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The Inhibitive Effects of Gemini Imidazoline Surfactants on Copper in Hydrochloric Acid Solution

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Gemini imidazoline surfactant was synthesized from the lauric acid and triethylenetetramine. The inhibitive effects of Gemini imidazoline surfactants on copper in hydrochloric acid solution were studied by electrochemical polarization and impedance spectroscopy. It was found that the inhibitive efficiency depended on the structure of surfactants inhibitive, concentrations of surfactants inhibitive, solution concentration effect were investigated. The results show that Gemini imidazoline surfactant has excellent inhibitive effect on copper in hydrochloric acid solution, and the inhibitive efficiency of Gemini imidazoline in hydrochloric acid solution is better than traditional monomer imidazoline surfactant. The higher the concentration of Gemini imidazoline surfactant, the better the inhibitive effect is.The lower the concentration of acid solution, the higher the inhibitive efficiency is.

Keywords: Gemini; Imidazoline; Surfactants; HCl; Inhibition

1. INTRODUCTION

In the recent years, the application fields of Gemini surfactants have gradually expanded, among which Gemini imidazoline surfactants have attracted a lot of attention because of their excellent properties[1]. Gemini imidazoline surfactant is a new representative surfactant developed in the recent years. Gemini surfactant is composed of two surfactant monomers connected by a binding group. The two end chains of the monomer can be different lengths. The two end chains of the monomer can be cationic, anionic or non-ionic, and the binding group can be in any form like short, long, soft, rigid, aliphatic or aromatic[2-4]. Compared with traditional single-chain surfactants, Gemini surfactants have good solubility and viscoelasticity in solvents. Its critical micelle concentration is usually one to two orders of magnitude lower than the corresponding single-chain surfactants. Because of its good storage stability, viscosity, dispersibility and fabric conditioning properties, Gemini surfactants are used in laundry industry and other industrial fields[5].Gemini imidazoline surfactants have special physical and chemical properties, such as high density, low Krafft point, unique rheological properties,good compatibility and self-assembly ability, etc[6-9].Therefore, Gemini imidazoline surfactants play an important role in gene and drug delivery[10],chemical applications, porous materials and other fields[11-16].

Researchers have done a lot of research on the design and synthesis of Gemini imidazoline surfactants.Divya Bajpai Tripathy[9, 17] and Yuanhong X[18] used microwave synthesis method, which greatly improved the synthesis efficiency,and studied its surface properties. A. Migahed[19, 20] team studied the effect of the length of the ligand on the sustained release performance. The results showed that the shorter the ligand,the better the sustained release effect. Huaiyu Yang[21] team studied the relationship between the sustained release and temperature. They concluded that the sustained release increased with the increase of temperature. The Jianbin Huang[22] research group made a comparative study of Gemini and Bola. The results showed that the cephalic groups played an important role, and how the cephalic groups of Gemini and Bola surfactants affected their properties. The changes is still under research. Fan Weiyu[23] has combined and studied its emulsifying performance and applied it to emulsified asphalt. The results showed that only a small amount of such surfactants can meet the requirements of emulsified asphalt. Zhang Guanghua's research group[24, 25] synthesized and confirmed its good sustained-release effect on tinplate. Yangjiang[26] project combined and studied the anti-corrosion performance of the compound. The results showed that the slow-release performance of the compound is better.

In fact, these studies focused on the effects of interval length, surface properties and sustained release effects. Although some synthetic methods are efficient, they greatly increase the instability of their synthesis. There is no efficient or stable synthetic method for the time being. Because the cost is so high and synthesis is quite time consuming, most of the research still remains at the molecular level such as some basic surface properties. Only Gemini type is studied such as different chain length, different link base, and compounding. Few single-chain molecules have been compared with Gemini-type molecules, which makes the excellent structure of Gemini molecule obscure. In order to be close to the actual production and life better, and reflect the excellent structure of Gemini imidazoline surfactant more effectively, the sustained release performance of Gemini imidazoline surfactant was studied, and compared with the single-chain molecule, so that it can reflect the difference more intuitively between a single-chain molecule and Gemini molecule.

The cationic Gemini surfactant of imidazoline quaternary ammonium salt was synthesized from lauric acid, diethylenetriamine (DETA) and modifier 1,3-dibromopropane. Its potential polarization curve and electrochemical impedance were measured. The corrosion inhibition performance of imidazoline surfactant monomer and Gemini surfactant in the hydrochloric acid medium was studied. The corrosion inhibition performance of pure copper in the hydrochloric acid medium was studied by changing the concentration of imidazoline inhibitor and hydrochloric acid. The sustained release effect of Gemini imidazoline surfactant in acidic medium was evaluated.

In this paper, a reasonable and stable synthetic route has been explored through experiments. The products have been successfully synthesized. At the same time, the corrosion inhibition performance of the product was studied.

2. EXPERIMENTAL AND COMPUTATIONAL SECTION

2.1 Materials and Main instrument

2.1.1 Materials

Lauric acid、Diethylenetriamine (DETA)、xylene、zinc power、dimethyl-carbonate、1,3-dibromopropane、acetone,all these reagents are analytical reagent (AR).

