A1 the fatty acids are in a more random arrangement than in B1, a block copolymer containing poly(12-hydroxystearic acid) crystalline at ambient temperatures. A2 has little tendency to order compared with A1, whereas B1 is more ordered.

On the intramolecular scale A3 is more ordered than A1 but less so than B1. Like A2 it contains PEG 600 but the hydrophobe is polyisobutenylsuccinic anhydride, a viscous liquid very different from the poly(12-hydroxystearic acid) of B1, showing no tendency to crystallize, even at temperatures as low as -40 C, whereas the latter crystallizes near 40 C. Polyisobutylene itself forms a coiled chain with the methyl groups staggered around a central axis; it is suggested that it is this effect that gives the polyisobutenyl groupings in A3 the tendency to form a separate phase, but no giant domains are observed, as the total structure is random.

For the degrees of order necessary for liquid crystal formation to occur, alignment of the surfactant molecules is required. Because polyoxyethylene chains interact favorably with each other, they produce the alignment necessary for the formation of a liquid crystal. This alignment is encouraged by the surfactant molecules having a regular structure. B1, an XYX block copolymer, exemplifies the molecular arrangement for this regular repeatable structure to occur, whereas with A2 the lack of regular molecular structure combined with shortness of the polyoxyethylene chains means there are insufficient interactions to produce the alignment necessary for the formation of liquid crystals.

This work was part of a project to determine the efficacy of these compounds as emulsifiers. B1 is an excellent oil+water+electrolyte emulsifier, and the low angle X-ray spacing found is in agreement with the thickness of its bilayer lipid film (6). However, better emulsifiers for the same system do not show liquid crystalline behavior in the same temperature range.

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Surface Properties in Binary Mixtures of Aerosol OT and Nonionic Surfactants

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ABSTRACT

The surface tensions of the mixed systems consisting of sodium di-2-ethylhexyl sulfosuccinate (Aerosol OT) and homogeneous polyoxyethylene glycol-n-dodecyl ether (nED; $n=5$ to 8) were measured in the absence and presence of sodium chloride (NaCl) by a modified Wilhelmy plate method.

In the case of the system containing nED and Aerosol OT, the nED concentrations were fixed at various amounts, and all the surface tension curves approached 32 or 33 mN/m as the concentration of Aerosol OT approached 1×10^{-3} mol/l. On the other hand, when the Aerosol OT concentration was fixed at 1×10^{-3} mol/l, the surface tension curves for the mixed system showed a flat portion in the range where mixed micelle in the solution and the two dimensional ones on the water surface were formed. Further, molecular interaction parameter β was calculated by the Rosen extension of the regular solution treatment of Rubingh. The average values of β in the nED-AOT systems increased as the value of n increased. The values of β increased with increasing activity of AOT.

In the case of the system containing 6ED, AOT and NaCl, the surface tension curves showed inflection points shifted to higher concentrations of AOT as the concentration of nED increased.

INTRODUCTION

There have been many studies on the interactions of nonionic and anionic surfactants in aqueous solution (1-11), in which the mixed micelle formation of mixtures has been studied by measuring the surface tension and by solubilization methods (1-11).

Recently, Meguro et al. (7,12) reported the properties of mixed micelles among sodium dodecyl sulfate (SDS), sodium tetradecyl sulfate (STS) and a series of homogeneous polyoxyethylene glycol-n-dodecyl ether (nED), and they have confirmed that the surface tension curves of nonionic surfactants have a flat portion in combination with SDS or STS. Further, they have interpreted the flat portion as the mixed micelles region.

On the other hand, sodium di-2-ethylhexyl sulfosuccinate (AOT) is an important practical anionic surfactant widely used in nonaqueous as well as in aqueous solutions. Although the micelle formation and solution behavior of AOT in water have been studied by several workers (13-16), there are a few reports about the interactions of AOT with other surfactants.

In this work, the surface properties of mixtures of nonionic surfactants and AOT were investigated by measuring their surface tensions and by calculating the interactions by measuring their surface tensions and by calculating the interaction parameter between them. The effect of salt on the mixtures containing nonionic and AOT surfactants also was examined.

