♣ Solution Properties of Mixed Surfactant System.
Sodium Dodecyl Sulfate and Alkyl Polyoxyethylene Ether System

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The effect of oxyethylene groups in a nonionic surfactant on the solution properties of anionic-nonionic systems is described; these systems are sodium dodecyl sulfate (SDS) - hexadecyl polyoxyethylene ethers ($C_{16}POE_n$, where n=10, 20, 30 and 40). The degree of ionic dissociation of the mixed micelles decreases with increasing numbers of oxyethylene groups in the nonionic surfactant. As polyoxyethylene chain lengths increase, the electrical conductivities of the mixed surfactant solutions decrease, in spite of the decrease in activation energy for conduction. The radius of the mixed micelle with the electric double layer is larger for a nonionic surfactant having a shorter polyoxyethylene chain length than for one having a long polyoxyethylene chain. This may be attributed to the fact that the mixed micelle is formed more easily by a nonionic surfactant with a shorter polyoxyethylene chain length than by one with a longer chain.

Nonionic surfactants are widely used as solubilizers and/or emulsifiers. Most commercial surfactants are polydisperse preparations containing a distribution of chain lengths for the polyoxyethylene chain. Only recently have synthetic, monodisperse substances, including a single polyoxyethylene chain length, become readily available. The polar portion of these nonionic surfactants consists of a long, uncharged polyoxyethylene chain which is much bigger than the hydrophobic group. Therefore, it is not surprising that the solution properties of these surfactants are considerably different from those of the ionic surfactants and/or of the nonionic surfactants having a short polyoxyethylene chain.

Most practical surfactant systems are mixed. Hence, understanding how surfactants interact in mixed micelles is essential for the many industrial applications of surfactants. Mixed surfactant systems are also of great theoretical interest. It can be speculated that, in solutions including mixtures of surfactants, the tendency to form aggregated structures would be substantially different than in solutions having only the pure surfactants. Many recent papers have been published on solution properties of mixed surfactant systems (1-5).

We have also reported the solution properties of anionic-nonionic surfactant mixed systems (alkyl polyoxylated carboxylate-alkyl polyoxyethylene ethers), based on electrical (6-8) and surface tension measurements (9-11), and have investigated the mixed micelle formation. We found that the formation of a mixed micelle in these systems was dependent on the molecular characteristics of the nonionic surfactant.

In this paper, sodium docedyl sulfate (SDS) is used as a typical anionic surfactant. We report the solution properties (pNa, electrical conductivity, dielectric constant and surface tension) of the mixed surfactant systems: SDS-$C_{16}POE_n$ (n=10, 20, 30 and 40). We discuss the differences in the solution properties due to the different polyoxyethylene chain length in nonionic surfactants.

EXPERIMENTAL

Materials: Anionic surfactant. Sodium dodecyl sulfate (SDS, $C_{12}H_{25}OSO_3Na$) was purchased from Tokyo Kasei Kogyo Co., Ltd., Tokyo. It is more than 99.5% pure. This material was extracted with ether and recrystallized from ethanol.

Nonionic surfactant. Alkyl poly(oxyethylene) ethers ($C_{16}POE_n$, $C_{16}H_{33}O(C_2H_4O)_nH$; n=10, 20, 30 and 40) were supplied by Nihon Surfactant Industries Co., Ltd., Tokyo. These have a narrow molecular weight distribution. Their purities were ascertained by surface tension measurements.

Water used in this experiment was twice distilled and then deionized by an ion-exchange instrument (NANO Pure D-1791 of Barnstead Co., Ltd., Boston, Massachusetts); its resistance was about 18.0 meg-ohm·cm and its pH was 6.7.

Methods; Preparation of mixed surfactant solutions. Into several 100-ml beakers, 25-ml portions of an SDS solution (2.0 × 10^{-2} mol/1) were placed, followed by addition of a $C_{16}POE_n$ solution (2.0 × 10^{-2} mol/1). All mixed surfactant solutions had a SDS: $C_{16}POE_n$ molar ratio of 1:1. The mixtures were diluted step by step with water. They were stirred for 2 hr in a thermostated bath at 30.0 C in order to establish equilibria.

Determination of pNa values. The pNa values of aqueous solutions of single and/or mixed surfactant systems were measured at 30±0.1 C using a pH meter (Model M-130, Corning Co., Ltd.) with a Na-glass electrode (A017NA, Corning Co., Ltd.). A saturated KCl-calomel electrode was used as reference.

