Phase Behavior of the Microemulsion Systems Containing Alkyl Polyglucoside and Hexadecyl-trimethyl-ammonium Bromide

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The composition and the solubilization ability of the microemulsion systems containing mixed surfactants of alkyl polyglucoside (APG) and hexadecyl-trimethyl-ammonium bromide (CTAB) were studied by using the $\varepsilon\sim\beta$ fishlike phase diagram method. Synergistic effect in terms of the composition of the interfacial layer and the solubilization ability was observed in microemulsion systems APG + CTAB + alcohol + alkane + NaCl aqueous solution. In the interfacial layer of the microemulsions the number of surfactant molecules notably decreases, resulting in the significant improvement in the solubilization ability of the system.

The effects of the carbon chain length of the alkanes and salinity on the $\varepsilon\sim\beta$ fishlike phase diagrams of APG (CTAB) + alcohol + alkane + NaCl solution microemulsion systems were also discussed.

Introduction

The nonionic surfactants alkyl polyglucosides (abbreviated as APG or C_iG_j) have peculiar properties and good biodegradability[1,2] and, therefore, are well suited to cosmetic preparations, cleaning products, food technology, and so on. It can be used as preferred substitute of the traditional surfactants such as LAS, AES, etc.[3,4] APG can be commingled with many ionic surfactants (LAS, AES, AS, CTAB, CPB, etc). This can enhance the cleaning ability, emulsification ability, the viscosity, etc. and reduce the irritation to skin. Hierrezuelo et al.[5] studied the properties of mixed surfactant systems of APG surfactant decanoyl-*N*-methylglucamide (MEGA-10) and SDS in aqueous solutions. It was found that the hydration of the mixed micelles decreases and the repulsive interaction between the headgroups of SDS increases as the charged headgroups of SDS component increase its participation in the mixed micelle. Ashok et al.[6] studied the effect of mixed surfactant system of APG and sodium lauryl sulfate (SLS) on dissolution rate enhancement of poorly water-soluble drug aceclofenac, and illustrated the advantage of mixed surfactant systems over the individual surfactants. Results obtained from physicochemical evaluation (the decrease in the cmc values and higher negative value of β-parameters) suggested the existence of synergism between surfactants blends. The synergistic behavior can be explained, by assuming strong interaction between head groups of nonionic−ionic surfactants and hydrophobic−hydrophobic attraction of two surfactants. However, the number of studies concerned with the mixture of APG with other surfactants is rather scarce.[5]

Substantial amounts of oil and water can be solubilized in microemulsions, which are composed of oil, water, surfactant, and alcohol. The solubilization ability of microemulsions can be improved by using mixed surfactants,[7−9] since they can extract properties superior than the individual ones. Addition of ionic surfactant to nonionic surfactant-based microemulsions can reduce the temperature sensitivity of the nonionic surfactant and also influence the phase behavior of the microemulsions.[10,11] In addition, the short-chain alcohols in the microemulsion systems distribute between the bulk oleic phase, aqueous phase and the interfacial layer, thereby changing the interfacial curvature and obtaining the composition of the interfacial layer. However, if the solubility of alcohols in the bulk oleic and aqueous phases is high, the solubilization ability of the microemulsion system decreases.[12]

The Winsor phase diagram method[13,14] and fishlike phase diagram method[15,16] were often used to study the phase behavior of microemulsions. In this paper, an $\varepsilon\sim\beta$ fishlike phase diagram[17,18] was chosen to investigate the phase behavior of microemulsions formed by composite surfactants of APG and hexadecyl-trimethyl-ammonium bromide (CTAB). The variations of the solubility of alcohols in the bulk oleic and aqueous phases, the composition of the interfacial layer and the solubilization ability of the microemulsion systems with the molar fraction of APG (X_{APG}) were investigated. The research will be beneficial to the comprehension of the relationship between the solubilization ability of microemulsions, the solubility of alcohols and the composition of the interfacial layer, and further the optimization of the microemulsion formations.

