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Effect of the Composition of Mixed Oils on the Phase Behavior and Solubilization Ability of Microemulsion Systems

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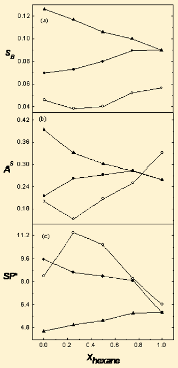
ABSTRACT: The phase behavior and the solubilization ability of the microemulsion systems containing mixed oils: sodium lauryl sulfate (SDS) + butan-1-ol + oil mixture (hexane + decane, hexane + cyclohexane, hexane + methylbenzene, methylbenzene + decane, methylbenzene + cyclohexane and methylbenzene + tetrachloroethene) + NaCl aqueous solution were studied using the ε–β fishlike phase diagram method. The synergism in the solubilization ability was observed in the microemulsion systems containing mixtures of methylbenzene + hexane, methylbenzene + cyclohexane, and methylbenzene + tetrachloroethene, respectively. The solubility of the alcohol in the oil and aqueous phases and the composition of the interfacial layer in the microemulsion systems containing oil mixtures were also investigated.



■ INTRODUCTION

Microemulsions are isotropic, transparent, and thermodynamically stable systems composed of water, oil, surfactant, and cosurfactant.[1,2] The solubilization of single-component oil and oil mixtures containing similar oil components in microemulsion systems has been widely studied.[3−6] Li et al.[7] studied the effect of different types of oils on the formation of microemulsions and found that the formation of the optimal microemulsion system is related to the polarity of oils. Methylbenzene with a stronger polarity is more likely to penetrate into the interfacial layer than alkanes, resulting in the lower salinities needed to form optimal microemulsions.

The relationship between the polarities of chlorocarbons as oil phase of microemulsions and the phase behavior was investigated.[8] It was found that less polar chlorocarbons would form classical Winsor-type phase behavior with the anionic surfactants. Moreover, the optimal solubilization of the chlorocarbons in microemulsion systerms was increased in the order of trichloroethene (TCE) > tetrachloromethane (CCl_4) > tetrachloroethene (PCE). Other common chlorocarbons are unable to form Winsor III type microemulsions with these surfactants. The electronegativity of the atoms in oil molecules also has an effect on the formation of microemulsions.[3,9−11] The solubilization ability of microemulsions was enhanced when the oil containing atoms with higher electronegativity was used as the oil phase. The solubilization ability of microemulsions with different oils was in the oil order: tetrachloromethane > dichloromethane > hexane. That is, the solubilization ability of the microemulsion with chlorocarbon as oil was increased as compared to the systems with alkane as oil. The explanation was that chlorine atom has higher electronegativity and is partially negatively charged in chlorocarbon molecules. Therefore, chlorocarbon molecules are more likely to penetrate into the interfacial layer and are prone to form microemulsions. In addition, the oils with shorter carbon chain lengths have a higher solubilization ability in the microemulsion systems,[4,12,13] and it is also explained by the increased penetration ability of the oils with shorter carbon chain lengths.

Moreover, it was found that mixtures of alkanes with different carbon chain lengths and many other types of hydrocarbons can be treated as a single pseudocomponent and no observable difference occurs upon solubilization.[14,15] Szekeres et al.[5,6,16−18] studied the modeling solubilization of oil mixtures in anionic surfactant-based microemulsions. Mixtures of polar and nonpolar oils were used as the oil phase, and long-chain alcohols were added to the microemulsions as lipophilic linkers. These alcohols should also be considered as oils of oil mixture, since they are not miscible with water, and remain primarily on the oil side of the microemulsion. When the volume fraction of dodecan-1-ol is below 0.2, regular Winsor type microemulsions would form, and above 0.2, regular Winsor type III microemulsions cannot form. Mathematical models were used to calculate the solubilization ability for optimum microemulsions in accordance with the experimental results.

Tetradecane was separately mixed with hexane, benzene, butylbenzene, and octylbenzene as oils to form microemulsions. The molar fraction of tetradecane in the binary mixture was varied from 0 to 1. The result showed that (alkyl) benzene molecules preferentially entered the interfacial layer. Thus small (alkyl) benzene molecules[19,20] are favorably selected.