Determination of electrical conductivity and dielectric constant. Instruments for the measurements of electrical conductivities and dielectric constant were wide-frequency bridge (TR-C type), GC box (YS-1 type), and the oscillator (WBG-11 type) of Ando Electric Co., Tokyo, Japan. The methods for measurements were the same as those described previously (12).

Determination of surface tension values. The surface tensions of aqueous solutions of the surfactant systems were measured at 30±0.1 C using a Wilhelmy type surface tensiometer (A-3, Kyowa Scientific Co., Ltd., Tokyo) with a platinum plate.

RESULTS

The pNa values. The pNa values of aqueous solutions

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of SDS alone and of SDS mixed with $C_{16}POE_n$ are plotted against the logarithm of the concentraton of SDS in Figure 1. Each curve has been displaced downwards by ΔpNa units.

In the case of SDS alone, the pNa value decreases with an increasing concentration of SDS, and a break point appears in the vicinity of the critical micelle concentration (CMC, 8.0×10^{-3} mol/1). In the case of the SDS-$C_{16}POE_n$ mixed surfactant system, the break point disappears gradually as the number of oxyethylene groups in the nonionic surfactant decreases down to n=10.

Electrical conductivities. Figure 2 shows frequency dependence of A. C. electrical conductivities in SDS-$C_{16}POE_n$ mixed surfactant solutions at 30 C.

The electrical conductivity does not change with frequency until 1 MHz. The values of electrical conductivity increase with decreasing numbers of oxyethylene groups in the nonionic surfactant. A similar tendency was recognized in results for sodium 3,6,9-trioxaicosanoate (ECL)-$C_{16}POE_n$ systems (8).

Dielectric constants. Figure 3 represents dielectric constants in SDS-$C_{16}POE_n$ mixed surfactant solutions as a function of frequency at 30 C.

The dielectric constants for all solutions exhibit remarkable dependence on frequency. The dielectric constants, ϵ', could not be measured at lower frequencies because of electrostatic polarization in the immediate neighborhood of the electrode surface, but could be measured above 220 Hz. The dielectric dispersion is found to be of the Debye-type. The process used to check for electrostatic polarization has been described previously (12-14).

In the lower frequency range (below 50 kHz), the dielectric constant increases as the number of oxyethylene groups in the nonionic surfactant decreases,
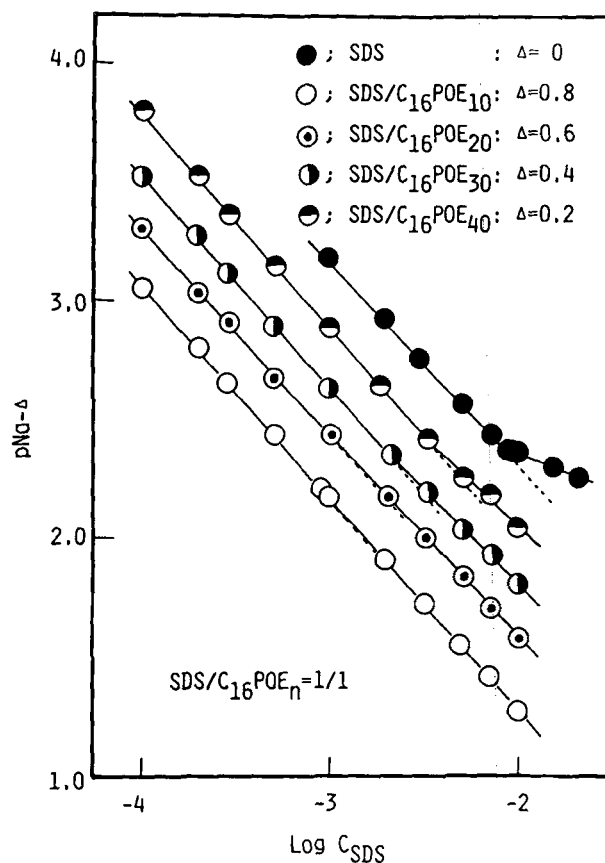

