

Water Solubilization Capacity and Volume-Induced Percolation of Sodium Bis(2-ethylhexyl)sulfosuccinate Microemulsions in the Presence of 1-Alkyl-3-Methylimidazolium Chloride Ionic Liquids

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S Supporting Information

ABSTRACT: The water solubilization capacity of sodium bis(2-ethylhexyl)sulfosuccinate (AOT) in isooctane microemulsions in the presence of chloride anion-based ionic liquids (ILs), such as 1-ethyl-3-methylimidazolium chloride (emimCl), 1-butyl-3-methylimidazolium chloride (bmimCl), and 1-octyl-3-methylimidazolium chloride (omimCl), was investigated. The water solubilization capacity is enhanced at low IL concentrations but decreases at high concentrations of IL. Meanwhile, the water solubilization capacity increases with the increase of chain length of ILs at low IL concentrations. The effect of ILs on the volume-induced percolation of AOT microemulsions was also studied. For the microemulsions, with an increasing volume fraction of dispersed phase (Φ), no percolation phenomena can be observed at high concentrations of IL, whereas the percolation conductance occurs at low IL concentrations and the percolation threshold (Φ_p) increases with the increase of IL concentrations. For the IL-containing microemulsions, the Φ_p increases with increasing IL chain length under the same conditions.

INTRODUCTION

Microemulsions are optically transparent, isotropic, thermodynamically stable mixtures of two immiscible liquids (nonpolar and polar) stabilized by amphiphilic molecules or surfactants. They have been extensively applied in many fields, such as chemical reactions, material science, separation science, and pharmaceutical industry.^{1–3}

Ionic liquids (ILs), which are typically composed of organic cations and organic/inorganic anions, have received a great deal of attention. They have some unusual properties, such as nonvolatility, nonflammability, and high thermal stability.^{4,5} Due to these special properties, ILs have been widely used in organic synthesis as solvents or catalysts.^{6–8} Recently it has been found that ILs can play an important role in water-in-oil (w/o) microemulsions. Lü et al.⁹ measured the critical concentrations and the critical temperatures for the microemulsions of water/1-butyl-3-methylimidazolium tetrafluoroborate (bmimBF₄)/sodium bis(2-ethylhexyl)sulfosuccinate (AOT)/decane with various compositions of bmimBF₄. Liu and co-workers showed that the concentration of a model IL bmimBF₄ can act as an effective interfacial control parameter for tuning the formation of microemulsions. It revealed that a smaller amount of IL needed than normal NaCl made ILs the most effective additives for curvature adjustment in ionic microemulsions.¹⁰ Rojas et al. investigated the influence of the IL 1-ethyl-3-methylimidazolium hexylsulfate (emimHexSO₄) on the spontaneous formation of microemulsions with ionic surfactants, which showed a significant increase of the transparent phase region by adding the IL.¹¹

In the water-based microemulsions, water solubilization capacity is an important property due to their practical applications. The water solubilization capacity depends on many factors, such as the oil chain length, the presence of cosurfactant, and the

salinity of the system.¹² It has been shown that the water solubilization capacity in the AOT/alkane system can be improved by the addition of salts. Hou and Shah¹³ studied the effect of salinity on the solubilization capacity of water in water/oil (w/o) microemulsions by both theoretical considerations and experimental observations. They explained the maximization in solubilization capacity as competition between two opposing factors, the curvature of the interface and the attractive interaction among the droplets. The limiting amounts of solubilized aqueous NaCl, NaNO₃, MgCl₂, and AlCl₃ in AOT/isooctane solution as a function of the ionic strength of the electrolytes were investigated by Hamada et al.¹⁴ An optimal ionic strength was reported in each system, and the results were interpreted in terms of salting-in and salting-out phenomena as well as from the counteracting effects of attractive intermolecular interaction and interfacial bending stress. The effects of NaCl on the water solubilization behavior of mixed microemulsions formed by AOT and nonionic surfactants were also reported by Liu et al.¹⁵ and Mitra and Paul.¹⁶ In this work the water solubilization capacity of AOT microemulsions in the presence of ILs is presented.