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Polar and amphiphilic oils were added to microemulsion systems to increase the solubilization of hydrophobic oils.[5,17] The research on the solubilization of the dodecan-1-ol−limonene binary oil mixture showed that synergism in the solubilization ability exists due to the large polarity difference between dodecan-1-ol and limonene.

In practical applications, the oil mixture is often used. It should be useful to predict the solubilization ability of oil mixtures rather than single-component oil in microemulsion systems. In this paper, a $\varepsilon-\beta$ fishlike phase diagram[21] was used to characterize the phase behavior of the microemulsion system containing binary oil mixtures. The research aims to provide more insight to understand how the change in oil components affects the solubilization ability. In addition, the solubility of alcohol and the composition of the interfacial layer of the microemulsion system containing oil mixtures were also investigated.

■ EXPERIMENTAL SECTION

Materials and Apparatus. Sodium lauryl sulfate (SDS, CAS No. 151-21-3, with mass fraction purity >0.99) purchased from Beijing Drug Group Chemical Reagent Company, China, was analytical reagent (AR) grade and crystallized twice before use.

Butan-1-ol, hexane, decane, cyclohexane, methylbenzene, and tetrachloroethene (with mass fraction purity >0.99) were all AR grade. Butan-1-ol, hexane, decane, cyclohexane, methylbenzene, and tetrachloroethene were purchased from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China and 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) and an electrically heated thermostatic bath (accurate to 0.1 K, model CS501, Jiangsu, China) were used in the experiments.

Methods. The samples were prepared by weighing different mass quantities of surfactant into a series of Teflon-sealed glass tubes, and then the same mass quantities of water and mixed oils were added into the tubes. Two oil components are mixed in proportion in advance. Then a different amount of butan-1-ol was added into these tubes. The uncertainty of all samples weighed above was ± 0.0001 g. The above preparation process was repeated while the mass ratios of the oils were fixed at different values.

Representative samples were chosen and allowed to reach phase equilibrium in a thermostatic water bath in about 1 week's time. The volumes of each phase were recorded by visual observation. The volume of each phase was recorded with an accuracy of ± 0.02 mL.

■ RESULTS AND DISCUSSION

$\varepsilon-\beta$ Fishlike Phase Diagrams of SDS + Butan-1-ol + Hexane + Decane (Cyclohexane, Methylbenzene) + NaCl Solution (w = 0.025 or 0.05) Systems. In a microemulsion system of surfactant (S) + oil (O) + cosurfactant (A) + NaCl solution (W), the following composition variables were defined: α represents the mass fraction of oil to water plus oil in the system, $\alpha = m_O/(m_O + m_W)$, β the mass fraction of the surfactant in the whole system, $\beta = m_S/(m_S + m_A + m_O + m_W)$, and ε the mass fraction of the alcohol in the whole system, $\varepsilon = m_A/(m_S + m_A + m_O + m_W)$. m_A, m_S, m_O, and m_W are the masses of alcohol, surfactant, oil, and water, respectively. Keeping temperature (T), pressure (P), and α (0.5) constant, β was plotted horizontally and ε vertically, a $\varepsilon-\beta$ fishlike phase diagram could be obtained.[22]

Figure 1 shows the $\varepsilon-\beta$ fishlike phase diagrams for SDS + butan-1-ol + hexane + decane (cyclohexane, methylbenzene) + NaCl solution (w = 0.025 or 0.05) microemulsion systems.

