

Adsorption Behavior of Fatty Alcohol Ether Sulfonate at Different Interfaces

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Abstract The equilibrium surface tension, dynamic surface tension, and interfacial tension (IFT) of fatty alcohol ether sulfonates (C_mE_nSO) were measured to investigate their adsorption behavior. The effect of NaCl and $CaCl_2$ concentrations on the IFT was also studied. The results showed that the number of EO units has no significant effect on the critical micelle concentration (CMC) and CMC decreases with increasing the length of the hydrophobic group. The surface tension at the CMC increases with the increase of the number of EO units and the length of the hydrophobic group. At dilute surfactant concentration, the adsorption process for C_mE_nSO is controlled by diffusion; at higher concentration, it becomes a mixed diffusion-kinetic adsorption mechanism. The IFT between C_mE_nSO solution and dodecane remains around 10^{-1} mN/m over a wide range of electrolyte concentrations (NaCl concentration from 25 to 210 g/L, $CaCl_2$ concentration from 0.1 to 10 g/L).

Keywords Adsorption behavior · Anionic-nonionic surfactants · Surface tension · Interfacial tension

Introduction

Surfactant flooding is an important enhanced oil recovery (EOR) method. Injecting surfactant solution helps to mobilize the residual crude oil from the reservoir by reducing the oil–water interfacial tension (IFT) [1–9]. During surfactant flooding, traditional anionic surfactants such as petroleum sulfonate and alkylbenzene sulfonate have been employed for common oil reservoirs [10–14]. Unfortunately, high levels of divalent ions (Ca^{2+} and Mg^{2+}) in the connate water in some petroleum reservoirs could make these anionic surfactants precipitate, so it is important to develop anionic surfactants for use in high salinity oil reservoirs [10]. Ethoxylated sulfonates, as a kind of anionic-nonionic surfactant, have a higher resistance to electrolytes and chemical stability, and these excellent properties have attracted wide interest. However, only a few publications have systematically studied the adsorption behavior of fatty alcohol ether sulfonate at different interfaces [10, 15–23].

The aim of this work was to investigate the surface and interfacial tension of fatty alcohol ether sulfonates (C_mE_nSO). The effects of the number of ethylene oxide (EO) units, the length of the hydrophobic group, and the electrolytes (NaCl and $CaCl_2$) on the adsorption behavior of C_mE_nSO are systematically discussed.

Experimental Sections

Materials

Fatty alcohol ethoxylate was kindly supplied by Sinolight Chemicals Co., Ltd, China. Sodium isethionate was supplied by Huanggang Yongan Pharmaceutical Co., Ltd.,

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China. Ethyl acetate, ethanol, dodecane, KOH, CaCl₂, and NaCl were obtained from Tianjin Kemiou Chemical Reagent Co., Ltd., China. All materials were used as received.

Synthesis

A 500-mL flask was charged with 0.3 mol fatty alcohol ether, 0.03 mol KOH, and 80 mL cyclohexane. The mixture was heated under reflux for 2 h to remove H₂O. Then 0.25 mol sodium isethionate and 80 mL *n*-decane were added and the mixture was heated at reflux for 90 min [21]. The synthesis of C_{*m*}E_{*n*}SO is shown in Scheme 1, in which R is the hydrophobic chain of C_{*m*}H_{2*m*+1}, *n* is the number of EO units, and *m* is the number of carbon atoms in the hydrophobic chain. For C₁₂₁₄E_{*n*}SO, the content of C₁₂ and C₁₄ in the mixture of C₁₂₁₄ is about 75 and 25%, respectively.

About 5.0 g crude product was dissolved in a mixture of 100 mL water and 50 mL ethanol. Then the mixed solution was extracted with 50 mL ethyl acetate in a separatory funnel. The top layer was removed, and the bottom layer was extracted successively five times with 50 mL ethyl acetate. The bottom layer was evaporated to remove water and ethanol, and then the remaining was filtered and washed with ethanol to desalt it [10, 21]. The purity of the product was above 90%.

Structure Characterization

Figure 1 shows the negative ion MS of C_{*m*}E_{*n*}SO ionized by ESI. Taking C₁₆E₆SO as an example (Fig. 1d), the molecular mass of C₁₆E₆SO was 328 + 44*p*, where *p* represents the actual number of EO units in C_{*m*}E_{*n*}SO. Figure 1d shows the ions at *m/z* 328 + 44*n* – 23, corresponding to the molecule [M – Na][–], which indicates that the number of EO units in C₁₆E₆SO varies from 1 to 16.

