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Physical Properties in Aqueous Solutions for a Series of Alkyltrimethylammonium Salicylates (C12TA-Sal through C16TA-Sal): From a View Point of Drag Reduction

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Abstract

Compounds for a series of alkyltrimethylammonium salicylates (C12TA-Sal through C16TA-Sal) were synthesized. Their physical properties in aqueous solutions were investigated by conductometry, viscometry, vortex inhibition, viscoelastic recoil and swirling decay time from a view point of drag reduction. For critical micelle concentrations (CMC) obtained for a series of compounds by conductometry, it was found that a linear relation of the form, $\log(\text{CMC}) = 4.088 - 0.305 \text{*Nc}$ (Nc: carbon number in the alkyl chain), holds. From the viscosity measurement, all the compounds showed viscosity increase above their CMC. Vortex inhibition was observed above the CMC for the compounds with the chain length longer than C13. Viscoelastic recoil was observed above the concentration of one and a half times the CMC for the compounds with alkyl chain length longer than C14.

Keywords

Drag Reduction, Vortex Inhibition, Viscoelastic Recoil, CMC

1. Introduction

Drag reduction or Toms effect is a flow phenomenon in which a reduction in turbulent friction occurs by the addition of a small amount of polymer or surfactant into a turbulent flow. The drag reduction in pump work was shown to be almost more than 60% which may provide significant savings for electric power or in energy use. Thus, much

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attention is focused in this field [1] [2] [3] [4]. Rheological properties of polymers and surfactant solutions are similar in the drag reduction. However, polymers in solution were broken by mechanical stress such as in pump, while in surfactant solution, micelles are reassemble into micelles after being degraded. Therefore, surfactants have been utilized in a district's heating and cooling systems to reduce pumping power in Japan [4] [5] [6].

As for screening of the surfactant for the drag reduction, vortex inhibition and viscoelastic recoil were used. When pure water in a beaker is stirred by a magnetic stirrer, a vortex forms and reaches the bottom of the beaker. After addition of a testing surfactant in the beaker, if the solution becomes viscoelastic, the vortex will disappear. This phenomenon is called vortex inhibition and used for the screening [7] [8]. While, when a test solution in a beaker is stirred by a magnetic stirrer and the stirrer is stopped, the solution will slows down in the direction of the swirling motion. If the solution is viscoelastic, it will stop, then, swirl in the opposite direction [9]. This phenomenon is written in some studies as recoil of bubbles [10] [11] [12], elastic recoil [13], or recoil phenomenon [14] [15]. However, as for a keyword of elastic recoil or recoil phenomenon, it has another meaning in medical term which means the rebound of the lungs after having been stretched by inhalation. Furthermore, there is another term of elastic recoil detection which refers to as forward recoil scattering in a nuclear technique in materials science to obtain elemental concentration depth profiles in thin films. Thus, avoid the confusion in the present phenomenon in the viscoelastic surfactant solution, we will use a new term of "viscoelastic recoil".

For major part of the drag reducing agents, mixtures of cationic surfactants and organic counterions have been used; especially the mixture of hexadecyltrimethylammonium bromide (cetyltrimethylammonium bromide; C16TAB, CTAB) and sodium salicylate (SalNa). Authors in the present study (Itoh and Tamano) have reported the velocity measurement in turbulent boundary layer by use the mixture of C16TAB/ SalNa [16].

Since aqueous solution of the mixture of C16TAB/SalNa is a ternary system, Gravsholt has reported the pioneering work of binary system (C16TA-Sal and water) without NaBr together with the homologous isomers of organic counterion [10]. She also has studied the properties of C16TA-Sal by using Linear Dichroism spectroscopy [17] and rheopectic behaviour [18]. Following her report, Angel *et al.* have studied the physical properties for even carbon number ATA-Sals (C10-C16) in aqueous solutions [19]. Imae *et al.* [20] [21] [22] and Hashimoto *et al.* [23] have reported the spinnability for C14TA-Sal and C16TA-Sal. Moreover, Imae *et al.* have reported the unique properties of C16TA-Sal layers adsorbed on glass beads [24]. In these studies, the origin of viscoelasticity in aqueous solution of C14TA-Sal and C16TA-Sal was ascribed to the formation of rod-like micelles. Recently, Alfaro *et al.* have reported the detailed analysis for the phase and rheological behaviour of the C16TA-Sal in the diluted, semi-diluted, and concentrated aqueous solutions together with the temperature dependence [25].

In the present study, we synthesized a series of alkyltrimethylammonium (ATA)-Salicylates (Sal), C12TA-Sal through C16TA-Sal, including odd number of the hydro-

carbon chain. Furthermore, we dealt with the physical properties of ATA-Sals in aqueous solutions from a view point of the drag reduction by using conductometry, viscometry, vortex inhibition, viscoelastic recoil, and swirling decay time.

