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ORIGINAL CONTRIBUTION

Comparison of the effects of methyl- and chloro-substituted salicylate counterions on drag reduction and rheological behavior and micellar formation of a cationic surfactant

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Abstract We studied drag reduction, rheological behavior, and cryogenic transmission electron microscopy (cryo-TEM) imaging of aqueous solutions of a cationic surfactant, Arquad 16–50 (5 mM) (commercial cetyltrimethylammonium chloride, CTAC), with two series of isomer counterions at 5 mM: 3-, 4-, and 5-methylsalicylate, and 3-, 4-, and 5-chlorosalicylate. All these systems demonstrated viscoelastic behavior at room temperature and showed effective drag reduction in the temperature range of 20–70 °C. The additional Cl- or CH_3- group on the salicylate extended the upper effective drag reduction temperature limit 5–10 °C higher than that of sodium salicylate solution. The 4-methylsalicylate, and 3- and 5-chlorosalicylate solutions are effective drag reducers down to 5 °C, the lowest temperature tested in the flow loop. All of the methyl isomers had very similar rheological behaviors, while the chloro isomers exhibited dramatic differences. The 5-chlorosalicylate system had near zero N_1, but did show viscoelastic behavior by showing recoil after swirling and overshoot at shear startup. Cryo-TEM images show that four of the isomer systems formed threadlike micellar nanostructures, but 3-methylsalicylate aggregated into vesicles and open vesicles. The 3-chloro and 5-chlorosalicylate showed vesicles with a few spherical micelles and threadlike micelles. It is postulated that the vesicles were transformed into threadlike micelles under shear and that this structure made them effective drag reducers.

Introduction

In dilute solutions, counterion chemical structures have significant impact on the shape and size of micelles of cationic surfactants (Zana 1991), and the degree of the ion binding affects the rigidity of the threadlike micelles (Cates and Candau 1990). A fraction of the counterions is tightly bound to the micelle surface while the remainder forms a diffuse electrical double layer in the space surrounding the micelles (Clint 1992). If the bound counterion is an aromatic counterion, it inserts between the headgroups into the micelle, at a depth that depends on the counterion chemical structure. Its negative charge diffuses the positive charges on the ammonium headgroup, fostering the formation of threadlike micelles. It increases the surfactant packing parameter, $p = v/a_0 l_c$, where v is the volume of the hydrocarbon chain, a_0 is the interfacial headgroup area, and l_c is the fully extended chain length (Israelachvili 1991). Values of $1/3 < p < 1/2$ favor long cylindrically shaped micelles. Nonpenetrating counterions like halides are less effective (Magid 1998).

The effect of counterions on micelle formation was investigated by light scattering, electric birefringence, NMR, rheology, and neutron light scattering experiments for the system cetylpyridiniumsalicylate (CPySal) (Hoffmann et al. 1982). The authors reported that binding of the counterions to the headgroups reduced the mutual repulsion of the aggregates and increased the length of the micelles. At rest, the axes of the threadlike micelles were randomly oriented, but they

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strongly aligned under shear. The alignment was lost when shear ceased.

Sodium salicylate (NaSal) is the most commonly used counterion to induce viscoelasticity in cationic surfactant solutions. NaSCN, 2-aminobenzenesulfonate, perfluorobutyrate ($C_4HF_7O_2$), and 4-methylsodiumbenzoate were listed by Manohar (Manohar 1998) as counterions that can produce viscoelasticity with cetyltrimethylammonium bromide solutions. Other aromatic counterions like mono- and dichlorobenzoate (meta or para-substituted), nitrobenzoate, tosylate (p-methylbenzenesulfonate), and hydroxynapthalenecarboxylates (HNC) are known to induce viscoelasticity when combined with cationic surfactants (Magid 1998). Benzenesulfate, 4-chlorobenzoate, 5-chlorosalicylate, 2,6-hydroxybenzoate, 1-OH-2-naphthoate and 3-OH-2-naphthoate, 3,4 dichlorobenzoate, 3,4 dimethylbenzoate, and 3,5 dichlorobenzoate are also effective binding counterions for cationic surfactants, which promote drag reduction (reduction in energy dissipation in turbulent flows of aqueous solutions) (Chou 1991) (Lin et al. 2001). Cleary et al. suggested that "to be effective as a counterion, a candidate compound should be unbranched and amphiphilic and should possess a structure such that its negative charge is highly dispersed over its headgroup." (Cleary et al. 1991) Chou also proposed that hydrophilic and hydrophobic groups on the aromatic rings of counterions should be located as far apart as possible to foster large micellar length and obtain good drag reduction, so that the hydrophilic groups can associate with the polar ammonium headgroup while the hydrophobic group is associated with the hydrocarbon tail inside the micelles (Chou 1991).

