



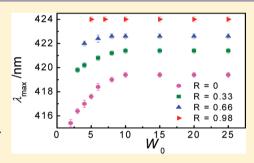
Effect of the Ionic Liquid 1-Butyl-3-Methylimidazolium Tetrafluoroborate on the Properties of Water + Triton X-100 + **Hexanol + Cyclohexane Microemulsions**

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Supporting Information

ABSTRACT: A study was carried out concerning the effects of an ionic liquid (IL) 1-butyl-3-methylimidazolium tetrafluoroborate (bmimBF₄) on the properties of nonionic surfactant-based H₂O + Triton X-100 (TX-100) + hexanol + cyclohexane microemulsions. Several properties of microemulsions, such as polarity, conductivity, viscosity, and droplet size, were investigated with the mass ratio of TX-100/hexanol/cyclohexane = 3:2:7. With the addition of IL, the polarity of the microemulsions increases. Conductivity results reveal that the onset water content of electrical percolation decreases with the presence of IL and continues to decrease as the amount of IL increases. The viscosity of microemulsions increases with the increasing amount of IL. Dynamic light scattering (DLS) measurements indicate that the droplet sizes of micro-



emulsions increase with the addition of IL. All results presented in this study suggest that the IL is solubilized into the polar core of microemulsions.

INTRODUCTION

Ionic liquids (ILs) are molten salts that generally consist of organic cations and organic/inorganic anions. They receive great attention as a class of neoteric solvent because of their special physical and chemical properties, such as low volatility, nonflammability, and high thermal stability. 1-4

Microemulsions are optically transparent, isotropic, thermodynamically stable dispersions of two immiscible liquids stabilized by surfactants or a surfactant/cosurfactant mixture.⁵ They have been an interesting subject for many years due to their broad applications in a variety of fields, such as separation, chemical reactions, and material preparation.⁶⁻⁹

In recent years, microemulsions containing ILs have become an interesting topic. It has been found that ILs can play an important role in water-in-oil (w/o) microemulsions. Liu et al. showed that the concentration of a model IL 1-butyl-3methylimidazolium tetrafluoroborate (bmimBF₄) can act as an effective interfacial control parameter for tuning the formation of microemulsions. It revealed that a smaller amount of IL than of normal NaCl is needed, which indicated that ILs are very effective additives for curvature adjustment in ionic microemulsions. 10 Lü and co-workers measured the critical concentrations and the critical temperatures for the microemulsions of water + bmimBF₄ + sodium bis(2-ethylhexyl)sulfosuccinate (AOT) + decane with various compositions of bmimBF₄. 11 Rojas et al. investigated the influence of the IL 1ethyl-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.¹² In the previous study, we have demonstrated that ILs have a significant impact on the water solubilization capacity and conductivity percolation of AOTbased aqueous microemulsions. 13,14

However, the effect of ILs on the nonionic surfactant-based aqueous microemulsions remains unclear. To the best of our knowledge, there is no report on the properties of nonionic surfactant-based aqueous microemulsions in the presence of ILs. Taking into account that nonionic surfactants are often used in practical applications, the aim of the research was to investigate the effect of ILs on the properties of nonionic surfactant-based aqueous microemulsions. In this work, the properties of H₂O + Triton X-100 (TX-100) + hexanol + cyclohexane microemulsions, such as polarity, conductivity, viscosity, and droplet size, in the presence of an IL, bmimBF₄, were investigated.

EXPERIMENTAL SECTION

Materials. TX-100 (polyoxyethylene tert-octylphenyl ether with 9.5 ethoxy groups, molecular biology grade, mass fraction purity >0.99) was obtained from Sigma-Aldrich and dried under vacuum at 353.15 K for 4 h before use. Hexanol (mass fraction purity >0.985), cyclohexane (mass fraction purity >0.995), and methyl orange (MO) were all purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). The

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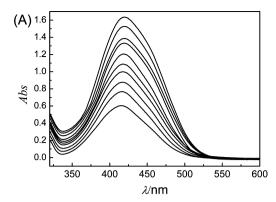
IL bmimBF₄ (mass fraction purity >0.99) was purchased from Lanzhou GreenChem ILS, LICP, Chinese Academy of Sciences (China). High purity water was provided by Wahaha Co., Ltd. (Hangzhou, China).

