

Short Communication

Study on Corrosion Resistance of Carbon Steel in HF/HCl Acid Mixture in Presence of *Lycoris radiata* and *Lycoris chinensis* Leaf Extract as Environmentally Friendly Corrosion Inhibitor

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The nitrogen and oxygen elements in natural plant protein can supply electrons. The concrete hole solution in the protein hydrolysis produced by the amino and carboxyl groups is good for electron groups, which slows down the corrosion. In this work, *Lycoris radiata* and *Lycoris chinensis* leaves were extracted, and environmentally-friendly corrosion inhibitors were prepared. The performance of corrosion inhibitors were evaluated with weight loss method and electrochemical method. This work aimed to investigate the anti-corrosion properties of single plant extracts, and prepare a composite corrosion inhibitor. The experimental results reveal that the strongest synergistic effect can be presented when *Lycoris radiata* and *Lycoris chinensis* are paired in a 2:3 ratio.

Keywords: Carbon steel; Plant extract; Acid solution; Corrosion; Polarization

1. INTRODUCTION

Steel is one of the primary building materials because of its abundant raw material sources, low price, as well as simple and mature production process. Under conventional circumstances, the concrete is protective of the reinforcing steel embedded in it, because the highly alkaline pore solution of calcium hydroxide, potassium and sodium ions produced by the hydration of cement can passivate the surface of the reinforcing steel to form a thick protective oxide film [1–3]. However, the carbonation caused by pH reduction or contamination brought by chloride will destroy the passivation film caused by the occurrence of corrosion of reinforcing steel, and then the accumulation of corrosion products will generate sudden expansion stress, resulting in cracking and spalling [4–6]. There are various protection and repair methods and technologies for the corrosion prevention of reinforcement, such as the application of internal corrosion inhibitor, the use of corrosion-resistant reinforcement and epoxy-coated

reinforcement [7], as well as cathodic protection method, realkalization technology, electrochemical dechlorination, etc.

Corrosion inhibitor for steel is a substance that is added to concrete in sufficient quantity to stop or slow down the corrosion of buried reinforcing steel and has no adverse effect on the performance [8–11]. Its substantial role is to stop or retard the electrochemical reaction of the corrosion of reinforcing steel and to reduce the corrosive effect of aggressive ions entering the reinforcing steel. Inorganic corrosion inhibitors, represented by calcium nitrite, molybdate, and phosphate, generally passivate the surface of reinforcing steel or form a deposition film on its surface to perform excellent corrosion inhibiting effect in steel [12,13]. However, with the increasing awareness of environmental protection, especially low energy-consumption and zero-emission, most inorganic corrosion inhibitors have been limited in their application due to their toxicity or high costs, such as traditional high-effect corrosion inhibitors based on phosphate, chromate, and nitrite. Moreover, phosphate corrosion inhibitors are rich in phosphate, making lake and river nutrients polluted, thus its widespread application is limited [14–16]. With the urgent demand of modern society for environmental indicators such as low energy-consumption, zero-emission, etc., some harmful, toxic, high-cost corrosion inhibitors will gradually be restricted and banned [17–19]. In recent years, environmentally friendly corrosion inhibitors have received extensive attention of domestic and foreign researchers and became one of the main directions of future development of corrosion inhibitors. Corrosion inhibitors extracted from plants are natural and harmless, which can realize the sustainable use of resources [20]. Hence it will be the new direction of research and development of corrosion inhibitors in the future.

This paper focuses on the corrosion of reinforcement caused by chloride salts in steel. Based on the competition of *Lycoris radiata* and *Lycoris chinensis* leaf extracts with chloride ions on the surface of steel for adsorption to hinder corrosion, effective corrosion inhibitors were explored. In this work, the change of corrosion rate of carbon steel was investigated under the conditions of changing the concentration, acidity, time, and temperature of *Lycoris radiata* and *Lycoris chinensis* extracts with the loss-in-weight method and electrochemical method. The best ratio of synergistic corrosion inhibitors were selected according to the corrosion-inhibiting effects.

