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Study of Ionic Liquid Microemulsions: Ethylammonium Nitrate/TritonX-100/ Cyclohexane

In this study, ionic liquid (IL), specifically ethylammonium nitrate (EAN), was used instead of water to form nonaqueous microemulsions with cyclohexane and the nonionic surfactant Triton X-100 (TX-100). The phase behavior of the ternary system was investigated, and the microemulsions of ionic liquid-in-oil (IL/O) and oil-in-ionic liquid (O/IL) and the bicontinuous microregion were identified through traditional electrical conductivity measurement. The micropolarities of the IL/O microemulsions were determined via UV–Vis spectroscopy with methyl orange as an absorption probe. Results indicated that the polarity of the reverse micelles remained constant but that of the IL/O microemulsions increased when IL pools were formed. Fourier transform infrared spectroscopy was used to study the interaction mechanism between TX-100 and EAN molecules in IL/O microemulsions. We demonstrated that IL/O microemulsions may be promising for application due to the unique features of ILs and microemulsions.

Key words: Ionic liquid, microemulsion, microstructure, Triton-X 100

Untersuchung von Mikroemulsionen mit ionischer Flüssigkeit: Ethylammoniumnitrat/TritonX-100/Cyclohexan. In dieser Untersuchung wurde eine ionische Flüssigkeit (IL), genauer Ethylammoniumnitrat (EAN), anstelle des Wassers eingesetzt, um eine nicht wässrige Mikroemulsion aus Cyclohexan und dem nichtionischen Tensid Triton-X 100 (TX-100) herzustellen. Das Phasenverhalten des ternären Systems wurde untersucht. Die Mikrobereiche der ionischen Flüssigkeit in Öl (IL/O), die bikontinuierlichen Bereiche und die Mikroemulsionen aus Öl-in-ionischer Flüssigkeit (O/IL) wurden mittels üblicher Messungen der elektrischen Leitfähigkeit identifiziert. Die Mikropolaritäten der IL/O-Mikroemulsionen wurden bestimmt mittels UV-Vis-Spektroskopie, wobei Methylorange als Absorptionssonde verwendet wurde. Die Ergebnisse machten deutlich, dass die Polarität der Umkehrmizellen konstant blieb, dass aber die der IL/O-Mikroemulsion anstieg, wenn sich ein IL-Pool bildete. Die Fourier-Transformations-IR-Spektroskopie wurde für die Untersuchung des Wechselwirkungsmechanismus zwischen den TX-100- und den EAN-Molekülen in den IL/O-Mikroemulsionen eingesetzt. Wir haben gezeigt, dass IL/O-Mikroemulsionen für die Anwendung vielversprechend sein können, in Anbetracht der einzigartigen Eigenschaften von ILs und Mikroemulsionen.

Stichwörter: Ionische Flüssigkeit, Mikroemulsion, Mikrostruktur, Triton-X 100

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1 Introduction

Microemulsions are thermodynamically stable and macroscopically homogeneous mixtures that are usually composed of two or more immiscible liquids. These liquids are stabilized by a surfactant film at the liquid–liquid interface [1]. Thus far, microemulsions have been widely used in various fields, such as in chemical reactions and the synthesis of nanomaterials [2, 3]. However, the surfactants in microemulsions generally form a palisade layer between the water and oil constituents, thus rendering the structure heterogeneous on a microscopic scale.

Recent attempts have been made to prepare and study nonaqueous microemulsions by replacing the water incorporated into traditional microemulsions with other nonaqueous solvents. These novel microemulsions have attracted much research interest from both the theoretical and practical viewpoints. In particular, ionic liquids (ILs) have received much attention because of their special properties, including low volatility, nonflammability, and high thermal stability [4–8]. As ideal alternative solvents, ILs have been widely applied in chemical reactions, separations, electrochemical applications, biopolymers, and molecular self-assembly [9–11]. ILs have been employed as substitutes for water or organic solvents in preparing novel microemulsions given that ILs are immiscible with water or nonpolar organic solvents [12–17]. For example, Han et al. [6] discovered that the hydrophilic IL 1-butyl-3-methylimidazolium tetrafluoroborate ($bmimBF_4$) can replace water in generating a nonaqueous IL microemulsion with the surfactant Triton X-100 (TX-100). The results of freeze-fracture electron microscopy indicated that the shape of microemulsion droplets was similar to that of classic water-in-oil (W/O) microemulsion droplets. Eastoe et al. investigated further the microemulsion system via small-angle neutron scattering and determined that microemulsion droplets increased consistently in volume as micelles swelled progressively with the addition of $bmimBF_4$. This behavior is consistent with that of classic W/O microemulsions [18]. The effect of different linear-chained alcohols on phase stability of IL microemulsions was studied and it was found that alcohols having short chain lengths were optimum cosurfactants in IL microemulsions [19]. With long chain lengths, ILs can be further used as surfactants in microemulsion or aqueous media [20, 21].

