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ORIGINAL ARTICLE

Surface Activities of Three Anionic Gemini Surfactants Derived from Cyanuric Chloride: Effect of a Branched Hydrophobic Chain

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Abstract Sodium 4,6-(2-(*N*,*N*-bis-ethylhexylamino)-1,3,5-triazine-4,6-yl-amino) ethane sulfonate (IXC_8), Sodium 4,6-(2-(*N*,*N*-bis-octylamino)-1,3,5-Triazine-4,6-yl-amino) ethane sulfonate (XC_8) and 2,2′-(6,6′-(ethane-1,2-diylbis(azanediyl) bis(4-(octylamino)-1,3,5-triazine-6,2-diyl)) bis(azanediyl))diethane sulfonate (C_8-2-C_8) were synthesized from cyanuric chloride. The surface activity and application properties of these surfactants (XC_8, IXC_8 and C_8-2-C_8) were discussed. The values of CMC, γ_{CMC}, pC_{20}, Γ_{max}, and A_{min} calculated from surface tension measurement at 30 °C indicate that the surface activity of IXC_8, which has two branched hydrophobic carbon chains, has lower γ_{CMC} (26.8 mN m^{-1}) than the other investigated surfactants and excellent wetting ability.

Keywords Gemini surfactants · Branched hydrophobic carbon chains · Surface tension · Wetting ability · Emulsification stability · Lime soap dispersing power

Introduction

Gemini surfactants are made up of two amphiphilic moieties connected at the level of, or very close to, the head groups by a spacer group of various natures: hydrophilic or hydrophobic, rigid or flexible. Compared to the conventional monomeric surfactant, the corresponding Gemini surfactants generally present a significantly lower critical micelle concentration (CMC), a lower surface tension recorded at the CMC (γ_{CMC}), and a greater ability to increase viscosity of the diluted aqueous solution [1–3].

In recent years, many Gemini surfactants have been reported [4–12], but the hydrophobic chains of most of these gemini surfactants are straight. There are relatively few reports concerning gemini surfactants with branched hydrophobic chains. Thus, it is important to synthesize Gemini surfactants with branched hydrophobic carbon chains and study their properties. In our former research, sodium 4,6-(2-(*N*,*N*-bis-octylamino)-1,3,5-triazine-4,6-yl-amino) Ethane Sulfonate (XC_8) with two branched hydrophobic carbon chains was synthesized. The surface activity and thermodynamic properties of micellization were investigated [13]. Triazine acts as a linker for the hydrophilic head group to the hydrophobic tail. The molecular structure of these geminis looks like the English letter X, so we call them X-type gemini surfactants. The three chlorines of the cyanuric chloride react with –NH_2 step by step at high yield, so the reaction is quite simple [14–17].

To further investigate the effect of branched hydrophobic carbon chains, in this paper, another X-type gemini surfactant, sodium 4,6-(2-(*N*,*N*-bis-ethylhexylamino)-1,3,5-triazine-4,6-yl-amino) ethane sulfonate (IXC_8) with two branched hydrophobic carbon chains, was synthesized. Moreover, the properties of XC_8, IXC_8, C_8-2-C_8 and sodium dodecyl sulfate (SDS) were compared in this work. The structures of X-type alkyl sulfonate gemini surfactants (XC_8 and IXC_8) and gemini C_8-2-C_8 are shown in Fig. 1. The effect of the structure of alkyl chain on the surface properties were also studied. In addition, their wetting ability, emulsification ability and lime soap dispersing power were investigated.

