Rheological Behavior of Aqueous Solutions of An Ionic Liquid As A Surfactant

ALI PING, PEIPEI GENG, JUNHONG ZHANG, JIE LIU, DEZHI SUN, XUDONG ZHANG, QINGXIA LI, JIFENG LIU, and XILIAN WEI*
Shandong Provincial Key Laboratory of Chemical Energy Storage and Novel Cell Technology, Liaoaocheng University, Liaoaocheng, Shandong, P. R. China

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The rheological properties of aqueous solutions of an ionic liquid as a surfactant, 1-tetradecyl-3-methylimidazolium bromide (C_{14}mimBr), in the presence of sodium salicylate (NaSal), have been studied by rheological measurements. For these C_{14}mimBr/NaSal systems, zero-shear viscosity as a function of NaSal concentration shows the maxima behavior. The effect of the concentration ratio, C_{NaSal}/C_{C_{14}mimBr}, on the maximum was determined, and the scaling relations were obtained. Network structures could be formed in the aqueous C_{14}mimBr solutions containing NaSal. 

**Keywords:** Ionic liquid, Sodium salicylate, Rheological property, Wormlike micelles, $^1$H NMR

**Introduction**

In recent years, ionic liquids (ILs) have attracted increasing attention due to the rapid development of green chemistry (1–8). ILs are a class of organic molten electrolytes whose physical and chemical properties can be tailored by judicious selection of cation, anion, and substituent. They have specific properties such as negligible vapor pressures, high ion conductivity, outstanding catalytic properties, nonflammability, and stability at quite high temperatures, up to 300°C or higher.

Long-chained 1-alkyl-3-methylimidazolium salts (C_{8-16}mim-X) are typical ILs. Because of the close resemblance of the alkyls in the ILs’ molecules to the long hydrocarbon chains of conventional surfactant molecules, they have been extensively studied in the field of colloid and interface science. The aggregates such as micelles (9–14), lyotropic liquid-crystalline (15–17), and microemulsions (18) formed in aqueous solutions and other solvents have been reported by many scholars. Recently, some studies on worm-like micelles formed by long-chained imidazolium surfactant-like ILs have been reported, for instance, salt-induced viscoelastic wormlike micelles formed in surface active ionic liquid aqueous solution studied by Zheng and co-workers (19). In the present paper, rheological methods and $^1$H NMR spectra were employed to investigate the formation and viscoelastic properties of wormlike micelles of a surface active IL, C_{14}mimBr in aqueous solution in the presence of sodium salicylate (NaSal). The aim is to understand the effect of the concentration change of C_{14}mimBr/NaSal on the rheological properties of wormlike micelles and to provide more useful information for their practical applications.

**Experimental**

**Materials**

The IL, 1-tetradecyl-3-methylimidazolium bromide (C_{14}mimBr), was prepared and purified according to the procedure reported by Dupont et al. (20). The 1-methylimidazole and an excess molar amount of appropriate alkyl bromide were dissolved in dichloromethane, and the mixture was stirred at 75–80°C for 48 h. The dichloromethane was then removed by the use of rotary evaporator under reduced pressure. The product was purified by recrystallization from ethyl acetate at least five times and then dried under vacuum for 1 day. The purity of the product was ascertained by $^1$HNMR spectrum in CDCl$_3$. Sodium salicylate (NaSal) (Shanghai Experimental Reagent Co., Ltd.) was analytical grade reagent and was used without further purification. The water used in solution preparation was redistilled from alkaline potassium permanganate, which insures that the surface tension of water is 72.7 mN m$^{-1}$ at 20°C.