Fig. 1. Variation of pNa vs logarithm of concentration for pure SDS and for SDS-$C_{16}POE_n$ mixed surfactant systems (a molar ratio of 1/1) at 30 C. Mixed surfactant systems are indicated in the figure.
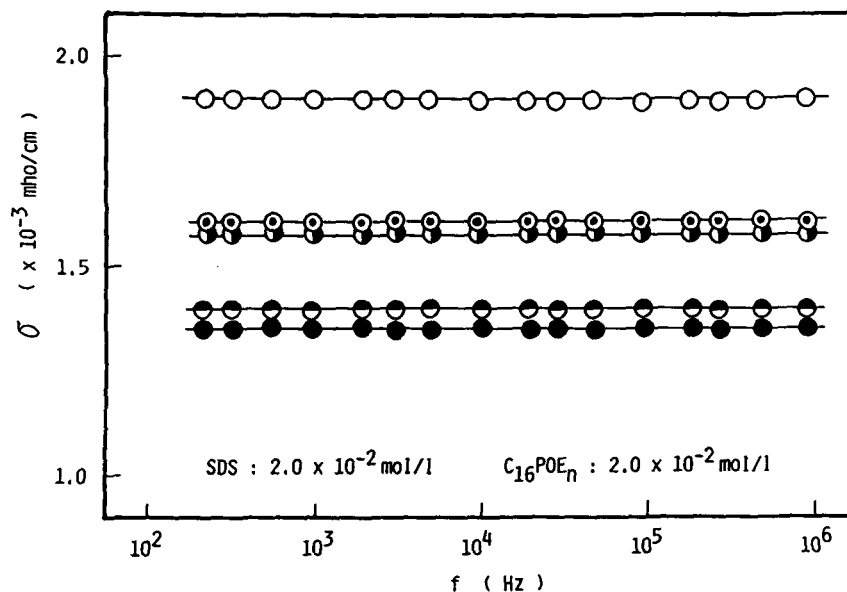

Fig. 2. Frequency dependence at AC electrical conductivities in SDS-$C_{16}POE_n$ mixed surfactant solutions (a molar ratio of 1/1 at 30 C. Total concentration is 4.0×10^{-2} mol/1. ●, SDS; ○, SDS-$C_{16}POE_{10}$; ⊙, SDS-$C_{16}POE_{20}$; ◑, SDS-$C_{16}POE_{30}$; ○, SDS-$C_{16}POE_{40}$.
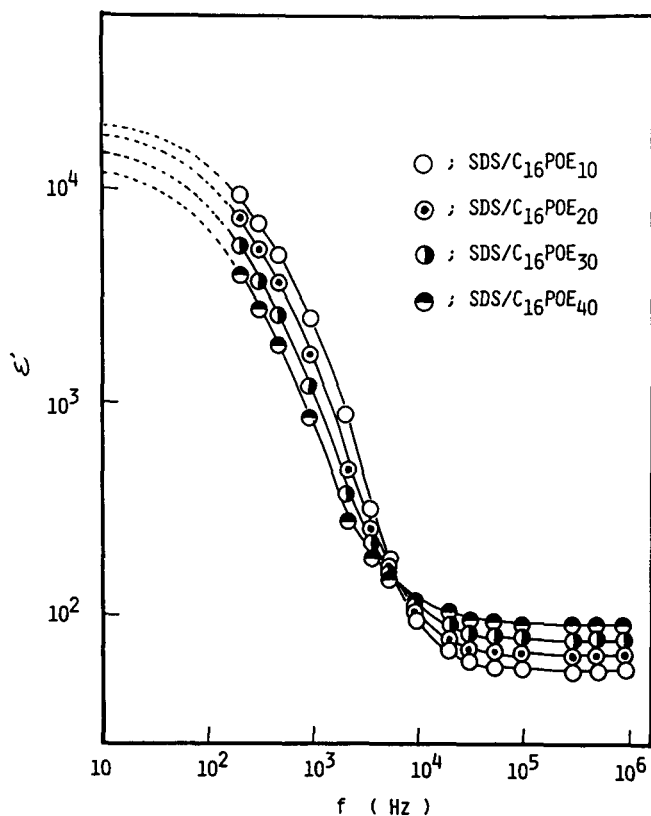
Fig. 3. Frequency dependence of dielectric constants in SDS-C$_{16}$POE$_n$ mixed surfactant solutions (a molar ratio of 1/1) at 30 C. Total concentration is 4.0 × 10^{-2} mol/1.

while in the higher frequency range (above 50 kHz) the dielectric constant decreases as the number of oxyethylene groups decreases.

