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Effect of Alkane/Water Ratios on the Phase Behavior and the Solubilization of Microemulsion Systems Containing Hexadecyltrimethylammonium Bromide

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Supporting Information

ABSTRACT: Phase behaviors and related physicochemical properties of the microemulsion systems containing hexadecyltrimethylammonium bromide + alcohol + alkane + brine were studied at various alkane/water volume ratios. The alcohol solubility (ε_B) increases proportionally to the alkane/water ratio. Concurrently, the alcohol mass fraction in the interfacial layer (A^S) increases, and the solubilization ability (SP*) of the system decreases. When the length of the carbon chain of the alkane molecules increases, the solubility of alcohol in the water and oil phases and the mass fraction of alcohol in the interfacial layer increase, while the solubilization ability of the microemulsion systems decreases.

INTRODUCTION

Microemulsions are thermodynamically stable multicomponent dispersions consisting of water, alkane, surfactant, and cosurfactant. Because of their special properties, for example, ultra low interfacial tension,[1−3] microemulsions are widely used in various fields, such as separation and concentration, polymerization reactions,[4] organic and biorganic synthesis,[5] electrochemical processes and the synthesis of nanoparticles,[6,7] and so forth. Cationic surfactant hexadecyltrimethylammonium bromide (CTAB) based microemulsions can be used as hosts for enzymes and microreactors for polymerization and preparation of nanosize particles.[8,9]

The influencing factors of the phase behavior of microemulsion systems, such as alcohol, alkane, salinity, and temperature have been widely studied.[10−18] In this paper, the effect of alkane/water volume ratio α (defined as the volume fraction of alkane in alkane and water mixture) on the phase behavior of microemulsion system hexadecyltrimethylammonium bromide/alcohol/alkane/brine was studied. This will be of great significance both in the further understanding of the phase behavior and the practical use of the microemulsion systems.

Much work has been done by researchers on the effect of alkane/water ratio on the phase behavior of microemulsion system. Tien et al.[19] examined the influence of alkane volume fraction θ_o in alkane and water mixture on the phase inversion of Winsor type microemulsion systems[20] containing sodium dodecylbenzenesulfonate (SDBS) and pentan-1-ol and found that phase inversion occurs Winsor II→Winsor III→Winsor I with increasing θ_o values. Kartsev et al.[21] measured the properties of density, isothermal compressibility, and so forth of microemulsion systems water/octane/sodium dodecyl sulfate (SDS)/pentan-1-ol in a wide range of alkane/water ratios and found that the alkane/water ratio has a notable effect on these properties. Paul and Mitra[22] studied the solubilization ability of heptane/water in polyoxyethylene 10 cetyl ether (trademarked name: Brij-56)/butan-1-ol/heptane/water systems at two different alkane/water mass ratio α values (0.25 and 0.75) and found that the solubilization ability of the microemulsion phase is reduced significantly at a higher mass fraction of the alkane (α = 0.75). The monomeric solubility of the surfactant in alkane and water at various α values was studied[23] for the simple system H_2O/dodecane/*n*-butyl monoglycol ether (C_4E_1), and it was concluded that the alkane/water mass ratio might be the key to describing the main features of these microemulsion systems. The fish-shaped region in the phase diagram of the ternary system water/1-butyl-3-methylimidazolium hexafluorophosphate (ionic liquid)/polyoxyethylene octyl phenyl ether (Triton X-100) was also influenced by ionic liquid/water ratios.[24]

Overall, the effects of alkane/water volume (or mass) ratios in microemulsion systems on the phase inversion, the microemulsion properties, the solubilization ability, the monomeric solubility of the surfactant, and the fish-shaped region in the fish-like phase diagram have been researched. Nevertheless, a systematic and quantitative description of microemulsion phase behavior at various α values is necessary to the application of microemulsion systems in industry fields.