Surface Tension Measurements

The measurements of static surface tension were carried out on the KRÜSS K12 processor tensiometer at 25.0 ± 0.1 °C. Surfactant solutions were prepared using deionized doubly distilled water.

The dynamic surface tension (DST) was determined using a KRÜSS bubble pressure tensiometer BP100 method at 25.0 ± 0.1 °C.

Interfacial Tension Measurements

IFT of surfactant aqueous solution/dodecane was measured using a TX-500C interfacial tensiometer at 30.0 ± 0.1 °C.

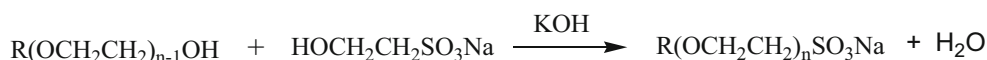
Results and Discussion

Equilibrium Surface Tension

The surface tensions of the aqueous solutions of C_{*m*}E_{*n*}SO were measured to evaluate their surface activities. The plots of surface tension (γ) versus log surfactant molar concentrations (log*C*) of C_{*m*}E_{*n*}SO are shown in Fig. 2, from which it can be seen that the surface tension initially decreases with increasing surfactant concentration, and then a plateau appears. The critical micelle concentration (CMC) is recorded as at the break point of the two linear portions of the γ versus log*C* plots. Table 1 lists the surface properties of C_{*m*}E_{*n*}SO obtained from these plots. The CMC of C_{*m*}E₆SO decreases with increasing number of carbon atoms in the hydrophobic group, which is similar to the traditional surfactants. The CMC increases with increasing number of EO units for typically nonionic surfactants containing EO units [24]. However, a slight decrease in CMC with an increase in the number of EO units is observed for the C_{*m*}E_{*n*}SO surfactants. Micelle formation is the result of two opposing forces. The higher the number of EO units is, the stronger the hydrophilicity which inhibits micelle formation. However, introduction of EO groups increases the molecular length, which increases the surface area of micelle and decreases the surface charge density of the micelle, which contributes to micelle formation. The net effect of increasing EO leads to no significant effects on the CMC with increasing the number of EO units.

The γ_{cmc} of C_{*m*}E_{*n*}SO increases with increasing the number of EO units and the length of the hydrophobic group because the higher the number of EO units is, the larger the volume of the hydrophilic group, which leads to a looser arrangement of surfactant molecular at the air/water interface [10]. When the number of carbon atoms in a straight-chain hydrophobic chain exceeds 16, the “–CH₃” density decreases and γ_{cmc} increases because of the coiling of the long chain [24].

Surface excess concentration (Γ_{max}) in mol/cm² and minimum area per molecule (A_{min}) in Å² were calculated from the following equations [24]:



Scheme 1 Synthetic route of C_{*m*}E_{*n*}SO

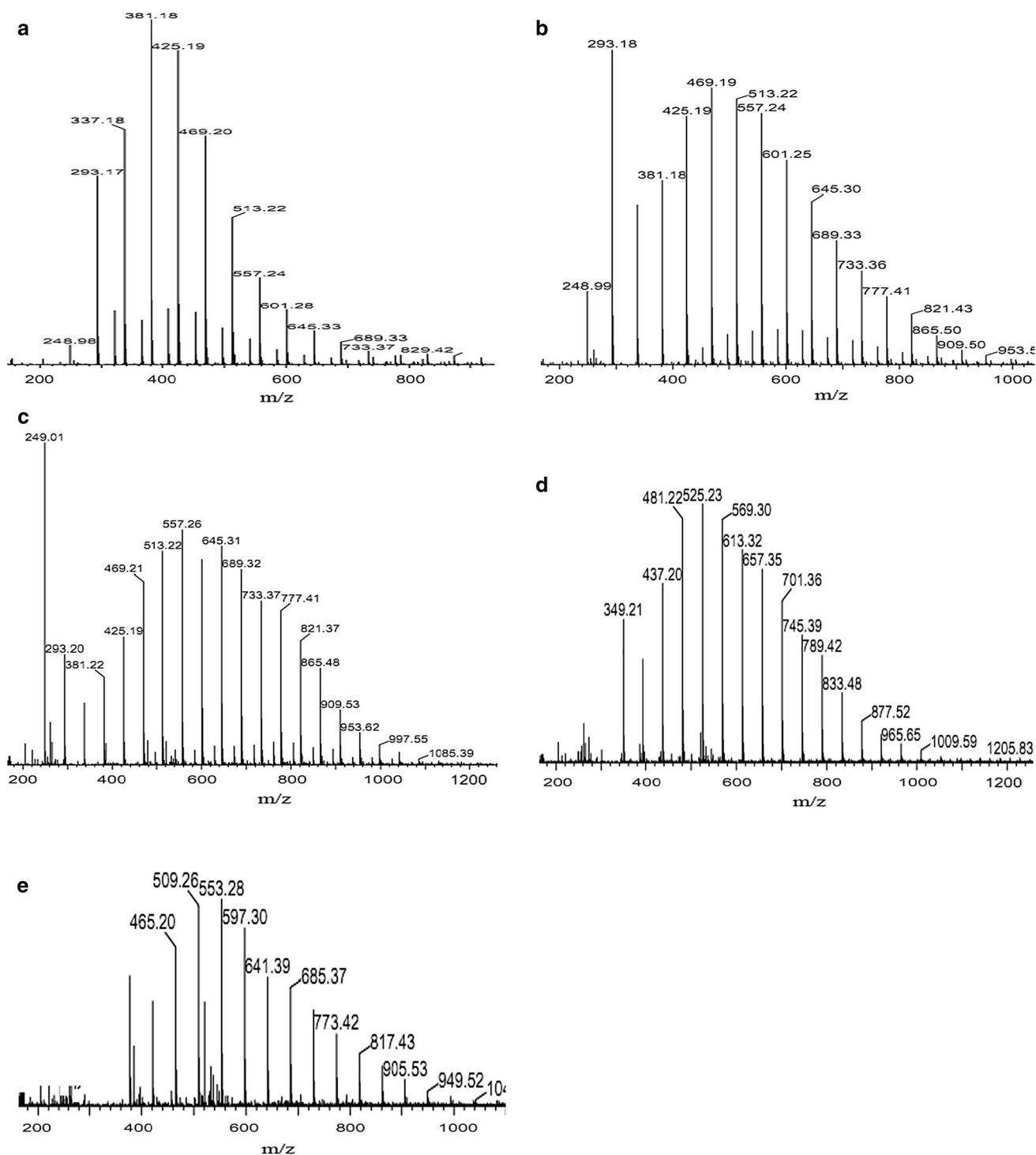


Fig. 1 ESI-MS of C_mE_nSO : **a** $C_{1214}E_4SO$, **b** $C_{1214}E_6SO$, **c** $C_{1214}E_8SO$, **d** $C_{16}E_6SO$, **e** $C_{18}E_6SO$

$$\Gamma_{\max} = -\frac{1}{4.606RT} \left(\frac{d\gamma}{d\log C} \right)_T, \quad (1)$$

$$A_{\min} = \frac{10^{16}}{N_A \Gamma}, \quad (2)$$

where R is $8.31 \text{ J mol}^{-1} \text{ K}^{-1}$, N_A is Avogadro's constant, T is the absolute temperature, and $d\gamma/d\log C$ is the slope below the CMC from the surface tension isotherms. Γ_{\max} is the maximum adsorption quantity of surfactant at the air/

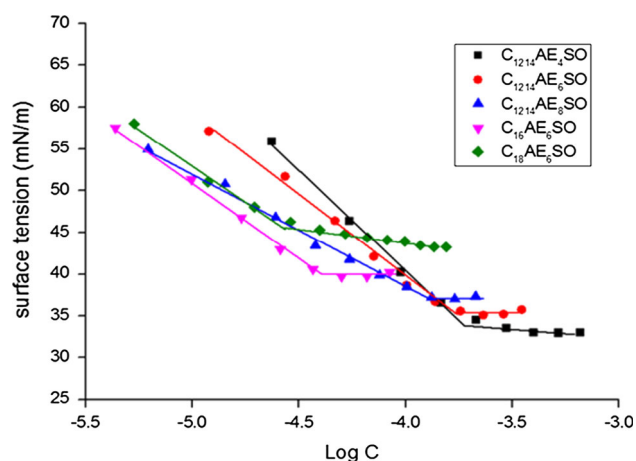


Fig. 2 Surface tension versus log molar concentration of C_mE_nSO

water interface. A_{\min} gives information on the degree of packing of surfactant molecules at the air/water interface. As can be seen from Table 1, as the number of EO units and the number of carbon atoms in the hydrophobic group of C_mE_nSO increase, A_{\min} increases and Γ_{\max} decreases. The reasons are as follow: the EO chain immerses in the aqueous solution in the form of a coil, whose cross-sectional area increases with increasing the number of EO units [10, 24]; coiling of the long chain occurs when the number of carbon atoms in the hydrophobic chain exceeds 16, with a consequent increase in cross-sectional area of the molecule.