Apparatus and Procedures. UV–vis spectra were recorded on a Shimadzu UV-2550 spectrophotometer with the quartz cell placed in a thermostatted cell holder. The uncertainty in the measurement of the absorbance was less than \pm 0.002. The conductivity measurement was performed with a DDS-11A conductivity meter (Shanghai Precision & Scientific Instrument Co., Ltd.). The viscosity was measured with a Brookfield DV-II + Pro Cone/Plate viscometer. The diameters of the microemulsion droplets were determined by dynamic light scattering (DLS, Zetasizer Nano ZS, Malvern Instruments, UK) with a laser light wavelength of 633 nm and a scattering angle at 173°. The sample was filtered through a 0.45 μm Millipore membrane filter to remove possible dust prior to measurement. All measurements were made at (298.15 \pm 0.1) K.

Sample Preparation. According to the literature, ^{15,16} for the preparation of stable microemulsions, which possess a large water solubilization capacity, the mass ratio of TX-100 and cyclohexane was fixed at 3:7, and the mass ratio of TX-100 and hexanol was fixed at 3:2. The microemulsions were obtained by solubilizing the desired amount of water to the TX-100 + hexanol + cyclohexane solution. W_0 was represented as the molar ratio of water to TX-100. bmimBF₄ was added to the microemulsions, and the mixtures were then stirred until the dispersion became transparent. The amount of bmimBF₄ in the microemulsions was expressed as the molar ratio of bmimBF₄ to TX-100 (R). The uncertainty in the determination of the fraction of the samples was less than \pm 1 %.

■ RESULTS AND DISCUSSION

Polarity. The polarity of the $H_2O + TX-100 + hexanol +$ cyclohexane microemulsions in the absence and presence of IL bmimBF₄ was investigated by UV-vis spectroscopy using MO as an absorption probe. The shift in the wavelength of maximum absorption (λ_{max}) of MO is a sensitive measurement of the local environment. As a red-shift indicator, the λ_{max} of MO shifts to longer wavelengths as the media polarity increases. The effect of the successive addition of water on the absorption spectra of MO in H₂O + TX-100 + hexanol + cyclohexane microemulsions is shown in Figure 1A, and the corresponding variation of λ_{\max} with W_0 is shown in Figure 1B. With the increase of the W_0 value, the λ_{\max} of MO increased until it reached a stable value. Such a phenomenon has already been reported and explained by Qi and Ma²⁰ for H₂O + TX-100 + hexanol + cyclohexane microemulsions. It can be ascribed to the different types of water present in the microemulsions. In the TX-100 microemulsions, MO is located in the polar core but outside the water pool. MO preferentially penetrates the interfacial layer with its conjugated azo group embedded between the ethylene oxide (EO) groups of the polar chain of the TX-100 or the hydration shell and with its terminal SO₃Na group anchored in the water pool.²⁰ At small W_0 , most water added acts as bound water to hydrate the EO chains of the TX-100. Therefore, with the increase of the water content, the polarity of the polar core increases, which reflects in the increase of the λ_{\max} of MO. When the W_0 reaches a higher value, more and more water is present in its free form, and the water pool begins to form. With the further increase of the water content, water accumulates in the water pool and



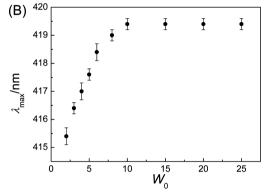


Figure 1. (A) Measurements of absorbance (Abs) of MO at different wavelengths (λ) in H₂O + TX-100 + hexanol + cyclohexane microemulsions as a function of water content (W_0). From bottom to top, the curves are: W_0 = 2 (a), W_0 = 3 (b), W_0 = 4 (c), W_0 = 5 (d), W_0 = 6 (e), W_0 = 8 (f), W_0 = 10 (g), W_0 = 15 (h), W_0 = 20 (i), and W_0 = 25 (j). Probe concentrations from a to j: 2.49·10⁻⁵, 2.88·10⁻⁵, 3.27·10⁻⁵, 3.66·10⁻⁵, 4.04·10⁻⁵, 4.47·10⁻⁵, 4.74·10⁻⁵, 4.98·10⁻⁵, 5.37·10⁻⁵, and 5.58·10⁻⁵ M, respectively. (B) Variation of wavelength of maximum absorption ($\lambda_{\rm max}$) of MO in H₂O + TX-100 + hexanol + cyclohexane microemulsions with water content (W_0).