2. EXPERIMENTAL

2.1. Materials

The steel bar ($\Phi 6\text{mm}$) purchased from Dongguan Yimaxin Metal Co. was cut into sections about 100 mm long. Sodium chloride, sodium hydroxide, hydrochloric acid, hydrofluoric acid, ethanol, and potassium hydroxide were purchased from Shanghai State Pharmaceutical. Both *Lycoris radiata* and *Lycoris chinensis* were purchased from local flower gardens and identified by the Nanjing Botanical Garden.

2.2. Preparation of extracts from *Lycoris radiata* and *Lycoris chinensis*

The two dried plant tissues were ground and sieved through a 350 μm sieve, and 100 g of the sieved plant powder was dissolved in 1 L of water. 2 g of α -amylase was added to the water and the pH was adjusted to about 6.0 with 2 M of NaOH. Afterwards, the solution was heated in a water bath at 70°C and stirred with a magnetic rotor for 2 h. It was then washed with water to precipitate three times before use. 10 g of the above-washed precipitate was taken and dissolved in 100 mL of acetone, being extracted for 30 min and centrifuged for 15 min to obtain the precipitate for use. The above precipitate was dissolved in an aqueous solution that is 10 times the mass of 70% ethanol, after which it was dissolved in a water bath at 60°C and stirred with magnetic rotor for 2 h. The precipitate was obtained after 15-min centrifugation. The above precipitate was dissolved in a solution that is 10 times the mass of 0.1 M NaOH solution and heated and stirred for 2h in a water bath at 60°C. The solution was centrifuged for 15 min to obtain the supernatant for use, and the precipitate was repeatedly alkaline and dissolved once to obtain the supernatant for use. The pH of the supernatant at night was adjusted to about 4.5 with dilute industrial hydrochloric acid (10 wt.%) to obtain the suspension solution, which was centrifuged for 15 min to obtain the precipitate. The precipitate was then washed with deionized water to remove the chloride ions and dried at 40°C to obtain the plant extract.

2.3. Pre-treatment of reinforcing steel bar

The steel bars were first placed in an acid solution that the ratio of hydrochloric acid (36 wt.%) to water is 1:2 by volume until the surface iron oxide was dissolved and removed. The bars were sanded with 400 mesh, 600 mesh, 800 mesh, and 1000 mesh sandpaper until they were close to white iron, after which they were cleaned and dried successively with distilled water and ethanol. A nickel wire was welded on one end of each bar and both ends were sealed with epoxy resin to avoid and mitigate the effects of crevice corrosion or tip corrosion (the exposed area of the bar cross-section is 1 cm^2). The bars were finally sealed with an oil dip, washed, and dried with ethanol and water before use in the corrosion test.

2.4. Weight loss determination

The carbon steel bar was sanded smooth with sandpaper, washed with water, and then wiped with anhydrous ethanol and acetone. The carbon steel bar was accurately weighed after cold air drying (W_0). A certain concentration of hydrofluoric acid, hydrochloric acid, or HF/HCl acid mixture (containing 10% HCl and 3% HF) was placed in a beaker with plant extract (3 g was added in every 100 mL acid) and a steel bar into a beaker. After 12 h of reaction at room temperature, the carbon steel specimens were washed with distilled water, dried, and weighed (W). The weight loss of the steel bar was calculated before and after the reaction. The corrosion rate (V) of carbon steel and the corrosion inhibition rate (η) of the extraction solution were calculated with the following notation:

$$\text{Corrosion rate: } V = (W_0 - W) / S \cdot t$$

2.5. Electrochemical determination

All electrochemical experiments were conducted with a CHI660E electrochemical workstation and a three-electrode system was applied for analysis. A saturated calomel electrode (SCE), a Pt electrode, and a steel bar were used as the reference electrode, counter electrode, and working electrode, respectively. We determined the polarization curves of plant extracts in acid systems for carbon steel in different temperature environments on an electrochemical analyzer. Corrosion inhibition efficiency was determined by the change in magnitude of corrosion current. Potentiodynamic polarization measurements were performed by scanning the potentials at a constant scan rate of 1 mV/s. The Tafel slopes of the cathode and anode were adopted to determine the performance of the inhibitor.

