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Investigation of 4-Amino-3-Hydrazino-5-Mercapto-1,2,4-Triazole as Corrosion Inhibitor for C38 Steel in Hydrochloric Acid Medium

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ABSTRACT

The inhibition effect of 4-Amino-3-hydrazino-5-mercapto-1,2,4-triazole (AHMT) against the corrosion of C38 steel in 1 M HCl solution was studied by weight loss, electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization. Inhibition efficiency increased with inhibitor concentration and it attains an efficiency of more than 87% at 10^{-2} M at 308K. Polarization curves show that AHMT is a mix-type inhibitor. As the inhibitor concentration increased, the charge transfer resistance of C38 steel increased and double layer capacitance decreased. The adsorption of AHMT molecules on the C38 steel surface obeys Langmuir adsorption isotherm. The adsorption free energy of AHMT on C38 steel (-34.88 kJ / mol) involves two types of interaction, chemisorption and physisorption of the inhibition on the metal surface. The results obtained from the different methods are in good agreement.

Keywords: Steel, Hydrochloric acid, Triazole, Polarisation curves, Impedance, Langmuir.
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1. INTRODUCTION

Hydrochloric acid solution is widely used as acid wash solution for removing rust and scale from the steel surface. However, the strong corrosivity of hydrochloric acid needs to be controlled by an appropriate corrosion inhibitor. The use of inhibitors is one of the most practical methods for protecting materials against corrosion, especially in acidic media [1]. Most well-known acid inhibitors are organic compounds containing nitrogen, sulfur, and oxygen atoms. Among them, N-heterocyclic compounds are considered to be the most effective corrosion inhibitors [2]. Up to now, various N-heterocyclic compounds are reported as good

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♠Correspondingauthor, e-mail: azarrouk@gmail.com corrosion inhibitors for iron or steel in acidic media, such as imidazoline derivatives [3–5], 1,2,3-triazole derivatives [6], 1,2,4-triazole derivatives [2,7–10], benzotriazole [11–14], bipyrazole derivatives [15–17]. Influence of some mercapto functional compounds including mercapto triazole derivatives [18], 2-aminophenyl-5-mercapto- 1-oxa-3,4-diazole [19], 2-amino-5 mercapto-1,3,4-thiadiazole [20], 5-amino-3-mercapto-1,2,4-triazole [21], 4-salicylideneamino- 3-phenyl-5-mercapto-1,2,4-triazole [22], 6-methyl-5-[m-nitro styryl]-3- mercapto-1,2,4-triazine [23], some mercapto oxadiazole derivatives [24] on corrosion of steel in hydrochloric acid solution have been studied with weight loss, polarization and EIS.

In present study, 4-Amino-3-hydrazino-5-mercapto-1,2,4-triazole (AHMT) was investigated as an inhibitor for the corrosion of C38 steel in 1M hydrochloric acid (HCl) using mass loss, potentiodynamic polarization and Electrochemical impendence spectroscopy (EIS). The molecular structure is shown as follows:

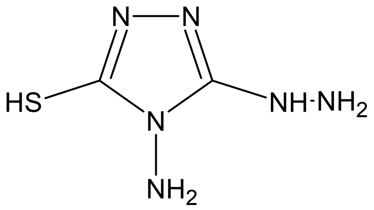

Figure 1. The molecular structure of AHMT.

2. EXPERIMENTAL DETAILS

2.1. Materials and Reagents

C38 Steel strips containing (0.09 % P; 0.38 % Si; 0.01 % Al; 0.05 % Mn; 0.21 % C; 0.05 % S and the remainder iron) were used for electrochemical and gravimetric studies. Prior to all measurements, are abraded with a series of emery paper from 180 to 1200 grade. The specimens are washed thoroughly with bidistilled water degreased and dried with acetone. The aggressive solution (1M HCl) was prepared by dilution of Analytical Grade 37 % HCl with double-distilled water. 4-Amino-3-hydrazino-5-mercapto-1,2,4-triazole (AHMT) is analytical grade.

2.2. Measurements

2.2.1. Weight loss measurements

Gravimetric measurements are carried out in a double walled glass cell equipped with a thermostated cooling condenser. The solution volume is 50 cm^3. The steel specimens used have a rectangular form (1.6cm × 1.6cm × 0.07cm). The immersion time for the mass loss is 6h at 308K.

