Synthesis of New Aromatic Azo-Schiff Compound as Carbon Steel Corrosion Inhibitor in 1 M H₂SO₄; High Efficiency at Low Concentration

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Abstract: Schiff base derivative (AS) namely (4-((E)-(4-hydroxy-3-((E)-(phenylimino)methyl)phenyldiazenyl)benzonitrile) was synthesized and tested as corrosion inhibitor for the carbon steel alloy in 1M sulphuric acid at different concentration (0.005, 0.01, 0.02, 0.04 and 0.08 mM) over temperature rang (303-333) K. Potentiodynamic polarization, weight loss, atomic force microscopy (AFM) and scanning electron microscopy (SEM) techniques have been used in the study. The results revealed that the inhibition efficiency increases as inhibitor concentration increases and decreases with the increasing the temperature. The maximum inhibition recorded efficiency 98% and 97.5% by potentiodynamic polarization and weight loss methods respectively at 303 K in the presence of 0.08 mM of AS. $E_{corr}$ values Proof that AS act as mixed type inhibitor. Adsorption isotherm of AS on the carbon steel surface obeys Langmuir adsorption isotherms model.

Keywords: Azo; Schiff base; Carbon steel; Corrosion inhibitors; Organic inhibitors, Acidic corrosion.

1. Introduction:

Acids are widely used in industry for different purposes such as cleaning of metallic parts of machines, tanks and pipelines; corrosion is the accompanying problem with these processes, it is the damage caused to the metal due to the aggressive attack of the acid when it come in contact with the metal surface (1-3). Organic corrosion inhibitors are used as one of the most efficient, practical and cost effective techniques for metal protection from the effects of acids (4,5). The molecules of the inhibitors are adsorbed onto the metal surface by different fashions forming a protective file that isolate the metal surface from the acidic medium and inhibit the corrosion process (6). The organic compounds that include heteroatoms in their structures (like sulphur, oxygen, nitrogen etc), aromatic ring and conjugated multiple bonds ($\pi$ systems) can act as good corrosion inhibitors since they are rich with adsorption centres that facilitate their attachment to the metal surface (7-9). Schiff bases have been proved to be effective inhibitors for the acidic corrosion because of its $\text{–C=N}–$ group in which N atom is eligible to form coordinate-covalent bond by its unshared electrons, whereas the $\pi$-bond capable to interact with the metal surface (10). Azo compounds that contain $\text{–N=N–}$ group are also used as corrosion inhibitors the azo group capable to form a chelate complex with the metal surface and for the guard layer (11).

In a previous work we have reported the use of azo-schiff compound as efficient carbon steel corrosion inhibitor in 1 M H₂SO₄(12). In the present work we report the synthesis and use of new azo-schiff derivative as carbon steel corrosion inhibitors, the new inhibitor differs from the
previously reported one by it does not have the electron donating methoxy group in its structure to determine whether this group has a significant effect on the corrosion inhibition efficiency.

2. Experimental

2.1. Specimens Preparation:

Carbon steel specimens with cylindrical shape (24 mm diameter and 3 mm height). Its elements compositional are 0.22% C, 0.23% Si, 0.83% Mn, 0.006% P, 0.008% S, 0.79% Cr, 0.33% Mo, 0.08% Ni, 0.02% Al, 0.19% Cu and Fe were used as working electrode. The specimens were grounded and polished by mechanical machine with deferent grade of emery papers (80 - 3000) and then the grounded surface was treatment with polishing paste (diamond suspension, 1 μm) to obtain a mirror like surface. Finally the specimens were rinsed in distilled-water then in absolute ethanol in ultrasound water bath.

2.2. Inhibitor synthesis:

Scheme 1 illustrates the synthesis steps of the target compound (AS):

Scheme 1: Synthesis of AS

Compound 3 and AS was prepared according to procedure in literature procedure(12,13), then (0.5 gm, 1.99 mmol) of compound 3 was dissolved in 5 ml of DCM, aniline (0.245 gm, 1.99 mmol) was dissolved in 25 ml DCM, solutions were mixed and catalytic amount of glacial acetic acid was added. The mixture was reflexed for 5 h, then the product was filtered off and washed by hot ethanol.

FT-IR (KBr, cm⁻¹): ν (3431, OH), (2218, C≡N), (1617, C=N), (1485, N=N), (1591, C=C).

