Influence of Inorganic Salts and pH on Surface Activity of Biodegradable Amido Gemini Amine Oxide Surfactant

Duojiao Fu, Xinru Jia, Ran Wei, Hongqin Liu*, and Baocai Xu

School of Light Industry, Beijing Key Laboratory of Flavor Chemistry, Beijing Higher Institution Engineering Research Center of Food Additives and Ingredients, Beijing Technology and Business University, No. 11 Fucheng Road, Beijing 100048, People’s Republic of CHINA

Abstract: The present study was designed to provide information regarding the influences of pH and inorganic salts on the surface activity of the amido Gemini amine oxide surfactants, the formula of which was $\text{C}_n\text{H}_{2n+1}\text{CONH(CH}_3\text{)}_3\text{N}^+\text{O}^-\text{(CH}_3\text{)}_2\text{-(CH}_3\text{)}_2\text{N}^+\text{O}^-\text{(CH}_3\text{)}_2\text{HNCOC}_n\text{H}_{2n+1}$ ($n=11, 13, 15, \text{and } 17$). Their biodegradability was also investigated. The results showed that the critical micelle concentration (CMC) and the corresponding surface tension ($\gamma_{\text{CMC}}$) changed regularly with the increase of the mass concentration of NaCl and CaCl$_2$. The optimal CMC and $\gamma_{\text{CMC}}$ can be obtained by adjusting the inorganic salt content. Besides, the CMC near the isoelectric point of the surfactant is higher than the CMC within the isoelectric point. Furthermore, the biodegradability test revealed that the prepared surfactants had good biodegradability which was above 96% after 7 days.

Key words: amido Gemini amine oxide surfactant, biodegradability, surface activity, pH and inorganic salts responsive

1 Introduction

Gemini surfactants, which consist of two hydrophobic chains and two hydrophilic groups in a molecule, have the characteristics of low critical micelle concentration (CMC), great efficiency in reducing the surface tension of water, stronger synergistic effect with the combination of monomer surfactant, good solubilization, wettability and foaming properties$^{1, 2}$. Because Gemini surfactants have more excellent physical and chemical properties, they have been rapidly developed in various fields such as petrochemicals, nanomaterials and mesoporous materials, soil remediation, detergents, and biomedical applications$^{2, 3, 4}$. The use of the surfactants has been firmly raised with the increasing demand for a wide range of industrial, household and cosmetic products$^8$. However, the surfactants remaining in water or soil can be detrimental for aquatic flora and fauna, microorganisms, terrestrial plants and animals, and even humans, such as the destruction of the function and structure of organism membranes, extensive damage to seedling growth, activation or inhibitory effect on enzymes, and disrupting normal physiological functions$^{5-12}$. For these reasons, when the new surfactants are designed and synthesized, it is very necessary to consider their biodegradability, and the surfactants with less pollution to the environment are more likely to be commercialized. Some studies have shown that the surfactants containing amide bond have better biodegradability, because they have corresponding enzymes to biodegrade them in nature$^{3, 13}$. With the increasing application of Gemini surfactants, the study of biodegradability of Gemini surfactants has been paid more attention. Gawali et al. exploited a series of new imidazoline cationic Gemini surfactants and their biodegradability could reach about 60%-70% in 28 days$^{14}$. Tehrani-Bagha et al. investigated a series of cationic Gemini surfactants with ester bonds inserted between the hydrocarbon tails, while the results clearly showed that the Gemini surfactants were more resistant to biodegradation and could not be classified as readily biodegradable. Furthermore, the authors of this study reported another novel type of cationic Gemini surfactants with an ester bond inserted into a short spacer and found that structural modification in the spacer of Gemini surfactants appears to be the key to the biodegradability of surfactants$^{15}$. Although some Gemini surfactants can be classified as readily biodegradable, the biodegradation rate is difficult to reach 90%, and there are few reports on the biodegradability of Gemini amine oxide surfactant.

Amine oxide surfactants are a type of amphoteric surfa-
tants, due to the existence of ionizable groups and interaction of protonation, charge reversal or chemical bonding, amine oxide surfactants have pH-responsive behavior affecting their surface activity. Thus, great interests have been shown in the study of the surface activity of amine oxide surfactants at a range of pH. Besides, the addition of salts is a convenient approach to tune the aggregation behavior of surfactants through changing the electrostatic interaction of ionic headgroups. Compared to traditional single chain amphoteric surfactants, Gemini surfactants have a lower tolerance to inorganic salts because of the stronger chemical interactions with each other via the headgroup. The increasing number of headgroups not only provides unique aggregation behavior and interfacial properties for surfactants but also offers multiple interaction sites with their opposite charged additives.