leaves the microenvironment of the azo group and thus $\lambda_{\rm max}$ of MO unchanged.

The effect of the successive addition of water on the absorption spectra of MO in microemulsions in the presence of IL bmimBF₄ was also investigated, which are given in Figures S1 to S3 in the Supporting Information (SI). The corresponding variation of λ_{\max} with W_0 is shown in Table 1 and Figure 2. As shown in Figure 2, the λ_{max} of MO increases initially with increasing W_0 value and reaches a stable value with a further increase of W_0 value. These behaviors are similar to the results of microemulsions in the absence of IL. However, the W_0 value needed to get the stable value of λ_{max} of MO decreases with the increasing amount of IL bmimBF₄. The possible reason may be explained as follows: when bmimBF₄ is added to the microemulsions, $bmimBF_4$ molecules are bound to the EO groups of TX-100,21 which decreases the bound water. Therefore, in the presence of IL and increasing IL amount, the water needed to form a water pool decreases, and the W_0 value needed to get the stable value of λ_{\max} of MO decreases. Figure 2 also shows that the λ_{max} of MO increases with the increasing amount of IL at the same W_0 value, indicating that the polarity of the microemulsions increases with the addition of IL. It can be interpreted as follows: the IL bmimBF₄ is a polar solvent, which is highly immiscible with cyclohexane. ²² As a result, the added IL is only solubilized into the polar core of microemulsions. The λ_{max} of MO in bulk IL

Table 1. Wavelength of Maximum Absorption (λ_{max}) of MO in H₂O + TX-100 + Hexanol + Cyclohexane Microemulsions with the Mass Ratio of TX-100/Hexanol/Cyclohexane = 3:2:7 at Different Amounts of the IL bmimBF₄ (R) and Water Content (W_0)

W_0	$\lambda_{\rm max}/{ m nm}$	W_0	$\lambda_{\rm max}/{\rm nm}$	W_0	$\lambda_{\rm max}/{\rm nm}$		
R = 0							
2	415.4 ± 0.3	6	418.4 ± 0.3	20	419.4 ± 0.2		
3	416.4 ± 0.2	8	419.0 ± 0.2	25	419.4 ± 0.2		
4	417.0 ± 0.3	10	419.4 ± 0.2				
5	417.6 ± 0.2	15	419.4 ± 0.2				
R = 0.33							
3	419.8 ± 0.2	8	421.2 ± 0.2	20	421.4 ± 0.2		
4	420.2 ± 0.2	10	421.4 ± 0.2	25	421.4 ± 0.2		
6	420.8 ± 0.2	15	421.4 ± 0.2				
R = 0.66							
4	422.0 ± 0.2	10	422.6 ± 0.2	25	422.6 ± 0.2		
6	422.4 ± 0.3	15	422.6 ± 0.2				
8	422.6 ± 0.2	20	422.6 ± 0.2				
R = 0.98							
5	424.0 ± 0.2	10	424.0 ± 0.2	20	424.0 ± 0.2		
7	424.0 ± 0.2	15	424.0 ± 0.2	25	424.0 ± 0.2		

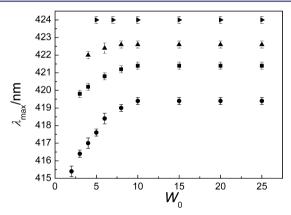


Figure 2. Variation of wavelength of maximum absorption (λ_{\max}) of MO in H_2O + TX-100 + hexanol + cyclohexane microemulsions with water content (W_0) at different amounts of IL bmimBF₄; $R: \bullet$, 0; \blacksquare , 0.33; \blacktriangle , 0.66; \blacktriangleright , 0.98.

bmimBF₄ is 430 nm,²¹ which is larger than that in H₂O + TX-100 + hexanol + cyclohexane microemulsions (419.4 nm). It reveals that bmimBF₄ is more polar than these microemulsions. Therefore, with the addition of bmimBF₄, the polarity of the microemulsions increases, which reflected in the increasing λ_{max} of MO.

