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*J. Tamilarasi, C. Joseph Kennady, K. Velmurugan***CORROSION INHIBITION OF MILD STEEL IN HYDROCHLORIC ACID BY USING NEWLY SYNTHESIZED 3-(1H-BENZO[D]IMIDAZOL-2-YL) QUINOLIN-2-THIOL****Department of Chemistry, Karunya University, Karunya Nagar, Coimbatore, Tamil Nadu, India**

Newly synthesized 3-(1H-benzo[d]imidazol-2-yl) quinolin-2-thiol derivative (BIQTH) was studied as a corrosion inhibitor by using a weight loss method and electrochemical methods. The weight losses of mild steel samples proved that BIQTH is an efficient corrosion inhibitor for mild steel in 1 M HCl at the temperature of 28°C. The corrosion rate of mild steel in 1 M HCl was decreased with increasing concentration of inhibitor. Potentiodynamic polarization studies indicated that BIQTH inhibitor displays predominantly a cathodic type of inhibition. AC impedance measurements showed that larger values of charge transfer resistance and lower values of double layer capacitance with change in the concentration of the inhibitor resulted from the adsorption of the inhibitor molecules and the formation of a protective layer on the mild steel surface. The adsorption of inhibitor obeyed Langmuir isotherm. The surface morphology of the mild steel in the presence of the inhibitor was studied by means of scanning electron microscopy.

Keywords: weight loss method, inhibitor, potentiodynamic polarization method, electrochemical impedance spectroscopy, scanning electron microscopy.

Introduction

Acid solutions are mainly used in industry for cleaning, pickling and descaling which causes the corrosion of materials under the electrochemical action of surrounding environments. Iron and its alloys are widely used for industrial purposes. But the disadvantage of mild steel is that it is easily affected by the corrosion environment [1]. The organic compounds containing such atoms as nitrogen, oxygen, sulfur and π -electrons are reported as very effective corrosion inhibitors in acidic medium because of their low toxicity and strong chemical activity [2]. The inhibition efficiency growth is in the following range of heteroatoms: O, N, S, P [3]. The inhibition mechanism of organic molecule mainly depends on the adsorption [4]. Organic molecules inhibit the corrosion by adsorption of inhibitor on metal surface thereby reducing the corrosion rate [5].

One of the best practical methods to avoid corrosion in acidic medium is by using corrosion inhibitor [6]. The corrosion process will mainly depend on the chemical structure of the inhibitor molecule and the nature, analyzing media, charge of metallic surface [7]. The inhibitor compounds containing both N and S atoms proved to show greater inhibition efficiency compared to those

compounds containing single heteroatoms [8].

In consideration of these findings, newly synthesized 3-(1H-benzo[d]imidazol-2-yl) quinolin-2-thio (BIQTH) was used as a corrosion inhibitor for mild steel in 1 M HCl medium. The structure of the synthesized compound is shown in Fig. 1. In this quinoline compound, thiol group (-SH) was attached in the second position and benzimidazole was attached in the third position. This synthesized compound was followed by our group [9]. It was investigated as a corrosion inhibitor in this work by means of weight loss method, potentiodynamic polarization method and electrochemical impedance spectroscopy.

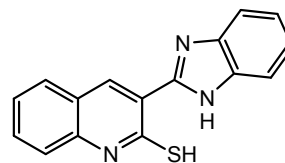


Fig. 1. Molecular structure of 3-(1H-benzo[d]imidazole-2-yl) quinoline-2-thiol

Materials and methods

The mild steel samples had the following chemical composition (wt.%): C 0.27, S 0.006, P 0.008, S 0.08, Mn 0.34, the remainder was Fe. Rectangular

form of coupons with the dimensions of 7.5 cm×2.5 cm×2 cm was taken for weight loss measurements. The surface of metal coupons was mechanically polished, cleaned with a grade of emery papers and then cleaned thoroughly with acetone, washed with twice-distilled water and finally dried at room temperature. The used acid solutions were made from A.R grade (1 M HCl) with twice-distilled water. The corrosion rate was calculated from the weight loss measurements in mm/year.