2.1.2 Main instrument

ALPHA FT-IR, NMR spectrometer, Electrochemical workstation (CHI600E), motor agitator, Digital display constant temperature oil bath pot.

2.2 Synthetic route （Synthesis of the Gemini imidazole compound）

Diethylenetriamine (DETA) was mixed with lauric (1:1.2 molar ratio) in xylene as the solvent at 140°C, add some zinc power as a catalyzer, drop diethylenetriamine (DETA) into the flask slowly and react for 2h; install a water separator, and increase the temperature to 200°C,then react for 8h. When the theoretically calculated amount of water was removed,the solvent was removed by distillation, the temperature of distillation is controlled at 140°C. Products are yellow oily imidazoline intermediates. Control the temperature in the range of 80-90°C, add a certain amount of dimethyl-carbonate for 5h, whose molar quantities was the same as intermediates. Single chain imidazoline quaternary ammonium salt was produced. Add some 1,3-dibromopropane as the 1/2 molar quantities of intermediates, react for 8h, the product is red-brown sticky liquid. Control the temperature under 50°C, add a few acetones, take a hot filtration and extract zinc powder. Stand the solution quietly and wait for the crystal (take a sucking filtration after solid precipitation appeared), wash the crystal several times with acetone. The product is khaki solid, which is Gemini imidazolinium surfactants. (LG is 1,3-di(1-methyl-1-ethylamino-2-n-undecyl-4,5-dihydro-imidazoline) propanegemini which is the gemini cationic imidazoline surfactants based on lauric acid and LM is 1-enthylamino-2-n-undecyl-4,5-dihydro-iminazoline which is the cationic monomeric surfactants based on lauric acid.[10]) The specific process is shown in Fig.1. FTIR spectra of synthesized cationic LG are given in Figure 2. 1H-NMR spectra of synthesized Cationic LG are given in Figure 3.

