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Wormlike micellar solutions formed by an anionic surfactant and a cationic surfactant with two head groups†

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Innovation in the molecular structure of surfactants is important for the preparation of soft materials with novel properties. In this study, we synthesized a cationic surfactant, N^1,N^1,N^1,N^1,N^3,N^3,N^3-pentamethyl-N^3-(3-stearamidopropyl)propane-1,3-diammonium bromide, hereafter referred to as C18-DQA. Unlike conventional cationic surfactants, C18-DQA contains two quaternary ammonium head groups and a long-saturated alkyl chain equal to a chain length of 21 carbon atoms. C18-DQA exhibits a low Krafft point of ~0 °C and a water solubility >1000 mM at 25 °C. The critical micelle concentration (cmc) of C18-DQA was determined to be 0.59 mM using the Nile red method. C18-DQA was mixed with sodium laurate (SL) at different molar ratios to produce transparent solutions with excellent viscoelasticity over a wide concentration range. The 1 : 1.5 molar ratio C18-DQA/SL mixed solutions exhibited gel-like behavior for a total surfactant concentration of 2.88 wt% (75 mM). The solution with a total surfactant concentration of 300 mM (120 mM C18-DQA and 180 mM SL) achieved a maximum zero-shear viscosity (η_0) of 4224 Pa s. Cryogenic transmission electron microscopy analysis revealed the formation of extremely long wormlike micelles, with a cross-sectional diameter of 5 nm and contour length >3 μm, in the mixed solutions. C18-DQA and SL molecules were drawn close by electrostatic attractions, leading to a suitable molecular geometry for the extensive growth of wormlike micelles. This work will act as an important reference for the future preparation of highly viscoelastic solutions by mixing cationic and anionic surfactants. The proposed system is also expected to have potential applications in cosmetic formulations, home care products, and oilfield fracturing fluids.

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1. Introduction

At suitable conditions, surfactants with specific molecular structures can form rod-shaped or wormlike micelles in an aqueous solution. The entanglement of these elongated aggregates results in the formation of three-dimensional networks, which significantly increase the solution viscoelasticity. In the past decades, wormlike micellar solutions have been applied in a wide range of fields, such as friction reduction,[1–3] drug delivery,[4,5] and petroleum extraction.[6–8]

Gemini surfactants or surfactants with long alkyl chains[9,10] can form wormlike micelles under particular conditions by themselves.[11–14] Considering the effect of ionic strength, the salt-free catanionic surfactant systems were also developed. For instance, the tetradecyltrimethylammonium hydroxide (TTAOH) and lauric acid (LA) mixed system can not only form wormlike micelles, but also exhibit rich phase behavior depending on the concentration ratio or temperature.[15,16] Most surfactants can only form such micelles in the presence of additives, such as inorganic salts and other types of surfactants.[17–19] In ionic surfactant solutions, the oppositely charged ions or surfactants can effectively screen head group charges of a surfactant through electrostatic attractions, forming elongated aggregates. For instance, the zero-shear viscosity (η_0) of 1-hexadecyl-3-methylimidazolium bromide (C16mimBr) and sodium toluate (NaTos) mixed solution increases from ~0.01 to 6.1 Pa s as the NaTos concentration increases from 20 to 30 mM for a fixed C16mimBr concentration of 40 mM.[19] The addition of *trans*-cinnamate (*trans*-NaCA) into a photo-responsive solution containing *N*,*N*′-bis(2-(dodecanoyloxy)ethyl)-*N*,*N*,*N*′,*N*′-tetramethyl-1,3-propane diammonium dibromide (C12DEG3) leads to the growth of wormlike micelles, substantially increasing the solution viscosity after ultraviolet irradiation.[20]

Carboxylate surfactants are a class of anionic surfactants with an $RCOO^-$ salt head group and an alkyl tail. They are widely