Although many IL properties can be "tuned" over wide ranges by varying the anion and cation types, the solubility in nonpolar solvents is likely to be overcome by the formation of ionic liquid-in-oil (IL/O) microemulsions if ILs are highly polar [22]. Thus, these liquids may be used as the polar cores of microemulsions. IL microemulsions are significant in that they generate hydrophobic or hydrophilic nanodomains with nanostructured surfactant assemblies. Therefore, microregions can be constructed as either reaction or extraction media in microheterogeneous systems. Microemulsions with ILs as polar cores dispersed in an oil-continuous phase may be applied in various fields given the unique features of ILs and microemulsions [6, 23–25]. Intensive investigation should be conducted into the microstructure and the structural transitions of novel IL microemulsions. In addition, ultraviolet–visible light (UV–Vis) spectroscopy can be employed to detect the polar core of IL microemulsions. If a hydrophilic IL, such as $bmimBF_4$, is substituted for water and forms an IL/O non-aqueous microemulsion in which IL is conductive and oil is an insulating medium, then the system accords with the theoretical description of percolation theory. Thus, the different microregions of IL microemulsions can be determined via conductivity.

In the current study, the phase behavior of an ethylammonium nitrate (EAN)/TX-100/cyclohexane ternary microemulsion system is investigated, to our knowledge, EAN is firstly used with TX-100 to form IL microemulsions. Three microregions, namely, IL/O, bicontinuous, and oil-in-IL (O/IL), are differentiated via electrical conductivity. The micropolarity of IL/O microemulsions is investigated further through UV–Vis spectroscopy with methyl orange (MO) as a probe, and the conformational structure of this microemulsion is determined via Fourier transform infrared (FTIR) spectroscopy. Moreover, ionic salts and biochemical reagent could be solubilized into IL/O microemulsion droplets, indicating that IL/O microemulsions have potential application in the production of metallic or semiconductor nanomaterials, and in biological extractions.

2 Experimental

2.1 Materials

TX-100 with a purity of 98.7% was purchased from Jinan Pengyuan Biological Technology Co., Ltd. Cyclohexane (CR grade) was obtained from the Beijing Chemical Factory. All chemicals were used directly as received.

2.2 Synthesis of RTIL and Ethylammonium nitrate

Ethylammonium nitrate (EAN) was synthesized as described by Evans et al. [26], that is, through the slow addition of $\sim$3 M nitric acid to an ethylamine solution (CR grade) while this solution was stirred and cooled in an ice bath. Most of the water was removed with a rotary evaporator, and the final traces of water were extracted with a lyophilizer (Martinchrister ALPHA 1-2). The resultant product was stored in a vacuum desiccator; the melting point of this product was approximately 286 K, and its density at room temperature was roughly $1.2\ g \cdot mL^{-1}$. These properties agreed with those reported previously [22]. Residual water content was determined to be approximately 0.6 wt% as per Karl Fischer titration findings. Proton nuclear magnetic resonance (^1H NMR) and FTIR results confirmed that the resultant product is EAN: ^1H NMR: α-H (2H, 3.10 ppm), ω-H (3H, 1.32 ppm), and ^{13}C NMR: α-C (34.56 ppm), ω-C (11.36 ppm); IR (Liquid membrane, cm^{-1}), 3,066.75 (υ_{N-H}), 1,358.13 (υ_{N-O}).

The structure of EAN is displayed in Fig. 1; the spectra of EAN are shown in Figs. 2 and 3.