EXPERIMENTAL SECTION

Materials and Apparatus. Hexadecyltrimethylammonium bromide (CAS No. 57-09-0, CTAB with mass fraction purity >0.99), purchased from National Drug Group Chemical Reagent Company, China, was analytical reagent (AR) grade and crystallized twice before use. Hexane, octane, decane, and butan-1-ol (with mass fraction purity >0.99) were all AR grade. Hexane, octane, and butan-1-ol (analytically pure) were purchased from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China and without further purification. Decane (with mass fraction purity >0.99) was purchased from Alfa Aesar Johnson Matthey, USA

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and used without further purification. NaCl (with mass fraction purity >0.99) was AR grade. Doubly distilled water was used.

An analytical balance (accurate to 0.0001 g, model FA 1104, Shanghai, China), an electrically heated thermostatic bath (accurate to 0.1 K, model CS501, Jiangsu, China), and an 811 ultra centrifuge were used in this experiment.

Methods. The samples were prepared by weighing NaCl solution ($w = 0.025$, density $\rho_W = 1.025\ \text{g}\cdot\text{mL}^{-1}$ at 303 K) and alkane (densities $\rho_O = 0.650, 0.702$, and $0.730\ \text{g}\cdot\text{mL}^{-1}$ at 303.15 K, for hexane, octane, and decane, respectively[25]) with different fixed α values [$\alpha = v_O/(v_O + v_W) = m_O\rho_W/(m_O\rho_W + m_W\rho_O)$, v_O and v_W are the volumes of alkane and water, m_O and m_W are the masses of alkane and water in the system, respectively] into a series of Teflon-sealed glass tubes. Then different masses of surfactant were added into the tubes, and at last alcohol was added into the tubes with the help of a microsyringe. The surfactant masses were fixed at respective values while the alcohol varied monotonically. The accuracy of all samples weighed above was ± 0.0001 g.

All of the Teflon-sealed glass tubes were shaken vigorously and ultracentrifuged and then placed in a thermostatic bath (303.15 ± 0.1 K) for about one week. The volume of each phase was continuously monitored by visual observation[26] until an equilibrium volume was reached and recorded with an accuracy of ± 0.05 mL.

If the mass fraction of alcohol in the system ε, $\varepsilon = m_A/(m_S + m_A + m_O + m_W)$, was used as the vertical coordinate, and the mass fraction of surfactant in the system β, $\beta = m_S/(m_S + m_A + m_O + m_W)$, was used as the horizontal coordinate (m_A, m_S, m_O, and m_W are the masses of alcohol, surfactant, oil, and water in the whole system, respectively), an ε−β fishlike phase diagram can be plotted.

RESULTS AND DISCUSSION

ε−β Fishlike Phase Diagrams at Different α Values. The ε−β fishlike phase diagrams for the microemulsion systems CTAB + butan-1-ol + alkane + NaCl solution ($w = 0.025$) at different α values were obtained and shown in Figure 1.

Figure 1 shows the vertical sections through the phase prism at different fixed α values.[27] The shape of the phase diagram obtained from such a section is that of a "fish". The hydrophile−lipophile property of the microemulsion system is just balanced on the lines[28] joining points B and E in Figure 1. The microemulsion systems on these lines solubilize equal amounts of water and oil and are called the optimum microemulsion systems. Several kinds of fishlike phase diagrams have been reported.[15,26,29] These phase diagrams can use temperature as a variable[26] and can also use the mass fraction of alcohol in the surfactant and alcohol mixture, δ, or the mass fraction of alcohol in the system ε (in this paper) as a variable.[15,29] In these phase diagrams, both the increase in temperature and the addition of alcohol can facilitate the phase inversion of microemulsion systems.

In Figure 1, the middle-phase microemulsion forms at point *B* ("fish head", β_B, ε_B) and disappears at point *E* ("fish tail", β_E, ε_E). Increasing ε at constant β causes a series of phase inversions Winsor I ($\underline{2}$) → III (3) → II ($\overline{2}$).