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used in home care products and detergents owing to their mild, non-toxic, and biocompatible properties. These surfactants can also be used to produce viscoelastic solutions through the formation of wormlike micelles.[21] However, for example, mixed solutions of sodium laurate (SL) and cetyltrimethylammonium bromide (CTAB) have been found to have $\eta_0 \leq 20$ Pa s for all the investigated molar ratios.[22] Sodium decanoate and CTAB mixed systems were found to exhibit an η_0 of only ~100 Pa s, even for a high total surfactant concentration of 171 mM. The behavior of the above-mentioned systems suggests that the viscoelasticity of carboxylate surfactant solutions[23–25] is quite weak compared to other catanionic surfactant systems.[26,27] According to the packing parameter theory,[28] a suitable molecular geometry is the prerequisite for effective wormlike micelle formation. Favorable viscoelastic properties are commonly achieved by adding salts to mixed carboxylate and cationic surfactant systems.[29,30] For example, the η_0 of sodium oleate ($C_{18}H_{33}NaO_2$) and octyl trimethylammonium bromide ($C_{11}H_{26}BrN$) mixed system was higher than 1000 Pa s at the presence of 0.3–0.4 M sodium carbonate (Na_2CO_3).[31]However, this method is not always effective as excessive salts can often lead to surfactant precipitation.[32] Therefore, Optimizing the structure and formulation of surfactants is necessary for the preparation of viscoelastic solutions containing common carboxylate surfactants. In this study, we synthesized N^1,N^1,N^1,N^3,N^3,N^3-pentamethyl-N^3-(3-stearamidopropyl)propane-1,3-diammonium bromide, a novel quaternary ammonium surfactant, abbreviated as C18-DQA. Unlike conventional quaternary ammonium surfactants, C18-DQA contains two hydrophilic ammonium head groups. A series of transparent solutions with excellent viscoelasticity were obtained by mixing C18-DQA and sodium laurate (SL) for a wide range of molar ratios and concentrations. Cryo-transmission electron microscopy (Cryo-TEM) revealed the formation of extremely long wormlike micelles within these solutions. The proposed system enriches the formulation of viscoelastic solutions containing carboxylate surfactants. The designing principle of this novel cationic surfactant can also inspire the preparation of catanionic surfactant systems.

2. Materials and methods

2.1. Materials

Stearic acid ($CH_3(CH_2)_{16}COOH$) was from TCI Chemical Industry Development Co., Ltd (Shanghai, China); methanol (analytical grade) was from Sinopharm Chemical Reagent Co., Ltd (China); 3-dimethylaminopropylamine was from Aladdin Reagent Co., Ltd (Shanghai, China); and SL (99%) was from Adamas. (3-bromopropyl)trimethylammonium bromide was synthesized in our lab following a standard procedure.[33]

2.2. Synthesis

The C18-DQA synthesis process is shown in Fig. 1 (detailed synthesis steps mentioned in the ESI†). In brief, the steps were as follows: firstly, stearic acid was reacted with methanol to obtain methyl stearate. The purified methyl stearate was further reacted with 3-dimethylaminopropylamine to produce *N*-(3-(dimethylamino)propyl)stearamide, which was then mixed with (3-bromopropyl)trimethyl ammonium bromide using ethanol as the solvent. This mixture was refluxed for 2 d, and the final product, C18-DQA, was obtained through recrystallization.

2.3. Sample preparation

Aqueous solutions with designated C18-DQA and SL concentrations were prepared using water (ρ = 18.2 MΩ cm) purified by a Milli-Q water purification system. All prepared samples were kept in sealed glass vials and incubated at 25 ± 0.1 °C for at least 24 h prior to further measurements.

2.4. Fluorescence probe measurements

A 1 mM Nile red methanol solution was made. 20 μL of the solution was transferred into a series of 10 mL bottles, which were then blow-dried with N_2. Subsequently, 6 mL of the C18-DQA aqueous solutions of different concentrations were separately added to these bottles. The samples were sonicated for 2 h followed by shaking for 24 h at 25 °C. The fluorescence of the solutions was measured using a spectrofluorometer (CARY Eclipse, VARIAN). The excitation and emission wavelengths used were 540 and 550 nm, respectively. The bandwidths of both slits were set to 5 nm.

