Study of the Corrosion Inhibitive Effect and Adsorption Process of Two Azo-Aldehydes on Carbon Steel in 1 M H₂SO₄

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Abstract. Two aromatic aldehydes with azo linkage (AS1 and AS2) were assessed for their inhibitive effect on carbon steel corrosion in 1 M H₂SO₄. The inhibition efficiency was studied by gravimetric and electrochemical analysis. The effects of concentration and temperature on the inhibition efficiency have also been studied. The obtained results showed that both of the used compounds can be considered as efficient carbon steel corrosion inhibitors. The compound which has methyl group in its structure (AS2) has higher inhibition efficiency (93.11% with 0.2 mM at 303 K by electrochemical analysis) than the one with cyano group (AS1). The adsorption isotherms of the inhibitors were studied; it was found that the adsorption of the two compounds obeys Langmuir isotherm. The studied inhibitors are mixed type inhibitors and adsorbed onto the surface of the carbon steel by a physisorption process. SEM and AFM techniques were used to study the effect of the aggressive solution on the morphology of the metal surface in the absence and presence of the used inhibitors.

1. Introduction:
Corrosion is one of the serious problems that the industry faces. It is defined as the destructive attack of the environment on the metals or alloys causing serious damages to the metallic parts of the equipment, especially ones made from steel, therefore, the prevention of this phenomenon became an interesting research area [1,2]. Acids are widely used in industry for different purposes such as instance oil-well acidizing, acid pickling and cleaning [3-6]. The necessity of the use of acids to treat metals makes the development materials that have the ability to reduce the corrosion requisite. Such materials are called corrosion inhibitors, among them, organic compounds that contain delocalized π electrons, hetero atoms like N, O, S and P either in the side chain or in the ring and polar functional groups such as (–NO₂, –SH, –
CHO, –C=O, –CN, –NH2, –OH etc.), such functionalities act as adsorption centers and make the molecules able to be adsorbed on the metal surface forming a protective layer that reduces the effect of the aggressive acid solution on the metal surface [7-12]. The planarity of the molecules and presence of π electrons and proton donors increases the affinity to the metal surface and make the formed layer more stable and consequently more efficient as corrosion inhibitors [13,14]. Such structural requirements are available in the azo dyes, the presence of the azo linkage (–N=N–) between two aromatic rings gain the molecule planarity, furthermore the presence the π electrons and nitrogen atoms facilitate the attachment of these molecules on the metal surface [15]. Such compounds are adsorbed onto the metal surface either by electrostatic interaction (physisorption) or by formation a coordination complex (chemisorption) to form a layer which acts as guard that protects the metal from the attack of the aggressive acidic environment [16]

As a apart of our interest in the synthesis of azo compounds and corrosion inhibitors [17,18], here work reports the use of two aromatic azo aldehydes as carbon steel corrosion inhibitors for carbon steel in 1 M H2SO4, and we report a comparative study between the one which has nitrile group (AS1) and the other which has methyl group (AS2) to determine the effect of such substituents on the corrosion inhibition behavior at different concentrations and temperatures.

2. Experimental
2.1. Preparation of Steel Specimens:
A carbon steel rod was cut into cylindrical specimens (2.5 cm diameter and 3 mm thickness) as working electrode which consists of (wt.%): C (0.21%), Si (0.22%), Mn (0.82%), P (0.01%), S (0.01%), Cr (0.78%), Mo (0.32%), Ni (0.08%), Cu (0.19%), and Fe balance. The specimens were then mechanically grounded and polished with deferent grades of emery papers (60 - 3000) followed by treatment with microcloth and polishing paste (diamond suspension, 3μm) in order to obtained a mirror-like surface. Finally, washed with distilled water, then with absolute ethanol under ultrasonic conditions and kept in a desiccator.
2.2 Synthesis of Compounds AS1 and AS2:

The title compounds were synthesized according to literature procedure [19], Scheme 1.