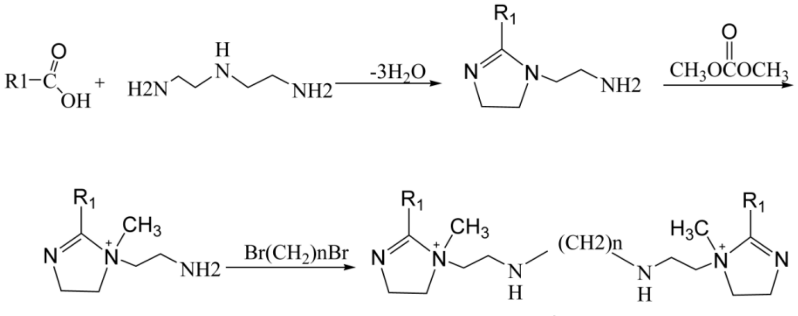

Figure 1. Synthesis route of LG

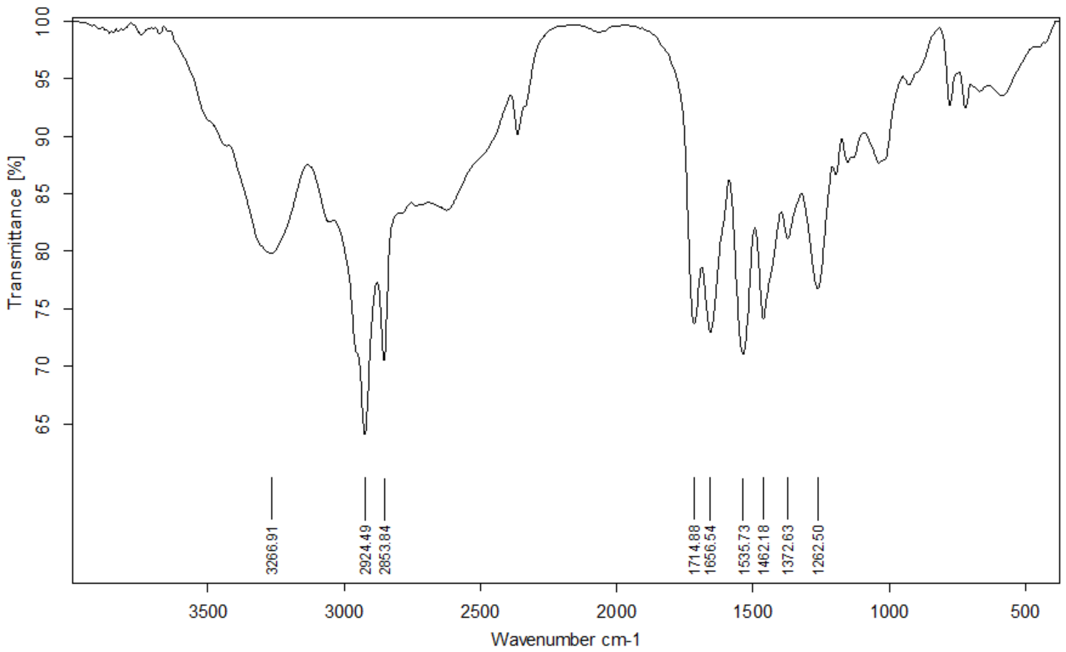

Figure 2. FT-IR spectrum of LG

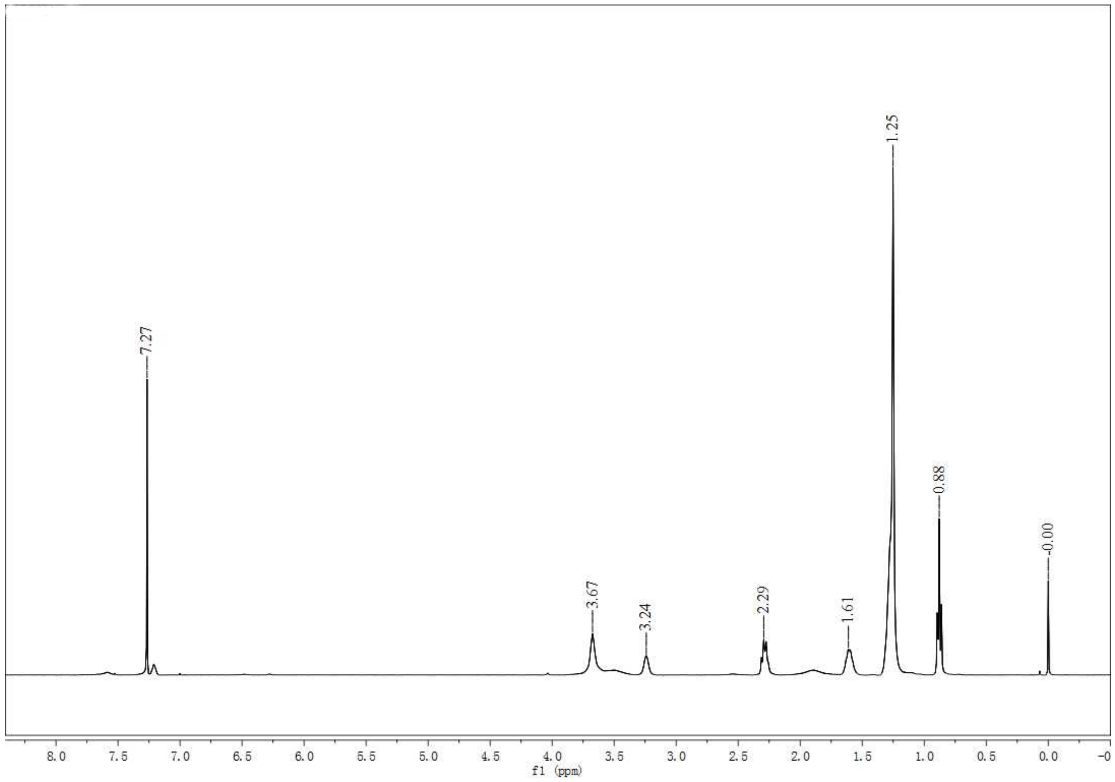

Figure 3. ^{1}HNMR spectrum of LG

In the Figure 2, FTIR spectrum of the prepared inhibitors shows the appearance of the peak at 1535.73 cm-1 due to the imidazole ring in inhibitor.One peak at 3266.91cm-1 is ascribed for V_{N-H} in the inhibitor, 2924.49cm-1 corresponding to CH_2.1656.54 cm-1 for C=N,1535.73 cm-1,corresponding to the (N-H)bending absence of C=O band at 1714.88 cm^{-1} confirms the cyclization of amide compound as seen in Fig. 2.The FT-IR charts of the prepared inhibitors show the expected functional groups in agreement with the reported work[11, 12] .

In the Figure 3, ^{1}H-NMR spectrum of inhibitor was given in Fig.2.Peaks at 7.27ppm correspond to the proton of -CONH- group whereas signal at 1.25ppm confirms the presence of CH2 group adjacent to amide linkage.Peaks at 3.67 and 2.29ppm correspond to the N-CH_2-CH_2-N and N-CH_3 methylene groups of the imidazoline ring.The peak at 1.25ppm shows the presence of -CH_2- adjacent to carbonate linkage, present in the spacer group. The results of ^{1}H-NMR confirmed the proton distribution in the prepared Gemini surfactant.

2.3 Electrochemical system

Three-electrode system was used. The auxiliary electrode (CE) is platinum electrode (platinum wire), the reference electrode (RE) is saturated calomel electrode (SCE), and the working electrode (WE) is copper electrode. Copper (d=2.0 mm, puratronic, Alfa aesar, 99.999%) was sealed with epoxy resin, one end of which was exposed to solution, and copper electrode was made.Before each experiment, the working electrodes were carefully ground with sandpaper of different meshes to make the surface smooth and smooth. Then the polishing residue is removed by cleaning, and then cleaned and dried with distilled water, acetone and anhydrous ethanol respectively. CHI660E electrochemical workstation was used for electrochemical testing and electrochemical impedance spectroscopy analysis. All potentials reported in this paper are referred to SCE.

