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Asymmetric azo Compounds synthesis, Characterization and Study them as Corrosion Inhibitors for Carbon Steel in Acidic Medium

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Abstract

New two azo compounds were synthesised from a coupling reaction of 3-tert-butyl-4-hydroxy anisol with 4-amino acetophenone or 4-aminobenzosulfonic acid to produce (*E*)-1-(4-((3-(tert-butyl)-2-hydroxy-5-methoxyphenyl) diazenyl) phenyl) ethan-1-one (1), and sodium (*E*)-4-((3-(tert-butyl)-2-hydroxy-5-methoxyphenyl) diazenyl) benzenesulfonate (2) . These compounds were characterized using FT-IR, 1HNMR, and mass spectroscopy. Compounds 1 and 2, were tested as inhibitors toward corrosion phenomenon of carbon steel in 1 M HCl solution based on potentiodynamic polarisation techniques and scanning electron microscopy (SEM). Show results that using high concentrations of compounds enhanced inhibitory efficiency(IE) which was reduced when the temperature was increased. Tafel polarization curves showed that corrosion rate was decreased with an increasing concentration of compounds. The values of an inhibitory efficiency (IE) were calculated by using several techniques and found that the order of IE of the inhibitors synthesized was inhibitor 2 high than inhibitor 1.

Keywords: Azo Compounds, Corrosion inhibition, Potentiodynamic Polarization, SEM.

1. Introduction

Carbon steel is an iron alloy that corrodes readily in an acidic environment. Acidic solutions are commonly employed in chemical laboratories and in a variety of industrial procedures, including acid pickling, acid cleaning, acid descaling, and oil-wet cleaning, to name a few. Carbon steel is also utilized in the chemical and allied industries to handle alkaline, acid, and salt solutions under various conditions[1–3]. Inhibitors are frequently used in industrial operations to ensure that metals do not dissolve in acid solutions. Organic molecules containing O, S, and/or N atoms have long been considered potent inhibitors[4,5]. These organic molecules adsorb on metallic surfaces[6]. Adsorption is primarily dependent on specific physicochemical properties of the inhibitor, such as functional groups, electron density at the donor atom, - orbital character, and the molecule's electronic structure[6,7]. Organic inhibitors that can effectively reduce or avoid the corrosion effects of metals and metallic alloys are considered as a beneficial way to achieve this goal. Many corrosive influences can be reduced or prevented from metal surfaces by using organic compounds to form a protective layer[8,9]. Azo compounds are a type of organic chemical that can be utilized to make a variety of synthetic organic molecules[10-12]. These compounds are found in various products, including textiles[13], leather, cosmetics, medicinal reagents, and also employed in the corrosion inhibition of metals and alloys because of their distinctive chemical structures [10,14]. Thus, in this study, the corrosion inhibition of carbon steel in 1 M HCl solution was studied in the presence and absence of the inhibitors (compounds 1, and 2) in different concentrations and temperatures, and evaluate their efficiency through using potentiodynamic polarization and scanning electron microscopy (SEM). Additionally, changes to inhibition efficacy in response to concentration and temperature changes were assessed. Subsequently, several thermodynamic characteristics were calculated.

2. Experimental

2.1. Metal coupons composition and medium

Test has been performed on carbon steel (CS) of the following composition: 0.3% C, 1.2% Mn, 0.05%

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P, 0.06% S, and the remainder is Fe. 1 M HCl was prepared by dilution of HCl (BDH grade, 37%) with distilled water.