Both the chemical structure of the counterion and its molar ratio to the cationic surfactant affect the effectiveness of a cationic surfactant as a drag reducer, its rheological behavior, and the nanostructures formed (Chou 1991) (Lu 1997). A counterion with a hydrophilic group substituted for a hydrogen atom on the hydrophobic benzene ring may interact with the surfactant to promote formation of long threadlike micelles. Counterions with one substituted hydrophilic group on the benzene ring, such as −OH, −SO3, −COO, −SO2, were tested for drag reduction effectiveness with the cationic surfactant, Kemamine Q-2983C [$C_{22}H_{43}$-N(CH_3)_3Cl] (Chou 1991). Results showed that the phenol ($C_6H_5CO^-$) system showed only 30 % drag reduction, while benzenesulfonate ($C_6H_5SO_3^-$) gave viscoelastic properties and effective drag reducing solutions (>50 % drag reduction). The SO_3^- group substituted on the benzene ring was the only single-substituent group found to be an effective drag reducing counterion.

A number of aromatic counterions with two substituted groups were quite effective in promoting drag reduction. NaSal has two adjacent hydrophilic groups, COO⁻ and OH. Other counterions with one hydrophilic and one hydrophobic group on the benzene ring, like tosylate (p-methylbenzenesulfonate), 4-methylbenzoate and 4-

fluorobenzoate, 4- chlorobenzoate, 4- iodobenzoate, and 4-bromobenzoate behave like NaSal as do 2-aminobenzoate and 2-nitrobenzoate (Chou 1991) (Zakin et al. 2007) (Ge et al. 2008). Here, we report our study of a commercial cationic surfactant, cetyltrimethylammonium chloride (CTAC) with counterions with Cl- or CH3-substituted groups at different positions on the salicylate benzene ring, carried out to determine the influence of counterion configuration on the properties of the surfactant solutions and to compare the influence of the more hydrophobic Cl- with the CH3- group.

Experimental section

Materials

The cationic surfactant, Arquad 16–50 [$C_{16}H_{33}N(CH_3)_3Cl$], cetyltrimethylammonium chloride (CTAC), is a commercial product donated by Akzo-Nobel. 3-chlorosalicylic acid, 4-chlorosalicylic acid, 5-chlorosalicylic acid, 3-methylsalicylic acid, 4-methylsalicylic acid, and 5-methylsalicylic acid were purchased from Aldrich Chemical Co. An appropriate amount of NaOH (purchased from Mallinckrodt, AR grade) was used to neutralize each acid. All the Arquad 16–50 (5 mM)/counterion (5 mM) solutions were clear after preparation at room temperature.

Drag reduction measurements

Turbulent drag reduction experiments were carried out in a recirculation system containing a 122-cm long, 0.617-cm inner diameter stainless steel test section. The system was temperature-controlled to allow experiments to be run from 2 to 150 °C. For a detailed description of this apparatus, refer to Lu (Lu 1997).