\[ \text{AS1: Yield (0.65 gm, 65%). m.p. 206-208°C. FT-IR (KBr, cm}\textsuperscript{-1}): \nu (3114), (3096), (2227), (1668), (1573), (1618).} \]

\[ \text{1H NMR (400 MHz, DMSO-d}\textsuperscript{6}) \delta 11.65 (s, 1 H), 10.41 (s, 1 H), 8.14 (m, 1H), 8.23 (d, } J = 8.9 \text{ Hz), 7.90-7.79 (m, } J = 10.0 \text{ Hz}, 7.24 (dd, } J = 7.8, 2.9 \text{ Hz).} \]

\[ \text{13C NMR (101 MHz, DMSO-d}\textsuperscript{6}) \delta: 190.72, 164.69, 154.45, 145.21, 134.28, 130.50, 124.98, 123.24, 123.49, 119.08, 118.95, 113.32. \]

\[ \text{AS2: Yield (0.68 gm, 68%). m.p. 153-154°C. FT-IR (KBr, cm}\textsuperscript{-1}): \nu 3189, 1660, 1570, 1618.} \]

\[ \text{1H NMR (400 MHz, DMSO-d}\textsuperscript{6}) \delta 11.62 (s, 1H), 10.40 (s, 1H), 8.19 (s, } J = 2.6 \text{ Hz), 8.10 (d, } J = 8.9, 2.6 \text{ Hz), 7.85 } - 7.77 (m, 2H), 7.42 (d, } J = 8.1 \text{ Hz), 7.21 (d, } J = 8.9 \text{ Hz, 1H), 2.43 (s, 3H).} \]

\[ \text{13C NMR (101 MHz, DMSO-d}\textsuperscript{6}) \delta 191.07, 164.11, 150.44, 145.06, 141.71, 130.42, 130.07, 124.08, 123.11, 122.84, 119.09, 21.50. \]

2.3 Preparation of solutions:

Different concentrations (0.1, 0.2, 0.4, and 0.6 mM) of AS1 and AS2 were prepared, 0.6 mmol was dissolved it in DMSO (2 mL) and 0.02 g of tween 80 was added to improve the solubility of the test compounds, then volume was brought up to 1000 mL with of 1.0 M H\textsubscript{2}SO\textsubscript{4} to get 0.6 mM concentration which was then used as stock solution for the preparation of the other concentrations by serial dilution.

2.4 Weight Loss Analysis:

The tested specimens were weighed accurately and then immersed in 200 mL of the test solution that contain different concentration of the synthesized inhibitors for 3 h at 303K. And then washed by distilled water and ultrasonically cleaned with absolute ethanol. The inhibition efficiency (Ƞ\textsubscript{w},\%) was calculated according to equation (1) [20].
\[ \eta_{\text{%}} = \frac{w_o - w}{w_o} \times 100 \ldots \ldots (1) \]

Where \( \eta_{\text{%}} \) is the percentage of the efficiency of inhibition, \( W_o \) and \( W \) are the weight loss in the presence and absence of inhibitors.

2.5 Potentiostatic Polarization Measurements:
Potentiostatic polarization measurements were conducted in a cell consists of three electrodes, reference electrode (saturated calomel electrode SCE), counter electrode (platinum electrode), and working electrode (carbon steel specimens). The working electrode was dipped into the test solution for 24 min with a time step of 1 sec and left to attain steady-state open circuit potential (OCP). Potential range was (-200 to +200) mV relative to the OCP at a scan rate of 0.3 mV s\(^{-1}\). The corrosion potential (\( E_{\text{corr}} \)) and corrosion current density (\( I_{\text{corr}} \)) were measured by Tafel method. The inhibition efficiency (\( \eta_{\text{%}} \)) was calculated as in equation (2) [21].
\[ \eta(\%) = \frac{I_{\text{corr}} - I_{\text{inh}}}{I_{\text{corr}}} \times 100 \ldots \ldots (2) \]

Where \( \eta(\%) \) is the ratio of the efficiency of inhibition \( I_{\text{corr}} \), \( I_{\text{inh}} \) is corrosion current density in the absence and presence of inhibitors.