2.2.2. Electrochemical measurements

- *Electrochemical cell*

The electrolysis cell was Pyrex of cylinder closed by cap containing five openings. Three of them were used for the electrodes. The working electrode was C38 steel with the surface area of 1 cm^2. Before each experiment, the electrode was polished using emery paper until 1200 grade. After this, the electrode was cleaned ultrasonically with distillate water. A saturated calomel electrode (SCE) was used as a reference. All potentials were given with reference to this electrode. The counter electrode was a platinum plate of surface area of 1 cm^2. The aggressive medium used here is 1M HCl solution was prepared with concentrated HCl and distilled water. The organic compound tested is triazole derivative. Its molecule formula is shown in Fig. 1. The concentration range of this compound was 1×10^{-2} to 1×10^{-5} M.

- *Polarization measurements*

The working electrode was immersed in test solution during 30 minutes until a steady state open circuit potential (E_{ocp}) was obtained. The polarization curve was recorded by polarization from -800 mV to -200 mV under potentiodynamic conditions corresponding to 1 mV/s (sweep rate) and under air atmosphere, at frequencies between 100 kHz and 10 mHz was superimposed on the rest potential. The potentiodynamic measurements were carried out using Tacussel Radiometer PGZ 100, which was controlled by a personal computer.

3. RESULTS AND DISCUSSION

3.1. Gravimetric Measurements

Three parallel rectangular C38 steel specimens of were used for the determination of corrosion rate. The coupons, initial mass using an analytic balance was recorded before immersion in 80 ml open beakers containing 50 mL of corrodent (1M HCl) without and with different concentrations of AHMT. The specimens were taken out, washed, dried, and reweighed accurately. The average mass loss of the three parallel C38 steel specimens could be obtained. The corrosion rates of have be C38 steel en determined for 6h immersion period at 308K from mass loss, using Eq. (1) where Δm is the mass loss, S is the area, and t is the immersion period [25]. The percentage protection efficiency E_w(%) was calculated according the relationship Eq. (2) where W and W_{inh} are the corrosion rates of steel without and with the inhibitor, respectively [26]:

$$W = \frac{\Delta m}{S.t} \quad (1)$$

$$E_W\% = \frac{W - W_{inh}}{W} \quad (2)$$

The value of percentage inhibition efficiency (%IE) and corrosion rate (CR) obtained from mass loss method at different concentrations of AHMT in 1M HCl at 308K are summarized in Table 1. It is evident from the Table 1 that the corrosion rate decreases and inhibition efficiency increases with increase in AHMT concentration.

Table 1. Corrosion parameters for steel in aqueous solution of 1M HCl in absence and presence of different concentrations of AHMT from mass loss measurements at 308K for 6h.

Inhibitor	Conc (M)	W(mg/cm^2.h)	E_w (%)	Θ
Blank	1	1.070±0.005	-	-
AHMT	1×10^{-5}	0.863±0.017	19.4±1.9	0.194
	1×10^{-4}	0.331±0.017	69.1±1.7	0.691
	5×10^{-4}	0.254±0.016	76.3±1.6	0.763
	1×10^{-3}	0.183±0.016	82.9±1.6	0.829
	5×10^{-3}	0.158±0.014	85.2±1.4	0.852
	1×10^{-2}	0.134±0.008	87.5±0.8	0.875

The variations in the inhibition efficiency and corrosion rate with AHMT concentration shown in Fig 2 suggest that AHMT inhibits steel at all the concentration range used in the study. Maximum inhibition efficiency was reported at 1×10^{-2}M concentration of triazole.

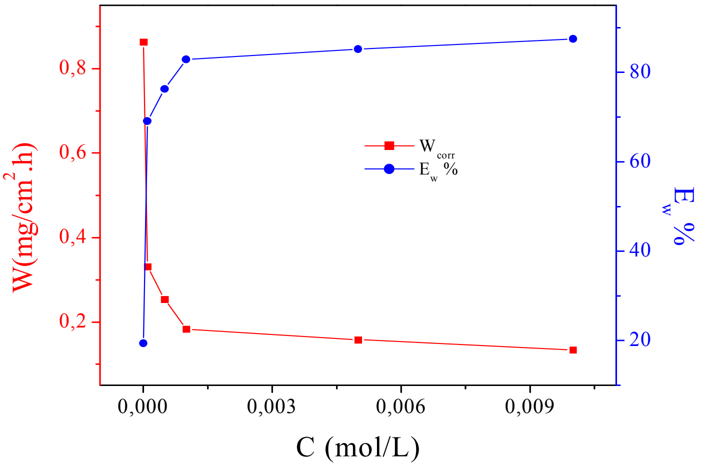

Figure 2. Variation of inhibition efficiency and corrosion rate in 1M HCl on C38 steel surface without and with different concentrations of triazole derivative