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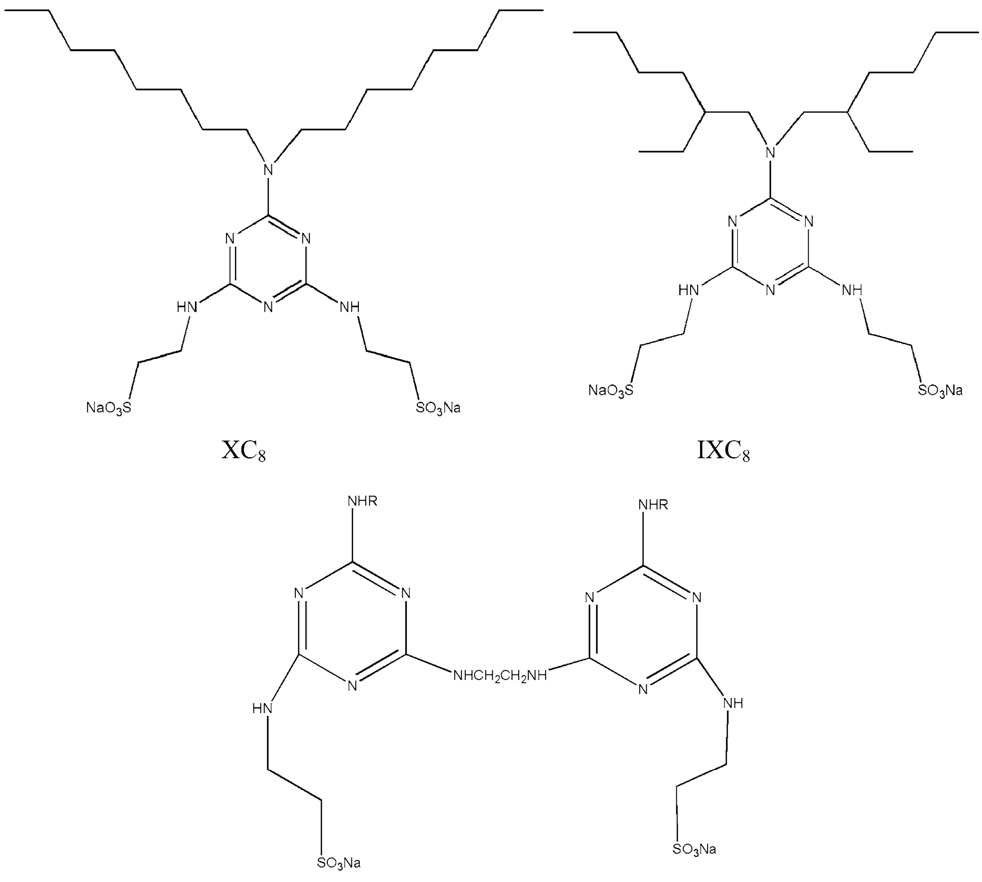

XC$_8$

IXC$_8$

C$_8$-2-C$_8$

Fig. 1 Structures of XC$_8$, IXC$_8$ and C$_8$-2-C$_8$

Materials and Methods

Materials

XC$_8$ was prepared according to Ref. [13]. C$_8$-2-C$_8$ was prepared according to Ref. [15]. Cyanuric chloride and taurine were purchased from Johnson Matthey Corporation. Bis(2-ethylhexyl)amine (99 %) was purchased from J&K Scientifics Ltd (Beijing). All the other reagents were of AR grade. Ultra-pure water was used to prepare the solutions in all experiments.

Synthesis of IXC$_8$

IXC$_8$ was synthesized from cyanuric chloride, taurine and bis(2-ethylhexyl)amine. The synthetic procedure and purification was similar to that reported in the literature [13]. The structure was confirmed by ^{1}H NMR, ^{13}C NMR, ESI-MS, IR spectra and Elemental analysis. ^{1}H NMR and ^{13}C NMR were recorded with a Bruker ARX600 NMR Spectrometer (Bruker BioSpin Corporation, Switzerland), ESI-MS were recorded with a Fourier transform ion cyclotron resonance mass spectrometer for electrospray ionization–mass spectrometry (Bruker Corporation, America), IR spectra were recorded with an FTIR-8400S Spectrometer (Shimadzu Corporation, Japan), Elemental analysis was recorded with a Vario EL Spectrometer (Elementar Corporation, Germany).

