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Synthesis of two chiral surfactants by a simple method and the surface activity evaluation



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ABSTRACT

In order to expand the application range of surfactants, two chiral surfactants named CTACYT and CTATYS were synthesized by reacting cetyltrimethylammonium chloride (CTAC) with L-cysteine and L-tyrosine, respectively by a simple method. Studies show that the foaming ability, foam stability and emulsifying properties of both CTACYT and CTATYS are superior to CTAC. The corrosion inhibition efficiencies of CTACYT and CTATYS are 96.72% and 90.22%, respectively, which are higher than that of CTAC. The new chiral surfactants remain the chiral character, and the specific optical rotations of CTACYT and CTATYS were 8.1° and 11.6°, respectively. This work provides a new and easy way to the synthesis of chiral surfactants, which will benefit the related research in this field.

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1. Introduction

Quaternary ammonium cationic surfactants are widely used in many fields, such as textiles, cosmetics and petrochemical industries [1-3]. The prepared cationic surfactants display greater aqueous solution properties as compared to other cationic surfactants, such as minimum surface tension, lower critical micelle concentration (CMC), higher foaming ability, better emulsifying performances, and higher corrosion inhibition efficiency [4,5]. Experimental study found that the structure of surfactant has a significant impact on its properties, such as washing, wetting, dispersing, emulsifying, solubilizing, foaming and so on. It is widely used in the fields of washing products, chemical industry, medicine, textile, remediation of petroleum oil contaminated soil [6] and so on. With the continuous development of economy, the level of chemical industry in China has been greatly improved, with the growth of our demand and application of surfactants. In order to effectively promote the application of surfactants in various fields, we need to strengthen the chemical structure of surfactants [7]. In recent years, researchers have expanded their range of applications by complexing cationic surfactants with other types of surfactants. However, few studies have attempted to rationally design functional cationic surfactants by incorporating functional groups into conventional cationic surfactants [8–12]. Depending on the functional group, functional cationic surfactants can react to various environmental stimuli to achieve their desired effects and applications. Due to scientific curiosity, our study uses cetyltrimethylammonium chloride (CTAC) as a raw material to react with L-cysteine and Ltyrosine, respectively to synthesize two kinds of chiral surfactants (CTACYT, CTATYS) by simple method. Here, we studied the foaming properties, the emulsifying properties and the corrosion inhibition properties of two kinds of chiral surfactants.

2. Experimental

2.1. Materials

Cetyltrimethylammonium chloride (CTAC), L-cysteine and L-Tyrosine were purchased from China National Pharmaceutical Group. Other chemicals were purchased from Xi'an Chemical Reagent Factory, and were used without further purification.

2.2. Synthesis of CTACYT and CTATYS

Sodium hydroxide reacted completely with cetyltrimethylammonium chloride (molar ratio of 1:1) at room temperature using methanol as a

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solvent. Then, the supernatant reacted with L-cysteine and L-tyrosine, respectively (molar ratio of 1:1), was heated to reflux for 2 h with stirring, and the mixture was evaporated and dried. Finally, two new surfactants (CTACYT, CTATYS) were obtained. The synthetic chemical equations for CTACYT and CTATYS are shown in Fig. 1. The products were recrystallized in ethanol to get the purified product for the following characterization and evaluation. ¹H NMR of CTACYT (D₂O, 400 MHz), δ : 3.80 (1H, dd, *J* = 7.2 Hz), 3.30 (9H, s), 3.23–3.24 (3H, m), 2.98 (1H, m), 2.55 (1H, b), 1.73 (2H, dd, *J* = 7.2 Hz), 1.65 (1H, b), 1.29–1.32 (26H, m), 0.96 (3H, t, *J* = 7.2 Hz); ¹H NMR of CTATYS (D₂O, 400 MHz), δ : 6.95 (2H, d, *J* = 6.8 Hz), 6.68(2H, d, *J* = 6.8 Hz), 5.93 (1H, b), 3.87 (1H, dd, *J* = 7.2 Hz), 3.30 (9H, s), 3.23–3.24 (3H, m), 2.97 (1H, m), 2.72 (1H, b), 1.73 (2H, dd, *J* = 7.2 Hz), 1.29–1.32 (26H, m), 0.96 (3H, t, *J* = 7.2 Hz), 1.29–1.32 (26H, m), 0.96 (3H, t, *J* = 7.2 Hz), 1.29–1.32 (26H, m), 0.96 (3H, t, *J* = 7.2 Hz), 1.29–1.32 (26H, m), 0.96 (3H, t, *J* = 7.2 Hz), 1.29–1.32 (26H, m), 0.96 (3H, t, *J* = 7.2 Hz), 1.29–1.32 (26H, m), 0.96 (3H, t, *J* = 7.2 Hz), 1.29–1.32 (26H, m), 0.96 (3H, t, *J* = 7.2 Hz), 1.29–1.32 (26H, m), 0.96 (3H, t, *J* = 7.2 Hz), 1.29–1.32 (26H, m), 0.96 (3H, t, *J* = 7.2 Hz).