3. RESULTS AND DISCUSSION

3.1 Electrochemical measurements

The corrosion inhibition efficiency (%) can be calculated by the following equation[27, 28].

$$\eta\% = \frac{i^0_{corr} - i_{corr}}{i^0_{corr}} \times 100\%$$

In this equation,i^o_{corr} and i_{corr} are respectively the current density corresponding to the blank copper electrode and the copper electrode covered with imidazoline film.The inhibition efficiency also can be calculated using the polarization resistance from the following equation[27, 28]:

$$\eta\% = \frac{R_P - R_P^0}{R_P} \times 100\%$$

where R_p and R_p^0 are the polarization resistance values with and without n-Bpys, respectively.

3.1.1 Different inhibitors

Figure 4 shows the potentidynamic polarization curves and impedance curves diagrams of copper electrodes containing or without surfactants obtained in 1M/L HCl solution. The electrochemical parameters are shown in Table 1 and Table 2. The results show that the corrosion rate of copper is decreased by adding inhibitor. When the anode polarization curve and cathode polarization curve are shifted to the right obviously in the solution containing inhibitor. Compared with the current density measured in non inhibitor solution, the current density of anode and cathode decreased at a single potential[29].From the data of polarization curve in Table 1, when other conditions are consistent, the slow-release efficiency of LM is 46.05%, and that of LG is 87.98%. The release efficiency of LG is much higher than that of LM. Similarly, in the impedance data in Table 2, the slow-release efficiency of LM is 49.41%, and that of LG is 96.38%. The results are consistent with the trend of polarization curve. The results show that LG is better than LM when the conditions are the same.

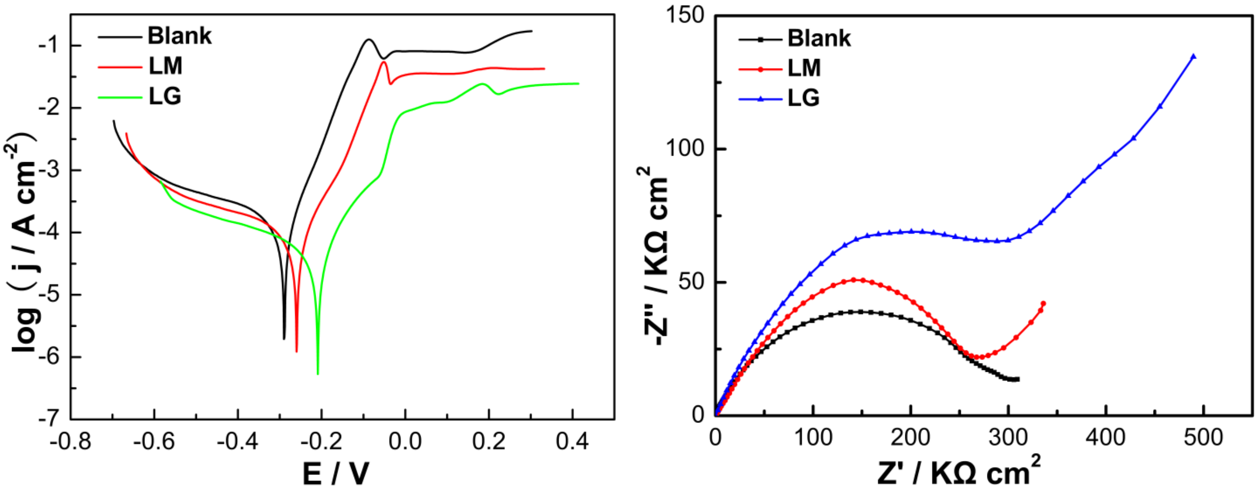

Figure 4. In 1M/L HCl solution, the slow-release effects of inhibitor-free, LM and LG on copper corrosion inhibition with the same concentration were compared at 25°C.

Table 1. Electrochemical parameters and inhibition efficiency obtained from polarization curves for copper in 1M/L HCl solution, the slow-release effects of inhibitor-free, LM and LG on copper corrosion inhibition with the same concentration were compared at 25°C.

Inhibitors	-E_{corr}(V)	ba(mV dec^{-1})	-bc(mV dec^{-1})	i_{corr}(μA cm^{-2})	η%
Blank	0.288	40	73	90.57	
LM	0.261	44	63	48.86	46.05%
LG	0.211	30	48	10.88	87.98%

Table 2. Electrochemical parameters and inhibition efficiency obtained from impedance curves for copper in 1M/L HCl solution, the slow-release effects of inhibitor-free, LM and LG on copper corrosion inhibition with the same concentration were compared at 25°C.