**Apparatus and Experimental Procedure**

**Rheological Measurements**

Samples were prepared at the given molality of C_{14}mimBr/NaSal, and then they were homogenized by a vortex mixer at
50°C and then they were stored in a water bath at 25°C for at least 1 week to ensure equilibration before performing measurements. All the samples were centrifuged to remove suspended air bubbles before being tested. Rheological measurements were performed on a stress controlled rheometer (AR2000ex, TA instruments, USA) with cone-plate geometry. The cone is made of standard ETC steel with a cone diameter of 20 mm and cone angle of 2°. The gap between the center of the cone and plate is 50 μm. The measuring unit was equipped with a temperature controlling unit (Peltier plate) providing rapid change of the temperature and giving accurate temperature value (uncertainty: ±0.05°C) over an extended time in this work. Frequency sweep measurements were performed at a given stress σ₀ (chosen in the linear domain where the amplitude of the deformations is very low) in the frequency (ω) region varying from 0.01 to 600 rad s⁻¹, the steady measurements in the shear rate (γ) region from 0.01 to 1000 s⁻¹. Each measurement was repeated twice to check the reproducibility (better than 5%). Oscillatory shear rheology of viscoelastic micelle systems at low shear frequency generally follows Maxwell fluid behavior with a single stress relaxation time, τ_R. The storage modulus, G′, the loss modulus, G″, and the complex viscosity, |η*(ω)| are given by the following relations (21–23):

\[
G' = \frac{(\omega \tau_R)^3}{1 + (\omega \tau_R)^2} G'_\infty
\]

\[
G'' = \frac{\omega \tau_R}{1 + (\omega \tau_R)^2} G'_\infty
\]

\[
|\eta*(\omega)| = \left(\frac{G'^2 + G''^2}{\omega}\right)^{\frac{1}{2}}
\]

where τ_R is the relaxation time estimated from ω_c⁻¹, and ω_c is the frequency at which two moduli are equal (16). At high ω, the G’ attains a limiting value called a plateau modulus, G'_∞. If the G’ does not give a constant limiting value in some surfactant systems, the value of G'_∞ may be estimated from the modulus value at ω_c, using the relation G'_∞ = 2G'_max, where G'_max is the viscosity modulus at shear frequency ω_c. The η₀ is the zero-shear-rate viscosity.

**1H NMR Measurements**

The 1H NMR spectra were obtained with an MP - 400 nuclear magnetic resonance spectrometer (Varian Company, USA) at the proton resonance frequency 400.15 MHz. The spectra were all determined in deuterium oxide (containing TMSP as an internal reference).

**Results and Discussion**

**Steady-State Viscosity**

Transparent, gel-like solutions were formed in all determined samples. The steady shear rate viscosities of the aqueous solutions of C₁₄mimBr in the existence of NaSal were determined, at C₁₄mimBr concentrations 80, 90, and 100 mmol·kg⁻¹ (50, 70, and 150 mmol·kg⁻¹ see ESI 1) and within the range of NaSal concentration from 40 mmol·kg⁻¹ to 75 mmol·kg⁻¹ (Fig. 1). It can be seen from the curves in Fig. 1 that all the sample solutions show similar behaviors irrespective of C₁₄mimBr or NaSal concentrations. At low shear rate, the shear viscosity is independent of shear rate and a viscosity plateau was observed, showing a behavior of typical Newtonian fluid. By extrapolating the plateau values to zero shear rate, the zero-shear viscosity (η₀) can be directly obtained according to the Crareau model. With increasing shear rate, the solution viscosity exhibits shear thinning behavior above the critical shear rate γ_c (at which shear thinning appears), which has been taken as evidence for the formation of long micelles or network structure (24, 25). Variation of η₀ versus the NaSal concentration is shown in Fig. 2.

It has been observed that with increasing concentration of NaSal, the η₀ of the mixed solutions increases, and reached a maximum then the viscosity drops off drastically to a plateau region for each surfactant concentration, indicating the one-dimensional growth of the micelles and the more structured system (25). This phenomenon is expected and is similar to the behavior of some conventional ionic surfactant/salt aqueous systems (26-30). Significantly, the viscosity maximums appear at low NaSal contents, the peak occurs at a NaSal/C₁₄mimBr mole ratio of about 0.6 where there is roughly one NaSal molecule for about two C₁₄mimBr molecules in the micelle. Thus, the peak does not reflect an optimal extent of charge neutralization, indicating the longest micelle or the strongest network structure is formed at this ratio. This phenomenon is similar to that reported by Zheng et al (19), that is, the behavior of CsTAB/NaSal aqueous systems (27) and some conventional ionic surfactant/salt aqueous systems (28-30). However, the difference is that the concentration of additive salt is lower than that in other systems.

