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Miscibilities of methanol and ethanol with dodecylammonium chloride in the adsorbed film and micelle

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Abstract The surface tension of the aqueous solutions of methanol–dodecylammonium chloride (DAC) and ethanol–DAC mixtures has been measured as a function of the total molality of the mixture $\hat{m}$ and the mole fraction of DAC $\hat{X}_2$ in the mixture at 298.15 K under atmospheric pressure. The compositions of the adsorbed film at 50, 40, and 30 $\mathrm{mN\,m^{-1}}$, and that of the micelle at the critical micelle concentration (CMC) have been evaluated by applying the thermodynamic equations derived previously and shown in the form of phase diagrams of adsorption and micelle formation. It has been found that (i) methanol is hardly miscible in the adsorbed film and micelle, and (ii) ethanol molecules are very slightly incorporated into adsorbed film of DAC at high-surface tension and into the micelle, although hardly miscible in the adsorbed film at low-surface tension. By comparing the corresponding phase diagrams of ethanol-2-(octylsulfinyl)ethanol (OSE) mixture, furthermore, it has been shown that there exists a difference in the miscibility of ethanol between DAC and OSE.

Key words Miscibility – adsorbed film – micelle – phase diagram – effect of alcohol

Introduction

The adsorption and micelle formation of the nonionic–ionic surfactant mixtures have been investigated extensively, because the interaction between the nonionic and ionic molecules results in the interesting behavior [1–4]. The mixtures of alcohol and ionic micelle-forming surfactant may belong to this category and have been studied by many authors [5–28] from the viewpoint of how alcohol molecules influence the critical micelle concentration of surfactant, the degree of ionization of micelle, the aggregation number, the heat of micelle formation, and so on as an additive or cosolvent. Then the results have been explained by considering the micelle solubilized alcohol and the solvent structure changed by added alcohols. Therefore, it would be very helpful to investigate the alcohol–ionic surfactant mixtures from another viewpoint that alcohol is regarded not as an additive but as one component of the mixture and then understand the effect of alcohol in terms of the miscibility of alcohol with surfactant in the adsorbed film and micelle, since a similar strategy has been applied satisfactorily to the alcohol–nonionic surfactant, 2-(octylsulfinyl)ethanol (OSE), mixtures [29–32] and elucidated that (i) ethanol molecules are excluded by OSE molecules in the adsorbed film and micelle, (ii) hexanol and octanol molecules are miscible and interact with OSE molecule attractively, and (iii) the mole fraction of alcohol in the micelle is different from that in the adsorbed film at the CMC for hexanol and octanol, whereas not appreciably for ethanol.

In the present work, we have measured the surface tension of the aqueous solution of the mixtures of methanol–dodecylammonium chloride (DAC) and ethanol– DAC, and examined the phase diagrams of adsorption and micelle formation constructed by evaluating the compositions of the adsorbed film and micelle thermodynamically.

Experimental

Dodecylammonium chloride (DAC) was synthesized and purified according to the method described previously [33]. Methanol and ethanol were purified by distilling the products (Nacalai Tesque, Inc.) after refluxing them with calcium hydride. Water used for preparing the solution was distilled triply: the second and third distillations were done from alkaline permanganate solution.

The surface tension was measured by the drop volume technique described previously [34]. Temperature was kept at 298.15 K within 0.01 K by immersing the experimental apparatus in thermostated water. The error inherent in the measurement was $\pm 0.05\,\mathrm{mN\,m^{-1}}$.

In this study, methanol or ethanol was assigned to component 1 and DAC to component 2. Since DAC molecule dissociates into ions in the aqueous solution, it is appropriate to adopt the total molality $\hat{m}$ of the mixture of alcohol and DAC defined by

$$\hat{m} = m_1 + 2m_2 \tag{1}$$

and the mole fraction of DAC $\hat{X}_2$ in the mixture defined by

$$\hat{X}_2 = 2m_2/\hat{m} \tag{2}$$

as the variables, where m_1 and m_2 are the molalities of alcohol and DAC, respectively [31, 35, 36]. The measurements of surface tension were made as a function of $\hat{m}$ at various $\hat{X}_2$.