IXC$_8$: yield 85 %; FT-IR (KBr, cm^{-1}): 3341, 2928, 2900, 2853 1573,1519, 1446, 1183,1119, 1053, 755; Elementary analysis (found/calculated)/%: C (45.05/45.23), H (7.25/7.26), N (13.74/13.76); ESI-MS negative(*m*/*z*): 587.3 [M–Na]$^-$; ^{13}C NMR (600 MHz, DMSO–d$_6$, d/ppm): 11.11 $\mathrm{[2\text{-}CH_2CH(CH_2\underline{C}H_3)CH_2CH_2CH_2CH_3]}$, 14.35$\mathrm{[2\text{-}CH_2CH(CH_2CH_3)CH_2CH_2CH_2\underline{C}H_3]}$, 23.03 $\mathrm{[2\text{-}CH_2CH(CH_2CH_3)\text{-}CH_2CH_2\underline{C}H_2CH_3]}$, 23.86 $\mathrm{[2\text{-}CH_2CH(\underline{C}H_2CH_3)CH_2CH_2CH_2CH_3]}$, 28.64$\mathrm{[2\text{-}CH_2CH(CH_2CH_3)CH_2\underline{C}H_2CH_2CH_3]}$, 30.49 $\mathrm{[2\text{-}CH_2CH(CH_2CH_3)\underline{C}H_2CH_2CH_2CH_3]}$, 37.10 $\mathrm{[2\text{-}CH_2\underline{C}H(CH_2CH_3)CH_2CH_2CH_2CH_3]}$, 50.92 $\mathrm{[2\text{-}\underline{C}H_2\text{-}CH(CH_2CH_3)CH_2CH_2CH_2CH_3]}$, 165.68$\mathrm{[2\underline{C}\text{-}Cl]}$, 166.01$\mathrm{[\text{–}N{=}\underline{C}\text{–}N{=}]}$, 37.13 $\mathrm{[2\text{-}\underline{C}H_2CH_2SO_3Na]}$, 51.08$\mathrm{[2\text{-}CH_2\underline{C}H_2\text{-}SO_3Na]}$; ^{1}H NMR (600 MHz, DMSO-d$_6$, d/ppm): 0.81 [t, 12H, $\mathrm{2\text{-}NCH_2CH(CH_2\mathbf{C\underline{H}_3})(CH_2)_3\mathbf{C\underline{H}_3}}$], 1.18–1.22 [m, 16H, $\mathrm{2\text{-}NCH_2CH(\mathbf{C\underline{H}_2}CH_3)(\mathbf{C\underline{H}_2})_3CH_3}$], 1.73–1.77 [m, 2H, $\mathrm{2\text{-}NCH_2\mathbf{C\underline{H}}(CH_2CH_3)(CH_2)_3CH_3}$], 3.48 [d, 8H, $\mathrm{2\text{-}N\mathbf{C\underline{H}_2}CH(CH_2CH_3)(CH_2)_3CH_3}$, $\mathrm{2\text{-}NH\mathbf{C\underline{H}_2}CH_2SO_3Na}$], 2.65[t, 4H, $2NHC\underline{H_2}CH_2SO_3Na$], 6.27 [t, 2H, $2N\underline{H}CH_2CH_2SO_3Na$].

Measurements

Equilibrium Surface-Tension

The surface tensions of aqueous solutions of surfactant were measured with a K12 automatic tensiometer (Kross Corporation, Germany) by using the Wilhelmy plate technique. The temperature was at 30 ± 0.1 °C. The results of surface tension measurements are shown in Fig. 2. CMC values and the surface tensions corresponding to CMC (γ_{CMC}) were determined from the curves of surface tension *versus* the logarithm of surfactant concentrations ($\gamma \sim \log C$). The maximum surface excess (Γ_{max}) at the air–water interface was calculated by using the Gibbs adsorption equation [18], as follows,

$$\Gamma_{max} = -\frac{1}{2.303nRT}\left(\frac{d\gamma}{d\log C}\right)_T \qquad (1)$$

where R is the gas constant (8.314 J mol^{-1} K^{-1}), T is the absolute temperature (K), and ($d\gamma/d\log C$) is the slope in the surface tension isotherm when the concentration is near the CMC. The value of n is taken as 3 for a dimeric surfactant made up of a divalent surfactant ion and two univalent counterions in the absence of a swamping electrolyte [19]. In order to extract the minimum surface area occupied by a surfactant molecule (A_{min}) at the air–water interface, Eq. 2 was used,

$$A_{min} = \frac{10^{14}}{N_A \Gamma_{max}} \qquad (2)$$

where N_A is Avogadro's number, A_{min} is in nm^2, Γ_{max} is in $\mu mol\ m^{-2}$.

