

Rapid Synthesis and Evaluation of a Cheaper Corrosion Inhibitor for Steel in HCl Solution

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ABSTRACT: A corrosion inhibitor suitable for industrial production and field application was rapidly synthesized from benzene-1,2-diamine, benzaldehyde, methanol, hydrochloric acid, and FeCl_3 . The mechanism of corrosion and its inhibition was proposed. We investigated the effect of the main reactants on corrosion rate, and optimal synthetic composition (benzene-1,2-diamine 10 g, benzaldehyde 10 g and ferric chloride hexahydrate 8 g) of the inhibitor was determined by using an orthogonal experimental design. The corrosion inhibition performances for N80 and Q235 steel in acid medium and oilfield water-injection were studied by employing the weight loss method. The inhibition efficiency for N80 steel reached to 99.24% in 15 wt.% HCl solution for 4 hours at 90 °C at the dosage of 1.2 wt.%, while in the water-injection, the efficiency for Q235 steel was 82.4% at 50 °C for 7 days at the concentration of 30 mg/L. Corrosion rates in acid medium and water-injection showed that the inhibitor has good corrosion inhibitive behavior for the steel surface and the inhibition efficiency increased with increasing concentration. Three kinds of compounds with synergistic corrosion inhibition were introduced to study, and ammonium bromide exhibited the best synergy. Attempts were made to fit the test values to different isotherms, it was found that the adsorption of the inhibitor on Q235 steel surface obeys Langmuir adsorption isotherm. The calculated value ΔG_{ads}^0 indicated that the adsorption reaction could spontaneously occur and the inhibitor molecules could be well adsorbed on Q235 steel surface in oilfield water-injection at 50 °C.

KEYWORDS: Corrosion inhibitor; Orthogonal experimental design; Adsorption; Corrosion mechanism; Synergism.

INTRODUCTION

In China, the price of most of the corrosion inhibitors used for oil wells acidizing is generally between \$1800 and \$2300 per ton. Once the requested performance of corrosion inhibitor is harsh, the price reaches even more

than \$2900 per ton. However, applications of corrosion inhibitors do more good than harm. In 2000, the investigation report of corrosion losses in China exposed that the economic loss caused by corrosion in various

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industries reached up to \$30 billion, which accounted for 3.6% of the gross domestic product in the year. In addition, corrosion brought about a lot of security issues, 75% of the accidents in the petrochemical industry originated from the stress cracking of metal caused by corroding [1].

The main ingredients of wild steel are iron and its alloys, which are widely used in engineering applications [2-4]. Meanwhile, mild steel is also the main structural material in oilfields. Due to the presence of carbon dioxide, dissolved oxygen, and hydrothion in excessive water of oilfields, moreover, the temperature and flow rate of the water can also cause corrosion on the surface of mild steel [5]. Especially in oil wells acidizing and its plug removal, as well as in industrial acid pickling and surface treatment [6-8], corrosion becomes more serious. This is because that hydrogen ion of the acid can react with iron, resulting in dissolution of metal, then the corrosion gets under way. In order to reduce the corrosion of metal, corrosion inhibitor is added into the acid solution, which can be adsorbed on the surface of MS to form a chemical adsorption film in the acid solutions [9, 10]. The film as a barrier can prevent hydrogen ion contacting with the surface of mild steel to protect it from corrosion [11].

Compounds containing electronegative functional groups, π -electrons and heteroatoms like sulfur, nitrogen, oxygen, and phosphorus as well as aromatic rings in their structures, and some plant extract also exhibit good corrosion inhibitive ability in acid media [12-15]. In recent years, a lot of work related to various corrosion inhibitors were carried out, and a considerable amount of effective inhibitors were reported to have been synthesized [16]. Meanwhile, many electrochemical methods such as potentiodynamic polarization, electrochemical impedance spectroscopy, and linear polarization resistance were used for evaluating the properties of inhibitor. However, the limiting factors that complex production processes, high production cost, poor temperature resistance, and scarce materials source restrict the development of most of the corrosion inhibitors.