2. Experimental Section

Materials: Compounds for a series of ATA-Sals (C12 through C16) were synthesized by an ion exchange column of IRA400J CL (Organo Co) similar to the method by Gravsholt [10] using the corresponding alkyltrimethylammonium bromide (ATAB) of C12 through C16 with Salicylic acid (Sal). The obtained compounds were recrystallized two times from acetone and small portion of ethanol. Compounds of C12TAB, C14TAB, and C16TAB were purchased from Tokyo Kasei Co. Compounds of C13TAB and C15TAB were synthesized by the coupling with the corresponding n-alkyl bromide (C13Br and C15Br) and trimethylamine, following the method of Birdi [26], respectively. The C13Br, C15Br, and Sal were purchased from Wako Pure Chemicals Co.

Purity: Purities of the ATA-Sals were checked by a portable ion meter IM-32P (TOA-DKK) for Br^- . The amounts of Br^- in all the ATA-Sals were less than 0.8 $mg \cdot L^{-1}$ of the detection limit of the ion meter.

Conductometry: Conductance measurements were taken with a conductivity meter CM-40V (TOA-DKK) using CG-7001PL with cell constant of 0.0995. A surfactant was progressively added to water in a beaker placed on a magnetic stirrer, and the conductance was measured after thorough mixing and temperature equilibrium at $25^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$.

Viscometry: Viscosity measurements were taken using a Ubbelohde viscosimeter (Shibata Scientific Technology Ltd) at 25° C \pm 0.1°C. Various sample solutions were prepared in a beaker with the unit of mmol·kg⁻¹ just before each experiment.

Vortex inhibition, Viscoelastic recoil, and Swirling decay time (SDT): Pure water (200 mL) was placed in a 200 mL glass beaker on a magnetic stirrer with 35 mm stir bar, which was kept at 1000 rpm. A surfactant was added progressively in the beaker at 25° C \pm 0.1°C. The average depth of the vortex was measured by a ruler behind the beaker. Additionally, the phenomenon of viscoelastic recoil was also observed and SDT, which was the time between stopping the stirrer and the starting the recoil, was measured.

3. Results and Discussion

3.1. Critical Micelle Concentration (CMC)

There are so many experimental methods to obtain CMC of surfactant in aqueous solution due to the difference of monomer-micelle equilibrium, such as surface tension, electric conductivity, refraction and so on [27]. Since different experimental methods may reflect the monomer-micelle transition to different extents, we confined the experimental method to the measurement of conductivity. When conductivity is plotted against concentration, two almost straight lines are obtained and the intersection is considered to be the CMC. Figure 1 shows the conductivity-concentration for C16TA-

Sal and the distinct break is regarded as the CMC. **Table 1** shows the CMC values for a series of ATA-Sals in this study together with their mother compounds of ATABs. As for the CMC values for ATABs except for C13TAB, the reported values are as follows: C12TAB, 14.2 - 15.8 mmol·L⁻¹ [27]-[35]; C14TAB, 3.60 - 3.943 mmol·L⁻¹ [27]-[33] [35] [36]; C15TAB, 1.7 mmol·L⁻¹ only obtained by fluorimetry [35]; C16TAB, 0.81 - 0.9642 mmol·L⁻¹ [27]-[33] [37]. In the present study, the CMC values obtained for ATABs were good agreement with the values in the literature except for the unit of mmol·kg⁻¹.

For C16TA-Sal, Gravsholt has reported the CMC value as 0.152 mmol·L⁻¹ at 25°C [10]. While, Angel *et al.* have reported the CMC values for C12TA-Sal, C14TA-Sal, and C16TA-Sal as 2.85, 0.625, and 0.15 mmol·L⁻¹ at 25°C, respectively [19]. Furthermore, Imae *et al.* have reported the CMC value as 0.15 [21] [24] for C16TA-Sal and Hashimoto *et al.* 0.69 for C14TA-Sal and 0.10 mmol·L⁻¹ for C16TA-Sal at 25°C [23]. In the present study, the CMC value for C12TA-Sal, C14TA-Sal, and C16TA-Sal was 2.72, 0.63, and 0.16 mmol·kg⁻¹, respectively (**Table 1**), indicating that these were in good agreement with the values reported in the literature.

As for the CMC for C13TA-Sal and C15TA-Sal, there are no values in the literature. However, Klevens has reported that, in a homologous series, the CMC values normally decrease logarithmically with the number of carbon atoms (Nc) in the chain according to the following equation, log(CMC) = A - B*Nc, where A and B are constant specific

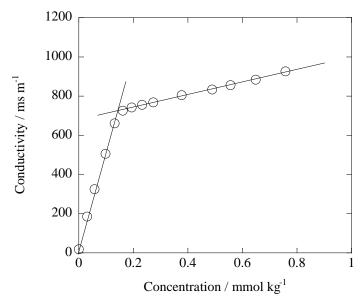


Figure 1. Concentration dependence of conductivity of C16TA-sal.