Cryo-TEM

Surfactant solution specimens were prepared for cryo-TEM in a controlled temperature and humidity chamber, by applying a small drop of the studied solution onto a perforated carbon film supported on a copper electron microscope grid, blotting it to form a thin (~0.2 μm) liquid film, and rapidly plunging the grid into liquid ethane at its freezing point, thus vitrifying the liquid (Talmon 2007). By avoiding water crystallization, the nanostructure is not disturbed, and the images obtained reflect the true nanostructures in the original solutions. However, one should bear in mind that blotting to form the thin film subjects of the solution to very high rates of shear. Images of the vitrified samples were obtained with a Philips CM120 transmission electron microscope operated at 120 kV, using an Oxford CT 3500 cooling holder, and an FEI T12 TEM with a Gatan 626 cryo-holder operated at about −180 °C.

Images were recorded with a cooled CCD camera at nominal objective lens underfocus of approximately 2 μm, with low electron doses to minimize electron-beam radiation damage to the specimen.

The profile of the vitrified film suspended over the polymer grid is important for analysis of the micrograph: the film has a thickness distribution as shown in Fig. 1.

Figure 1 shows the schematic of a cross-section of the film thickness distribution over a hole in the perforated carbon film.

The thicker part near the edge may reach 200–400 nm in thickness, and the thinner part may be as thick as 10 nm. It is important to recognize that different portions of the film are subject to different shear rates during blotting.

Rheological measurements

Shear viscosity

Shear viscosity measurements were carried out with a Rheometrics RFSII Couette rheometer, equipped with sensitive transducers allowing measurements at a shear rates as low as 0.02 s^{-1}. The Couette cell had a cup diameter of 34 mm and bob diameter of 32 mm, giving a fixed gap of 1 mm. The bob length was 32 mm. Temperature was controlled by a fluid bath to ±0.05 °C. Viscosity at each shear rate was calculated as the average of clockwise and counterclockwise measurements. After each reading, the waiting time before taking the data at the next shear rate was 20 s, to allow the system to reach equilibrium.

Swirl decay time

Viscoelastic systems display recoil after swirling, a property that can be utilized in quick screening tests; 60 ml of solution was placed in a 100-ml beaker. A one-inch magnetic stirring bar was rotated in the solution at about 700 rpm. The time for recoil to begin after the stirring is abruptly stopped is denoted as the swirl decay time (SDT).
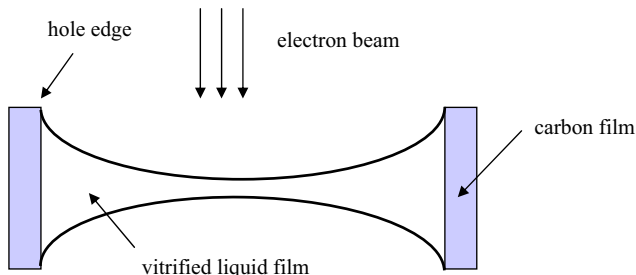

Fig. 1 Schematic of the film thickness distribution for the cryo-TEM grid

First normal stress difference measurements

First normal stress differences, N_1, were measured at 20 ± 2 °C using a Rheometric Scientific, Inc. RMS-800 rheometer with a cone-and-plate testing device. The normal force and shear stress on the upper fixed cone (50 mm in diameter and 0.04 radians cone angle) were measured. Due to the limited sensitivity of the transducers, the lowest shear rate tested was 25 s^{-1}. Also, no measurements could be made at shear rates above 800 s^{-1} because of foaming of the surfactant solutions.

Inertia effects are very significant for N_1 values less than 1000 Pa in the range of shear rates of interest. Thus, all first normal stress normal stress difference results were corrected for inertial effects using the following equation given by Macosko (Macosko 1994):

$$N_{1\text{corrected}} = N_{1\text{reading}} + 0.15\rho\Omega^2 R_p{}^2 \tag{1}$$

where ρ is solution density, Ω is angular velocity, and R_p is cone radius. Because the inertial force is in the opposite direction to the normal force, the correction term, $0.15\rho\Omega^2 R_p{}^2$, was added to the rheometer output, giving an increase in $N_{1\text{corrected}}$.