This work was carried out on the basis of the previous research reported in the literature [17, 18]. Due to the fact that the solubility of benzene-1,2-diamine is very low in water and HCl solutions at room temperature, resulting in that it cannot dissolve well in HCl solutions, the application of the novel corrosion inhibitor

is impeded. In order to facilitate the on-site application, in the synthesis process of the corrosion inhibitor, commercial hydrochloric acid solution and methanol were introduced into this experiment to make benzene-1,2-diamine soluble. The work aimed to determine the inhibition features of the inhibitor on steel surface using weight loss method, and put forward a synthesis method of a cheaper inhibitor suitable for industrial production and field application, the production cost of which is less than \$900 per ton.

EXPERIMENTAL SECTION

Materials

N80 and Q235 steel specimens were used in two kinds of corrosion tests, all of which were pre-treatment by mechanically polished the surface with 1200 grades of emery paper prior to each experiment, then cleaned successively using absorbent cotton in acetone and ethyl alcohol, dried at dry cabinet for balance weight before weighing them. All the used chemical materials except HCl solution for the synthesis of corrosion inhibitor in the present study were of analytical grade. The accuracy of electronic scales was 0.0001 gram.

A 15 wt.% hydrochloric acid solution was pre-prepared by using dilution of commercial grade 37% HCl with distilled water in acidizing corrosion inhibition test. Corrosion solution was put into a wide-mouth bottle. In the experiment, the steel specimens were hung to immerse in the corrosion solutions through perforating its pore canal by a string.

Methods

According to the literature [19], corrosion tests in a static state for the acidizing inhibitor were carried out at the temperature of 90 degrees centigrade for 4 hours, here the N80 steel specimens were put to use. The corrosion rate (v) was determined by weight loss of tested specimens using a hanging plate method, which could be calculated from the following equation:

$$v = \frac{10^6 \times \Delta m}{S \times \Delta t} \quad (1)$$

Where Δm and S are the weight loss (g) and surface area (mm^2) of steel specimens respectively, Δt is the corrosion time (days).

The corrosion inhibition efficiency was calculated from the following equation:

$$\eta = \frac{V_0 - V}{V_0} \times 100\% \quad (2)$$

Where η is the corrosion inhibition efficiency, and v_0 and v correspond to the corrosion rates of steel specimens in the uninhibited and inhibited hydrochloric acid solutions, respectively.

Synthesis

In order to solve the insoluble problem of benzene-1,2-diamine in water, a synthetic method was put into practice as follows:

Step I: 10 grams of benzene-1,2-diamine were dissolved in 50 grams of methanol at 50°C and the process would take about 15-20 minutes, then 6 grams of commercial hydrochloric acid solution were added in the prepared solution under stirring. Solid ferric chloride hexahydrate was used to pre-prepare for 10 wt.% tap water solution so that it became more soluble.

Step II: 10 grams of benzaldehyde were added in the solution of step I under stirring. Due to the fact that the reaction between benzene-1,2-diamine and benzaldehyde is rapid, this step has to be carried out immediately in 10 minutes. Otherwise, a solidified reactant will form about 20 minutes later, which is difficult to deal with.

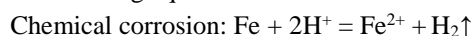
Step III: Ferric chloride hexahydrate solution prepared in Step I was quickly added in the solution of Step II under stirring. Finally, the appearance of the obtained corrosion inhibitor became dark purple.

After preliminary work, we found that methanol should be five times as heavy as benzene-1,2-diamine, and water in inhibitor solution is seven times as heavy as benzene-1,2-diamine. If the mass of methanol and water are too little, a solid insoluble production can ultimately form. The optimal mass of commercial hydrochloric acid solution should be sixty-percent of benzene-1,2-diamine.