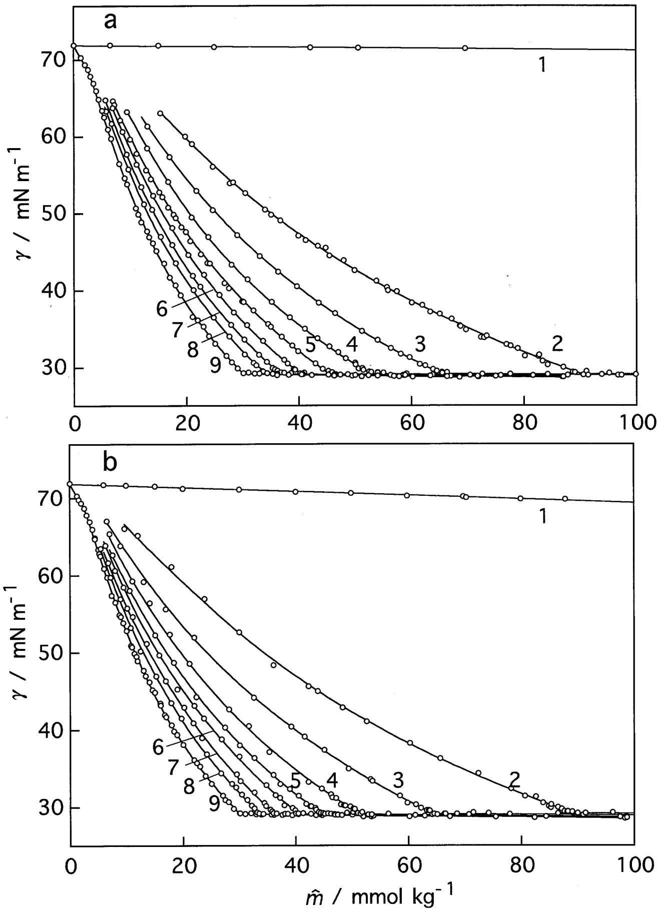


Fig. 1 (a) Surface tension vs. total molality curves of methanol–DAC system at constant composition: (1) $\hat{X}_2 = 0$; (2) 0.334; (3) 0.461; (4) 0.571; (5) 0.661; (6) 0.750; (7) 0.825; (8) 0.888; (9) 1. (b) Surface tension vs. total molality curves of ethanol–DAC system at constant composition: (1) $\hat{X}_2 = 0$; (2) 0.334; (3) 0.461; (4) 0.581; (5) 0.667; (6) 0.734; (7) 0.809; (8) 0.882; (9) 1

Results and discussion

Figure 1 shows the surface tension vs. total molality curves at constant composition of the mixture. The surface tension vs. total molality curves have a break point caused by the micelle formation. In this study, the total molality given by Eq. (1) at the break point is defined as the value of the critical micelle concentration (CMC) $\hat{C}$. The $\hat{m}$ values (open circles) at a given surface tension picked from γ vs. $\hat{m}$ curves and $\hat{C}$ values (filled circles) are plotted against $\hat{X}_2$ in Figs. 2 and 3, respectively. It can be seen that both $\hat{m}$ and $\hat{C}$ values increase greatly with increasing the mole fraction of alcohol in the mixture. Although the large increase in $\hat{C}$ value seems to be in contrast with decrease in CMC reported by the other authors for the alcohol–surfactant mixtures [8–10, 12–15, 18, 20, 24], this apparent discrepancy arises from the definition of the CMC: they defined the CMC as the concentration of the surfactant at the break point $C_2(=\frac{1}{2}\hat{C}\hat{X}_2)$. In Fig. 4, the C_2 values are plotted against the alcohol concentration $C_1(=\hat{C}(1-\hat{X}_2))$. The C_2 value is practically independent of methanol concentration and decreases very slightly with increasing ethanol concentration.

At first, let us focus our attention on the adsorbed film at the water/air interface and evaluate the total surface density difined by

$$\hat{\Gamma}^{\mathrm{H}} = \Gamma_1^{\mathrm{H}} + \Gamma_{2+}^{\mathrm{H}} + \Gamma_{2-}^{\mathrm{H}} \ , \tag{3}$$

where the subscripts 1, 2+ , and 2− represent alcohol, dodecylammonium ion, and chloride ion, respectively, and Γ_i^{H} is the excess number of moles of chemical species i per unit area defined with reference to the two dividing planes so as to make $\Gamma_{\mathrm{W}}^{\mathrm{H}}$ of water and $\Gamma_{\mathrm{A}}^{\mathrm{H}}$ of air zero [31, 36]. Here