Results and discussion

Drag reduction

Figure 2 shows the drag reduction results for Arquad 16–50 (5 mM)/3-chlorosalicylate (5 mM). The drag reduction data for the other solutions are not shown because all of the graphs are qualitatively similar, the only differences being the effective temperature ranges, which are summarized in Table 1. The effective drag reduction temperature range was defined
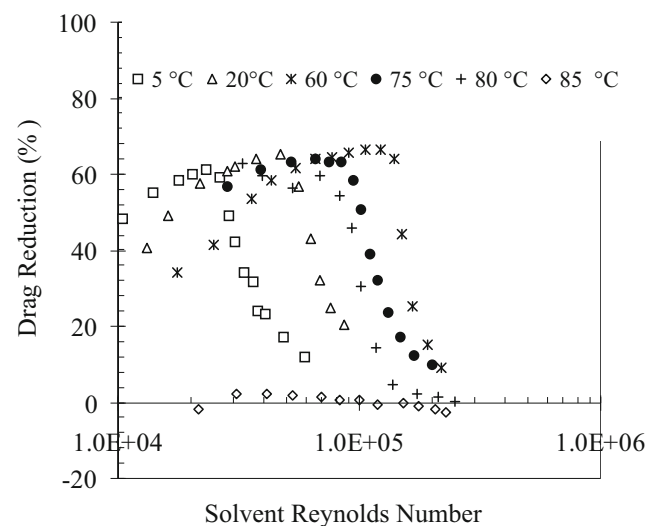

Fig. 2 Drag reduction of Arquad 16–50 (5 mM)/3-chlorosalicylate (5 mM) system

Table 1 Effective drag reduction temperature range of Arquad 16–50 (5 mM)/chloro- or methylsalicylate (5 mM)

Counterion	3-Cl-	4-Cl-	5-Cl-	3-CH₃-	4-CH₃-	5-CH₃-
Temperature (°C)	5–80	10–80	5–80	20–80	5–70	10–70

by the temperatures where the percent drag reduction exceeded 50 % and was 5–80 °C for this solution.

All these systems are effective drag reducers in the temperature range from 20–70 °C, while the Arquad 16–50 (5 mM)/ NaSal (5 mM) solution has an effective drag reduction temperature range of 10–65 °C. The 4-CH₃-salicylate, and 3-Cl- and 5-Cl-salicylate solutions were effective down to 5 °C, the lowest temperature tested in the flow loop, while the 4-Cl-salicylate and 5-CH₃-salicylate were effective to 10 °C. In all cases, the addition of the Cl- or CH₃- group to salicylate raised the upper effective drag reduction temperature limit compared with salicylate.

Cryo-TEM images

Cryo-TEM images show the nanostructures of the drag reducing micellar solutions. For the CH₃-salicylate series, the 4-CH₃-salicylate solution shows aligned threadlike micelles (Fig. 3a), caused by the shear during blotting. A threadlike micellar network is seen in the 5-CH₃-salicylate systems (Fig. 3b).

However, the 3-CH₃-salicylate system shows vesicles and open vesicles (Fig. 3c). A detailed study of this 3- CH₃-salicylate system under shear was previously reported (Zheng et al. 2000). Their cryo-TEM images showed evidence of a shear-induced nanostructure transition from vesicles to threadlike micelles due to the shear caused by blotting. Some vesicles reformed if the solution was allowed to relax on the grid for 10 s after blotting before vitrifying the solution. The solution completely transformed back to vesicles after 30 s of relaxation on the grid after blotting, before vitrification. Their hypothesis was that "the straining actions of flow disrupt vesicles, distort the distribution of, or strip, the counterions around vesicle fragments, shift the binding and dissociation equilibrium of counterions, alter the local preferred curvature of the surfactant assemblies, and this induces structural instability of the fragments that leads to their reconstruction into networks of branching threadlike micelles" (Zheng et al. 2000). This system shows viscoelastic behavior at room temperature because threads form when the solution is sheared.

