ORIGINAL ARTICLE



Synthesis and Solution Properties of a Double-Tailed Quaternary Ammonium Surfactant with a Protrudent Head Group

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Abstract Modification of the molecular structure of surfactants is an effective method for exploring their selfassembly. A double-tailed quaternary ammonium surfactant with a protrudent head group, namely 2-octyldecyltrimethylammonium bromide (2-ODTAB) was synthesized, and the solution properties were investigated by surface tension, dynamic light scattering, and cryogenic TEM. A comparative study was also performed on the traditional double-tailed homologue surfactants dioctyldimethylammonium bromide (8-8), didecyldimethylammonium bromide (10-10), and didodecyldimethylammonium bromide (12–12). The results showed that 2-ODTAB was more effective at lowering surface tension and in forming stable vesicles than traditional double-tailed surfactants with similar alkyl chain length. The reason is attributed to the improved structure of 2-ODTAB, in which the two alkyl tails are connected to the ionic head group by one carbon atom. This structure imparts more freedom to the head group and thus favors formation of more stable aggregates at low concentration. In addition, the lower limit of the alkyl chain length of the double-tailed surfactants for forming stable vesicles was illustrated.

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² School of Environmental and Chemical Engineering, Shanghai University, Shanghai 200444, People's Republic of China Keywords Double-tailed surfactant \cdot Molecular configuration \cdot Aggregation

Introduction

Surfactant molecules composed of a polar part and a nonpolar part have received extensive interest both in theoretical studies and in industrial applications. Depending on the molecular structure and solution environment, surfactants can self-assemble to form various aggregates such as micelles, vesicles, wormlike micelles [1], and even spongelike coacervates [2]. These aggregates of different morphologies endow the surfactant solutions with useful properties such as solubilization [3-6], viscoelasticity [7], and controlled release. Construction of novel aggregates is thus a hot topic in the field of surfactant research. Among the conditions that contribute to aggregate formation, molecular structure is a key factor that determines the selfassembly behavior of surfactants [8-10]. According to Israelachvili's theory [11], the aggregate morphology can be roughly predicted by the value of the critical packing parameter, $P = v/a_0 l$, where v is the volume of hydrophobic part, a_0 is the optimum head group area, and *l* is the extended alkyl chain length. This theory emphasizes the importance of molecular geometry in surfactant aggregation. By rationally selecting the head group and the chemical structures of hydrophobic tail, one often obtain novel surfactants with expected aggregation behaviors.

Vesicles are closed surfactant bilayers usually with a larger aggregate size than that of micelles. Because of their trapping properties, vesicles are widely used in biomimetic systems and drug delivery systems [12, 13]. Vesicles can also act as microreactors or nanoreactors for the preparation of nano-scaled particles and materials [14, 15]. If a

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vesicle system is composed of several molecular species, synergistic effects among multiple components usually dominate the aggregation process. Thus a single-component system is preferred for learning more about the effect of molecular structure on vesicle formation. In the absence of any additives, double-tailed surfactants generally have a greater tendency to form vesicles than those with a single tail [16]. For example, 1.4-bis(2-ethylhexyl)sulfosuccinate (AOT) is an anionic surfactant that can form vesicles in aqueous solutions and reversed micelles in apolar solvents without adding any cosurfactants. Didode-[17] cyldimethylammonium bromide (DDAB) is a cationic surfactant that can spontaneously form vesicles and lamellar phases under different conditions [18–20]. Because of its strong ability to form vesicles, DDAB is also used in mixed surfactant systems for investigating aggregate transitions [21-23]. It has been observed that in a DDAB molecule, the hydrophilic quaternary ammonium ion locates at the middle of the hydrophobic chain. When dispersed in water, the two hydrophobic parts separated by the quaternary ammonium ion tend to come into contact with each other to reduce the system energy. This configuration is generally adopted in sequential adsorption and in the aggregation process. This indicates that the dynamic molecular configuration and the resulting steric effect may affect the manner in which surfactants interact. For example, Xie and Zhao [24] discovered that a Gemini surfactant with a dibenzene spacer can form large aggregates with a loose structure at very low concentrations.

Because of the rigidity of the long spacer, the molecules tend to adopt a configuration suitable for the formation of aggregates with low curvatures. Since the dynamic molecular configuration is very sensitive to the factors affecting aggregation such as molecular structure, two surfactants with even subtle difference in their structures may behave quite differently in self-assembly [25, 26], showing a rather complex structure–property relationship. Investigation on the double-tailed synthetic surfactants with modified structures, therefore, may give more details of special properties of the vesicular systems.