2.6 Surface Analysis:
The carbon steel samples were immersed in the acid solution (1M H\(_2\)SO\(_4\)) for 3 hours in absence and presence the optimum concentrations of inhibitors (0.6 mM of AS\(_1\) and 0.2 mM of AS\(_2\)). After that the specimens were washed by absolute ethanol, dried and then tested by scanning electron microscope (SEM, Quanta 450 FEI, voltage 25 kv, spot size 3 mm and magnification range 2228 – 2382 x with 20 \( \mu \)m scale bar) and atomic force microscope (AFM), and atomic force microscope (AFM, AA3000 Scanning Probe Microscope).

3. Results and Discussion:

3.1 Gravimetric Analysis:
To test the effectiveness of the azo-aldehydes AS\(_1\) and AS\(_2\) as carbon steel corrosion inhibitors in acidic medium, steel specimens were treated with 1 M H\(_2\)SO\(_4\) as aggressive solution in the absence and presence of different concentrations of the test compounds (0.1, 0.2, 0.4 and 0.6 mM) at 303 K for 3 h, with
monitoring the weight of the specimens before and after treatments to determine the corrosion inhibition efficiency ($\eta_w\%$). The obtained results (Table 1) revealed that for AS1 the corrosion inhibition efficiency increases with increasing the concentration and the maximum efficiency was recorded as 82.07% with the highest used concentration (0.6 mM). While for compound AS2 the maximum efficiency was recorded as 90.12% at 0.2 mM then dropped down to 84.66 and 84.01% with the concentrations 0.4 and 0.6 mM respectively.

Table 1: Gravimetric results of the carbon steel corrosion in 1 M H$_2$SO$_4$ at various concentrations of AS1 and AS2 inhibitor at 303 K at 3 h.

| Compound | Concentration (mM) | $\eta_w\%$ |
|----------|--------------------|------------|
| AS1      | 0.1                | 68.46      |
|          | 0.2                | 76.67      |
|          | 0.4                | 79.48      |
|          | 0.6                | **82.07**  |
| AS2      | 0.1                | 78.47      |
|          | 0.2                | **90.12**  |
|          | 0.4                | 84.66      |
|          | 0.6                | 84.01      |

3.2 Electrochemical Analysis:

Electrochemical (potentiostatic polarization) analyses were implemented for more detailed studies about the type and mechanism of action of the corrosion inhibitors AS1 and AS2. The same range of concentrations were used, furthermore, the effect of temperature on the inhibition efficiency was studied by running the experiments at different temperatures (303, 313, 323 and 333 K) for each concentration of AS1 and AS2 in the aggressive acidic medium.

Table 2 shows the obtained electrochemical parameters including corrosion potential ($E_{corr}$), corrosion current ($I_{corr}$), Tafel slopes [anodic ($\beta_a$) and cathodic ($\beta_c$)], inhibition efficiency ($I\%$) and surface coverage ($\Theta$), these parameters were measured at 303 K for blank (no inhibitor added), AS1 and AS2. Figures 1 shows the generated Tafel curves in the absence and presence of different concentrations of AS1 and AS2 at 303 K. It is clear that the significant decrements in the $I_{corr}$ values refer to the action of both of AS1 and AS2 as corrosion inhibitors. There are two indicators for concluding that both AS1 and AS2 act as mixed type inhibitors when compared with blank, first the negative (decrement) of the values of both anodic and
cathodic currents at the same time [22,23] second, for the $E_{corr}$ values the displacements were found to be in the range of 12 mV (less than 85 mV) [24,25,26].

For AS1, the inhibition efficiency increases with increasing the concentration, reaching the maximum of 86.88% at 0.6 mM, that means increasing the concentration increase the number of molecules being adsorbed on the metal surface forming more active protecting layer [27] whereas further increase in the AS1 concentration causes slightly decrease in the inhibition efficiency. while for AS2, the maximum inhibition efficiency 93.11% was recorded at 0.2 mM then decreased with increasing the concentration, this could be attributed to the fact that there is a limit for amount of AS2 to be adsorbed on the metal surface, beyond that limit the unadsorbed molecules dissolves in the aggressive solution and increases its effect on the metal. However, AS2 is more effective than AS1 at lower concentration.

