.31
0.4996	43.06	41.70	40.33	38.97
0.5977	41.55	40.25	38.96	37.66
0.7005	40.33	39.12	37.91	36.70
0.8034	39.30	38.10	36.90	35.69
0.9096	38.38	37.16	35.95	34.73
1.0000	37.68	36.46	35.24	34.02

Table 2. Surface Tension γ (mJ·m^{-2}) of Methylmorpholine (3) + Water (1) Mixtures at Temperatures from (298.15 to 328.15) K

	T/K			
x_3	298.15	308.15	318.15	328.15
0.0000	72.01	70.41	68.79	67.17
0.0082	69.64	67.64	65.75	63.98
0.0256	54.94	53.18	51.42	49.66
0.0481	50.58	48.64	46.70	44.76
0.0539	49.42	47.71	46.00	44.30
0.0747	46.86	45.35	43.83	42.32
0.0992	44.62	43.24	41.87	40.49
0.1962	38.66	37.36	36.05	34.75
0.2972	34.90	33.66	32.43	31.19
0.3950	32.72	31.54	30.35	29.16
0.4993	31.33	30.17	29.02	27.86
0.5952	30.56	29.47	28.37	27.28
0.6979	29.81	28.73	27.65	26.57
0.8028	29.24	28.14	27.03	25.93
0.8925	28.81	27.69	26.57	25.45
1.0000	28.22	27.20	26.18	25.15

group on the nitrogen atom is the same for this concentration range. This observation is consistent with the results for aqueous solutions of organic compounds with an alkyl end group.[21−26,29−31]

The variation of the surface tension of (water + morpholine) mixtures with temperature was linear. A linear variation of surface tension with temperature for pure compounds was found by Jasper[28] and for aqueous solutions of linear amino alcohols by many workers.[21−26] The temperature dependence of the surface tension is presented as

$$\gamma = K_1 + K_2T \tag{1}$$

where γ is the surface tension, K_1 and K_2 are fitting coefficients, and T is the absolute temperature. These coefficients for (morpholine + water) and (methylmorpholine + water) mixtures are given in Tables 3 and 4, respectively.

2.4. Surface Thermodynamics.

Thermodynamic properties of the surface of the solutions are obtained by the following equations[25,29−31]

$$S^S = -\left(\frac{\partial\gamma}{\partial T}\right)_{x,P} \tag{2}$$

where S^S is the surface entropy and

$$H^S = \gamma - T\left(\frac{\partial\gamma}{\partial T}\right)_{x,P} \tag{3}$$

where H^S is the surface enthalpy. The surface entropy values are obtained from the slopes of the temperature plots of the surface tension. The surface entropy values are constant in the temperature range of (298.15 to 328.15) K. The composition variations of the surface entropy for aqueous solutions of morpholine and methylmorpholine are presented in Figure 2. The composition dependence of the surface entropy shows two maxima for the (morpholine + water) and one for (methylmorpholine + water) systems.