Experimental Section

Materials and Apparatus. The alkyl polyglucoside (APG or C_iG_j) with a decyl group as the hydrocarbon chain was synthesized[19] by our research group, and its average number of glucose units in the hydrophilic headgroup was measured by gas chromatography to be 1.46 ($C_{10}G_{1.46}$). Hexadecyl-trimethyl-ammonium bromide (CAS No. 57-09-0, CTAB with mass fraction purity >0.99), purchased from National Drug Group Chemical Reagent Company, China, was A.R. grade and crystallized twice before use. Hexane, octane, decane, dodecane and butan-1-ol (with mass fraction purity >0.99), etc., were all A.R. grade. NaCl (with mass fraction purity >0.99) was A.R. grade. Doubly distilled water was used.

An FA 1104 electron balance, a 501 super thermostat and a 811 ultra centrifuge were used in the experiment.

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10.1021/je1007444

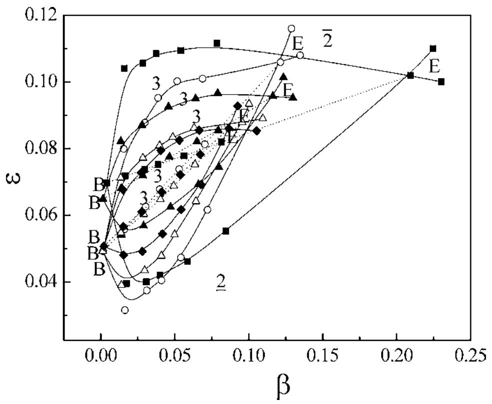

Figure 1. $\varepsilon\sim\beta$ fishlike phase diagrams of microemulsions formed by APG and CTAB mixture with different mole ratios X_{APG} of APG to CTAB, for APG(CTAB) + octane + butan-1-ol + NaCl solution ($w = 0.025$) systems. ■, 1.0 (X_{APG}); ○, 0.7 (X_{APG}); ▲, 0.5 (X_{APG}); △, 0.3 (X_{APG}); ◇, 0 (X_{APG}). ($\underline{2}$, Winsor I; $\bar{2}$, Winsor II; and 3, Winsor III microemulsion. B: beginning point; E, end point of the three-phase region. The solid line: the borderline of the "fish body"; The dashed line: the centerline of the "fish body".)

Methods. Samples were prepared by weighing equal mass of oil and aqueous solution into a series of Teflon-sealed glass tubes, and fixed amount of surfactant was also weighted into these tubes. Then different amount of butan-1-ol was added into these tubes. The above preparation process was repeated while the amount of surfactant was fixed at different values. The accuracy of all samples weighed above was ± 0.0001 g.

All the tubes were fully submerged and placed into a thermostatic water bath at (313.15 ± 0.01) K, and were left to equilibrate for about 1 week, until the phase equilibrium was approached. The volume of each phase was recorded with an accuracy of ± 0.02 mL.

Results and Discussion

$\varepsilon\sim\beta$ Fishlike Phase Diagrams of APG + CTAB + Butan-1-ol + Octane + NaCl Solution ($w = 0.025$) Systems. In APG(S_1) + CTAB(S_2) + octane (O) + butan-1-ol (A) + NaCl solution ($w = 0.025$, W) microemulsion systems, m_{s_1}, m_{s_2}, m_a, m_o, and m_w represent the mass of APG, CTAB, alcohol, oil, and aqueous solution, respectively. The symbols, α, β, and ε were defined as $\alpha = m_o/(m_w + m_o)$, $\beta = (m_{s_1} + m_{s_2})/(m_w + m_o + m_{s_1} + m_{s_2} + m_a)$, and $\varepsilon = m_a/(m_w + m_o + m_{s_1} + m_{s_2} + m_a)$, respectively.

The temperature T ((313.15 ± 0.01) K) and pressure P (normal atmospheric pressure) were held constant, and $\alpha = 0.5$ in all experiments. β was plotted horizontally and ε vertically, and then an $\varepsilon\sim\beta$ fishlike phase diagram could be obtained.

Figure 1 shows the $\varepsilon\sim\beta$ fishlike phase diagrams of microemulsions formed by APG and CTAB mixture with different mole ratios X_{APG} of APG to CTAB, for APG + CTAB+ octane + butan-1-ol + NaCl solution ($w = 0.025$) microemulsion systems.