1H NMR (400 MHz, DMSO-d6) δ (ppm): 14.07 (s, 1H, H-7), 9.16 (d, J = 4.1 Hz, 1H, H-4), 8.34 (d, J = 4.5 Hz, 1H, H-8), 8.09 - 8.00 (m, 3H, H-2,16,12), 8.00- 7.93 (m, 2 H, H-13,15), 7.57 – 7.46 (m, 4 H,H-20,21,23,24), 7.36 (apparently m, 1 H, H-1), 7.25 – 7.11 (m, 1 H, H- 22).

13C NMR (101 MHz, DMSO-d6) δ 165.63(C-6), 163.01 (C-8,11), 145.19 (C-19), 134.28 (C- 3), 130.04 (C-13,15), 129.46(C-21,23) , 128.01(C-22), 127.91(C-4) 123.37(C-12,16), 121.87(C-20,24), 119.76(C-5), 118.95(C-17), 114.34(C-1) , 113.10(C-14).

2.3. Solutions preparation:
A stock solution of inhibitor solution with concentration 0.08mM was prepared by dissolving 0.029 gm of AS in 2 ml of DMSO and 0.01 gm of tween-80 was added to obtain a clear solution, then brought up the volume to 1000 ml with 1.0 M sulphuric acid. Different concentrations (0.005, 0.01, 0.02 and 0.04 mM) were prepared by dilution.

2.4. Potentiodynamic Polarization Measurements:

The potentiostatic polarization measurements carried out by a three electrodes potentiostat circuit. Saturated calomel electrode Hg / Hg₂Cl₂ used as reference electrode, Pt electrode used as auxiliary electrode. The carbon steel specimen was connected to the working electrode with exposure area of 1 cm². The working electrode was then dipped in the test solution for (30 min with time step of 2 sec). The changing in electrode potentials was ±250 mV(14) and sweep rate 0.3 mVs⁻¹. By the extrapolation of Tafel plots segments, the corrosion current density ($I_{corr}$) was measured; the inhibition efficiency (%) was calculated according to equation (1):

$$\eta \% = \frac{I_{corr\text{-inhibitor}} - I_{corr}}{I_{corr}} \times 100$$  \hspace{1cm} (1)

where $I_{corr}$ and $I_{corr\text{-inhibitor}}$ represent corrosion current density without and with AS respectively.

2.5. Weight loss and surface study:

Accurately weighed specimens were immersed in the test solutions (200 ml, without and with different inhibitor concentrations) for 3 h at 303 K. After this immersion period the samples washed by absolute ethanol in ultrasound water bath and accurately weighed. The inhibition efficiency $\eta \text{w\%}$ calculated as in equation (2):

$$\eta \text{w\%} = \frac{W_o - W}{W_o} \times 100$$  \hspace{1cm} (2)

where $W$ and $W_o$ represent the weight loss for the specimen after immersion with and without inhibitors.

The morphology of the test samples was analysed by SEM and AFM techniques after immersion in the aggressive solution with and without the inhibitor (0.08 mM, at 313 K).

3. Results and Discussion:

3.1. Electrochemical studies (Tafel plots):

In order to understanding the effect of the synthesized inhibitor (AS) on the carbon steel corrosion in 1 M H₂SO₄, the potentiodynamic polarization measurements were done in presence of different concentrations of AS over the temperature rang 303-333 K. Table 1 displays the measured experimental data including corrosion potential ($E_{corr}$), corrosion current density ($I_{corr}$), cathodic and anodic Tafel slopes ($\beta_c$ and $\beta_a$), inhibition efficiency ($\eta \%$) and surface coverage ($\Theta$).

It was found that the corrosion current density decreases with the addition of the AS to the aggressive media at constant temperature and increases as temperature increases with slight displacement in the $E_{corr}$ values. From the value of the shift in the $E_{corr}$ we can conclude that AS acts as mixed-type inhibitor due to the shift was less than 85 mV when compared the $E_{corr}$ of different inhibitor concentrations at constant temperature with the $E_{corr}$ of blank solution(17). That
implies the AS inhibitor work as a retardant for the anodic reaction of carbon steel (dissolution reaction) and at the same time as a retardant for the hydrogen evolution reactions\(^{(18)}\). It is clear that the variation in the \(\beta_a\) values more than in it in the \(\beta_c\) values, that refers to formation of inhibitor complexes with Fe in the low and/or higher oxidation state on the carbon steel surface, and indicate that more inhibition effect of AS on the anodic process than on the cathodic process\(^{(19)}\). All of that is an explanation for the reasons of increasing the inhibition efficiency with increasing the inhibitor concentration and decreasing the inhibition efficiency with increasing the temperature range. Figure (1)