**Table 2: Electrochemical parameters for carbon steel corrosion in 1 M H2SO4 in the presence of different concentrations AS1 and AS2 at 303 K.**

| Inhibitor | Conc. (mM) | $-E_{corr}$ (mV) | $I_{corr}$ ($\mu$A/cm²) | $\beta_a$ (mVdec⁻¹) | $-\beta_c$ (mVdec⁻¹) | $\eta$% | $\Theta$ |
|-----------|-----------|-----------------|-----------------|-----------------|-----------------|--------|--------|
| Blank     | 0         | 386.0           | 7120            | 60.6            | 85.4            | 0      | 0      |
| AS1       | 0.1       | 386.4           | 1360            | 44.2            | 103.3           | 80.89  | 0.8089 |
| AS1       | 0.2       | 388.0           | 1260            | 40.7            | 87.8            | 82.30  | 0.8230 |
| AS1       | 0.4       | 385.5           | 1010            | 34.0            | 66.8            | 85.81  | 0.8581 |
| AS1       | **0.6**   | **380.7**       | **933.52**      | **30.5**        | **47.9**        | **86.88**| **0.8688** |
| AS2       | 0.1       | 393.5           | 664.30          | 33.1            | 105.3           | 90.66  | 0.9066 |
| AS2       | **0.2**   | **375.5**       | **490.26**      | **18.3**        | **63.2**        | **93.11**| **0.9311** |
| AS2       | 0.4       | 391.6           | 844.95          | 34.8            | 73.6            | 88.13  | 0.8813 |
| AS2       | 0.6       | 378.3           | 893.90          | 26.3            | 101.3           | 87.44  | 0.8744 |
Figure 1: Potentiostatic polarization curves for carbon steel in 1 M H₂SO₄ in the absence and presence of AS1 (left) and AS2 (right) at 303 K

For each used concentration of both AS1 and AS2, the effect of temperature was studied by measuring the electrochemical parameters and inhibition efficiencies at higher temperatures (313, 323 and 333 K), the results revealed that increasing the temperature reduces the inhibition efficiency, and this could be the result of desorption process that happens at higher temperatures [28,29], the effect of increasing the temperature on the inhibition efficiency of AS1 is greater than it on AS2, the minimum inhibition efficiency of AS1 was found to be 33.04% with concentration of 0.1 mM at 332 K, while at the same concentration and temperature the inhibition efficiency of AS2 was 75.30%, Figure 2 and Figure 3 show the relationship between inhibition efficiency and temperature.
3.3 Adsorption Isotherms:

The adsorption behaviour of AS1 and AS2 onto carbon steel surface has been investigated over the temperature range (303-333) K, several adsorption isotherm were tested, however, a linear relationship was found between $C_{inh}/\theta$ and $C_{inh}$ indicating the adsorption process of both inhibitors obey Langmuir isotherms, equation 1, Figures 4 and 5.

Figure 2: The effect of temperature on the inhibition efficiency of AS1

Figure 3: The effect of temperature on the inhibition efficiency of AS2.
The equilibrium constant ($k_{ads}$), enthalpy change ($\Delta H_{ads}$), Gibbs free energy ($\Delta G_{ads}$) and standard entropy change ($\Delta S_{ads}$) values were determined by equations 1, 2, 3 and 4 [30,31,32,33].

$$\frac{C_{inh}}{\theta} = \frac{1}{k_{ads}} + C$$ .........................1

$$\log k_{ads} = \left( -\frac{\Delta H_{ads}}{2.303 R T} \right) + \text{Constant}$$ ..................2

$$\Delta G_{ads} = -RT \ln(55.5 k_{ads})$$ ......................3
Where $C_{inh}$ is concentration of th inhibitor, $\theta$ is the fraction of the surface covered, $k_{ads}$ is the equilibrium constant of the adsorption process, $T$ is the absolute temperature and $R$ is the universal gas constant. The numerical value of 55.5 represents the molar concentration of water in acid solution [34,35,36]. Figures 6 and 7 represent relationship of $\log k_{ads}$ versus $1/T$ for the adsorption process of AS1 and AS2 respectively.