2.3. Surface tension measurement

Hanging ring method is one of the frequently used methods for measuring the surface tension of different surfactant solutions. At room temperature, a Krüss-K6 tensiometer (Krüss K100, Germany) was used to measure the surface tension by means of different surfactant solutions prepared at concentrations ranging from 10 to 5000 mg/l [7]. The platinum ring was often washed using distilled water to obtain a standard surface tension value of the distilled water. All surface tension measurements were repeated thrice to reduce the error [8].

2.4. Foaming ability measurement

The foaming ability is usually measured by the Waring Blender method. 100 ml solution was poured into a 500 ml container with a high speed stirrer, and the time $t_{1/2}$ (half-life) represents stabilization [9]. Each experiment was repeated at least three times [10]. The microstructure of the foam was characterized under a microscope using a polarizing microscope (DM4500P LFD, Germany).

2.5. Emulsification

Three 20 ml stoppered graduated cylinders were taken, 2% different surfactant solutions with 10 ml of dehydrated and degassed crude oil were added in a volume ratio of 1:1, and the cylinder was sealed and placed at 60 °C [11,12]. The box was taken out for 60 min and each

tube was shaken evenly 200 times, which was immediately placed on the test tube rack vertically and placed back in the 60 $^{\circ}$ C incubator. Then, the timing started and the water separated in the test tube was recorded at volume intervals. The formula for calculating the water extraction rate is:

$$E_d = \frac{V_W}{V} \times 100\%$$

Where:

*E*_d-water extraction rate (%); *V*_w-water output (ml); *V*-the water phase volume when preparing emulsion (mL).

2.6. Corrosion inhibition measurement

A weight loss technique was used to evaluate the inhibiting efficiency of the four surfactant solutions at a certain concentration in HCl solution at 60 °C for 4 h [13]. The experiment was carried out with a 40 mm \times 13 mm \times 2 mm mild steel sample. The sample was polished with 360#, 600#, 1200# sandpaper on a smooth plane before the experiment, and then the polished sample was cleaned in distilled water and later washed with absolute ethanol. Finally, it was washed with acetone. They were dried by degreasing cold air, placed in a dryer, weighed with an electronic balance 5 min prior to the experiment, and then the length, width, thickness, diameter of the hole were measured with a vernier caliper. These samples were immersed in 5% HCl solution, and different surfactant solutions were added at some concentration at 60 °C for 4 h [14].

Electrochemical measurements were conducted in a conventional three-electrode thermostated cell. The electrode was inserted into a Teflon tube and isolated with polyester so that only its section (0.5 cm²) was allowed to contact the aggressive solutions. A platinum disk as counter electrode and standard calomel electrode (SCE) as the reference electrode have been used in the electrochemical studies. The potentio-dynamic curves were recorded using a CS350 system connected to a personal computer. The working electrode was first immersed in the test solution for 60 min to establish a steady state open circuit potential. After measuring the open circuit, potential dynamic polarization curves were obtained with a scan rate of 0.5 mV/s. Corrosion rates (corrosion



Fig. 1. Synthetic chemical equations for CTACYT and CTATYS.

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current densities) were obtained from the polarization curves by linear extrapolation of the anodic and cathodic branches of the Tafel plots at points 100 mV more positive and more negative than the E_{corr} .