Surface tension. Figure 4 depicts the change in surface tension with concentration of each pure surfactant. As can be seen from Figure 4, each surface

tension value decreases with increasing concentration, but remains constant (C$_{16}$POE$_{10}$, 35.8 dyne/cm; C$_{16}$POE$_{20}$, 41.6 dyne/cm; C$_{16}$POE$_{30}$, 44.2 dyne/cm; C$_{16}$POE$_{40}$, 48.0 dyne/cm) above the critical micelle concentration (CMC). The CMC's of nonionic surfactants are considerably lower than those of SDS (8.0 × 10^{-3} mol/1). Moreover, as polyoxyethylene chain lengths increase, the CMC's of the nonionic surfactant decrease (C$_{16}$POE$_{40}$, 7.0 × 10^{-6} mol/1; C$_{16}$POE$_{30}$, 1.2 × 10^{-5} mol/1; C$_{16}$POE$_{20}$, 1.7 × 10^{-5} mol/1; C$_{16}$POE$_{10}$, 2.5 × 10^{-5} mol/1). A similar trend is observed for alkyl polyoxylated carboxylate (15) and ethoxysulfate surfactants (16). Yanagida et al. (17,18) emphasize that as the polyoxyethylene chain length in the nonionic surfactant becomes extremely large, the spiral configurational contribution due to the TGT (trans-Gauche-trans) conformation changes the property of oxyethylene groups in it. Moreover, the effectiveness increases as the number of oxyethylene groups in the nonionic surfactant decreases.

Figure 5 demonstrates the variation of the surface tension with the total concentration in mixed surfactant solutions of SDS-C$_{16}$POE$_n$ systems. In the case of the SDS-C$_{16}$POE$_{40}$ system, the surface tension decreases with increasing concentration, even beyond a break point (first transition), in the vicinity of the CMC for C$_{16}$POE$_{40}$ alone. For the case of the SDS-C$_{16}$POE$_{30}$ and SDS-C$_{16}$POE$_{20}$ systems, when the concentration increases, the surface tension initially decreases, reaches a plateau through the first transition, breaks again at the second transition, and decreases again. On the other hand, in the case of the SDS-C$_{16}$POE$_{10}$ system, it shows a minimum just before the second transition, in the vicinity of the CMC for SDS alone.

DISCUSSION

The degree of ionic dissociation of mixed micelle. As can be seen from Figure 1, a break point appears
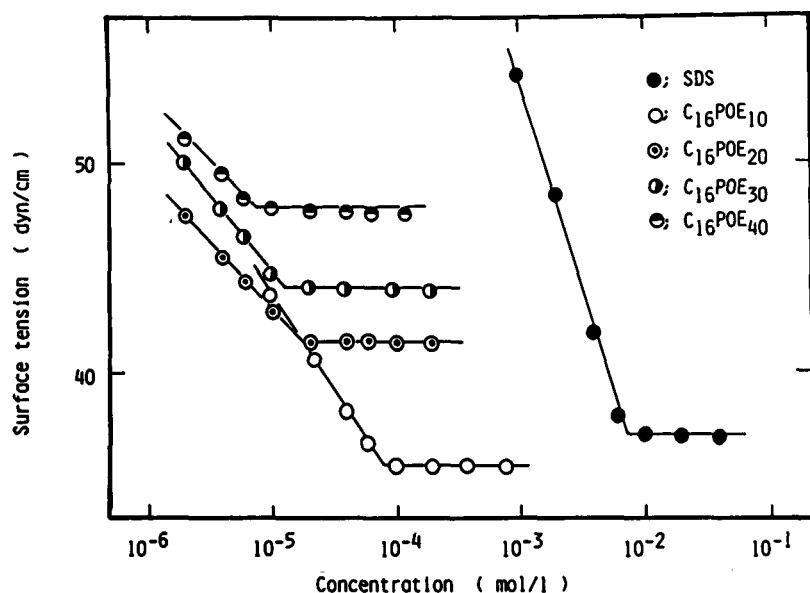