Electrochemical studies were conducted in a conventional three-electrode Pyrex-glass cell with platinum wire as a counter electrode; a saturated calomel electrode (SCE) was used as a reference electrode. Polished mild steel specimen with the surface area of 1cm² was exposed to 100 ml of 1 M HCl and used for electrochemical studies. The electrochemical measurements were carried out by using electrochemical work station CHI660C. Potentiodynamic polarization measurements were performed at a scan rate of 10 mV/s. The electrode impedance spectra were recorded in the frequency range from 0.1 kHz to 10000 MHz with AC amplitude of 5 mV relative to an open circuit potential (OCP) value in 1 M HCl. The circuit fitment for impedance graphs were made by using Z-view software. All the graphs were fitted by using origin software 8. The working electrode surface was further characterized by using scanning electron microscope JEOL-MODEL 6390.

Results and discussion

Weight loss method

The weight losses of mild steel specimen in 100 mL of HCl solution with and without various concentrations of inhibitors were determined after 3 hours of immersion period. After immersion, the mild steel specimen was washed with twice distilled water, dried at room temperature. All the experiments were carried out three times and the mean value of corrosion rate was reported. The inhibition efficiency (IE, %) was calculated by using the following equation:

$$IE = \frac{W_0 - W}{W_0}, \quad (1)$$

where W is the weight loss in the presence of inhibitor and W_0 is the weight loss in the absence of inhibitor.

The corrosion rate and inhibition efficiency in the presence and absence of inhibitor are given in Table 1. The corrosion rate of mild steel was decreased with increasing the concentration of inhibitor up to 5–25 ppm (Fig. 2)

Table 1

Variation of corrosion rate, inhibition efficiency and surface coverage of mild steel at different concentrations of BIQTH in 1 M HCl determined by weight loss method at 28°C for 3 hours

Concentration of BIQTH, ppm	Corrosion rate, mm/y	Inhibition efficiency, %	Surface coverage, C/θ
Blank	0.73	–	–
5	0.24	67.12	7.44
15	0.15	79.45	18.87
25	0.09	88	28.40

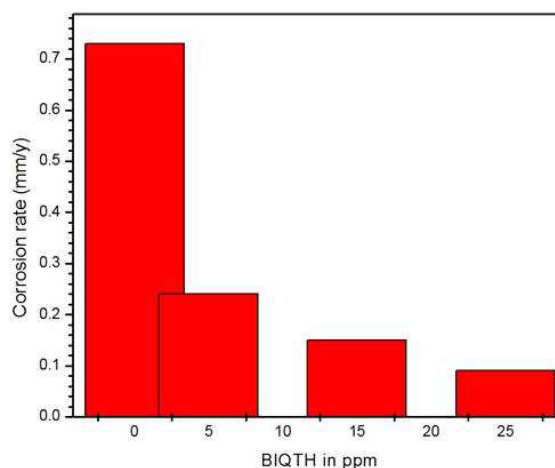


Fig. 2. Variation of corrosion rate versus concentrations of BIQTH at 28°C

Moreover, protection efficiency increased (88%) with increasing the concentration of BIQTH up to 25 ppm (Fig. 3).

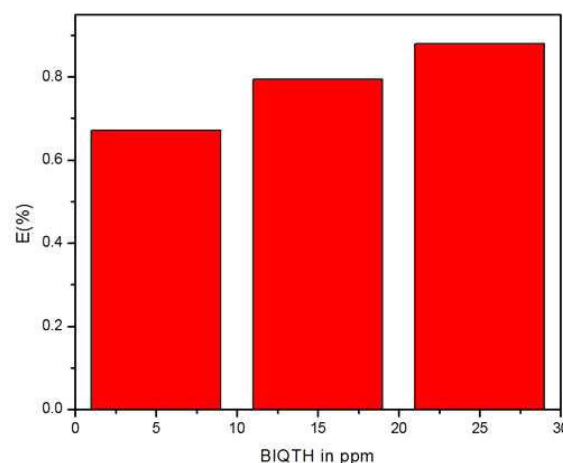


Fig. 3. Variation of inhibition efficiency versus concentrations of BIQTH at 28°C