In the present work we designed and synthesized a novel double-tailed surfactant as shown in Fig. 1, named 2-octyldecyltrimethylammonium bromide (2-ODTAB), in which the quaternary ammonium group is connected to the double tails by only one carbon atom. In 2-ODTAB, the quaternary ammonium ion is not exactly located at the middle of a linear hydrophobic chain. The peculiarity in aggregation of the novel surfactant was investigated and compared with traditional double-tailed surfactants like DDAB (12-12) and its homologues, dioctyldimethylammonium bromide (8-8), and didecyldimethylammonium bromide (10-10), where both hydrophobic chains are directly connected to the quaternary ammonium head group, for understanding the effects this modification would bring. Unlike the vesicular systems mentioned above [18-20, 22, 23], the comparative study mainly focused on the evolution of aggregates at low concentrations (just around the critical aggregation concentration), a region that has not been fully recognized.



Fig. 1 Reaction scheme for the synthesis of 2-octyldecyltrimethylammonium bromide (2-ODTAB)

Experimental

Materials

2-ODTAB, dioctyldimethylammonium bromide (8–8), didecyldimethylammonium bromide (10–10), and didodecyldimethylammonium bromide (12–12) were synthesized in our laboratory. The main reagents used for the synthesis, diethyl malonate (99 %), sodium hydride (60 %), 1-bromooctane (98 %), lithium aluminum hydride (97 %), *p*toluenesulfonyl chloride (*p*-TsCl) (99 %), and anhydrous lithium bromide (99 %), were all purchased from Aladdin Reagent Co., Ltd. (Shanghai, China) and used as received. Other reagents and solvents were all analytical grade.

Synthesis

The synthetic procedures are shown in Fig. 1. The detailed synthetic procedure is reported in the supplementary material. A general process is as follows: Diethyl malonate was reacted with 1-bromooctane to get diethyl 2-octylmalonate with the assistance of sodium hydride. Diethyl 2-octylmalonate was reacted with 1-bromooctane to get diethyl 2.2-dioctylmalonate. After a two-step decarboxylation, 2-octyldecanoic acid was obtained. 2-Octyldecanoic acid was reduced to 2-octyldecanol with lithium aluminum hydride. 2-Octyldecanol was then reacted with p-TsCl and anhydrous lithium bromide to obtain 9-(bromomethyl)heptadecane, which was then quaternized with trimethylamine to yield the crude product. The crude product was purified by column chromatography pretreated with NaBr and methanol [27]. The final product 2-ODTAB was obtained as gel-like solid under vacuum.

¹H NMR (400 MHz, DMSO) δ 3.18 (d, J = 8.0 Hz, 2H), 3.04 (s, 9H), 1.86 (s, 1H), 1.41–1.17 (m, 28H), 0.85 (t, J = 6.8 Hz, 6H).

Elemental analysis: Anal. Calcd. for $C_{21}H_{46}BrN \cdot H_2O$: C 61.44; H 11.79; N 3.41. Found: C 61.27; H 11.87; N 3.32.

Purity determined by HPLC: single peak, nearly 100 %.

Methods

Equilibrium surface tension was measured by a DCAT-21 tensionmeter (DataPhysics, USA) with a Pt–Ir Du Noüy ring at 25 °C. Dynamic light scattering of solutions was measured with a Brookhaven Instrument, which was composed of a BI-200SM goniometer, a BI-9000AT digital correlator (522 channels), and a photomultiplier detector. The apparent hydrodynamic radius of aggregate ($R_{h,app}$) was obtained from the Stokes–Einstein equation:

$$R_{\rm h,app} = \frac{k_B T}{6\pi \eta_0 D_{app}}$$

where $k_{\rm B}$ is the Boltzmann constant, *T* is absolute temperature, η_0 is the solvent viscosity, and D_{app} is the apparent translational diffusive coefficient, which can be obtained from the intensity-intensity time correlation function using the CONTIN program. The solutions for DLS measurement were all equilibrated for 10 days at 25 °C before measurement. The sample solutions of 3.5 µL for cryogenic TEM were dropped on a 200-mesh holey grid and were blotted for 4 s in a chamber at 100 % humidity using an FEI Vitrobot Mark IV. The aggregates were observed by an FEI Titan Krios electron microscope (300 kV).

Results and Discussion

Surface Activity

Like single-tailed surfactants, double-tailed surfactants adsorb at the air-water interface to form adsorption layers. This results in a decrease of the equilibrium surface tension, as shown in Fig. 2. With increasing surfactant concentration, the surface tension of all surfactant solutions decreases and then gradually levels off. The break point is denoted as critical aggregation concentration (*cac*), at which surfactant begins to form aggregates. The surface-active parameters obtained from the surface tension isotherms are listed in Table 1. For the homologue surfactants 8–8, 10–10, and 12–12, the variation of *cac* with the carbon atom number (*n*) in the alkyl chains can be expressed with the empirical formula log (*cac*) = A – Bn, as shown in Fig. 3, where A is an empirical constant which depends on temperature and hydrophilicity of the surfactant, and