The percolation of conductance is an important and interesting physical phenomenon in w/o microemulsion. A manifold (100 to 1000 times) increase in conductance can take place after a threshold volume fraction of the dispersant at a constant temperature or after a threshold temperature at a constant composition.^{17–19} Recently, the effects of additives on volume-induced or temperature-induced conductance percolation of microemulsions have been examined. It has been shown that

Received: June 28, 2011

Accepted: August 2, 2011

Published: August 22, 2011

Table 1. Water Solubilization Capacity (W_0) of AOT Microemulsions with Three Chloride Anion-Based ILs at Various IL Concentrations (C_{IL})

C_{IL}		C_{IL}		C_{IL}	
mol·kg ⁻¹	W_0	mol·kg ⁻¹	W_0	mol·kg ⁻¹	W_0
emimCl					
0.000	47	0.050	99	0.090	52
0.010	53	0.060	118	0.100	44
0.020	60	0.070	138	0.120	36
0.030	69	0.080	160	0.150	30
0.040	83	0.085	64	0.200	24
bmimCl					
0.000	47	0.045	182	0.081	35
0.010	54	0.050	144	0.101	29
0.020	62	0.055	83	0.151	22
0.030	79	0.061	61	0.201	18
0.040	126	0.071	44		
omimCl					
0.000	47	0.033	172	0.070	33
0.010	55	0.035	148	0.080	29
0.020	67	0.040	92	0.100	25
0.025	80	0.050	54	0.150	20
0.030	127	0.060	40	0.200	17

certain additives can promote or retard the occurrence of percolation phenomenon, while others may remain totally ineffective.^{20–23} Among the salts, the bile salts sodium cholate (NaC) and deoxycholate (NaDC) assist percolation by reducing either the threshold volume fraction of water or the threshold temperature, whereas sodium salicylate (NaSal) and NaCl retard percolation by increasing the threshold volume fraction or temperature.^{24–26} However, the influence of ILs on the volume-induced or temperature-induced percolation of microemulsions remains unclear.

In this work, we investigated the water solubilization capacity and volume-induced percolation of AOT microemulsions in the presence of chloride anion-based ILs with different chain lengths. These results might be helpful to understand the effect of ILs on the behavior and properties of w/o microemulsions.

EXPERIMENTAL SECTION

Materials. AOT (mass fraction purity ≥ 0.99) was obtained from Sigma-Aldrich (USA), dried at 60 °C under vacuum, and used without further purification. The IL 1-ethyl-3-methylimidazolium chloride (emimCl, mass fraction purity 0.98) was obtained from Sigma-Aldrich (USA). The ILs including 1-butyl-3-methylimidazolium chloride (bmimCl, mass fraction purity > 0.99) and 1-octyl-3-methylimidazolium chloride (omimCl, mass fraction purity > 0.99) were purchased from Lanzhou GreenChem ILS, LICP, Chinese Academy of Sciences (China). The water mass fraction of ILs was determined by a Karl Fischer titrator, which are 0.008 for emimCl, 0.003 for bmimCl, and 0.001 for omimCl. The trace water in ILs was taken into account when preparing the aqueous IL solution. Isooctane (mass fraction purity ≥ 0.99) was purchased from Shanghai Jingchun Reagent Co., Ltd. (China).

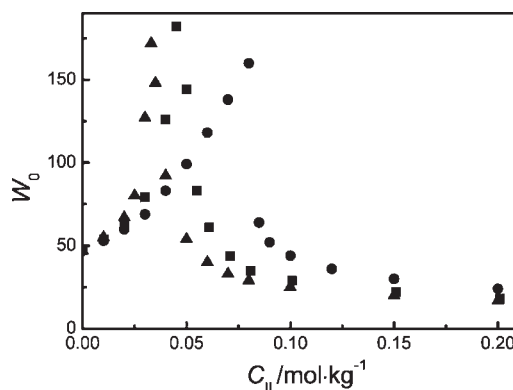


Figure 1. Water solubilization capacity (W_0) of AOT microemulsions with various concentration of ILs (C_{IL}): ●, emimCl; ■, bmimCl; ▲, omimCl.

High purity water was purchased from Hangzhou Wahaha Co., Ltd. (China).