According to literature,[28,30] some parameters of the ε−β fishlike phase diagrams were calculated from Figure 1 and listed in Table 1. β_B and ε_B values can represent the average solubilities of surfactant and alcohol in the water and oil phases equilibrated with the microemulsion phase, respectively. β_i and ε_i, the mass fractions of surfactant and alcohol contained in the interfacial layer, respectively, are defined as

$$\beta_i = \frac{m_S^i}{m_W + m_O + m_S + m_A},\ \varepsilon_i = \frac{m_A^i}{m_W + m_O + m_S + m_A} \qquad (1)$$

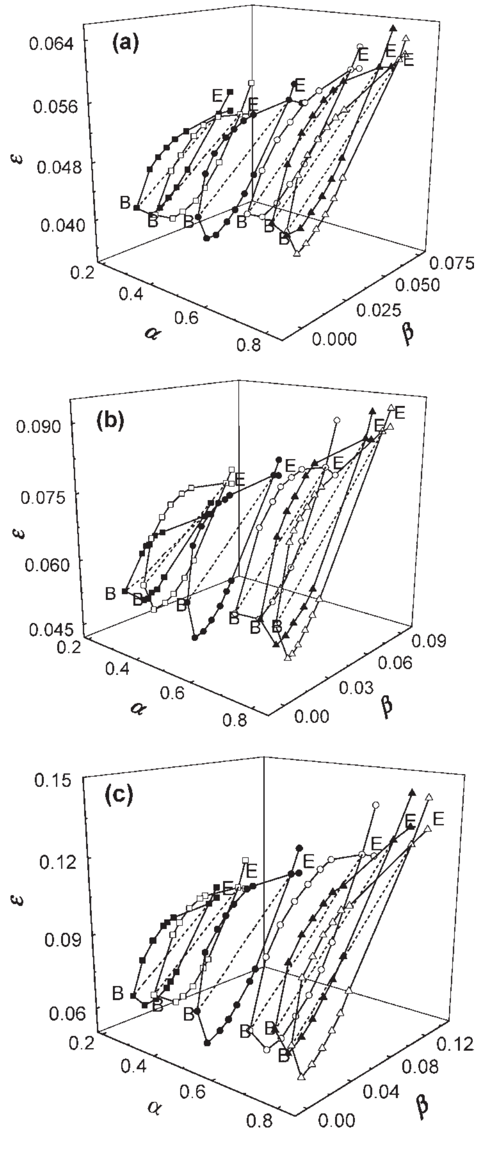

Figure 1. ε−β fishlike phase diagrams for microemulsion systems CTAB + butan-1-ol + alkane + NaCl solution at different α values ($w = 0.025$). (a) hexane, (b) octane, (c) decane. α: ■, 0.25; □, 0.33; ●, 0.50; ○, 0.67; ▲, 0.75; △, 0.80.

m_A, m_S, m_O, and m_W are the mass of alcohol, surfactant, alkane, and water in the whole system, respectively. m_S^i and m_A^i represent the mass of CTAB and the alcohol in the interfacial layer. β_i and ε_i reveal the composition of the balanced interfacial layer, and the mass fraction of the alcohol molecules in the interfacial layer, which is composed of the surfactant and alcohol mixture, A^S can be calculated from β_i and ε_i values. In addition, β_E and ε_E describe the minimum concentrations of the surfactant and alcohol, respectively, needed to establish a macroscopically homogeneous phase at a given α value.

Properties of the ε−β Fishlike Phase Diagrams at Different α Values. *Solubility of the Alcohol (ε_B).* β_B values in Table 1 are very small compared to β_i values. This suggests that CTAB is mainly incorporated into the interfacial layer and a smaller amount of CTAB is solubilized into the alkane or water phase. However, the large ε_B values in Table 1 indicate that the alcohol is readily soluble in the water or alkane phase apart from their entering into the interfacial layer. The variation of the solubility

Table 1. Physical-Chemical Parameters of β_B, ε_B, β_E, ε_E, β_i, and ε_i for Microemulsion Systems CTAB + Butan-1-ol + Alkane + NaCl Solution (w = 0.025) at Different α Values

