Effects of Isomeric Alcohols and Oils on the Properties of the Microemulsion Systems Formed by Alkyl Phenol Poly(oxyethylene ether)

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The effects of three isomeric alcohols, butan-1-ol, butan-2-ol, and 2-methyl-propan-2-ol, on the phase behavior and physicochemical properties of the pseudoquaternary microemulsion systems alkyl phenol poly(oxyethylene ether) (OP) + butan-1-ol (butan-2-ol, 2-methyl-propan-2-ol) + hexane (heptane, octane) + 0.05 NaCl solution were investigated with an $\varepsilon-\beta$ fishlike phase diagram method. The properties studied include the composition of the interfacial layer A^S, the solubilities of alcohols $A^{O,W}$, and the solubilization ability SP^* of the microemulsion systems. With the branched chain increasing, the A^S and $A^{O,W}$ values are in the order: butan-1-ol < butan-2-ol < 2-methyl-propan-2-ol, but the SP^* values are in the inverse order. These results were discussed. The effects of oils on the phase behavior and above physicochemical properties were also investigated.

Introduction

Microemulsions are clear, stable, isotropic liquid mixtures of oil, water, surfactant, and cosurfactant. Microemulsions have been identified as having enormous potential in many technical applications, such as tertiary oil recovery (EOR), cosmetics, foods, pesticides and coating of materials,[1,2] pharmaceutics,[3] nanoparticle synthesis,[4,5] extraction,[6,7] and so on. Studies on the phase behavior and properties of microemulsions have been continually done by scientific researchers.[8−10]

The ionic surfactant-based microemulsions would show effective detergency under normal conditions,[11−13] and they are widely used in many industrial and household fields. Nonionic surfactants are considered superior to ionic surfactants in some aspects. They are typically more hydrophobic than ionic surfactants and less sensitive to high electrolyte concentrations. Nonionic surfactant-based microemulsion systems have been widely studied.[14,15]

The nonionic surfactant alkyl phenol poly(oxyethylene ether) (OP) consists of oxyethylene groups as the polar head and octylphenyl as the hydrophobic part. OP can be used as one of the templates in the synthesis of polymers and other materials.[16,17] Zhai et al.[18] demonstrated that the synthesis of MCM-48 using the mixture of cationic CTAB (cetyltrimethyl ammonium bromide) and nonionic OP as a cotemplate is capable of providing highly ordered products with a range of unit cell sizes by simply changing the OP + CTAB ratios. Ge et al.[19] prepared composite particles by the solvent evaporation method and examined the effects of concentration of OP as a nonionic surfactant on the morphology of these anisotropic composite particles.

In contrast to the above-mentioned applications in a wide range of fields, there has been limited research work done on the fundamental properties of the OP based microemulsions. In the formation process of microemulsions, alcohols as cosurfactant and oils play important roles. The often-used cosurfactants in microemulsion systems are medium chain length alcohols, such as butanol and pentanol. The other alternatives are the alcohols with branched chains such as butan-2-ol and pentan-2-ol.[20,21] The role of alcohols with the straight chain in microemulsion systems has also been well-validated.[22−25] However, the effect of the branched alcohols on the microemulsion formations was less studied.

In this paper, the effects of alcohol isomers (butan-1-ol, butan-2-ol, and 2-methyl-propan-2-ol) and oils on the phase behavior and solubilization of OP-based microemulsion systems were studied in detail, with the use of an $\varepsilon-\beta$ fishlike phase diagram.

Experimental Section

Materials. Nonionic surfactant alkyl phenol poly(oxyethylene ether) (OP, with mass fraction purity > 0.98) was purchased from Shanghai Chemical Reagent Company, China. Butan-1-ol, butan-2-ol, 2-methyl-propan-2-ol, hexane, heptane, and octane (with mass fraction purity > 0.99) were purchased from Sinopharm Chemical Reagent Company, Ltd., China, and are of analytical grade. The inorganic salt (NaCl with mass fraction purity > 0.99) is of analytical grade and was purchased from Sinopharm Chemical Reagent Company Ltd. Water was doubly distilled after being ion-exchanged.

An FA 1104 electronic balance, a 601 super thermostat, and a TDL-5B centrifuge were used in this experiment.

Methods. The microemulsion samples were prepared by weighing a fixed quantity of the surfactant OP into a series of Teflon-sealed glass tubes, and then an equal quantity of 0.05 NaCl solution and oil (hexane, heptane, or octane) was also added into each of the tubes. Then all tubes were scanned with alcohol content, and the three-phase region was determined.