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MATERIALS AND METHODS

Sodium di-2-ethylhexyl sulfosuccinate (AOT) and a series of polyoxyethylene glycol-n-dodecyl ether (nED; n=5 to 8) were supplied by Nikko Chemical Company, Tokyo, Japan.

AOT was a reagent (NIKKOL OTP-100s) whose purity was above 98%. The sample was confirmed to be highly pure by the absence of a minimum in the surface tension log of concentration curve. The surface tension curve obtained in this experiment agreed well with that obtained with an elaborately purified sample by Williams et al. (13). Its critical micelle concentration (CMC) was 2.5×10^{-3} mol/l in water at 25 C.

Nonionic surfactants (nED) were highly pure as confirmed by gas liquid chromatography (GLC), thin layer chromatography (TLC) and surface tension measurements. The water was purified by passing through Milli-Q system (Nihon Millipore Co.) until its specific conductivity fell below 10^{-7} Ω^{-1} cm^{-1}.

The surface tension was determined by a modified Wilhelmy type surface tensometer Shimadzu ST-1. The surface tension of solution of mother liquor was measured at 25.0 C. The establishment of equilibrium was checked by reported measurements in 5-min intervals until a constant surface tension value was obtained for 15 min.

RESULTS AND DISCUSSION

Surface Tension of the Mixed Aqueous Solution of AOT and Nonionic surfactants

The surface tension-log of activity curves of AOT are shown in Figure 1 as a function of added concentration of 6ED. The concentrations of nonionic surfactant in Figure 1 range from below (6.8×10^{-5} mol/l) to above the CMC. When the concentrations of 6ED were changed below the CMC, each of the curves started from the surface tension values of the respective 6ED solutions. The surface tension decreased with increasing concentration of AOT, finally reaching the same values after the CMC of AOT.

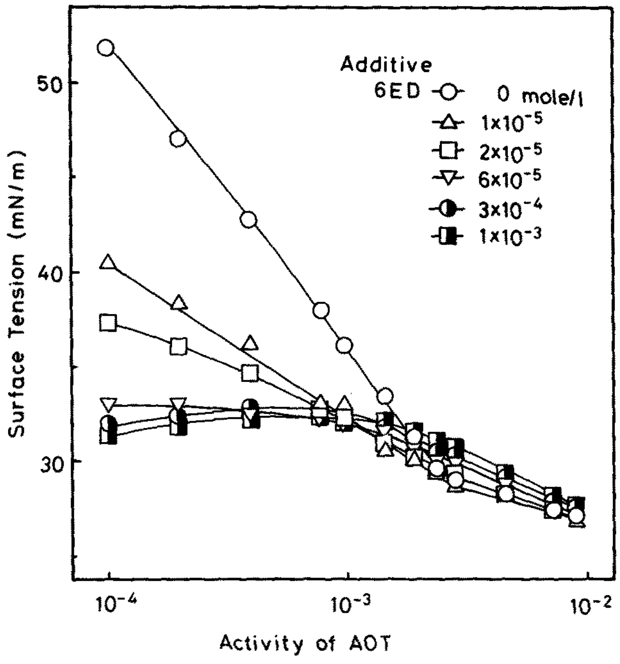

FIG. 1. The surface tensions of AOT as a function of 6ED.

On the other hand, when the concentrations of 6ED were fixed above the CMC, the surface tension of the mixture became nearly equal and coincided with the surface tension at the CMC of 6ED in the low concentration region of AOT, indicating that 6ED molecules occupy most of the surface. The surface tension of the mixture at higher concentration of AOT approaches that of AOT alone above the CMC of AOT. This fact can be explained by the increase in the molecular ratio of AOT/6ED being occupied at the surface. Each curve at different concentrations of 6ED coincides with the same values when the concentration of AOT is 1×10^{-3} mol/l. The surface tension of this mixed system differed significantly from the result obtained for the mixture of sodium dodecyl sulfate (SDS) and nonionic surfactants (7,11). The surface tension of SDS in the presence of nonionic surfactants is similar to the surface tension behavior of SDS containing a certain contaminant, such as dodecanol (7), but the surface tension curves of AOT-6ED systems do not show a minimum value near the CMC. r_{CMC} (surface tension at the CMC of a single surfactant) of the nonionic surfactants 5ED, 6ED, 7ED and 8ED are between r_{CMC} of SDS and of AOT, whereas r_{CMC} of SDS is larger than that of AOT. It is noteworthy that at the concentration of AOT of 1×10^{-3} mol/l, all of the mixtures exhibit the same surface tension values as those observed by Akasu et al. (7).