![Figure 1](image-url)

**Fig. 1.** Potentiodynamic polarization curves for carbon steel in 1 M H\(_2\)SO\(_4\) in the absence and presence of AS at 303 K

| Table 1: Polarization parameters for carbon steel corrosion in 1 M H\(_2\)SO\(_4\) in the presence of different concentrations AS at different temperatures. |
|---|---|---|---|---|---|---|---|
| Tem. K | Inhibitor Con.mM | \(-E_{corr}\) (mV) | \(I_{corr}\) (\(\mu\)A/cm\(^2\)) | \(\beta_c\) (mVdec\(^{-1}\)) | \(-\beta_c\) (mVdec\(^{-1}\)) | \(I_\%\) | \(\theta\) |
3.2. Weight loss method:

The results of gravimetric analyses were implemented to confirm the results obtained from potentiostatic polarization measurements, Table 2 includes the recorded corrosion inhibition

| Concentration (g/L) | 303 | 313 | 323 | 333 |
|---------------------|-----|-----|-----|-----|
| 0                   | 386.0 | 7120.0 | 60.6 | 85.4 | 0 | 0 |
| 0.005               | 384.8 | 7620.1 | 88.5 | 158.8 | 0 | 0 |
| 0.01                | 384.9 | 8850.0 | 59.6 | 86.3 | 0 | 0 |
| 0.02                | 380.6 | 9110.0 | 64.5 | 101.8 | 0 | 0 |
| 0.04                | 386.0 | 1870.0 | 51.5 | 105.8 | 73.3 | 0.733 |
| 0.08                | 385.8 | 3857.8 | 49.6 | 75.9 | 49.4 | 0.494 |
| 0.1                 | 386.7 | 6575.5 | 67.1 | 109.8 | 25.7 | 0.257 |
| 0.005               | 378.2 | 9030.0 | 55.4 | 73.3 | 0.87 | 0.0087 |
| 0.01                | 383.9 | 1220.0 | 34.3 | 49.2 | 82.8 | 0.82 |
| 0.02                | 379.8 | 2876.6 | 51.4 | 134.3 | 62.2 | 0.622 |
| 0.04                | 385.1 | 5660.0 | 45.4 | 61.8 | 36.5 | 0.365 |
| 0.08                | 386.3 | 7440.0 | 63.3 | 88.9 | 18.3 | 0.183 |
| 0.1                 | 396.6 | 413.1 | 38.3 | 88.8 | 94.7 | 0.94 |
| 0.02                | 390.0 | 2080.0 | 44.6 | 86.3 | 72.7 | 0.727 |
| 0.04                | 385.6 | 4920.0 | 47.2 | 86.6 | 44.4 | 0.444 |
| 0.08                | 387.5 | 7310.0 | 63.0 | 88.3 | 19.7 | 0.197 |
| 0.1                 | 402.5 | 286.14 | 33.3 | 103.2 | 95.9 | 0.95 |
| 0.02                | 387.3 | 1870.0 | 46.0 | 82.8 | 75.4 | 0.754 |
| 0.04                | 386.0 | 4660.0 | 52.3 | 74.1 | 47.3 | 0.473 |
| 0.08                | 382.8 | 6960.0 | 62.3 | 121.4 | 23.6 | 0.236 |
| 0.1                 | 387.1 | 136.94 | 18.3 | 29.8 | 98.0 | 0.98 |
| 0.02                | 397.1 | 875.12 | 43.4 | 101.8 | 88.5 | 0.885 |
| 0.04                | 386.4 | 3200.0 | 64.0 | 185.5 | 63.8 | 0.638 |
| 0.08                | 385.4 | 6930.0 | 53.1 | 68.9 | 23.9 | 0.239 |
efficiencies at different concentrations of AS, the maximum inhibition efficiency was 97.5% at 0.8mM.