Figure 4a shows the entangled threadlike micellar networks of the 4-Cl-salicylate system, while the 3-Cl- and 5-Cl-salicylate systems (Fig. 4b, c) exhibit vesicles and threadlike micelles. We postulate here also that vesicles change into threadlike micelles under high shear, similar to the 3-CH₃-salicylate system, resulting in drag reducing behavior.
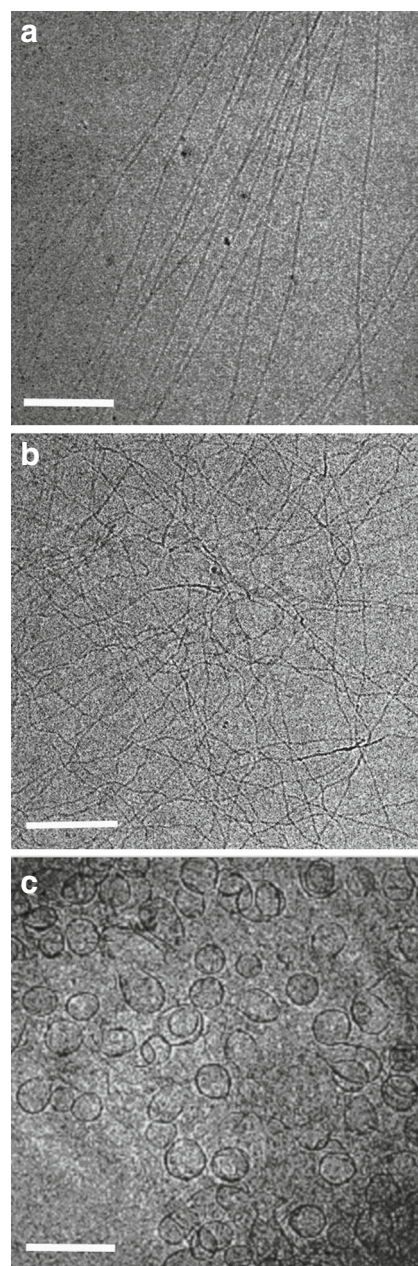
Fig. 3 Cryo-TEM images of Arquad 16/50 (5 mM) with 4-methylsalicylate (5 mM) (**a**), 5-methylsalicylate (5 mM) (**b**), and 3-methylsalicylate (5 mM) (**c**). *Scale bars* are 100 nm

It has been suggested that there are two types of viscoelastic surfactant solutions. The first and more common type of viscoelasticity is a result of the threadlike structure present in the surfactant solution at rest. Viscoelastic behavior is already present in the stagnant solution (Hoffmann et al. 1996), and the entanglements between threadlike micelles are an essential reason for the elasticity, as it is in polymer solutions (Shikata et al. 1997). In the other type, threadlike micelles are induced by shear. Below a characteristic critical shear stress, the surfactant solution behaves like a non-viscoelastic fluid, while above this critical shear, these solutions become viscoelastic.
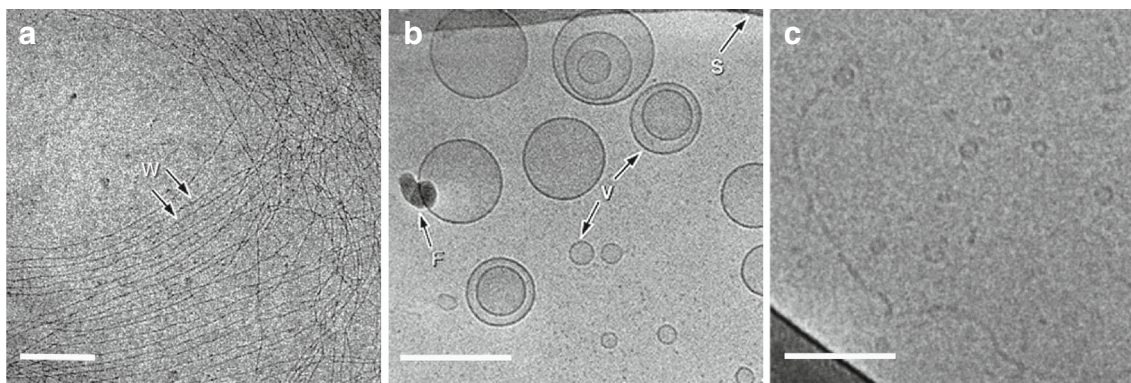
Fig. 4 Cryo-TEM images of Arquad 16/50 (5 mM) with 4-chlorosalicylate (5 mM) (**a**) and 5-chlorosalicylate (5 mM) (**b**). *F* frost, *V* vesicles, *M* spherical micelles, *W* threadlike micelles, *S* support grid. *Scale bars* are 100 nm

In this case, the nanostructure of the micelles changes under shear; the shear causes a new structure to form with viscoelastic properties.