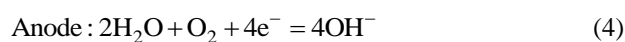
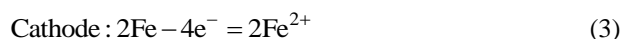
RESULTS AND DISCUSSION

Mechanism of corrosion and its inhibition

Generally, there are two main types of corrosion occurring on the surface of metal: chemical corrosion and electrochemical corrosion, which could be depicted as the following equation:



Electrochemical corrosion:



Corroding of steel surface in acid medium is considered as that iron is consumed by hydrogen ions. In the process of generating hydrogen, continuous dissolution of iron occurs on the steel surface. Surface adsorption is able to block the case. Generally, the adsorption of inhibitor molecules on MS surface is attributed to the combined action of physical adsorption and chemical adsorption [20-22]. When inhibitor molecules are adsorbed on steel surface by electrostatic attraction, which is a reversible process, desorption is liable to occur in the process. Compared to physical adsorption, chemical adsorption generate coordination bond between inhibitor molecule and iron atom on the steel surface, the intermolecular attraction in which is more intense and stable [23-24]. The iron atom has empty orbit where could be occupied by lone pair electrons supplied from sulfur, oxygen, nitrogen and phosphorus atoms in the inhibitor molecular structure [25-27]. The adsorption process may be described in Fig. 1. The combination of coordination bond indicates that the inhibitor molecules will strongly adsorb on the steel surface. The stronger the adsorption is, the higher corrosion inhibition efficiency will be. The strength of adsorption depends on the inhibitor molecular space structure and free-electron supplying-capacity [28].

Generally, efficient organic inhibitor molecules contain abundant lone pair electrons and there are electron-repelling groups next to these free electrons. When the inhibitor is introduced into an acid solution, adsorbed water molecules on the surface of the steel are replaced by the inhibitor molecules until the adsorption achieves a new equilibrium, and the adsorbed inhibitor molecules desorbing from the steel surface are not easy [29-30]. Then a firm chemical adsorption film is established for preventing steel surface from corroding. Meanwhile, synergism is an effective method for improving the inhibitive ability.

Effect of the main reactants

After preliminary work, in order to investigate the optimal composition of the inhibitor compound,

Table 1: Variables and their levels.

Factors	Levels of each variable		
	1	2	3
(A) Benzene-1,2-diamine (g)	8	10	12
(B) Benzaldehyde (g)	8	10	12
(C) FeCl ₃ ·6H ₂ O (g)	6	8	10

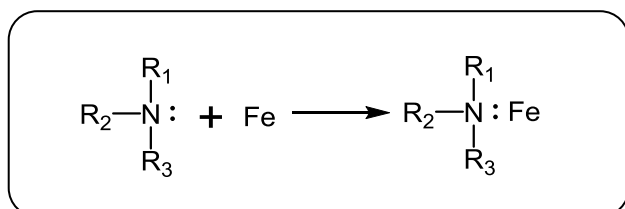


Fig. 1: Adsorption between nitrogen and iron atom, where R is hydrogen or alkyl.

an orthogonal experimental design was adopted to study the impacts of the content of main materials in the reactants on inhibition efficiency. In this study, the content of benzene-1,2-diamine, benzaldehyde and ferric chloride hexahydrate in the inhibitor reactants were regarded as the main variables while the other conditions remained unchanged in the synthesis process mentioned above. The inhibition efficiency was calculated by using the method mentioned in the industrial standard about acidification corrosion inhibitor. Table 1 shows the three variables and their levels. The data and their corresponding test results of the orthogonal experiment are listed in Table 2. As seen in Table 2, it can be found that the order of influence of each variable is A>B>C for corrosion rate, and the optimal levels for each factor is A₂B₂C₂. The results indicate that the optimal synthesis composition of the corrosion inhibitor is as follows: the dosage of benzene-1,2-diamine, benzaldehyde and ferric chloride hexahydrate is 10 g, 10 g, 8 g, respectively. The calculated mole ratio of benzene-1,2-diamine and benzaldehyde involved in the reaction is almost 1:1. Overall considering the references 1 and 2, the reaction equation may be depicted in Fig. 2.