Fig. 4. Variation of surface tension vs concentration for pure surfactant at 30 C.
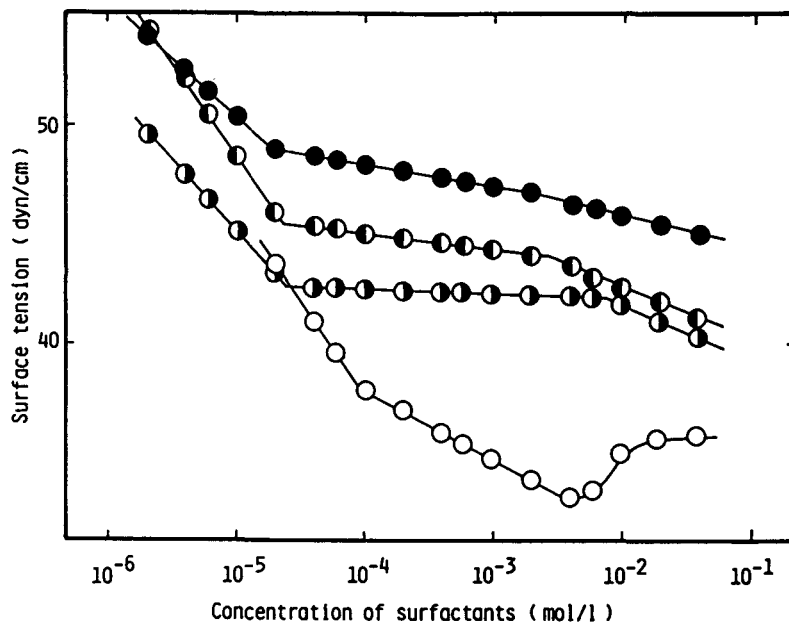
Fig. 5. Variation of surface tension vs concentration for mixed surfactant systems (a molar ratio of 1/1) at 30 C. $\bigcirc$, SDS-$C_{16}POE_{10}$; $\ominus$, SDS-$C_{16}POE_{20}$; $\ominus$, SDS-$C_{16}POE_{30}$; $\bullet$, SDS-$C_{16}POE_{40}$.

in the vicinity of the CMC with SDS alone, and it gradually disappears as the number of oxyethylene groups in the nonionic surfactant decreases. This is due to the fact that the degree of ionic dissociation of the mixed micelle increases as the number of oxyethylene groups in the nonionic surfactant decreases. We could thus determine the degree of ionic dissociation of mixed micelle by using the following method obtained by Tokiwa et al. (19).

The activity a of counter ion (Na^+) in mixed surfactant solution can be expressed by the following equation, suggested by Botre et al. (20):

$$a = \gamma \, [C_0 + \alpha \, (\, C - C_0 \,) \,] \qquad [1]$$

Here γ is the activity coefficient of the counterion (Na^+), α the degree of ionic dissociation of mixed micelle, C_0 the critical micelle concentration (CMC), and C the concentration of surfactant (SDS). With the assumption that γ is constant above the CMC and differentiating equation [1] with respect to C:

$$(\, \partial a / \, \partial c \,)_\gamma = \gamma \, [\, \alpha + (\, C\text{-}C_0 \,) \, (\, d\alpha/dc \,) \,] \qquad [2]$$

In the case of $d\alpha/dC = 0$, equation [2] becomes

$$da/dC = \gamma \cdot \alpha \qquad [3]$$

Figure 6 shows the curves of the activity of Na^+ ions vs concentration of SDS. These curves are obtained by replotting the pNa vs log concentration of SDS curves shown in Figure 1.

As can be seen in this figure, in the region of concentrations higher than 8.0×10^{-3} mol/1, the slope of the curves is almost constant. The degree of ionic dissociation of the mixed micelle (α) which was obtained by using this slope of the linear part of the a vs C curves is shown in Table 1. For the pure SDS, the value is 0.21, which is only one-half the ECL value done previously (6-8). When nonionic surfactants are added into the solution, the values increase. More-

TABLE 1

The Degree of Ionic Dissociation of Micelle (α) in $C_{16}POE_n$-SDS Mixed Surfactant Solutions[a]

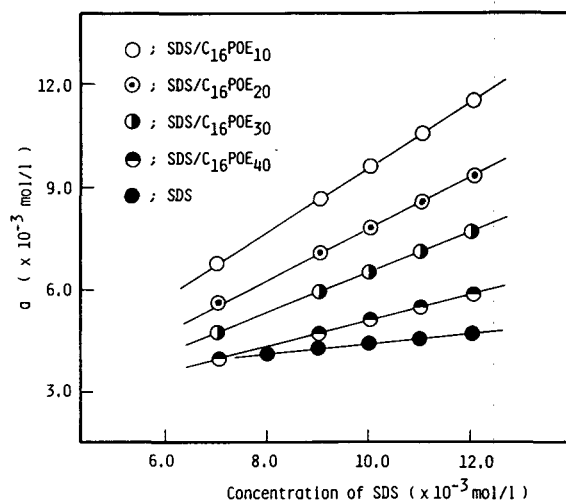| Mixed systems	α
$C_{16}POE_{10}$ - SDS	0.74
$C_{16}POE_{20}$ - SDS	0.64
$C_{16}POE_{30}$ - SDS	0.54
$C_{16}POE_{40}$ - SDS	0.49
SDS	0.21

[a]Temperature, 30 C; molar ratio, SDS/$C_{16}POE_n$ = 1/1.