Inhibitors	R_S (Ω cm^2)	R_p (Ω cm^2)	C_{dl}		η %
			Y_0(Ω^{-1}cm^{-2}S^n)	n	
Blank	0.0795	19.56	0.0507	0.0467	
LM	0.0927	38.67	0.0117	0.3217	49.41
LG	0.6231	540.40	0.0013	0.4979	96.38

It is noted that, when the inhibitor is present, the copper corrosion is inhibited effectively[30]. When the concentration of surfactant is the same, the polarization curve of LG moves to the right of LM, and the higher the capacitance arc of EIS curve, the better the corrosion inhibition effect. This is because the adsorption mechanism of imidazoline surfactants is that the polar functional groups adsorb on the metal surface, while the non-polar functional groups form a dense protective film as part of the tail opposite to the metal surface. Compared with LM, LG has a unique structure with two hydrophilic groups and two hydrophobic groups, which makes the protective film more compact. Thus we can see that imidazoline surfactants have excellent corrosion inhibition performance, and the corrosion inhibition performance of imidazoline Gemini surfactants is better than that of traditional single-chain imidazoline inhibitors. Specific parameters are shown in Table 1 and Table 2.

3.1.2 Concentration of different inhibitors

The effect of gemini imidazoline inhibitor concentration (100, 200, 300, 400 and 500 mg/L) on the corrosion inhibition performance was studied in hydrochloric acid solution with 1 mol/l and other conditions unchanged.

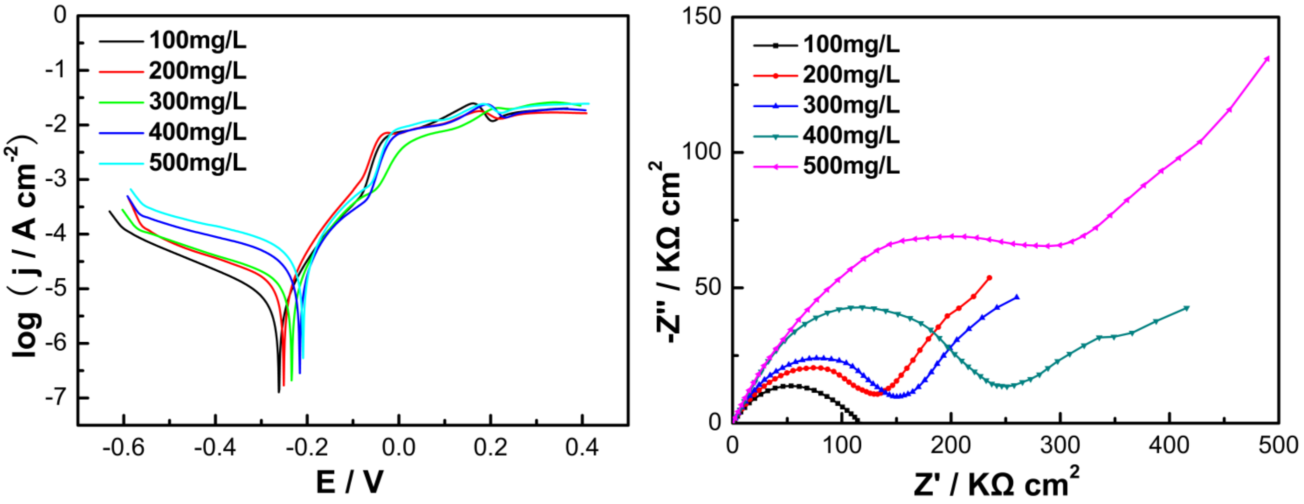

Figure 5. Polarization and impedance curves of LG with different concentrations in 1M/L HCl solution at 25℃

As shown in Figure 5, it can be found that the more the surfactant concentration, the more positive the potentidynamic polarization curves moves, and the higher the capacitance arc of impedance curves the better the corrosion inhibition effect. This indicates that with the increase of surfactant concentration, the corrosion inhibition effect is also significantly improved. The results show that the size of high frequency semicircle in impedance curves increases with the addition of inhibitor in the solution, and further increases with the increase of inhibitor concentration. As the semicircle of high frequency capacitance is related to the charge transfer reaction at the interface, the corrosion resistance of steel can be improved by increasing the concentration of inhibitor. The low frequency straight line is attributed to the Warburg impedance of diffusion, which indicates that the formed inhibitor film may affect the diffusion process of corrosive substances.[31]. The electrochemical parameters are shown in Table 3 and Table 4. It can be seen from the data of polarization curve in Table 3 that with the increase of LG concentration from 100mg/L to 500mg/L, the release efficiency will increase, from 28.30% to 87.98%. At the same time, it can be seen from the data of impedance curve in Table 4 that under the same conditions, with the increase of LG concentration, the release efficiency increased from 76.47% to 96.38%. The trend is the same as that of polarization curve data, indicating that when other conditions are the same, the release efficiency increases with the increase of LG concentration. The protective effect of film-forming inhibitor is that with the increase of inhibitor concentration in solution, the adsorption amount of corrosion inhibitor on metal surface increases, the film-forming effect increases, and the slow-release effect becomes better.