3 Characterization

The phase behavior of the system was determined by direct observation. A low-frequency conductivity meter (Model DDSJ-308A, Shanghai Cany Precision Instrument Co., Ltd.) was used to measure the conductivities at 25 °C. FTIR spectra were recorded in KBr pellets with $2\ cm^{-1}$ resolution using a Bruker VERTEX 70 spectrometer. UV–Vis spectra were measured at approximately 25 °C with a U-4100 (HITACHI, Japan) UV–Vis spectrometer.

4 Results and Discussion

4.1 Phase Diagram

Figure 4 shows the phase behavior of an EAN/TX-100/cyclohexane microemulsions system at (25.0 ± 0.1) °C. A continuous single-phase region extends from the IL corner to the cyclohexane corner, and the residual region marked “Two phases” is a two-phase region, i. e., a microemulsion equilibrium with an excess EAN or cyclohexane phase. In the single-phase region, an IL/O microemulsion is formed at low IL concentrations, and a gradual structural transition occurs in the microemulsion when IL concentration increases. Thus, structures may transition from IL/O microdroplets to bicontinuous structures and finally to O/IL microdroplets. Therefore, the microstructure transition of a single-phase region can be studied through specific characterization.

4.2 Electrical Conductivity Measurements and Microregions of IL Microemulsions

Microemulsions are thermodynamically stable mixtures. Single-phase microemulsions exhibit various microstructures; ordered microstructures, such as oil-in-water or W/O microdroplets, can form traditional microemulsions with high water or oil content. These microstructures are similar to the structure of micelles with large microemulsion droplets. A bicontinuous microstructure, such as a network of water tubes in an oil matrix or a network of oil tubes in a water matrix, has been detected in the intermediate regions. Different microemulsion regions can be studied via conductivity measurement based on percolation theory.

Conductivity measurement is a simple technique that is commonly used to investigate microstructures and structural changes according to the percolation theory. This method has generally been employed to determine microemulsion microstructures. A static percolation model has been developed to describe the percolation mechanism in traditional microemulsions; this mechanism attributes percolation to the generation of a bicontinuous water structure. The open water channel is presumably responsible for electrical conduction [27, 28]. In addition, a dynamic percolation model has been established based on the unique interactions among water droplets or micelles [28]. From this perspective, these interactions between water globules may facilitate the formation of percolation clusters.

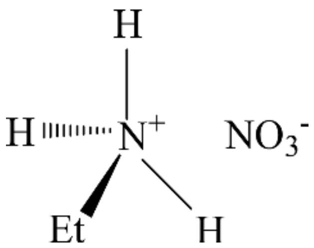

Figure 1 Molecular structure of EAN

IL is used instead of water in IL microemulsions, and the microstructures of such microemulsions can be investigated through conductivity measurement. Unlike aqueous microemulsion, IL has high conductivity; thus, IL microemulsions apply oil content in the titration phase. When the cyclohexane concentration increases, the electrical conductivity of IL microemulsions changes in three stages. As shown in Fig. 5, first, an initial nonlinear increase is observed with the addition of cyclohexane because TX-100 forms a membrane at the interface of cyclohexane and EAN; thus, an O/IL microemulsion is produced. The subsequent decrease is attributed to the formation of a bicontinuous structure as a result of the structure transition in clustered inverse microdroplets. This transition is ascribed to the progressive growth and interconnection of the oil in IL microdomains. The final decrease with increasing cyclohexane content corresponds to the appearance of an oil-continuous, microemulsion-type media. That is, an IL/O microemulsion forms with high oil content. Consequently, we can confirm the microregions of IL microemulsions by varying the weight ratio of EAN:TX-100.

4.3 UV–Vis Spectroscopy

Methyl orange (MO) molecules are preferentially located in the polar outer shell of TX-100 aqueous microemulsions. This shell is composed of oxyethylene (OE) chains [29]. Therefore, MO can be used to probe the environmental po-