2.5. Krafft temperature

The 1 wt% surfactant aqueous solution was refrigerated at ~0 °C for 3 d. The solution remained clear and no precipitates were observed, indicating that the Krafft temperature of C18-DQA was ~0 °C.

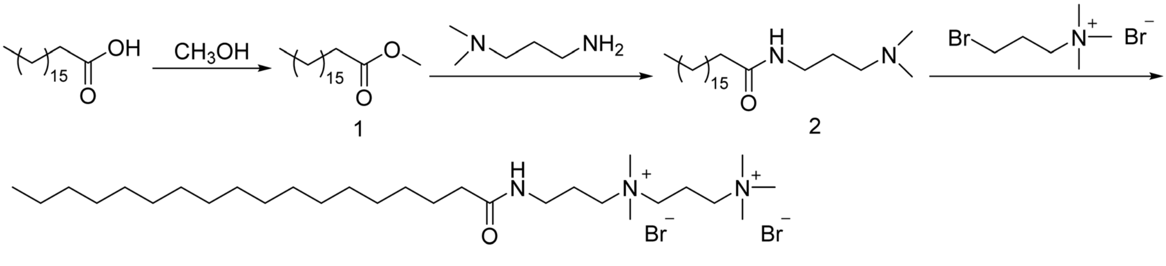

Fig. 1 C18-DQA synthesis process.

2.6. Rheological measurements

The rheological tests were performed on the DHR-3 rotational rheometer (TA instrument). The standard-steel cone-plate detector had a diameter of 40 mm and a cone angle of 2°. For the steady-state rheological tests, the minimum and maximum shear rates were set to 1.0×10^{-3} and 600.0 rad s^{-1}, respectively. Before the oscillatory rheological test, a stress sweep was carried out at a frequency of 6.28 rad s^{-1}. The strain value was set at 2% to ensure that the sample was in the linear viscoelastic region in the subsequent tests.

2.7. Cryogenic transmission electron microscopy (Cryo-TEM)

Cryo-TEM samples were prepared in a controlled environment vitrification system (CEVS). A micropipette was used to load 3 μL of the sample onto a TEM copper grid, which was blotted with two pieces of filter paper to form thin films suspended on the mesh holes. Then, the sample was quickly plunged into a reservoir of liquid ethane cooled by nitrogen at −165 °C. The vitrified samples were then stored in liquid nitrogen until they were transferred to a cryogenic sample holder (Gatan 655) and examined using a Talos F200C transmission electron microscope (200 kV) at ∼−174 °C. The images were recorded on a Gatan multiclan CCD and processed using a digital micrograph.

3. Results and discussion

The physical and chemical properties of the C18-DQA/SL mixed solutions were investigated. At 25 °C, C18-DQA exhibited a high aqueous solubility of ∼1000 mM (38.5 wt%; Fig. S4, ESI†), attributed to its two hydrophilic head groups. The Krafft point of C18-DQA was ∼0 °C, significantly lower than that of octadecyl trimethyl ammonium bromide (OTAB; 36 °C),[34] which endows C18-DQA with a large range of application temperature. The critical micelle concentration (cmc), determined by the Nile red fluorescence probe method, was found to be ∼0.58 mM (Fig. S5, ESI†), which is consistent with the results of the Du Noüy ring method (0.59 mM, Fig. S6, ESI†), suggesting excellent aggregability of C18-DQA.