Figure 2 shows the surface tension curves of 6ED in the presence of AOT below the CMC of AOT. The surface tension decreased as the concentrations of AOT increased in the low concentration region, and each surface tension curve had a knick point. The concentration of 6ED at the knick point, which will be regarded as the critical concentration of mixed micelle formation between 6ED and AOT, decreased with increasing AOT concentration. When the concentration of added AOT reached 1×10^{-3} mol/l, the plateau was maintained over the wide range on the 6ED concentration and the two break points appeared at 2.4×10^{-5} mol/l and 2.7×10^{-3} mol/l. These results suggest that 6ED interacts strongly with AOT at 1×10^{-3} mol/l. The diagram is given schematically in Figure 3.

Region I represents the concentration below 2.4×10^{-5} mol/l (A). The decrease in surface tension suggests that the two kinds of surfactant adsorb on the surface of the solution without forming micelles in the solution.

On the other hand, the mixed micelles consisting of AOT and 6ED will be considered to exist in region II having a flat portion in the curve between 2.4×10^{-5} mol/l (A) and 2.7×10^{-3} mol/l (B). The existence of the plateau between the break points implies that mixed micelles will be formed in this region, as reported previously (7-9,11,20). This suggests that the CMC of mixed micelle begins to be formed from 2.4×10^{-5} mol/l (A) in the mixed solution. Shick (4) and Takasawa (11) have reported that the CMC of mixed

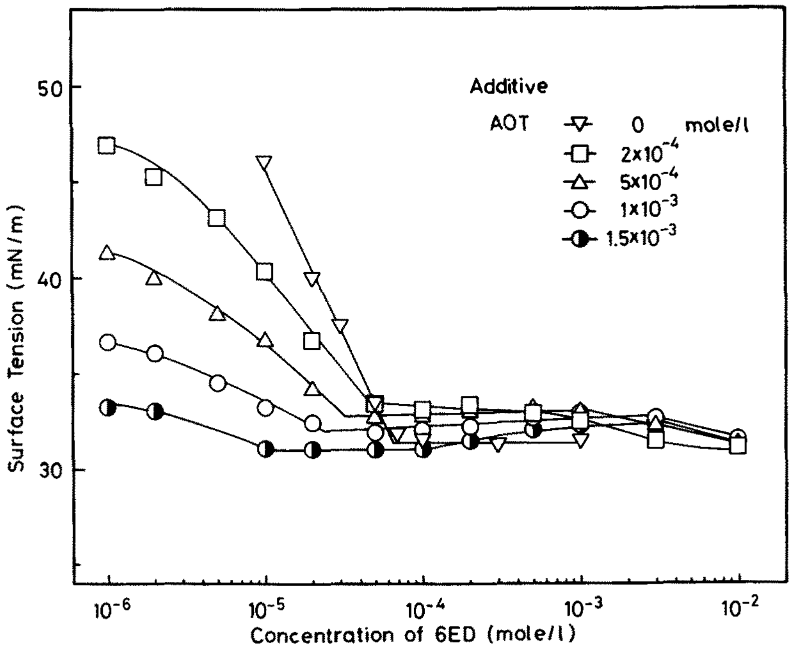

FIG. 2. The surface tensions of 6ED as a function of AOT additive concentrations below the CMC (2.5 × 10^{-3} mol/l).