A higher inhibition efficiency of BIQTH indicates strong bond formation between steel and acid solution. The adsorption of inhibitor on the surface of mild steel is mainly due to the heteroatoms, such as sulfur and nitrogen, benzo-imidazole ring, high electron density of the molecule and also planarity of the molecule. It is known that the compounds containing nitrogen and sulfur in the molecule adsorb stronger than those containing only nitrogen or sulfur, thereby decreasing the corrosion rate of mild steel in 1 M HCl [10].

Potentiodynamic measurements

The potentiodynamic polarization technique was carried out by gain the knowledge of kinetics of the cathodic and anodic reactions. Figure 4 shows potentiodynamic polarization curves obtained in 1 M HCl at the temperature of 28°C in the presence and absence of the studied inhibitor at different concentrations, the working electrode being pre-polarized to attain steady-state for 30 minutes. After that, the electrodes were subjected to the measure of the corrosion potential (E_{corr}), corrosion current (I_{corr}), anodic Tafel slopes (b_a), cathodic Tafel slopes (b_c) and polarization resistance (R_p). The values of inhibition efficiency (E_i , %) was calculated by using the following formula:

$$E_i = \frac{\text{LPR} - \text{LPR}^0}{\text{LPR}^0}, \quad (2)$$

where LPR is the linear polarization resistance with inhibition and LPR^0 is the linear polarization resistance without inhibition.

Table 2 shows the potentiodynamic polarization parameters of mild steel in 1 M HCl at 28°C in the presence and absence of the inhibitor under study. The addition of BIQTH to the acid solution realists in slight shifting the anodic polarization values (b_a), whereas shifting the cathodic polarization values (b_c) is more pronounced. The current density values were decreased linearly with increasing the polarization resistance compared to blank polarization resistance value ($49.1 \Omega \times \text{cm}^2$ and $345 \Omega \times \text{cm}^2$, respectively). Thus, the cathodic reaction kinetics dominated. The maximum inhibition efficiency of BIQTH in 1 M

HCl increased to 86%. This higher inhibition efficiency proves that the inhibition of BIQTH occurs by the formation of chelate compounds with Fe. An organic compound containing C–S bond undergoes oxidative addition at the iron surface interface [11]. Hence, BIQTH was adsorbed on metal surface through hydrogen evolution reaction with anodic dissolution of metal. The above results show that BIQTH acts as good cathodic inhibitor.

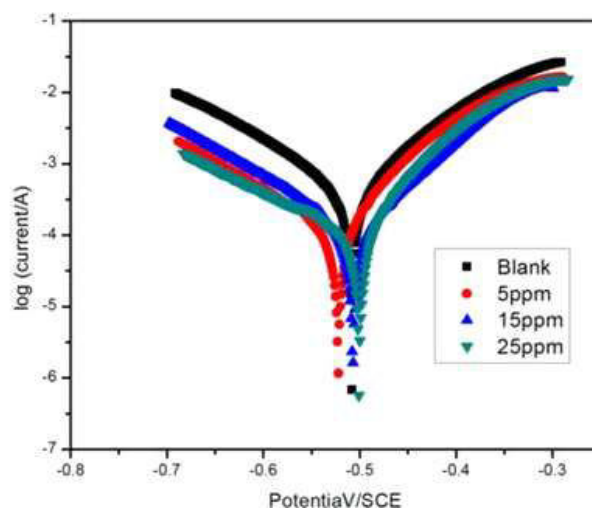


Fig. 4. Tafel polarization curves for mild steel obtained in 1 M HCl solution containing various concentration of BIQTH at 28°C

