

Synthesis a New Heterocyclic Compound through Domino Reactions, Identification, and Study them as Corrosion Inhibitor for Carbon Steel in brine water

Mushtaq J. Meften, Adnan S. Abdulnabi

Abstract— This study comprises three parts; the first is the synthesis new heterocyclic compound through domino reactions, and this compound 2-oxa-4, 8,10-trithia-6,12,15-triaza-tricyclo [9.3.1] pentadeca-1 (15),11,13-triene. The second part, is the identification of this compound by several techniques such as, Infra- red spectrophotometer (FT - IR), ^1H and ^{13}C nuclear magnetic resonance spectrophotometer (NMR), Gas chromatography - mass spectrophotometer (GC-Mass), and Elemental analysis (CHNS). The last part is the study of the prepared compound as corrosion inhibitor for carbon steel alloy (N-80) in brine water as a corrosive medium, by several techniques such as potentiodynamic polarization (Tafel Plot), electrochemical impedance spectrophotometer (EIS), energy dispersive X-ray fluorescence spectrophotometer (EDX), and atomic force microscopy (AFM). Inhibition efficiency increases with increasing inhibitor concentrations to attain a maximum value of 88.89% .

Index Terms— Carbon steel alloy, EDX, Inhibitors, Heterocyclic compound, AFM, Electrochemical Impedance Spectroscopy (EIS), Brine water, Formaldehyde.

I. INTRODUCTION

Corrosion is degradation of materials properties due to chemical or electrochemical reaction with their environments. In general, metallic corrosion produces in its initial stage soluble metal ions in water, and then, the metal ions develop into solid corrosion precipitates such as metal oxide, and hydroxide [1], [2]. Corrosion generally may be divided into two types: wet corrosion, and dry corrosion. Dry corrosion where the corrosive environment is a dry gas. It is also frequently called chemical corrosion and the best-known example is high temperature corrosion where occurs at higher temperature (above the boiling point of water), and in the absence of an aqueous phase (dry corrosion)[3], [4]. Corrosion phenomenon in the petroleum industry occurs in a two media of water, and hydrocarbon, so that the presence of a thin layer of water leads to corrosion. Thus, rigorous elimination of water reduces the corrosion rate to a negligible

value [5]. L. Xiang-Hong, and X. Xiao-Guang studied the inhibition effect of two pyrimidine derivatives, 2- hydroxyl pyrimidine (HP), and 2-mercaptopyrimidine (MP), on the corrosion of carbon steel (CRS) in $1 - 5 \text{ mol}\cdot\text{L}^{-1}$ HCl investigated by weight loss, potentiodynamic polarization curves, and electrochemical impedance spectroscopy (EIS). The results show that HP and MP are both good inhibitors of corrosion of carbon steel in $1 \text{ mol}\cdot\text{L}^{-1}$ HCl solution. Inhibition efficiency increases with inhibitor concentration, and decreases with hydrochloric acid concentration [6].

The aim of this study, synthesis a new heterocyclic compound by domino reactions, then evaluate it as corrosion inhibitor for carbon steel alloy in brine water. Heterocyclic compound which prepared has N, S, and O atoms, thus formation a protective film on surface of carbon steel alloy. The basis of the synthesis, we used a multicomponent thiomethylation reaction of amine with formaldehyde, and H_2S gas [7], [8].

II. EXPERIMENTAL

INSTRUMENTS

In this was used ^1H and ^{13}C NMR spectrophotometer, model Bruker AM 500. Also was used GC- mass spectrophotometer model GCMS-QP2010 Ultra (Shimadzu) (Supelco 5ms capillary column 60000×0.25 ; carrier gas helium; oven temperature programming from 40°C to 150°C at a rate of $10 \text{ deg}/\text{min}$; injector temperature 260°C ; ion source temperature 200°C). Infrared spectrophotometer model FTIR-8400 (Shimadzu) in the $(400-4000) \text{ cm}^{-1}$. Energy dispersive X-ray Fluorescence Spectrometer (EDX) was used to obtain activity of these compounds as corrosion inhibitor model EDX-800HS (Shimadzu), the EDX tests were performed at Voltage $15 - 50 \text{ kv.}$, current $20 - 100 \text{ mv.}$, each experiment in the instrument needs to be from 10 to 15 minutes, and the measurements were carried out under vacuum pressure.

