

Influence of Substructures on the Spreading Ability and Hydrolysis Resistance of Double-Tail Trisiloxane Surfactants

Zhongli Peng · Caifeng Lu · Minling Xu

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Abstract Four types of novel double-tail trisiloxane surfactants of the general formula $\text{Me}_3\text{SiOSiMeR}^1\text{OSiMe}_3$ ($R^1 = -(\text{CH}_2)_3\text{NR}^2\text{CH}_2\text{CH}(\text{OH})\text{CH}_2(\text{OCH}_2\text{CH}_2)_x\text{OCH}_3$; $R^2 = -\text{CH}_2\text{CH}(\text{OH})\text{CH}_2\text{OCH}_2(\text{CH}_2)_y\text{CH}_3$, $-\text{CH}_2(\text{CH}_2)_3\text{CH}_3$, $-\text{CH}_2\text{CH}_2\text{CH}(\text{CH}_3)_2$; $x = 8.4, 12.9, 17.5, 22$; $y = 2, 6$), have been synthesized. Their structures were characterized by proton and carbon nuclear magnetic resonance. Most of them are able to reduce the surface tension of water to less than 24 mN/m at concentration levels of 10^{-5} mol/L and 10^{-4} mol/L. The emphasis was on the influence of substructures on their spreading ability and hydrolysis resistance. The results showed that a weaker hydrophilicity of a surfactant molecule, a larger molar ratio of methyl to methylene in the whole hydrophobic groups, more flexible hydrophobic groups and introduction of a methyl group in the spacer can all improve the spreading ability of the double-tail trisiloxane surfactant solutions on low-energy solid surfaces. The double-tail trisiloxane surfactants **1F** and **2F** are stable for more than 270 days in a neutral environment (pH 7.0). The hydrolysis resistance of the double-tail trisiloxane surfactants can be improved by a weaker hydrophilicity of the surfactant molecule, and a larger volume of the hydrophobic groups.

Keywords Double-tail surfactants · Trisiloxane · Spreading properties · Hydrolysis resistance

Abbreviations

HRA Hydrolysis resistant ability
SA Spreading ability

CMC	Critical micelle concentration
^1H NMR	Proton nuclear magnetic resonance
^{13}C NMR	Carbon nuclear magnetic resonance
γ	Surface tension
γ_{sv}	Solid/vapour interfacial energies
γ_{cmc}	The surface tension of surfactant solution at CMC
PTC	Phase transfer catalyst
HLB	Hydrophile/lipophile balance
Γ_{max}	Surface excess concentration
a_{m}^{s}	Surface area per molecule
ΔG_{mic}^0	Standard free energy of micellization

Introduction

Trisiloxane surfactants are usually denoted $\text{M}(\text{D}'\text{E}_n)\text{M}$ where M stands for the trimethylsiloxy group $(\text{CH}_3)_3\text{SiO}_{1/2}$, D' stands for $-\text{O}_{1/2}\text{Si}(\text{CH}_3)(\text{R})\text{O}_{1/2}$, where R is a polyoxyethylene group attached to the silicon by way of a propyl spacer, and E_n stands for polyoxyethylene, $-(\text{CH}_2-\text{CH}_2\text{O})_n\text{H}$ —[1]. This type of surfactant is effectively able to reduce the surface tension of water to approximately 21 mN/m. However, conventional hydrocarbon surfactants can only reduce the surface tension of water to about 30 mN/m [2] because of the arrangement of methylene groups on the water surface. Additionally, certain trisiloxane surfactant solutions are able to wet and spread rapidly on low-energy solid surfaces. This is called 'superwetting' or 'superspreading' [3]. The superspreading performance of the trisiloxane surfactants has found extensive use in agricultural adjuvants. Many studies of the spreading mechanism of trisiloxane surfactants have been carried out since their performance was discovered [2, 4–8]. There are a few different trends on the relationship of

Z. Peng (✉) · C. Lu · M. Xu
Department of Chemical Engineering, Huizhou University,
516007 Huizhou, China
e-mail: zsupzl@126.com

the structure with the spreading properties of trisiloxane surfactants. For example, a bulkier and more polar carbohydrate unit and the incorporation of hydrophilic spacer elements can reduce the trisiloxane surfactants spreading ability (SA) [9]; it seems that only the T-shaped trisiloxane surfactants are able to spread rapidly on solid surfaces, while flexible linear chain surfactants cannot do so [10]; their spreading performance is apparently affected by the HLB values and the molecular volume of the surfactants [8] and the structure of the trisiloxanyl units (hammer-like, linear) is not a critical parameter for the spreading of the trisiloxane surfactants as long as surfactant bilayers can be formed [11].