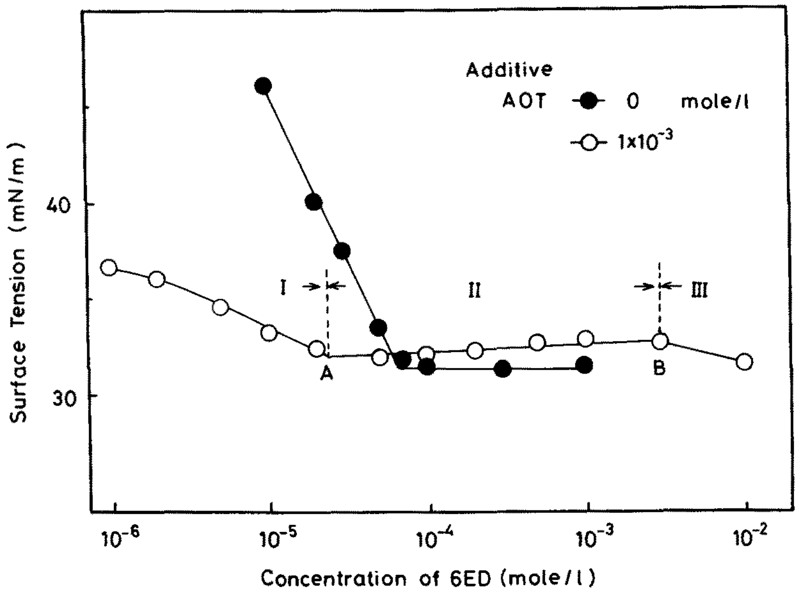

FIG. 3. The surface tensions of 6ED at a constant concentration of AOT.

micelle would be lowered more than that of the separate surfactant.

Region III represents the concentration above 2.7 × 10^{-3} mol/l (B). Some single nonionic surfactant micelle or nonionic surfactant rich mixed micelles are confirmed to be formed according to the results (8,9) obtained with the TCNQ solubilization method.

Figure 4 shows the surface tension curves of a series of nED with ethylene oxide chain lengths from 5 to 8 in the presence of 1 × 10^{-3} mol/l AOT at 25 C. The data indicate that 5ED, 7ED and 8ED show behavior similar to 6ED, and the mixed micelle concentration increases with an increase of the ethylene oxide chain length in nonionic surfactants.

From the facts mentioned above, the existence of the same surface tension region in the mixture of AOT and nonionic surfactants is like that found by Akasu et al. (7)

Calculation of Molecular Interaction Parameter; β

The regular solution treatment of Rubingh (17) for the calculation of the composition of mixed micelles in aqueous solution containing a mixture of two surfactants was extended to the calculation of the composition of the surface phase by Rosen (18, 21). The following two equations are necessary to obtain the values of β.

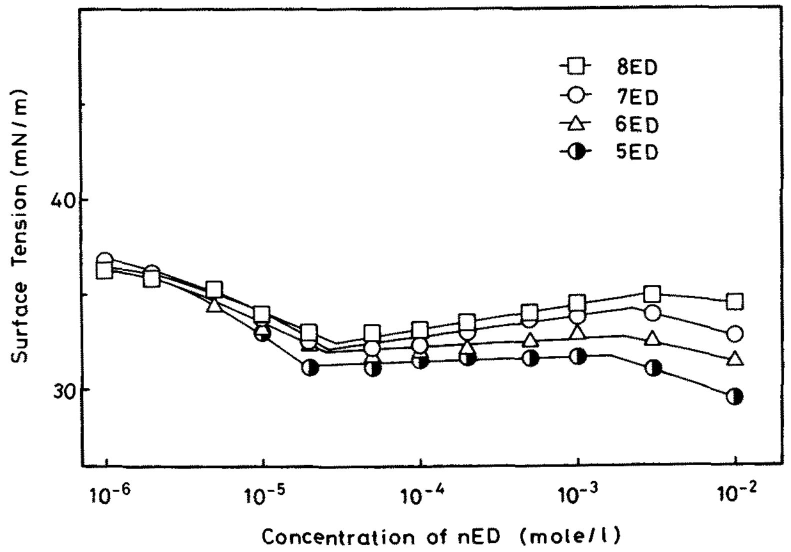

FIG. 4. The surface tensions of homogeneous nED at a constant concentration of AOT.