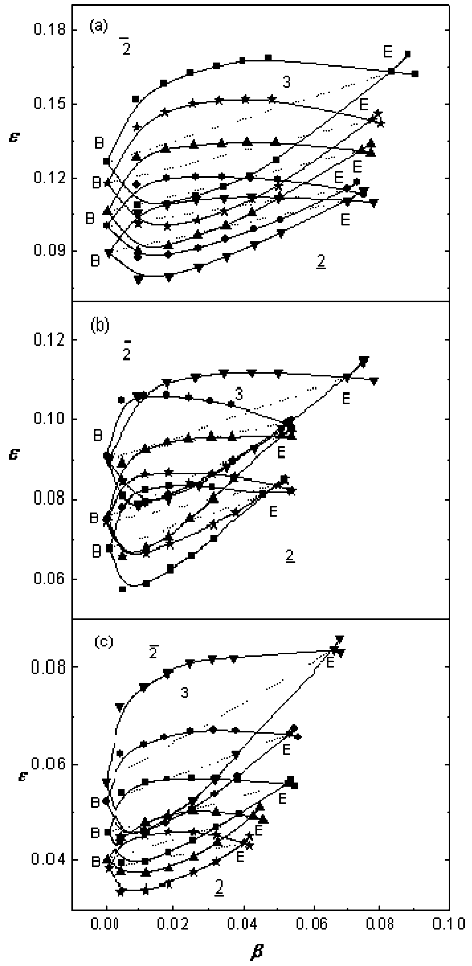

Figure 1. $\varepsilon-\beta$ fishlike phase diagrams of microemulsions systems SDS + butan-1-ol + hexane + decane (cyclohexane, methylbenzene) + NaCl solution at 303 K. X_{hexane}: ■, 0; ★, 0.25; ▲, 0.5; ●, 0.75; ▼, 1. (a) Hexane + decane (0.025 NaCl), (b) hexane + cyclohexane (0.025 NaCl), (c) hexane + methylbenzene (0.05 NaCl).

It can be seen from Figure 1 that increasing ε at constant β causes a phase inversion of Winsor I ($\underline{2}$) → III (3) → II ($\bar{2}$). At the "fish head" (point B), the three-phase microemulsion would start to form. At the "fish tail" (point E), the three-phase and single-phase regions intersect, where equal amounts of water and oil are solubilized in a single-phase microemulsion.

According to Figure 1, as the mass fraction of hexane increases, the "fish" would move upward. To evaluate the effect of the composition of mixed oils on the physicochemical properties of the microemulsion systems, some parameters related to the phase diagrams were obtained from Figure 1, according to literature,[13] and listed in Table 1.

In Table 1, (β_B, ε_B) and (β_E, ε_E) were the coordinates of the "fish head" and the "fish tail", respectively, and calculated by the hydrophile−lipophile balance (HLB) plane equation,[22,23]

Table 1. Values of β_B, ε_B, β_E, ε_E, β_i, and ε_i for Microemulsion Systems SDS + Butan-1-ol + Hexane + Decane (Cyclohexane, Methylbenzene) + NaCl Solution at 303 K[a]

X_{hexane}	β_B	ε_B	β_E	ε_E	β_i	ε_i
SDS + Butan-1-ol + Hexane + Decane + NaCl Solution (w = 0.025)						
0.00	0.0001	0.126	0.083	0.163	0.083	0.054
0.25	0.0002	0.117	0.078	0.144	0.077	0.037
0.50	0.0002	0.106	0.075	0.131	0.074	0.031
0.75	0.0001	0.100	0.070	0.115	0.070	0.030
1.00	0.0008	0.090	0.070	0.111	0.069	0.024
SDS + Butan-1-ol + Hexane + Cyclohexane + NaCl Solution (w = 0.025)						
0.00	0.0001	0.070	0.046	0.078	0.045	0.012
0.25	0.0002	0.073	0.050	0.086	0.050	0.018
0.50	0.0002	0.080	0.051	0.093	0.051	0.019
0.75	0.0003	0.090	0.052	0.104	0.052	0.021
1.00	0.0008	0.090	0.070	0.111	0.069	0.024
SDS + Butan-1-ol + Hexane + Methylbenzene + NaCl Solution (w = 0.05)						
0.00	0.0006	0.046	0.053	0.056	0.052	0.013
0.25	0.0008	0.038	0.040	0.044	0.039	0.007
0.50	0.0002	0.040	0.043	0.049	0.043	0.011
0.75	0.0001	0.052	0.054	0.066	0.053	0.018
1.00	0.0001	0.056	0.066	0.084	0.066	0.033

[a] Uncertainties: $\beta_B \pm 0.0001$; $\varepsilon_E \pm 0.002$; ε_B, β_E, β_i, and $\varepsilon_i \pm 0.001$.