It is well known that at a constant surfactant concentration, additive salt screens the electrostatic repulsions among the polar head groups, resulting in micellar growth. Aromatic counterions such as Sal⁻ can be strongly adsorbed at the hydrophilic–hydrophobic interface and reduce the curvature of the micelles, effectively leading to an increase in the viscosity of the solutions. The rodlike micelles grow further in size with increasing NaSal concentration, they become flexible and can curve freely; therefore, the micelles become long and, accordingly, the viscosity rapidly increases until a maximum. At the peak, a possible electrostatic interaction between molecules is shown in Fig. 3 according to the molar ratio of NaSal/C₁₄mimBr about 0.6, at which the most stable and optimal structure might be formed.

In order to further elucidate the interaction between the cation of C₁₄mimBr and the counterion, 1H NMR spectra have been used to investigate the position of salicylate counterions existing in the micelles. ESI 2 show the 1H NMR signals for the C₁₄mimBr-NaSal/D₂O systems at the concentration of 100 mmol·kg⁻¹ C₁₄mimBr. The bottom spectrum is the 1H NMR result of 80 mmol·kg⁻¹ NaSal in D₂O and the second one is that of 100 mmol·kg⁻¹ pure C₁₄mimBr. The other curves are the spectra of 100 mmol·kg⁻¹ C₁₄mimBr in D₂O coexisting with different relative amount of NaSal. As the concentration of
Fig. 1. Curves of apparent viscosity ($\eta$) versus shear rate ($\dot{\gamma}$) as a function of NaSal concentration (as denoted) for $C_{14}\text{mimBr}$ solution at different concentrations (a) 80 mmol kg$^{-1}$, (b) 90 mmol kg$^{-1}$ and (c) 100 mmol kg$^{-1}$ at 25°C, respectively.

Fig. 2. Zero-shear viscosity of $C_{14}\text{mimBr}$/NaSal solutions as a function of NaSal concentration for surfactant concentrations of 50, 70, 80, 90, 100, and 150 mmol kg$^{-1}$ ($t = 25°C$).

$C_{14}\text{mimBr}$ is much higher than the cmc ($2.6 \times 10^{-3}$ mmol kg$^{-1}$), the values of observed chemical shift, $\delta$, can be considered by comparing them with those of the micellized. Table 1 shows the chemical shift changes of all proton resonances. With increasing NaSal concentration, the chemical shifts of the 3, 5 H' and 20 H signals move slightly toward down-field relative to those of the protons in the free NaSal solution and $C_{14}\text{mimBr}$ ones, while those of 4H' and 6H' hardly shifted. However, the signals of 4H~8H in $C_{14}\text{mimBr}$ molecule shift to up-field relative to the pure $C_{14}\text{mimBr}$ solution. The changes in chemical shifts of 4H and 5H are the maximal among those of protons in all function group, the next are those of 7H and 8H. These facts and the aforementioned experimental results show that the possible connection between $C_{14}\text{mimBr}$ and NaSal molecules may be primarily caused by an ion pair (see Fig. 3). On the other hand, hydrogen bonds can also form between some Sal$^-$ and Br$^-$ ions in solution. Both factors are conducive to the mixed systems to form longer micelles and pseudo-network structure. Thus, the number of ion pairs between $C_{14}\text{mimBr}$ and NaSal gradually increases with increasing the concentration of NaSal until the appearance of maximum viscosity (the optimal structure formation). After the maximum viscosity, hydrogen bonds can form between the hydroxyl group of NaSal molecule and Br$^-$ in the solution with increasing NaSal concentration, the optimal ion pair structure are partially broken, resulting in rapidly reducing the viscosity of the system until an appropriate concentration where the values of $\eta_0$ and $|\eta|^*$ remain constant, suggesting that the micellar shape and size will no longer change. From the absolute value of the zero-shear viscosity, we judge that the micelles in the solution are short rod-like in size. The three-dimensional pseudo-network may be formed over long distances by the pseudo-linkages between short rod-like micelles; therefore, the solutions show low viscosity.