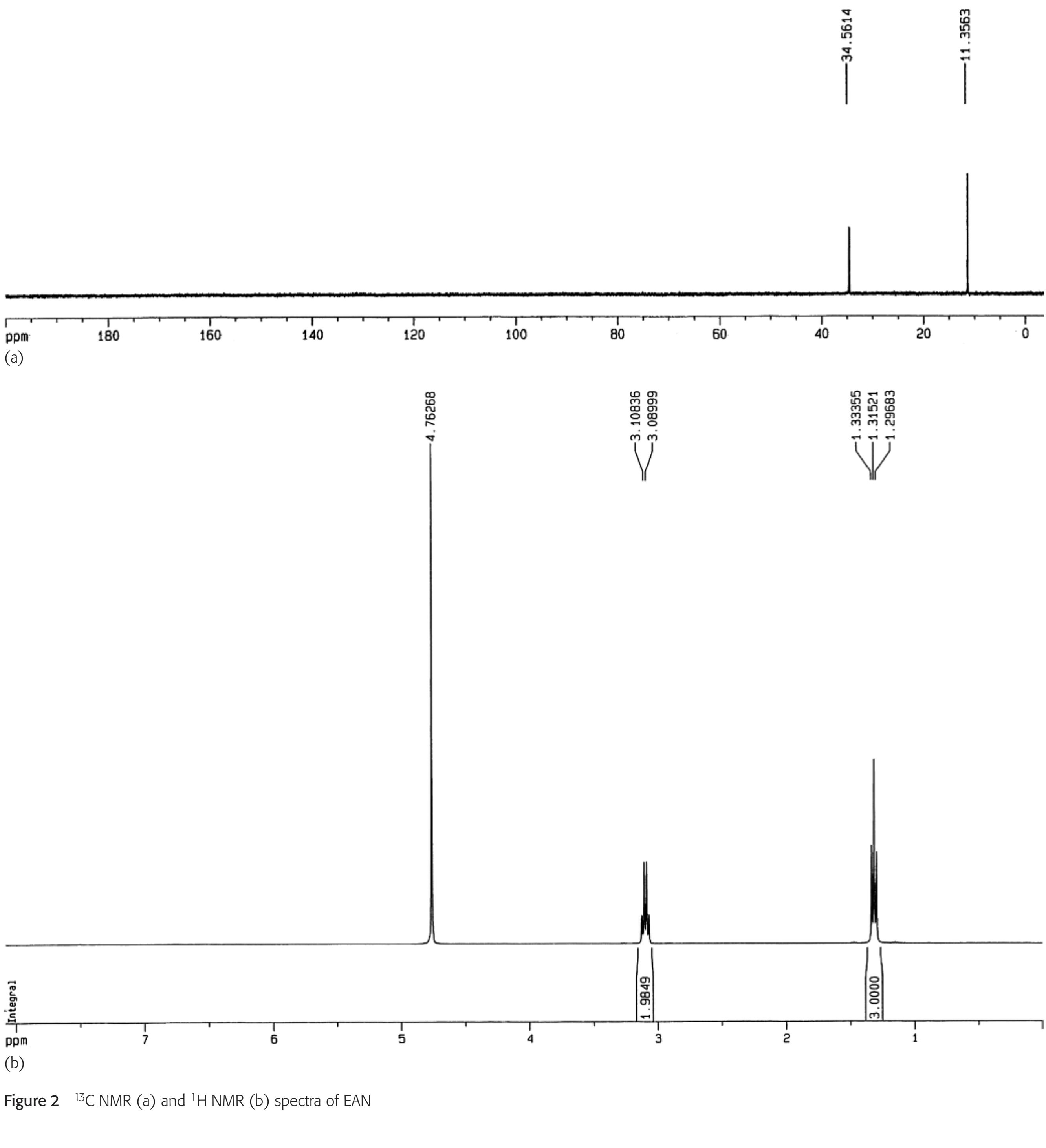

Figure 2 ^{13}C NMR (a) and ^{1}H NMR (b) spectra of EAN

larity of EAN-in-cyclohexane reverse microemulsions. The interior polarity of this microemulsion can be reflected by the absorption maximum (λ_{max}) of the UV–Vis spectroscopy data, and the absorption peak red shifts when the polarity of the environment in which the MO is located increases.