β_i and ε_i in Table 1 are defined as

$$\beta_i = \frac{m_S^i}{m_W + m_{O1} + m_{O2} + m_S + m_A}$$

$$\varepsilon_i = \frac{m_A^i}{m_W + m_{O1} + m_{O2} + m_S + m_A} \qquad (1)$$

m_{O1} and m_{O2} represent the mass of hexane and decane (cyclohexane, methylbenzene), respectively. m_S^i and m_A^i represent the mass of SDS and the alcohol in the interfacial layer. β_i and ε_i indicate the mass fractions of surfactant and cosurfactant in the interfacial layer in the whole microemulsion system.

Effect of the Composition of Mixed Oils on the Properties of the Microemulsion Systems. *Solubilities of Alcohols in the Microemulsion Systems.* The mean solubilities of the alcohol, S_B [$= \varepsilon_B = m_{A,B}/(m_O + m_W + m_{A,B} + m_{S,B})$, where $m_{S,B}$ and $m_{A,B}$ refer to the masses of surfactant and alcohol, respectively, at the "fish head" in the microemulsion system], are shown in Figure 2a.

Figure 2a shows the variation of the values of S_B with X_{hexane} (mass fraction of hexane in alkane mixture), for microemulsion systems SDS + butan-1-ol + hexane + decane (cyclohexane, methylbenzene) + NaCl solution.

The alcohol is distributed among the oil phase, aqueous phase, and the interfacial layer in the microemulsion systems, thereby changing the interfacial curvature and obtaining the composition of the interfacial layer. It can be seen from Figure 2a that the solubility of butan-1-ol decreases with increasing X_{hexane} in the microemulsion systems containing the oil mixture (hexane + decane). This could be attributed to the contraction of the average carbon chain length as the mass fraction of hexane in alkane mixture increases. Therefore, the force of interaction between the alcohol and the alkane molecules would decrease,[12] resulting in the reduction of the mean solubility of the alcohol.

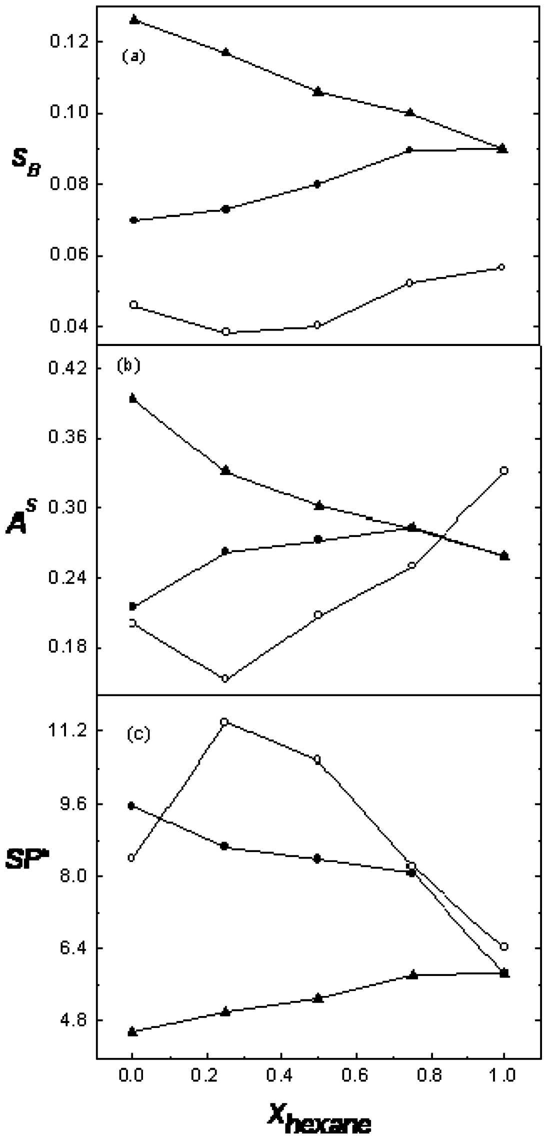

Figure 2. Effect of the composition of mixed oils on S_B (a), A^S (b), and SP* (c) values of the microemulsion systems SDS + butan-1-ol + hexane + decane (cyclohexane, methylbenzene) + NaCl solution. ▲, hexane + decane; ●, hexane + cyclohexane; ○, hexane + methylbenzene.