The above procedure was repeated at different fixed quantities of the surfactant OP; a series of three-phase regions were determined.

Representative samples were chosen and allowed to equilibrate in a water bath at (323.15 ± 0.01) K for about 1 week. At last the volumes of each phase were no longer changed, and the initially formed unstable macroemulsions changed to the thermodynamically stable microemulsion systems. The volumes of each phase were recorded by visual observation.

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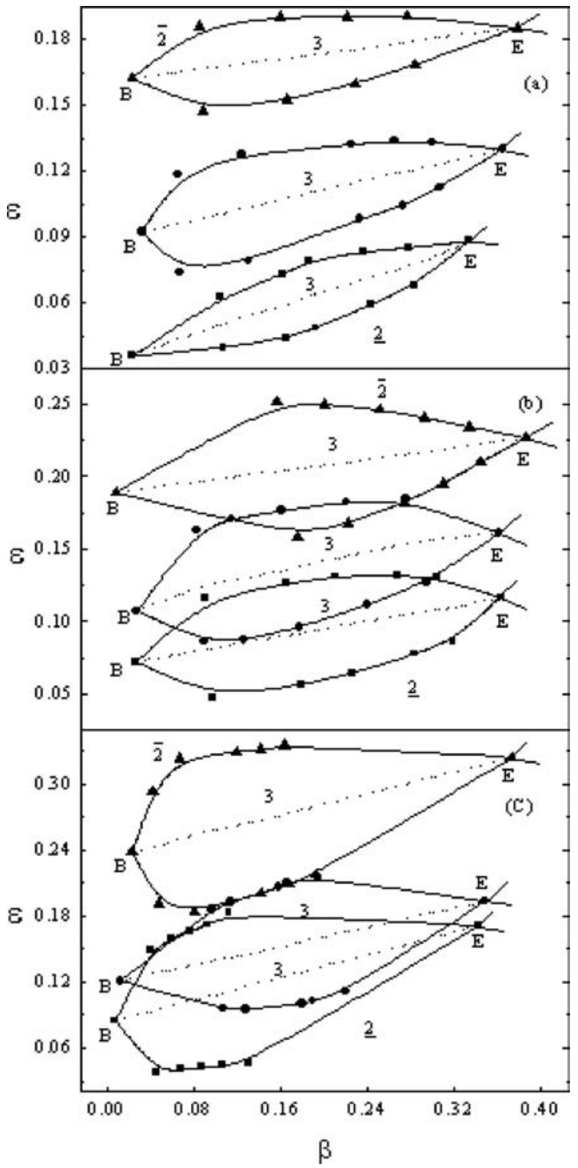

Figure 1. $\varepsilon-\beta$ fishlike phase diagrams for the microemulsion systems OP + alcohol + alkane + 0.05 NaCl solution: (a) hexane, (b) heptane, (c) octane; ■, butan-1-ol; ●, butan-2-ol; ▲, 2-methyl-propan-2-ol.

Results and Discussion

ε−β Fishlike Phase Diagrams of the Microemulsion Systems Containing Alkyl Phenol Poly(oxyethylene ether). For the quaternary microemulsion system of surfactant (S) + oil (O) + alcohol (A) + salt solution (W), the following composition variables were defined.[26,27] The symbol α represents the mass fraction of oil in brine plus oil, $\alpha = (m_O)/(m_O + m_W)$, β represents the mass fraction of surfactant in the whole system, $\beta = (m_S)/(m_W + m_O + m_S + m_A)$, and ε means the mass fraction of alcohol in the whole system, $\varepsilon = (m_A)/(m_S + m_A + m_W + m_O)$.

The $\varepsilon-\beta$ fishlike phase diagrams for the OP + butan-1-ol (butan-2-ol, 2-methyl-propan-2-ol) + hexane (heptane, octane) + 0.05 NaCl solution system at (323.15 ± 0.01) K and $\alpha = 0.5$ were shown in Figure 1 (see Supporting Information, Section S1).

With increasing β values in Figure 1, a three-phase region forms at the "fish head" (point B, β_B, ε_B) and terminates at the "fish tail" (point E, β_E, ε_E) where equal amounts of water and oil are solubilized in a single-phase microemulsion.

Figure 1 shows the inversions of the microemulsion phases of the three basic types. With ε increasing at constant β values, the phase type changes in sequence Winsor I ($\underline{2}$, oil-in-water) → Winsor III (3, bicontinuous microemulsion) → Winsor II ($\bar{2}$, water-in-oil).