Fig. 2 Equilibrium surface tensions of 2-ODTAB (filled square), 8-8 (unfilled circle), 10-10 (unfilled triangle), and 12-12 (unfilled inverted triangle) at 25 °C

Table 1Surface-activeparameters of four surfactants a25 °C

s at	$cac \pmod{L^{-1}}$	$\gamma_{cmc}~(mN~m^{-1})$	p <i>C</i> ₂₀	$\Gamma_{\rm max}~(\mu {\rm mol}~{\rm m}^{-2})$	A _{min} (nm ²)
2-ODTAB	1.56	23.7	3.94	2.14	0.78
8-8	18.6	26.2	2.77	2.16	0.77
10-10	1.82	25.8	3.82	2.25	0.74
12-12	0.23	28.6	4.91	1.95	0.85



Fig. 3 Logarithmic *cac* against the number of carbon atoms in the alkyl chain

B reflects the contribution of adding a methylene group to the alkyl chain. A larger B value corresponds to a greater contribution. Generally methyl groups connected directly to the quaternary ammonium nitrogen atoms are not considered as hydrophobic. From Fig. 3, B = 0.238 was obtained for 8-8, 10-10, and 12-12 homologues, which is smaller than that (B = 0.3) of conventional single-tailed surfactants. This is reasonable because for a double-tailed surfactant the relative increase in alkyl chain length, which determines the aggregation ability, by adding a methylene group is smaller than that for a single-tailed surfactant. The cac of 2-ODTAB, which contains only 18 carbon atoms, is close to that of 10-10 containing 20 carbon atoms, indicating that 2-ODTAB has a stronger ability to aggregate than the 10–10, and the lack of a carbon atom connecting to the quaternary ammonium head group does not affect the aggregation ability of double-tailed surfactants. This principle can be applied to design surfactants with stronger aggregation ability without lengthening the hydrophobic chains. The structure of 2-ODTAB also shows advantages in the ability to lower surface tension, as revealed by its $\gamma_{\rm cmc}$ shown in Table 1, which is lower than that of the homologues 8-8 to 12-12. This may be attributed to closer packing of 2-ODTAB at the air-water interface [28] than that of the other three double-tailed surfactants.

Aggregate Morphology

Double-tailed surfactants are well known to form vesicles in aqueous solutions. The resulting solutions usually look bluish or "turbid", which often causes a misunderstanding that the surfactants are poorly soluble. The presence of ordered vesicles can be identified by dynamic light scattering (DLS) or TEM methods [29]. DLS is a powerful tool for investigating the properties such as the hydrodynamic radius and size distribution of dispersed colloid particles. For associated colloids such as surfactant aggregates, the obtained information can be used to identify the aggregate shapes and existing states [30]. The mechanisms of selfassembly of the surfactants can then be speculated.

The time correlation functions measured by DLS and intensity-fraction distributions calculated by the CONTIN model at different concentrations are given in the supplementary material. The calculated apparent mean hydrodynamic radii $R_{\rm h}$ based on DLS results for the four surfactants are shown in Fig. 4. The corresponding cac value for each surfactant is marked with a dotted line. 12-12 is a widely investigated vesicle-forming surfactant, which can form both small vesicles with diameters of ca.10 nm [18] and multi-wall vesicles [19]. As shown in Fig. 4d, $R_{\rm h}$ of 60 nm was detected by DLS below the cac, indicating that aggregates are present. Cryogenic TEM measurements confirm this observation (Fig. 5a). No large aggregates are observed in the micrograph. Only a few aggregates of 10- to 20-nm diameters are present, which may be small vesicles that have been investigated previously [23]. When the concentration exceeds the cac, $R_{\rm h}$ shows a rapid increase, indicating growth in vesicle population and size. 10-10 solution shows a similar phenomenon to that of 12-12 (Fig. 4c). Vesicles formed by 10-10 were observed with cryogenic TEM at the concentration of 6 mM (Fig. 6b).

An unexpected phenomenon was discovered in 8–8 solutions (Fig. 4b). Below the *cac*, an increase in R_h with increasing concentration is revealed by DLS, which is similar to its homologue surfactants 10–10 and 12–12. A sharp decrease in R_h was then observed when the concentration exceeds the *cac*. The aggregates at 30 mM with an R_h of 2 nm are confirmed to be micelles by cryogenic TEM (Fig. 6c). This seems to be contrary to conventional wisdom concerning surfactant aggregation because an





Fig. 5 Proton NMR spectrum of 8–8 at different concentrations. **a** 10 mM, **b** 25 mM, and **c** 35 mM



Fig. 6 Cryogenic TEM images of a 12–12 at 0.1 mM, b 10–10 at 6 mM, c 8–8 at 30 mM, d 2-ODTAB at 3 mM, e vesicle fusion of 2-ODTAB at 3 mM, and f vesicle fission of 2-ODTAB at 10 mM

increase in concentration usually facilitates growth of larger aggregates. Since the large size aggregates revealed by DLS are not observed by cryogenic TEM, we believe that the large aggregates below the *cac* of 8–8 solution have unusual morphologies.