$$\frac{(X_{B,s})^2 \ln \dfrac{a_B}{a_B^\circ\, x_{B,s}}}{(1 - x_{B,s})^2 \ln \dfrac{a_M}{a_M^\circ\,(1 - x_{B,s})}} = 1 \qquad [1]$$

$$\beta = \frac{\ln \dfrac{a_B}{a_B^\circ\, x_{B,s}}}{(1 - x_{B,s})^2} \qquad [2]$$

Where $x_{B,s}$ is the mole fraction of surfactant ion B in the surface phase and a_B and a_M are the activity of the surfactant ion B and of the nonionic surfactant M, respectively, in the surface phase. a_B° and a_M° are the activity of surfactant ion B and of nonionic surfactant M required to produce the same surface tension in an aqueous solution containing only surfactant AB and nonionic surfactant M, respectively. For aqueous solutions of nonionic surfactant at concentrations of less than 1×10^{-3} mol/l, molar concentrations may be substituted for activities without significant error. For ionic surfactants at concentration of less than 1×10^{-2} mol/l, activity coefficients can be calculated by means of the extended Debye-Hückel equation. Equation [1] can be solved iteratively for $x_{B,s}$. By using the values of $x_{B,s}$, the molecular interaction parameter, β, is obtained from equation [2]. The values of β and of the mole fraction of anionic surfactant in the surface phase are shown in Tables I through VIII.

From the data for systems containing constant activity of AOT (9.66×10^{-4} mol/l), given in Tables I-IV, the average values are −0.9 for the 5ED-AOT system, −1.2 for the 6ED-AOT system, −1.5 for the 7ED-AOT system and −2.6 for the 8ED-AOT system. These results for nED-AOT systems suggest that deviations from the ideal increase with increas-

TABLE I

Surface Mole Fraction and Molecular Interaction Parameter Values in 5ED-AOT Mixtures

Conc. of 5ED (M)	Activity of AOT	S.T. (mN/m)	X(5ED)	B
(× 10^{-6})	(× 10^{-4})			
1.00	9.66	36.3	0.12	−1.5
2.00	9.66	36.1	0.15	−0.95
5.00	9.66	34.5	0.27	−1.0
10.00	9.66	33.4	0.36	−0.7
20.00	9.66	31.3	0.49	−0.5

TABLE II

Surface Mole Fraction and Molecular Interaction Parameter Values in 6ED-AOT Mixtures

Conc. of 6ED (M)	Activity of AOT	S.T. (mN/m)	X(6ED)	B
(× 10^{-6})	(× 10^{-4})			
1.00	1.97	47.0	0.17	−0.6
2.00	1.97	45.4	0.30	−1.0
5.00	1.97	43.2	0.45	−0.9
10.0	1.97	40.4	0.57	−1.0
20.0	1.97	36.8	0.68	−1.2
1.00	4.88	41.4	0.11	−0.9
2.00	4.88	40.1	0.22	−1.4
5.00	4.88	38.2	0.34	−1.5
10.0	4.88	36.8	0.43	−1.1
20.0	4.88	34.3	0.54	−1.1
1.00	9.66	36.7	0.09	−1.3
2.00	9.66	36.1	0.14	−1.3
5.00	9.66	34.6	0.25	−1.4
10.0	9.66	33.3	0.34	−1.3
20.0	9.66	32.5	0.42	−0.6
1.00	14.4	33.3	0.12	−2.3
2.00	14.4	33.1	0.14	−1.7

TABLE III

Surface Mole Fraction and Molecular Interaction Parameter Values in 7ED-AOT Mixtures

Conc. of 7ED (M)	Activity of AOT	S.T. (mN/m)	X(7ED)	β
($\times 10^{-6}$)	($\times 10^{-4}$)			
1.00	9.66	36.9	0.07	−1.4
2.00	9.66	36.3	0.12	−1.6
5.00	9.66	35.3	0.20	−1.5
10.0	9.66	33.8	0.29	−1.7