The $\varepsilon\sim\beta$ phase diagram in Figure 1 is called fishlike phase diagram according to its shape.[20−22] The "fish head" (point B) is the beginning point of the middle-phase microemulsion and the "fish tail" (point E) is the intersection between the three- and single-phase regions. Increasing ε at constant β causes a series of phase inversions Winsor I ($\underline{2}$) → III (3) → II ($\bar{2}$).

In Figure 1, the $\varepsilon\sim\beta$ fishlike phase diagrams of the microemulsions formed by APG, CTAB, and APG−CTAB mixture, respectively, separate from each other in space. In order to evaluate the effect of the mixed surfactants on the physical−chemical properties of the microemulsion systems, some parameters related to the phase diagrams were obtained from Figure 1, according to ref 21 and listed in Table 1.

In Table 1, (β_B, ε_B) and (β_E, ε_E) are the coordinates of the "fish head" and the "fish tail", respectively.

β_i and ε_i are defined as $\beta_i = (m_{s_{1,i}} + m_{s_{2,i}})/(m_w + m_o + m_{s_1} + m_{s_2} + m_a)$ and $\varepsilon_i = m_{a_i}/(m_w + m_o + m_{s_1} + m_{s_2} + m_a)$. m_{s_1}, $m_{s_{s_2}}$, and m_{a_i} represent the mass of APG, SDS, and the alcohol in the interfacial layer. β_i and ε_i reveal the composition of the interfacial layer of the microemulsion.

The detailed discussion about (β_B, ε_B), (β_E, ε_E), and (β_i, ε_i) appears in the next section.

Physical−Chemical Properties of the Composite Surfactant Microemulsion Systems. Solubilities. β_B values in Table 1 reveals the total solubilities of surfactants APG and CTAB in the aqueous and oleic phases. It can be seen that β_B values are very small compared to β_i values. This phenomenan indicates that two surfactants are mainly incorporated into the interfacial layer, and the solubilities of the two surfactants in the aqueous or oleic phase can be ignored.

ε_B values in Table 1 shows the solubility of alcohol in the whole microemulsion system. However, ε_B and ε_i have comparable values, this elucidates that an amount of alcohol dissolves in the aqueous or oleic phase, besides their entering into the interfacial layer for the composite surfactant microemulsion systems APG (CTAB) + butan-1-ol + octane + NaCl solution ($w = 0.025$).

Composition of the Interfacial Layer. The mass fraction of alcohol in the interfacial layer which was composed of surfactant and alcohol, A^S, can be calculated from β_i and ε_i values

$$A^S = \frac{\varepsilon_i}{\beta_i + \varepsilon_i} \tag{1}$$

As 1 g of oil and 1 g of aqueous solution were just dissolved in the same microemulsion system, the total number of moles of surfactants (S_M^S, APG + CTAB), the number of moles of alcohol (A_M^S), and the molar fraction of alcohol X_A^S ($X_A^S = A_M^S/(S_M^S + A_M^S)$) in the interfacial layer can be obtained from β_i and ε_i values.

Figure 2 shows the relationship between S_M^S(A_M^S) and X_{APG} (Figure 2a), and between A^S (X_A^S) and X_{APG} (Figure 2b) of microemulsion systems APG + CTAB + butan-1-ol + octane + NaCl solution ($w = 0.025$).

Table 1. Parameters Related to the Phase Diagrams of APG (CTAB) + Butan-1-ol + Octane + NaCl Solution ($w = 0.025$) Microemulsion Systems Calculated from the $\varepsilon\sim\beta$ Fishlike Diagrams