Fig. 2 Total molality vs. composition curves at constant surface tension: (a) methanol–DAC system; (b) ethanol–DAC system: (1) $\gamma = 50 \mathrm{~mN~m^{-1}}$; (2) 40; (3) 30; (—○—) $\hat{m}$ vs. $\hat{X}_2$; (– – –) $\hat{m}$ vs. $\hat{X}_2^{\mathrm{H}}$

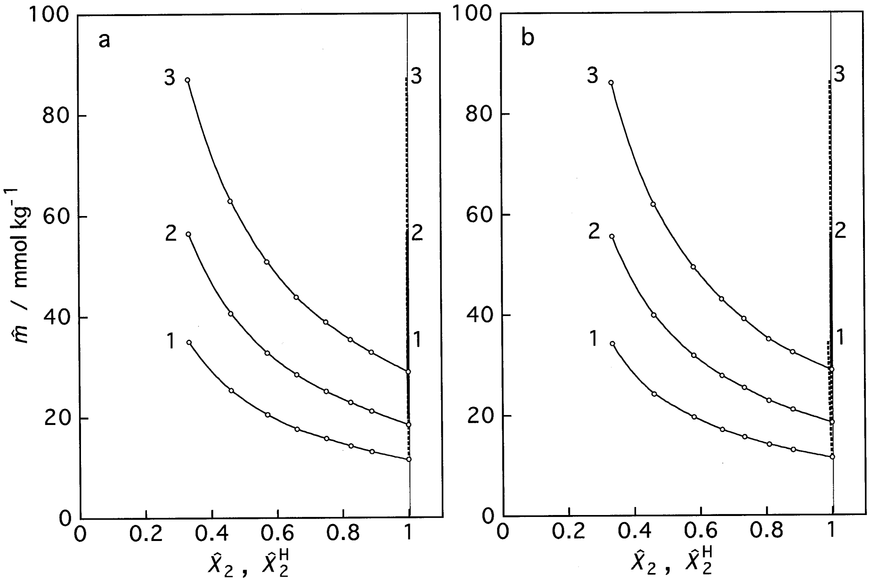
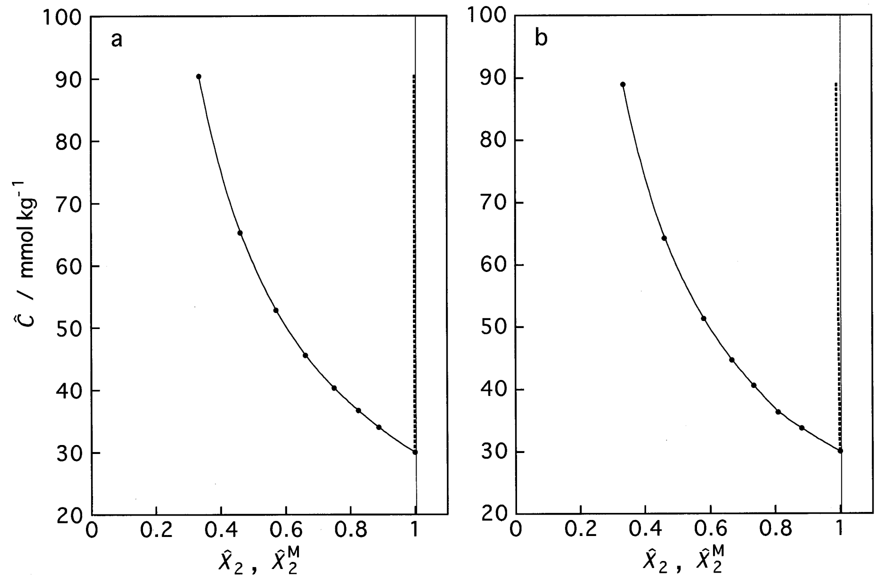


Fig. 3 Critical micelle concentration ($\hat{C}$) vs. composition curves: (a) methanol–DAC system; (b) ethanol–DAC system: (—●—) $\hat{C}$ vs. $\hat{X}_2$; (– – –) $\hat{C}$ vs. $\hat{X}_2^{\mathrm{M}}$



it should be emphasized that the adsorbed film is defined theoretically by containing the counter ion distributed in the interfacial region so as to satisfy the electrical neutral condition ($\Gamma_{2+}^{\mathrm{H}} = \Gamma_{2-}^{\mathrm{H}}$). The $\hat{\Gamma}^{\mathrm{H}}$ value is calculated by applying the equation [31, 36].