Rheological behavior

Shear viscosity

Shear viscosities of all solutions were measured at room temperature (22 ± 2 °C) as shown in Fig. 5. All the CH_3-salicylate solutions have very similar behavior. After showing shear thinning behavior at low shear rates, they all show a rise in apparent viscosity, referred to as shear-induced viscosity caused by shear-induced structures (SIS), starting at a shear rate of about 30 s^{-1} and reaching a peak at about 100 s^{-1}. At higher shear rates, shear thinning was observed. In contrast, the Cl-salicylat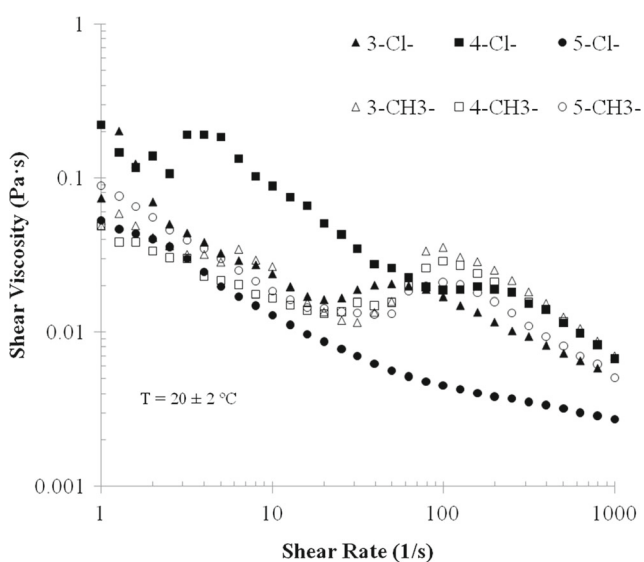e series shows different trends for counterions with different chloro positions on the benzene ring. The 3-Cl-salicylate solution showed a trend similar to the CH_3-salicylate series, with SIS starting to form at a shear rate of about 25 s^{-1} and a peak near 60 s^{-1} followed by shear thinning. The 4-Cl-salicylate system shows SIS at a low shear rate, shear thinning and a smaller second SIS region at shear rates between 60–250 s^{-1} followed by shear thinning. The 5-Cl-salicylate system has the lowest apparent viscosity showing shear thinning of about one order of magnitude over the shear rate range of 1 to 1000 s^{-1} with no SIS. Thus, while the position of the methyl group (3, 4, or 5) on the salicylate does not have much influence on the shear viscosity, the position of the chloro group makes a big difference.

A chlorine atom attached to the benzene ring has a radius of about 1.76 Å, while that of CH_3 is 1.91 Å, so there is a small difference in their sizes (Hart 2001). The electronegativity of carbon is 2.5, hydrogen is 2.2, and chlorine is 3.2 on the Pauling scale (Wade 1991). Hydrogen's electronegativity is similar to that of carbon, and the C–H bond is usually considered to be non-polar. The C–CH_3 bond is also non-polar, so the −CH_3 group, while hydrophobic, has weak electronegativity, and its position on the salicylate ring does not greatly affect its interaction with the CTAC headgroup and thus has little effect on the macroscopic rheological properties. However, at its least favorable 3- position, it apparently increases the effective tail volume, which increases the packing parameter causing, Arquad 16–50 (5 mM) with 3-CH_3-salicylate (5 mM) to form vesicles in the quiescent state. In contrast, when the chlorine is bonded to a carbon on the benzene ring, the bonding pair electrons are attracted more strongly to the chlorine atoms. Thus, its position on the salicylate ring dramatically influences the counterion effect on the micellar nanostructure and on the macroscopic rheological behavior as described above. Since the chloro influence at the 3 position close to the salicylate polar group is small, its shear viscosity behavior and other properties are similar to the methylsalicylates.