Table 3. Electrochemical parameters and inhibition efficiency obtained from polarization curves for copper in 1mol/l hydrochloric acid solution with different concentrations of LG inhibitor at 25℃

C(mg L^{-1})	-E_{corr}(V)	ba(mV dec^{-1})	-bc(mV dec^{-1})	i_{corr}(μA cm^{-2})	η%
100	0.266	35	27	64.86	28.39%
200	0.253	32	25	49.54	45.30%
300	0.235	27	25	23.28	74.30%
400	0.218	21	20	15.81	82.54%
500	0.211	30	48	10.88	87.98%

Table 4. Electrochemical parameters and inhibition efficiency obtained from impedance curves for copper in 1mol/l hydrochloric acid solution with different concentrations of LG inhibitor at 25℃

C (mg L^{-1})	R_S (Ω cm^2)	R_p (Ω cm^2)	C_{dl}		η %
			Y_0($Ω^{-1}cm^{-2}S^n$)	n	
100	0.7000	83.1	0.0011	0.4902	76.47
200	0.8084	173.9	0.0589	0.6532	88.75
300	0.5600	201.1	0.0014	0.5055	90.27
400	1.2916	348.7	0.0008	0.4984	94.39
500	0.6231	540.4	0.0013	0.4979	96.38

3.1.3 Concentration of different solution

The inhibition effect of the same concentration of Gemini imidazoline inhibitor in different hydrochloric acid solutions (0.5, 1.0, 2.0, 3.0 and 4.0 mol/L) was studied.

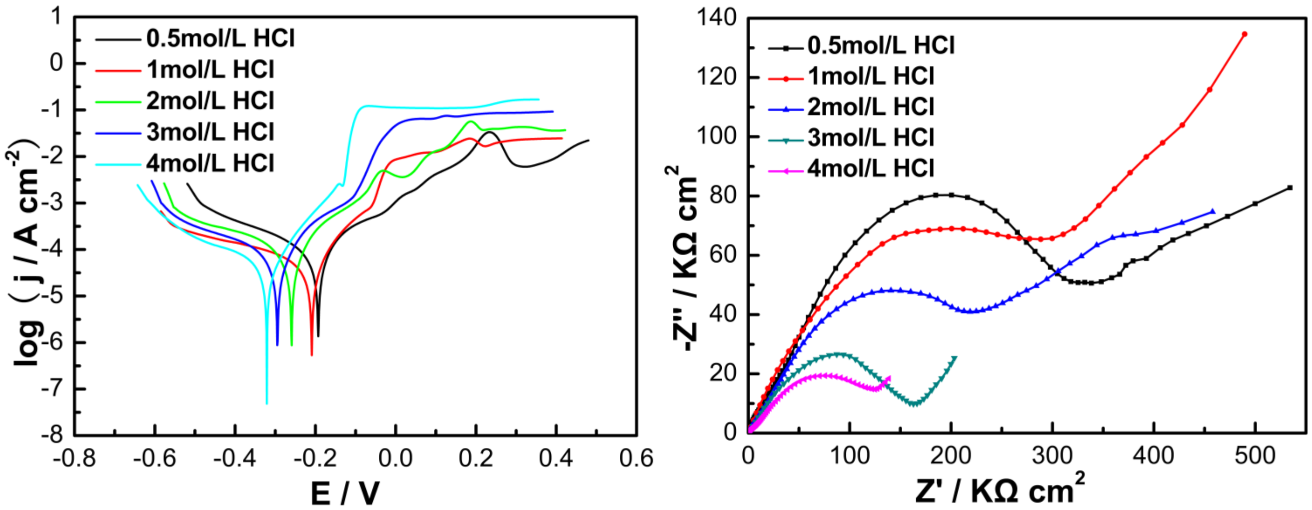

Figure 6. Polarization and impedance curves of 500mg/L LG in different concentrations of hydrochloric acid solution at 25℃.

The corrosion process of hydrochloric acid may affect the content of $CuCl^{2-}$ on the surface of copper electrode and indirectly affect the quality of self-assembled film. In order to confirm this conjecture, different concentrations of $CuCl^{2-}$ were produced by dropping the same concentration of LG on the surface of copper electrode in different concentrations of hydrochloric acid solution[32,33]. As shown in Fig.6, it is found that with the increase of HCl concentration, the corrosion current density decreases and the curve moves negatively. This indicates that the higher the concentration of hydrochloric acid is, the stronger the corrosion will be when other conditions remain unchanged. This is because with the increasing concentration of HCl solution, the amount of Cl^- in the solution increases, which promotes the reaction to proceed in a positive direction, which promote the production of such corrosion products as $CuCl^{2-}$,and making the corrosion degree greater. Specific parameters are shown in Table 5 and Table 6. It can be seen from the data of polarization curve in Table 5 that when the LG concentration is constant, the release efficiency decreases from 88.65% to 58.89% with the increase of hydrochloric acid concentration. From the data of impedance curve in Table 6, it can be seen that under the same conditions, the slow-release efficiency is reduced from 96.50% to 36.71%, and the trend of slow-release effect is consistent with the polarization curve. The results showed that the higher the concentration of hydrochloric acid, the worse the release effect. With the increase of hydrochloric acid concentration, the content of $CuCl^{2-}$ on the surface of copper electrode increases, and the corrosion of the self-assembled film increases obviously. Therefore, it is reasonable to think that the corrosion process of hydrochloric acid has a great influence on the quality of the self-assembled film formed on the surface of the copper electrode, which is mainly due to the different content of $CuCl^{2-}$ produced on the surface of the electrode[33].