α	β_B	ε_B	β_E	ε_E	β_i	ε_i
			CTAB + Butan-1-ol + Hexane + NaCl Solution (w = 0.025)			
0.25	0.002 ± 0.001	0.041 ± 0.001	0.048 ± 0.002	0.051 ± 0.002	0.046 ± 0.001	0.012 ± 0.001
0.33	0.002 ± 0.001	0.041 ± 0.001	0.049 ± 0.002	0.052 ± 0.002	0.047 ± 0.001	0.013 ± 0.001
0.50	0.001 ± 0.001	0.043 ± 0.001	0.052 ± 0.002	0.055 ± 0.002	0.050 ± 0.001	0.015 ± 0.001
0.67	0.001 ± 0.001	0.046 ± 0.001	0.059 ± 0.002	0.060 ± 0.002	0.058 ± 0.001	0.018 ± 0.001
0.75	0.001 ± 0.001	0.046 ± 0.001	0.062 ± 0.002	0.060 ± 0.002	0.061 ± 0.001	0.019 ± 0.001
0.80	0.001 ± 0.001	0.046 ± 0.001	0.064 ± 0.002	0.062 ± 0.002	0.064 ± 0.001	0.020 ± 0.001
			CTAB + Butan-1-ol + Octane + NaCl Solution (w = 0.025)			
0.25	0.006 ± 0.001	0.051 ± 0.001	0.060 ± 0.002	0.065 ± 0.002	0.054 ± 0.001	0.019 ± 0.001
0.33	0.006 ± 0.001	0.055 ± 0.001	0.062 ± 0.002	0.073 ± 0.002	0.056 ± 0.001	0.022 ± 0.001
0.50	0.006 ± 0.001	0.055 ± 0.001	0.064 ± 0.002	0.077 ± 0.002	0.059 ± 0.001	0.027 ± 0.001
0.67	0.005 ± 0.001	0.056 ± 0.001	0.066 ± 0.002	0.080 ± 0.002	0.062 ± 0.001	0.030 ± 0.001
0.75	0.005 ± 0.001	0.056 ± 0.001	0.077 ± 0.002	0.087 ± 0.002	0.073 ± 0.001	0.036 ± 0.001
0.80	0.004 ± 0.001	0.056 ± 0.001	0.077 ± 0.002	0.089 ± 0.002	0.074 ± 0.001	0.038 ± 0.001
			CTAB + Butan-1-ol + Decane + NaCl Solution (w = 0.025)			
0.25	0.006 ± 0.001	0.065 ± 0.001	0.074 ± 0.002	0.093 ± 0.002	0.069 ± 0.001	0.035 ± 0.001
0.33	0.006 ± 0.001	0.068 ± 0.001	0.083 ± 0.002	0.101 ± 0.002	0.077 ± 0.001	0.041 ± 0.001
0.50	0.003 ± 0.001	0.069 ± 0.001	0.088 ± 0.002	0.111 ± 0.002	0.085 ± 0.001	0.050 ± 0.001
0.67	0.003 ± 0.001	0.070 ± 0.001	0.107 ± 0.002	0.120 ± 0.002	0.104 ± 0.001	0.062 ± 0.001
0.75	0.002 ± 0.001	0.072 ± 0.001	0.111 ± 0.002	0.127 ± 0.002	0.109 ± 0.001	0.066 ± 0.001
0.80	0.001 ± 0.001	0.070 ± 0.001	0.112 ± 0.002	0.126 ± 0.002	0.111 ± 0.001	0.069 ± 0.001

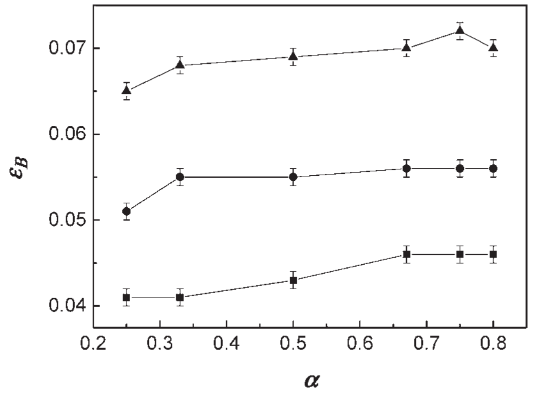

Figure 2. Effect of α values on the solubility of alcohol (ε_B) in the water and oil phases for systems CTAB + butan-1-ol + alkane + NaCl solution (w = 0.025). ■, hexane; ●, octane; ▲, decane.