According to Figure 1, different isomeric alcohols have notable effect on the $\varepsilon-\beta$ fishlike phase diagrams. As the branched chains of the alcohols increase, the "fish" moves upward, and at the same time, the parameters of the phase diagrams change accordingly (see Table 1).

Calculation of the Parameters for the Phase Diagrams. In Figure 1, the coordinates of the "fish head" (β_B, ε_B) and the "fish tail" (β_E, ε_E) can be calculated by the hydrophile−lipophile balance (HLB) plane equation.[27,28]

Then, the mass fractions of alcohol and surfactant contained in the balanced interfacial layer in the whole system, β_i [$\beta^i = (m_S^i)/(m_W + m_O + m_S + m_A)$] and ε_i [$\varepsilon_i = (m_A^i)/(m_W + m_O + m_S + m_A)$] ($m_S^i$ and m_A^i refer to the masses of surfactant and alcohol, respectively, in the interfacial layer), were obtained from the "fish head" (β_B, ε_B) and the "fish tail"(β_E, ε_E).[27] (β_B, ε_B), (β_E, ε_E), and (β_i, ε_i) were all listed in Table 1 and discussed as follows.

Effects of the Isomeric Alcohols on the Properties of the Microemulsion Systems. Mass Fraction of Alcohols in the Balanced Interfacial Layer, A^S. The mass fraction of the alcohol in the balanced interfacial layer, A^S, can be calculated from the β_i and ε_i values in Table 1.

$$A^S = \frac{m_A^i}{m_A^i + m_S^i} = \frac{\varepsilon_i}{\beta_i + \varepsilon_i} \qquad (1)$$

The effect of the isomeric alcohols on the A^S values was plotted in Figure 2.

Figure 2 shows that in microemulsion systems with hexane, heptane, and octane as the oil phase, respectively, the composition of the interfacial layer, A^S, depends on the isomeric alcohols and increases in the order: butan-1-ol < butan-2-ol < 2-methyl-propan-2-ol.

As a cosurfactant, the alcohol molecules with a short carbon chain play an important role in the formation of microemulsions. They penetrate into the interfacial layer, reducing the interfacial tension and improving the hydrophilicity of the interfacial layer.[25,29,30] Thus, it can promote the change in the interfacial curvature, resulting in the transformation of phase types.

In Figure 2, the butan-1-ol molecule with a straight carbon chain has a higher penetration ability, making the interfacial layer effectively more hydrophobic. Thus, it has higher efficiency to change the curvature of the interfacial layer. As a result, less butan-1-ol is needed to balance the hydrophile−lipophile property of the interfacial layer.

Butan-2-ol and 2-methyl-propan-2-ol molecules in Figure 2 have two and three methylic branched chains, respectively, and the penetration ability of their molecules is lower than that of butan-1-ol, resulting in their larger A^S values.

Solubilities of Alcohols in the Microemulsions. The values of the coordinates of the "fish head" (β_B, ε_B) reveal the solubilities of surfactant and alcohol in the whole system, respectively.[31]

The β_B values in Table 1 are very small as compared with β_i values. This shows that surfactant is mainly solubilized in the interfacial layer. The amount solubilized in oil and water phases can be ignored. Compared with ε_i values, ε_B cannot be ignored. As a result, the alcohol partitions between the interfacial layer

Table 1. Physical Parameters (Mass Fraction) of the Microemulsion Systems OP + Alcohol + Alkane + 0.05 NaCl Solution