X_{APG}	β_B	ε_B	β_E	ε_E	β_i	ε_i
0	0.002 ± 0.001	0.051 ± 0.001	0.087 ± 0.002	0.086 ± 0.002	0.085 ± 0.001	0.042 ± 0.001
0.3	0.001 ± 0.001	0.049 ± 0.001	0.096 ± 0.002	0.088 ± 0.002	0.094 ± 0.001	0.046 ± 0.001
0.5	0.002 ± 0.001	0.055 ± 0.001	0.116 ± 0.002	0.096 ± 0.002	0.115 ± 0.001	0.050 ± 0.001
0.7	0.002 ± 0.001	0.049 ± 0.001	0.122 ± 0.002	0.106 ± 0.002	0.120 ± 0.001	0.066 ± 0.001
1.0	0.004 ± 0.001	0.070 ± 0.001	0.209 ± 0.002	0.102 ± 0.002	0.206 ± 0.001	0.050 ± 0.001

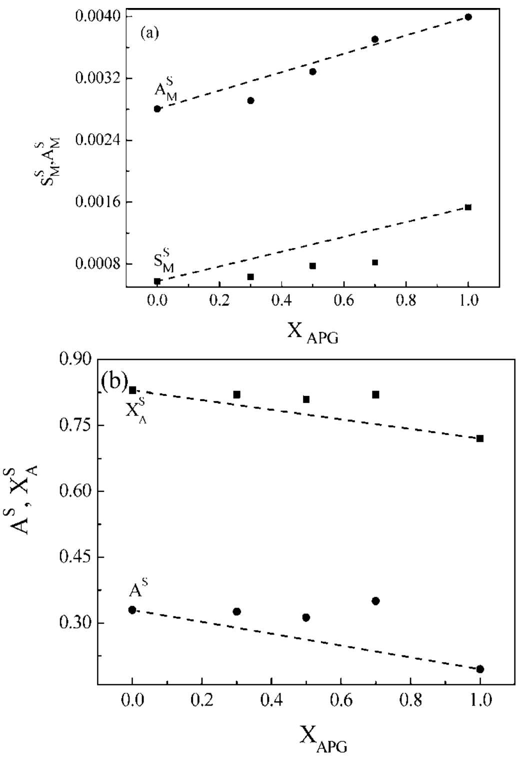

Figure 2. Plot of $S_M{}^S$ and $A_M{}^S$ vs X_{APG} (a) and A^S and $X_A{}^S$ vs X_{APG} (b) of microemulsion systems APG(CTAB) + butan-1-ol + octane + NaCl solution ($w = 0.025$).

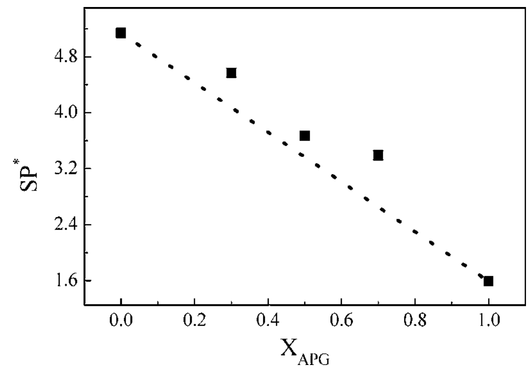

Figure 3. Plot of SP* vs X_{APG} of microemulsion systems APG(CTAB) + butan-1-ol + octane + NaCl solution ($w = 0.025$).