C_{20} is the concentration of surfactant required to produce a 20 mN/m reduction in the surface tension of water, and the negative logarithm of the C_{20} (pC_{20}) represents the efficiency of adsorption of surfactant [10, 24]. The larger the pC_{20} value is, the more efficiently the surfactant is adsorbed and the surface tension is reduced [10, 24]. As listed in Table 1, the pC_{20} for C_mE_nSO increases as the number of EO units increases. The pC_{20} for C_mE_nSO has a maximum value when the length of the hydrophobic group increases up to 16 carbon atoms.

The CMC/C_{20} ratio shown in Table 1 is an index of micellization relative to adsorption onto the water/air interface [10, 24]. It can be seen that the CMC/C_{20} ratio for C_mE_nSO increases with increasing the number of EO units, which means that increasing the number of EO units inhibits micellization more than adsorption. The CMC/C_{20}

ratio for C_mE_nSO decreases with increasing the number of carbon atoms in the hydrophobic group, which means that an increase in the length of hydrophobic group facilitates micellization more than adsorption. Usually, anionic surfactants with a single straight-chain hydrophobic group show low CMC/C_{20} ratios of 3 or less, while polyoxyethylene nonionic surfactants show ratios of about 7 or more [24]. From Table 1, it can be seen that increasing the number of EO units in C_mE_nSO increases their nonionic surfactant characteristics, while increasing the length of the alkyl chain of C_mE_nSO increases their ionic surfactant characteristics.

Dynamic Surface Tension (DST)

The DST as a function of time for C_mE_nSO is shown in Fig. 3, from which it can be seen that the time required to attain the equilibrium surface tension decreases with increasing surfactant concentration. For $C_{1214}E_nSO$ solution of very low concentration, there is an induction region at the beginning; whereas, for $C_{16}E_6SO$ and $C_{18}E_6SO$, there is still an induction region even at higher surfactant concentration such as 5 mM. In addition, when the surfactant concentration is the same, the induction region becomes smaller with increasing the number of EO units and decreasing the length of the hydrophobic group.

Apparent Diffusion Coefficient

The Ward and Tordai model is usually used to analyze the DST. Equation (3) allows one to quantitatively analyze the process:

$$\Gamma(t) = 2C_0\sqrt{\frac{Dt}{\pi}} - 2\sqrt{\frac{D}{\pi}} \int_0^{\sqrt{t}} C_s d(\sqrt{t-\tau}), \quad (3)$$

where $\Gamma(t)$ is the surface excess concentration at time t , D is the apparent diffusion coefficient, C_0 is the bulk surfactant concentration, C_s is the concentration in the sub-surface, and τ is a dummy variable of integration [10, 25, 26]. Values of D at short time and long time could be obtained from Eqs. (4) and (5), respectively,

$$\text{Short time: } \gamma(t)_{t \rightarrow 0} = \gamma_0 - 2nRTC_0\sqrt{\frac{Dt}{\pi}}, \quad (4)$$

Table 1 Surface properties of C_mE_nSO

Surfactant	γ_{cmc} (mN/m)	CMC (mol/L)	A_{\min} (\AA^2)	Γ_{\max} ($10^{10} \times \text{mol/cm}^2$)	pC_{20}	CMC/C_{20}
$C_{1214}E_4SO$	33.87	1.91×10^{-4}	78	2.14	4.48	5.77
$C_{1214}E_6SO$	35.38	1.74×10^{-4}	98	1.70	4.59	6.77
$C_{1214}E_8SO$	37.31	1.29×10^{-4}	143	1.16	4.95	11.50
$C_{16}E_6SO$	39.97	4.07×10^{-5}	106	1.56	5.05	4.57
$C_{18}E_6SO$	45.60	2.69×10^{-5}	111	1.50	4.98	2.57