$$\hat{\Gamma}^{\mathrm{H}} = -(\hat{m}/RT)(\partial\gamma/\partial\hat{m})_{T,p,\hat{X}_2} \qquad (4)$$

to the γ vs. $\hat{m}$ curves shown in Fig. 1. The results are shown in the form of $\hat{\Gamma}^{\mathrm{H}}$ vs. $\hat{m}$ plot at constant $\hat{X}_2$ in Fig. 5. It is observed that the $\hat{\Gamma}^{\mathrm{H}}$ values near the CMC at different $\hat{X}_2$ are similar to each other for both systems. This suggests that the alcohol molecules do not influence appreciably the closest packing of molecules in the adsorbed film. Here it is worthwhile to note that also the surface density of dodecylammonium ion Γ_{2+}^{H}, which was evaluated by combining the values of $\hat{\Gamma}^{\mathrm{H}}$ with those of $\hat{X}_2^{\mathrm{H}}$ given later, does not change appreciably by adding methanol and ethanol molecules within a concentration studied here.

The next step is to examine the composition of the adsorbed film. The mole fraction of DAC in the adsorbed film is defined by the excess numbers of moles per unit area

$$\hat{X}_2^{\mathrm{H}} = (\Gamma_{2+}^{\mathrm{H}} + \Gamma_{2-}^{\mathrm{H}})/(\Gamma_1^{\mathrm{H}} + \Gamma_{2+}^{\mathrm{H}} + \Gamma_{2-}^{\mathrm{H}})\,. \qquad (5)$$

Then the mole fraction of alcohol is equal to $1 - \hat{X}_2^{\mathrm{H}}$. The $\hat{X}_2^{\mathrm{H}}$ value can be obtained by estimating the slope of the

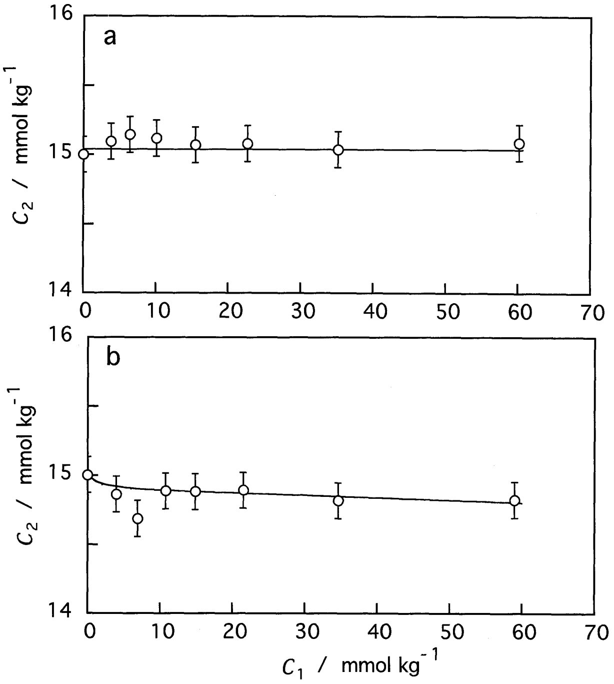


Fig. 4 Critical micelle concentration of DAC (C_2) vs. molality of alcohol (C_1) curves: (a) methanol–DAC system; (b) ethanol–DAC system

line tangent to the $\hat{m}$ vs. $\hat{X}_2$ curve in Fig. 2 and using the equation [31, 36]

$$\hat{X}_2^{\mathrm{H}} = \hat{X}_2 - (\hat{X}_1\hat{X}_2/\hat{m})(\partial\hat{m}/\partial\hat{X}_2)_{T,p,\gamma} \,. \tag{6}$$

The results are shown in the form of the $\hat{m}$ vs. $\hat{X}_2^{\mathrm{H}}$ plot in Fig. 2 (broken line); the diagram constructed by $\hat{m}$ vs. $\hat{X}_2$ and $\hat{m}$ vs. $\hat{X}_2^{\mathrm{H}}$ plots is referred to as the phase diagram of adsorption, and has been demonstrated to be greatly useful for making investigation into the miscibility and interaction of two kinds of surfactants in the adsorbed film [31, 36]. It is seen that the $\hat{X}_2^{\mathrm{H}}$ values are almost unity over a wide range of $\hat{X}_2$ and $\hat{m}$. This suggests that, despite positive adsorption of alcohols from their pure aqueous solutions, methanol and ethanol molecules are hardly miscible with DAC in the adsorbed film, and is closely related to the finding that the largest $\hat{\Gamma}^{\mathrm{H}}$ value near the CMC does not change appreciably with $\hat{X}_2$. However, looking carefully at Fig. 2(b). we note that the $\hat{X}_2^{\mathrm{H}}$ value of the ethanol system at 50 mN m^{-1} seems to decrease very slightly from unity; ethanol molecules are very slightly incorporated into adsorbed film. Therefore, it is said that there exists a small difference in miscibility of alcohols with DAC molecules between methanol and ethanol molecules at a high-surface tension and therefore at a relatively low-surface density of surfactants.