Table 5. Electrochemical parameters and inhibition efficiency obtained from polarization curves for copper with 500mg/L LG in different concentrations of hydrochloric acid solution at 25°C.

C(mg L^{-1})	$-E_{corr}$(V)	ba(mV dec^{-1})	-bc(mV dec^{-1})	i_{corr}(μA cm^{-2})	η%
0.5	0.196	57	59	10.28	88.65%
1	0.211	30	48	10.88	87.98%
2	0.258	52	47	25.06	72.33%
3	0.294	50	65	31.84	64.84%
4	0.319	25	55	37.23	58.89%

Table 6. Electrochemical parameters and inhibition efficiency obtained from impedance curves for copper with 500mg/L LG in different concentrations of hydrochloric acid solution at 25°C.

C (mg L^{-1})	R_S (Ω cm^2)	R_p (Ω cm^2)	C_{dl}		η %
			Y_0($\Omega^{-1}cm^{-2}S^n$)	n	
0.5	0.6467	560.1	0.0009	0.535	96.50
1	0.6231	540.4	0.0013	0.4979	96.38
2	0.4703	452.4	0.0021	0.809	95.67
3	0.4594	60.27	0.0024	0.4473	67.09
4	0.7148	30.91	0.0008	0.4946	36.71

3.2 Inquiry into Inhibiting Mechanism by Surface Morphology Method

The results of photographic volumetric microscopy are shown in the figure. Figure 7 shows a panoramic view of the surface of copper electrode magnified 25 times, and Figure 8 shows a local view of the surface of copper electrode magnified 50 times.

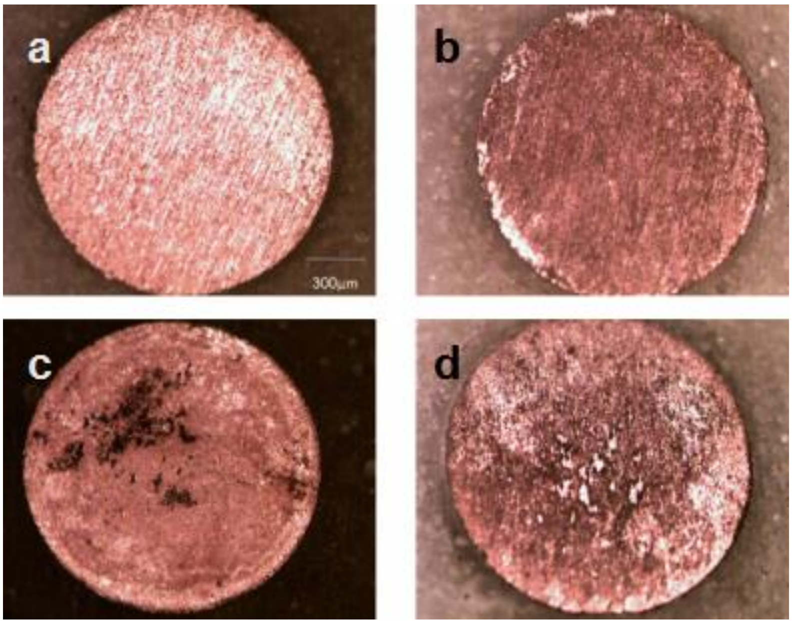

Figure 7. The overall picture of copper surface enlarged 25 times under different conditions

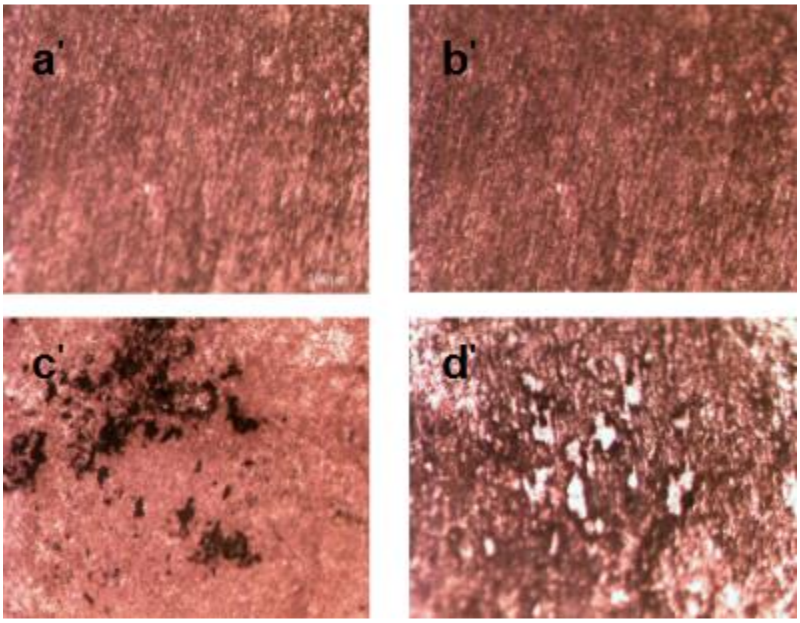

Figure 8. Local graphs of copper surface enlarged 50 times under different conditions