2.2. Synthesis of (E)-1-(4-((3-(tert-butyl)-2-hydroxy-5 methoxyphenyl)diazenyl)phenyl)ethan-1-one (1).

Solution of 4-amino acetophenone (0.675g, 5mmol) dissolved in a dilute HCl solution (concentrated HC l: H2O, 1:1 v/v) was cooled in an ice bath (≤ 5 °C). To above content, NaNO2 (0.38 g) dissolved in water (10 mL) was added dropwise. This mixture was continually stirred under an ice bath. To above mixture, 3-tert-butylhydroxyanisolesol (0.901g, 5mmol) was gradually added. The resulting mixture was chilled in an ice bath and stirred additionally for 20 min. The precipitated product was then filtered and recrystallized from ethanol. Finally, the desired product was dried in the oven at 50 °C for 24 h.

Dark orange solid, yield: ٨٦%, m.p. 150-152 °C. IR (υ, cm−1): 1575 cm-1 (N=N), 1681cm-1 (C=O), 1597 cm-1 (aromatic C=C) and 3415 cm-1 (O-H) Phenolic. 1H-NMR (500 MHz, DMSO) δ (ppm): 1.42 (s, 9H ,t-But), 2.64 (s, 3H , CH3), 3.77 (s, 3H , OCH3), 11.42(s, 1H , OH) and 7– 8.1 (m ,6H , Ar-H). MS [EI]+ m/z 326.2 [M]+.

2.3. Sodium (E) – 4 - ((3-(tert-butyl) – 2 – hydroxyl - 5 methoxyphenyl) diazenyl) benzenesulfonate (2)

Solution of 4-aminobenzosulfonic acid (0.865g, 5mmol) dissolved in a dilute HCl solution (concentrated HCl: H 2O, 1:1 v/v) was cooled in an ice bath (≤ 5 °C). To above content, NaNO2 (0.38 g). dissolved in water (10 mL) was added dropwise. This mixture was continually stirred under the ice bath. To the above mixture, 3-tert-butyl-hydroxyanisoleol (0.901g, 5mmol) was gradually added. The resulting mixture was chilled in an ice bath and additionally stirred for 20 min. The precipitated product was then filtered and recrystallized from ethanol. Finally, the desired product was dried in the oven at 50 °C for 24 h.
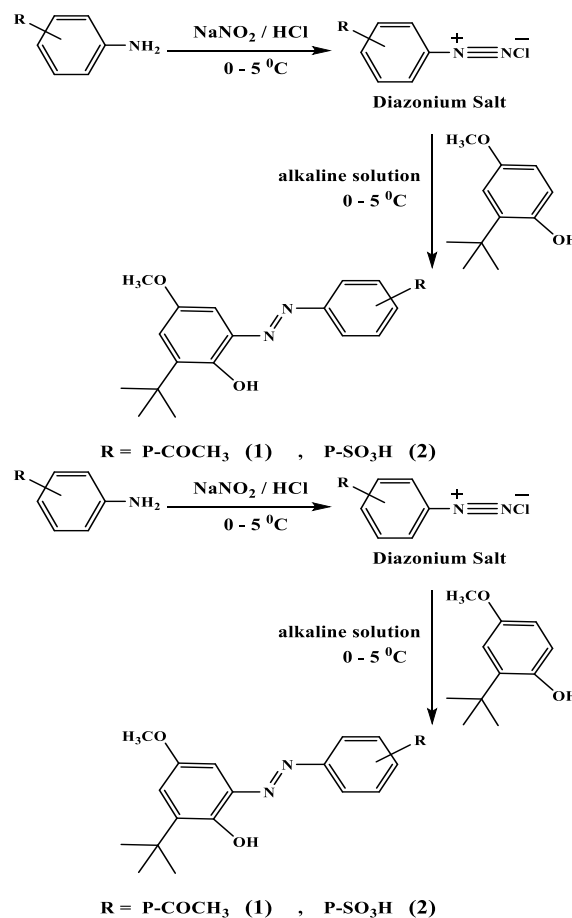
Blood Red solid, yield: 70%, m.p. 294-296°C. IR (υ, cm−1): 1454 cm-1 (N=N), 1126-1309 cm-1 (SO2), 1614 cm-1 (aromatic C=C) and 3452 cm-1 (O-H) Phenolic. 1H-NMR (500 MHz, DMSO) δ (ppm): 1.41 (s, 9H, t-But), 3.76 (s, 3H, OCH3), 11.42(s, 1H , OH) and 6.98– 8 (m ,6H , Ar-H), MS [EI]+ m/z 386 [M]+.