Table 1. CMC of ATAB and ATA-Sal (m·mol·kg⁻¹).

Nc	12	13	14	15	16
ATAB	15.5	7.6	3.8	1.9	0.90
ATA-Sal	2.72	1.32	0.63	0.34	0.16

to a homologous series [38]. Figure 2 shows the relations between Nc and the CMC values for both the series of ATAB and ATA-Sal: ATABs, A = 4.881, B = 0.307; ATA-Sals, A = 4.088, B = 0.305. It is found that, for both the series of ATABs and ATA-Sals, the slopes of B are almost the same and may be taken to be log 2 with sufficient accuracy [38].

Angel *et al.* have reported that the conductivity-concentration plots for the systems of hexadecylpyridinium salicylate and teteradecylpyridinium salicylate, (C16Py-Sal and C14Py-Sal), showed two breaks [19]. They have concluded that the first break was the CMC of the systems where they begin to form micelles. Additionally, the second break was assigned to the transition concentration c_t above which the systems form rod-like micelles. They have also reported no c_t values for C12TA-Sal, C14TA-Sal, and C16TA-Sal at 25°C [19]. However, Ohlendorf *et al.* have reported the CMC and c_t at various temperatures [39]. In their report, the CMC and c_t were of equal concentration at 20°C (2.54 mmol·L⁻¹) and 30°C (2.93 mmol·L⁻¹) for C12TA-Sal and at 20°C (0.583 mmol·L⁻¹) for C14TA-Sal, while at 30°C, c_t observed for C14TA-Sal (1.14 mmol·L⁻¹) and C16TA-Sal (0.474 mmol·L⁻¹). In the present study, we have observed only one break in the conductivity-concentration plots at 25°C for a series of ATA-Sals (**Figure 1** for C16TA-Sal).

In the detailed report by Alfaro *et al.* [25], there are three types of critical concentrations: cmc_1 , formation of spherical micelle; cmc_2 , the sphere-to-prolate ellipsoidal transition; mgc, formation of wormlike micelle by using the electrical conductivity, surface tension, sound velocity, and Sudan II absorbance measurements. Average values for cmc_1 and cmc_2 were 3.5×10^{-4} and 1.2×10^{-3} (wt.%) at 35° C, corresponding to 0.0083 and 0.028 mmol, respectively. Their method to prepare sample solutions was the dilution of concentrated surfactant solution. In the present study, since we used the progressive addition of the sample surfactant to a solution, it was too difficult to obtain the critical concentration values of the cmc_1 and cmc_2 in these highly dilute regions (**Figure 1**). However, Alfaro *et al.* have reported the value of mgc as 1.1×10^{-2} (wt.%), corres-

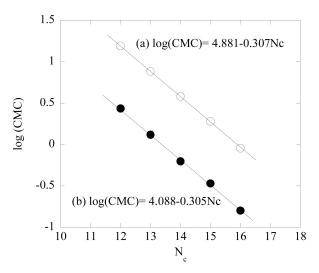


Figure 2. logCMC vs crabon number: (a) ATAB, (b) ATA-Sal.

ponding to 0.26 mmol. Especially, the mgc value from the specific electrical conductivity and Sudan II absorbance was 0.7×10^{-2} (wt.%), corresponding to 0.17 mmol, was in good agreement with the CMC values in the present study and the previous studies [10] [19] [21] [23] [24].

3.2. Viscosity

Figure 3 shows the viscosity-concentration dependence for a series of ATA-Sals. The horizontal axis is the concentration in the unit of mmol·kg⁻¹. Angel *et al.* have studied the viscosity for even number ATA-Sals (C12-C16) by measuring the complex viscosity with an oscillating viscometer and that the viscosity increased rapidly by several orders of magnitude [19]. This viscosity increase is ascribed to the length of the rods in a micelle. In the present study, it is found that the viscosity increased rapidly for C15TA-Sal and C16TA-Sal. However, it is hard to appreciate the relation between the viscosity and the CMC. Thus, **Figure 4** shows the viscosity-concentration dependence with the normalized values divided by the respective CMC (**Table 1**) in the horizontal axis. The viscosity for ATA-Sals was found to change from the respective CMC. As for the compounds of the shorter chain length of C13TA-Sal and C14TA-Sal, the viscosities were gradually increased and drastically increased for C15TA-Sal and C16TA-Sal.