Additionally, the trisiloxane surfactants currently available exhibit a poor resistance to hydrolysis [12, 13]. Some of them hydrolyze rapidly when placed in an aqueous environment where the pH value is below 5 or above 9, and are stable only for 40 days even in a neutral aqueous environment (pH 7.0). This shortcoming limits their application as agricultural adjuvants, because they are likely to lose their efficacy as pesticides emulsifiers during transportation or storage. Therefore, the development of hydrolysis resistant superspreading surfactants is a quite important issue in formulating pesticide products.

Nevertheless, the study of hydrolysis resistant silicone surfactants has rarely been reported. A kind of hydrolysis resistant trimethylsiloxane surfactant was mentioned by Wagner et al. [14]. However, its aqueous solutions can only spread effectively on solid surfaces whose solid/vapor interfacial energies (γ_{sv}) are no less than 40 mN/m, which indicates that it does not match the agricultural adjuvant requirements. Recently, hydrolysis resistant disiloxane surfactants [12] and trisiloxane surfactants [13] have been synthesized by Leatherman MD et al. Unfortunately, these two types of siloxane surfactant can only reduce the surface tension of water to about 23 mN/m. Moreover, the siloxanes used to synthesize the above hydrolysis resistant surfactants have a very special structure, and they are not easily available on the market. As a consequence, the industrial production of these hydrolysis resistant siloxane surfactants is not easy.

It is known that certain double-tail hydrocarbon chain surfactants, e. g., sodium bis (2-ethylhexyl) sulfosuccinate, exhibit an ability to reduce the surface tension of water, to wet and spread on low-energy surfaces which are superior to those of the corresponding single-tail surfactants [15–18]. It is also known that the incorporation of a methyl group in the spacer of a trisiloxane surfactant is able to improve its hydrolysis resistance [13]. Consequently, to accumulate the previously mentioned features, we synthesized a series of new double-tail trisiloxane surfactants [19]. It was found that their hydrolysis resistant ability (HRA) is greatly improved with respect to original single-

tail trisiloxane surfactants, but that their spreading performance is not satisfactory.

As a further contribution, this paper reports on the synthesis and interfacial properties of four types of novel double-tail trisiloxane surfactants, and analyzes the influence of substructure on the SA and HRA.

Experimental Procedures

Materials

Type A single-tail trisiloxane surfactants (**1A**, **2A**, **3A**, and **4A**), whose structures are shown in Scheme 1, were synthesized as described in our previous report [19]. Paraffin wax was purchased from Shanghai Specimen and Model Factory, China. All of other chemicals were of analytical grade. Water was doubly distilled.

Synthesis

The synthesis route to the double-tail trisiloxane surfactants is shown in Scheme 1. Procedures (a) and (b) were carried out in accordance with references [20] and [21], respectively. One difference is that the used solvent is toluene, rather than methanol in procedure (b). Procedures (c) and (d) were carried out in the light of the reference [19].

Four types (**E**, **F**, **G** and **H**) and sixteen double-tail trisiloxane surfactants (**1E**, **2E**, **3E**, **4E**, **1F**, **2F**, **3F**, **4F**, **1G**, **2G**, **3G**, **4G**, **1H**, **2H**, **3H** and **4H**) were prepared. Their molecular structures are shown in Scheme 1.