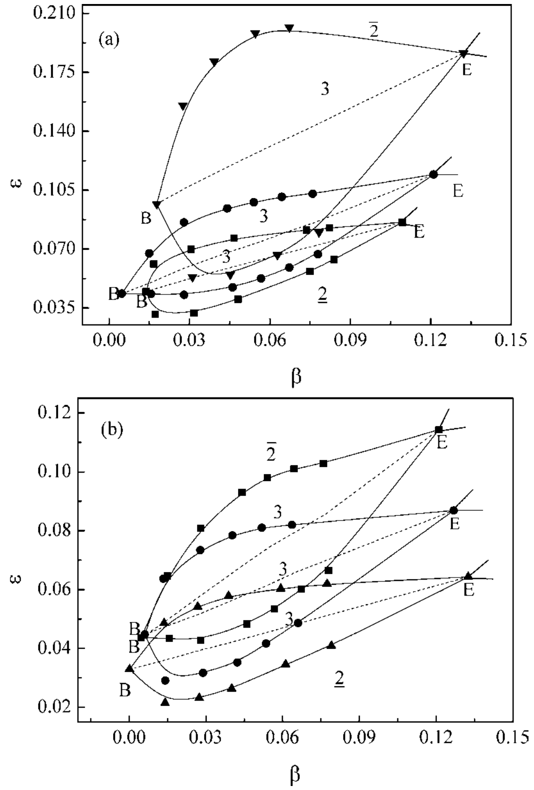

Figure 4. Effects of alkanes (a) and salinity (b) on the ε - β fishlike phase diagrams for APG (CTAB) + alkane + NaCl solution systems at (313.15 ± 0.01) K. (a) APG (CTAB) (mole ratio = 7:3) + butan-1-ol + alkane + NaCl solution ($w = 0.025$). ■, hexane; ●, octane; ▼, dodecane. (b) APG (CTAB) (mole ratio = 7:3) + butan-1-ol + octane + NaCl solution ■, 0.025 (NaCl); ●, 0.050 (NaCl); ▲, 0.100 (NaCl). ($\underline{2}$, Winsor I; $\bar{2}$, Winsor II; and 3, Winsor III microemulsion. B: beginning point; E, end point of the three-phase region. The solid line: the borderline of the "fish body"; The dashed line: the centerline of the "fish body".)

It can be seen from Figure 2a that as the molar fraction of APG (X_{APG}) increases, in comparison with the pure APG and CTAB microemulsion systems, the total number of moles of surfactants (S_M^S, APG + CTAB) decreases. This result suggests the existence of synergism between surfactant mixture on regulating the curvature of the interfacial layer.

Figure 2a also shows that the moles of alcohol (A_M^S) in the interfacial layer decrease a little compared with the moles of surfactants S_M^S in the composite surfactant microemulsion systems.

The different trends of changes of S_M^S and A_M^S with X_{APG} in Figure 2a result in the variation of the mass percentage (A^S) and the mole fraction (X_A^S) of alcohol in Figure 2b. That is, in the interfacial layer of the mixed-surfactants based microemulsion system, both values of A^S and X_A^S are large compared with that of the pure APG and CTAB microemulsion systems.

In summary, due to the fact that the surfactant predominantly enters the interfacial layer for the mixed-surfactants based microemulsion systems APG + CTAB + butan-1-ol + octane + NaCl solution ($w = 0.025$), the reduction in the number of the surfactant molecules resulted in an improvement of the solibilization ability of the system(see next section).

Solubilization Ability. At point E in Figure 1, the three- and single-phase regions intersect. This point reflects the minimum quantity of the surfactant needed to solubilize one gram of oil and one gram of aqueous solution into the microemulsion phase. That is, point E reveals the solubilization ability of the systems. Through β_E and ε_E values, the solubilization parameter of the microemulsion phase SP*,[23] the mass of oil solubilized in the microemulsion phase per gram of surfactant, can be calculated

$$SP^* = \frac{1 - \beta_E - \varepsilon_E}{2\beta_E} \qquad (2)$$

SP* values represent the solubilization ability of the system.

Figure 3 shows the relationship between SP* and X_{APG} of microemulsion systems APG (CTAB) + butan-1-ol + octane + NaCl solution ($w = 0.025$).

Figure 3 indicates that the solubilization ability SP* of the microemulsion system decreases with the increased molar ratio X_{APG} of APG to CTAB. However, SP* values do not decrease linearly, but larger than the linear relation predicts with the increase of X_{APG} values. The result in Figure 3 suggests the

Table 2. Effects of Alkanes and Salinity on the Physical−Chemical Parameters for APG (CTAB) (Molar Ratio = 7:3) + Alcohol + Alkane + NaCl Solution Systems at (313.15 ± 0.01) K