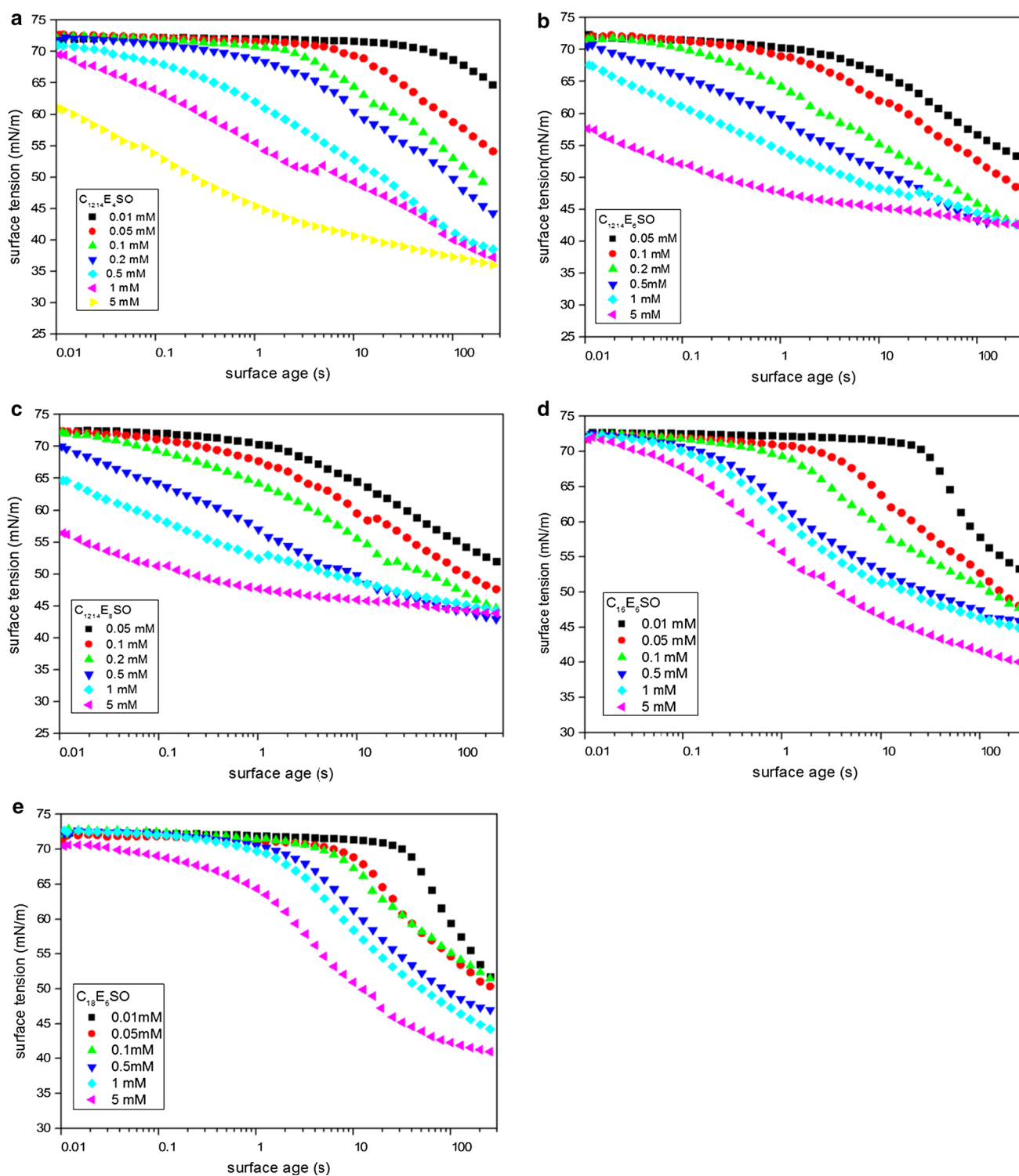


Fig. 3 Dynamic surface tension of C_mE_nSO solutions with various concentrations: **a** $C_{1214}E_4SO$, **b** $C_{1214}E_6SO$, **c** $C_{1214}E_8SO$, **d** $C_{16}E_6SO$, **e** $C_{18}E_6SO$

$$\text{Long time: } \gamma(t)_{t \rightarrow \infty} = \gamma_{eq} + \frac{nRT\Gamma_{eq}^2}{C_0} \sqrt{\frac{\pi}{4Dt}}, \quad (5)$$

where R is $8.31 \text{ J mol}^{-1} \text{ K}^{-1}$, C_0 is the surfactant concentration, T is the absolute temperature, $\gamma(t)$ is the surface

tension at time t , γ_0 is the surface tension of the solvent, γ_{eq} is the equilibrium surface tension at infinite time, $n = 2$ for ionic surfactant, and Γ_{eq} is the equilibrium surface excess concentration [10, 26–28]. Figure 4 shows the plots of DST versus t , $t^{1/2}$ (short time) and $t^{-1/2}$ (long time) for