3.2. Potentiodynamic Polarization Studies

Fig. 3 shows polarization curves for C38 steel in 1M HCl without and with different concentrations of triazole derivative. The anodic and cathodic current–potential curves were extrapolated up to their interaction point where corrosion current density (I_{corr}) and corrosion potential (E_{corr}) are obtained [27]. The electrochemical parameters such as corrosion current density (I_{corr}), corrosion potential (E_{corr}), cathodic Tafel slopes (β_c) and inhibition efficiency obtained from polarization measurements are listed in Table 2. In the case of polarization method the relation determines the inhibition efficiency (E_I%):

$$E_I\% = (1 - \frac{I_{corr}}{I^{\circ}_{corr}}) \times 100 \qquad (3)$$

Where $I°_{corr}$ and I_{corr} are the uninhibited and inhibited corrosion current densities, respectively, determined by extrapolation of cathodic Tafel lines to corrosion potential.

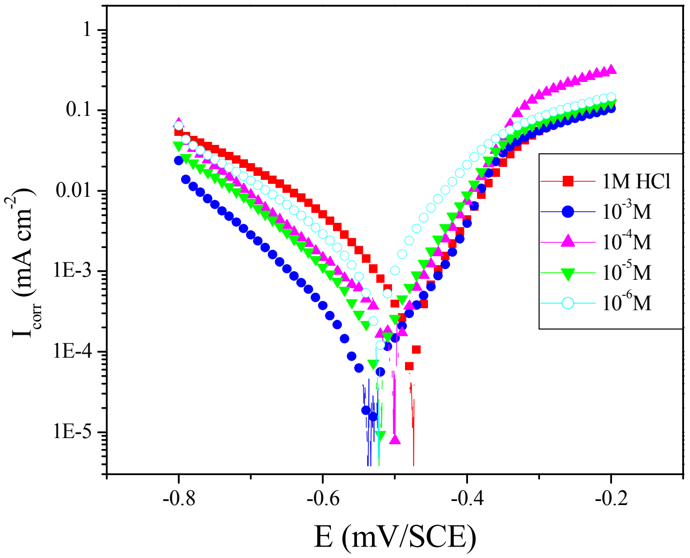

Figure 3. Polarisation plots of steel in 1M HCl for various concentrations of AHMT.

Table 2. Potentiodynamic polarization parameters for the corrosion of steel in 1M HCl without and with different concentrations of AHMT.

Inhibitor	Conc (M)	$-E_{corr}$ (mV/SCE)	$-\beta_c$ (mV/dec)	I_{corr} ($\mu A/cm^2$)	E_I (%)
HCl	1	475.9	175.6	1077.8	-
AHMT	1×10^{-5}	523.4	184.0	910.5	15.52
	1×10^{-4}	520.8	138.2	360.2	66.58
	1×10^{-3}	500.6	129.4	255.7	76.27
	1×10^{-2}	535.8	129.2	151.9	**85.90**

It is clear from the results that the addition of inhibitor causes a decrease of the current density. The values I_{corr} of steel in the inhibited solution are smaller than those for the inhibitor free solution (Table 2). The parallel cathodic Tafel plots obtained in Fig. 3 indicate that the hydrogen evolution is activation-controlled and the reduction mechanism is not affected by the presence of inhibitor. The change in cathodic Tafel slope (β_c) values (Table 2), suggest that the reaction mechanism of the hydrogen reduction is not the same in the absence and presence of inhibitor. The anodic effect is well shown when curves are compared at $E-E_{corr}$. The inhibiting action is increased with the concentration to reach the maximum value of 85.90% at the 10^{-2} M. The addition of AHMT shifted the E_{corr} value towards the negative direction. This indicates that the AHMT compound acts as a mixed type inhibitor. The Tafel polarization method confirmed the main weight loss results, giving some additional information.

3.3. Electrochemical Impedance Spectroscopy (EIS) Studies

Nyquist plots of triazole compound in 1 M HCl solutions in the absence and presence of various concentrations of AHMT are given in Fig.4. The impedance spectra show that a single semicircle and the diameter of semicircle increases with increasing inhibitor concentration. These diagrams exhibit that the impedance spectra consist of one capacitive loop at high frequency, the high frequency capacitive loop was attributed to charge transfer of the corrosion process [28]. Various parameters such as charge-transfer resistance (R_{ct}), double layer capacitance (C_{dl}), f_{max} and inhibition efficiency were obtained from impedance measurements and are shown in Table 3. R_{ct} values were calculated from the difference in impedance at lower and higher frequencies as suggested by Tsuru et al. [29]. C_{dl} values were calculated from the frequency at which the imaginary component of impedance was maximum (Z_{im} max) using the reaction:

$$C_{dl} = \frac{1}{2\pi f_{\max} R_{ct}} \tag{4}$$

Where $f_{\max}$ is the frequency at which the imaginary component of impedance is maximum. The inhibition efficiency got from the charge-transfer resistance is calculated by the following relation:

$$E_{R_{ct}}\% = \frac{R_{ct} - R^{\circ}_{ct}}{R_{ct}} \tag{5}$$

Where R_{ct} and R°_{ct} represent the resistance of charge transfer in the presence and absence of inhibitor, respectively. R_{ct} is the diameter of the loop. The R_{ct} values of this investigated triazole derivative increase with increasing inhibitor concentration.