To distinguish the preferential locations of the probe used, probes are selected based on their insolubility in cyclohexane and their favorable solubility in TX-100 and EAN. To elucidate the polarity of the IL microemulsion microenvironment, the UV–Vis spectrum of the MO in TX-100/cyclohexane reverse micelles is studied first because MO molecules are preferentially located in the polar outer shell that is composed of OE chains. Thus, MO is solubilized in the inner core of TX-100/cyclohexane reverse micelles. Figure 6 shows the UV–Vis spectrum of the MO in TX-100/cyclohexane reverse micelles at different surfactant concentrations. The MO band intensity increases, but the maximum adsorption of this band (λ_{max} = 415 nm, spectra a–c) remains independent of surfactant concentration. This finding indicates that the micropolarity of TX-100/cyclohexane reverse micelles is lower than that of pure TX-100 (λ_{max} = 422 nm, spectrum d) and that of pure EAN (λ_{max} = 438 nm, spectrum e). The probe is located in a more nonpolar environment in this case than in TX-100; this outcome may demonstrate that cyclohexane penetrates the palisade layer of TX-100 during reverse micelle formation. For comparison, EAN is added to TX-100/cyclohexane reverse micelle aggregates to form EAN-in-cyclohexane microemulsions. The adsorption spectra of MO in this microemulsion are shown in Fig. 7. The maximum adsorption of MO at R = 0.87, 0.99, 1.23, 1.36, 1.50, 1.63 (R = [EAN]/[TX-100]) is 421, 423, 424, 424, 424, and 426 nm, respectively. The maximum adsorption in microemulsions is lower than that in pure EAN and higher than that in pure TX-100, except when R = 0.87. Therefore, IL pools begin to form when the EAN content in microemulsions increases. EAN penetrates into the interfacial membrane; as a result, the micropolarity of the area in which MO is located increases gradually.

4.4 FTIR Spectroscopy

Microstructure transition is further examined by FTIR spectroscopy. This method has been used extensively to determine the state and structure of solubilized water in reverse microemulsions. The water in traditional microemulsions exhibits behavior that differs significantly from that

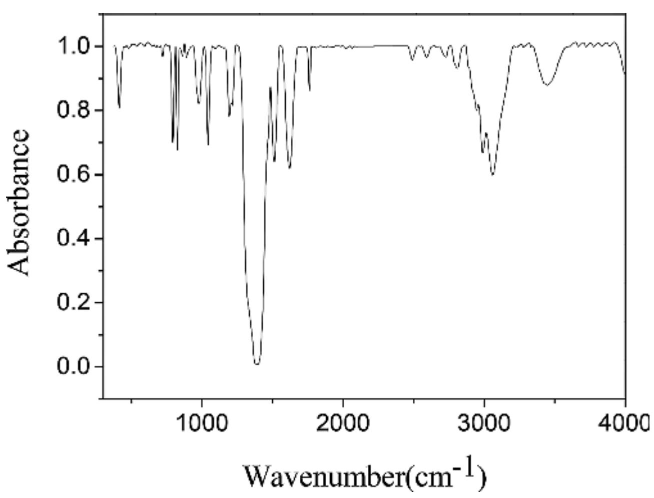

Figure 3 FTIR spectrum of EAN

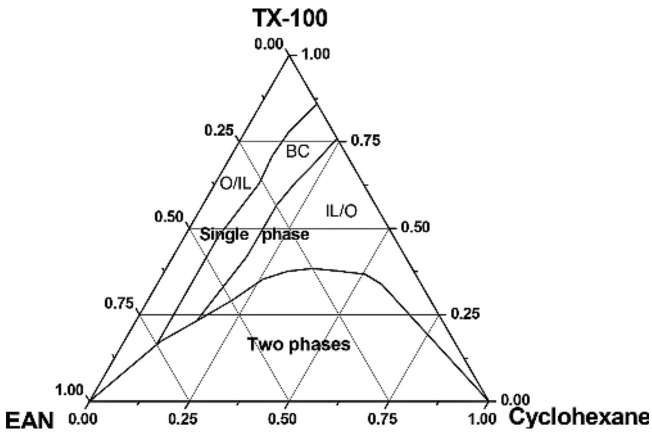

Figure 4 Phase diagram of the EAN/TX-100/cyclohexane microemulsion system at (25.0 ± 0.1) °C