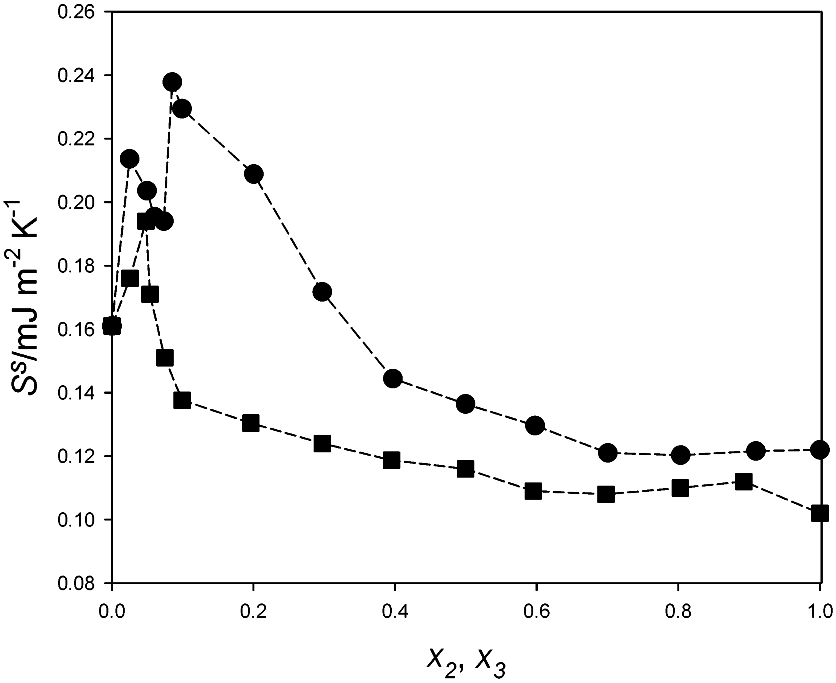

Figure 2. Surface entropy of mixtures in the temperature range (298.15 to 328.15) K: ●, water (1) + morpholine (2); ■, water (1) + methylmorpholine (3).

Table 3. Surface-Tension Parameters K_1 and K_2 from the Linear Fitting of the Surface Tension of the Binary Mixtures of Morpholine (2) + Water (1) in the Temperature Range of (298.15 to 328.15) K

x_2	K_1/mJ·m^{-2}	K_2/mJ·m^{-2}·K^{-1}
0.0000	120.03	−0.161
0.0125	125.50	−0.191
0.0251	129.09	−0.214
0.0496	122.48	−0.203
0.0600	119.15	−0.195
0.0735	117.44	−0.194
0.0854	129.85	−0.238
0.0991	126.11	−0.229
0.2005	113.48	−0.209
0.2973	98.43	−0.172
0.3969	87.69	−0.144
0.4996	83.73	−0.136
0.5977	80.19	−0.130
0.7005	76.41	−0.121
0.8034	75.17	−0.120
0.9096	74.63	−0.122
1.0000	74.05	−0.122

Table 4. Surface-Tension Parameters K_1 and K_2 from the Linear Fitting of the Surface Tension of the Binary Mixtures of Methylmorpholine (3) + Water (1) in the Temperature Range of (298.15 to 328.15) K

x_3	K_1/mJ·m^{-2}	K_2/mJ·m^{-2}·K^{-1}
0.0000	120.03	−0.161
0.0082	125.84	−0.189
0.0256	107.41	−0.176
0.0481	108.42	−0.194
0.0539	100.35	−0.171
0.0747	92.00	−0.151
0.0992	85.64	−0.138
0.1962	77.54	−0.130
0.2972	71.75	−0.124
0.3950	68.11	−0.119
0.4993	65.80	−0.116
0.5952	63.18	−0.109
0.6979	62.01	−0.108
0.8028	62.16	−0.110
0.8925	62.20	−0.112
1.0000	58.72	−0.102

The same trend has been found for (water + alcohol) mixtures. Gliński et al.[29] have found one maximum for (water + *tert*-butyl alcohol) and two maxima[30] for (water + propanol) mixtures at very low concentrations of the alcohols. This kind of surface entropy variation has been related[31] to a molecular organization on the water surface. This will depend on the conformational possibility of the alkyl group of the alcohol. In the case of methylmorpholine, the methyl group has only one configurational shape, which is comparable to the *tert*-butyl group of the *tert*-butyl alcohol, which has one maximum.[29] Morpholine has no end alkyl group, but still there are two maxima. We need more studies to bring some light to this case.

The surface enthalpy values were obtained by eq 3, and they are given in Table 6 for morpholine and methylmorpholine solutions at 298.15 K. The composition dependence of the surface enthalpy for (water + morpholine) and (water + methylmorpholine) mixtures at 298.15 K are shown in Figure 3. The trend of the surface enthalpy of these mixtures against the composition is similar to the surface entropy for (water + morpholine), while in the case of (water + methylmorpholine), it becomes less pronounced. This behavior has been reported for aqueous solutions of alcohols.[29−31]