Methods. The solution of 0.142 mol·kg⁻¹ AOT in isooctane was prepared at room temperature. For examining the water solubilization, the desired amount of water or aqueous IL solution was gradually injected into the AOT solution with a microsyringe under stirring until permanent turbidity resulted. Water content (W_0) is represented as the molar ratio of water to AOT.

The conductivity (κ) of microemulsions was measured by using a DDS-11A digital conductometer (Shanghai Leici Xinjing Instrument Company, China). The dependence of conductivity on volume fraction of dispersed phase (Φ) was measured by dropwise addition of water or aqueous IL solution into AOT solution. Here, Φ is defined as the volume fraction of the dispersed phase (water or aqueous IL solution), which is different from the volume fraction of droplets (i.e., the volume fraction of dispersed phase and surfactant)¹⁸ in the microemulsion system,

$$\Phi = \frac{V_d}{V_d + V_A} \quad (1)$$

where V_d and V_A are the volume of the dispersed phase (water or aqueous IL solution) and the AOT/isooctane solution, respectively. Each conductivity value reported was an average of three measurements. The experiments were carried out in a magnetically stirred, jacketed glass vessel. A constant temperature (308.15 ± 0.1 K) was maintained by circulating water through the outer jacket from a thermostatically controlled water bath.

The volume percolation threshold (Φ_p) was obtained by the sigmoidal Boltzmann fitting (SBF) procedure described by Hait et al.,²⁷

$$\log \kappa = \frac{\log \kappa_i - \log \kappa_f}{[1 + \exp(\Phi - \Phi_p)/d\Phi]} + \log \kappa_f \quad (2)$$

where Φ and Φ_p are the volume fraction of the dispersed phase in the system and that at the percolation threshold, respectively, and κ_i and κ_f are the initial and final specific conductance of the system, respectively.

RESULTS AND DISCUSSION

Water Solubilization Capacity. The water solubilization capacity of AOT in isooctane microemulsions in the absence and presence of ILs is summarized in Table 1 and Figure 1.

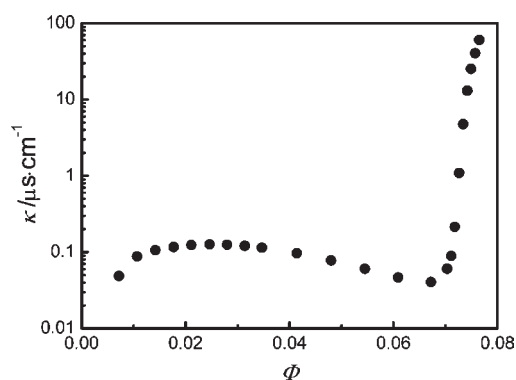


Figure 2. Dependence of conductivity (κ) on the volume fraction of water (Φ) in the microemulsions at 308.15 K.

As seen from Figure 1, the water solubilization capacity increases initially with increasing IL concentration and reaches a maximum and then decreases with a further increase of IL concentration. The behavior of exhibiting a maximum water solubilization capacity at a specific IL concentration is similar to the results reported for the AOT systems in the presence of inorganic electrolytes.^{14,28} The effect of IL on water solubilization capacity of AOT system can be interpreted as follows: On one hand, the addition of salt decreases the attractive interaction among droplets by making the interfacial layer more rigid due to closer packing of polar groups, which increases the water solubilization capacity.¹³ On the other hand, the addition of salt diminishes the effective polar area of the surfactant by decreasing the thickness of the electrical double layer around the polar group. This increases the natural negative curvature of the surfactant monolayer, which is also called “packing parameter” (P). P is defined as ν/al , where ν is the effective volume of a surfactant molecule, a is the effective area of its polar head, and l is the length of its hydrocarbon chain.¹⁶ As P increases, the water solubilization capacity decreases.¹³ Because of the two counteracting factors, a maximum value of water solubilization capacity is observed. As the concentration of salt becomes higher, the latter effect should play a dominant role, which explains the further decrease in water solubilization capacity.