2.7. Specific rotation

The rotation of a substance depends on its properties, its concentration, the temperature at which it is measured, the wavelength of the light used and the thickness of the liquid layer of the substance transmitted. Therefore, the specific rotation Dt is usually used to represent the optical rotation of a compound with a fixed wavelength, the formula for calculating the specific rotation is as follows:

 $[\alpha]_{D}^{t} = \frac{\alpha}{Lc}$ Where: α -rotation. t-Temperature at the time of measurement; L-Length of sample tube (DM); *c*-concentration (g/100 ml).

3. Results and discussion

3.1. Surface activity

As shown in Fig. 2, the minimum interfacial tension values of CTACYT and CTATYS are almost the same as that of CTAC at high concentration. However, the critical micelle concentrations of CTACYT and CTATYS were slightly higher than that of CTAC. When the critical micelle concentration is reached in the surfactant solution, the surface tension value of the surfactant molecules in the water is almost no longer reduced and slightly increased [15]. As shown in Table 1, the surface tension of the three kinds of surfactants (CTAC, CTACYT, CTATYS) in the critical micelle concentration is 37.4, 37.1 and 37.0 mN/m, respectively.

3.2. Foaming ability

Previous papers have reported that foaming ability and foam stability are related to the properties of interfacial membranes and interfacial tension [16-18]. When bubbles with the same total surface area are produced, the low surface tension helps to produce the foam; however, it can also bubble and burst. The findings of Sonin et al. [19] insist that the high-frequency interfacial elasticity has a significant impact on the film thinning process, that is, foam stabilization is mainly due to the elasticity of the interface film that defines the two phases. This study also demonstrates the fact that foaming stability is closely related to



Fig. 2. Surface tension-concentration curve of different surfactants at 25 °C.

Table 1	
The CMC and surface tension of different surfactants.	

Surfactant	CMC/mg/l	The minimum surface tension/mN/m
CTAC	8.27	37.4
CTACYT	8.68	37.1
CTATYS	8.47	37.0

the high interfacial elasticity of the film [20,21]. At room temperature, the cationic surfactants assimilate to form a double layer, and the hydrophilic head base and liquid membrane form a hydrated layer. In this study, the stability was measured based on the initial foam height (H_0) and the height of the foam after 5 min (H_5) after mechanical stirring at room temperature. The results are shown in Table 2. The foaming capacity of the three surfactants is different, and the foaming height of the same surfactant increases with the increase in concentration, reaching a maximum of H₀ and H₅ when the concentration is approximately 2000 mg/l. Above this concentration, H₀ and H₅ remain almost constant. Therefore, 2000 mg/l is known as the best concentration of foaming ability and foam stability of different surfactant solutions. CTACYT has the best foaming ability, with H₀ reaching 21.4 ml and H₅ reaching 20.2 ml, hence the foam volume of CTACYT only decreased by 0.2 ml. CTATYS's foam stability reached 98.17%. In order to further understand the stability of the surfactants, the Waring Blender method was used to determine the concentration of 2000 mg/l surfactant solution. A 100 ml solution and distilled water were used as the solvent, and the stirring speed was 7000 r/min for 3 min. Then, the foam height and half-life of the different surfactant solutions having the same concentration were observed. Fig. 3 shows that CTACYT has a significant foaming capacity, which agrees with the results of the agitation method [22].

Table 2

The foaming performance of different concentration of different surfactants.

Concentration of surfactant /mg/l	ion of surfactant /mg/l Foam height of Ross-l							
	CTAC		CTAC		CTACYT		CTATY	′S
	H ₀	H_5	H ₀	H_5	H ₀	H_5		
400	12.7	11.6	12.8	11.9	13.5	12.8		
600	14.0	13.1	15.2	14.8	15.9	15.2		
1000	16.8	15.7	17.3	16.2	17.5	16.7		
2000	18.3	18.0	21.4	21.2	21.8	21.4		
3000	18.2	17.8	19.4	19.0	20.3	19.6		



Fig. 3. The foaming performance of different surfactants with the same concentration.