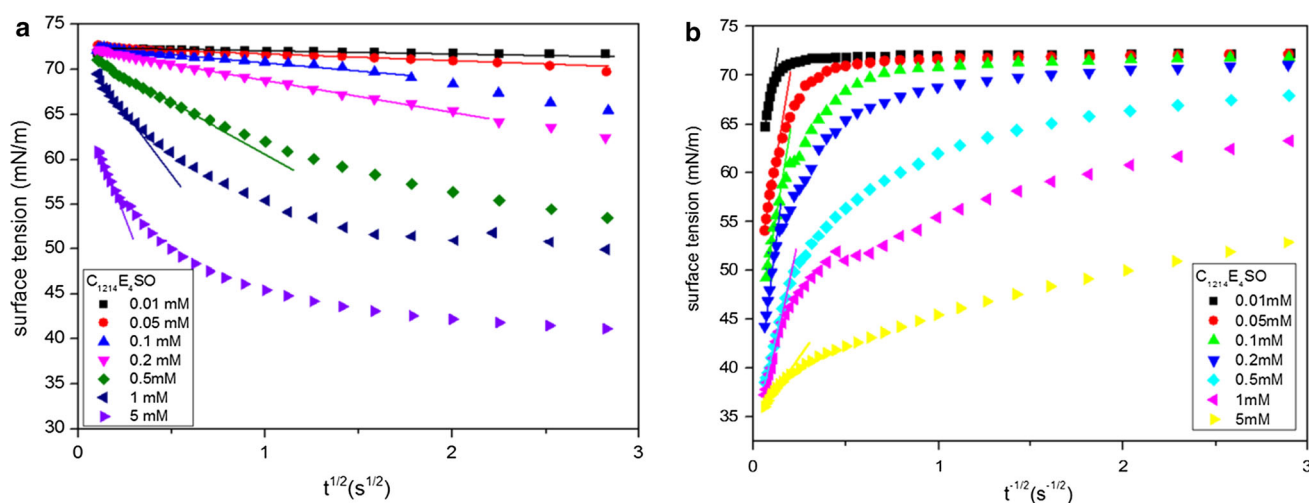


Fig. 4 Dynamic surface tension of $C_{1214}E_4SO$ as a function of **a** short-time ($t^{1/2}$) and **b** long-time ($t^{-1/2}$)

$C_{1214}E_4SO$ at different concentrations, and it can be seen that these plots exhibit linear behavior. Similar plots were obtained for the other C_mE_nSO (not shown).

The apparent diffusion coefficients of C_mE_nSO with various concentrations obtained from the gradients of the plots based on Eqs. (4) and (5) are shown in Fig. 5. Figure 5a shows that for $C_{1214}E_nSO$ the diffusion coefficients at short time (D_{short}) increase first and then decrease with increasing the concentration, while for $C_{16}E_6SO$ and $C_{18}E_6SO$ the D_{short} decreases with increasing the concentration. In addition, at the same concentration the D_{short} increases with increase of the number of EO units, and decreases with increase of the number of carbon atoms in the hydrophobic group. Figure 5b shows that the diffusion coefficients of C_mE_nSO decrease almost linearly with increasing the concentration. The ratio of D_{long}/D_{short} could reflect the adsorption mechanism. When the ratio is relatively close to 1, the adsorption process is diffusion-controlled. When it is far less than 1, the adsorption process is a mixed diffusion-kinetic adsorption mechanism [29, 30]. As seen in Fig. 5c, the ratio of D_{long}/D_{short} decreases with increasing the concentration when the concentration is less than 1 mM, and only at low concentration is the ratio close to 1, which indicates that there is a significant adsorption barrier with increasing the concentration of C_mE_nSO .

Interfacial Tension

Figure 6a shows the influence of NaCl concentration on IFT between C_mE_nSO solutions and dodecane. The results shows that IFT decreases with increasing NaCl concentration because NaCl could decrease the electrostatic repulsive force between intermolecular headgroups [10, 20]. As a result, C_mE_nSO concentration increases at

the dodecane/water interface, which leads to the decrease of IFT. For $C_{1214}E_nSO$, the IFT increases with the increase of the number of EO units because of increasing the cross-sectional area of C_mE_nSO . The effect of increasing NaCl concentration on IFT decreases with increasing the number of EO units because the higher the number of EO units is, the more obvious the nonionic surfactant characteristic is, which is insensitive to electrolyte. For C_mE_6SO , the IFT decreases first and then increases with increasing the length of the hydrophobic group, which may contribute to the coiling of the long chain of $C_{18}E_6SO$, with a consequent increase in cross-sectional area of the molecule and decrease in interaction between surfactant and oil.

Figure 6b shows the influence of concentration of $CaCl_2$ on IFT. It can be seen that the IFT does not significantly change with increasing the $CaCl_2$ concentration from 1 to 10 g/L. The trend of the change of IFT for the $C_mE_nSO/CaCl_2$ /dodecane system is similar that of the $C_mE_nSO/NaCl$ /dodecane system with increasing the number EO units and the length of the hydrophobic group. Usually, the IFT has a minimum value in a certain electrolyte concentration [20, 31]. However, it is worth noting that the IFT of C_mE_nSO has no minimum value except for $C_{16}E_6SO$, which indicates that the IFT of C_mE_nSO is not sensitive to electrolyte concentration because C_mE_nSO possess more nonionic surfactant characteristics than ionic surfactant characteristics as a result of the EO group.