Electrochemical impedance measurement

The corrosion behavior of mild steel in 1 M HCl solution for different concentrations of BIQTH was investigated at the temperature of 28°C. Table 2 shows the potentiodynamic polarization parameters of mild steel in 1 M HCl at 28°C in the presence and absence of the inhibitor under study. The addition of BIQTH to the acid solution realists in slight shifting the anodic polarization values (b_a), whereas shifting the cathodic polarization values (b_c) is more pronounced. The current density values were decreased linearly with increasing the polarization resistance compared to blank polarization resistance value ($49.1 \Omega \times \text{cm}^2$ and $345 \Omega \times \text{cm}^2$, respectively).

Table 2

Tafel plots parameters for mild steel in 1 M HCl at different concentrations of BIQTH at the temperature of 28°C

Concentration of BIQTH (ppm)	E_{corr} (mV vs. SCE)	$I_{\text{corr}} \times 10^4$ ($\mu\text{A cm}^{-2}$)	b_a (mV dec ⁻¹)	$-b_c$ (mV dec ⁻¹)	R_p ($\Omega \times \text{cm}^2$)	E_i (%)
Blank	-512	4.807	0.0998	0.1192	49.1	–
5	-522	1.307	0.0868	0.1282	172.3	71.50
15	-501	1.156	0.0820	0.1586	203.4	76.00
25	-448	0.583	0.05652	0.2525	345	85.76

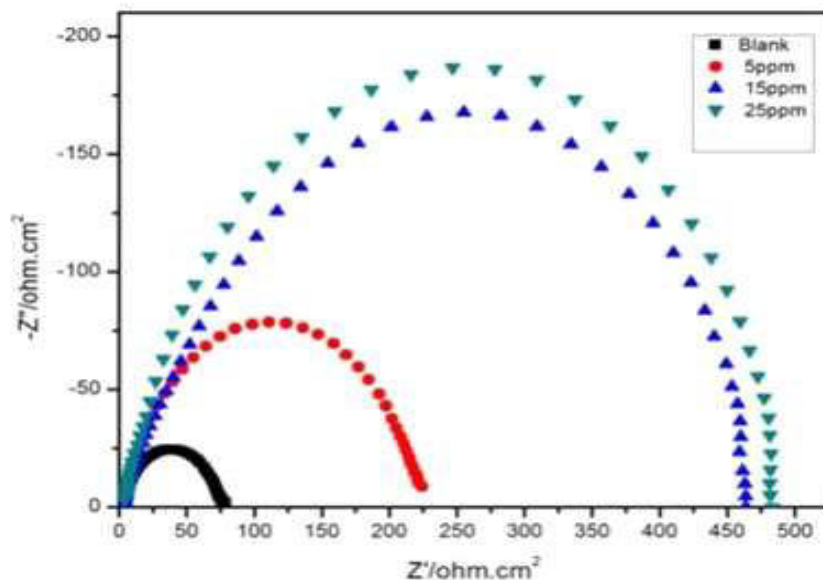


Fig. 5. Nyquist plots for mild steel obtained in 1 M HCl solution containing various concentration of BIQTH at 28°C

Thus, the cathodic reaction kinetics dominated. The maximum inhibition efficiency of BIQTH in 1 M HCl increased to 86%. This higher inhibition efficiency proves that the inhibition of BIQTH occurs by the formation of chelate compounds with Fe. An organic compound containing C–S bond undergoes oxidative addition at the iron surface interface [11]. Hence, BIQTH was adsorbed on metal surface through hydrogen evolution reaction with anodic dissolution of metal. The above results show that BIQTH acts as good cathodic inhibitor. C by using electrochemical impedance spectroscopy. The charge transfer resistance was calculated from the diameter of the semi-circle in Nyquist plots. The charge transfer resistance was obtained by the following formula

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}}, \quad (3)$$

where C_{dl} is the capacitance double layer, R_{ct} is the charge transfer resistance and f_{max} is the Z'' maximum frequency.

