ORIGINAL ARTICLE



# Novel Synthesized Gemini Surfactant as Corrosion Inhibitor for Carbon Steel in HCl Solution

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Abstract A quaternary ammonium gemini surfactant containing an ester spacer, namely (diethylhexanedioate)diyl-a, w-bis(dimethyl myristyl ammonium bromide) (14-DEHA-14) was synthesized using a two-step procedure with dimethyl hexanedioate, dimethylaminoethanol, and 1-bromotetradecane. The chemical structure of the compound was confirmed by infrared (IR) spectrum, <sup>1</sup>H nuclear magnetic resonance (NMR), and elemental analysis. The critical micelle concentration was determined by conductivity and tensiometry measurements, and its inhibition effect on corrosion of carbon steel in dilute (1.0 M) hydrochloric acid solution was investigated by weight loss measurements. The results show that the synthesized gemini surfactant acts as an excellent corrosion inhibitor in 1.0 M HCl solution. The adsorption of the inhibitor via chemical adsorption onto the carbon steel surface obeyed the Langmuir adsorption isotherm. A possible corrosion mechanism of the compound is discussed in relation to the calculated thermodynamic parameters and adsorption isotherm.

**Keywords** Corrosion inhibition  $\cdot$  Gemini surfactant with ester spacer  $\cdot$  CMC  $\cdot$  Adsorption mechanism

# Introduction

Use of corrosion inhibitors is one of the most convenient means for protecting metals against corrosion, especially in acidic media [1, 2]. Most organic corrosion inhibitors interact with the metal surface through heteroatoms, such as nitrogen, oxygen, phosphorus, sulfur, aromatic rings, and multiple bonds, which can markedly change the corrosion resistance of the metal [3–6]. The inhibition efficiency is mainly due to the compatibility and affinity of the organic inhibitors with the metal surface, and their chemical structure [7]. Among organic inhibitors, oxygenated compounds and quaternary ammonium compounds are considered to be excellent corrosion inhibitors for many metals and alloys in various aggressive solutions [8–14]. As a result, exploitation of novel gemini surfactants containing oxygen atoms may be a good choice.

As a new generation of surfactants, gemini surfactants have attracted increasing research attention. Gemini surfactants consist of two hydrophobic chains and two polar headgroups covalently linked by a spacer. It has been demonstrated that gemini surfactants exhibit superior properties compared with conventional surfactants, such as lower critical micelle concentration (CMC) values, higher surface activity, better solubility, etc. [15–18]. In recent years, increasing research attention has been devoted to the corrosion inhibition behavior of gemini surfactants for highly viscous or aggressive media.

We report herein the synthesis of a quaternary ammonium gemini surfactant containing an ester spacer. The CMC was investigated by conductivity and tensiometry measurements. The inhibition effect of this gemini surfactant for carbon steel was evaluated by weight loss measurements in 1.0 M hydrochloric acid solution. The results show that this novel corrosion inhibition system is

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very efficient for carbon steel in 1.0 M hydrochloric acid solution. The possible adsorption mechanism of the gemini surfactant onto the surface of carbon steel in acidic medium was also explored.

# **Experimental**

# Synthesis of Gemini Surfactant

# Synthesis of Compound A

A mixture of dimethyl hexanedioate (9.80 g, 56.32 mmol) with dimethylaminoethanol (30.07 g, 337.92 mmol) in the presence of dibutyltin dilaurate (1.69 g, 2.67 mmol) as catalyst was stirred for 6 h at reflux. Then, the methanol byproduct and residual dimethylaminoethanol were removed under reduced pressure. The residue was purified by column chromatography to give **A** as white solid (9.00 g, yield 62.50 %).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ ppm: 1.66–1.73 (m, 4H, CCH<sub>2</sub>CH<sub>2</sub>C), 2.33–2.56 (t, 4H, 2× COCH<sub>2</sub>), 3.48 (s, 12H, 4× CH<sub>3</sub>), 4.14–4.17 (t, J = 7.2 Hz, 4H, 2× NCH<sub>2</sub>), 4.46–4.72 (m, 4H, OCH<sub>2</sub>). IR (KBr, cm<sup>-1</sup>): 2947, 2822, 1736, 1460, 1383, 1172, 1041.