In our previous works, it was found that the prepared amido Gemini amine oxide surfactants with the formula C\(_{1n}\)H\(_{2n+1}\)CON(H\(_2\))\(_n\)O\(^-\)(CH\(_2\))\(_x\)-(CH\(_2\))\(_y\)-O\(^-\)(CH\(_2\))\(_n\)NHCOCH\(_3\), \(n = 11, 13, 15,\) and 17\) had high surface activities and were highly effective aqueous surfactants. In this paper, their surface properties are further explored. The effect of pH and inorganic salt on the surfactant properties is also investigated. The results can be useful for the selection of technically efficient surfactants with less environmental impact.

### 2 Material and Methods

#### 2.1 Materials

Sodium hydroxide, hydrochloric acid, sodium chloride, calcium chloride, acetic acid, sodium acetate, disodium hydrogen phosphate, sodium dihydrogen phosphate and boric acid, A.R., were purchased from Sinopharm Chemical Reagent Beijing Co., Ltd. \(n\)-Decane and benzophenone, A.R., were provided by J&K Scientific Ltd. The high-purity water (\(ρ = 18.25 \text{ MO} \cdot \text{cm}^{-1}\)) was obtained by the ultrapurity laboratory water purification system.

#### 2.2 Synthesis of amido Gemini amine oxides surfactants

These four amido Gemini amine oxides surfactants were synthesized according to our previous paper, which are shown in Table 1.

#### 2.3 Biodegradability of amido Gemini amine oxide surfactant

The biodegradability of these amido Gemini amine oxide surfactants was determined according to the Chinese standard GB/T 15818-2018 test method for biodegradability of surfactants. 3.0 g ammonium chloride, 1.0 g dipotassium phosphate, 0.25 g magnesium sulfate, 0.25 g potassium chloride, 0.002 g ferrous sulfate and 0.3 g yeast extract were dissolved in 1000 mL water to prepare a basic nutrient solution. The concentration of the active sludge suspensions was 10,000-20,000 mg/L. 0.1 g synthesized compound or test of reference was dissolved in 100 mL water. 5 mL test solution and 500 mL basic nutrient solution were introduced into the degradation bottle, which were vibrated for 7 days at 25 ± 0.3°C, then the biodegradability was measured.

The calculation formula of the surfactant biodegradability \(D\) was as follows:

\[
D = \frac{ρ_0 - ρ_x}{ρ_0} \times 100
\]

where \(ρ_0\) is the initial concentration of amido Gemini amine oxides surfactants before degradation, mg/L; \(ρ_x\) was their concentration after degradation \(x\) time, mg/L, which was determined by spectrophotometry using orange II dye. The complex compound formed by zwitterionic surfactant and orange II dye under the buffer condition of pH = 1 was soluble in chloroform, and then the absorbance of the test solution was measured by a spectrophotometer in a 485 nm wavelength.

#### 2.4 Surface tension measurements

The surface tension measurements were carried out by using the Delta-8 high-throughput surface tension meter at 25°C. The 4 target products were prepared into an aqueous solution with a concentration of 0.001 mol·L\(^{-1}\) with high-purity water, respectively, and then kept in a water bath at 25°C for 40 min. The measurement was repeated 3 times for each sample. The measured value of each sample was the average of triplicate measurements, and the standard deviation was less than 0.02 mN·m\(^{-1}\).

#### 2.5 Surface tension measurements under different inorganic salt concentrations

The aqueous solutions of NaCl and CaCl\(_2\) with different concentrations (1, 2, 3, 5, 7, 9 and 10 mg·L\(^{-1}\)) were prepared, and then were used to prepare surfactants aqueous solutions with a concentration of 0.001 mol/L. The pre-
pared solution was dispersed by ultrasonic for 1 h, and then left standing at constant temperature for 2 h. Afterwards, the surface tension was measured at 25°C and the relevant parameters were calculated.

2.6 Surface tension measurements under different pH values

The pH values of the amido Gemini amine oxide surfactant solution (concentration of 0.001 mol/L) were adjusted to 4.05, 7.45 and 10.15 with the buffer solutions of acetic acid/sodium acetate (0.2 mol/L, v/v = 2:1), disodium hydrogen phosphate/sodium dihydrogen phosphate (0.05 mol/L, v/v = 1:4), and borax (0.05 mol/L)/sodium hydroxide (0.2 mol/L) (v/v = 1:1), respectively. Afterwards, the surface tension was measured at 25°C and the relevant parameters were calculated.