3.3. Vortex Inhibition

After addition of a testing surfactant in a beaker on a magnetic stirrer, the vortex disappeared, if the solution became viscoelastic. In this study, the compound of C12TA-Sal hardly ever showed the vortex inhibition. Figure 5 shows the height of the vortex for ATA-Sals except for C12TA-Sal for various concentrations. The horizontal axis is also normalized by the respective CMC. It is found that the vortex inhibition

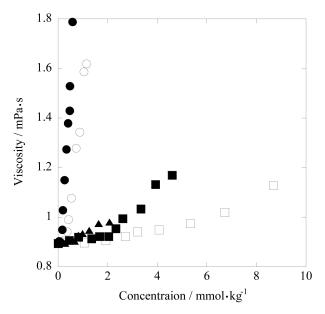


Figure 3. Concentration dependence of viscosity for ATA-Sal: C16, ●; C15, ○; C14, ♠; C13, ■; C12, □.

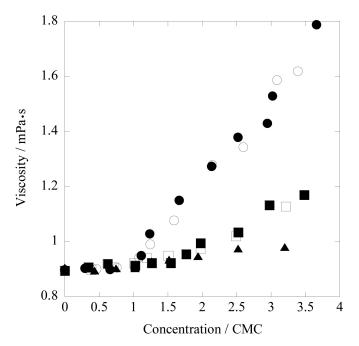


Figure 4. Concentration dependence of viscosity for ATA-Sal: C16,
•; C15,
○; C14,
•; C13,
•; C12,
□.

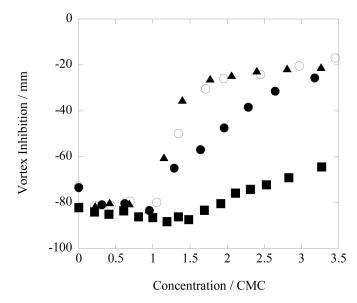


Figure 5. Vortex Inhibition for a series of ATA-Sal: C16, ●; C15, ○; C14, ▲; C13, ■.

starts from the CMC for these compounds. However, for C14TA-Sal and C15TA-Sal, the vortex disappeared just above the CMC and almost constant height at around the concentration of one and half of the CMC. On the other hand, for C16TA-Sal, the vortex gradually disappeared just starting the CMC and almost constant height at around three times the CMC (ca. $0.50 \text{ mmol}\cdot\text{kg}^{-1}$), indicating that the growth of the rods completed at this concentration starting from the CMC, not the transition concentration c_r

3.4. Viscoelastic Recoil and SDT

Viscoelasticity was detected by the method of visually observing recoil of small air bubble in a beaker after swirling was stopped [9]. Viscoelastic recoil was observed neither for C12TA-Sal nor C13TA-Sal. **Figure 6** shows the SDT for C14TA-Sal, C15TA-Sal, and C16TA-Sal with the increase in concentration. The horizontal axis is also normalized by CMC, showing that the viscoelastic recoil was observed from the concentration of one and a half the CMC for these compounds and almost constant at around twice the CMC. Since the shorter the SDT the stronger the viscoelasticity, it is found that the compounds of C15TA-Sal and C16TA-Sal has the strong viscoelasticity above the concentration of twice the CMC; 0.7 mmol·kg⁻¹ (280 ppm) for C15TA-Sal, 0.35 mmol·kg⁻¹ (150 ppm) for C16TA-Sal.

4. Conclusion

CMC for a series of ATA-Sals showed the linearity with the number of carbon atoms (N_c) in the chain according to the equation, $\log(\text{CMC}) = \text{A} - \text{B*N}_c$; A = 4.088, B = 0.305. Additionally, transition concentration (c_i) from spherical micelle to rod-like micelle was not observed for these compounds. In case of viscosity, the value for C15TA-Sal and C16TA-Sal drastically increased with increase in concentration above the CMC, while the compounds of the shorter chain length below C14 gradually increase. For the compounds with the longer alkyl chain of C14TA-Sal, C15TA-Sal, and C16TA-Sal showed the vortex inhibition starting from the CMC. These compounds showed the viscoelastic recoil and the almost constant SDTs were observed for C15TA-Sal and C16TA-Sal above the concentration of twice the CMC. Thus, it is reconfirmed that C16TA-Sal is the most suitable compound for drag reduction from the

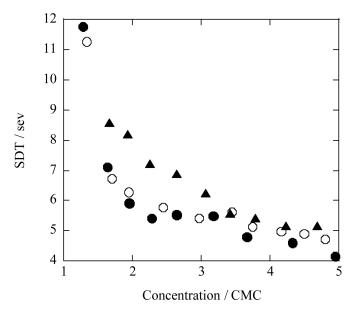


Figure 6. Swirl Decay Time for a series of ATA-Sal: C16, ●; C15, ○; C14, ▲.

view point of both the characteristic property and the concentration in diluteness.

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