# Synthesis of 14-DEHA-14

A mixture of compound A (3.00 g, 10.44 mmol) and 1-bromotetradecane (8.72 g, 31.49 mmol) in acetone (30 mL) was refluxed for 24 h. After the reaction mixture had cooled to room temperature, the crude product was isolated by filtering and then recrystallized twice from a mixture of acetone and acetonitrile to give **14-DEHA-14** as white crystal (8.04 g, yield 91.60 %).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  ppm: 0.86–0.89 (t, J = 6.8 Hz, 6H, 2(CH<sub>2</sub>)<sub>11</sub>CH<sub>3</sub>), 1.25–1.36 (m, 44H, 2(CH<sub>2</sub>)<sub>11</sub>CH<sub>3</sub>), 1.70–1.77 (m, 8H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub> and 2CH<sub>2</sub>CH<sub>2</sub>(CH<sub>2</sub>)<sub>11</sub>), 2.44 (m, 4H, 2CH<sub>2</sub>COO), 3.48 (s, 12H, 4NCH<sub>3</sub>), 3.62–3.66 (t, J = 7.6 Hz, 4H, 2 NCH<sub>2</sub>CH<sub>2</sub>(CH<sub>2</sub>)<sub>11</sub>), 4.15 (t, J = 7.2 Hz, 4H, 2OCH<sub>2</sub>CH<sub>2</sub>N), 4.63 (m, 4H, 2OCH<sub>2</sub>CH<sub>2</sub>N); IR (KBr, cm<sup>-1</sup>): 2919, 2850, 1723, 1473, 1381, 1274, 1153; Anal. Calcd. Found %: C, 59.81; H, 10.63; N, 3.56; Theory %: C, 59.82; H, 10.21; N, 3.32.

#### **Electrical Conductivity Measurements**

The conductivity of solutions of the synthesized gemini surfactant was measured as a function of concentration at  $308 \pm 0.1$  K using a DDS-11D conductivity meter (Shanghai Precision Scientific Instrument Co.). Each measurement was repeated twice [19]. Triple-distilled

water was added to the surfactant solution to adjust the surfactant concentration.

#### **Measurements of Surface Tension**

The surface tension of gemini surfactant solutions was measured using the drop-volume technique at  $308 \pm 0.1$  K [20]. All solutions were prepared with triple-distilled water. The surface tension of pure water was measured as being  $71.99 \pm 0.05$  mN/m at 298.0 K. The critical micelle concentration (CMC) and surface tension at the CMC ( $\gamma_{CMC}$ ) were determined from the break point of the surface tension versus logarithm of concentration curve. From the curve of surface tension versus concentration, the amount of surfactant adsorbed ( $\Gamma_{max}$ ) and the area occupied by a surfactant molecule ( $A_{min}$ ) at the air–water interface were calculated according to the following Gibbs adsorption Eqs. 1 and 2 [21]:

$$T_{\max} = -\left(\frac{1}{2.303nRT}\right) \left(\frac{d\gamma}{d\log C}\right),\tag{1}$$

$$A_{\min} = \frac{10^{16}}{N_{\rm A}\Gamma_{\rm max}},\tag{2}$$

where *R* is the gas constant (8.314 J mol<sup>-1</sup> K<sup>-1</sup>), *T* is the absolute temperature (K), *C* is the surfactant concentration (mol L<sup>-1</sup>), and  $d\gamma$ /dlog*C* is the slope below the CMC of the surface tension versus concentration curve. The value of *n* (the number of species at the interface whose concentration changes with the surfactant concentration) is taken as 3 [15, 22, 23]. *N*<sub>A</sub> is Avogadro's number (6.02 × 10<sup>23</sup> mol<sup>-1</sup>).

# **Test Solutions**

All tests were carried out in 1.0 M hydrochloric acid solution without or with addition of the synthesized quaternary ammonium gemini surfactant. Gas chromatography spectrometry was used to check the presence of hydrolysis for the gemini surfactant with ester spacer hydrolyzed in 1.0 M HCl solution at 308 K for half an hour in advance, and no peaks for hydrolysis products were observed. The test solutions were directly prepared by using reagentgrade hydrochloric acid and distilled water. During the measurement, the temperature of the test solutions was controlled by using a water bath with thermostatic control, and all experiments were performed under static conditions.