3. RESULTS AND DISCUSSION

The corrosion inhibition effect of *Lycoris radiata* and *Lycoris chinensis* extracts on carbon steel in acid solution is shown in Table 1. From the data in the table, it can be seen that both *Lycoris radiata* and *Lycoris chinensis* extracts have a good effect on the corrosion inhibition of carbon steel. Extracting active ingredients from natural plants as corrosion inhibitors and replacing traditional corrosion inhibitors with new corrosion inhibitors that are widely available, inexpensive, environmentally friendly, and non-toxic to human beings is an idea that has emerged from the research field of steel corrosion [21–23]. Current research has shown that some substances have corrosion-inhibiting properties and potential in concrete, such as substances containing polyphenolic compounds such as alkaloids, flavonoids, and tannins, as well as substances with atoms and groups capable of providing lone pairs of electrons such as sugars, amino acids, organic acids, and vegetable oil esters [24,25].

Table 1. The corrosion inhibition efficiency of 3% *Lycoris radiata* and *Lycoris chinensis* extract on carbon steel in 5% HCl, 5% HF and 5% HF/HCl acid mixture.

Plant	Corrosion rate (g·m ² ·h)	η (%)
5% HCl		
Blank	11.501	-
<i>Lycoris radiata</i>	1.021	91.44
<i>Lycoris chinensis</i>	0.977	91.51
5% HF		
Blank	7.584	-
<i>Lycoris radiata</i>	1.847	85.54
<i>Lycoris chinensis</i>	1.511	86.07
5% HF/HCl acid mixture		
Blank	12.151	-
<i>Lycoris radiata</i>	0.787	88.89
<i>Lycoris chinensis</i>	0.805	87.79

In this study, the corrosion inhibition properties of *Lycoris radiata* in HF/HCl acid mixture was first explored. Different concentrations of *Lycoris radiata* extracts were added to 5% HF/HCl acid mixture in the order of 0%, 0.5%, 1%, 1.5%, 2%, 2.5%, 3%, 3.5%, 4%, 4.5%, 5%, 5.5% and 6%. The temperature in the reaction environment was 25°C, and the reaction time was 12 h. The weight loss of carbon steel was examined and the curve between corrosion rate and *Lycoris radiata* extraction concentration was plotted (Figure 1A). As shown in the figure, the corrosion rate of *Lycoris radiata* increases with the concentration of the extract. When the concentration of *Lycoris radiata* extract is greater than 2%, the corrosion rate changes less, the value almost levels off, and the carbon steel surface is in the adsorption saturation state. Therefore, the concentration of *Lycoris radiata* extract was finally determined to be 3% for the following experiments.

Changes in acidity can also affect the corrosion of metals [26]. As shown in Figure 1B, when the soil acidity is below 5%, the corrosion rate is $1.0\text{ g}\cdot\text{m}^{-2}/\text{h}$. Therefore, the acid tolerance range of *Lycoris radiata* extracts is below 5%. Lower acidity causes slower corrosion.

Figure 1C shows the curves of corrosion rate and time of carbon steel when 3% *Lycoris radiata* extract was added. Increasing the experimental time can enhance the protective effect on carbon steel, which indicates that the *Lycoris radiata* extracts are time resistant.

Temperature is one of the main factors in evaluating corrosion resistance [27,28]. Figure 1D presents the relationship between corrosion rate and temperature from 25°C to 60°C. It can be seen from the figure that the curve tends to rise with temperature. When the temperature exceeds 40°C, the curve increases more obviously. It can be noted that the temperature resistance of *Lycoris radiata* extract is weak, and it is recommended to be used in the environment at 40°C.