The effect of chain length of ILs on the water solubilization capacity is also presented in Figure 1. For the ILs, the water solubilization capacity increases with the increase of chain length at low IL concentrations, which is similar to the effect of propanol to heptanol on the AOT microemulsions.¹³ As these alcohols, IL can act as a cosurfactant in w/o microemulsions stabilized by ionic surfactant.¹⁰ The long-chain IL makes the interfacial layer more rigid; therefore, the water solubilization of microemulsions varies in the order omimCl > bmimCl > emimCl for low IL concentrations. Besides, the maximum water solubilization is obtained at a lower IL concentrations for an IL with a longer chain length. This conclusion is also consistent with the results of propanol to heptanol in AOT microemulsions.¹³ It also reveals that the increase of the natural negative curvature is greater for ILs with a longer chain.

Volume-Induced Percolation. The variation of conductivity of AOT in isoctane microemulsions as a function of volume fraction of dispersed phase (water) is shown in Figure 2. As shown in Figure 2, initially, the conductivity increases with the addition of water and reaches a maximum, then followed by a decrease with increasing Φ , and reaches a minimum. The final

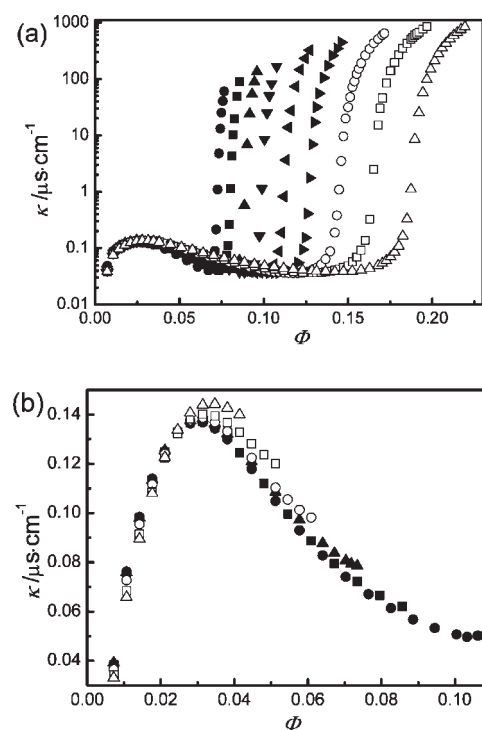


Figure 3. Dependence of conductivity (κ) on the volume fraction of dispersed phase (Φ) in the microemulsions at 308.15 K with various concentrations of emimCl: (a) ●, 0 mol·kg⁻¹; ■, 0.010 mol·kg⁻¹; ▲, 0.020 mol·kg⁻¹; ▼, 0.030 mol·kg⁻¹; left-pointing triangle, 0.040 mol·kg⁻¹; right-pointing triangle, 0.050 mol·kg⁻¹; ○, 0.060 mol·kg⁻¹; □, 0.070 mol·kg⁻¹; △, 0.080 mol·kg⁻¹; (b) ●, 0.085 mol·kg⁻¹; ■, 0.090 mol·kg⁻¹; ▲, 0.100 mol·kg⁻¹; ○, 0.120 mol·kg⁻¹; □, 0.150 mol·kg⁻¹; △, 0.200 mol·kg⁻¹.

dramatic increase of electrical conductivity with the further addition of water indicates the existence of a percolation phenomenon. Such conductance behavior has already been reported and explained by Liu et al.,^{29,30} Li and Li,³¹ and Chakraborty and Moulik.³² for AOT in heptane and isoctane microemulsions.

The effect of IL on the conductance behavior of AOT microemulsions was investigated. Figure 3 shows the variation of conductivity as a function of Φ in the presence of emimCl with varying IL concentrations. As shown in Figure 3a, the onset Φ for the percolation conductance increases with the presence of emimCl and continues to increase as the concentration of emimCl increased. This indicates that emimCl can retard the occurrence of percolation phenomenon, which has the same effect as NaCl.^{25,33} The addition of salt makes the interfacial layer more rigid, which hinders the exchange of water and ions between droplets, thus retarding the occurrence of percolation phenomenon. However, as the concentration of emimCl becomes higher, no percolation phenomena can be observed within the range of water solubilization capacity (Figure 3b); the system becomes unstable much earlier than the percolation state reached. There are same conductance behaviors for bmimCl and omimCl, which are given in Figures S1 and S2 in the Supporting Information (SI).