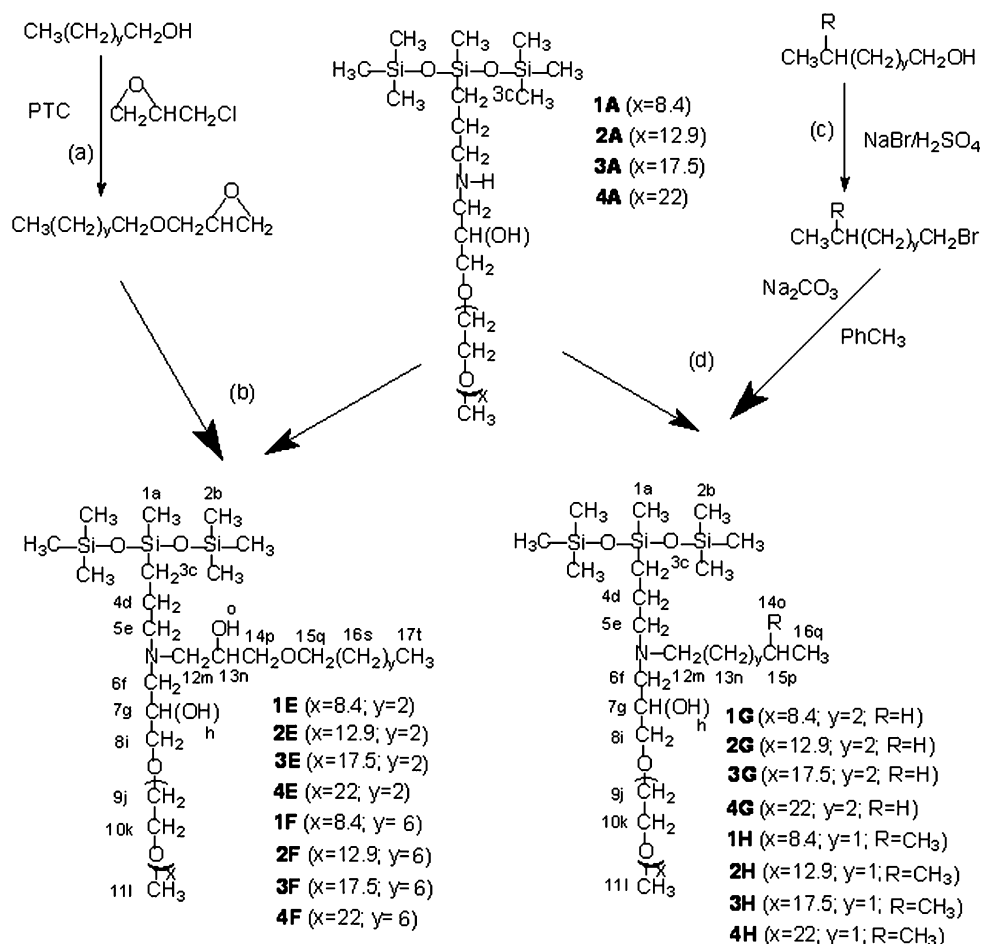
The ^1H - and ^{13}C -NMR spectra of double-tail trisiloxane surfactants prepared were analyzed according to the rule that different chemical environments of **H** and **C** result in different chemical shifts, and by comparing the chemical shifts of related compounds [19, 22, 23]. The assignments of the chemical shifts in ^1H - and ^{13}C -NMR spectra of **1E**, **1F**, **1G** and **1H** are listed in Tables 1 and 2.

Structural Characterization

^1H - and ^{13}C -NMR spectroscopy analyses were carried out with a Varian Mercury-plus 300 spectrometer in CDCl_3 .

Surface Activity and Hydrolysis Resistant Ability (HRA) Determination

Surface activity experiments were all carried out under constant atmospheric conditions (32 ± 2 °C room temperature, $60 \pm 3\%$ relative humidity). Aqueous solution surface tension (γ) values were obtained by the Wilhelmy plate method using a BZY-1 completely automatic surface tensiometer (Shanghai Equity Instruments Factory, China). The critical micelle concentration (CMC) values, the

Scheme 1 Synthesis route to double-tail trisiloxane surfactants**Table 1** $^1\text{H-NMR}$ spectral data (δ) of compounds **1E**, **1F**, **1G** and **1H**

H	1E (δ)	1F (δ)	1G (δ)	1H (δ)
a	-0.078	-0.039	-0.148	-0.078
b	-0.007	0.046	0.082	0.008
c	0.361	0.439	0.315	0.455
d	1.412	1.524	1.318	1.417
e	2.426	2.217–2.492	2.435	2.525
f	2.426	2.217–2.492	2.435	2.525
g	3.725	3.914	3.820	3.760
h	5.174/6.056/6.056	5.957/6.198	5.823/6.036	5.251/5.912/6.151
i	4.009	3.787	3.376	3.465
j	3.538	3.602	3.471	3.565
k	3.538	3.602	3.471	3.565
l	3.268	3.332	2.435	3.295
m	2.426	2.217–2.492	3.344	2.525
n	3.725	3.914	1.381	1.417
o	5.174/6.056/6.056	5.957/6.198	1.091	0.826
p	4.009	3.787	1.091	1.176
q	3.341	3.395	0.725	0.826
s	1.159	1.240		
t	0.790	0.837		

surface tension at CMC (γ_{cmc}), the SA and HRA were determined as reported elsewhere [19].