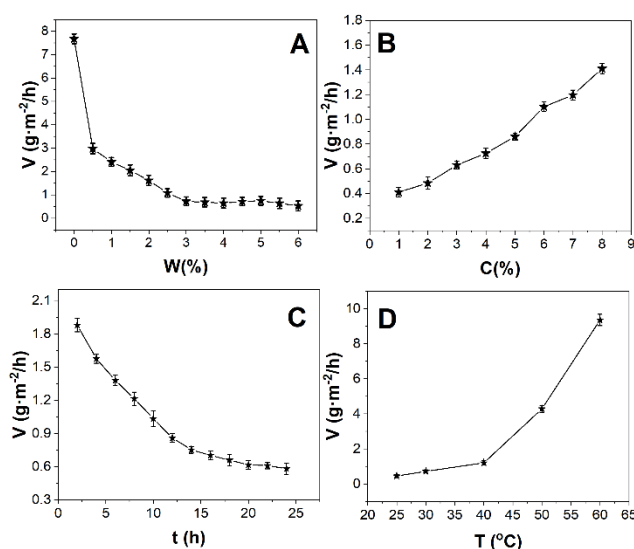


Figure 1. (A) Relationship between the corrosion rate and the concentration of *Lycoris radiata* extract. (B) Relationship between the corrosion rate and the concentration of HF/HCl acid mixture. (C) Relationship between the corrosion rate and corrosion time. (D) Relationship between the corrosion rate and temperature.

After determining the optimal conditions for using *Lycoris radiata* extracts, we controlled the temperature with a water bath and measured the polarization curves at different temperatures (Figure 2). Table 2 shows the corresponding electrochemical corrosion parameters. It can be found that the corrosion currents are smaller after *Lycoris radiata* extract was added than that before it was added. The Tafel slopes of both cathode and anode change, indicating that their corrosion processes are inhibited, and therefore *Lycoris radiata* extract can be a mixed corrosion inhibitor. The above experimental results reveal that in 5% HF/HCl acid mixture, 3% of *Lycoris radiata* extract at 40°C can play a good corrosion inhibition with a corrosion inhibition rate of about 92%.

Table 2. Electrochemical corrosion parameters deduced from the polarization curves with 3% *Lycoris radiata*.

Temperature (°C)	System	E_{cor} (V)	I_{cor} (mA/cm ²)	β_a (mV/dec)	β_c (mV/dec)
25	HF/HCl acid mixture	-0.369	0.055	0.542	0.603
	HF/HCl acid mixture + <i>Lycoris radiata</i>	-0.312	0.023	0.932	0.637
30	HF/HCl acid mixture	-0.322	0.069	0.579	0.532
	HF/HCl acid mixture + <i>Lycoris radiata</i>	-0.316	0.048	0.643	0.614
40	HF/HCl acid mixture	-0.319	0.072	0.457	0.524
	HF/HCl acid mixture + <i>Lycoris radiata</i>	-0.303	0.049	0.868	0.679
50	HF/HCl acid mixture	-0.310	0.079	0.503	0.569
	HF/HCl acid mixture	-0.300	0.040	1.100	1.257

	+				
		<i>Lycoris radiata</i>			
60	HF/HCl acid mixture	-0.302	0.083	0.538	0.544
	HF/HCl acid mixture	-0.301	0.057	0.915	1.495
	+				
	<i>Lycoris radiata</i>				