Fig. 5 Shear viscosity of Arquad 16–50 (5 mM)/methyl- and chlorosalicylate (5 mM) at room temperature

Swirl decay

The swirl decay time room temperature measurement results (Table 2) show that all the systems are viscoelastic with strong recoil after swirling at room temperature.

Normal stress

First normal stress differences, N_1, are shown Fig. 6. Data for the methylsalicylate series show increasing N_1 with shear rate until about 600 s^{-1} and then fall off. Similar to its effect on shear viscosity, the chlorine atom position has a major effect on the normal stress of the chlorosalicylate solutions. The 5-Cl- system, which had no SIS, showed near zero N_1 in the shear rate range tested and a swirl decay time of 2.5 s. These results corroborate results for other systems that display no SIS and also show no N_1 in the range of shear rates measured (Zakin et al. 2007) (Qi et al. 2009). While 2.5 s is one of the longest recoil times for these solutions, it does, nonetheless, indicate that the solution is viscoelastic. In other words, solutions showing recoil can have near zero N_1. The 4-Cl- system, whose swirl decay time is 2.6 s, has low N_1 until the shear rate exceeds 500 s^{-1}, above its second SIS. The 4-CH$_3$-salicylate solution, which has the shortest swirl decay time among the methylsalicylates, has the highest N_1 below 100 s^{-1}.

Transient stress

Transient stress behavior under shear was investigated for the 3-CH$_3$-salicylate and 5-Cl-salicylate solutions whose nano-structures are vesicles. Figure 7 shows the stress development of the 3-CH$_3$- system under shear rates of 50, 100, 200, 300, and 600 s^{-1}. Stress overshoots were observed at all the shear rates tested. The growth of the stress was slower at lower shear rates, indicating that the transition of vesicles to threadlike micelles under shear takes a longer time at lower shear rates, suggesting that a threshold of shear energy is required to reach the peak shear stress. This transition time was quite short at shear rates greater than 200 s^{-1}. Similar behavior was observed in the 5-Cl- system. Moderate stress overshoots were observed for this low viscosity system, and the magnitude increased with shear rate. This further confirms that this solution has viscoelastic properties despite its zero N_1 values.

To sum up, the rheological measurements show that the position of the methyl group on the salicylate has less influence on rheological behavior, such as shear viscosity and
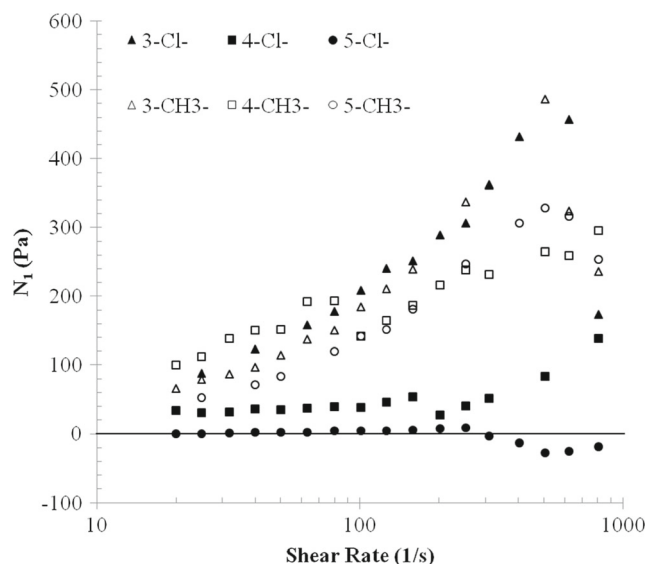

Fig. 6 First normal stress difference, N$_1$, of Arquad 16–50 (5 mM)/ methyl- and chlorosalicylate (5 mM) at room temperature

viscoelasticity of the methylsalicylate solutions, than the position of the more electronegative chloro group which has a greater impact on the rheological behavior of the chlorosalicylate solutions, presumably because the more electronegative Cl- penetrates deeper into the micelle core. The 5-Cl-salicylate system has low shear viscosity and near zero N_1; yet, it is viscoelastic. The 4-Cl-salicylate system has high shear viscosity and modest N_1, while the 3-Cl-salicylate behaves like the methylsalicylate series.