Table 3. Corrosion parameters obtained by impedance measurements for steel in 1M HCl at various concentrations of AHMT.

Inhibitor	Conc (M)	R_s (Ω cm^2)	R_{ct} (Ω cm^2)	$f_{\max}$ (Hz)	C_{dl} (μF/cm^2)	$E_{R_{ct}}$ (%)
HCl	1	1.61	31.04	63.34	80.99	-
AHMT	1×10^{-5}	1.72	35.17	63.29	71.74	13.31
	1×10^{-4}	1.79	91.33	31.60	55.19	66.01
	1×10^{-3}	1.47	115.30	25.00	55.24	73.08
	1×10^{-2}	1.88	132.10	25.00	48.22	**76.50**

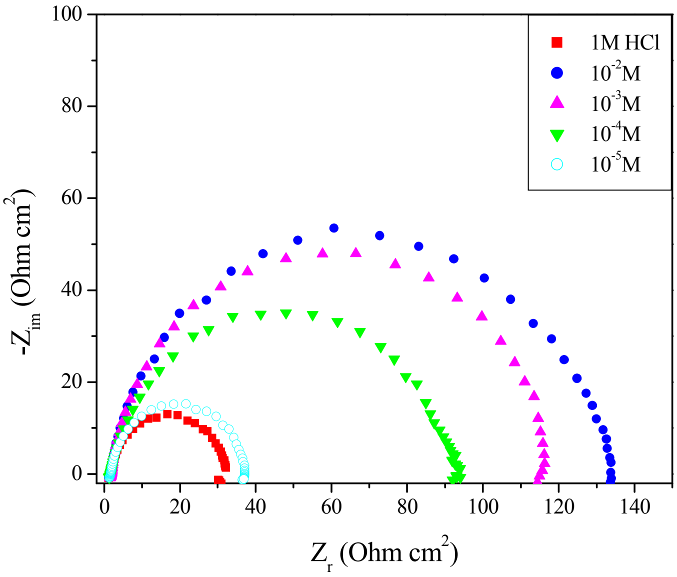

Figure 4. Nyquist diagrams for C38 steel in 1M HCl containing different concentrations of AHMT at 308K.

EIS spectra were analyzed using the equivalent circuit in Fig. 5, which was used previously to model the iron/acid interface [30]. The impedance parameters derived from these figures are given in Table 3. R_s is the solution resistance, R_{ct} is the charge transfer resistance, C_{dl} is the double layer capacitance.

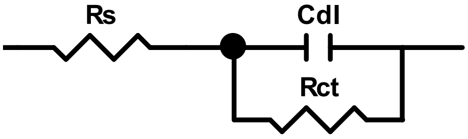

Figure 5. Equivalent circuit model for system C38 steel/1 M HCl/inhibitor.

Table 3 shows that R_{ct} values increased and C_{dl} decreased with concentration. The decrease in C_{dl} values indicating that the inhibitors function by adsorption at the metal/solution interface by gradual displacement of water molecules originally and/or chloride ions on the C38 steel surface [31] leading to a protective solid film, inhibiting species or both on the C38 steel surface, then decreasing the extend of dissolution reaction [32, 33]. This decrease of C_{dl} concentration can be explained by the decrease in local dielectric constant and/or an increase in the protective layer thickness of electrode surface. In addition, the inhibition efficiencies, got from electrochemical impedance measurements, increase with concentration and show the same trend as those obtained from polarization measurements.