3. Results and discussion
3.1. Chemistry

In this work, diazonium salts were prepared by a diazonation reaction of 4-amino acetophenone and 4-aminobenzosulfonic acid in the presence of an aqueous HCl and sodium nitrite. The fresh diazonium compounds were coupled with 3-tert-

butylhydroxyanisolesol to synthesize compounds 1 and 2 respectively, as shown in Scheme 1.

Scheme 1: Synthesis of azo compounds 1 and 2

The structures were confirmed by IR, 1H-NMR, and mass spectroscopy. FT-IR spectrum of compound 1 shows strong bands at 1575 cm-1 and 1597 cm-1 that were assigned to ν(N=N) and (aromatic C=C) groups, respectively 1681cm-1 (C=O), 3415 cm-1 ν(OH) phenolic.The spectrum of compound 2 shows bands at 1126-1309 cm-1 and 1454 cm-1 that we assigned to the (SO2) and ν(N=N) groups respectively, 1614 cm-1 (aromatic C=C), 3452 cm-1 ν(OH) phenolic.

The 1HNMR spectrum of compound **1** shows signals at δ = 1.42 ppm (s, 9H, t-But), 2.64 (s, 3H , CH3), δ = 3.77 ppm (s, 3H , OCH3), the aromatic protons appear as multi-signals in the region δ = 7– 8.1 ppm (m ,6H , Ar-H) and δ = 11.42 ppm (s, 1H , OH). 1HNMR spectrum of compound **2** shows a signals at δ = 1.41 ppm (s, 9H ,t-But)**,** δ = 3.76 ppm (s, 3H , OCH3), the aromatic protons appeared as multiplet signals in the region δ = 6.98– 8 ppm (m ,6H , Ar-H) and δ = 11.42 ppm (s, 1H , OH). The mass spectra of compounds confirm the presence of molecular ion peak at m/z 326.2 for compound **1** and m/z 386 for compound **2**.

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3.2. Potentiodynamic Polarization Measurements

The results that obtained from the potentiodynamic polarization measurements study for the corrosion of carbon steel in the absence or the presence of azo compounds 1 and 2 in 1 M HCl solutions are given in Figures 1 and 2.

The varied polarization parameters such as corrosion current (Icorr), corrosion potential (Ecorr), anodic and cathodic Tafel slopes (-βa and -βc), As well as inhibition efficiency (IE%), have been obtained from potentiodynamic polarization studies on carbon steel in acidic mediums as shown in Table (1).

computations were conducted using the magnitude of corrosion current determined using the extrapolation approach, as specified in equation (1)[15,16]. These tests were performed at several temperatures (298, 308, and 318 K), using many inhibitor (1 and 2) concentration levels (1×10^{-4}, 5×10^{-4}, 1×10^{-3}, and 5×10^{-3} M).

$$IE\% = \frac{(I° \, corr - I \, corr)}{I° \, corr} \times 100 \, ... (1)$$

Where Icorr and I°corr are inhibited and uninhibited density values respectively of corrosion current. The results indicated that inhibitory efficiency increased when the concentration was increased, In addition. additionally, the highest inhibition efficacy was 89.18% at the concentration 5x10-3 M and temperature 298 K of compound 2. Furthermore, the results indicated that temperatures above 298 K reduced the inhibition efficiency; meanwhile that azo synthesized compounds act as a mixed inhibitor on cathodic and anodic changes simultaneously[17].