Fig. 6. Change of activity with concentration for pure SDS and for SDS-$C_{16}POE_n$ mixed surfactant systems (a molar ratio of 1/1) at 30 C.

over, the values of α increase with a decrease in the number of oxyethylene groups in the nonionic surfactant. A similar trend was recognized in results for ECL-C$_{16}$POE$_n$ systems (8). The effect of nonionic surfactant on the rate of increase of α is larger for SDS mixed systems (maximum, 3.5 times) than for ECL mixed ones (maximum, 2 times).

As you can see from Figure 1, the slope of pNa vs log concentration of the SDS curve below CMC for SDS alone is coincident with that for SDS mixed with C$_{16}$POE$_n$. The slope of each curve is almost equal to that of the curve above CMC for the SDS-C$_{16}$POE$_{10}$ system. Thus, the degree of ionic dissociation of SDS monomer does not change by mixing with C$_{16}$POE$_n$ and is almost equal to 1.

Therefore, the disappearance of the break point upon decreasing the number of oxyethylene groups in Figure 1 can be attributed to an increase in the degree of ionic dissociation of mixed micelles.

The activation energy of conduction. As can be seen in Figure 2, the electrical conductivities of mixed surfactant systems at a molar ratio of C$_{16}$POE$_n$/SDS=1/1 increase when the number of oxyethylene groups decreases. The number of effective carriers for conduction seems to increase with an increasing degree of ionic dissociation of SDS in micelle.

Next, we have obtained the activation energy for conduction for the mixed surfactant systems, in order to discuss the mechanism of electrical conduction.

At a molar ratio of C$_{16}$POE$_n$/SDS=1/1 the Arrhen-

ius plots of conductivity of aqueous solutions of pure SDS and of SDS mixed with C$_{16}$POE$_n$ are shown in Figure 7.

The electrical conductivities increase with increasing temperature; these plots are linear, as suggested by Rasch and Hinrichsen (21), in conformity with the following equation:

$$\sigma = \sigma_0 \exp(-\Delta E/RT) \qquad [4]$$

where σ is the electrical conductivity, σ_0 the constant for a given surfactant, R the gas constant, T the absolute temperature and ΔE the activation energy for conduction.

Table 2 shows the activation energies of SDS alone and of SDS mixed with C$_{16}$POE$_n$; these energies have been obtained from the slope in Figure 7.

The activation energy for conduction may decrease with an increasing degree of ionic dissociation or the number of effective carriers or both. As seen in Table 2, the activation energies for conduction decrease as the number of oxyethylene groups in the nonionic surfactant increases, in spite of the increase in the degree of ionic dissociation. Those of the SDS-C$_{16}$POE$_{40}$ mixed surfactant system are equal to the values in the pure SDS system.

We reported recently that the activation energy for conduction of sodium alkyl sulfates (22) and of an anionic-nonionic mixed surfactant system (6) above the CMC was governed by the surface charge density of the counterion of micelle. That is, the activation energy for conduction begins to decreae with increasing surface charge of the counterion of the micelle.

Recently, we have also reported the activation energy for conduction of ECL-C$_{16}$POE$_n$ mixed surfactant systems (8). We found that the energy increased as the polyoxyethylene chain length in the nonionic surfactant decreased. This may be attributed to the fact that the surface charge densities of counterions of mixed micelles decrease as the size of mixed micelles becomes larger.