2.5. Surface Binding. Conners and Wright[32] developed a useful model for aqueous solutions of the organic compounds where the bulk and the surface phases are considered separately.[25,32] They presented a general form as

$$\frac{(\gamma - \gamma_1)}{(\gamma_1 - \gamma_2)} = x_2\left(1 + \frac{bx_1}{1 - ax_1}\right) \tag{4}$$

where γ, γ_1, and γ_2 are the surface tension of the mixture, of water, and of the morpholine, x_1 and x_2 are the mole fractions of water and morpholine, and "*a*" and "*b*" are the fitting coefficients. The left side of the equation is called

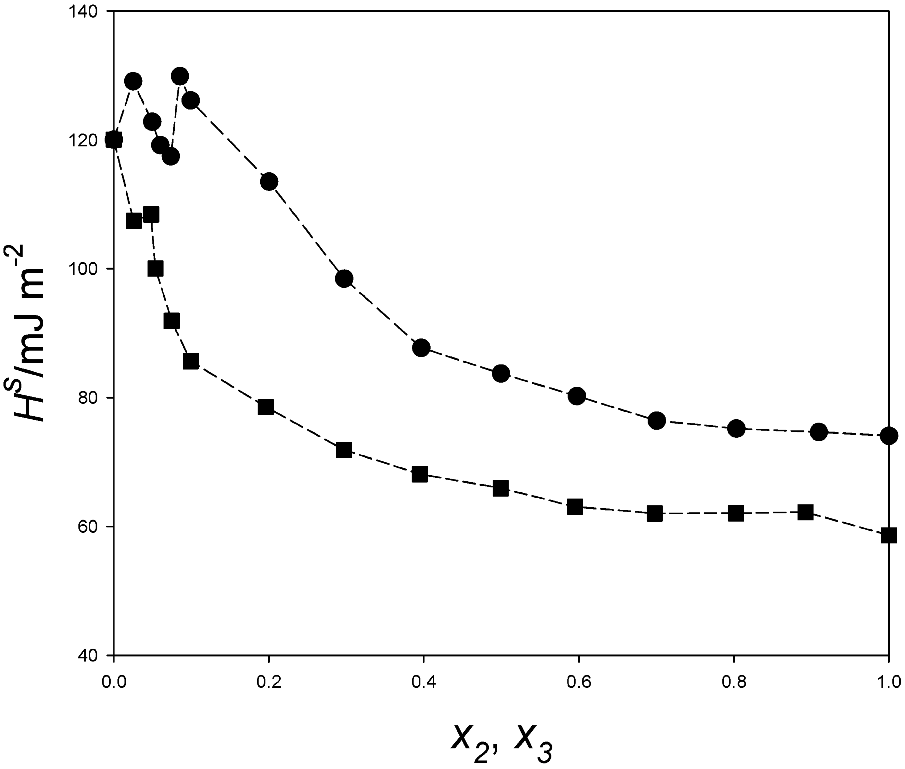

Figure 3. Surface enthalpy of mixtures at 298.15 K: ●, water (1) + morpholine (2); ■, water (1) + methylmorpholine (3).

Table 5. Surface Entropy S^S of Morpholine (2) + Water (1) and Methylmorpholine (3) + Water (1) Mixtures in the Temperature Range of (298.15 to 328.15) K

x_2	S^S/mJ·m^{-2}·K^{-1}	x_3	S^S/mJ·m^{-2}·K^{-1}
0.0000	0.16	0.0000	0.16
0.0155	0.20	0.0256	0.18
0.0251	0.21	0.0481	0.19
0.0496	0.20	0.0539	0.17
0.0600	0.20	0.0747	0.15
0.0735	0.19	0.0992	0.14
0.0854	0.24	0.1962	0.13
0.0991	0.23	0.2972	0.12
0.2005	0.21	0.3950	0.12
0.2973	0.17	0.4993	0.12
0.3969	0.14	0.5952	0.12
0.4996	0.14	0.6979	0.11
0.5977	0.13	0.8028	0.11
0.7005	0.12	0.8925	0.11
0.8034	0.12	1.0000	0.10
0.9096	0.12		
1.0000	0.12		