Thermal Stability

Thermal stability of surfactants is a material property because the surfactants will degrade at high temperature which leads to loss of surface activity [32]. The TG curves

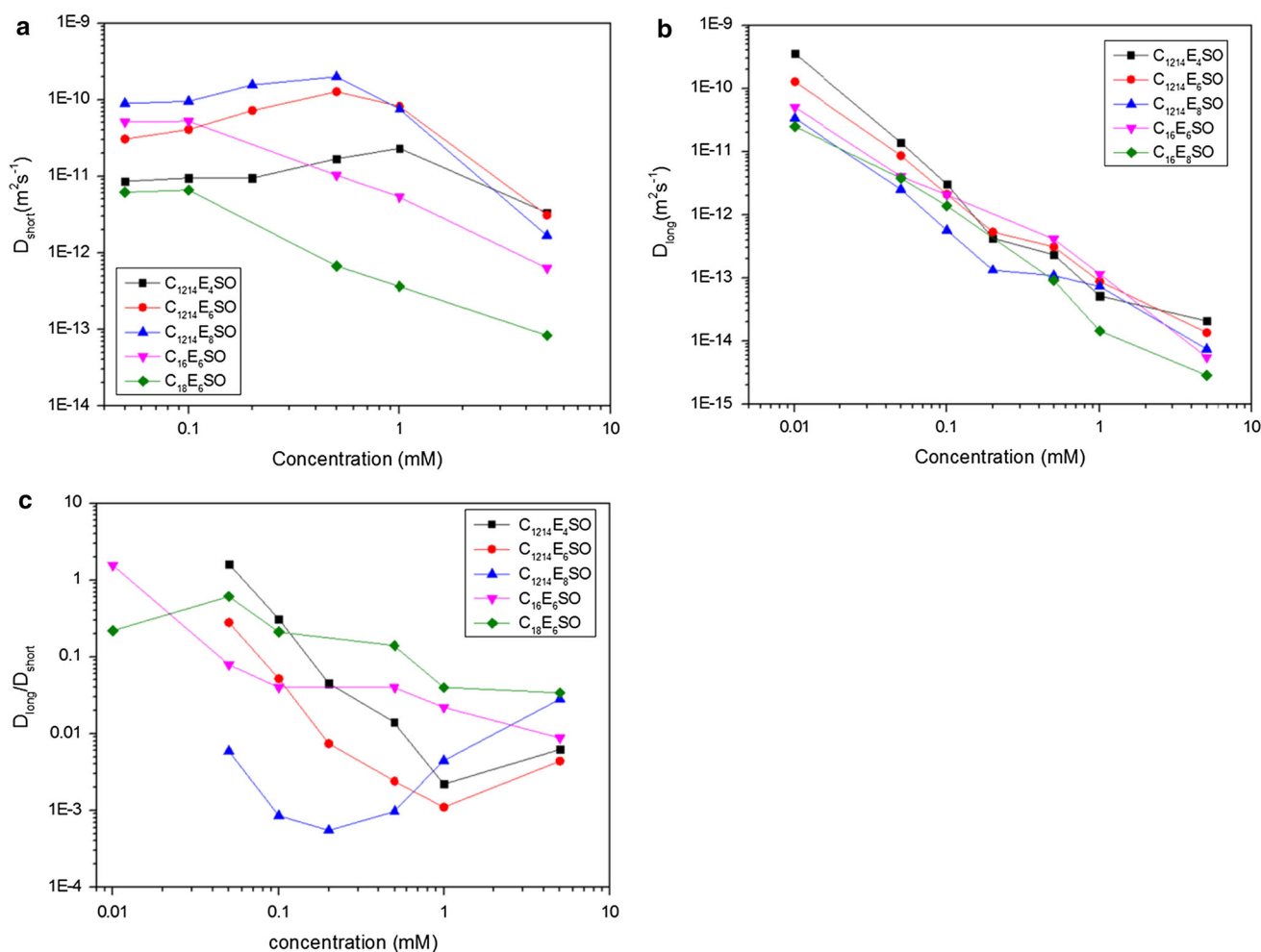


Fig. 5 Apparent diffusion coefficients of C_mE_nSO with different concentrations, **a** D_{short} , **b** D_{long} , and **c** D_{long}/D_{short}