	β_B	ε_B	β_E	ε_E	β_i	ε_i	A^S	SP*
APG (CTAB) (= 7:3) + Butan-1-ol + Alkane + NaCl Solution (w = 0.025)								
hexane	0.014 ± 0.001	0.045 ± 0.001	0.109 ± 0.002	0.086 ± 0.002	0.098 ± 0.001	0.048 ± 0.001	0.328 ± 0.002	3.68 ± 0.02
octane	0.005 ± 0.001	0.044 ± 0.001	0.121 ± 0.002	0.114 ± 0.002	0.117 ± 0.001	0.079 ± 0.001	0.404 ± 0.002	3.16 ± 0.02
dodecane	0.018 ± 0.001	0.097 ± 0.001	0.132 ± 0.002	0.186 ± 0.002	0.119 ± 0.001	0.112 ± 0.001	0.486 ± 0.002	2.57 ± 0.02
APG (CTAB) (= 7:3) + Butan-1-ol + Octane + NaCl Solution								
0.025 NaCl	0.005 ± 0.001	0.044 ± 0.001	0.121 ± 0.002	0.114 ± 0.002	0.117 ± 0.001	0.079 ± 0.001	0.404 ± 0.002	3.16 ± 0.02
0.050 NaCl	0.006 ± 0.001	0.045 ± 0.001	0.127 ± 0.002	0.087 ± 0.002	0.122 ± 0.001	0.050 ± 0.001	0.290 ± 0.002	3.10 ± 0.02
0.100 NaCl	0.001 ± 0.001	0.033 ± 0.001	0.133 ± 0.002	0.064 ± 0.002	0.132 ± 0.001	0.037 ± 0.001	0.210 ± 0.002	3.03 ± 0.02

existence of synergism in the solubilization of the microemulsion systems between the two surfactants.

Effects of Alkanes and Salinity on the $\varepsilon\sim\beta$ Fishlike Phase Diagrams. The effects of the carbon chain length of the alkanes and the salinity of the aqueous phase on the $\varepsilon\sim\beta$ fishlike phase diagrams of APG (CTAB) (molar ratio = 7:3) + alkane + NaCl solution systems were shown in Figure 4, the related physical-chemical parameters were listed in Table 2.

Figure 4 shows that different alkanes and salinity have notable effects on the $\varepsilon\sim\beta$ fishlike phase diagrams. The "fish" body moves upward as the increase in the carbon chain length of the alkane molecules or decrease in NaCl contents. Accordingly the parameters of the phase diagrams were calculated and listed in Table 2.

Table 2 shows that ε_i and β_i values increase with the increase in the carbon chain length of the alkane molecules, and hence, SP* decreases.

The long chain alkane molecules were not easy to penetrate the interfacial layer to increase the lipophilicity of the interfacial layer.[24] Therefore, more surfactant and alcohol molecules enter into the interfacial layer, and hence the solubilization ability of the microemulsion (SP*) decreases.

The increase in the NaCl content has notable influence on the $\varepsilon\sim\beta$ phase diagram owing to the "salting-out effect".[25] The fact that the values of ε_i and A^S notablely decrease with NaCl contents (Table 2) demonstrates that the number of alcohol molecules that insert into the interfacial layer decreases. However the solubilization ability (SP*) of the microemulsion system remains relatively unchanged.

Conclusions

The composition and the solubilization of the composite surfactant microemulsion systems containing nonionic surfactant alkyl polyglucoside (APG) and cationic surfactant hexadecyltrimethyl-ammonium bromide (CTAB) were studied with an $\varepsilon\sim\beta$ fishlike phase diagram.

APG and CTAB molecules are mainly incorporated into the interfacial layer, while alcohol molecules dissolve in the aqueous or oleic phase, besides their entering into the interfacial layer in microemulsion systems APG (CTAB) + alcohol + alkane + NaCl solution.

There is an observable decrease in the total number of APG and CTAB molecules in the interfacial layer of the APG and CTAB based microemulsion systems, resulting in an improvement of the solubilization ability of the system. APG and CTAB have shown significant synergistic effect on the solubilization ability SP^* of the microemulsion system.

As the carbon chain length of the alkane molecules decreases, the solubilization ability (SP*) of the microemulsion systems increaseses. The increase in the NaCl content notably deacreases the solubility of the alcohol, but the solubilization ability of the microemulsion system changes little.

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Received for review July 15, 2010. Accepted November 29, 2010. The authors are thankful for the financial support of the Natural Science Foundation of Shandong Province of China (Grant ZR2009BM036).

JE1007444