3 Results and Discussion

3.1 Biodegradability

In general, the mechanism of amine oxide surfactants biodegradation may follow two paths: a) terminal methyl group undergoes ω-oxidation to obtain ω-amino fatty acid, and then deaminates to the intermediates amine and ω-oxo fatty acid, or b) the intermediates alkanals and fatty acids are formed due to the fission of the C-N bond between amine group and alkyl chain, and finally CO₂ and NH₃ are generated through the following β-oxidation and N-de-methylation. The biodegradation of the synthesized amido Gemini amine oxide surfactants after degradation of 7 days at 25 ± 0.3°C is given in Table 2. We can see that all these prepared amido Gemini amine oxide surfactants have good biodegradability, and the biodegradation after 7 days is above 96%. So, they could be classified as easily biodegradable due to their high degradation rate obtained under the rigorous experiment process. Besides, it is worth noting that the biodegradability increases slightly with their hydrophobic carbon chain growth. From Table 3, we can also see that the CMC values of these amido Gemini

| Amido Gemini amine oxide surfactant | Absorbance[a] | Surfactant concentration after degradation (μg/mL) | Biodegradability D (%) |
|-------------------------------------|--------------|---------------------------------------------------|------------------------|
| C₁₂-2-C₁₂                          | 1.51         | 33.35                                             | 96.73                  |
| C₁₄-2-C₁₄                          | 1.37         | 30.27                                             | 96.97                  |
| C₁₆-2-C₁₆                          | 1.15         | 25.42                                             | 97.46                  |
| C₁₈-2-C₁₈                          | 0.89         | 19.70                                             | 98.03                  |

[a] measured in 485 nm wavelength.

| Inorganic salt | Salinity (mg/mL) | CMC × 10⁻⁵ (mol/L) | γ_{CMC} (mN/m) |
|----------------|------------------|-------------------|---------------|
|                | C₁₂-2-C₁₂ | C₁₄-2-C₁₄ | C₁₆-2-C₁₆ | C₁₈-2-C₁₈ | C₁₂-2-C₁₂ | C₁₄-2-C₁₄ | C₁₆-2-C₁₆ | C₁₈-2-C₁₈ |
| NaCl 0         | 3.98     | 9.12     | 12.60     | 15.00     | 28.13    | 30.14    | 32.88    | 34.81    |
| NaCl 1         | 3.89     | 9.07     | 12.03     | 14.59     | 27.66    | 29.17    | 31.53    | 34.21    |
| NaCl 3         | 2.89     | 8.91     | 11.86     | 14.31     | 27.06    | 28.89    | 31.15    | 33.97    |
| NaCl 5         | 2.72     | 7.54     | 11.47     | 14.01     | 26.22    | 28.73    | 30.69    | 33.39    |
| NaCl 7         | 1.32     | 7.17     | 11.19     | 13.83     | 25.29    | 28.06    | 30.02    | 33.03    |
| NaCl 9         | 1.21     | 6.86     | 10.99     | 13.42     | 24.36    | 27.43    | 29.78    | 32.21    |
| NaCl 10        | 2.56     | 7.61     | 11.71     | 14.09     | 26.08    | 28.51    | 30.26    | 33.22    |
| CaCl₂ 1        | 3.09     | 8.86     | 11.97     | 14.73     | 27.56    | 29.15    | 31.18    | 33.98    |
| CaCl₂ 3        | 2.45     | 7.79     | 11.53     | 14.26     | 27.55    | 28.73    | 30.57    | 33.32    |
| CaCl₂ 5        | 1.86     | 7.51     | 11.20     | 13.92     | 25.45    | 28.12    | 29.75    | 32.63    |
| CaCl₂ 7        | 1.51     | 7.13     | 10.02     | 13.65     | 24.25    | 27.58    | 29.02    | 31.97    |
| CaCl₂ 9        | 1.56     | 7.75     | 10.79     | 14.29     | 24.42    | 27.07    | 29.16    | 30.30    |
| CaCl₂ 10       | 2.92     | 7.49     | 11.47     | 14.13     | 25.51    | 28.61    | 28.77    | 33.74    |
amine oxide surfactants increase with the hydrophobicity of the alkyl chain increasing. For the same surfactant concentration, higher CMC surfactants have less number of micelles. So, the presence of free surfactants in the test medium is higher, and therefore the methanogenic microorganisms are more likely to attach to the surfactant and thus enhance surfactant biodegradation\textsuperscript{26}. The results of this study are consistent with those reported for other surfactants, for which greater CMC increase the biodegradability\textsuperscript{27,28}.