TABLE IV

Surface Mole Fraction and Molecular Interaction Parameter Values in 8ED-AOT Mixtures

Conc. of 8ED (M)	Activity of AOT	S.T. (mN/m)	X(8ED)	β
($\times 10^{-6}$)	($\times 10^{-4}$)			
1.00	9.66	36.2	0.12	−2.8
2.00	9.66	35.9	0.18	−2.9
5.00	9.66	35.3	0.20	−1.9

TABLE V

Surface Mole Fraction and Molecular Interaction Parameter Values in AOT-5ED Mixtures

Activity of AOT	Conc. of 5ED (M)	S.T. (mN/m)	X(AOT)	β
($\times 10^{-5}$)	($\times 10^{-5}$)			
9.89	1.00	40.8	0.28	−1.1
19.7	1.00	38.7	0.39	−1.2
39.1	1.00	36.4	0.49	−1.2
77.5	1.00	33.2	0.58	−1.4
96.6	1.00	32.4	0.62	−1.3
144.	1.00	31.0	0.70	−0.9

ing length of the ethylene oxide chain of nonionic surfactant, and that the interaction of nonionic surfactant and AOT increases with increasing length of the ethylene oxide chain of nED.

Further, the values of β obtained for system containing constant concentration of nED are shown in Tables V-VIII, and the average values of β are −1.2 for the 5ED-AOT system, −1.2 for the 6ED-AOT system, −2.1 for the 7ED-AOT system and −2.3 for the 8ED-AOT system. The magnitude of β with ethylene oxide chain length of nonionic surfactants shows tendency similar to that described above. Additionally, it is apparent from Table II that the absolute values of β for the systems containing a constant concentration of 6ED increase with increasing AOT activity, suggesting that the interaction of 6ED and AOT increases with increasing AOT activity.

Furthermore, the values of n, the coefficient in the Gibbs adsorption equation for ionic surfactant, were calculated by the use of the value $x_{B,s}$ obtained by equation [1].

TABLE VI

Surface Mole Fraction and Molecular Interaction Parameter Values in AOT-6ED Mixtures

Activity of AOT	Conc. of 6ED (M)	S.T. (mN/m)	X(AOT)	β
($\times 10^{-5}$)	($\times 10^{-5}$)			
9.89	1.00	40.7	0.16	−0.01
39.1	1.00	36.5	0.53	−1.7
77.5	1.00	33.3	0.61	−1.9
9.89	2.00	37.5	0.25	−1.6
39.1	2.00	34.9	0.42	−1.1
77.5	2.00	33.0	0.53	−0.84

TABLE VII

Surface Mole Fraction and Molecular Interaction Parameter Values in AOT-7ED Mixtures

Activity of AOT	Conc. of 7ED (M)	S.T. (mN/m)	X(AOT)	β
($\times 10^{-5}$)	($\times 10^{-5}$)			
9.89	1.00	42.0	0.41	−2.1
19.7	1.00	39.6	0.48	−2.4
39.1	1.00	36.9	0.57	−2.3
77.5	1.00	33.6	0.69	−3.9
9.89	2.00	39.0	0.33	−2.1
19.7	2.00	37.7	0.40	−1.8
9.89	6.00	35.0	0.09	−0.3
39.1	6.00	33.7	0.41	−1.5

TABLE VIII

Surface Mole Fraction and Molecular Interaction Parameter Values in AOT-8ED Mixtures

Activity of AOT	Conc. of 8ED (M)	S.T. (mN/m)	X(AOT)	β
($\times 10^{-5}$)	($\times 10^{-5}$)			
9.89	1.00	42.8	0.43	−2.1
19.7	1.00	40.4	0.52	−2.3
39.1	1.00	37.4	0.60	−2.6
77.5	1.00	35.1	0.70	−2.0

$$\Gamma^{B} = \left(\frac{-\partial r}{\partial \ln a_M} \right) \Big/ nRT \qquad [3]$$

$$\Gamma_{B} = \left(\frac{-\partial r}{\partial \ln a_M} \right) \Big/ nRT \qquad [4]$$

where Γ_B and Γ_M are the surface excess concentrations of the surface active ion B and of the nonionic surfactant M, respectively. From these values, the areas per surfactant ion at the air/solution interface can be calculated. The mole fraction of the surface active ion B at the air/solution interface

$$X_{B,s} = \frac{\Gamma_B}{\Gamma_B + \Gamma_M} \qquad [5]$$

and the value of n can be calculated by the use of equations [3], [4] and [5].