Inhibitive performance in acid medium

According to the method of the industrial standard, inhibition efficiency of inhibitor on steel was calculated via measuring the weight loss of specimen before and after immersing in 15 wt.% HCl solution for 4 hours

at 90 °C. As seen in Fig. 3, the inhibition efficiency increases with increasing inhibitor concentration. When the dosage of inhibitor is 1.2 wt.%, the inhibition efficiency reaches to 99.24% and its plot tends to gently continue increasing the inhibitor concentration, indicating that the inhibitor exhibits a good inhibitive behavior for mild steel surface in 15 wt.% HCl solution at 90°C.

Synergistic effect

Adsorption behavior on the metal surface can influence corrosion inhibition efficiency, which depends on the molecular structure of inhibitors, surface charge density and the potential of zero charge of the metal. Some compounds introduced into the acid solution may fill in the unadsorbed blanks and enhance the adsorption of inhibitors on the metal surface, thus the corrosion extent of steel get reduced, resulting in what is the so-called synergistic effect. Some pure compounds such as ammonium bromide (AB), propargyl alcohol (PRAL) and sodium dodecyl sulfate (SDS) have a certain corrosion inhibition on steel surface [31-33]. That ammonium bromide rather than cuprous iodide or potassium iodide was used as the synergistic compound is because both of them are very expensive, the price of which is as high to \$25000 per ton, while the price of ammonium bromide is only \$3800 per ton. Considering the treatment cost, the additive mass content of synergistic compounds in 15 wt.% hydrochloric acid solution was 0.1 wt.%. Attempts of introducing some synergistic compounds were made to investigate the synergistic effect, and the corresponding data are listed in Table 3. Table 3 exhibits corrosion inhibition efficiencies of the inhibitor cooperated with the three kinds of compounds. The experimental results show that, these compounds can enhance the degree of surface coverage, resulting in increase of corrosion inhibition efficiencies. Under the same additive mass amounts, the obtained corrosion inhibition efficiencies are in the order sodium dodecyl

Table 2: Orthogonal experimental design and its test results.

NO	Level			Corrosion rate/ g/(m ² ·h)
	A (g)	B (g)	C (g)	
1	8	8	6	13.13
2	8	10	8	5.55
3	8	12	10	7.59
4	10	8	8	4.60
5	10	10	10	3.21
6	10	12	6	6.44
7	12	8	10	7.56
8	12	10	6	5.03
9	12	12	8	3.37
K ₁	26.27 ^a	25.29	24.60	
K ₂	14.25	13.79	13.52	
K ₃	15.96	17.4	18.36	
k ₁	8.76 ^b	8.43	8.20	
k ₂	4.75	4.60	4.51	
k ₃	5.32	5.80	6.12	
R	4.01 ^c	3.83	3.69	
The order	A>B>C			
Optimal level	A ₂	B ₂	C ₂	

^a $K_i = \Sigma$ the amount of corrosion rate at A_i.

^b $k_i = K_i/3$.

^c $R = \max(k_i) - \min(k_i)$.

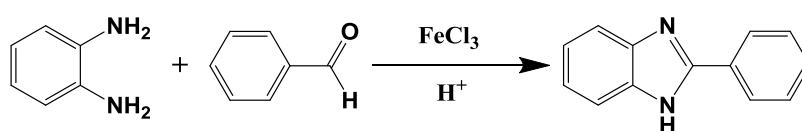


Fig. 2: Synthetic route of the inhibitor.

sulfate < propargyl alcohol < ammonium bromide. The appearance exposes that NH₄Br cooperates with the inhibitor in increasing the metal surface adsorption.