![Figure 6: $\log k_{ads}$ versus $1/T$ for AS1](image1)

![Figure 7: $\log k_{ads}$ versus $1/T$ for AS2.](image2)

Table 3 includes the obtained results, the values of $k_{ads}$ decrease with increasing the temperature and the significant effect of temperature on the $k_{ads}$ is observed in the case of AS1, while less effect was observed for AS2, that means the interaction between the molecules of AS2 and the metal surface is stronger and resists the effect of temperature making the formed protective layer more efficient corrosion inhibitor [37].
The negative values of $\Delta G_{ads}$ indicate that the adsorption of AS1 and AS2 on the carbon steel surface is spontaneous and also confirm the stability of the adsorbed layer, furthermore, the $\Delta G_{ads}$ (about $-20$ kJ mol$^{-1}$) values indicate that the adsorption is physisorption [38,39]. The negative $\Delta H_{ads}$ values refer to the adsorption process of AS1 and AS2 molecules onto carbon steel surface is an exothermic process [40].

Table 3: The calculated thermodynamic parameters for the adsorption of AS1 and AS2

| Compound | Temperature (K) | $k_{ads}$ (M$^{-1}$) | $\Delta G$ (kJ mol$^{-1}$) | $\Delta S$ (kJ mol$^{-1}$) | $\Delta H$ (kJ mol$^{-1}$) |
|----------|----------------|---------------------|---------------------------|--------------------------|-------------------------|
| AS1      | 303            | 73.52941            | -20.9443                  | -0.25636                 | -98.6211                |
|          | 313            | 18.48429            | -18.0424                  | -0.25744                 |                         |
|          | 323            | 5.995204            | -15.5952                  | -0.25705                 |                         |
|          | 333            | 2.138123            | -13.2235                  | -0.25645                 |                         |
| AS2      | 303            | 66.66667            | -20.6975                  | -0.15088                 | -25.0177                |
|          | 313            | 45.45455            | -20.3839                  | -0.14505                 |                         |
|          | 323            | 33.78378            | -20.2383                  | -0.14011                 |                         |
|          | 333            | 27.3224             | -20.2772                  | -0.13602                 |                         |

3.4 SEM Analysis:

The effect of the aggressive acidic solution on the carbon steel surface in the absence and presence of AS1 and AS2 was visualized by studying the surface morphology using SEM, Figure 8 shows the recorded micrographs, the damage caused by the effect of the acid was reduced remarkably when AS1 and AS2 are added to the solution indicating that both of them are forming a protective layers and acting as efficient corrosion inhibitors.
3.5 AFM Analysis:

Atomic force microscopy (AFM) was also used to study the effect of the acidic solution on the morphology of the carbon steel surface in the absence and presence of the optimum concentrations of AS1 and AS2 where samples were immersed for 3 h at 303 K. Figure 8 represents the 3D AFM micrographs of the test specimens; it is clear that the damage caused by the effect of the acidic solution was significantly reduced when the inhibitors AS1 and AS2 added to the solution. The determined values of the average surface roughness were found 0.9 nm for the polished specimen before treatment (Figure 9 (A)), 155 nm for specimen treated with acid in the absence of any inhibitor (Figure 9 (B)), while in the presence of AS1 (0.6 mM) and AS2 (0.2 mM) the calculated average roughness were 7.4 and 1.5 nm respectively (Figure 8 (C) and (D)), these results come in line with determined inhibition efficiencies ($\eta_w$ %) of AS1 and AS2 which are 82.07 and 90.12% respectively.

Figure 8: SEM micrographs of untreated (A), uninhibited (B), inhibited with AS1 (C) and inhibited with AS2 (D) carbon steel surfaces.
Figure 9: 3D AFM micrographs of the test specimens: before treatment (A), uninhibited (B), inhibited with AS1 (C) and inhibited with AS2 (D).

Conclusions:

In this work, the main conclusions were determined that the compounds AS1 and AS2 can be considered as efficient corrosion inhibitors for carbon steel in 1 M H₂SO₄. Compound AS2 with methyl substituent is more efficient than AS1 even at lower concentration, this could be attributed to the hydrolysis of the –CN group of compound AS1 converting to carboxyl group in the acidic environment which reduces the inhibition efficiency especially at higher concentrations.
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