Conductivity. Electrical conductivity is a useful tool to gain information of microemulsions, such as the variation of the droplet size and attractive interactions between droplets. 23,24 Figure 3 shows the variation of the conductivity of $H_2O + TX_100 + hexanol + cyclohexane microemulsions as a function of <math>W_0$ containing different amounts of the IL bmimBF₄ at 298.15 K. The presence of the IL bmimBF₄ can remarkably increase the conductivity of the microemulsions, which is possibly related to the high conductivity of IL. It is clear from Figure 3 that all conductivity versus W_0 variations present an electrical percolation phenomenon, which indicates the existence of large attractive interactions between droplets. The percolation threshold was obtained with the method described by Gao et al. 25 (see Figure S4 in SI) and is summarized in Table 2. As

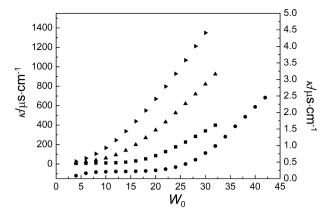


Figure 3. Variation of the conductivity (κ) of the microemulsions with water content (W_0) at different amounts of the IL bmimBF₄; R: \bullet , 0 (right vertical coordinate); \blacksquare , 0.33 (left vertical coordinate); \blacktriangle , 0.63 (left vertical coordinate); \blacktriangleright , 0.94 (left vertical coordinate).

Table 2. Percolation Threshold $(W_{0,p})$ of $H_2O+TX-100+Hexanol+Cyclohexane$ Microemulsions with the Mass Ratio of TX-100/Hexanol/Cyclohexane = 3:2:7 at Different Amounts of the IL bmimBF₄ (R)

R	$W_{0,\mathrm{p}}$
0	26.70 ± 0.14
0.33	17.29 ± 0.21
0.63	12.39 ± 0.21
0.94	9.81 ± 0.19

seen from Table 2, the percolation threshold decreases with the presence of IL and continues to decrease as the amount of IL increases. It indicates an increase of the attractive interaction between droplets with the addition of IL. With increasing IL content, the volume fraction of the droplets increases, and thus the percolation threshold decreases.

Viscosity. The viscosity of microemulsions, which can also provide the information of the variation of the attractive interactions between droplets, was investigated (Table 3).

Table 3. Viscosity (η) of H₂O + TX-100 + Hexanol + Cyclohexane Microemulsions with the Mass Ratio of TX-100/Hexanol/Cyclohexane = 3:2:7 at Different Amounts of the IL bmimBF₄ (R) and Water Content (W_0)

	η/mPa·s					
W_0	R = 0	R = 0.33	R = 0.66	R = 0.92		
5	3.57 ± 0.07	4.04 ± 0.08	4.90 ± 0.11	5.69 ± 0.13		
10	4.52 ± 0.10	5.35 ± 0.11	6.44 ± 0.14	7.41 ± 0.16		
15	5.84 ± 0.12	6.92 ± 0.15	8.50 ± 0.19	9.47 ± 0.20		
20	7.56 ± 0.16	9.45 ± 0.22	10.5 ± 0.23	12.1 ± 0.27		

Figure 4 shows the variation of viscosity as a function of W_0 in the absence and presence of bmimBF₄. As shown, the viscosity increases when either the W_0 value or IL amount increased. At each W_0 , the viscosity increases with the increasing amount of IL, which also indicates an increase of the attractive interaction between microemulsion droplets with the addition of IL. Additionally, the increase of viscosity is also due to the increase of microemulsion droplet size. ²⁶