#### Weight Loss Measurements

Gravimetric analysis was performed on an analytical balance (model HR 200, readability 0.1 mg, standard Tab carb

| <b>e 1</b> Composition of on steel panels | Element | С     | Р     | S     | Si    | Mn   | Cr    | Со    | Cu    | Fe      |
|---|---------|-------|-------|-------|-------|------|-------|-------|-------|---------|
| -   | Wt.%    | 0.252 | 0.082 | 0.051 | 0.296 | 1.26 | 0.031 | 0.395 | 0.378 | Balance |

deviation  $\pm 0.2$  mg). Carbon steel panels with area of  $9 \text{ cm}^2$  were abraded with a series of emery papers (grades 320-400-600-800-1000-1200), followed by successive washing with distilled water, ethanol, and acetone, and finally dried in a low-humidity atmosphere. The composition of the carbon steel panels is presented in Table 1. After precise weighing, the carbon steel panels were immersed in 200 mL of 1 M hydrochloric acid solution without or with addition of the synthesized quaternary ammonium gemini surfactant for different concentrations at 308 K. After keeping for 6 h, the steel samples were taken out from solution, rinsed two times with distilled water, degreased with ethanol and acetone, and finally dried in dry air and precisely weighed [24]. To achieve good reproducibility and high reliability, the experiments were carried out in triplicate under aerated conditions. The average weight loss of three parallel steel samples was calculated.

# **Results and Discussion**

# Synthesis of Gemini Surfactant Containing Ester Spacer

The synthesis route for the gemini surfactant **14-DEHA-14** containing ester spacer is shown in Scheme 1. It was easily prepared through a two-step procedure. First, dimethyl hexanedioate was reacted with stoichiometric excess of dimethylaminoethanol in the presence of dibutyltin dilaurate to generate the important intermediate compound **A**. Subsequently, the target compound **14-DEHA-14** was prepared with high yield of 91.60 % by reaction of the intermediate with 1-bromotetradecane. The chemical structure of **14-DEHA-14** was confirmed by IR, <sup>1</sup>H NMR, and elemental analysis.

# **Conductivity of Surfactant Solutions**

The CMC was determined as the concentration at the intersection of the linear portions of the plots [19]; the curves of conductivity versus concentration of the synthesized gemini surfactant at 308 K are shown in Fig. 1. The CMC value for the gemini surfactant was calculated as 0.281 mmol  $L^{-1}$ , which is about one or two orders of magnitude lower than those of traditional surfactants. This illustrates that the synthesized gemini surfactant easily forms micelles in aqueous solution.



Scheme 1 Synthetic route for 14-DEHA-14



Fig. 1 Conductivity (K) versus surfactant concentration (C)

# **Surface Chemical Properties**

In general, the CMC and the surface tension at the CMC ( $\gamma_{\rm CMC}$ ), usually taken to be the minimum surface tension achievable by a surfactant, are the most important parameters to characterize the surface activity of a surfactant [25]. The surface tension isotherm for **14-DEHA-14** versus molar concentration (*C*) at 308 K is shown in Fig. 2. The values of CMC and  $\gamma_{\rm CMC}$  of **14-DEHA-14** were determined from the intersection points in Fig. 2, and the pertinent data are summarized in Table 2.

As shown in Table 2, the CMC of **14-DEHA-14** was determined as 0.267 mmol  $L^{-1}$  at 308 K and the  $\gamma_{CMC}$  value in aqueous solution was 39.6 mN m<sup>-1</sup>. The CMC value of the gemini surfactant is about one or two orders of magnitude lower than those of traditional surfactants, suggesting that it forms micelles easily in bulk solution.



Fig. 2 Surface tension versus aqueous molar concentration of 14-DEHA-14

The  $\gamma_{CMC}$  of the gemini surfactant was lower than those for common surfactants. This shows that the gemini surfactant possessed high surface activity.

The values of  $\Gamma_{\text{max}}$  and  $A_{\text{min}}$  for **14-DEHA-14** are also shown in Table 2. The efficiency can be expressed by the logarithm of the surfactant concentration at which the surface tension of water is reduced by 20 mN m<sup>-1</sup>. The larger the pC<sub>20</sub> value, the greater the surfactant adsorption at the air–water interface [26]. As shown in Table 2, the pC<sub>20</sub> value of the synthesized gemini surfactant was 3.84 at 308 K. It is obvious that the Gemini surfactant shows strong adsorption at the air–water interface.