In addition, S_B would increase with increasing X_{hexane}, when hexane and cyclohexane are mixed in different mass ratios in Figure 2a.

The binary mixture of alkanes with different carbon numbers (ACN) follows simple mole fraction averaging rules for their microemulsion formation.[14,20] The EACN (equivalent alkane carbon number) of the alkane mixture ($EACN_{mix}$) can be obtained by $EACN_{mix} = X_1ACN_1 + X_2ACN_2$, where X represents the mole fraction of alkane to the alkane mixture. The equation could be modified to apply to any two components, with the general form as $EACN^*_{mix} = X_1^*(EACN_1^* - EACN_2^*) + EACN_2^*$.[20]

The EACN value of cyclohexane (3) is less than the EACN value of hexane (6).[20] That is, the values of $EACN^*_{mix}$ increase as X_{hexane} increases, and therefore the mean solubility of butan-1-ol increases, since the interaction between the alcohol and the alkane molecules increases.

S_B increases in general with increasing X_{hexane} for the microemulsion systems containing the oil mixture (hexane + methylbenzene) (EACN = 1)[20] (Figure 2a). This can also be attributed to the increase in the $EACN^*_{mix}$ values.

Mass Fraction of Alcohols in the Interfacial Layer. The mass fraction of alcohol in the interfacial layer, A^S, can be calculated from β_i and ε_i values.

$$A^S = \frac{\varepsilon_i}{\beta_i + \varepsilon_i} \tag{2}$$

The effect of the mixed oils on the A^S values was plotted in Figure 2b. Figure 2b shows that the A^S values show a similar trend of change as S_B values as X_{hexane} increases.

In the microemulsion systems containing the oil mixture [hexane + decane (EACN = 10)],[20,24] the $EACN^*_{mix}$ values decrease as X_{hexane} increases. Alkane molecules with short carbon chain have a strong ability to penetrate and enter the interfacial layer composed of surfactant and alcohol molecules. Therefore, a small amount of alcohol was needed to enter into the interfacial layer, resulting in the decrease in the A^S values.[12]

When the oil mixture (cyclohexane + hexane) was used as the oil phase, the A^S values of microemulsion increase as X_{hexane} increases. The circular cyclohexane molecule has a small molecular volume as compared to hexane molecule with straight chains, even though they contain the same carbon number. The small molecule can easily enter the interfacial layer and therefore needs less alcohol molecules in the interfacial layer.

A^S values increase in general in the microemulsion systems containing hexane and methylbenzene. This can also be attributed to the increase in $EACN^*_{mix}$ values as X_{hexane} increases.

Solubilization Ability of the Microemusion Systems. SP*, the solubilization parameter of the microemulsion phase, is often used to express the solubilization ability of the microemulsion system. SP* is defined as the mass of oil solubilized by each gram of surfactant in the microemulsion phase. β_E and ε_E values were used to calculated SP* values.

$$SP^* = \frac{1 - \beta_E - \varepsilon_E}{2\beta_E} \tag{3}$$

Figure 2c shows the relationship between SP* and X_{hexane} of the microemulsion systems.

In the microemulsion systems containing the oil mixture (hexane + decane), as X_{hexane} increases, the $EACN^*_{mix}$ values would decrease, and the oil molecules can easily enter the interfacial layer. Hence, the solubilization ability of microemulsion (SP*) increases.

As mentioned above, the larger hexane molecules are less likely to enter the interfacial layer as compared to cyclohexane molecule. Therefore, as X_{hexane} values increase, the solubilization ability of the system decreases for the microemulsion systems containing the oil mixture (hexane + cyclohexane).