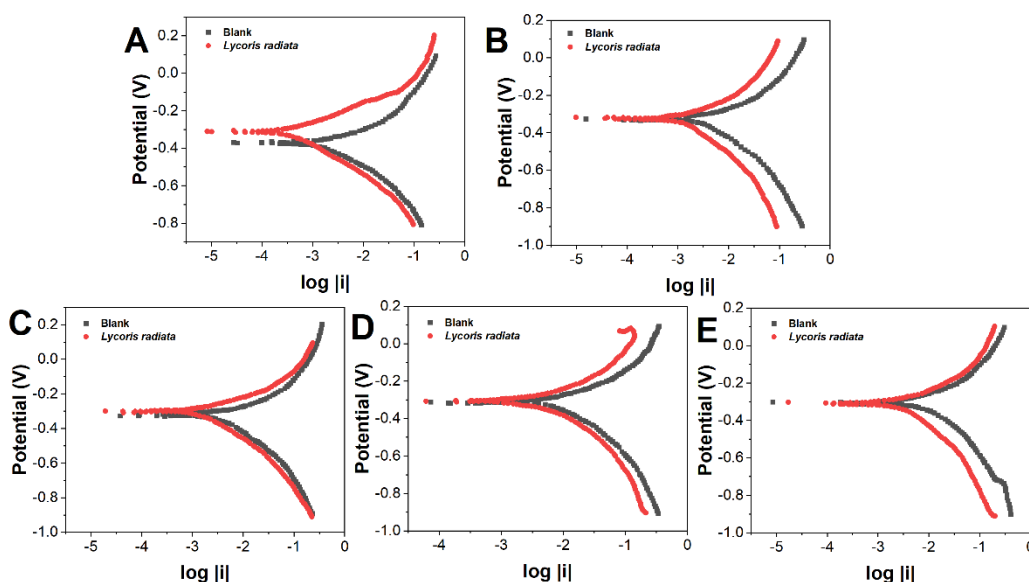


Figure 2. Polarization curves of the rebar in the HF/HCl acid mixture (5%) with 3% *Lycoris radiata* extract under (A) 25°C, (B) 30°C, (C) 40°C, (D) 50°C and (E) 60°C at a 1 mV/s scan rate.

Afterwards, the corrosion inhibition properties of *Lycoris chinensis* in HF/HCl acid mixture were explored. Different concentrations of *Lycoris chinensis* extracts were added to 5% HF/HCl acid mixture, and the temperature in the reaction environment was 25°C with a reaction time of 12 h. The weight loss of carbon steel was examined and the curve between corrosion rate and *Lycoris chinensis* extraction concentration was plotted (Figure 3A). It can be noted from the figure that the degree of corrosion gradually decreases with the increase of *Lycoris chinensis* concentration. When the concentration of *Lycoris chinensis* extract is greater than 3%, the curve becomes smooth, which means that the carbon steel surface is saturated with adsorption at this time. Therefore, the concentration of *Lycoris chinensis* extract was also determined to be 3% for the following experiments.

Changes in acidity can also affect the corrosion of metals [29]. As shown in Figure 3B, the curve increases with the concentration of HF/HCl acid mixture, and the acid tolerance range of *Lycoris*

chinensis extracts is up to the concentration of 5% HF/HCl acid mixture. The corrosion rate is below $1.0\text{g}\cdot\text{m}^{-2}/\text{h}$.

Figure 3C shows the curves of corrosion rate and time of carbon steel as 3% *Lycoris chinensis* extract was added. The corrosion rate decreases with time, and the corrosion inhibition effect of *Lycoris chinensis* extract on carbon steel is stable after 12 h of reaction time.

Figure 1D presents the relationship between corrosion rate and temperature from 25°C to 60°C . As can be seen from the figure, the increase in temperature severely affects the corrosion of carbon steel, especially above 40°C .

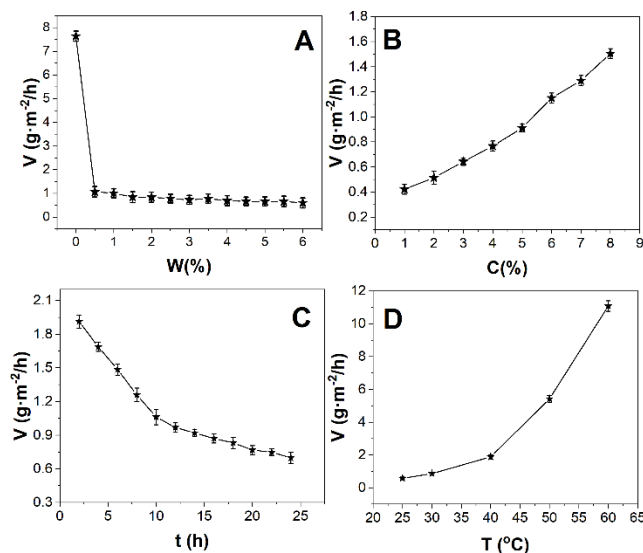


Figure 3. (A) Relationship between the corrosion rate and the concentration of *Lycoris chinensis* extract. (B) Relationship between the corrosion rate and the concentration of HF/HCl acid mixture. (C) Relationship between the corrosion rate and corrosion time. (D) Relationship between the corrosion rate and temperature.