	β_B	ε_B	β_E	ε_E	β_i	ε_i
			OP + Alcohol + Hexane + 0.05 NaCl Solution			
butan-1-ol	0.001 ± 0.001	0.036 ± 0.001	0.329 ± 0.001	0.088 ± 0.002	0.066 ± 0.001	0.329 ± 0.002
butan-2-ol	0.010 ± 0.001	0.092 ± 0.001	0.361 ± 0.001	0.130 ± 0.002	0.078 ± 0.001	0.355 ± 0.002
2-methyl-propan-2-ol	0.001 ± 0.001	0.162 ± 0.001	0.375 ± 0.001	0.185 ± 0.002	0.100 ± 0.001	0.375 ± 0.002
			OP + Alcohol + Heptane + 0.05 NaCl Solution			
butan-1-ol	0.026 ± 0.001	0.072 ± 0.001	0.362 ± 0.001	0.117 ± 0.002	0.075 ± 0.001	0.347 ± 0.002
butan-2-ol	0.026 ± 0.001	0.107 ± 0.001	0.362 ± 0.001	0.161 ± 0.002	0.102 ± 0.001	0.366 ± 0.002
2-methyl-propan-2-ol	0.002 ± 0.001	0.189 ± 0.001	0.418 ± 0.001	0.230 ± 0.002	0.148 ± 0.001	0.417 ± 0.002
			OP + Alcohol + Octane + 0.05 NaCl Solution			
butan-1-ol	0.007 ± 0.001	0.085 ± 0.001	0.342 ± 0.001	0.171 ± 0.002	0.125 ± 0.001	0.338 ± 0.002
butan-2-ol	0.012 ± 0.001	0.121 ± 0.001	0.348 ± 0.001	0.193 ± 0.002	0.129 ± 0.001	0.342 ± 0.002
2-methyl-propan-2-ol	0.023 ± 0.001	0.239 ± 0.001	0.373 ± 0.001	0.324 ± 0.002	0.226 ± 0.001	0.363 ± 0.002

and the water or oil phase,[31,32] in addition to the interfacial layer as discussed above.

The mean solubilities of the alcohol, $A^{O,W} = \varepsilon_B$ ($\varepsilon_B = (m_{A,B})/(m_W + m_O + m_{S,B} + m_{A,B})$, where $m_{S,B}$ and $m_{A,B}$ refer to the masses of surfactant and alcohol, respectively, at the "fish head"), in the microemusion system are shown in Figure 3.

It can be seen from Figure 3 that the mean solubility $A^{O,W}$ of the alcohol in different microemulsion systems with different oils is in the same order: 2-methyl-propan-2-ol > butan-2-ol > butan-1-ol.

2-Methyl-propan-2-ol is miscible with water as well as most common organic solvents, and it forms an azeotrope. The sterically hindered tertiary butyl group imparts its large solubility and stability compared to butan-1-ol and butan-2-ol. In microemulsion systems in Figure 3, the effect of this steric hindrance can be observed. However, the difference is that alcohol dissolved in water or oil would reach distribution equilibrium with the alcohol entering into the interfacial layer. That is, the solubility $A^{O,W}$ represents the mass fraction of alcohol dissolved in the water and oil phases in the total system at the point the interfacial layer just gets hydrophile−lipophile balanced.

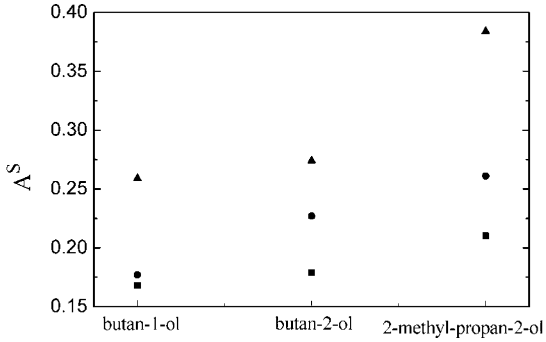

Figure 2. Effect of the isomeric alcohols on the A^S values in microemulsion systems OP + alcohol + alkane + 0.05 NaCl solution. ■, hexane; ●, heptane; ▲, octane. (See Supporting Information, Section S2.)

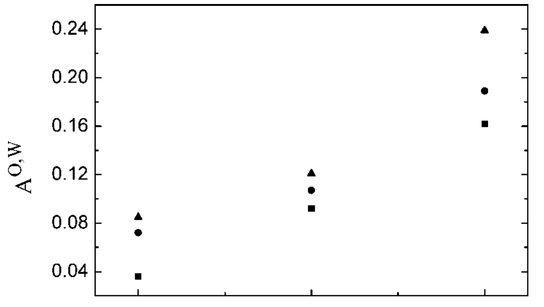

Figure 3. Effect of the isomeric alcohols on the mean solubility of alcohol, ε_B, in microemulsion systems OP + alcohol + alkane + 0.05 NaCl solution. ■, hexane; ●, heptane; ▲, octane. (See Supporting Information, Section S3.)

Solubilization Ability of the Microemulsion Systems. Substantial amounts of oil and water were solubilized in microemulsion phase. The solubilization ability of the microemulsion systems can be expressed as the optimum solubilization parameter SP*. SP* is defined as the mass of oil solubilized by per gram of surfactant in the microemulsion phase.