The C18-DQA aqueous solutions acted as Newtonian fluids for a wide concentration range. The solution with a C18-DQA concentration of 200 mM exhibited a zero-shear viscoelasticity of only 0.001 Pa s (Fig. S7, ESI†). Upon adding SL, the C18-DQA solutions showed a visible increase in viscoelasticity. For convenience, the phase behavior of the C18-DQA/SL mixed solutions with different molar ratios is described using only the C18-DQA concentration hereafter (Fig. 2). Increasing the SL molar ratio significantly enhanced the solution viscoelasticity. The 1 : 1, 1 : 1.5, and 1 : 2 molar ratio C18-DQA/SL solutions began exhibiting gel-like behavior at a C18-DQA concentration of 30, 21, and 10 mM. The 1 : 2 molar ratio solution turned turbid at a C18-DQA concentration of 90 mM (Fig. S8, ESI†). The simple phase diagram revealed that for a wide range of molar ratios, the aggregates formed by C18-DQA and SL efficiently increased the viscoelasticity of the solutions, an unprecedented observation compared to previously reported wormlike micelle systems constructed with SL and other cationic surfactants.[35,36] As shown in Fig. 3, at a fixed C18-DQA concentration, the zero-shear viscosity of the mixed solutions increased with increasing the molar ratio of SL : C18-DQA, indicating an optimized molecular geometry for forming wormlike micelles. This phenomenon was also reflected by their dynamic rheological behavior, which was shown in Fig. S9 (ESI†). For molar ratio 1 : 1 C18-DQA/SL system, the storage modulus and loss modulus crossed with each other at rather high oscillatory frequency. This means that the wormlike micelles formed are rather short. In the 1 : 1.5 and 1 : 2 C18-DQA/SL system, the crossover was expected to appear at very low frequencies, indicating the presence of very long wormlike micelles. The rheological behavior of these solutions with different molar ratios showed similar behavior. Comparatively, the 1 : 1.5 system showed the more obvious viscoelastic rheological behavior than that of 1 : 1 system. The concentration range of 1 : 1.5 system in a transparent solution state is also wider than that of 1 : 2 system. Therefore, the 1 : 1.5 molar ratio C18-DQA/SL mixed solutions were selected for detailed investigation.

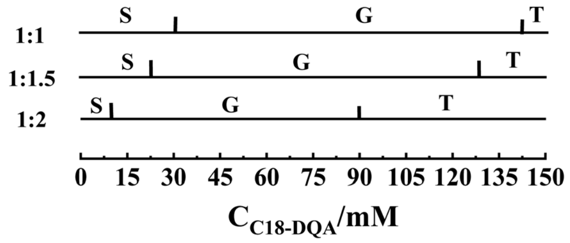

Fig. 2 Simple phase diagram of C18-DQA/SL mixed solution for different molar ratios at 25 °C (S denotes sol, G denotes gel, and T denotes turbid state).

As shown in Fig. 4, for C18-DQA concentrations <20 mM, the 1 : 1.5 C18-DQA : SL mixed solutions exhibited good fluidity. For C18-DQA concentrations >30 mM, the solutions can hold their weight in an inverted tube. Further increase in surfactant concentrations led to the formation of highly viscoelastic solutions, as indicated by the gradually decreasing curvature of the

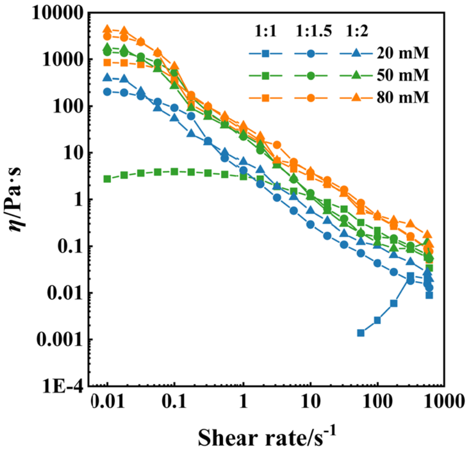

Fig. 3 Viscosity (η) as a function of the shear rates for different C18-DQA concentrations at different molar ratio with SL. The concentration of C18-DQA and molar ratio are shown in the figure.