3.3. Microstructure of foams

An optical microscope was used to characterize the microstructure of a large amount of foam produced by 2000 mg/l of three surfactant solutions at a stirring speed of 7000 r/min for 3 min [23]. The results are shown in Figs. 4. It is evident from the figures that the foams produced by the different surfactant solutions exhibit different shapes and volumes. The foam microstructure of CTAC and CTACYT is spherical, and the foam microstructure of CTATYS is an irregular polygon, indicating that the structure of the amino acid has different effects on the microstructure of the foam. This can be ascribed to the fact that foam stability is controlled by the liquid film discharge process. Compared with other surfactants, CTAC has the fastest drainage process and CTACYT has the slowest drainage process; hence, CTACYT produces foam with high stability. Two hydrophilic groups have been added to CTACYT and CTATYS. One functional group is an amino group $(-NH_2)$ and the other is a carboxyl group (-COO⁻), in order that the molecular structure makes CTACYT and CTATYS more hydrated than conventional anionic (cationic) ionic surfactants. When adsorbed at the gas-liquid interface, the molecules of the liquid film can be hydrogen-bonded or entangled with each other, so that the surface film has certain strength, thereby providing better foaming ability and foam stability.

3.4. Emulsification power

The water evolution rate may reflect the emulsifying ability of the surfactant, that is, the smaller the water evolution rate, the stronger the emulsifying ability of the system to be tested, and on the contrary, the worse the emulsifying ability of the system. The stability of the emulsion formed between the surfactant solution and the oil phase depends on the chemical structure of the surfactant and oil [24]. The emulsifying ability of the surfactant is strongest at the micelle concentration. Therefore, we have studied the emulsifying ability of three surfactants at different micellar concentrations, as shown in Fig. 5. With the extension of time, the water evolution rate of the emulsion formed by the CTAC solution and dehydrated degassed crude oil increased continuously. After 20 min of rapid demulsification, the water evolution rate of the emulsion solution reached over 80%. However, the water evolution rate of the emulsion formed by the CTACYT and CTATYS solutions and the dehydration and degassing crude oil was less than 60% at 20 min. Also, the water evolution rate had a tendency to be stable with the prolongation of time. It can be seen that CTACYT and CTATYS had strong emulsifying ability and could form stable emulsion with dehydrated and degassed crude oil. Also, for the emulsifying ability, the separation time become longer and the emulsifying ability was improved. With the introduction of amino acids, the hydrophilicity increases, the adsorption is saturated, a dense film is formed at the water/oil interface, and the strength of the interfacial film is increased, which makes the separation time longer; that is, the emulsifying ability becomes stronger.



Fig. 5. The emulsion capability of surfactants in the CMC with same concentration.

3.5. Corrosion inhibition

Corrosion inhibitors are the most commonly used method of inhibiting metal corrosion. In recent years, with the improvement of human environmental awareness and the development of sustainable development, the research and development of new and efficient environmental inhibitors has become a hot spot [25]. The inhibition efficiency obtained by weight loss method of chiral cationic surfactants, CTACYT and CTATYS, is 96.72% and 90.22%, respectively, which is superior to that of CATC. Probably because there are not only nitrogen atoms in the molecular structure of CTACYT, but also sulfur atoms. The synergistic action of these two ions makes the Fe-interface more favorable for the adsorption of charged CTACYT molecules, making them easier to coadsorb with metal ions [26,27], and making them more effective in corrosion inhibition. Therefore, it is inferred that the adsorption of CTACYT on the metal surface may be a chemical adsorption in addition to physical adsorption, while the adsorption of CTATYS is physical adsorption.

The anodic and cathodic polarization curves for a mild steel electrode at 298 K were shown in Fig. 6. Table 4 shows the electrochemical corrosion kinetic parameters, i.e., corrosion potential (E_{corr}), cathodic and anodic Tafel slopes (β_a , β_c) and corrosion current density I_{corr} obtained by extrapolation of the Tafel lines. The inhibition efficiency (%) is also calculated from the following equation:

$$E(\%) = \frac{I_{corr} - I_{corr(i)}}{I_{corr}} \times 100$$
⁽¹⁾

where I_{corr} and $I_{\text{corr}(i)}$ are corrosion current densities obtained in the absence and presence of inhibitors, respectively. As it was expected both



Fig. 4. Microscope morphology of CTAC(a), CTACYT(b) and CTATYS(c).