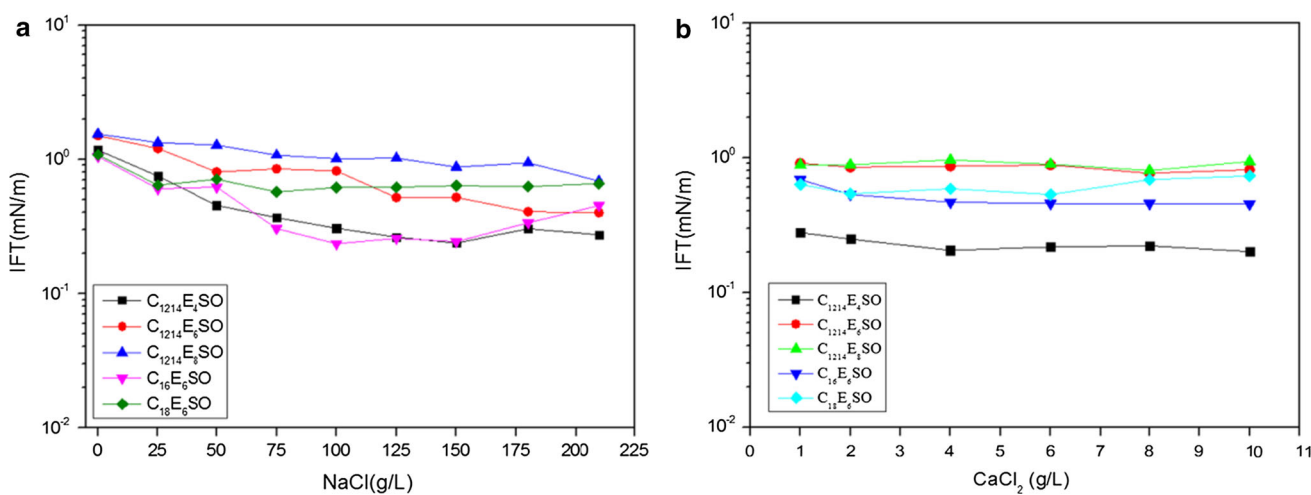


Fig. 6 Effect of electrolytes concentration on IFT between C_mE_nSO solutions and dodecane

of C_mE_nSO under nitrogen are shown in Fig. 7, from which it can be seen that three stages of degradation are evident. The second stage corresponds to rapid decomposition.

Thermal stability of C_mE_nSO decreases in the order $C_{1214}E_8SO > C_{1214}E_6SO = C_{16}E_6SO = C_{18}E_6SO > C_{1214}E_4SO$. The C_mE_nSO with the longer EO chain exists in the form of

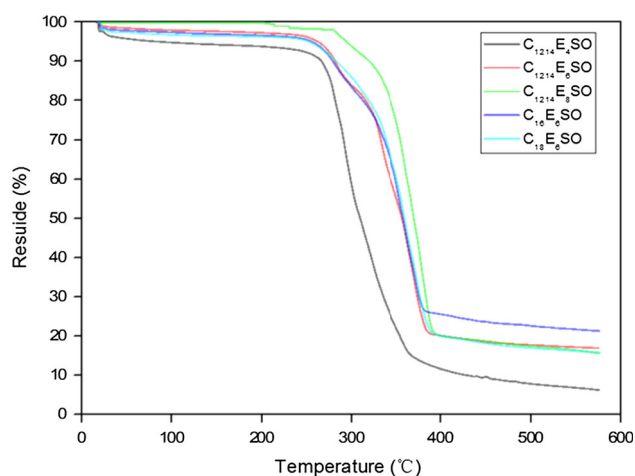


Fig. 7 Thermogravimetric analysis of C_mE_nSO at 10 °C/min under nitrogen

a coil, which inhibits the thermal decomposition. Thus, the main factor that affects the thermal stability of C_mE_nSO is the number of EO units rather than the length of carbon chain.

Conclusion

The surface tension and interfacial tension of fatty alcohol ether sulfonates (C_mE_nSO) at the air/water interface and dodecane/water interface were investigated. The main results can be concluded as follows:

1. Increasing the length of the hydrophobic group of C_mE_nSO leads to decreasing the CMC and increasing the γ_{CMC} and A_{min} ; increasing the number of EO units has no significant effect on CMC and leads to increasing the γ_{CMC} and A_{min} .
2. At lower concentration, the adsorption process is diffusion-controlled; at higher concentration, a mixed diffusion-kinetic mechanism controls the adsorption process.
3. The IFT increases with an increase of the number of EO units, and decreases first and then increases with increasing the length of the hydrophobic group at the studied concentrations.
4. Thermal stability of C_mE_nSO increases in the order $C_{12}E_4SO < C_{12}E_6SO = C_{16}E_6SO = C_{18}E_6SO < C_{12}E_8SO$.

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