Conclusions

1. The addition of Cl- or CH$_3$- on the salicylate ring at the 3-, 4-, or 5- positions promotes higher upper effective drag

Table 2 Swirl decay time of Arquad 16–50 (5 mM)/salicylate (5 mM) at room temperature

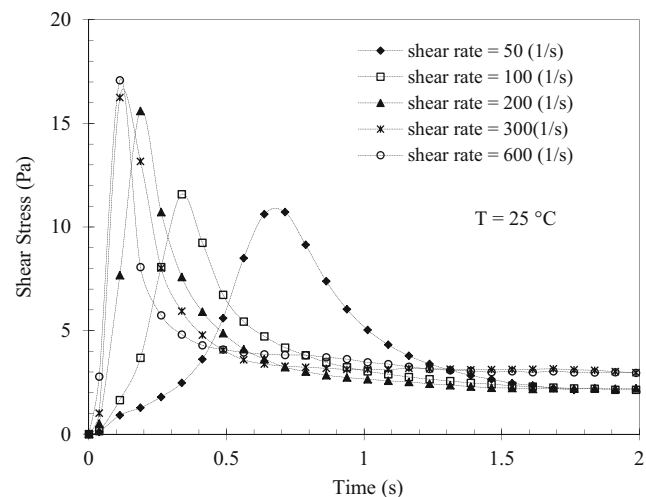| Counterion	3-Cl-	4-Cl-	5-Cl-	3-CH$_3$-	4-CH$_3$-	5-CH$_3$-
SDT (s)	1.2	2.6	2.5	2.1	1.3	1.5

Fig. 7 Transient shear stress development under different shear rates of Arquad 16–50(5 mM)/3-methylsalicylate (5 mM) at room temperature

reduction temperature limits than NaSal when present as a counterion for the cationic surfactant, Arquad 16–50 (commercial CTAC), at concentrations of 5 mM for both the counterion and surfactant.

2. All the 3-, 4-, and 5-Cl- and 3-, 4-, and 5-CH$_3$- salicylate solutions show some viscoelastic character. The position of the methyl substituent group has little effect on shear viscosity, SIS, and first normal stress difference, N_1, but the position of the chloro substituent group on the salicylate ring has a dramatic influence on rheological behavior. The rheological behavior of the 3-Cl- solution is similar to that of the three methyl substituted groups. High shear rate is required before high N_1 is obtained for the 4-Cl- solution. The 5-Cl- solution, which displayed no SIS and showed near zero N_1 but fairly low SDT, showed stress overshoot. The absence of both SIS and N_1 in this drag reducing system in the shear rate range tested corroborates similar observations in several other systems (Lu et al. 1997) (Zakin et al. 2007) (Qi et al. 2009).

3. Cryo-TEM images of the 4- and 5-CH$_3$-salicylate systems and the 4-Cl-salicylate system show threadlike micelles, while 3-CH$_3$-salicylate shows vesicles and open vesicles. The 3- and 5-Cl-salicylate systems show vesicles and threadlike micelles. It is postulated that systems with vesicles in the quiescent state change their nanostructures to threadlike micelles under shear.

4. The presence of threadlike micelles that form networks may be the cause of viscoelasticity in most of these solutions. However, it should be noted that a soyatrimethylammonium surfactant (5 mM) with sodium salicylate (12.5 mM) with a threadlike micellar network solution was nonviscoelastic (Lu et al. 1997) (Zakin et al. 2007). Moreover, threadlike micellar structure at rest is not a necessity. Threads can be induced by shear of vesicles, and thus, the solution can exhibit viscoelasticity and drag reduction. Also, micellar solutions which have near zero N_1 can show other viscoelastic behavior, e.g., in SDT and stress overshoot measurements.

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