3.2 Effect of inorganic salts on the surface activity of the amido Gemini amine oxide surfactants

The inorganic salt ionic strength has a certain impact on the surface properties of the surfactants. The $\gamma$-lgC curves of C$_{12}$-2-C$_{12}$ aqueous solutions at different concentrations of NaCl and CaCl$_2$ (298 K) are shown respectively in Figs. 1 and 2. By analyzing the plateau region and the break point of the plots, the surface tension at the CMC ($\gamma$$_{CMC}$) and CMC values are determined separately. There are no minimum points on the surface tension chart, which demonstrate that the purity of these synthesized surfactants is very well. Since the influence of NaCl and CaCl$_2$ on the CMC and $\gamma$$_{CMC}$ of the amido Gemini amine oxide surfactants with different carbon chain lengths is basically the same, the $\gamma$-lgC curves of C$_{14}$-2-C$_{14}$, C$_{16}$-2-C$_{16}$ and C$_{18}$-2-C$_{18}$ aqueous solutions at distinct concentrations of NaCl and CaCl$_2$ are not presented here. Their CMC and $\gamma$$_{CMC}$ are listed in Table 3. It can be found from Table 3 that the CMC and $\gamma$$_{CMC}$ of the surfactants decrease as the mass concentration of the inorganic salt increases, when the mass concentration of the inorganic salt is less than 7 mg/mL. The reason may be that after the addition of inorganic salt, the hydration film around the micelle is broken, and the diffusion electric double layer around the ionic group is compressed, which weakens the repulsion between the head group charges and makes the arrangement closer\textsuperscript{29}. Another reason may be that the anions and cations of inorganic salts will compete with surfactants for water molecules. Therefore, the affinity of water molecules and surfactant molecules is greatly weakened, which makes it easier to aggregate between surfactant molecules and reduces the CMC of the surfactant solution\textsuperscript{30,31}. However, when the mass concentration of the inorganic salt reaches 9 mg/mL or 10 mg/mL, the CMC of the surfactant solution raises and the solution becomes turbid. It may be that the electrostatic effect causes the N$^+$ in the molecule to be repelled to the interface by more and more Na$^+$ and Ca$^{2+}$, which leads to an increase of CMC\textsuperscript{32}. In addition, the higher the cation valence, the greater the reduction in CMC, indicating that the multivalent cations under high salinity greatly reduce the surface activity of the surfactant\textsuperscript{33,34}. Furthermore, the results also show that the $\gamma$$_{CMC}$ values of these synthesized amido Gemini amine oxide surfactants are between 24.25 and 34.81 mN/m at distinct concentrations of NaCl and CaCl$_2$, indicating that their surface activities are higher.

The high surface activity of amido Gemini amine oxide surfactants with salt tolerance may have great application prospects in modern oil exploration such as enhancement of oil recovery from depleted reservoirs\textsuperscript{35-37}.

3.3 Effect of pH on the surface activity of the amido Gemini amine oxide surfactants

Figure 3 shows the surface tension of the amido Gemini amine oxide surfactant at different pH values. Amido Gemini amine oxide surfactant, as a kind of amphoteric surfactant, contains inseparable positive and negative charge centers connected by bridges, so it can show unique isoelectric point properties in solution\textsuperscript{38}. The isoelectric
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points of the synthesized surfactants are pH 4.0-10.5, which were reported in our previous paper \(^{21}\). When the pH is within the isoelectric point range, the values of CMC and \(\gamma_{\text{CMC}}\) are the lowest, and when the pH value is higher and lower than the isoelectric point, the values of CMC and \(\gamma_{\text{CMC}}\) are briefly increase. This is because in the isoelectric point range, the amido Gemini amine oxide surfactant exists in the form of ylide \(\text{N}^+\text{O}^-\) with the least charge. Therefore, the gas-liquid interface is arranged most closely, and the values of CMC and \(\gamma_{\text{CMC}}\) are reduced obviously. When lower or higher than the isoelectric point, amine oxide surfactant molecules in water exhibit ionicity due to the protonation of N-O. At pH = 4.05, the properties of amido Gemini amine oxide surfactant are closer to cationic surfactants, and the micellization of the protonated form should be disfavored by the electrostatic repulsion among the positively charged headgroups \(^{30}\), so the values of CMC and \(\gamma_{\text{CMC}}\) increase. At pH = 10.15, the amido Gemini amine oxide surfactant mainly exists in amphoter form, the deviation from the isoelectric point region increases the electrostatic charge of the amido Gemini amine oxide, which lead to the surface activity slightly reduced.

4 Conclusion

In summary, the effects of inorganic salts and pH values on the surface activity of the amido Gemini amine oxide surfactants with different carbon chain length have been characterized. The results show that their \(\gamma_{\text{CMC}}\) values are between 24.25 and 34.81 mN/m at distinct concentrations of NaCl and CaCl\(_2\), indicating that their surface activities are higher. When the mass concentration of the inorganic salt is less than 7 mg/mL, the values of CMC and \(\gamma_{\text{CMC}}\) of the surfactants decrease as the mass concentration of the inorganic salt increases. In addition, the effect of CaCl\(_2\) on their surface activities is greater than that of NaCl. From the point of view of the effect of pH, the values of CMC and \(\gamma_{\text{CMC}}\) are the lowest within the isoelectric point range. Studies also have shown that the synthetic surfactants have a good biodegradability, which was above 96% after 7 days, and the biodegradability raises slightly with an increase of the hydrophobic alkyl chain from 12 to 18.

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