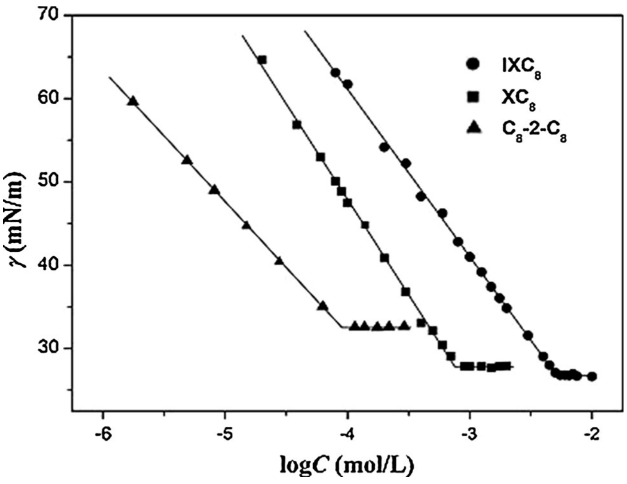

Fig. 2 Surface tension *versus* the logarithms of molar concentration plots for XC_8, IXC_8 and C_8-2-C_8 at 30 °C

Krafft Temperature

The Krafft temperature (T_K) was obtained by heating the surfactant solution until a clear solution was obtained [20]. In our experiment, all surfactant concentrations were 0.6 wt% (at least twice the CMC of the studied gemini surfactant).

Wetting Ability

Surfactant solutions (0.1 wt%) were prepared and kept at 25 °C. The wetting ability of the samples was measured by the Draves test [18]: a 5 g skein of gray, naturally waxed cotton yarn (54-in. loops containing 120 threads), is attached to a 3 g hook. The hook was totally immersed in a tall cylinder of surfactant solution using a weight tied to the hook by a thread. The surfactant solution displaces the air in the skein by the immersion wetting process, and when sufficient air has been displaced, the skein suddenly sinks in the cylinder. The better the wetting agent, the shorter the time required for sinking to occur. The wetting time was obtained by the average value over 10 measurements.

Emulsification Ability

According to [21] 0.1 wt% surfactant solutions were prepared and kept at 25 °C. A 40-ml sample solution and an equal volume of liquid paraffin were placed in a flask. The flask was shaken vigorously up and down for 5 min, and then kept still for 1 min. This was repeated for 5 times and then the solution was poured into a 100-ml measuring cylinder; the time of separating 10 ml water from the system was recorded. Emulsification ability was obtained from an average over 5 measurements.

Lime Soap Dispersing Power

Surfactant solutions (0.25 wt%) were prepared and kept at 25 °C . Lime soap dispersing power was determined using the Borghetty test [22]. A test tube was filled with 5 ml (0.5 wt%) sodium oleate solution, 10 ml hard water (containing 600 ppm Ca^{2+} and 400 ppm Mg^{2+}) and an initial small amount of surfactant solution. Then, water was added until the total volume was 30 ml. The test tube was stoppered and inverted 20 times, allowed to stand for 30 s. Eventually, the test tube was checked from visual inspection whether the lime soap deposit was dispersed into the solution or not. This test procedure was repeated under the same conditions but increasing amounts of surfactant solution until the minimum amount of the surfactant causing the lime soap deposit to disperse was obtained. There, the ratio of the minimum amount of surfactant and the weight of sodium oleate was called the lime soap dispersing power (LSDP), as follows:

$$\mathrm{LDSP}\,(\%) = \frac{\text{minimum weight required of the surfactant}}{\text{weight of sodium oleate}} \times 100 \tag{3}$$

According to this test method, a surfactant with a low LSDP is more effective than one with a high LSDP.