$$n = \frac{(-\partial r/\partial \log a_{AB})_M \, (1 - x_{B,s})}{x_{B,s}(-\partial r/\partial \log a_M)_{AB}} \qquad [6]$$

The area per molecule, σ_B at the air/solution interface is given by

$$\sigma_B = \frac{1}{N\Gamma_B} \qquad [7]$$

where N is Avogadro's number.

In Table IX, the values of the coefficient n and areas per AOT ions, σ_{AOT} are listed. From these results, σ_{AOT} increases with increasing concentration of 6ED. This is because the mole fraction of AOT decreases in the surface film and the mole fraction of 6ED in the surface film increases with increasing the concentration of 6ED.

TABLE IX

The Coefficient of Gibbs Adsorption Equation and the Areas per Surfactant ion in AOT-6ED Mixtures

Activity of AOT	Conc. of 6ED (M)	S_{AOT}	S_{6ED}	n	AOT (Å²)
(× 10^{-4})	(× 10^{-5})				
1.97	1.00	7.9	8.7	1.2	139
1.97	2.00	4.3	9.9	0.89	194
4.88	1.00	8.2	6.4	0.96	111
4.88	2.00	5.9	7.4	0.97	156
9.65	1.00	8.4	3.7	1.1	127
9.65	2.00	7.1	3.9	1.3	174

$S_{AOT} = (-\partial\gamma/\partial \log a_{AOT})c_{6ED}$.
$S_{6ED} = (-\partial\gamma/\partial \log c_{6ED})a_{AOT}$.

Effect of Sodium Chloride

The surface tension vs. log of AOT concentration curve in the presence of 0.04 mol/l sodium chloride is shown in Figure 5. The values of the surface tension and the CMC of AOT decreased with the addition of sodum chloride, which is in good agreement with the result obtained by Knox et al. (19). This depression of the CMC is due mainly to the decrease in the thickness of the ionic atmosphere surrounding the ionic head groups in the presence of sodium chloride.

On the other hand, the addition of sodium chloride lowered the surface tension of 6ED and reduced the CMC value, but had a relatively small effect compared to the ionic surfactant. The decrease in CMC by salt solution has been interpreted in terms of salting out mechanism (23, 24). The addition of salt changes the solvent property and decreases the hydration of the ethylene oxide chain of 6ED (25-27).

The surface tension curve for AOT in 2 × 10^{-5}, 3 × 10^{-4} and 1 × 10^{-3} mol/l 6ED containing sodium chloride of 0.04 mol/l is shown in Figure 5. The CMC of AOT in 0.04 mol/l NaCl solution increases with the concentration of 6ED, indicating that the addition of salts would affect the solubilizing power of ionic