Results and Discussion

Interfacial Properties

The surface excess concentration (Γ_{max}) and the surface area per molecule (a_{m}^{s}) were computed by applying Eqs. 1

and 2 in the steeply downward section of the tension–log concentration plot just below the CMC. The standard free energy of micellization (ΔG_{mic}^0) of the double-tail trisiloxane surfactants was calculated by equation (3) [19, 22].

$$\Gamma_{\text{max}} = -\frac{1}{2.303RT} \left(\frac{\partial \gamma}{\partial \log C} \right)_T \quad (1)$$

$$a_{\text{m}}^{\text{s}} = \frac{10^{16}}{N_{\text{A}} \Gamma_{\text{max}}} \quad (2)$$

$$\Delta G_{\text{mic}}^0 = RT \ln \left(\frac{\text{CMC}}{55.5} \right) \quad (3)$$

Table 2 ^{13}C -NMR spectral data (δ) of compounds **1E**, **1F**, **1G** and **1H**

C	1E (δ)	1F (δ)	1G (δ)	1H (δ)
1	−0.194	−0.341	−0.023	−0.445
2	2.247	1.834	2.189	1.767
3	14.265	14.038	14.101	13.583
4	24.880	24.143	23.730	23.388
5	59.204	58.961	53.069	52.752
6	62.300	58.961	59.134	58.802
7	68.155	68.203	68.789	68.674
8	74.171	74.033	74.356	74.165
9	70.752	70.412	70.700	70.383
10	72.117	71.853	72.064	71.771
11	57.927	57.656	57.699	57.409
12	62.300	58.961	52.151	51.802
13	69.627	69.403	24.422/29.857	29.498
14	73.543	73.282	–	14.936
15	71.562	71.158	20.013	22.563
16	31.980/19.403	19.711–31.762	15.302	14.936
17	15.236	14.972	–	–

where $R = 8.3144 \text{ J/mol K}$, N_{A} is Avogadro's number, Γ_{max} and a_{m}^{s} are in mol/cm^2 and $\text{\AA}^2/\text{molecule}$, respectively. The data of the CMC, γ_{cmc} , Γ_{max} , a_{m}^{s} , ΔG_{mic}^0 and SA of the double-tail surfactants are listed in Table 3.

The CMCs of the double-tail trisiloxane surfactants are in the 10^{-5} – 10^{-4} mol/L range. Most of their γ_{cmc} values are below 24.0 mN/m, and some of them are even less than 21 mN/m, which denotes a high effectiveness in reducing the surface tension of water.

In general, the CMC and γ_{cmc} values of polyethoxylated surfactants increase with the increase of the number of ethylene oxide units, a trend which has been attributed to a stronger hydrophilicity and a more voluminous hydrophilic group [24]. Basically, except for the unobvious variation of the γ_{cmc} values of the **H** type trisiloxane surfactants (**1H**, **2H**, **3H** and **4H**), the variations of the CMC, γ_{cmc} and a_{m}^{s} values of the other types of the double-tail trisiloxane surfactants obey the above-mentioned rule, and corroborate the trend found for the trisiloxane surfactants [19]. The obviously lower γ_{cmc} values of the **H** type of double-tail