Results and Discussion

Krafft Temperature

The Krafft point measures the solubility of ionic surfactants. Table 1 lists the Krafft point of XC_8, C_8-2-C_8, IXC_8 and SDS. From Table 1, one can see that the Krafft points of XC_8, C_8-2-C_8 and IXC_8 are lower than SDS, and are all below 10 °C. This indicates that XC_8, C_8-2-C_8 and IXC_8 show good solubility in water. And such low Krafft temperatures permit the use of these surfactants in cold water.

Equilibrium Surface Tension

Table 1 lists the values of the CMC, γ_{CMC}, Γmax, A_{min}, pC_{20} of XC_8, IXC_8, C_8-2-C_8 and sodiumdodecyl sulfate (SDS). The γ_{CMC} of XC_8 and IXC_8 is lower than the γ_{CMC} of C_8-2-C_8, but their CMC's are higher than the CMC of C_8-2-C_8. The reason may be that molecular volumes of XC_8 and IXC_8 are smaller than C_8-2-C_8 that they adsorb more tightly at the air–water interface. Similarly, more C_8-2-C_8 diffuses into the bulk for forming micelles at low concentrations [23–25].

From Table 1, it can be found that the CMC value of IXC_8 is 4.89 mmol L^{-1}, higher than that of XC_8 0.759 mmol L^{-1}. However, the γ_{CMC} of IXC_8 is lower than that of XC_8. The reasons may be attributed to two branched hydrophobic carbon chains of IXC_8. It is well known that branched chains have weaker intermolecular cohesive forces than straight chains, which prevents the packing of the hydrocarbon chains in a closed-packed manner [26]. At the same time, more ethyl substituents presented on the carbon chain in IXC_8 restricts the rotational degree of freedom around C–C bond which causes the chains to tilt and prevents them from packing in a close manner. So, The CMC of IXC_8 is higher than that of XC_8. Secondly, the IXC_8 has 4 methyl groups that increase coverage and density of the hydrophobic surface so that the surface of solution is closer to that of the liquid hydrocarbon [27].

Efficiency (pC_{20}) and Minimum Surface Area (A_{min})

The pC_{20} value measures the efficiency of adsorption of surfactant at the air–water interface. Higher pC_{20} value indicate that the surfactant adsorbs more efficiently at the interface and reduces the interface tension more efficiently [18]. The pC_{20} value was calculated by using the following equation,

$$pC_{20} = \frac{\gamma_0 - 20 - \gamma_{\mathrm{CMC}}}{2.303nRT\Gamma_{\max}} - \log c \tag{4}$$

where γ_0 is the surface tension of water, c is the surfactant concentration.

The values of pC_{20} of XC_8, IXC_8 and C_8-2-C_8 are also listed in Table 1. The pC_{20} of XC_8 is lower than the pC_{20} of C_8-2-C_8. This may be attributed to the configuration of the surfactant molecules at the air–water interface when surfactants just begin to absorb at the interface. XC_8 is a surfactant where the hydrophilic group and hydrophobic group are linked by a rigid triazine ring spacer. When the bulk concentration is pre-CMC, the surfactant which has a rigid spacer may lie on the interface so that these molecules adsorb loosely at the interface [28]. The pC_{20} of IXC_8 is lower than the pC_{20} of XC_8. This indicates that the pC_{20} of the surfactant which has two straight hydrophobic carbon chains is higher than its isomers which has two branched hydrophobic carbon chains. Compared with SDS, the values of pC_{20} of XC_8, IXC_8 and C_8-2-C_8 are much larger. Therefore, the anionic Gemini surfactants are more efficient at reducing surface tension.