Table 6. Surface Enthalpy H^S of Morpholine (2) + Water (1) and Methylmorpholine (3) + Water (1) Mixtures in the Temperature Range of (298.15 to 328.15) K

x_2	H^S/mJ·m^{-2}	x_3	H^S/mJ·m^{-2}
0.0000	121	0.0000	121
0.0155	129	0.0256	107
0.0251	123	0.0481	108
0.0496	119	0.0539	100
0.0600	117	0.0747	92
0.0735	130	0.0992	86
0.0854	126	0.1962	78
0.0991	113	0.2972	72
0.2005	98	0.3950	68
0.2973	88	0.4993	66
0.3969	84	0.5952	63
0.4996	80	0.6979	62
0.5977	76	0.8028	62
0.7005	75	0.8925	62
0.8034	75	1.0000	59
0.9096	74		
1.0000	74		

Table 7. Surface-Tension Parameters of Aqueous Solutions of Morpholine and Methylmorpholine at Temperatures from (298.15 to 328.15) K

T/K	a	b	K
	Morpholine (2) + Water (1)		
298.15	0.884	0.778	7.6
308.15	0.897	0.773	8.7
318.15	0.911	0.766	10.2
328.15	0.921	0.762	11.0
	Methylmorpholine (3) + Water (1)		
298.15	0.952	0.885	19.9
308.15	0.949	0.899	18.8
318.15	0.948	0.910	18.3
328.15	0.947	0.919	18.0

the reduced surface tension (γ_{red}), and by defining the quantity $R = \gamma_{\text{red}}/x_2$, eq 4 can be arranged as

$$\frac{x_1}{R-1} = \frac{1}{b} - \frac{ax_1}{b} \tag{5}$$

This equation applies to all the aqueous solutions, (water + alkanolamines)[25] and other organic aqueous solutions.[32] The binding constant K can be obtained by

$$K = \frac{a}{1-a} \tag{6}$$

In Table 7, a, b, and K values from eqs 5 and 6 are given. This shows that parameters a, b, and K are temperature dependent. The binding constants K, of morpholine and methylmorpholine, for temperatures from (298.15 to 328.15) K are shown in Figure 4. This figure shows that the binding constant for morpholine and methylmorpholine on the water surface varies with temperature. It increases with temperature for morpholine, while it decreases for methylmorpholine. This means that a temperature increase makes the water surface a more favorable site for morpholine and a less favorable site for methylmorpholine. We

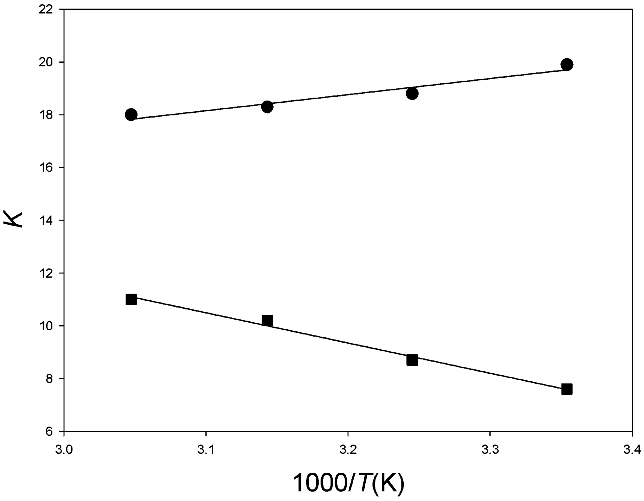

Figure 4. The binding constant on the water surface as a function of temperature: ●, water (1) + morpholine (2); ■, water (1) + methylmorpholine (3).

need more data on the surface tension of the aqueous solutions of morpholines with larger alkyl groups to understand this temperature effect.

3. Conclusions

Surface-tension studies are a useful technique for obtaining the surface thermodynamics at the air−liquid interface. The temperature studies allow us to obtain surface entropy and enthalpy values. These properties are more sensitive to the surface properties than the surface tension itself. The surface entropy behavior at low concentrations reflects a nonrandom distribution of the morpholine and methylmorpholine molecules on the water surface. Similar behavior has been found for mixtures of propanol and *tert*-butyl alcohol with water.

Note Added after ASAP Posting. This article was released ASAP on 3/20/2004. The first column headings in Tables 2 and 3 were transposed. The paper was reposted 3/24/2004.

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