3.4. Adsorption Isotherm and Thermodynamic Parameters

The adsorption on the corroding surfaces never reaches the real equilibrium and tends to reach an adsorption steady state. However, when the corrosion rate is sufficiently small, the adsorption steady state has a tendency to become a quasi-equilibrium state. In this case, it is reasonable to consider the quasi-equilibrium adsorption in thermodynamic way using the appropriate equilibrium isotherms [34]. The efficiency of this triazole as a successful corrosion inhibitor mainly depends on its adsorption ability on the metal surface. So, it is essential to know the mode of adsorption and the adsorption isotherm that can give valuable information on the interaction of inhibitor and metal surface. The surface coverage values, Θ ($\Theta = IE/100$) for different concentrations of AHMT was used to explain the best adsorption isotherm. A plot of C/Θ versus C (Fig. 6) gives a straight line with an average correlation coefficient of 0.99993 and a slope of nearly unity (1.14) suggests that the adsorption of triazole derivative molecules obeys Langmuir adsorption isotherm, which can be expressed by the following equation:

$$\frac{C_{inh}}{\Theta} = \frac{1}{K_{ads}} + C_{inh} \qquad (6)$$

Where C_{inh} is inhibitor concentration and K_{ads} is the equilibrium constant for the adsorption–desorption process.

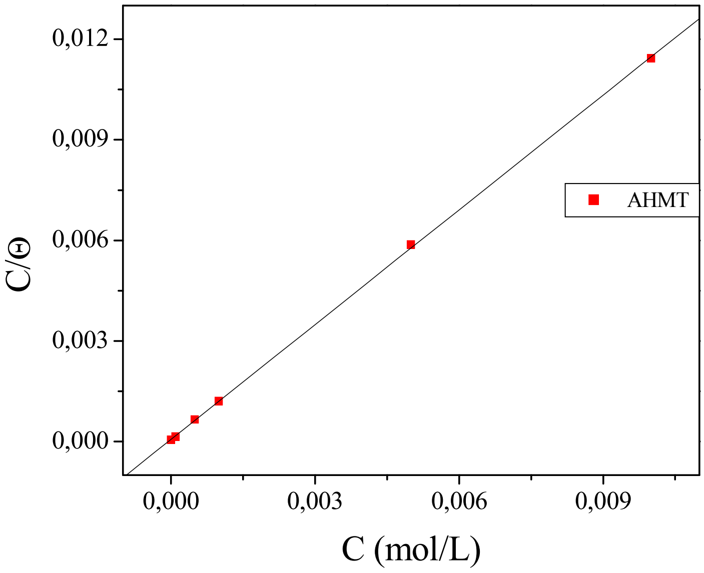

Figure 6 . Langmuir isotherm for the adsorption of AHMT on the surface of steel in 1M HCl.

The thermodynamic parameters from the Langmuir adsorption isotherm are listed in Table 4, together with the value of the Gibbs free energy of adsorption ΔG°_{ads} calculated from the equation:

$$\Delta G^{\circ}_{ads} = -RTLn(55.5K_{ads}) \qquad (7)$$

Where R is the universal gas constant, T the thermodynamic temperature and the value of 55.5 is the concentration of water in the solution [35].

The value K_{ads} calculated from the reciprocal of intercept of isotherm line as $1.48498 \times 10^4\ M^{-1}$. The high value of the adsorption equilibrium constant reflects the high adsorption ability of this inhibitor on C38 steel surface.

Table 4. Thermodynamic parameters for the adsorption of AHMT in 1M HCl on the C38 steel at 308K.

Inhibitor	Slope	K_{ads} (M^{-1})	R^2	ΔG°_{ads} (KJ/mol)
AHMT	1.14	14849.79	0.99993	-34.882

From Eq. (7), the ΔG°_{ads} was calculated as -34.882 kJ mol^{-1}. The negative value of standard free energy of adsorption indicates spontaneous adsorption of triazole molecules on C38 steel surface and also the strong interaction between inhibitor molecules and the metal surface [36, 37]. Generally, the standard free energy values of -20 kJ mol^{-1} or less negative are associated with an electrostatic interaction between charged molecules and charged metal surface (physical adsorption); those of -40 kJ mol^{-1} or more negative involves charge sharing or transfer from the inhibitor molecules to the metal surface to form a co-ordinate covalent bond (chemical adsorption) [38, 39]. The value of ΔG°_{ads} in our measurement is -34.882 kJ mol^{-1} (in Table 4), it is suggested that the adsorption of this triazole derivative involves two types of interaction, chemisorption and physisorption [40].

CONCLUSION

- Inhibition efficiencies increase with increasing inhibitor concentration.
- Adsorption mechanism of the AHMT on C38 steel in a 1M shydrochloric acid solution at 308K was a combination of both physisorption and chemisorption.
- Polarization curves indicated that the AHMT behaves mainly as mixed-type inhibitor. Impedance method indicates that AHMT adsorbs on the C38 steel surface with increasing transfer resistance and decreasing of the double-layer capacitance.
- The inhibition efficiencies determined by mass loss, potentiodynamic polarization and EIS techniques are in reasonably good agreement.

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