Here we show another method of analysis of experimental data to confirm the results of composition of the adsorbed film. Instead of the $\hat{m}$ and $\hat{X}_2$, let us choose m_1 and m_2 as the variables. Since the total differential of γ is represented at constant temperature T and pressure p by [31, 36]

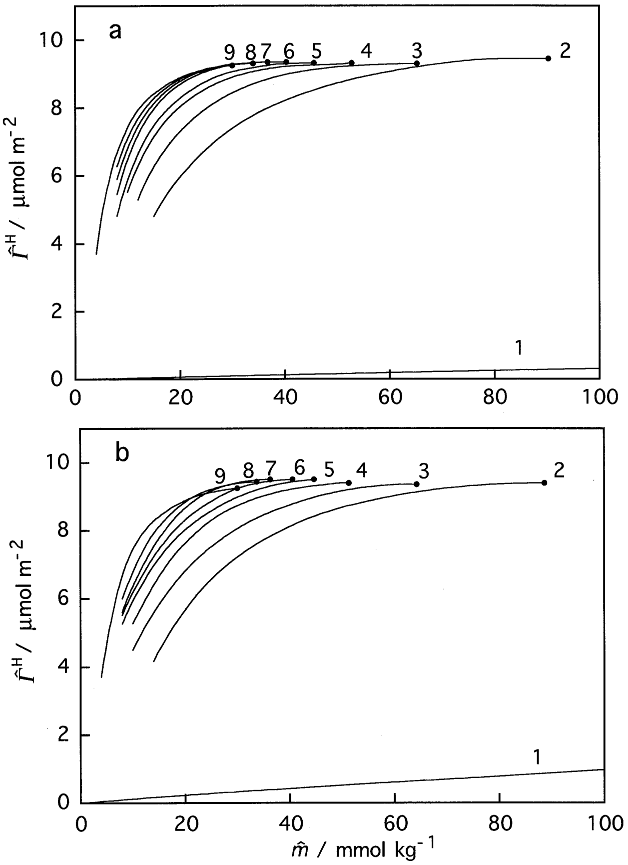


Fig. 5 (a) Total surface density vs. total molality curves of methanol–DAC system at constant composition: (1) $\hat{X}_2 = 0$; (2) 0.334; (3) 0.461; (4) 0.571; (5) 0.661; (6) 0.750; (7) 0.825; (8) 0.888; (9) 1. (b) Total surface density vs. total molality curves of ethanol–DAC system at constant composition: (1) $\hat{X}_2 = 0$; (2) 0.334; (3) 0.461; (4) 0.581; (5) 0.667; (6) 0.734; (7) 0.809; (8) 0.882; (9) 1

$$\mathrm{d}\gamma = -\Gamma_1^{\mathrm{H}}\mathrm{d}\mu_1 - \Gamma_{2+}^{\mathrm{H}}\mathrm{d}\mu_{2+} - \Gamma_{2-}^{\mathrm{H}}\mathrm{d}\mu_{2-} \,, \tag{7}$$

where μ_i is the chemical potential of chemical species i in the aqueous solution, we obtain

$$\mathrm{d}\gamma = -RT\hat{\Gamma}^{\mathrm{H}}\{(\hat{X}_1^{\mathrm{H}}/m_1)\mathrm{d}m_1 + (\hat{X}_2^{\mathrm{H}}/m_2)\mathrm{d}m_2\} \,. \tag{8}$$

Here the assumption of an ideal dilute solution of chemical species i is introduced

$$\mathrm{d}\mu_i = (RT/m_i)\mathrm{d}m_i \,. \tag{9}$$

From Eq. (8), another relation to evaluate the $\hat{X}_2^{\mathrm{H}}$ value is derived

$$\hat{X}_2^{\mathrm{H}} = 1/\{1 - (m_1/m_2)(\partial m_2/\partial m_1)_{T,p,\gamma}\} \,. \tag{10}$$