The Φ_p values of each microemulsion system with varying IL concentrations are determined by the SBF procedure and summarized in Table 2. As seen in Table 2 and Figure 4, Φ_p increases with increasing chain length of the ILs at the same IL concentration. The results also reveal that long-chain IL makes

Table 2. Volume Percolation Threshold (Φ_p) of AOT Microemulsions with Three Chloride Anion-Based ILs at Various IL Concentrations (C_{IL})

C_{IL}		C_{IL}	
mol·kg ⁻¹	Φ_p	mol·kg ⁻¹	Φ_p
emimCl			
0.000	0.0729 ± 0.0001	0.050	0.1282 ± 0.0002
0.010	0.0807 ± 0.0001	0.060	0.1466 ± 0.0002
0.020	0.0895 ± 0.0001	0.070	0.1665 ± 0.0002
0.030	0.0995 ± 0.0001	0.080	0.1896 ± 0.0003
0.040	0.1133 ± 0.0002		
bmimCl			
0.000	0.0729 ± 0.0001	0.030	0.1136 ± 0.0001
0.010	0.0828 ± 0.0001	0.040	0.1548 ± 0.0002
0.020	0.0939 ± 0.0001	0.045	0.2231 ± 0.0009
omimCl			
0.000	0.0729 ± 0.0001	0.025	0.1230 ± 0.0001
0.010	0.0836 ± 0.0001	0.030	0.1924 ± 0.0049
0.020	0.1040 ± 0.0001		

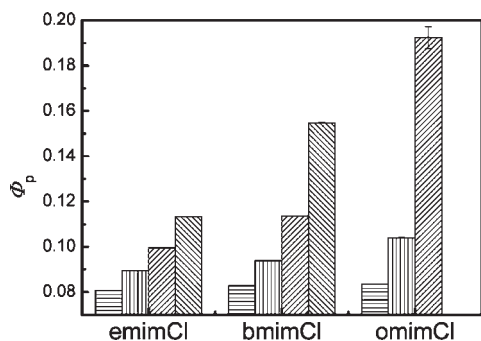


Figure 4. Volume percolation threshold (Φ_p) of AOT microemulsions with three chloride anion-based ILs at four IL concentrations: horizontal stripes, 0.010 mol·kg⁻¹; vertical stripes, 0.020 mol·kg⁻¹; diagonal stripes that travel upwards from left to right, 0.030 mol·kg⁻¹; diagonal stripes that travel downwards from left to right, 0.040 mol·kg⁻¹.

the interfacial layer more rigid, which delays the ion transfer more effectively and makes the percolation phenomenon appear later.

CONCLUSIONS

The effects of ILs, such as emimCl, bmimCl, and omimCl, on the water solubilization capacity and volume-induced percolation of AOT microemulsions were investigated. The water solubilization capacity is enhanced as a function of IL concentration at low concentrations of IL reaches a maximum and decreases for high IL concentrations. For the microemulsions, the water solubilization capacity increases with the increase of chain length of ILs at low IL concentrations. The volume percolation threshold (Φ_p) increases in the presence of IL and increases with the increase of the concentration of IL at low IL concentrations, whereas no percolation phenomena can be observed at high IL concentrations. The Φ_p value increases with increasing chain length of ILs under the same conditions.

ASSOCIATED CONTENT

S Supporting Information. The conductivity (κ) depends on the volume fraction of dispersed phase (Φ) in the microemulsions at 308.15 K with various concentrations of IL bmimCl or omimCl. Specific conductivity (κ) values at different volume fractions of water (Φ) in the microemulsions at 308.15 K. Specific conductivity (κ) values at different volume fractions of dispersed phase (Φ) in the microemulsions at 308.15 K with various concentrations of emimCl, bmimCl, or omimCl. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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Funding Sources

The authors are grateful for the financial support from the National Natural Science Foundation of China (Nos. 20936005 and 21076178) and the Zhejiang Provincial Natural Science Foundation of China (No. Y4080167).

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