In the figure, a is the whole picture of the copper electrode without corrosion, b is the whole picture of the copper electrode with LG, c is the whole picture of the copper electrode without corrosion after applying potential to test in the same acid solution, and d is the whole picture of the electrode after applying potential to test in the above acid solution after adding LG. The a',b',c',d' in the graph is the local image of the a,b,c,d enlarged in the graph. The corrosion inhibition mechanism was analyzed by comparing these graphs.

(1) Whether from the panoramic view or the local view, we can clearly see that the surface of the uncorroded copper electrode is relatively bright, the morphology is relatively regular, and the surface structure is relatively neat. With the corrosion in acid solution, the surface gloss becomes darker, many corrosion pits appear, and the surface flatness becomes worse, indicating that serious corrosion has taken place. After adding the LG, there is a layer of film on the surface of the electrode, and after the test, the film appears to ablate locally, while copper itself has no obvious signs of corrosion, which indicates that the LG has a good corrosion inhibition effect on the copper electrode, which is consistent with the results of electrochemical method[34].

(2) As can be seen from the two graphs of a and c, there is also a big gap between the applied potential and the non-applied potential. The corrosion of the applied potential surface is much more serious than that of the non-applied potential surface. These comparisons show that the size of the potential has a direct impact on the corrosion. From the Perspective of the electrochemical mechanism, the negative movement of the anode potential and the cathodic potential are discussed. The positive movement constitutes the corrosion potential of copper. After applying the potential, the corrosion becomes serious, which indicates that the negative shift of the anode potential becomes larger, and further indicates that the corrosion current caused by the corrosion potential in the anode region can dissolve rapidly. The addition of corrosion inhibitor controlled the corrosion, which indicated that water-soluble imidazoline slowed down the dissolution of the anode and positively moved the corrosion

potential[35-36]. From this point of view, the LG was the main anodic corrosion inhibitor to inhibit the dissolution of the anode, and so was the result of polarization curve analysis.

(3) As can be seen from the figures c' and d', there are many corrosion pits on the surface of the corroded electrode, which are not uniformly distributed. This is an obvious pitting phenomenon. Compared with the overall corrosion, pitting is more difficult to control. The reason of pitting corrosion on the surface of the copper electrode is that copper belongs to passivation metal and contains active anion Cl-, which selectively adsorbs on the surface of the copper metal, especially at relatively weak points, so that these points are more easily corroded, thus forming many pitting corrosions. The addition of LG improved the pitting corrosion obviously, which indicated that LG had a good inhibition effect on the pitting corrosion of metals. This indicates that LG has multi-center adsorption on the surface of the copper electrode, and preferentially adsorbed on the surface of the metal surface. As far as the physical and chemical mechanism is concerned, the imidazoline ring in LG contains N atoms and non-covalent electron pairs, while copper has empty D orbital, which forms the relationship between donor and acceptor, makes them form coordination bonds, and makes LG adsorb on copper[37]. This is a way of chemical adsorption. In addition, this may be due to the double bond pion electrons on the imidazoline ring, which makes the chemical adsorption easier to form.

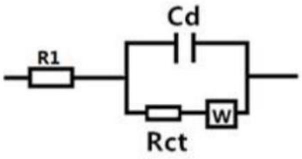

Figure 9. Equivalent circuit diagram about Impedance.

This is due to the formation of an adsorbed layer on the surface of the copper by the inhibitor molecule. The equivalent circuit diagram is roughly shown in Figure 9[14], where R1 is solution resistance, Rct is charge transfer resistance, W is concentration polarization resistance, and Cd is surface double layer capacitance. The equivalent circuit diagram can be understood as follows: the adsorption of inhibitor molecules leads to the increase of film resistance and capacitance characteristics, and then leads to the increase of charge transfer resistance and surface double layer capacitance, thus reducing the corrosion current and slowing down the corrosion. Thus, the impedance value is changed and the corrosion inhibition effect is achieved.

4. CONCLUSIONS

After the synthetic modification experiment, corrosion inhibition performance evaluation experiment and corrosion inhibition mechanism exploration experiment, the conclusions are as follows:

(1) when other conditions are unchanged, Gemini imidazoline inhibitors have better corrosion inhibition than their monomers.

(2) when other conditions are unchanged, the higher the concentration of the Gemini imidazoline inhibitor is, the better the corrosion inhibition effect will be.

(3) Gemini-imidazoline inhibitor has better corrosion inhibition effect on pure copper under low concentration acid environment when other conditions remain unchanged.

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