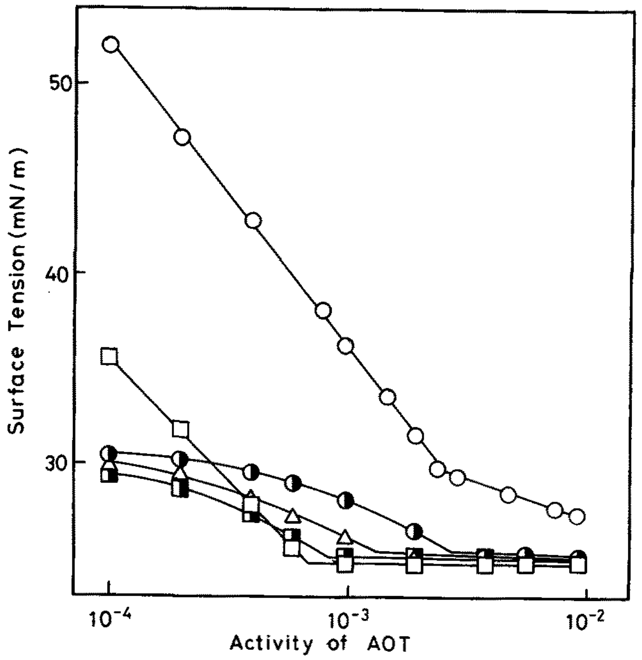

FIG. 5. The surface tensions of mixtures of AOT and 6ED in 0.04 mole/l NaCl; ○, AOT in distilled water; □, AOT in 0.04 mole/l NaCl; ◧, AOT in 2 × 10^{-5} mole/l 6ED containing NaCl at 0.04 mole/l; △, AOT in 3 × 10^{-4} mole/l 6ED containing NaCl at 0.04 mole/l; ◑, AOT in 1 × 10^{-3} mole/l 6ED containing NaCl at 0.04 mole/l.

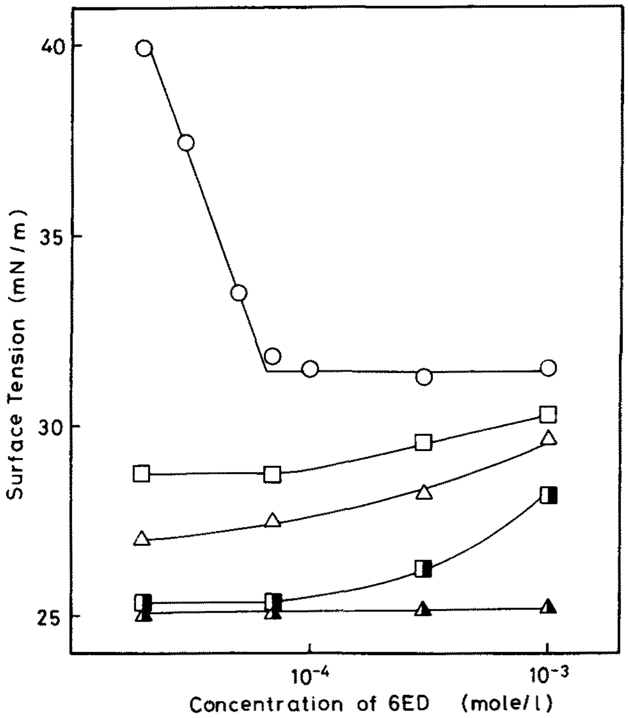

FIG. 6. The surface tensions of mixtures of 6ED and AOT in 0.04 mole/l NaCl; ○, 6ED in distilled water; □, 6ED in 2 × 10^{-4} mole/l AOT containing NaCl at 0.04 mole/l; △, 6ED in 4 × 10^{-4} mole/l AOT containing NaCl at 0.04 mole/l; ◧, 6ED in 1 × 10^{-3} mole/l at 0.04 nole/l; ▲, 6ED in 4 × 10^{-3} mole/l AOT containing NaCl at 0.04 mole/l.

surfactants (22,28). The concentrations of added 6ED were used below (2×10^{-5} mol/l) and above (3×10^{-4} and 1×10^{-3} mol/l) the CMC. The increase in the CMC of AOT in 0.04 mol/l salt solution by the addition of 3×10^{-4} mol/l or 1×10^{-3} mol/l of 6ED can be interpreted by the solubilization of AOT molecules into the 6ED micelles; the AOT molecules are solubilized into the 6ED micelles and, after solubilization saturated, the AOT micelles are formed.

In addition, the surface tension concentration curves of 6ED containing various concentrations of AOT in the presence of 0.04 mol/l NaCl are shown in Figure 6. The values of surface tension of the mixture increased as did the concentration of 6ED from the CMC of 6ED alone, indicating that the 6ED micelles would play an important role in 6ED-AOT-NaCl system.

The above results confirm that the addition of salt markedly affects the micellization of AOT and that the solubility of AOT in the presence of salt is increased by the addition of 6ED.

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