The way NaSal concentration varies with surfactant concentration at the viscosity maximum is shown in Fig. 4. In the log$C_{C_{14}\text{mimBr}}$—log$C_{\text{NaSal}}$ plot, we observed a single straight line between these two concentrations. Similar phenomena have also been observed from CPyCl/NaSal and CTAB/NaSal solutions (31, 32). The relationship obtained is:

$$\log(C_{C_{14}\text{mimBr}}) = 16.88 + 0.42C_{\text{NaSal}}$$ (4)
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![Fig. 3. Possible links between NaSal and C_{14}mimBr molecules at the peak.]

**TABLE 1** $^1$H NMR chemical shifts ($\delta$, ppm) of C_{14}mimBr (100 mmol·kg$^{-1}$) in the existence of different concentrations of NaSal at 25°C

| NaSal mmol·kg$^{-1}$ | NaSal chemical shifts (δ, ppm) | C_{14}mimBr chemical shifts (δ, ppm) |
|----------------------|--------------------------------|-------------------------------------|
| 0                    | 4H$^*$ 7.464 3',5'H 6H$^*$ 7.816 | 4H 7.611 5H 7.787 6H 3.969 4.294 1.925 0.841 |
| 70                   | 7.448 7.123 7.787 | 6.695 6.715 3.897 3.897 1.422 1.031 |
| 80                   | 7.427 7.117 7.774 | 6.689 6.709 3.885 3.885 1.432 1.037 |
| 90                   | 7.398 7.109 7.759 | 6.680 6.699 3.867 3.867 1.443 1.046 |

![Fig. 4. Molarity of the surfactant corresponding to maximum viscosity of the solution as a function of the salt concentration, C_{NaSal}, (t = 25°C).](image)

where $C_{C_{14}mimBr}$ and $C_{NaSal}$ are, respectively, the molarities of C_{14}mimBr and NaSal.

**Oscillatory Measurements**

Oscillatory measurements for each sample were carried out in suitable oscillatory stress where the elasticity modulus $G'$ and viscous modulus $G''$ is independent of the applied stress. The typical results of the storage moduli/loss moduli as functions of the stress ($\sigma$) for 90 mmol·kg$^{-1}$ C_{14}mimBr/45 mmol·kg$^{-1}$, 55 mmol·kg$^{-1}$, and 65 mmol·kg$^{-1}$ NaSal solutions are shown in Fig. 5. It can be seen that the values of $G'$ and $G''$ remain constant until the stress reaches the critical stress $\sigma_c$, a shear-thinning behavior took place at above $\sigma_c$, which is a characteristic property of wormlike micelles (33). The systems were more viscous than elastic at all the applied stresses. Most of the samples have

![Fig. 5. The storage moduli and loss moduli as functions of the applied stress at a constant angular frequency (6.28 rad/s) for 90 mmol·kg$^{-1}$ C_{14}mimBr/45 mmol·kg$^{-1}$, 55 mmol·kg$^{-1}$, and 65 mmol·kg$^{-1}$ NaSal solution at 25°C.](image)
the linear viscoelasticity up to about 10 Pa. Therefore, in subsequent frequency sweep measurements we chose a stress value of 1.0 Pa. It can also be observed that both the storage modulus ($G'$) and the loss modulus ($G''$) are independent of stress over the entire measurement range (with $G''$ dominating $G'$), indicating that these samples are viscous micellar solution.

Fig. 6a shows the dynamic rheological spectra of the 80 mmol·kg⁻¹ $C_{14}mimBr$ solutions as a representative system at different NaSal concentrations. The curves show the overlap of $G'$ and $G''$ as the frequency increasing, indicating that the materials are more viscous than elastic at low frequencies and more elastic than viscous at high frequencies. The crossing point of $\omega$-$G'$ and $\omega$-$G''$ curves move toward the higher frequency region, indicating lower relaxation time, and thus poor viscoelastic behavior. The $G'$ and $G''$ crossover occurs in the high frequency region, which corresponds to a fast relaxation process (34). An earlier report (35) mentioned that the higher the angular frequency at which $G'$ and $G''$ cross, the more difficult the formation of network structure. As frequency increases, both $G'$ and $G''$ increase and cross at a frequency $\omega_{C}$, which corresponds to the relaxation time being fitted by a simple Maxwell model. $G'$ appeared at maximum in $\omega_{C}$, then decreased.