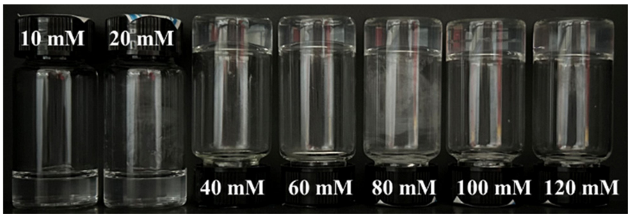

Fig. 4 Photographs of 1 : 1.5 molar ratio C18-DQA/SL mixed solutions for different C18-DQA concentrations at 25 °C.

meniscus. All solutions were transparent for the investigated concentrations, suggesting a well-dissociated state of the and 1 : 1.5 molar ratio C18-DQA/SL mixed solutions. When cationic and anionic surfactants are mixed, precipitates tend to form due to the strong electrostatic interactions between the surfactants' ionic head groups.[37,38] In comparison, C18-DQA and SL molecules showed good synergistic effects in the solutions without precipitation. This effect is possibly attributed to the unique molecular structure of C18-DQA, as it contains two cationic quaternary ammonium head groups. Over a wide range of molar ratios, the charge carried by the head group cannot easily be completely neutralized. In addition, the C18-DQA alkyl chain contains 21 carbon atoms in contrast to the 11 carbon atoms of the SL alkyl tails. The formed catanionic pairs can be extremely asymmetric in structure, reducing the possibility of precipitation. Therefore, compared to traditional cationic surfactants, C18-DQA exhibited excellent performance in a catanionic surfactant system.

Rheological behavior of 1 : 1.5 C18-DQA : SL mixed solutions was further investigated. A shear thickening behavior of mixed solutions was observed when C18-DQA concentrations were in the range of 2–6 mM [Fig. 5(a)]; the total surfactant concentrations were only 0.2–0.6 wt%. The shear thickening behavior is typically attributed to the formation of shear-inducing structures.[39–41] Large transient aggregates formed in the solution by the alignment of the smaller aggregates due to directional shear forces. This suggested the existence of smaller asymmetric aggregates at very low surfactant concentrations, showing the strong aggregation ability of C18-DQA and SL. For solutions containing C18-DQA ≥ 8 mM, a shear thinning behavior was observed, indicating the formation of wormlike micelles.

The 1 : 1.5 molar ratio C18-DQA/SL mixed solutions also showed remarkable viscoelasticity compared to other carboxylate surfactant solutions. Mixing cationic surfactant *N*-tetradecyl-*N*-(2-hydroxyethyl)dimethylammonium bromide (DM; 25 mM) and SL (30 mM) results in η_0 of this solution is only 0.6 Pa s.[42] In comparison, the 1 : 1.5 molar ratio C18-DQA/SL mixed solution with a total surfactant concentration of 50 mM exhibited $\eta_0 \sim 200$ Pa s. This value was even higher than that of the Gemini surfactant 12-3(OH)-12/SL (total surfactant concentration of 80 mM)[43] and CTAB/SL mixed solutions (total surfactant concentration of 130 mM).[36]

For a C18-DQA concentration of 80 mM, the η_0 of the solution reached ∼3000 Pa s, a property that is seldom found among carboxylate surfactant solutions.[44] Zhang *et al.*[16] prepared viscoelastic solutions exhibiting η_0 in several thousands Pa s using sodium erucate (NaOEr), a C22 anionic surfactant with a monounsaturated tail, in the presence of benzyl trimethyl ammonium bromide (BTAB) and tetramethyl ammonium bromide (TMAB). This indicates that a long alkyl tail in the surfactant structure is essential for high viscoelasticity of the solutions. In the present C18-DQA/SL systems, C18-DQA molecule contains an alkyl tail equal to about 21 carbon atoms.