This equation shows that also the dependence of m_2 on m_1 at fixed γ reveals the miscibility of alcohol in the adsorbed film. A decrease in m_2 with increasing m_1 indicates that the alcohol molecules are miscible in the adsorbed film ($\hat{X}_2^{\mathrm{H}} < 1$; $\Gamma_{\mathrm{W}}^{\mathrm{H}} = 0, \Gamma_1^{\mathrm{H}} > 0$), whereas an increase in m_2 that the alcohol molecules are excluded from the adsorbed film compared with water molecules ($\hat{X}_2^{\mathrm{H}} > 1$; $\Gamma_{\mathrm{W}}^{\mathrm{H}} = 0, \Gamma_1^{\mathrm{H}} < 0$). Furthermore, the constant m_2 implies that the adsorbed film is formed only by surfactant ($\hat{X}_2^{\mathrm{H}} = 1$; $\Gamma_{\mathrm{W}}^{\mathrm{H}} = 0, \Gamma_1^{\mathrm{H}} = 0$) and alcohol and water molecules are distributed similarly around the adsorbed film. Thus, the m_2 vs. m_1 plot is useful to look into the miscibility of alcohol qualitatively. In Fig. 6, the m_2 values are plotted against m_1 values at 50, 40, and 30 $\mathrm{mN\,m^{-1}}$. It is seen that the m_2 values are almost independent of m_1 values ($\hat{X}_2^{\mathrm{H}} = 1$), except for the very slight decrease of m_2 at 50 $\mathrm{mN\,m^{-1}}$ of the ethanol–DAC system. Therefore, the above-conclusion drawn from the phase diagram of adsorption is confirmed.

Now, let us proceed with evaluating the composition of the micelle. The mole fraction of DAC in the micelle is defined by

$$\hat{X}_2^{\mathrm{M}} = (N_{2+}^{\mathrm{M}} + N_{2-}^{\mathrm{M}})/(N_1^{\mathrm{M}} + N_{2+}^{\mathrm{M}} + N_{2-}^{\mathrm{M}}) , \tag{11}$$

where N_i^{M} is the number of the chemical species i in a micelle particle which is defined as an excess one by supposing that the spherical dividing surface around the micelle makes the excess number of moles of water zero [31, 35, 36]. Here, the micelle, as well as the adsorbed film, is defined theoretically by containing the counter ion distributed around micelle particle so as to statisfy the electrical neutral condition ($N_{2+}^{\mathrm{M}} = N_{2-}^{\mathrm{M}}$). The $\hat{X}_2^{\mathrm{M}}$ values evaluated by applying [31, 35, 36]

$$\hat{X}_2^{\mathrm{M}} = \hat{X}_2 - (\hat{X}_1\hat{X}_2/\hat{C})(\partial\hat{C}/\partial\hat{X}_2)_{T,p} \tag{12}$$

to the $\hat{C}$ vs. $\hat{X}_2$ curves are shown in the form of $\hat{C}$ vs. $\hat{X}_2^{\mathrm{M}}$ plot in Fig. 3 (broken line). The evaluated $\hat{X}_2^{\mathrm{M}}$ values have been also confirmed by examining the C_2 vs. C_1 curves in Fig. 4 through

$$\hat{X}_2^{\mathrm{M}} = 1/\{1 - (C_1/C_2)(\partial C_2/\partial C_1)_{T,p}\} \tag{13}$$

which was derived in a similar manner to Eq. (10). The diagram of $\hat{C}$ vs. composition curves in Fig. 3 is referred to as the phase diagram of micelle formation. For the methanol–DAC system, the $\hat{X}_2^{\mathrm{M}}$ value is almost unity and suggests that the micelle, as well as the adsorbed film, is formed virtually by the DAC molecules. Although we note for the ethanol–DAC system that it decreases very slightly from unity with increasing ethanol composition, it is said that the difference in the composition of the micelle, as well as that of the adsorbed film, is small between methanol and ethanol. Thus, the effect of the increment of the one methylene group of alcohol on the miscibility is not noticeable because the compositions are close to unity and therefore the interaction between hydrocarbon chains of alcohols and DAC molecules may not be essential for the miscibilities of methanol and ethanol in the adsorbed film and micelle.