of the alcohol in water and oil phases with α values for the microemulsion systems of CTAB + butan-1-ol + alkane + NaCl solution (w = 0.025) was shown in Figure 2.

Figure 2 shows a positive variation of the solubility of alcohol in the water and oil phases and α values. Alcohol is solubilized in both alkane and water phases. However, as a hydrophobic cosolvent, alcohol partitions mainly into the alkane phase. Therefore, as α values increase at the fixed total amount of alkane and water, more alcohol was solubilized in the alkane phase.[12] As a result, the solubility (ε_B) of the alcohol in the water and oil phases would increase as α values increase.[31] In short, the solubility of alcohol in alkane phase is higher than that in water phase, which results in the increase in the average solubility (ε_B) of alcohol in the water and oil phases, as the α values increase.

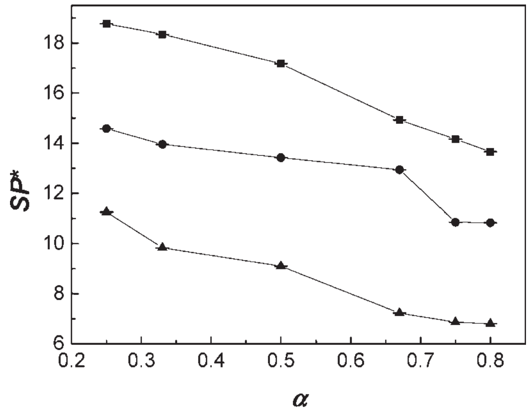

Figure 3. Effect of α values on the optimum solubilization parameters SP* for microemulsion systems CTAB + butan-1-ol + alkanes + NaCl solution (w = 0.025). ■, hexane; ●, octane; ▲, decane.

Solubilization Parameters SP.* The optimum solubilization parameter SP* of the microemulsion phase is defined here as the total mass of alkane and water mixture solubilized in the microemulsion phase per gram of surfactant.[32] According to this definition, SP* can be calculated from β_E and ε_E values.

$$\mathrm{SP}^* = \frac{1-\beta_E-\varepsilon_E}{\beta_E} \tag{2}$$

The variation of the optimum solubilization parameter SP* with α values for the microemulsion systems CTAB + butan-1-ol + alkanes + NaCl solution (w = 0.025) is shown in Figure 3.

Figure 3 indicated that the solubilization ability (SP*) of the microemulsion systems to water and alkane would be decreased

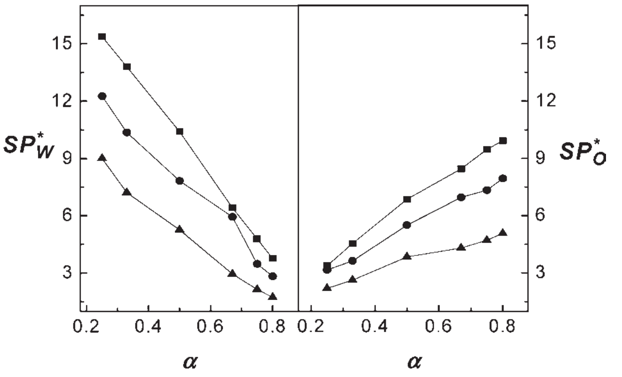

Figure 4. Plot of SP_W^* and SP_O^* vs α values of microemulsion systems CTAB + butan-1-ol + alkane + NaCl solution ($w = 0.025$). ■, hexane; ●, octane; ▲, decane.

as α values gets larger. That is, when the total mass of solubilized water and alkane remains unchanged, the larger the percentage mass of alkane becomes, the more surfactant would be needed to solubilize water and alkane.