Table 3. Electrochemical corrosion parameters deduced from the polarization curves using *Lycoris chinensis*.

Temperature (°C)	System	E_{cor} (V)	I_{cor} (mA/cm^2)	β_a (mV/dec)	β_c (mV/dec)
25	HF/HCl acid mixture	-0.369	0.055	0.542	0.603
	HF/HCl acid mixture + <i>Lycoris chinensis</i>	-0.322	0.032	0.632	1.001
30	HF/HCl acid mixture	-0.322	0.069	0.579	0.532

	HF/HCl acid mixture + <i>Lycoris chinensis</i>	-0.298	0.034	0.681	1.124
40	HF/HCl acid mixture	-0.319	0.072	0.457	0.524
	HF/HCl acid mixture + <i>Lycoris chinensis</i>	-0.297	0.062	0.569	0.525
50	HF/HCl acid mixture	-0.310	0.079	0.503	0.569
	HF/HCl acid mixture + <i>Lycoris chinensis</i>	-0.298	0.072	1.122	1.243
60	HF/HCl acid mixture	-0.302	0.083	0.538	0.544
	HF/HCl acid mixture + <i>Lycoris chinensis</i>	-0.298	0.079	1.594	2.978

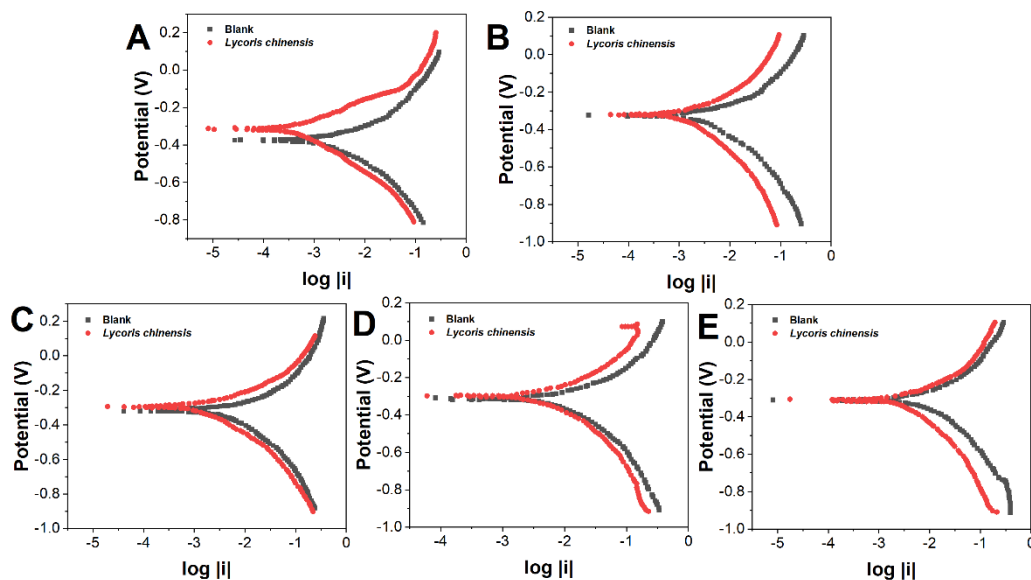


Figure 4. Polarization curves of the rebar in the HF/HCl acid mixture (5%) with 3% *Lycoris chinensis* extract under (A) 25°C, (B) 30°C, (C) 40°C, (D) 50°C and (E) 60°C at a 1 mV/s scan rate.