By comparing the phase diagram of adsorption with that of micelle formation, we note that there exists a little difference in the miscibility of ethanol between the micelle and the adsorbed film at a low-surface tension: the

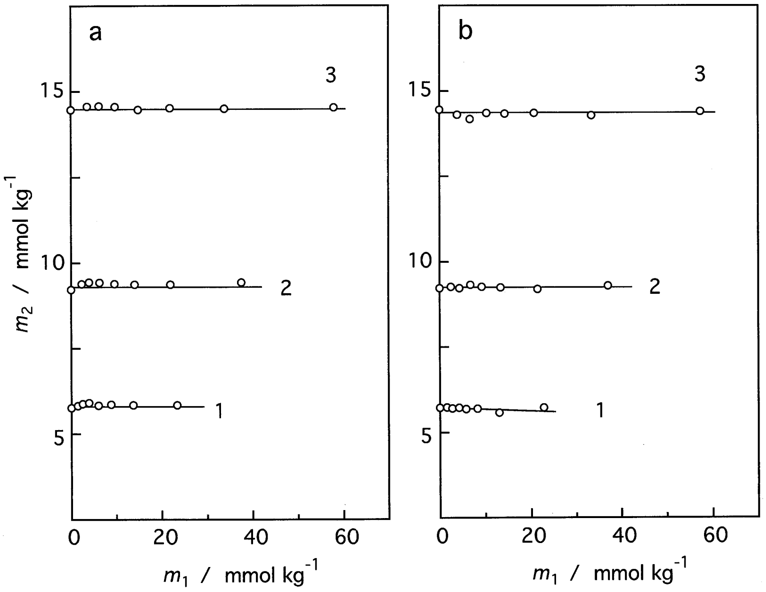


Fig. 6 Molality of DAC (m_2) vs. molality of alcohol (m_1) curves at constant surface tension: (a) methanol–DAC system; (b) ethanol–DAC system: (1) $\gamma = 50\ \mathrm{mN\,m^{-1}}$; (2) 40; (3) 30

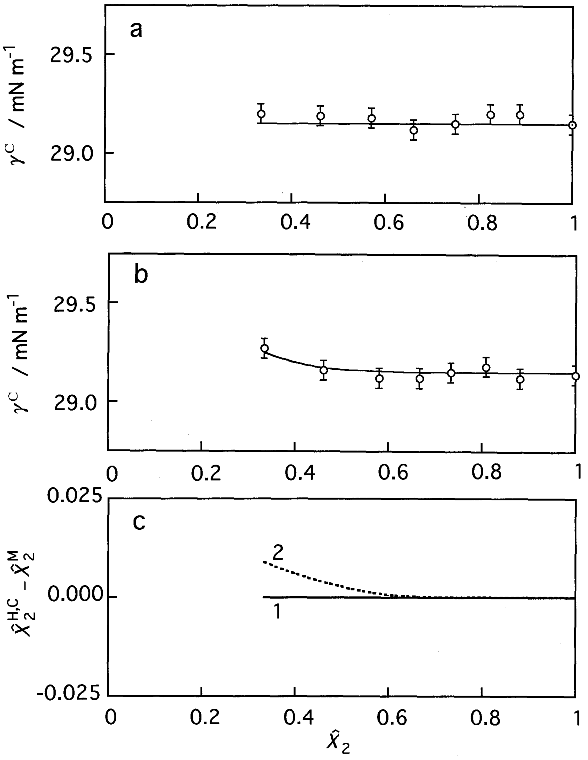


Fig. 7 (a) Surface tension at the critical micelle concentration (γ^C) vs. composition curve of methanol–DAC system. (b) Surface tension at the critical micelle concentration (γ^C) vs. composition curve of ethanol–DAC system. (c) Difference in the composition at the CMC between the adsorbed film and micelle vs. composition curves: (1) methanol–DAC system; (2) ethanol–DAC system