Compared with single-tailed surfactants with the same alkyl chain length, double-tailed surfactants have a stronger tendency to aggregate in water because of their more hydrophobic nature. However, at concentrations below the cac, the number of molecules is not enough to form stable aggregates. Although the contact of the two alkyl chains in one molecule is helpful for reducing the energy of the system, hydrophobic interactions with other molecules may be more effective. Thus, each molecule uses one of two chains to interact with that of other molecules, with the intramolecular hydrophobic interactions reduced instead. This is especially reasonable for the system of the 8–8 with short alkyl chain length because the cohesive hydrophobic force is rather low. As a result, large aggregates with a loose structure were formed. This condition is similar to network-like aggregates formed by a hexameric cationic quaternary ammonium surfactant at low concentrations reported by Fan et al. [31]. Aggregation in this way is a better choice for these double-tailed surfactants at low concentrations. With increasing surfactant concentration, these loose aggregates eventually transform to corresponding stable aggregates such as micelles or vesicles.

¹H NMR measurements were carried out to identify the aggregation state of 8-8. It is known that the signal of protons in the alkyl chain of a surfactant will move to lower field with decreasing polarity of the environment [31]. As shown in Fig. 5, the H1 and H2 signals, which belong to the alkyl chains of 8-8, remain almost the same below (Fig. 5a) and above (Fig. 5b) the cac. This indicates that the alkyl chain is already in a rather nonpolar environment before the formation of micelles, confirming the presence of large aggregates below the cac. With further increase in concentration (Fig. 5c), the downward shift of alkyl chain proton signals can be observed. H4 and H5, which are the protons near the ionic head group, show a rather obvious shift. This may be caused by close packing of surfactants in the aggregates and variation of polarity at the aggregate surface.

Compared with the homologue surfactants investigated above, 2-ODTAB showed stronger ability to form vesicles (Fig. 4a). Vesicles with diameters of 100-200 nm were observed by cryogenic TEM at 3 mM (Fig. 6d), which is just above its cac. Although the 10-10 has a cac close to that of 2-ODTAB, vesicles were observed at 6 mM, a much higher concentration than its corresponding cac. Considering that the number of carbon atoms in the hydrophobic chain of 10-10 is larger than that of 2-ODTAB, it is obvious that 2-ODTAB has a stronger ability to form vesicles. This fact demonstrates the superiority of the 2-ODTAB molecular structure in vesicle formation. Because the two alkyl tails in 2-ODTAB are connected by only one carbon atom to the ionic head group, there is more freedom for the head groups. Both the alkyl tails and head groups in 2-ODTAB can adopt configurations by suitable rotation or extension in vesicle bilayers, which accounts for the stability of vesicular aggregates.

The 2-ODTAB solutions were further investigated with cryogenic TEM. Although DLS gave rather narrow size distribution signals of aggregates, rich vesicular morphologies were observed. In a 2-ODTAB solution of 3 mM (Fig. 6d), vesicles with various diameters between 10 nm and nearly 300 nm all coexisted. Vesicle fusion phenomenon [32] was observed by chance (indicated by the arrow in Fig. 6e), where a small vesicle is swallowed by a large vesicle nearby to form a larger one. This is perhaps one of the pathways for the vesicle growth. With increasing surfactant concentration, DLS shows an increase in aggregate size. Larger vesicles with diameters of even 600 nm were present in 10 mM solution (Fig. 6f). Another phenomenon called vesicle fission [33] was also observed (indicated by the arrow in Fig. 6f). These data illustrate that although with only 18 carbon atoms in the hydrophobic chain, the double-tailed surfactant 2-ODTAB has the ability to form stable vesicles of different sizes at low concentrations as in other traditional vesicular solutions.

Conclusions

The solution properties of a newly synthesized doubletailed surfactant 2-ODTAB with a protrudent head group were investigated. The properties of the traditional doubletailed homologue surfactants in which two tails are directly connected to the ionic head group were also explored for comparison. All surfactants investigated formed vesicles above certain concentrations expect 8–8, which forms loosely packed large aggregates which then transform into small micelles with increasing concentration. Because of the head group peculiarity, 2-ODTAB forms more stable aggregates at low concentration compared with the traditional double-tailed surfactant 10–10 containing similar carbon atom number. The delicate molecular structure of 2-ODTAB endows the surfactant with pronounced self-assembly properties and reveals the importance of the linkage region that connects the polar head groups with the hydrophobic segments in a surfactant. These design principles can be applied rationally to synthesize surfactants with novel aggregation properties.

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