The solubilization ability of the microemulsion system containing the oil mixture (hexane + methylbenzene) was significantly enhanced. This result in Figure 2c suggests the existence of synergism in the solubilization of the microemulsion systems between hexane and methylbenzene. This phenomenon may be explained by the polarity difference between methylbenzene and hexane. The more polar oil component methylbenzene was preferentially solubilized from the oil mixture (methylbenzene + hexane).[17]

ε–β Fishlike Phase Diagrams for the Microemulsion Systems Containing Methylbenzene as Oil. As mentioned above, synergism exists between methylbenzene and hexane. The effect between methylbenzene and some other oils was investigated.

The ε–β fishlike phase diagrams of SDS + butan-1-ol + mixed oil (methylbenzene + decane, methylbenzene + cyclohexane, and methylbenzene + tetrachloroethene) + NaCl solution (w = 0.05) were plotted in Figure 3, and the related physicochemical parameters were listed in Table 2.

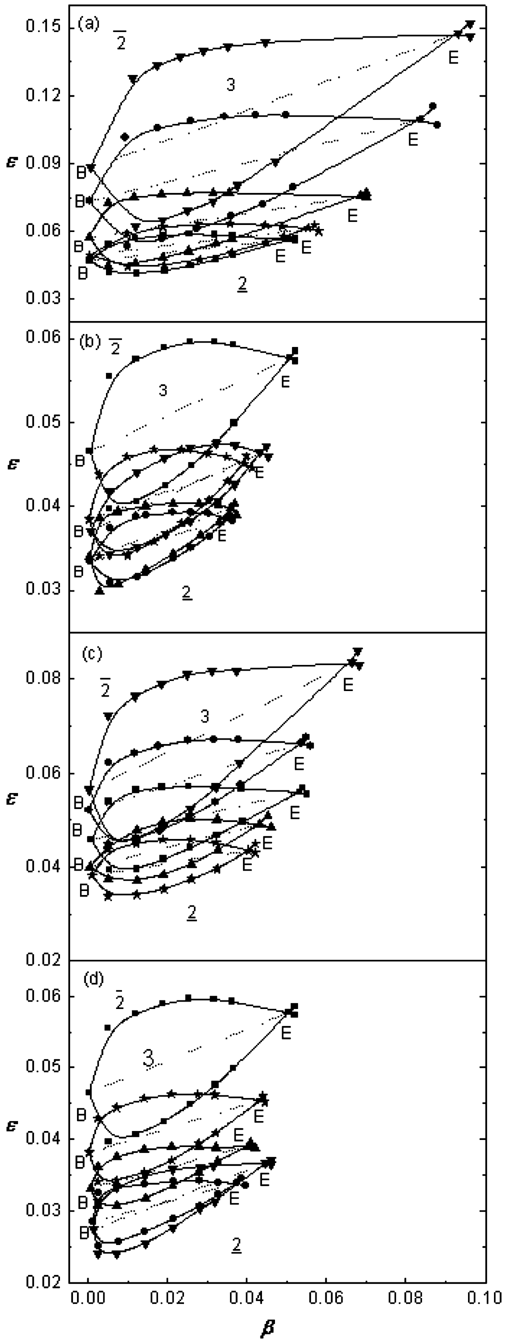

Figure 3. ε–β fishlike phase diagrams of SDS + butan-1-ol + methylbenzene + hexane (decane, cyclohexane, and tetrachloroethene) + NaCl solution (w = 0.05). $X_{methylbenzene}$: ▼, 0; ●, 0.25; ▲, 0.5; ★, 0.75; ■, 1. (a) Decane, 313 K; (b) cyclohexane, 313 K; (c) hexane, 303 K; (d) tetrachloroethene, 313 K.