Table 3 Aqueous surface activity of double-tail trisiloxane surfactants

Surfactant	CMC (mol/L)	γ_{cmc} (mN/m)	Γ_{max} (mol/cm^{-2})	a_{m}^{s} (\AA^2)	ΔG_{mic}^0 (KJ/mol)	SA
1E	0.82×10^{-4}	20.9	41.51×10^{-11}	40.0	−34.3	3.15
2E	3.37×10^{-4}	23.9	24.67×10^{-11}	67.3	−30.7	2.64
3E	4.22×10^{-4}	22.8	23.35×10^{-11}	71.1	−30.0	2.18
4E	3.14×10^{-4}	24.1	26.41×10^{-11}	62.9	−30.8	1.63
1F	0.21×10^{-4}	21.2	45.21×10^{-11}	36.7	−37.5	3.80
2F	0.28×10^{-4}	22.6	33.05×10^{-11}	50.3	−36.7	2.25
3F	1.89×10^{-4}	23.7	31.92×10^{-11}	52.0	−32.0	1.76
4F	4.65×10^{-4}	25.8	33.51×10^{-11}	49.6	−29.8	1.69
1G	0.12×10^{-4}	20.5	35.56×10^{-11}	46.7	−33.0	3.06
2G	2.43×10^{-4}	23.5	31.44×10^{-11}	52.8	−31.2	2.18
3G	3.74×10^{-4}	21.1	28.75×10^{-11}	57.9	−29.9	1.44
4G	3.98×10^{-4}	23.8	26.29×10^{-11}	63.2	−29.7	1.05
1H	1.77×10^{-4}	20.5	34.53×10^{-11}	48.1	−32.2	2.33
2H	2.31×10^{-4}	20.7	34.53×10^{-11}	48.1	−31.5	1.44
3H	3.44×10^{-4}	20.9	32.31×10^{-11}	51.4	−30.5	1.40
4H	4.33×10^{-4}	20.5	29.40×10^{-11}	56.5	−29.7	1.11

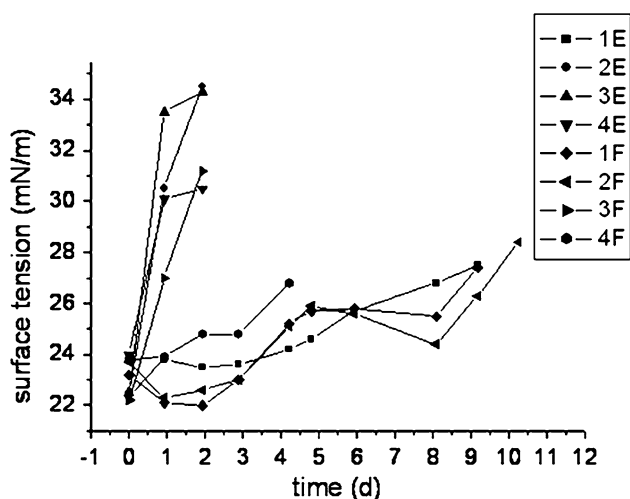


Fig. 1 Surface tension versus time plots for types **E** and **F** double-tail trisiloxane surfactants in pH 4.0 buffer

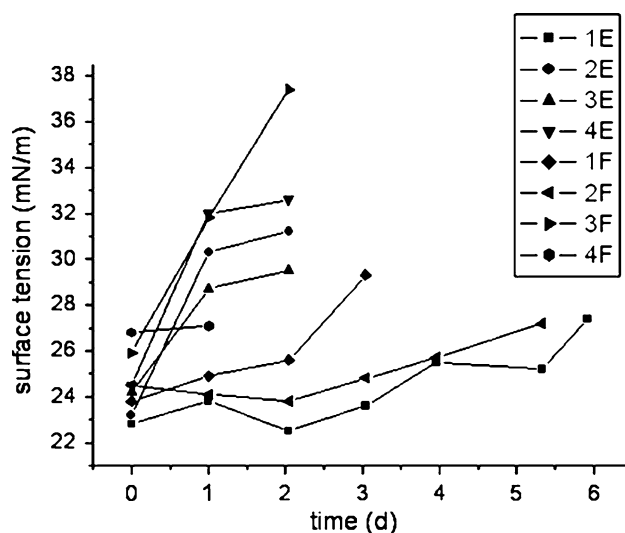


Fig. 3 Surface tension versus time plots for types **E** and **F** double-tail trisiloxane surfactants in pH 10.7 buffer

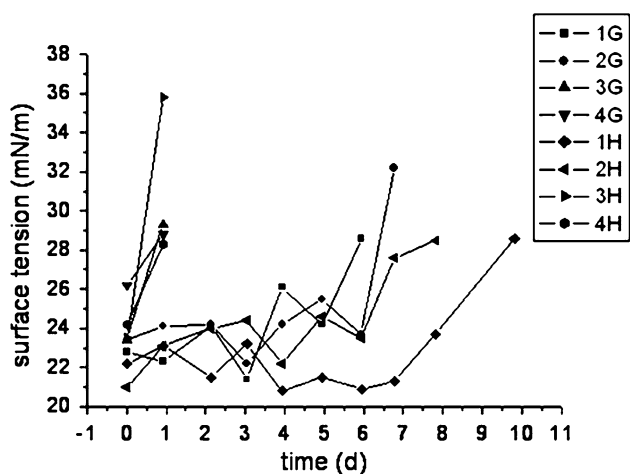


Fig. 2 Surface tension versus time plots for types **G** and **H** double-tail trisiloxane surfactants in pH 4.0 buffer