Sometimes, it is difficult to determine how “good” a Maxwell model being fitted by the data from the plots of $G'$ and $G''$ vs. $\omega$. A Cole–Cole plot (plot of $G''$ as a function of $G'$) provides a better picture of how well the data corresponds to a single relaxation time fitting to Maxwell model, which reveals the semicircle characteristic of a Maxwell fluid. It can be expressed as Eq. (5) according to Eqs. (1)–(3):

$$G''^2 + \left(G' - \frac{G_0}{2}\right)^2 = \left(\frac{G_0}{2}\right)^2$$

Fig. 6b shows a Cole–Cole plot from the data presented in Fig. 6a; the solid lines correspond to Maxwellian evolution. The experimental points are in relatively good accordance with Eqs. (1) and (2), except for $\eta_0 \propto \omega^{3.18}$ at higher angular frequencies, where we can see a departure in the measured evolution, corresponding to the contribution of the breathing and Rouse modes, which is an ordinary characteristics appearing in many wormlike micellar systems (27, 28, 33). The deviation of $G''$ from the model at the high $\omega$ region is another characteristic of wormlike micelles (36), which corresponded to the fact that wormlike micelles are in a dynamic equilibrium and there are rapid breaking and recombination processes (37). An analogous trend has been observed for some other ionic surfactant/salt viscoelastic solutions (28, 30, 31). This can also be confirmed using the Cox–Merz empirical rule (38), that is, for a solution with wormlike micelles and network structures, the steady-shear viscosity ($\eta$), and the magnitude of the complex viscosity ($|\eta^*|$) superimpose closely at equivalent values of the shear rate ($\dot{\gamma}$/s⁻¹) and angular frequency ($\omega$/rad s⁻¹). If the structure can survive with small oscillatory deformations, it may be ruptured by a large deformation. The $|\eta^*|$ is significantly larger than $\eta$ at high frequency and so this solution tends to depart from the Cox–Merz superposition at high frequency or high shear rate. The shear rate as a function of $\eta$ and the frequency versus $|\eta^*|$ are shown in Fig. 7 for 100 mmol·kg⁻¹ $C_{14}mimBr$/65 mmol·kg⁻¹ NaSal as a representative system. It is shown that $\eta$ and $|\eta^*|$ are in good agreement at 0.1–100 shear rate or $\omega$ region, but they separate and go different ways at very high shear rate, implying that the

![Fig. 6](image1)

**Fig. 6.** Variations of $G'$ (filled symbols) and $G''$ (open symbols) with the shear frequency for aqueous solutions of $C_{14}mimBr$ (80 mmol·kg⁻¹) (a) and the corresponding Cole–Cole plots (b) with different NaSal concentrations, the NaSal concentrations are expressed in the figures at 25°C.

![Fig. 7](image2)

**Fig. 7.** Shear-rate dependence of the steady-shear viscosity and frequency dependence of the magnitude of the complex viscosity for 100 mmol·kg⁻¹ $C_{14}mimBr$/65 mmol·kg⁻¹ NaSal solution (25°C).
network structures can be formed at \( \dot{\gamma} \) or \( \omega \) region from 0.1 to 100 and destroyed with increasing shear rate. However, the constant region of \( \eta \) or \( |\eta^*| \) is wider than that of other worm-like micelles system (16, 24–38), the structure of these systems is more stable, because the short rod-like micelle is not easy to be broken.

Fig. 8 depicts a plot of the steady and complex zero shear viscosities (\( \eta_0 \) and \( |\eta^*| \)) of \( C_{14\text{mimBr}} = 100 \text{ mmol kg}^{-1} \) solution versus NaSal concentration. The values of \( |\eta^*| \) are extrapolated from the data by averaging the low-frequency data points once they reach a plateau value and is given by Eq. 3. From Fig. 8 it can also be seen that the data for \( \eta (\dot{\gamma}) \) and \( |\eta^*| \) overlap each other, indicating that all solution follow Cox–Merz empirical rule at low shear rate or \( \omega \) region.