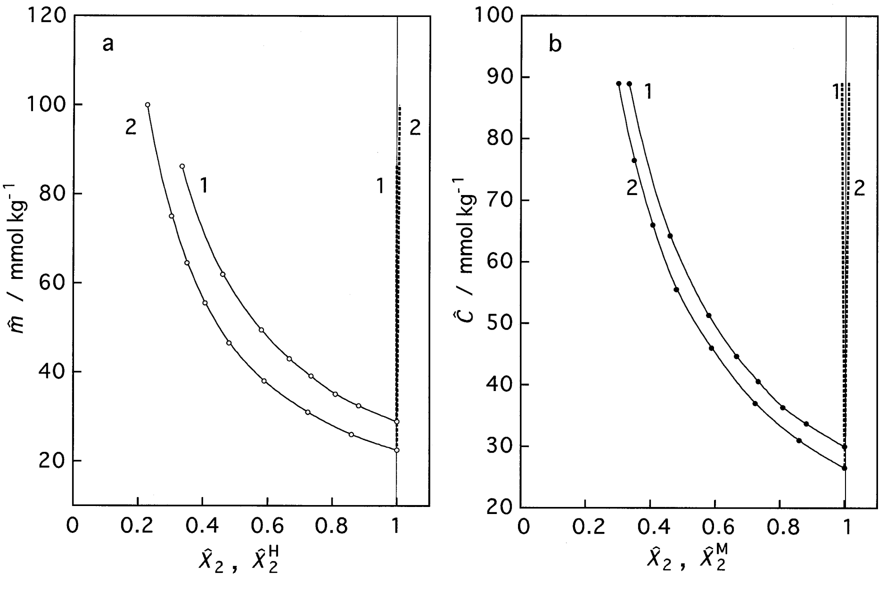
composition of micelle is $\hat{X}_2^M < 1$, whereas that of adsorbed film $\hat{X}_2^H \approx 1$. Furthermore, the difference was confirmed by calculating the compositions of the adsorbed film and micelle at the CMC from the dependence of the surface tension γ^C at the CMC on $\hat{X}_2$ by applying the following equation derived previously to the changes of γ^C with $\hat{X}_2$ shown in Figs. 7(a) and 7(b):

$$\hat{X}_2^{H,C} - \hat{X}_2^{M} = -(\hat{X}_1\hat{X}_2/RT\hat{\Gamma}^{H,C})(\partial\gamma/\partial\hat{X}_2)_{T,p}\,, \qquad (14)$$

where $\hat{X}_2^{H,C}$ and $\hat{\Gamma}^{H,C}$ is the $\hat{X}_2^H$ and $\hat{\Gamma}^H$ values at the CMC [31, 36]. The value of $\hat{X}_2^{H,C}$–$\hat{X}_2^M$ is plotted against $\hat{X}_2$ in Fig. 7(c); $\hat{X}_2^{H,C}$–$\hat{X}_2^M$ is positive at low $\hat{X}_2$ for the ethanol–DAC system. This may be caused by the difference in the geometrical structure between the adsorbed film and the micelle: the former is plane and the latter spherical. So the packing of head group may be looser in the spherical micelle than in the adsorbed film and then the ethanol molecules can penetrate slightly the micelle of DAC. The smaller value than unity at a high-surface tension, i.e., at a low-surface density, may be also attributable to the looser packing in the adsorbed film.

Finally, we compare the miscibility of ethanol in the ethanol–DAC system with that in the ethanol-2-(octylsulfinyl)ethanol (OSE) system, because OSE is one of typical nonionic surfactants and the ethanol–OSE mixture has been studied previously [30]. The phase diagrams of adsorption at 30 mN m^{-1} are compared in Fig. 8(a). It is noted that the $\hat{X}_2^H$ value of the ethanol–OSE system is larger than unity, while that of ethanol–DAC system does

Fig. 8 (a) Total molality vs. composition curves at 30 mN m^{-1}: (1) ethanol–DAC system; (2) ethanol–OSE system: (—○—) $\hat{m}$ vs. $\hat{X}_2$; (– – –) $\hat{m}$ vs. $\hat{X}_2^H$. (b) Critical micelle concentration ($\hat{C}$) vs. composition curves: (1) ethanol–DAC system; (2) ethanol–OSE system: (—●—) $\hat{C}$ vs. $\hat{X}_2$; (– – –) $\hat{C}$ vs. $\hat{X}_2^M$



not exceed unity. This suggests that ethanol molecules, compared to water molecule, are excluded by OSE molecules in the adsorbed film. In Fig. 8(b), the phase diagrams of micelle formation are further compared with each other. The composition of surfactant of the ethanol–OSE system is larger than unity, while that of the ethanol–DAC system is smaller than unity. Thus, it is said that the miscibilities of ethanol in the adsorbed film and micelle differ slightly from one another in the ethanol–DAC system, while they do not differ appreciably in the ethanol–OSE system. One probable reason may be that the electrical properties, such as charge density, of hydrophilic atmosphere of the ethanol–DAC system are different in micelle from those in adsorbed film because the packing of hydrophilic groups of DAC molecules in the micelle is looser than that of adsorbed film.

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