3.3. Effect of Temperature

The effect of increasing temperature on the corrosion rate of C-steel in 1 M HCl in the absence and presence of different concentrations of the synthesize azo compounds was investigated. Fig. (3) reveals that the corrosion rate increased and %IE decreased when the temperature was increased. This indicates that the rising of temperature caused decreasing the inhibition process the highest inhibition efficiency was obtained at 298K[18–20].
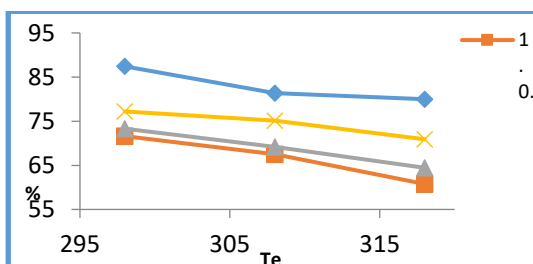

Figure 3: Effect of the temperature on the inhibitory efficiency in the presence of different concentrations of compound 2

3.4. Adsorption Isotherm

The adsorption of organic molecular data showed molecules interact with the surface of the electrode. Chemical and physical adsorption are two primary categories concerning organic molecular adsorption. It is electrolyte type, metal nature and characteristics, and inhibitor chemical arrangement affect adsorption. The surface coverage degree (θ) for several inhibitor reagent concentrations using 1 M HCl solution was computed through equation (2)[16,18]. And values of (θ) are shown in table (1).

$$\theta = \frac{I°coor - Icoor}{I°corr} \, ... (2)$$

Where Icorr and I°corr are inhibited and uninhibited corrosion current density values, respectively. The equation aligns well with the score due to the Langmuir isotherm, as specified by the general expression[22].

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \, ... (3)$$

Where C is an inhibitor to concentration (M), is surface coverage (θ), and K_{ads} is a constant corresponds to adsorptive equilibrium. Surface coverage (θ) was gained graphically to find an appropriate straight correlation line. The determinative coefficient (R^2) was 0.9978-٠,٩٩٩٨ listed in Table 2. The results suggested that the Langmuir adsorption isotherm can be used to explain inhibitory adsorption phenomena with an acceptable level of precision (Figs. 4 and 5). The excellent correlation that concerns with Langmuir adsorption isotherm helps to validate this method. The intercept value is used in reciprocal form to determine the equilibrium constant (K_{ads}) corresponding to the adsorption phenomenon. The equilibrium constant values are illustrated in Table 2.

The large magnitudes proposed that the selected of inhibitor has appreciable adsorption performance on carbon steel surface. The observations could be attributed to the presence of coordinated bonds between the synthesized compound and the d-orbitals of the iron molecules present on the steel surface.

3.5. Thermodynamic Parameters

The following equation gives the standard adsorption of free energy ($\Delta G°_{ads}$)[10].

$$\Delta G°_{ads} = -RT \ln (55.5 \, K_{ads}) \, ... (4)$$

The abbreviate T is the absolute temperature, R represents the gas constant (8.314 J mol^{-1} K^{-1}) and the value 55.5 refers to the water quantity in a solution that expressed M. The negative values of $\Delta G°_{ads}$ express spontaneous adsorption with regards to inhibitor molecule onto the surface of the steel. In general, when the values of $\Delta G°_{ads}$ are less than -20 kJ mol^{-1}, these values expressed for physisorption, as

well from an electrostatic reaction between the charged inhibitor molecules and charged metal surface, whereas values larger than -40 kJ mol^{-1} chemisorption from an electron transfer or sharing between the inhibitor molecules' orbitals and their corresponding orbitals of metal to form a coordinated bond[23]. In our work, the calculated $\Delta G°_{ads}$ values denoted that chemisorption (mixed adsorption) It was noted azo compounds afforded adsorption mechanism on carbon steel via a mixture of both physisorption and chemisorption processes in 1 M HCl solution [14]. The Van't Hoff equation is employed to calculate adsorption heat [24].