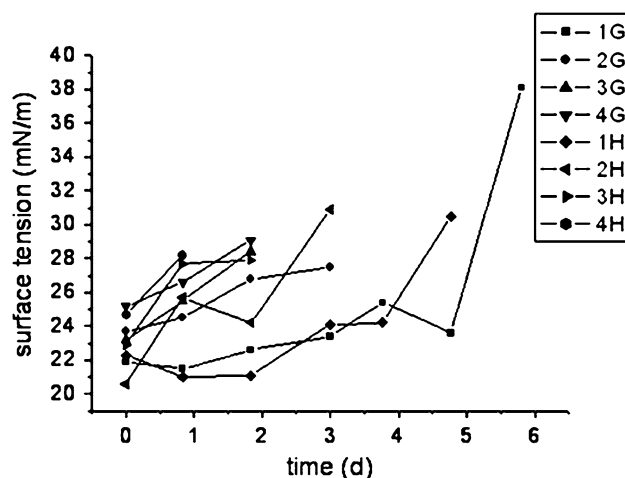


Fig. 4 Surface tension versus time plots for types **G** and **H** double-tail trisiloxane surfactants in pH 10.0 buffer

trisiloxane surfactants may be attributed to the additional low-energy methyl group in its hydrophobic groups.

Spreading Ability

The Spreading Ability (SA) values of the same type of the double-tail trisiloxane surfactants decrease with the increase of the number of ethylene oxide units (Table 3). The increase in ethylene oxide units make them bulkier and more polar, which results in a poorer SA of the trisiloxane surfactants [9]. We also noted that the SA performance of the double-tail trisiloxane surfactants with a lower γ_{cmc} value is not better than other species in the same type of the surfactants. This result is consistent with the literature [9]. As could be seen from Table 3, the SA of the double-tail trisiloxane surfactant, with the lowest CMC value, is the

strongest. Maybe it is easier to form special aggregates relevant to the turbidity of a surfactant solution [5–7]. The fact that the CMC value of **4E** is lower than that of **3E** indicates that the higher hydrophilicity of a double-tail surfactant molecule does not necessarily lead to a higher CMC value, and that other factors are also likely to affect the CMC of the double-tail surfactants.

For the different types of the double-tail trisiloxane surfactants, the γ_{cmc} values of **1G**, **2G**, **3G** and **4G** are higher than those of **1H**, **2H**, **3H** and **4H**, respectively. But the SA of the latter is superior to that of the former (Table 3). This result suggests that the presence of an additional methyl group is able not only to reduce the γ_{cmc} value of the **H** type of double-tail surfactants, but also to decrease the flexibility of the total hydrophobic group of

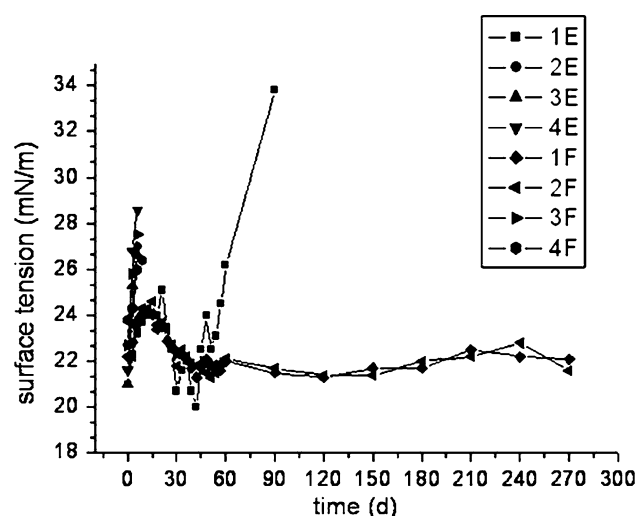


Fig. 5 Surface tension versus time plots for types **E** and **F** double-tail trisiloxane surfactants in pH 7.0 buffer