The surface charge density in the SDS-C$_{16}$POE$_n$ systems is thought to decrease due to the changes in the size of mixed micelles as the number of oxyethylene groups in the nonionic surfactant is increased. We have thus tried to determine the radius of the mixed micelle with the electric double layer.
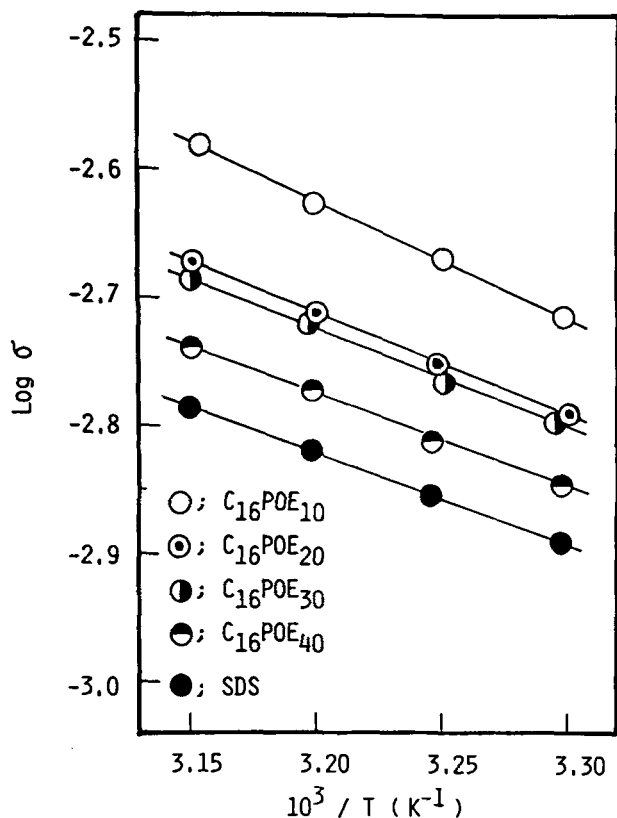

Fig. 7. Arrhenius plots of conductivity of aqueous solutions for pure SDS and for SDS-C$_{16}$POE$_n$=1/1 mixed surfactant systems. Total concentration is 4.0×10^{-2} mol/1.

TABLE 2

The Activation Energies for Conduction in C$_{16}$POE$_n$-SDS Mixed Surfactant Solutions[a]

Mixed systems	ΔE (KJ/mol)
C$_{16}$POE$_{10}$ - SDS	17.6
C$_{16}$POE$_{20}$ - SDS	14.6
C$_{16}$POE$_{30}$ - SDS	14.0
C$_{16}$POE$_{40}$ - SDS	13.8
SDS	13.2

[a]Molar ratio, SDS/C$_{16}$POE$_n$ = 1/1.

The radius of the mixed micelle with the electric double layer. Tokiwa et al. (23) reported that the mixed micelle formed with an anionic and a nonionic surfactant is a spherical micelle in which the hydrocarbon core is surrounded by polyoxyethylene chains. We shall assume that the mixed micelle formed in the SDS-$C_{16}POE_n$ mixed surfactant system is such a spherical micelle. With this assumption, it is possible to speculate that the changes in the size of the mixed micelle as well as the changes in the degree of ionic dissociation of the mixed micelle influence the surface charge density of the counterion of the mixed micelle. So, the radius of the micelle with the electric double layer formed in the aqueous solution of SDS alone and mixed with $C_{16}POE_n$ at a given concentration of SDS (2.0×10^{-2} mol/1— were obtained by using equations [5] and [6] (6-8, 12-14, 21)

$$\sigma = n \cdot e \cdot U \qquad [5]$$

$$R^2 = 2 \cdot U \cdot K \cdot T \cdot \tau/q \qquad [6]$$

Here, σ is electrical conductivity, n the number of effective carriers for conduction, e the electric charge of a single carrier, U the counterion surface mobility, K the Boltzmann constant, T the absolute temperature, τ the relaxation time, and q is 4.8×10^{-10} esu.

Because of the difference in the degree of ionic dissociation of mixed micelles formed in various mixed systems, the number of effective carriers (n) contributing to the electrical conduction is different. We determined n by using the calibration curve of the electrical conductivity and concentration of SDS, as in a previous paper (14). The relaxation time τ is calculated by the use of the Schwarz theory (24).

$$f_{max} = 1/2 \cdot \pi \cdot \tau \qquad [7]$$

Here, f_{max} is the frequency at which the dielectric absorption becomes maximum.

Table 3 represents τ, σ, U and R for aqueous solutions of SDS alone and of SDS mixed with $C_{16}POE_n$. As can be seen from Table 3, the radius of the mixed micelle with the electric double layer increases as the number of oxyethylene groups in the nonionic surfac-

tant decreases. Thus the surface charge density of counterions seems to decrease with an increase in the number of oxyethylene groups in the nonionic surfactant.

We reported that the migration of $C_{16}POE_n$ molecules from the micellar phase to the bulk phase seems to increase with an increase in the polyoxyethylene chain length (8). in the case of SDS mixed with $C_{16}POE_n$ at a molar ratio of $C_{16}POE_n/SDS=1/1$, the degree of ionic dissociation of mixed micelle and the activation energy for conduction decrease with an increase in the number of oxyethylene groups in $C_{16}POE_n$. This may be attributed to changes in the micellar sizes formed in SDS-$C_{16}POE_n$ mixed surfactant systems by the differences in hydrophilicity of $C_{16}POE_n$.