SP^* can be divided to the sum of SP_W^* (the mass of solubilized water in the microemulsion phase per gram of surfactant, $SP_W^* = m_W(1 - \beta_E - \varepsilon_E)/[(m_0 + m_W)\beta_E]$ and SP_O^* (the mass of solubilized alkane in the microemulsion phase per gram of surfactant, $SP_O^* = m_0(1 - \beta_E - \varepsilon_E)/[(m_0 + m_W)\beta_E]$, $SP^* = SP_W^* + SP_O^*$. Figure 4 shows the variation of the solubilization parameters SP_W^* and SP_O^* with α values.

The microemulsion can solubilize both water and alkane; as α values increase, SP_W^* significantly decreases, but SP_O^* increases slowly compared to SP_W^* values. The solubilization ability on water is more significant than that of alkane. On balance, the addition of alkane and the subtraction of water weaken the solubilization ability of the microemulsion system.

Composition of the Interfacial Layer. The interfacial layer was composed of surfactant and alcohol. The mass fraction of alcohol in the interfacial layer A^S can be calculated from β_i and ε_i values in Table 1 as follows[28,30]

$$A^S = \frac{\varepsilon_i}{\beta_i + \varepsilon_i} \qquad (3)$$

In the CTAB + butan-1-ol + alkane + NaCl solution ($w = 0.025$) system, when α values were varied, a relationship between the mass fraction of alcohol in the interfacial layer, A^S, and α values was observed, as shown in Figure 5.

The increase in A^S values with α values in Figure 5 indicates that more alcohol molecules accumulated at the interfacial layer to reach the hydrophilic−lipophilic balance of the interfacial layer, as the α values increase. The accumulation of alcohol at the interfacial layer would lead to a decrease in solubilization ability simultaneously as indicated in Figure 3.

Effect of Alkanes on the ε_B, A^S, and SP Values.* Figure 6 shows the effect of alkanes with different carbon chain lengths on the properties of the microemulsion systems including (a) the solubility of alcohol ε_B in the water and oil phases, (b) the composition of the interfacial layer A^S, and (c) the solubilization ability SP^* values for systems CTAB + butan-1-ol + alkane + NaCl solution ($w = 0.025$) at different α values.

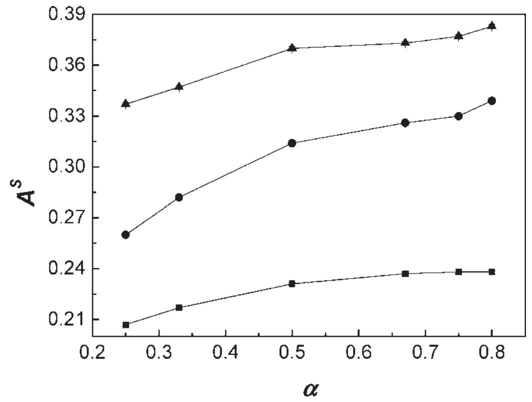

Figure 5. Plot of A^S vs α values of microemulsion systems CTAB + butan-1-ol + alkane + NaCl solution ($w = 0.025$). ■, hexane; ●, octane; ▲, decane.