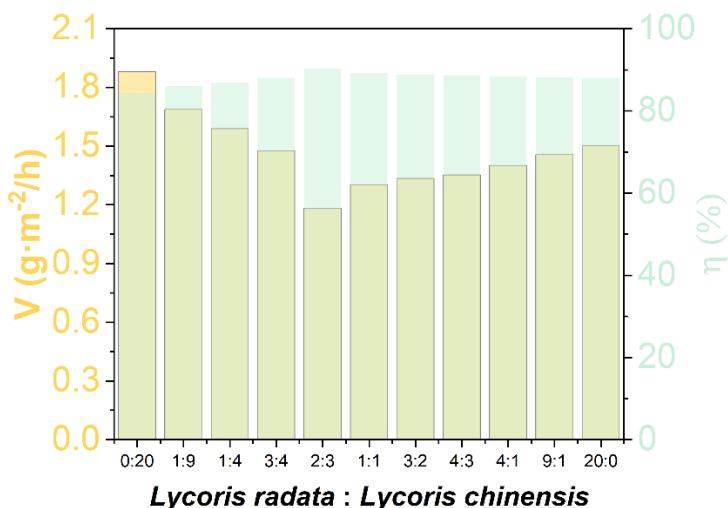


Figure 5. The corrosion rate of different ratios between *Lycoris radata* and *Lycoris chinensis* in 5% HF/HCl acid mixture.

After determining the optimal conditions of using *Lycoris chinensis* extracts, we controlled the temperature with a water bath and measured the polarization curves at different temperatures (Figure 4). Table 3 shows the corresponding electrochemical corrosion parameters. As seen from the figure and table, the properties of the added *Lycoris chinensis* are very similar to those of the *Lycoris radiata* extract, which means that it is also a mixed corrosion inhibitor.

Through the experiments, it is found that the extracts of *Lycoris radata* and *Lycoris chinensis* may have a synergistic effect and enhance the corrosion resistance of carbon steel. Figure 5 shows the anti-corrosion effect of different ratios of *Lycoris radata* and *Lycoris chinensis* in 5% HF/HCl acid mixture. The experimental results reveal that the performance of the mixed corrosion inhibitors is enhanced as the percentage of *Lycoris chinensis* increases. The best corrosion inhibition performance can be achieved as the ratio of *Lycoris radata* to *Lycoris chinensis* reaches 2:3. When the proportion of *Lycoris chinensis* continues to increase and the proportion of *Lycoris radata* continues to decrease, the corrosion rate of carbon steel begins to increase and the corrosion resistance efficiency decreases. The results suggest that *Lycoris radata* and *Lycoris chinensis* have synergistic effects.

Figure 6 presents the polarization curves of *Lycoris radata* (2%), *Lycoris chinensis* (2%) and *Lycoris radata*+*Lycoris chinensis* (1%+1%) extracts on carbon steel. Table 4 shows the corresponding electrochemical corrosion parameters. From the results, it can be inferred that adding *Lycoris radata* + *Lycoris chinensis* (1% + 1%) extracts has less rot candle current, compared with the case that a single extract of *Lycoris radata* or *Lycoris chinensis* is added. This indicates that *Lycoris radata* + *Lycoris chinensis* provides better anti-corrosion effect than single extract.

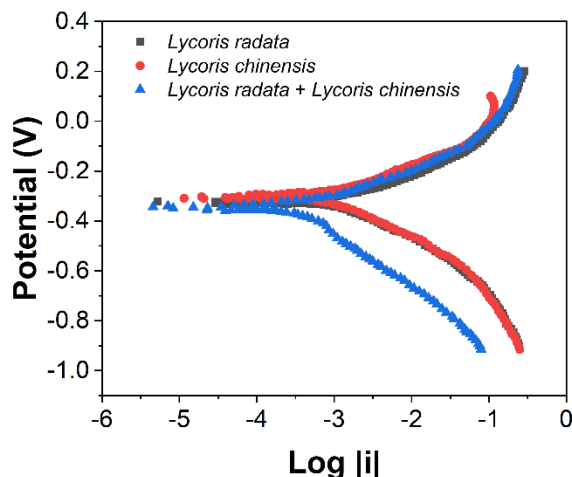


Figure 6. Polarization curves of the rebar in the HF/HCl acid mixture (5%) with *Lycoris radata* (2%), *Lycoris chinensis* (2%) and *Lycoris radata* + *Lycoris chinensis* (1%+1%) at a 1 mV/s scan rate.