# Weight Loss Measurements

The corrosion inhibition efficiency of the synthesized quaternary ammonium gemini surfactant was measured in dilute (1.0 M) hydrochloric acid solution on carbon steel surface at 308 K. The percentage values of corrosion rate (k) and inhibition efficiency (E) obtained by the weight loss method for different concentrations of the inhibitor are summarized in Table 3. The corrosion rate (k) and inhibition efficiency (E) were calculated from Eqs. 3 and 4 [12, 27, 28]:

$$k = \frac{m_1 - m_2}{S \times t},\tag{3}$$

 Table 3 Weight loss results of carbon steel corrosion in 1.0 M HCl at different concentrations of synthesized gemini surfactant

| $C \pmod{\mathrm{L}^{-1}}$ | Weight loss (mg) | $k (g m^{-2} h^{-1})$ | E~(%) | $\theta$ |
|----------------------------|------------------|-----------------------|-------|----------|
| 0                          | 66.46            | 18.46                 | 0     | 0        |
| $1.0 \times 10^{-7}$       | 58.28            | 16.19                 | 12.31 | 0.127    |
| $5.0 \times 10^{-7}$       | 37.67            | 10.46                 | 43.32 | 0.449    |
| $7.5 \times 10^{-7}$       | 32.15            | 8.93                  | 51.62 | 0.535    |
| $1.0 \times 10^{-6}$       | 19.16            | 5.32                  | 71.17 | 0.720    |
| $2.5 \times 10^{-6}$       | 8.61             | 2.39                  | 87.05 | 0.914    |
| $5.0 \times 10^{-6}$       | 5.56             | 1.49                  | 91.63 | 0.950    |
| $7.5 \times 10^{-6}$       | 4.79             | 1.33                  | 92.79 | 0.962    |
| $1.0 \times 10^{-5}$       | 4.28             | 1.19                  | 93.56 | 0.971    |
| $5.0 \times 10^{-5}$       | 2.80             | 0.78                  | 95.78 | 0.993    |
| $1.0 \times 10^{-4}$       | 2.68             | 0.74                  | 95.96 | 0.996    |
| $5.0 \times 10^{-4}$       | 2.40             | 0.67                  | 96.39 | 0.996    |
| $1.0 \times 10^{-3}$       | 1.99             | 0.55                  | 96.14 | 0.997    |

$$E(\%) = \frac{W_0 - W_i}{W_0} \times 100,$$
(4)

where  $m_1$  and  $m_2$  are the sample weight before and after immersion, respectively, *S* is the total area of the specimen, *t* is the corrosion time, and  $W_0$  and  $W_i$  are the weight loss in absence and presence of inhibitor, respectively.

Figure 3 shows the corrosion behavior of carbon steel in the presence of the synthesized gemini surfactant at 308 K. The results obtained from weight loss measurements of the quaternary ammonium gemini surfactant are listed in Table 3. The inhibition efficiency of the gemini surfactant increased with increasing inhibitor concentration in 1.0 M HCl solution. These results suggest that the inhibitor molecule acts through adsorption onto the metal surface. Thus, the inhibition efficiency may depend on the amount adsorbed and the surface coverage of the inhibitor [29, 30].

#### **Adsorption Isotherm**

From the adsorption isotherm, basic information about the interaction between the organic inhibitor and the carbon steel surface can be acquired. To obtain the isotherm, values of the fractional coverage as a function of inhibitor concentration must be determined. According to Eq. 5 [31, 32], the degree of surface coverage ( $\theta$ ) for the different

| Compound   | CMC <sup>a</sup> (mM) | $CMC^{b}\ (mM)$ | $\gamma_{\rm CMC}~({\rm mN/m})$ | $\Gamma_{\rm max}~(\times 10^6~{\rm mol/m^2})$ | $A_{\min} (\mathrm{nm}^2)$ | р <i>C</i> <sub>20</sub> |
|------------|-----------------------|-----------------|---------------------------------|--|----------------------------|--------------------------|
| 14-DEHA-14 | 0.267                 | 0.281           | 39.60                           | 1.11   | 1.50                       | 3.84                     |
| a          |                       |                 |                                 |  |                            |                          |