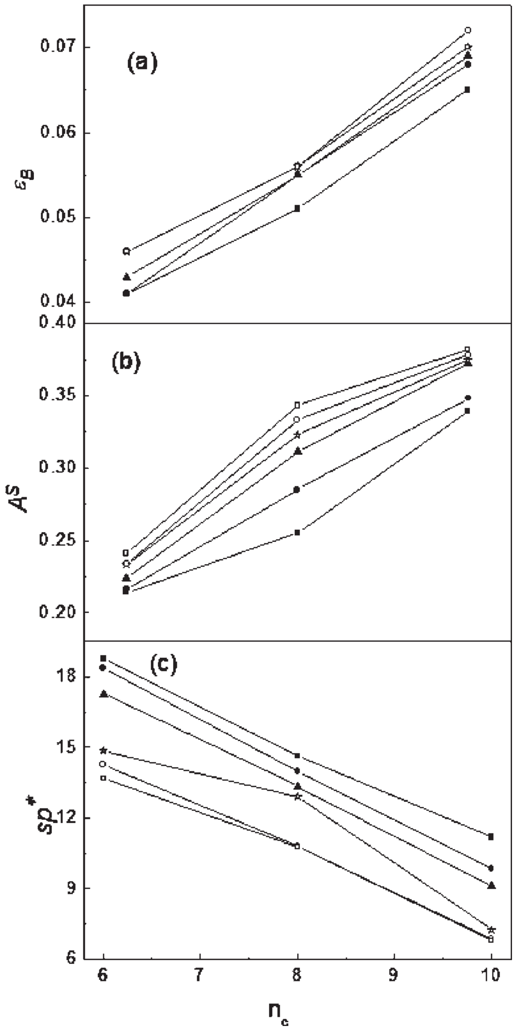

Figure 6. Effect of alkanes with different carbon chain lengths (n_c) on (a) ε_B, (b) A^S, and (c) SP^* values for systems CTAB + butan-1-ol + alkane + NaCl solution ($w = 0.025$) at different α values: ■, 0.25; ●, 0.33; ▲, 0.50; ☆, 0.67; ○, 0.75; □, 0.80.

It can be seen from Figure 6a that the solubility (ε_B) of the alcohol in the water and oil phase systems increases as the carbon chain length of the alkane molecules increases. This may be attributed to the increased interaction between the alcohol and the alkane molecules as the carbon chain length of the alkane molecules increases.

The alkane has notable influence on the composition of the interfacial layer (Figure 6b). When the length of the carbon chain of the alkane molecules increases, the mass fraction of alcohol (A^S) in the interfacial layer increases. This can be explained by the penetration and the hydrophobicity of the alkane molecules. The alkane molecules with the shorter carbon chain length are easier to penetrate the interfacial layer and improve the lipophile property of the interfacial layer, thereby a lesser amount of the alcohol was needed to solubilize all of the water and alkane in the microemulsion systems.

The microemulsion systems containing the alkane with a shorter carbon chain have a larger solubilization ability (Figure 6c). This may also be attributed to the penetration of the alkane molecules in the interfacial layer. As the carbon chain length of the alkane molecules increases, the penetration ability of the alkane molecules decreases, and fewer alkane molecules can enter into the interfacial layer which was composed of alcohol and surfactant molecules; therefore, more alcohol molecules are needed to balance the hydrophile−lipophile property of the interfacial layer. The amount of surfactant molecules needed to solubilize the water and alkane in the microemulsion system also increased, resulting in the decrease of the solubilization ability of the microemulsion systems.

■ CONCLUSIONS

The $\varepsilon-\beta$ fishlike phase diagram for microemulsion systems hexadecyltrimethylammonium bromide + alcohol + alkane + brine was plotted. Applying the hydrophilic−lipophilic balanced (HLB) plane equation to the phase diagrams, some important parameters of microemulsions, such as the composition of the interfacial layer, the solubilities of surfactant and alcohol, and the maximum solubilization ability of the microemulsion phase at various α values can be calculated.

The solubility of the alcohol in the microemulsion system increases as α values increase. In contrast, the solubilization ability of the microemulsion system on water and alkane decrease as the α values becomes larger. In addition, the mass fraction of the alcohol in the balanced interfacial layer increases as α values increase.

Alkanes have notable effects on the properties of the microemulsion systems. As the carbon chain length of the alkane molecules increases, the solubility of the alcohol and the mass fraction of the alcohol in the interfacial layer increases. However, the solubilization ability of the system on water and alkane in the microemulsion system would decrease.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information. $\varepsilon-\beta$ fishlike phase diagram data for Figure 1. This material is available free of charge via the Internet at http://pubs.acs.org.

■ AUTHOR INFORMATION

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