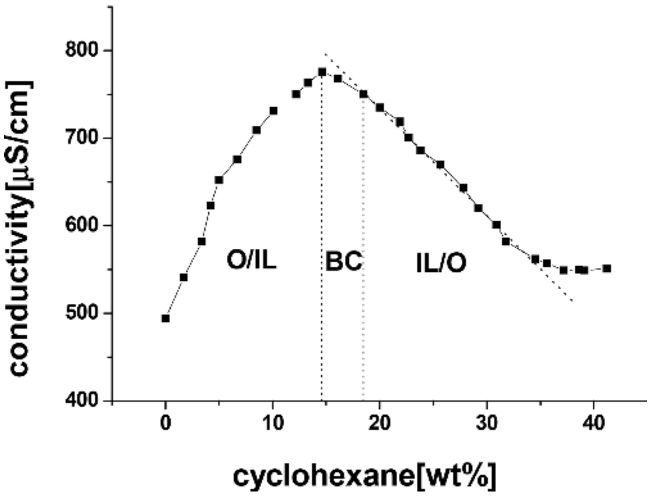

Figure 5 Electroconductivity curve of IL microemulsions versus cyclohexane content changes at 25 °C; EAN:TX-100 (wt%) = 3 : 7

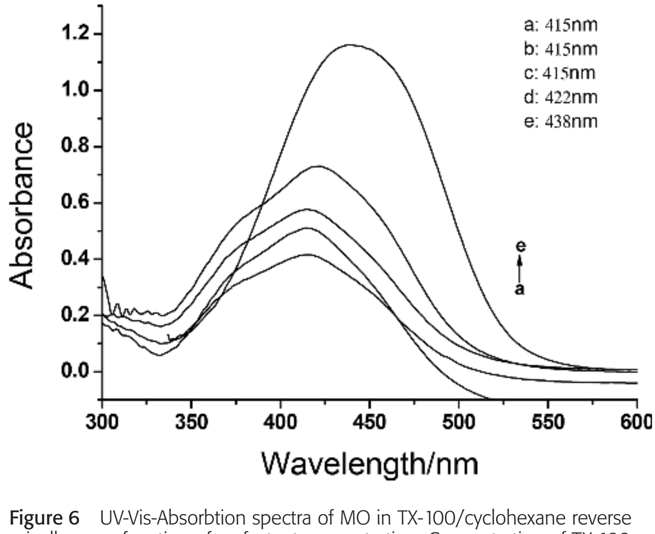

Figure 6 UV-Vis-Absorbtion spectra of MO in TX-100/cyclohexane reverse micelles as a function of surfactant concentration. Concentration of TX-100 (M): a) 0.32, b) 0.39, c) 0.47, d) pure TX-100, and e) EAN. To enhance the distinguishability of the spectra, different probe concentrations (M) were incorporated into the solutions. For (a) – (e): 2.5×10^{-5}, 3.125×10^{-5}, 3.75×10^{-5}, 5×10^{-5}, and 5×10^{-5}

of bulk water. The unusual behavior of the water in reverse microemulsions is attributed to its strong interaction with the hydrophilic groups of the surfactant molecule and the disruption of the three-dimensional hydrogen bonded network. The hydration affinity of surfactant head groups can be regarded as the driving force in the formation of microemulsion aggregates [30]. In aqueous microemulsions with the nonionic surfactant TX-100, an increase in water content induces the generation of an extended network of OE–water and water–water hydrogen bonds. This network stabilizes this conformation.

Figure 8 depicts the FTIR spectra of EAN (a), TX-100 (b), and IL/O microemulsions when R = 0 (c), 1.23 (d), 1.36 (e). The bands at 3058.03 cm^{-1} and 1395.22 cm^{-1} in Fig. 8(a) are clearly visible; these bands are attributed to the –NH and –NO stretching vibrations. In IL microemulsions, the band at 3448 cm^{-1} is almost covered by TX-100. The –NO stretching of EAN is observed at 1395.22 cm^{-1} and gradually shifts to 1378.04 cm^{-1} and 1376.61 cm^{-1} when R = 1.23 and 1.36, respectively. The –OH stretching of the TX-100 terminal hydroxyl group is at 3517 cm^{-1} when R = 0 and gradually shifts to 3450 cm^{-1} when R increases (up to R = 1.36). These findings may be attributed to the fact that in pure TX-100 or TX-100/cyclohexane reverse micelles, the terminal hydroxyl of TX-100 is hydrogen-bonded with the oxygen atoms of OE units or with the terminal hydroxyl groups of other TX-100 molecules. Upon adding EAN, however, the electronegative oxygen atoms of the OE units repel the electronegative nitrate group and strengthen the hydrogen-bond interaction in the process. As a result, an –OH stretching band is observed in the low-frequency region of the IR spectrum.