Miscibility of surfactants in the micellar state. Next, we have attempted to consider the miscibility of these surfactants in the micellar state by the surface tension values shown in Figure 5.

Rubingh (25), Rosen and Hua (26), and Motomura et al. (27) have proposed theoretical equations with respect to that mentioned above.

Rubingh (25) showed that the parameter related to the degree of molecular interaction between the two surfactants in the mixed micelle was calculated by equations analogous to equations [8] and [9].

$$X^2 \ln (C^* \, \alpha/C_1 X)/(1-X)^2 \ln [C^*(1-\alpha)/(C_1-X)] = 1 \quad [8]$$

$$\beta = \ln (C^* \alpha/C_1 X)/(1-X)_2 \qquad [9]$$

Here, X is the mole fraction of surfactant 1 in the mixed micelle, C^* the CMC of the mixed system, α the mole fraction of surfactant 1 in the total mixed solute, C_1 the CMC of pure surfactant 1, and β the interaction parameter.

Equation [8] must be solved numerically for X, whereupon substitution of this X into equation [9] results in immediate solution for β,

Rosen and Hua (26) have also mentioned that with a more negative β, the extent of synergism will be greater. So, we tried to calculate the negative number, (deviations from ideality), from CMC data shown in Figures 4 and 5. Table 4 lists some values of β.

As can be seen in Table 4, the values of β becomes more negative as the number of oxyethylene groups in the nonionic surfactant decreases. Namely, the mixed micelle is formed more easily by a nonionic surfactant including shorter polyoxyethylene chains

TABLE 3

Dielectric Parameters and the Radius of Mixed Micelles for a Given Concentration of SDS (2.0×10^{-2}M) and $C_{16}POE_n$ (2.0×10^{-2}M) in Mixed Surfactant Solutions at 30.0 C

Mixed systems	τ (sec) $\times 10^{-4}$	σ (mho/cm) $\times 10^{-3}$	U (cm^2/Vsec) $\times 10^{-4}$	R (Å)
$C_{16}POE_{10}$ - SDS	7.14	1.91	8.32	58.3
$C_{16}POE_{20}$ - SDS	6.42	1.61	8.25	55.1
$C_{16}POE_{30}$ - SDS	6.05	1.58	8.20	53.3
$C_{16}POE_{40}$ - SDS	5.79	1.41	8.28	52.4
SDS	5.62	1.35	8.09	51.0

TABLE 4

Molecular Interaction Parameters for Mixed Surfactant Systems

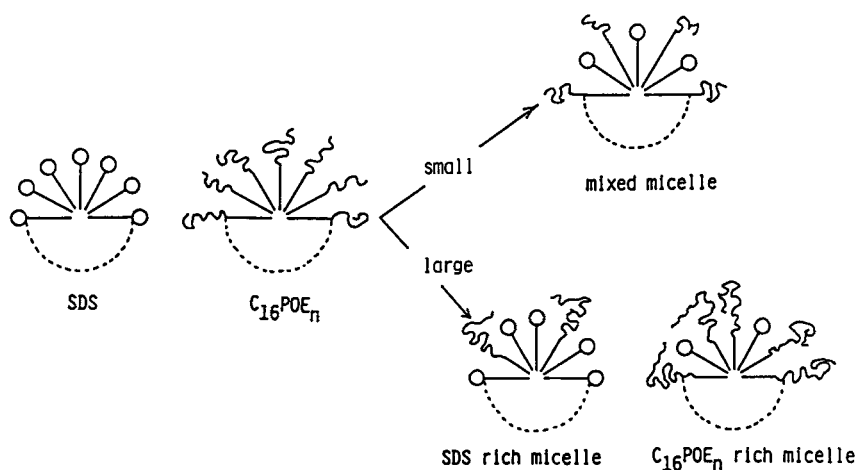| Mixed systems	$-\beta$
$C_{16}POE_{10}$ - SDS	6.58
$C_{16}POE_{20}$ - SDS	6.18
$C_{16}POE_{30}$ - SDS	4.29
$C_{16}POE_{40}$ - SDS	—

Fig. 8. Micellization models for SDS-C$_{16}$POE$_n$ mixed surfactant systems.

than by one having long polyoxyethylene chains.

Consequently, we would guess that micellization models for SDS-C$_{16}$POE$_n$ mixed surfactant system are demonstrated in Figure 8.

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