Droplet Size. The DLS method is used to study the size of the microemulsions at two typical compositions with and without IL. Figure 5 and Figure S5 of the SI show the variation

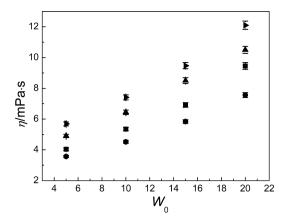


Figure 4. Variation of the viscosity (η) of the microemulsions with water content (W_0) at different amounts of the IL bmimBF₄; $R: \bullet$, 0; \blacksquare , 0.33; \blacktriangle , 0.66; \blacktriangleright , 0.92.

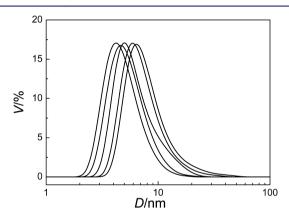


Figure 5. Variation of the drop size (diameter, D) and size distribution of the microemulsions by volume (V) with IL content (R) at $W_0 = 10$. From left to right, the curves are: R = 0 (a), R = 0.17 (b), R = 0.37 (c), R = 0.62 (d), and R = 0.90 (e).

of the size and size distribution of microemulsions with IL content (R) at $W_0 = 10$ and $W_0 = 15$, respectively. The corresponding droplet sizes are listed in Table 4. As shown in

Table 4. Droplet Size (Diameter, D) of H₂O + TX-100 + Hexanol + Cyclohexane Microemulsions with the Mass Ratio of TX-100/Hexanol/Cyclohexane = 3:2:7 at Different Amounts of IL bmimBF₄ (R) and Water Content (W_0)

W_0	R	D/nm	W_0	R	D/nm
10	0	5.0 ± 0.1	15	0	5.7 ± 0.1
10	0.17	5.6 ± 0.1	15	0.15	6.3 ± 0.2
10	0.37	6.6 ± 0.1	15	0.37	7.2 ± 0.2
10	0.62	7.7 ± 0.2	15	0.61	8.0 ± 0.3
10	0.90	8.5 ± 0.2	15	0.92	8.9 ± 0.2

Figure 6, the droplet sizes of the microemulsions increase with the addition of IL. Moreover, the droplet sizes show a nearly linear behavior as a function of the IL bmimBF₄'s amount, indicating that the added IL is solubilized into the polar core of microemulsions.

CONCLUSIONS

The properties of microemulsions of $H_2O + TX-100 + hexanol + cyclohexane$, such as polarity, conductivity, viscosity, and droplet size, in the presence of an IL bmimBF₄, were

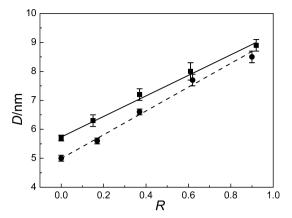


Figure 6. Variation of the drop size (diameter, D) of the microemulsions with IL content (R) at fixed W_0 ; W_0 : \bullet , 10; \blacksquare , 15. Solid and dashed lines represent the results of the linear regression analysis for the two cases, respectively.

investigated. The polarity of the microemulsions in the absence and presence of the IL bmimBF $_4$ was investigated by UV—vis spectroscopy using MO as an absorption probe. It shows that the polarity of the microemulsions is enhanced in the presence of ILs. The studies of conductivity and viscosity reveal an increase of the attractive interaction between microemulsion droplets with the addition of IL. DLS results indicate that the droplet sizes of the microemulsions increase in the presence of IL and increase with the increase of the amount of IL. All of the results indicate that the added IL is solubilized into the polar core of microemulsions.

ASSOCIATED CONTENT

S Supporting Information

Absorption spectra of MO in microemulsions of $\rm H_2O + TX-100 + hexanol + cyclohexane$ as a function of water content (W_0) at R=0.33, 0.66, and 0.98; determination of the electrical percolation threshold $(W_{0,p})$ of microemulsions; variation of the size and size distribution of the microemulsions with IL content (R) at $W_0=15$; specific conductivity of the microemulsions with W_0 at different amounts of the IL bmimBF₄. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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