The information on the degree of packing and the orientation of the adsorbed surfactant molecule at the air–water interface can be obtained by using A_{min} values. The effectiveness of the adsorption at the interface increases with a lower A_{min} value. Both $\Gamma_{\max}$ and A_{min} values are

Table 1 The surface properties of XC_8, IXC_8, C_8-2-C_8 and SDS

Surfactant	T_{Krafft} (°C)	γ_{CMC} (mN m^{-1})	CMC (mmol L^{-1})	pC_{20}	Γmax (μmol m^{-2})	A_{min} (nm^2)
IXC_8	5	26.8	4.89	3.56	1.16	1.45
XC_8	3	27.9	7.59×10^{-1}	4.40	1.30	1.28
C_8-2-C_8	8	32.7	8.91×10^{-2}	5.21	0.91	1.83
SDS[a]	16	32.5	8.20	2.50	3.16	0.53

[a] Reported in Ref. [18] at 25 °C

Table 2 Wetting ability, emulsification stability and lime soap dispersing power of XC_8, IXC_8 and C_8-2-C_8

Surfactant	Wetting ability (s)	Emulsification stability (s)	LSDP (%)
IXC_8	15	100	30
XC_8	40	126	10
C_8-2-C_8	118	179	20

listed in Table 1, the A_{min} of XC_8 is the smallest, followed by IXC_8, the largest is C_8-2-C_8. This result may be related to the molecular volume of the surfactant [18]. It also suggests that gemini surfactants with two branched hydrophobic carbon chains adsorb more loosely than Gemini surfactant with two straight hydrophobic carbon chains at the air–water interface.

Wetting Ability

Wetting is important in many processes, both industrial and natural. In many cases, wetting is a prerequisite for application. Wetting involves the interaction of a liquid with a solid, including the formation of a contact angle at the solid/liquid/fluid interface, the spreading of a liquid over a surface (displacing the fluid initially in contact with that surface), or the penetration of a liquid into a porous solid medium [29].

As it can be clearly seen from Table 2, the wetting ability of XC_8 is better than C_8-2-C_8 and the wetting ability of IXC_8 is better than XC_8. These results indicate that: (1) the smaller the molecular volume of the gemini surfactant, the stronger the wetting ability. (2) The higher the branch degree of hydrophobic carbon chain, the stronger the wetting ability.

Emulsification Ability

The emulsification ability of a surfactant is determined by the rate of diffusion of surfactant from bulk solution to the interface between oil and water and the physical properties of the adsorbed layers formed from surfactant molecules around the inner phase droplet. The two main factors determining emulsion ability are low surface tension and mechanical strength of the interfacial film, and the latter is more important to some extent. If the surfactant molecules arrange more tightly in the interfacial film, the mechanical strength of formed interfacial film is higher [18]. From Table 1, it can be seen that pC_{20} values of these three surfactants are C_8-2-C_8 is 5.21, XC_8 is 4.40 and IXC_8 is 3.56. The higher pC_{20} value indicates the surfactant adsorbs more efficiently at the interface and reduces the interface tension more efficiently. On the other hand, compared to C_8-2-C_8 and XC_8, branched carbon chains presented in IXC_8 molecule makes IXC_8 arranges relatively loosely in the interfacial film. Both lower efficient adsorption at the interface and a looser packing manner make the IXC_8 cause worse emulsification. From Table 2, the emulsification stability of C_8-2-C_8 is best, followed by XC_8, the worst is the IXC_8. This result indicates that IXC_8 molecules arrange more loosely in the interfacial film.

Lime Soap Dispersing Power

Lime soap dispersing agents (LSDA) are surfactants that enable soaps to act as effective laundry detergents in hard water without the deposition of insoluble calcium soap. For a surfactant to act as an LSDA, it must possess a bulky hydrophilic group and a straight-chain hydrophobic group. It is believed that, in the presence of hardness ions (Ca^{2+}, Mg^{2+}), the soap and LSDA form a mixed micelle that shows high surface activity, including detergency. The bulky hydrophilic group of the LSDA forces the mixed micelle to orient its hydrophilic group toward the aqueous phase. Soap micelles by themselves are believed to invert in hard water, then their hydrophobic group is oriented toward the aqueous phase, producing insoluble lime soaps [18]. Table 2 lists the LSDP values of XC_8, IXC_8, and C_8-2-C_8. It can be seen that the lime soap dispersing power of XC_8 is best.

Conclusions

Surface and interface properties of two X-type anionic gemini surfactants and a gemini surfactant derived from cyanuric chloride were investigated. The results showed that the gemini surfactant with two branched chains has a lower γ_{CMC} and an excellent wetting ability.

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