Fig. 6. The polarization curve of steel in HCl solution containing the surfactants at 20 mg/l.

anodic and cathodic reactions of mild steel electrode corrosion were inhibited by the increase of the surfactants. This result suggests that the addition of the surfactants reduces anodic dissolution and also retards the hydrogen evolution reaction [13], and CTACYT and CTATYS are much better than that of CTAC, which is consisted with the results above. Comparing the data from Table 3 and Table 4, we can find the inhibition efficiency obtained from potentiodynamic polarization are quite different from those calculated from weight-loss measurements, which is attributable to the fact that the weight-loss method gives average corrosion rate, whereas electrochemical method gives instantaneous corrosion rates. These differences may arise frequently because of the difference in the time required to form an adsorbed layer of inhibitors on metal surface [13,14].

3.6. Specific rotation

It can be seen from Table 5 that the modification of the proceeds of the chiral amino acid surfactant is slightly different from the rotation of cysteine, and the tyrosine ratio is almost unchanged. The interpretation of quaternary ammonium salt cationic opponents on the carbon atoms did not produce electronic polarization phenomenon. In the previous studies, they thought that the bigger the spiral electronic polarization, the more susceptible it is to the effects of light and electromagnetic fields, hence the optical rotation [28].

Table 3

Corrosion inhibition efficiency and corrosion rate of the surfactants at 20 mg/l.

Cationic surfactants	$W_{corr} \left(\mathbf{g} \cdot \mathbf{m}^{-2} \cdot \mathbf{h}^{-1} \right)$	E_w (%)
Blank	17.07	/
CTAC	1.93	88.70
CTACYT	0.56	96.72
CTATYS	1.67	90.22

Table 4

Potentiodynamic polarization parameters for the corrosion of steel in HCl solution containing the surfactants at 20 mg/l.

Cationic surfactants	$-E_{\rm corr}$ (mV)	I _{corr} (μA/cm ²)	$egin{aligned} & \beta_{a} \ (mV/dec) \end{aligned}$	$eta_{ m c}$ (mV/dec)	Corrosion rate (mm/a)	E (%)
Blank	0.46121	41.687	61.67	122.43	0.4891	/
CTAC	0.46541	25.703	55.65	121.42	0.3015	38.35
CTACYT	0.46502	12.022	48.54	131.32	0.1410	71.17
CTATYS	0.46432	10.715	53.43	133.32	0.1257	74.30

Table 5	
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Specific optical rotation of chiral amino acid surfactants.

Chiral substance	Solvent	c(mmol/l)	α	$[\alpha]t D$
L-cysteine	1 mol/l	1	$+0.16^{\circ}$	+8.0°
L-tyrosine	1 mol/l	1	-0.62°	-12.4°
CTACYT	1 mol/l	1	$+0.18^{\circ}$	+8.1°
CTATYS	1 mol/l	1	-0.58°	-11.6°

4. Conclusions

First of all, with the introduction of amino acids, the hydrophilicity increases, the adsorption is saturated, a dense film is formed at the water/oil interface, and the strength of the interfacial film is increased, so that the separation time becomes longer; that is, the emulsifying ability becomes stronger. Both CTACYT and CTATYS surfactants have better emulsifying ability and foam stability than CTAC. Secondly, the CTACYT and CTATYS surfactants have the functional group structure of amino acids, which has better corrosion inhibition effect than CTAC. The corrosion inhibition efficiencies of CTACYT and CTATYS are 96.72% and 90.22%, respectively. The result obtained by the electrochemical method is that the instantaneous corrosion inhibition rate is different from the average corrosion inhibition rate obtained by the weightlessness method, but the corrosion inhibition trends measured by the two methods are consistent. Besides, the new chiral surfactants remain the chiral character, and the specific optical rotations of CTACYT and CTATYS were 8.1° and 11.6°, respectively. This work will benefit the related research in this field.

CRediT authorship contribution statement

Gang Chen: Conceptualization, Methodology. Jiao Yan: Data curation, Writing - original draft. Yun Bai: Visualization, Investigation. Sanbao Dong: Software, Validation. Dengwei Liu: Investigation. Jie Zhang: Supervision. Shidong Zhu: Software. Chengtun Qu: Writing - review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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