The value of inhibition efficiency (E , %) was calculated by the following relationship:

$$IE(\%) = \frac{R_{inh} - R_t}{R_t}, \quad (4)$$

where R_{inh} is the charge transfer resistance in the presence of inhibitor and R_t is the charge transfer resistance in the absence of inhibitor.

Figure 5 shows that the impedance responses of inhibited and uninhibited systems. The Nyquist

plots contain depressed semi-circles whose size increased with increasing the concentration of inhibitor from 5 ppm to 25 ppm, and consequently the inhibition efficiency increases. The best inhibitory effect was observed at 25 ppm. This means that the charge transfer reaction is mainly controlled by the corrosion of metal and indicates roughness and other inhomogeneities of the metal surface [12].

The results of the electrochemical impedance spectroscopy were interpreted in terms of the equivalent circuit model given in Figure 6.

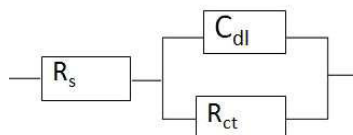


Fig. 6. Equivalent circuit model used to fit the impedance data

As can be seen from Table 3, the charge transfer resistance value (R_t) for blank 1 M HCl was 76 Ωcm^2 ; it increased by the addition of BIQTH (5–25 ppm). The maximum charge transfer resistance for BIQTH was 478 Ωcm^2 . This increasing charge transfer resistance by the addition of BIQTH proved the formation of a barrier film on the metal-solution interface surface with decreasing the capacitance double layer (C_{dl}) values which led to increasing the efficiency of the inhibitor.

Here the gradual removal of water molecules by the adsorption of organic molecules occurs on the metal surface with decreasing the extent of metal dissolution, it leads to decreasing the capacitance double layer [13]. These observations clearly explained the corrosion of mild steel in 1 M HCl,

which is mainly controlled by the adsorption of substituted compounds and heteroatoms on the mild steel surface. The results obtained from the weight loss method and polarization method also proved that BIQTH acts as a good corrosion inhibitor in an acid medium.

Table 3
Nyquist plots parameters for mild steel obtained in 1 M HCl solution containing various concentration of BIQTH at 28°C

Concentration of BIQTH (ppm)	R_{ct} ($\Omega \times \text{cm}^2$)	$C_{dl} \cdot 10^7$ ($\mu\text{F} \times \text{cm}^{-2}$)	IE (%)
Blank	76	2.524	–
5	243.992	0.7862	68.85
15	461.427	0.4157	83.52
25	477.633	0.4016	84.08

Adsorption isotherm

Adsorption plays an important role in inhibition mechanism. Experimentally obtained data were tested with various types of adsorption isotherms. The adsorption of an organic adsorbate at the metal solution interface can be explained by the competitive adsorption between the organic molecules in the aqueous solution and the water molecules on metal surface [14].

The Langmuir adsorption isotherm for monolayer adsorption is described by the following equation:

$$\frac{\theta}{1-\theta} = KC_{\text{inh}} \quad (5)$$

On rearranging the equation (5) we get the following equation:

$$\frac{C_{\text{inh}}}{\theta} = \frac{1}{K} + C_{\text{inh}} \quad (6)$$

Here θ is the surface coverage, C_{inh} is the concentration of inhibitor in the aqueous solution, and K is the equilibrium constant of the adsorption process.

The value of surface coverage θ can be obtained from the weight loss method for different concentrations of inhibitor (see above). The values of surface coverage were fitted by using various isotherms (Langmuir, Frumkin and Temkin); the isotherms are not shown in the paper. The best fit was obtained with the Langmuir isotherm. By plotting the graph C_{inh}/θ against C_{inh} the straight line was obtained according Eq. (6). The correlations

coefficient (R^2), was 0.99454 for the Langmuir plot. Thus, the adsorption of the studied inhibitor on the surface of mild steel in 1 M HCl at 28°C obeyed Langmuir isotherm.