Inhibitive performance in water-injection and absorptive behavior

The inhibitor was made into a water solution with a volume fraction of 10% to observe the solubility in oilfield water-injection. Being different from N80 steel

used in acidification corrosion test, Q235 steel specimens (50mm×25mm×2mm) were used here to investigate the corrosion inhibition performance of the inhibitor in the oilfield water-injection. The composition of the water samples is seen in Table 4. According to the method mentioned in the industrial standard [34] and the practical situation on site, the weight loss of Q235 steel was calculated before and after immersing in the oilfield water-injection for 7 days at 50°C, and the corrosion

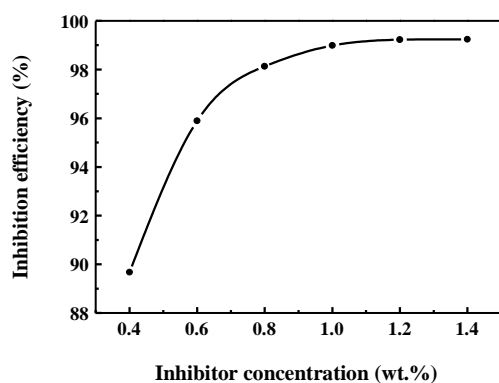
Table 3: Corrosion performance of inhibitor cooperated with three kinds of compounds, respectively.

C_{inh} (g/L)	C (AB) (g/L)	C (PRAL) (g/L)	C (SDS) (g/L)	v (g/(m ² ·h))	H %
0	0	0	0	396.98	-
9	0	0	0	7.38	98.14
9	1	0	0	4.21	98.94
9	0	1	0	4.72	98.81
9	0	0	1	5.68	98.57

Table 4: The composition of the water-injection samples.

pH	K ⁺ and Na ⁺	Mg ²⁺	Ca ²⁺	Cl ⁻	SO ₄ ²⁻	HCO ₃ ⁻	S ²⁻	Fe ³⁺	Total hardness	Mineralization degree	Total alkalinity	Suspended particles	SRB
	mg/L												ind./mL
6.76	2063	21.7	96.4	2951	52.8	731.1	25	1.42	3.3	5915	12	20	2500

where SRB is the abbreviation of Sulfate-Reducing Bacteria.

**Fig. 3: Effect of the inhibitor concentration on inhibition efficiency.**

inhibition efficiency is calculated from the following equation:

$$\eta = \frac{\Delta m_0 - \Delta m_1}{\Delta m_0} \times 100\% \quad (5)$$

Where Δm_0 and Δm_1 are the weight loss (g) of the specimens in oilfield water-injection without and with inhibitor, respectively.

The corrosion rate is calculated from the following equation:

$$r = \frac{365 \times 10 \times \Delta m}{S \times t \times \rho} \quad (6)$$

Where r is the corrosion rate (mm/year), Δm is the weight loss of test specimen (g), S is the surface area (cm²), t is the immersion time (day), ρ is the density

(g/cm³), the value 365 and 10 are the number of days in a year and unit conversion ratio of cm to mm, respectively.

The inhibition performance parameters are shown in Table 5. As seen in Table 5, the inhibition efficiency increases with increasing the inhibitor concentration. When the dosage is 30 mg/L, corrosion inhibition efficiency in oilfield water-injection is 82.4%, which meets the requirement (the required value is more than 70%) in the industrial standard.

The mechanism of corrosion inhibition may be explained as adsorption behavior. Adsorption of inhibitor on Q235 steel surface is accompanied by desorption, finally the two species achieve a balance when adsorbed inhibitor molecules form a saturated layer on the surface [35, 36]. In order to get more information about the adsorption, Freundlich and Langmuir isotherm equations were employed to fit the experimental data. It is found that the adsorption of the inhibitor on Q235 steel surface obeys the Langmuir adsorption isotherm equation:

$$\frac{C_{inh.}}{\theta} = \frac{1}{K_{ads}} + C_{inh.} \quad (7)$$

Where $C_{inh.}$ is the inhibitor concentration, K_{ads} is the adsorption equilibrium constant and θ is the surface coverage.

The surface coverage values for various concentrations of the inhibitor in oilfield water-injection are calculated in Table 5. The plot of $C_{inh.}/\theta$ against $C_{inh.}$ yields a straight line as shown in Fig. 4. The linear relationship depicted in Fig. 4 indicates that the

Table 5: Effect of inhibitor concentration on the inhibitive performance.