Table 2. Values of β_B, ε_B, β_E, ε_E, β_i, and ε_i for Microemulsion Systems SDS + Butan-1-ol + Methylbenzene + Decane (Hexane, Cyclohexane, Tetrachloroethene) + NaCl Solution[a]

$X_{\text{methylbenzene}}$	β_B	ε_B	β_E	ε_E	β_i	ε_i
SDS + Butan-1-ol + Methylbenzene + Decane + NaCl Solution (w = 0.05) at 313 K						
0.00	0.0005	0.088	0.093	0.148	0.093	0.074
0.25	0.0002	0.073	0.084	0.109	0.084	0.045
0.50	0.0001	0.058	0.068	0.076	0.068	0.023
0.75	0.0002	0.050	0.055	0.061	0.055	0.015
1.00	0.0002	0.046	0.050	0.058	0.050	0.014
SDS + Butan-1-ol + Methylbenzene + Cyclohexane + NaCl Solution (w = 0.05) at 313 K						
0.00	0.0003	0.037	0.043	0.046	0.043	0.011
0.25	0.0002	0.033	0.035	0.038	0.034	0.006
0.50	0.0001	0.034	0.036	0.040	0.036	0.007
0.75	0.0001	0.038	0.039	0.045	0.039	0.008
1.00	0.0002	0.046	0.050	0.058	0.050	0.014
SDS + Butan-1-ol + Methylbenzene + Hexane + NaCl Solution (w = 0.05) at 303 K						
0.00	0.0001	0.056	0.066	0.084	0.066	0.033
0.25	0.0001	0.052	0.054	0.066	0.053	0.018
0.50	0.0002	0.040	0.043	0.049	0.043	0.011
0.75	0.0008	0.038	0.040	0.044	0.039	0.007
1.00	0.0006	0.046	0.053	0.056	0.052	0.013
SDS + Butan-1-ol + Methylbenzene + Tetrachloroethene + NaCl Solution (w = 0.05) at 313 K						
0.00	0.0013	0.027	0.045	0.037	0.043	0.011
0.25	0.0010	0.029	0.038	0.034	0.037	0.006
0.50	0.0005	0.033	0.040	0.039	0.040	0.007
0.75	0.0002	0.038	0.044	0.046	0.043	0.009
1.00	0.0002	0.046	0.050	0.058	0.050	0.014

[a] Uncertainties: $\beta_B \pm 0.0001$; ε_B, β_E, ε_E, β_i, and $\varepsilon_i \pm 0.001$.

Physicochemical Properties for the Microemulsion Systems Containing Methylbenzene as Oil. Figure 4 shows the relationship between S_B, A^S, SP*, and the mass fraction of methylbenzene in oil mixture $X_{\text{methylbenzene}}$ of microemulsion systems SDS + butan-1-ol + methylbenzene + decane [cyclohexane, tetrachloroethene, and hexane (for comparison)] + NaCl solution (w = 0.05).

It can be seen from Figure 4 that for the microemulsion systems containing the oil mixture (methylbenzene + decane, methylbenzene + hexane), as $X_{\text{methylbenzene}}$ increases, the mean solubility S_B of butan-1-ol and the A^S values decrease significantly. For the microemulsion systems containing the oil mixture [methylbenzene + cyclohexane, methylbenzene + tetrachloroethene (EACN = 2.9)],[20,24] the S_B and A^S values would change less significantly. When $X_{\text{methylbenzene}}$ increases, the EACN*$_{\text{mix}}$ values of the mixture (methylbenzene + decane) decrease as in the mixture (methylbenzene + hexane) mentioned above. As thus, the force of interaction between the alcohol and the alkane molecules decreases, resulting in the decrease in the mean solubility of butan-1-ol. However, for the other oil mixture, the change of the EACN*$_{\text{mix}}$ values was relatively small, and the mean solubility of butan-1-ol changes at a slower rate.