$$\text{Log K}_{ads} = \frac{-\Delta H_{ads}}{2.303 \ RT} + \text{Cons} \dots \dots \dots (5)$$

Where $\Delta H°_{ads}$ denotes the adsorption heat constant, K_{ads} identifies the adsorptive equilibrium constant. herein the standard stable peak at 311.2 can be gained by losing versus 1/T (Fig. 6) in order to give a straight line as per Eq. (5) wherein the slope was equal to $- \Delta H°_{ads}/R$. The negative values of $\Delta H°_{ads}$ showed the occurrence of exothermic adsorption for the investigated compounds on the carbon steel surface. Equation (6) is employed to calculate the entropy of inhibitor adsorption $\Delta S°_{ads}$ [23].

$$\Delta G°_{ads} = \Delta H°_{ads} - T\Delta S°_{ads} \dots \dots \dots (6)$$

Table 2 demonstrates the obtained $\Delta S°_{ads}$ values. Positive values of $\Delta S°_{ads}$ denote that there was an increase in entropy along with the adsorption process, as expected with increasing in entropy as it follows endothermic adsorption which refers to the driving force about inhibitor adsorption onto the surface of carbon steel [25].
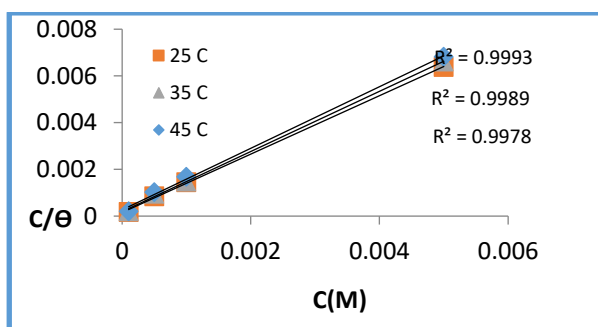

Figure 4: Langmuir plot for investigated compound 1 in 1M HCl solution for CS corrosion at various temperatures

4. SEM and EDX Analysis

The analysis of the morphology surface of the tested steel samples was performed in both the presence and absence of compounds (1 and 2) at an ambient temperature in order to obtain better understanding of the kind of interaction that would occur between the deposited organic inhibitor and the metal surface. As presented in (Fig. 7A), the analysis of SEM images for the steel sample introduced to 1 M HCl, before

exposing to the corrosive solution, parallel characteristics on the polished steel surface, usually linked to polishing scratches, (Fig. 7B- Blank) in the absence of inhibitors demonstrated rough surface as well as damage caused by corrosion via aggressive media[26]. In the same Fig. 7 (images D and E), the deposition of inhibitors on the steel surface is credited for the smooth surface, which also safeguards against corrosion. In the azo molecules, the interaction that exists between the iron, as well as the electron-rich species, is commonly employed to describe the efficient deposition of inhibitors on the surface of the metal. These groups, via blockage of the metal's obtained from carbon steel samples with and without the metal surface, suggesting enhanced inhibition efficiency since adsorption of azo compound molecules occurs on the metal surface. Finally, inhibitors also help to protect the surface against harsh acid solutions, as confirmed via SEM images These two morphological evaluations suggest effective adsorption property of the inhibitor molecules on the steel surface, via the formation of a protective film[29,30].
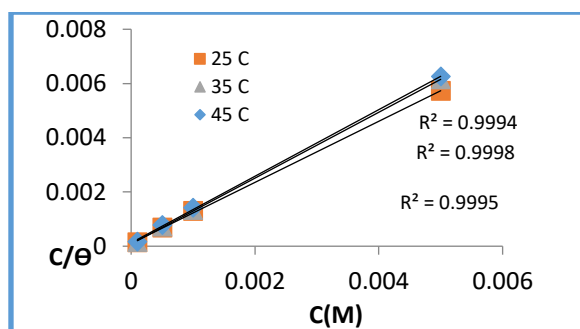