Dispersing performance	Inhibitor conc. (mg/L)	r (mm/year)	η (%)	θ (%)
Being homogeneously dispersed in water	Blank	0.145	-	-
	20	0.040	72.0	72.0
	25	0.034	76.5	76.5
	30	0.026	82.4	82.4
	35	0.025	83.1	83.1
	40	0.019	86.9	86.9

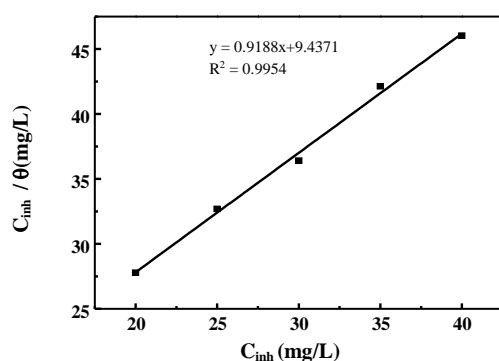


Fig. 4: Langmuir adsorption plots for steel in water-injection with different concentrations of inhibitor at 50 °C.

adsorption of inhibitor on Q235 steel surface obeys Langmuir adsorption isotherm. The linear regression coefficient is 0.9954, and the slope is 0.9188, both of them are close to 1, suggesting that the experimental results are well described by Langmuir adsorption isotherm. The adsorption-desorption equilibrium constant (K_{ads}) is determined from the C_{inh}/θ -intercept of the plot C_{inh}/θ versus C_{inh} , and the calculated value is 0.10596 L·mg⁻¹. K_{ads} is related to the standard free energy of adsorption (ΔG_{ads}^0), which can be described as the following equation:

$$\Delta G_{ads}^0 = -RT \ln(9.88 \times 10^5 K_{ads}) \quad (8)$$

Where R is the gas constant (8.314 J/mol·K), T is the absolute temperature (K), and the value 9.88×10^5 is the mass concentration of water at the experimental temperature in solution expressed in mg/L.

The calculated ΔG_{ads}^0 is -31.04 kJ/mol that is less than zero, indicating that the adsorption reaction can occur spontaneously and the inhibitor molecules can be

well adsorbed on Q235 steel surface in the oilfield water-injection at 50°C.

CONCLUSIONS

A corrosion inhibitor was rapidly synthesized from benzene-1,2-diamine, benzaldehyde, methanol, hydrochloric acid, and FeCl₃. In order to solve the insoluble problem of benzene-1,2-diamine to make the inhibitor suitable for industrial production and field application, methanol was chosen as the solvent, and hydrochloric acid was brought in the synthesis process to take part in the reaction. The orthogonal experimental results show that the optimal composition of the corrosion inhibitor is as follows: the dosage of benzene-1,2-diamine, methanol, hydrochloric acid, benzaldehyde, and 10 wt.% ferric chloride hexahydrate solution is 10 g, 50 g, 6 g, 10 g, 80 g, respectively. Under the required concentrations according to the different industrial standards of corrosion inhibition for the hydrochloric acid solution and oilfield water-injection, the inhibitor has a good inhibition performance for steel surfaces in both the two medium and the inhibition efficiency increases with increasing concentration. The synergistic test results indicate that ammonium bromide, propargyl alcohol, and sodium dodecyl sulfate can help inhibitor to enhance the corrosion inhibition efficiency, and the order of synergistic inhibition efficiencies are sodium dodecyl sulfate < propargyl alcohol < ammonium bromide. The adsorption of inhibitor on Q235 steel surface in oilfield water-injection at 50°C obeys Langmuir adsorption isotherm. It is found that the value of ΔG_{ads}^0 is negative and high (-31.04 kJ·mol⁻¹) indicating that the adsorption reaction can spontaneously occur and the inhibitor molecules can be well adsorbed on Q235 steel surface.

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