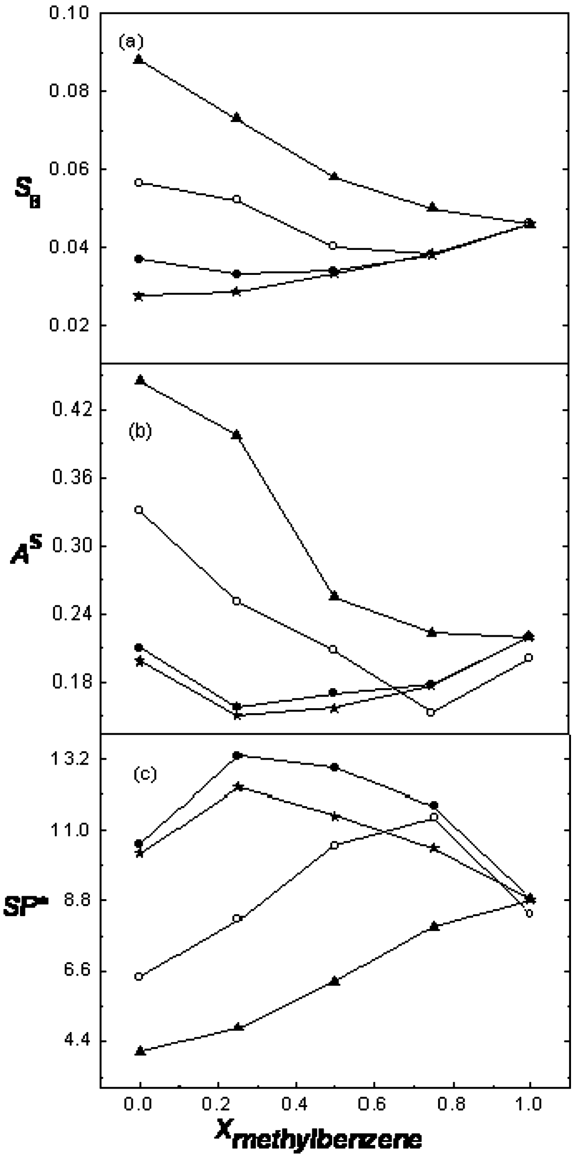

Figure 4. Physicochemical properties of the microemulsion systems SDS + butan-1-ol + methylbenzene + hexane (cyclohexane, decane, and tetrachloroethene) + NaCl solution (w = 0.05). ▲, decane; ●, cyclohexane; ○, hexane; ★, tetrachloroethene. (a) Mean solubility of the alcohol, S_B; (b) the mass fraction of butan-1-ol in the interfacial layer, A^S; (c) the solubilization ability of the microemulsion system, SP*.

As $X_{\text{methylbenzene}}$ increases, the EACN*$_{\text{mix}}$ values decrease for the microemulsion systems containing the oil mixture [methylbenzene + decane (hexane)]. The oil molecules can easily enter the interfacial layer, and hence the mass fraction of alcohol in the interfacial layer, A^S, would notably decrease.

Figure 4c suggests the existence of synergism between oil mixtures (methylbenzene + cyclohexane and methylbenzene + tetrachloroethene) on the solubilization ability (SP*) of the system, as in the microemulsion systems containing the oil mixture (methylbenzene + hexane). The solubilization ability of the microemulsion system containing the oil mixture (methylbenzene + decane) increases linearly with an increase in $X_{\text{methylbenzene}}$. However, no synergism was found in the microemulsion systems. It is predicted that this anomalous change could be associated with the weaker penetration ability of decane molecules due to their longer carbon chain length. Further studies could be done from this aspect.

CONCLUSIONS

The phase behavior and the solubilization ability of the microemulsion systems SDS + butan-1-ol + oil mixtures (hexane + decane, hexane + cyclohexane, hexane + methylbenzene, methylbenzene + decane, methylbenzene + cyclohexane, and methylbenzene + tetrachloroethene) + NaCl solution were studied using the $\varepsilon-\beta$ fishlike phase diagram method.

With increasing X_{hexane}, the solubility S_B of butan-1-ol in the microemulsion systems decreases when hexane + decane are used as oil phase but increases when hexane + cyclohexane or hexane + methylbenzene are mixed in different mass ratios. For the microemulsion systems containing oil mixtures (methylbenzene + decane and methylbenzene + hexane), as $X_{\text{methylbenzene}}$ increases, S_B values would significantly decrease. For the microemulsion systems containing oil mixtures (methylbenzene + cyclohexane and methylbenzene + tetrachloroethene), the S_B values would change gradually.

The mass fraction of alcohol in the interfacial layer, A^S, shows a similar trend of change as compared to S_B values when X_{hexane} and $X_{\text{methylbenzene}}$ increase.

Synergism exists between oil mixtures (methylbenzene + hexane, methylbenzene + cyclohexane, and methylbenzene + tetrachloroethene) on the solubilization ability (SP*) of the microemulsion systems.

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