Table (2) Weight loss results for the carbon steel corrosion in 1 M H$_2$SO$_4$ at various concentrations of AS at 303 K after 3 h of immersion.

| Concentration of AS (mM) | $\eta_w$ (%) |
|--------------------------|--------------|
| 0.005                    | 69.20        |
| 0.01                     | 84.60        |
| 0.02                     | 94.60        |
| 0.04                     | 96.66        |
| 0.08                     | 97.5         |

3.3. Adsorption isotherms:

The adsorption of AS on the carbon steel surface has been studied, the obtained results indicated that the adsorption obeys the Langmuir isotherms model (1) with correlation coefficients around (0.995), Figure (2). From Langmuir equation the equilibrium constant of the adsorption process ($K_{ads}$) values were calculated, the values of free energy $\Delta G_{ads}$ figure (3) and reaction enthalpy $\Delta H_{ads}$ were calculated according to equations (2) and (3) respectively $^{20,21}$.

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C$$  \hspace{1cm} (3)

$$\Delta G_{ads} = -RT \ln \ln (55.5 K_{ads})$$ \hspace{1cm} (4)

$$\log \log K_{ads} = \left(- \frac{\Delta H_{ads}}{2.303 RT}\right) + \text{Constant}$$ \hspace{1cm} (5)

Where $C_{inh}$ is concentration of the inhibitor, $\theta$ is the surface coverage, $T$ 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.
Figure. (2) Langmuir relationship for AS inhibitor adsorption in 1M sulfuric acid at different temperature in the range (303-333)K.

Figure. (3) Relationship of Log $K_{ads}$ versus 1/T for carbon steel with and without AS inhibitor in 1M sulfuric acid solution at different temperature in the range (303-333)K.

Table 3 shows the adsorption parameters $K_{ads}$, $\Delta G_{ads}$ and $\Delta H_{ads}$, their values give an illustration about the nature of adsorption process and the ability of the inhibitor to get adsorbed onto the metal surface. The values of $K_{ads}$ reflect the affinity of AS for the adsorption on the metal surface.
in which the higher $K_{ad}$ values refers to high ability for adsorption and better inhibition efficiency\cite{22,23}. The negative $\Delta G_{ads}$ values indicate the spontaneous inhibition process and high stability of the adsorbed layer of AS on the metal surface, furthermore, $\Delta G_{ads}$ values in this study were around $\sim$20 kJ mol$^{-1}$ which suggests the kind of adsorption was physisorption\cite{24}. The negative $\Delta H_{ads}$ values refer to the exothermic nature of the adsorption process and indicate that the inhibition efficiency decreases as temperature increases\cite{25}.

| Compound symbol. | Medium Con./M | Tem. (K) | $K_{ad}$ M$^{-1}$ | $\Delta G_{ads}$ kJ.mol$^{-1}$ | $\Delta H_{ads}$ kJ.mol$^{-1}$ |
|------------------|--------------|----------|------------------|------------------|------------------|
| AS               | 1M           | 303      | 588.235          | -26.182          | -89.003          |
|                  |              | 313      | 163.934          | -23.722          |                  |
|                  |              | 323      | 066.225          | -22.045          |                  |
|                  |              | 333      | 059.500          | -22.431          |                  |

3.4. Surface analysis:

As shown in the SEM micrographs (Figure 3), the serious damage which is caused by the effect of the acidic solution on the metal surface in the absence of AS was significantly decreased in its presence, this confirms that AS protects the surface of the metal surface from the attack of the aggressive acidic solution.

![SEM micrographs](image1)

Fig 4. Illustration of SEM micrograph (A) specimens surface immersed in blank (B) specimens surface before treatment with AS solution.

The AFM analysis came in line with the SEM results, the measured of the specimens before treatment was 0.895 nm, after treatment with 1 M H$_2$SO$_4$ the roughness was found to be 17.1 nm such increment is a common corrosion result. For specimen immersed in the acidic solution that containing AS (0.08 mM) the surface roughness was found to be 2.2 nm, such variation clearly
indicate that AS acts as excellent protective corrosion inhibitor, Figure 4 show the AFM 3D micrographs of non-treated (A), uninhibited (B) and inhibited (C) specimens.

![AFM Micrographs](image.png)

**Fig 5.** A represent to the polished specimens before treatment B represent to the specimens that immersed in blank solution C represent to the specimens that immersed in AS solution

**Conclusions:**

A new azo-schiff compound (AS) has been synthesized, the inhibition efficiency of AS for carbon steel corrosion in 1 M H$_2$SO$_4$ was investigated, tween 80 was used as surfactant to improve the solubility of the inhibitor. High inhibition efficiency was recorded (98% by potentiodynamic polarization and 97.5% by weight loss measurements) with relatively low concentration (0.08 mM).
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