Table 4. Electrochemical corrosion parameters deduced from the polarization curves of *Lycoris radata* (2%), *Lycoris chinensis* (2%) and *Lycoris radata*+*Lycoris chinensis* (1%+1%).

Temperature (°C)	System	E_{cor} (V)	I_{cor} (mA/cm ²)	β_a (mV/dec)	β_c (mV/dec)
25°C	Blank	-0.368	0.055	0.542	0.602
	<i>Lycoris radata</i> (2%)	-0.321	0.006	1.235	1.869
	<i>Lycoris chinensis</i> (2%)	-0.308	0.005	1.001	1.105
	<i>Lycoris radata</i> + <i>Lycoris chinensis</i> (1%+1%)	-0.344	0.001	2.667	4.782

In order to further verify that the corrosion inhibitor in the simulated pore solution can be adsorbed on the surface of the rebar to play a protective role, FTIR test was conducted on the extracted *Lycoris radata*+*Lycoris chinensis* powder and the surface of the rebar, with the results are shown in Figure 7. It can be seen from the figure that the surface adsorption of the soaked steel is dominated by the adsorption peaks of three functional groups, and the main functional groups of the surface adsorption of the steel are similar to those of the *Lycoris radata*+*Lycoris chinensis* extract. Among them, 1450 cm⁻¹ is the C-H stretching vibration of the alkane group [30], 1538 cm⁻¹ is the adsorption peak of the C=O stretching vibration of the characteristic peak of amide I, and 1655 cm⁻¹ is the characteristic peak of amide II, which is mainly the adsorption peak of C-N stretching with N-H bending [31]. The adsorption peak at 3400 cm⁻¹ is the characteristic peak of the hydroxyl group in the water molecule. Therefore, the adsorption of *Lycoris radata*+*Lycoris chinensis* on the surface of the reinforcing steel can be well demonstrated, which can effectively inhibits the corrosion of the reinforcing steel. This is in agreement with the results of previous studies on the adsorption of chitosan derivatives [32], banana peel extract [33], garlic extract [34] and other environmental corrosion inhibitors.

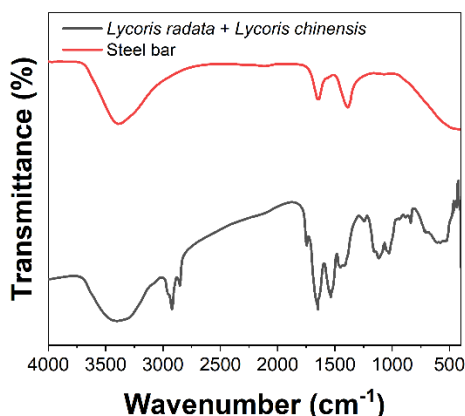


Figure 7. The FTIR spectra of pure *Lycoris radata* + *Lycoris chinensis* and adsorbate on steel bar.

4. CONCLUSION

To address the problem of chloride salt corrosion of reinforcing steel, this study extracted the leaves of *Lycoris radata* and *Lycoris chinensis* with alkali-soluble acid deposition method and prepared an environmentally friendly corrosion inhibitor suitable for steel protection. In addition, the corrosion inhibition effect of plant extracts on carbon steel in acid solution was investigated with weight loss method and the optimum addition amount was confirmed. Furthermore, the synergistic effect of *Lycoris radata* and *Lycoris chinensis* extracts was analyzed. Multi-component compounding of the extract mixture was carried out to find the optimal ratio.

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