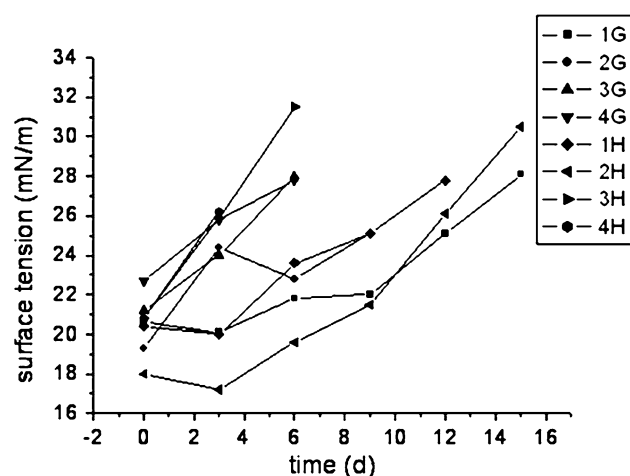


Fig. 6 Surface tension versus time plots for types **G** and **H** double-tail trisiloxane surfactants in pH 7.0 buffer

the surfactant. Consequently, the SA of **H** type of double-tail surfactants is poor.

By comparing the SA of the double-tail surfactants containing the same number of ethylene oxide units of the types **E** and **F**, little variations of their SA values are found, although the number of carbon atoms in their hydrophobic groups is not the same (Table 3). Consequently, it may be said that the influence of the number of carbon atoms in the hydrophobic groups of the double-tail trisiloxane surfactants on their SA is rather limited.

However, when compared with the **G** and **H** types of double-tail trisiloxane surfactants, the **E** and **F** types in which a flexible and hydrophilic spacer has been incorporated, exhibit an obviously improved SA (Table 3). This result demonstrates the impact of a flexible spacer on the SA of double-tail trisiloxane surfactants. Surfactant **1F** is found to have the highest SA in all prepared double-tail

trisiloxane surfactants. This feature may be attributed to its lowest CMC and the flexible spacer.

In contrast to the SA of the single-tail trisiloxane surfactants [19], the SA of the corresponding double-tail trisiloxane surfactants is relatively poor. This is mainly due to the decrease of the molar ratio of methyl group to methylene group in the latter hydrophobic groups, which leads to the increase in γ_{cmc} value. Additionally, the increase of a rigidly hydrophobic group of the latter trends to decrease the flexibility of the total surfactant molecule.

Hydrolysis Resistant Ability

As a whole, the hydrolysis resistant ability (HRA) of the same type of double-tail trisiloxane surfactants tends to decrease with an increase in the number of ethylene oxide units (Figs. 1, 2, 3, 4, 5, 6). However, in the different types of double-tail trisiloxane surfactants, the HRA of those surfactants having more carbon atoms (at constant ethylene oxide number) is higher, for example, the **F** and **E** types. The decrease in the number of ethylene oxide units or the increase in the number of carbon atoms in the hydrophobic group tends to weaken the hydrophilicity of a surfactant.

The HRA of **G** and **H** types of double-tail trisiloxane surfactants is better than those of the corresponding ones of **E** and **F** types in an acidic environment (pH 4.0), while it is poorer than those of the latter in alkaline or neutral pH solutions (Figs. 1, 2, 3, 4, 5, 6). This result suggests that the hydrolysis mechanism of the double-tail trisiloxane surfactants in an acidic environment is different from that in an alkaline or neutral environment.

According to previous results [19], the HRA of double-tail trisiloxane surfactants is obviously superior to that of the single-tail ones. For instance, the double-tail trisiloxane surfactant **1H** is stable for 8 days in an acidic environment (pH 4.0), and the surface tension values of aqueous solutions (0.1 wt.%) of the surfactants **1F** and **2F** are still less than 24.0 mN/m over 270 days in a neutral environment (pH 7.0). Therefore, it may be concluded that the incorporation of an additional hydrophobic group in the double-tail trisiloxane surfactants is able to improve their HRA.

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Author Biographies

Zhongli Peng is an associate professor of applied chemistry. He received a Ph.D. in Polymer Chemistry and Physics from the School of Chemistry and Chemical Engineering, Sun Yat-sen University, People's Republic of China. His research interests deal with organosilicon surfactants and emulsion (or microemulsion) preparation.

Caifeng Lu is an undergraduate student at Huizhou University. Her research involves the synthesis and application of surfactants.

Minling Xu is an undergraduate student at Huizhou University. Her research involves the synthesis and application of surfactants.