However, when the NaSal concentration is fixed at 80 mmol·kg\(^{-1}\), the variation of zero-shear viscosity (\( \eta_0 \)) with increasing \( C_{14\text{mimBr}} \) concentration is different from that of the system with fixed \( C_{14\text{mimBr}} \) (Fig. 9, the curve of steady shear rate viscosity of the mixed aqueous solutions as a function of \( C_{14\text{mimBr}} \) concentration are shown in ESI. 3). The curve can be divided into three domains. First, at lower \( C_{14\text{mimBr}} \) concentrations (<110 mmol·kg\(^{-1}\)), \( \eta_0 \) shows little increases; second, in the middle range of \( C_{14\text{mimBr}} \) concentrations from 110 mmol·kg\(^{-1}\) to 150 mmol·kg\(^{-1}\), the viscosity increases rapidly; and, finally, \( \eta_0 \) shows decrease with \( C_{14\text{mimBr}} \) concentration increasing. This can be explained from the viewpoint of micellar growth. Generally, in the range of lower surfactant contents, an increase in the concentration of surfactant can only lead to an increase in the size and the number of micelles, and viscosity of the solution cannot show evident strengthening. When the length of micelles has a further increase as the concentration falls in the range of higher surfactant contents, they become flexible and can curve freely, becoming wormlike micelles, which can result in a rapid increase in viscosity. In other words, the formation of wormlike micelles and network structures begins at a high surfactant concentration for the studied systems. Viscosity shows less dependence on the surfactant concentration in the range of higher \( C_{14\text{mimBr}} \) contents, owing to the equilibrium between the combination and dissociation of the aggregates, and, therefore, wormlike micelles show slower growth.

**Plateau Modulus \( G'_\infty \) and Relaxation Times \( \tau_R \)**

Variation of \( G'_\infty \) and \( \tau_R \) with the NaSal concentration in the system are shown in Fig. 10a and b. It can be observed that \( G'_\infty \) increases in the investigated concentration range (Fig. 10a) and the \( \tau_R \) possesses maximum points (Fig. 10b), corresponding to the maximum values of the zero-shear viscosity. The similar results in both \( G'_\infty \) and \( \tau_R \) with addition of additive were also reported in \( C_{16\text{mimBr}}/\text{NaSal} \) (16) and other surfactant system (25, 34). As \( G'_\infty \) usually depends on the number density of the aggregates and, therefore, reflects the mesh size of the network, the increase in the plateau modulus \( G'_\infty \) corresponds to the increase in the degree of entanglements and, hence, can be regarded as an evidence of one dimensional micellar growth. As for \( \tau_R \), it can be associated with the length of the aggregate for the wormlike micellar system undergoing stress relaxation by reputation, as indicated by Maxwellian behavior. Increase in these quantities may be associated with the micellar growth leading to longer micelles and increased number of entanglements, which makes the system more viscoelastic; that is, the bigger is the \( \tau_R \) value, the slower is the diffusing of wormlike micelles and also the longer the micellar size. The maxima observed in the \( \tau_R \) curves indicate that some structural changes occur, which allows a faster stress relaxation as discussed in the steady-state viscosity section.

**Conclusions**

This paper reports viscoelastic wormlike micellar solutions formed by a surfactant active ionic liquid \( C_{14\text{mimBr}} \) with NaSal in water at various surfactant concentrations. The zero-shear viscosity of these solutions as a function of NaSal concentration shows a maximum behavior. The NaSal concentrations corresponding to the maximum point in the viscosity curves vary linearly with
surfactant concentration. The strongest network structures are formed in the mixed systems of C14mimBr with NaSal at the molar ratio about 1:0.6. The 1H NMR spectra analysis indicates that the main interaction between C14mimBr and NaSal molecules arises from the formation of ion pair. The variations τ versus the NaSal concentration also show the similar trend. When NaSal concentration is fixed, the variations of zero-shear viscosity (η0) with increasing C14mimBr concentration are different from those in the systems at fixed C14mimBr concentration, indicating that the micellar structure did not change in the investigated concentration range.

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Supplemental Material

Supplemental data for this article can be accessed on the publisher’s website.

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