Figure 5: Langmuir plot for investigated compound 2 in 1M HCl solution for CS corrosion at various temperatures
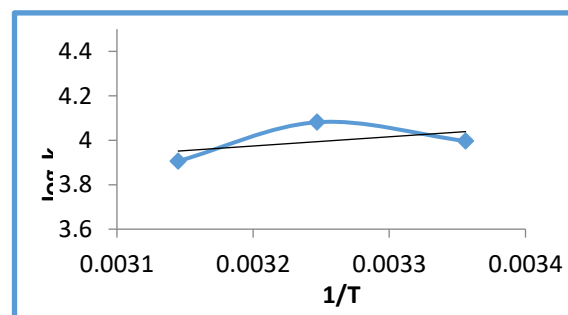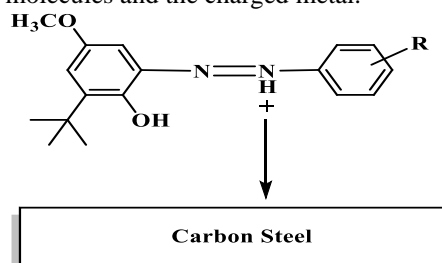

Figure 6: The relationship between Log K_{ads} and 1/T for carbon steel compound 2 in 1 M HCl solution at various temperatures.
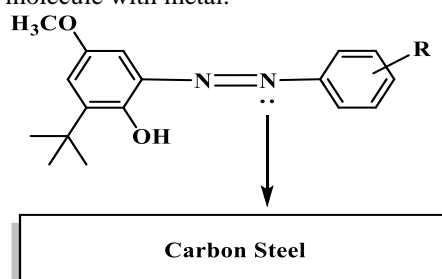
4.1. Mechanism of Inhibition

From the obtained electrochemical results, it was concluded that the azo compounds inhibit the corrosion of carbon steel in 1 M HCl by an

adsorption at the carbon steel solution interface. There is a general assumption to explain the adsorption of organic molecules that adsorb on the metal surface in three types, namely[31,32].
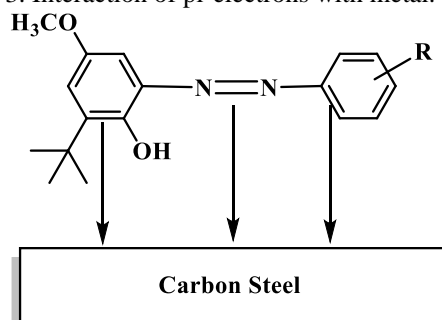
1. Electrostatic interaction between the charge of molecules and the charged metal.

2. Interaction of unshared electron pairs in the molecule with metal.

3. Interaction of pi-electrons with metal.

Conclusion

In summary, new two azo compounds were synthesized as effective corrosion inhibitors. These compounds were dissolved in 1 M HCl solution and adsorbed onto the carbon steel surface at varying concentrations and temperatures.

The best inhibitory efficiency (IE%) values were obtained at concentration 5×10^{-3} M and temperature 298 K. These azo compounds can be considered mixed inhibitors and followed the Langmuir adsorption isotherm. The results showed that inhibition efficiency increased with the concentrations, while decreased with increasing temperature.

As per the potentiodynamic polarized technique, the investigated compounds were identified to be mixed-type inhibitors. Thermodynamic studies confirmed the compounds (1 and 2) to be adsorptive inhibitors and their adsorption process was found to be spontaneous and follow Langmuir's adsorption isotherm. The values about the standard free energies of adsorption specify that the inhibitors get adsorbed to the carbon steel exposed to 1 M HCl via both physisorption and chemisorption processes, wherein chemisorption (mixed type adsorption) would be predominant.

Conflicts of interest

In accordance with our policy on Conflict of interest please ensure that a conflicts of interest statement is included in your manuscript here. Please note that this statement is required for all submitted manuscripts. If no conflicts exist, please state that "There are no conflicts to declare".

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