Scanning electron spectroscopy

The SEM micrographs for uninhibited and inhibited mild steel are shown in the Fig. 7. The metal exposed to 1 M HCl solution resulted in the formation of pits (Fig. 7, left). The metal exposed to 1 M HCl solution with the addition of 25 ppm BIQTH resulted in the disappearance of pits (Fig. 7, right). This was due to the formation of protective layer on metal surface in an inhibited acidic medium [15]. It can be concluded from above results that the corrosion does not occur in the presence of the proposed inhibitor.

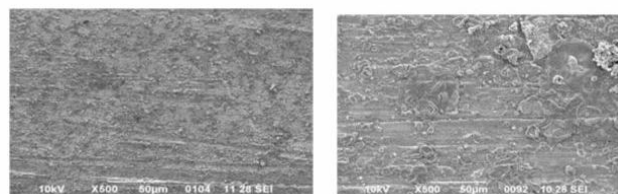


Fig. 7. SEM images of the surface of mild steel in 1 M HCl solution without (left) and with 25 ppm BIQTH (right)

Conclusions

According to the results of weight loss method, the corrosion rate decreased with increasing the concentration of inhibitor which led to increasing the inhibitor efficiency up to 88% at the concentration of 25 ppm.

BIQTH retards the anodic reaction to a large extent as compared with the cathodic reaction. From these observations, we concluded that BIQTH behaves as a cathodic inhibitor in the acidic solution.

As follows from the results of electrochemical impedance spectroscopy, the charge transfer resistance increased with increasing the concentration of the inhibitor, which confirms that BIQTH is a good inhibitor in 1 M HCl medium.

A good agreement was observed between the values obtained by weight loss method, potentiodynamic polarization method and electrochemical impedance spectroscopy.

Adsorption of BIQTH on the surface of mild steel followed Langmuir isotherm.

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ІНГІБУВАННЯ КОРОЗІЇ МАЛОВУГЛЕЦЕВОЇ СТАЛІ У ХЛОРОВОДНЕВІЙ КИСЛОТІ З ВИКОРИСТАННЯМ НОВОГО СИНТЕЗОВАНОГО 3-(1Н-БЕНЗО[Д]ІМІДАЗОЛ-2-ІЛ) ХІНОЛІН-2-ІОЛУ

Дж. Таміларасі, К. Джозеф Кеннаді, К. Вельмуруган

Нове синтезоване похідне 3-(1Н-бензо[д]імідазол-2-іл) хінолін-2-тіолу (VIQTH) було досліджено у ролі інгібітора корозії з використанням гравіметричного методу та електрохімічних методів. Гравіметричні вимірювання швидкості корозії зразків маловуглецевої сталі довели, що VIQTH є ефективним інгібітором корозії у розчині 1 М НСІ при температурі 28°C. Швидкість корозії маловуглецевої сталі в 1 М НСІ знижувалася при підвищенні концентрації інгібітора. Дослідження методом лінійної вольтамперометрії показали, що VIQTH є переважно інгібітором катодного типу. Зміннострумові імпедансні вимірювання показали, що більші значення опору перенесення заряду та менші значення ємності подвійного електричного шару при змінній концентрації інгібітора є результатом адсорбції органічних молекул і формування захисного шару на поверхні маловуглецевої сталі. Адсорбція описується рівнянням ізотерми Ленгмюра. Поверхнева морфологія маловуглецевої сталі у присутності інгібітора була вивчена методом сканувальної електронної мікроскопії.

Ключові слова: гравіметричний метод; інгібітор; метод лінійної вольтамперометрії; спектроскопія електродного імпедансу; сканувальна електронна мікроскопія.

CORROSION INHIBITION OF MILD STEEL IN HYDROCHLORIC ACID BY USING NEWLY SYNTHESIZED 3-(1H-BENZO[D]IMIDAZOL-2-YL) QUINOLIN-2-THIOL**J. Tamilarasi, C. Joseph Kennady, K. Velmurugan****Karunya University, Karunya Nagar, Coimbatore, Tamil Nadu, India**

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