Strong hydrophobic interactions occurred between C18-DQA molecules as well as SL molecules involved in self-assembly. The aggregates formed were quite difficult to dissociate during the entanglement process, resulting in the enhanced viscoelasticity of the solutions. The variation of η_0 with C18-DQA concentrations can be divided into dilute, semi-dilute, and concentrated regions [Fig. 5(b)]. For C18-DQA concentrations < 8 mM, η_0 increased gradually with the surfactant concentration, increasing sharply above 8 mM. Therefore, 8 mM was recognized as the overlap concentration, above which the

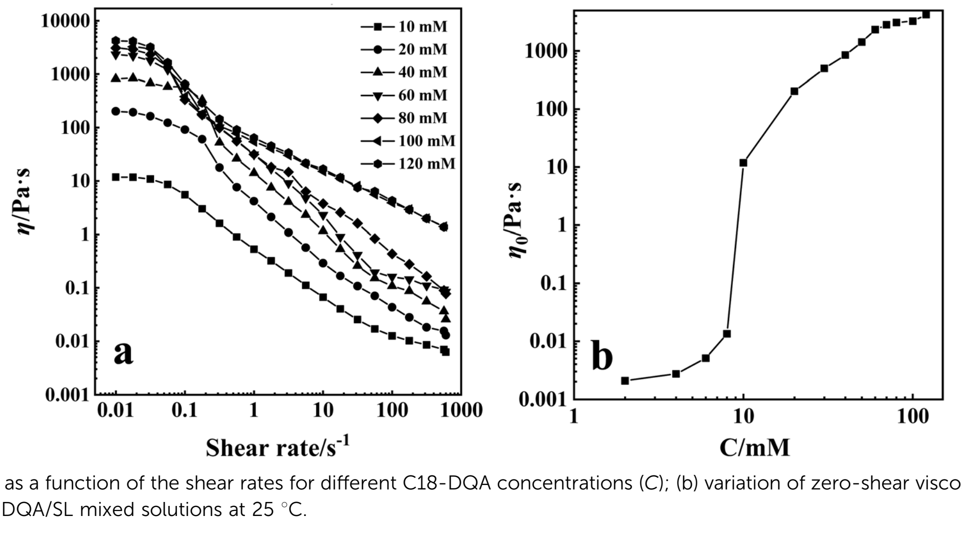

Fig. 5 (a) Viscosity (η) as a function of the shear rates for different C18-DQA concentrations (C); (b) variation of zero-shear viscosity (η_0) with C for the 1 : 1.5 molar ratio C18-DQA/SL mixed solutions at 25 °C.

wormlike micelles in the solutions began to grow rapidly and entangle. In several systems, η_0 initially increased with the concentration, reaching a maximum, followed by a subsequent decrease.[44] This phenomenon can be explained by the micellar branching in the solutions at high salt and surfactant concentrations. The joints of the branched micelles slide along the micelle body, aiding the relaxation process, and leading to a decrease in η_0. However, when C18-DQA concentrations >60 mM, η_0 saturated. No reduction of the viscosity was observed within the investigated concentration range, suggesting that the micellar branching is not the dominant process in the 1 : 1.5 molar ratio C18-DQA/SL mixed solutions.

The aggregate morphology of the 1 : 1.5 molar ratio C18-DQA/SL mixed solutions was investigated using Cryo-TEM. The sample solutions with a concentration of 10 and 15 mM (well above the overlap concentration) were selected. Extremely long, linear wormlike micelles with a cross-sectional diameter of 5 nm were found in the 10 mM solution, with some entangled structures [Fig. 6(a)]. The micelle ends could not be identified even under a large vision field, revealing highly efficient one-dimensional micelle growth. The average length of the micelles was estimated to be >3 μm. These long wormlike micelles were possibly responsible for the high viscoelasticity of the solutions. Although the total surfactant concentration was only 0.95 wt% (25 mM), the η_0 reached 10 Pa s [Fig. 5(a)], which is a rare finding among cationic and anionic surfactant mixed systems.[45–47] In the 15 mM solution [Fig. 6(c)], there was a significant increase in the number of wormlike micelles, along with an enhanced entanglement in comparison to the 10 mM solution [Fig. 6(b)].