At the fish tail, equal amounts of oil and water were solubilized in the microemulsion phase. The solubilization ability of the microemulsion systems reaches the maximum value at this point. SP* can be calculated by the coordinate values of the fish tail (ε_E, β_E).

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

The values of SP* of the microemulsion systems OP + butan-1-ol (butan-2-ol, 2-methyl-propan-2-ol) + hexane (heptane, octane) + 0.05 NaCl solution are plotted in Figure 4. According to SP* values in Figure 4, the solubilization ability of the microemulsion systems formed by different alkanes is in the order: butan-1-ol > butan-2-ol > 2-methyl-propan-2-ol.[33]

According to reports in the literature,[34−36] the mixing nonionic surfactants,[34] added salt,[35] and temperature,[36] and so forth, have a significant effect on the solubilization ability of microemulsion systems. Few studies have described the effect of isomeric alcohols on the solubilization ability. In microemulsion systems, alcohol molecules are inserted into the interfacial layer and change the elastic modulus of the mixed interfacial layer. It can be deduced that butan-1-ol molecules with a straight carbon chain in Figure 4 have a larger ability to penetrate into

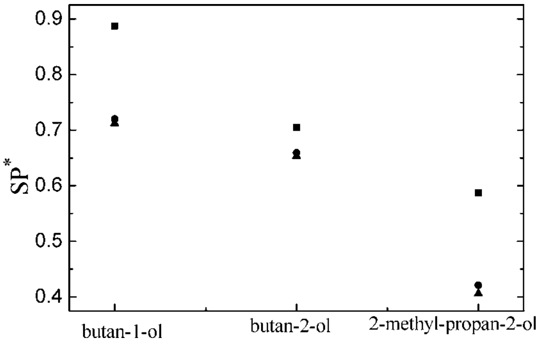

Figure 4. Effect of the isomeric alcohols on the solubilization parameters of different microemulsion systems OP + alcohol + alkane + 0.05 NaCl solution. ■, hexane; ●, heptane; ▲, octane. (See Supporting Information, Section S4.)

the interfacial layer, and it is easier to bend the elastic modulus of the interfacial layer than that of both butan-2-ol and 2-methylpropan-2-ol. Therefore, the microemulsion formed from butan-1-ol would show a better solubilization ability than the microemulsions formed from the other two alcohols with branched chains.

As seen above, three kinds of chemical data, the mean solubilities of the alcohol, $A^{O,W}$, the mass fraction of the alcohol in the balanced interfacial layer, A^S, and the optimum solubilization parameter, SP*, can be extracted from the $\varepsilon-\beta$ fishlike phase diagrams (Figure 1). These data are of great value in industrial applications. Similar to the oil recovery, the displacement or mobilization of residual nonaqueous phase liquids from the porous media requires ultralow interfacial tensions between aqueous and organic phases. These ultralow interfacial tensions can be achieved by the presence of a middle-phase microemulsion coexisting with both oil- and water-rich phases with a balanced interfacial layer.[37] The partitioning of alcohol in the optimum system would take place as mentioned above; this would affect the phase behavior of the system, especially with the change of water−oil ratio. So, an accurate selection of a surfactant−alcohol formulation for the mobilization of organic contaminants from soils requires consideration of the solubility of alcohol, $A^{O,W}$, the composition of the interfacial layer, A^S, and the solubilization ability, SP*, as major parameters.

Effect of Oils on the Phase Diagrams. Figure 5 shows the effect of alkanes with different carbon chain lengths on the properties of the microemulsion systems including A^S (a), $A^{O,W}$ (b), and SP* (c) values for the systems OP + butan-1-ol + alkane + 0.05 NaCl solution.

It can be seen from Figure 5 that, as the carbon chain length of the alkane molecules increases, A^S and $A^{O,W}$ increase, but SP* decreases.

As the oil phase of the microemulsion changes from hexane to heptane and octane, in the microemulsion systems in which the isomeric alcohols were used as cosurfactant, the mean solubility ($A^{O,W}$) of the alcohol in the water and oil phases increases. This may be explained by the penetration and the hydrophobicity of oil molecules. The increase in $A^{O,W}$ values may be attributed to the increased hydrophobicity of oil molecules, and thus the interaction between the alcohol and the alkane molecules as the carbon chain length of the alkane molecules increases.