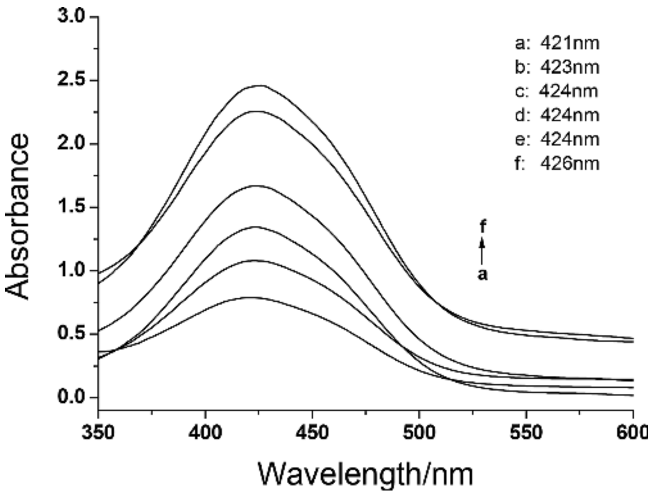

Figure 7 Absorbance spectra of MO in EAN-in-cyclohexane microemulsions as a function of EAN content when R = 0.87 (a), 0.99 (b), 1.23 (c), 1.36 (d), 1.50 (e), 1.63 (f). Probe concentrations (M) from (a)–(f) are as follows: 1.875×10^{-5}, 2.5×10^{-5}, 3.25×10^{-5}, 3.75×10^{-5}, 4.375×10^{-5}, 5×10^{-5}

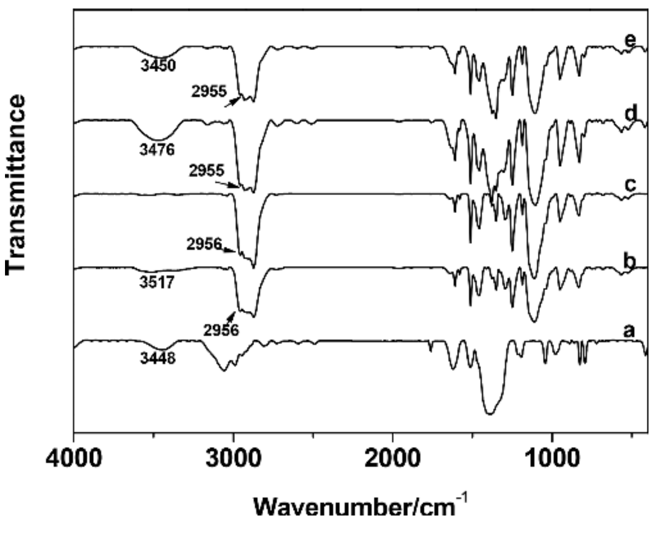

Figure 8 FTIR spectra of EAN/TX-100/cyclohexane microemulsions as a function of EAN content. Pure EAN (a), pure TX-100 (b), R = 0 (c), 1.23 (d), 1.36 (e)

5 Conclusions

A microemulsion system composed of IL EAN, nonionic surfactant TX-100, and cyclohexane was prepared in this study, and the phase behavior of this three-component system was determined. To determine the regions of IL/O microemulsion, the bicontinuous region, and the O/IL microemulsions, the transitions in the three types of single-phase microemulsions were detected via electroconductivity. The UV–Vis absorption spectra of methyl orange (MO) were obtained to study the micropolarities of EAN in cyclohexane (IL/O) microemulsions. The results showed that polarity was almost constant when the TX-100 concentration increased in reverse micelles. With the formation of IL pools, however, the polarity of the IL pool increased with EAN concentration. Nonetheless, the polarity in this case remained lower than that of bulk EAN. We therefore demonstrated that EAN penetrates the interfacial membrane. The FTIR spectra suggested that the –OH stretching of the TX-100 terminal hydroxyl group shifted to a low-frequency region with EAN addition, thus indicating that the electronegative oxygen atoms of OE units repel the electronegative nitrate group. These results showed that IL/O microemulsions may be used in nanomaterial processing, enzyme catalysis, and in other applications as reaction and extraction media.

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