The measurements of the storage (G') and loss (G'') moduli for the 1 : 1.5 molar ratio C18-DQA/SL mixed solutions were carried out within a typical frequency range of 0.01–100 rad s^{-1} at 25 °C (Fig. 7). For the 10 mM solution, a crossover appeared at $\sim$2 rad s^{-1}, with $G' > G''$ above 2 rad s^{-1} and *vice versa*. This behavior was similar to most wormlike micellar systems.[48,49] However, $G' > G''$ for solutions with concentrations $\geq$15 mM, indicating the crossover to be unavailable or in the very low angular frequency region. The solutions thus behaved elastically due to the entanglement of the seemingly unbreakable, extremely long wormlike micelles (Fig. 6). Once the number of micelles crosses a certain threshold, they formed transient three-dimensional networks through entanglements. The microscale network structures can resist deformation and restrict the free movement of water molecules, resulting in solution elasticity. With the increasing angular frequency, ω, G' approached a constant value, *i.e.*, the plateau modulus, G_0, which is related to the number density of the micelle entanglement points. G_0 increased with the surfactant concentration, suggesting an increase in the number of entanglements.

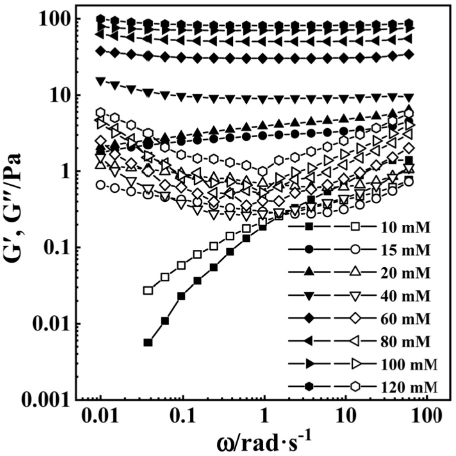

Fig. 7 Storage modulus, G', (black blocks) and loss modulus, G'', (white blocks) as a function of the oscillatory frequency of the 1 : 1.5 molar ratio C18-DQA/SL mixed solutions at 25 °C, the concentration of C18-DQA is shown in the figure.

The C18-DQA/SL mixed solutions exhibited extremely long wormlike micelles, resulting in gelation even at low surfactant concentrations. The unique molecular structure of C18-DQA is speculated to be responsible for this unique phenomenon. As previously mentioned, C18-DQA contains two cationic head groups that can combine with more than one anionic surfactant. For example, in the 1 : 1.5 molar ratio C18-DQA/SL mixed systems, a C18-DQA molecule combined with 1.5 SL molecules on average through electrostatic attractions (Fig. 8). The average molecular occupation area decreased largely, leading to a suitable molecular geometry for wormlike micelle formation.[16,50] In addition, the formed catanionic ion unit held a large volume of the hydrophobic group composed of 37.5 carbon atoms. Although the wormlike micelles are always undergoing a dynamic scission-recombination process, the former was suppressed by the strong