Measured by tensiometry

<sup>b</sup> Measured by conductometry

 Table 2
 Surface properties and micellization parameters of gemini surfactant



Fig. 3 Curve of E% versus concentration for 14-DEHA-14

concentrations of organic inhibitor was estimated from weight loss measurements:

$$\theta = \frac{W_0 - W_i}{W_0 - W_m},\tag{5}$$

where  $W_m$  is the smallest weight loss. The best correlation between the experimental results and isotherm functions at high concentrations of the organic inhibitor was acquired for the Langmuir adsorption isotherm, as shown by the following equation [33]:

$$\frac{C_{\rm inh}}{\theta} = \frac{1}{K_{\rm ads}} + C_{\rm inh},\tag{6}$$

where  $K_{ads}$  is the adsorption equilibrium constant of the adsorption/desorption process, and free energies of adsorption ( $\Delta G_{ads}$ ) were calculated using the following relationship [34, 35]:

$$\Delta G_{\rm ads} = -2.303 RT \log(55.5 K_{\rm ads}),\tag{7}$$

where *R* is the universal gas constant (the molar concentration of water in solution being 55.5). The plots of  $C_{inh}/\theta$  versus  $C_{inh}$  at 308 K (Fig. 4) show that the linear correlation coefficient ( $r^2$ ) and the slope are close to 1, confirming that adsorption of **14-DEHA-14** onto the carbon steel surface obeys the Langmuir adsorption isotherm. The inhibitor molecules adsorb onto the steel surface, forming a film that prevents corrosion induced by the medium. The adsorption parameters derived from Eqs. 6 and 7 are given in Table 4. The negative  $\Delta G_{ads}$  indicates that the inhibitor adsorption is a spontaneous process. The value of  $\Delta G_{ads}$  is below -40 kJ mol<sup>-1</sup>, which means that there is strong chemical adsorption between the charged inhibitor molecules and the carbon steel surface [36, 37].



Fig. 4 Langmuir adsorption plots for carbon steel in 1.0 M HCl solution with various concentrations of 14-DEHA-14

# Relationship Between CMC and Corrosion Inhibition

As shown in Fig. 4, the inhibition efficiency of the gemini surfactant increased with increasing inhibitor concentration (below the CMC) in 1.0 M HCl solution. However, when the inhibitor concentration was close to the CMC, increasing the inhibitor concentration led to relatively small changes in inhibition efficiency. Below the CMC, increasing the inhibitor concentration results in relatively high surface coverage. As the inhibitor concentration reaches the CMC, the amount of gemini surfactant molecule adsorbed onto the carbon steel surface reaches a maximum, and the inhibition efficiency is higher than 96.6 %. Additionally, with increasing inhibitor concentration (near the CMC), a multilayer may form on the carbon steel surface, which leads to little change in the inhibition efficiency. Therefore, when the CMC of the synthesized gemini surfactant is low, good corrosion inhibition can be achieved at low concentration.

# **Mechanism of Corrosion Inhibition**

The adsorption parameters above indicate that the adsorption behavior of the synthesized quaternary ammonium gemini surfactant is more complicated than that of traditional surfactants. To investigate the mechanism of corrosion inhibition, the different modes of adsorption available to the gemini surfactant are shown in Fig. 5. At extremely low concentrations of organic inhibitor, adsorption will take place through electrostatic interaction between the two ammonium groups (N<sup>+</sup>) and the negatively charged metal surface in acidic media (Fig. 5a). As the concentration of organic inhibitor increases, the interhydrophobic chain interactions will become stronger, which may lead to desorption of one or two ammonium groups from Table 4Adsorption parametersof gemini surfactant on carbonsteel surface at 308 K

| Linear correlation coefficient | Slope | $K_{\rm ads}~({ m M}^{-1})$ | $\Delta G_{\rm ads} \ ({\rm kJ} \ {\rm mol}^{-1})$ |
|--------------------------------|-------|-----------------------------|--|
| 0.9999                         | 1.043 | $7.38 \times 10^{7}$        | -56.69   |



Fig. 5 Model of gemini surfactant adsorption onto carbon steel surface

the metal surface (Fig. 5b) [12, 14]. When the concentration exceeds the CMC, increasing the gemini surfactant concentration will result in gradual formation of multilayers (Fig. 5c). However, it is obvious that variation of the concentration above the CMC has little effect on the corrosion inhibition. This is mainly because a change in the concentration above the CMC only results in additional coverage beyond the monolayer level, which is already sufficient for significant inhibition.

In addition, as shown in Table 4, using the  $\Delta G_{ads}$  value of -56.69 kJ mol<sup>-1</sup>, the potential can be calculated as 0.29 V according to the equation  $\Delta_r G = -nEF$ , where *F* is the Faraday constant and *n* is taken as 2 for the two ammonium groups (N<sup>+</sup>). This result indicates that carbon steel does not corrode easily under these experimental conditions.

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