The mass fraction of the alcohol in the interfacial layer (A^S) increases with increasing the carbon chain length of the alkane molecules. The alkane molecules with long carbon chain have a low ability to penetrate and enter the interfacial layer to improve the lipophile property of the interfacial layer. As such, more alcohol molecules were needed to enter into the interfacial layer, resulting in the increase in the mass fraction of alcohol in the interfacial layer (A^S).

On the other hand, as the penetration effect of the alcohol molecules becomes insignificant with the carbon chain length of the alkane molecules, the surfactant molecules needed to solubilize all of the water and oil in the microemulsion phase would increase. As a result, the solubilization ability of the microemulsion system would decrease.

Conclusions

The $\varepsilon-\beta$ fishlike phase diagrams of the OP-based microemulsion systems OP + butan-1-ol (butan-2-ol, 2-methylpropan-2-ol) + hexane (heptane, octane) + 0.05 NaCl solution were plotted. The effects of the isomeric alcohols and oils on the physicochemical properties of the microemulsion systems were investigated from the diagrams.

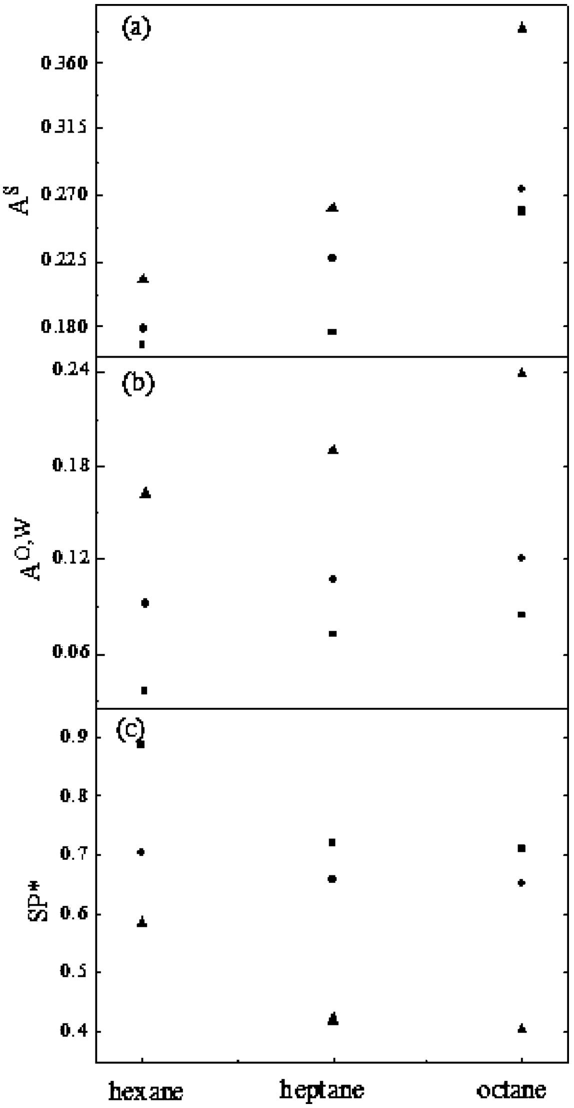

Figure 5. Effects of oils on A^S (a), $A^{O,W}$ (b), and SP* (c) values of the microemulsion systems OP + alcohol + alkane + 0.05 NaCl solution. ■, butan-1-ol; ●, butan-2-ol; ▲, 2-methyl-propan-2-ol.

The mass fraction of the alcohol in the balanced interfacial layer, A^S, increases in the order: butan-1-ol < butan-2-ol < 2-methyl-propan-2-ol.

The solubilities $A^{O,W}$ of the alcohol in different oil-based microemulsion systems are in the same order: butan-1-ol < butan-2-ol < 2-methyl-propan-2-ol, being opposite to the order of the penetration ability of these isomeric alcohol molecules into the interfacial layer.

The solubilization ability SP* of the microemulsion systems formed by different alkanes is in the inverse order: butan-1-ol > butan-2-ol > 2-methyl-propan-2-ol. It can be expained by the ability of alcohols to bend the elastic modulus of the interfacial layer.

As the carbon chain length of oil molecules increases, $A^{O,W}$ and A^S increase, and SP* decreases. This may be explained by the penetration and the hydrophobicity of the oil molecules.

Supporting Information Available:

Sections describing: (S1) the $\varepsilon-\beta$ fishlike phase diagram, (S2) the calculation of A^S values, (S3) the calculation of $A^{O,W}$ values, and (S4) the SP* values for microemulsion systems. This material is available free of charge via the Internet at http://pubs.acs.org.

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