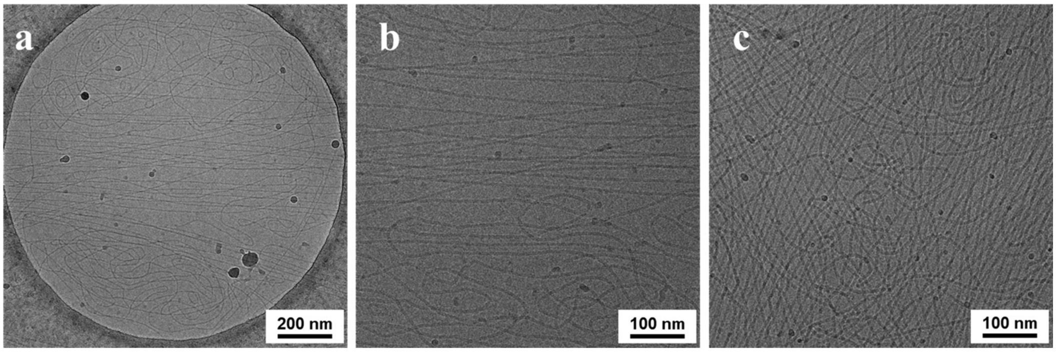

Fig. 6 Cryo-TEM images of 1 : 1.5 molar ratio C18-DQA/SL mixed solutions at a C18-DQA concentration of (a) and (b) 10 mM and (c) 15 mM.

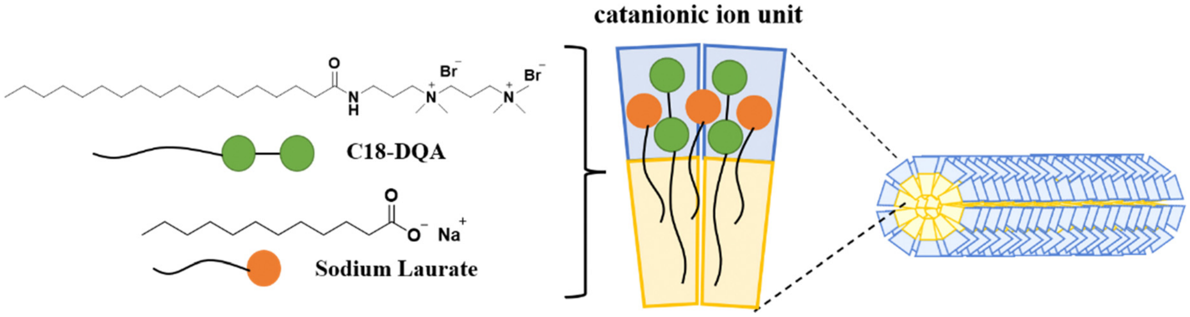

Fig. 8 Schematic Illustration of the formation of wormlike micelles by C18-DQA and SL.

van der Waals interactions generated by the large hydrophobic portions of the surfactants. The wormlike micelles formed in this manner remained unbroken and continuously increased in length.

Conclusions

In this study, a novel cationic surfactant, C18-DQA, containing a long alkyl tail and two quaternary ammonium head groups, was synthesized. Surfactants with long saturated hydrophobic chains are often insoluble in water at room temperature, which limits their applications. This problem was tackled by introducing an additional hydrophilic head group into the surfactant molecule. Due to the rich charges carried by these head groups, C18-DQA can form catanionic groups by combining with the SL anionic surfactant, resulting in transparent mixed solutions over a wide concentration range without precipitation. A small average molecular occupation area was achieved for the solutions, which is highly suitable for wormlike micelle formation. In addition, the strong van der Waals interactions induced by the large hydrophobic groups of the catanionic units suppress the micelle scission process and facilitate the formation of extremely long and unbreakable wormlike micelles. By comparing our C18-DQA/SL mixed solutions with other wormlike micellar systems, we infer that surfactants containing a long-saturated alkyl tail in a catanionic mixed system are beneficial for the formation of long wormlike micelles, increasing the solution viscoelasticity. The novel viscoelastic solutions presented in this study are expected to be applied in cosmetic formulations, home care products, and oilfield fracturing fluids.

Author contributions

Hongye Li: writing original draft, methodology, formal analysis, investigation. Zhengrong Lin: writing original draft, conceptualization, investigation. Zhao Chen: project administration, visualization, investigation. Zhenggang Cui: supervision, methodology. Lan Lei: project administration, visualization, funding acquisition. Binglei Song: methodology, writing – review & editing, investigation, funding acquisition.

Conflicts of interest

There are no conflicts to declare.

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