Atomic force micrograph (AFM), the surface morphology of carbon steel plates was investigated by (AFM) model Agilent 5500 AFM, USA. Imaging for this study was conducted in AC mode with PPP-NCL probes (Nanosensor, Switzerland). The resonance frequency and force constant of cantilever are 185 kHz , and 43 N/m , respectively. The samples were cleaned with distilled water, dried, and then used for AFM. Potentiodynamic polarization spectrophotometer, prepared compound evaluated as a corrosion inhibitor by using device from Gamry company model CH /300. Electrochemical impedance spectrophotometer prepared compound evaluated as a corrosion inhibitor by using device model PV70A. The elemental analysis for C, H, N, and S were carried out on elemental analyzer CHNS 628 from LECO Company.

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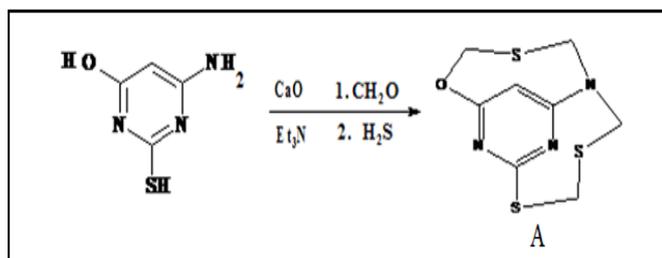
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Synthesis compound 2-oxa-4, 8, 10-trithia-6, 12, 15 - triaza - tricyclo [9.3.1] pentadeca-1 (15), 11, 13 - triene (A)

The mixture consisting of aqueous 10 % formaldehyde (12ml) (40mmol), 100 ml water, 4- amino -6- hydroxy -2-mercapto pyrimidine monohydrate (1.61 g) (10 mmol), 10 ml from calcium oxide solution at concentration 0.05 M (0.28g), and triethyl amine (1.4ml) (10 mmol), was stirred at temperature 0 °C with added bubbled hydrogen sulfide for 3 hours. The mixture was extracted with chloroform, organic layer neglected, and aqueous layer was neutralized by dilution HCl to produce yellow precipitate which was collected and dried. The percentage of purity compound and its yield were 77.12 %, and 88%, respectively. Its m.p. was 216-218°C. ¹H NMR (500 MHz, CDCl₃): δ =3.41 (s, 4H, S-CH₂-N); 3.92(s,2H, S-CH₂-S); 4.71(s, 2H, O-CH₂-S); 6.81(s,1H, C=CH). ¹³C NMR (500 MHz, CDCl₃): δ = 38.55 (S-CH₂-S); 57.76 (S-CH₂-N); 69.20 (O-CH₂-S); 121.40 (C=CH); 154.90 (N-C-N); 164.33(N=C-S); 166.03(N=C-O). MS, m / z (Irel (%)): 259 [M]⁺ (100); 213 [M-CH₂S]⁺ (86); 72 [CN₂S]⁺ (28); 126 [C₅H₄NSO]⁺ (6); 139 [C₄HN₃OS]⁺ (27); 80 [C₄H₄N₂]⁺ (52); 180 [MH-C₄H₄N₂]⁺ (54), 153 [C₅H₃N₃OS]⁺ (11); 109 [C₄HN₂S]⁺ (5). IR(cm⁻¹) 1240.86 C-N; 615.29 C-S; 2960.88 and 2930.15 C-H stretching aliphatic; 3040.54 C-H stretching aromatic; 1600.92 C=C; 1200.86 C-O; 1690.40 C=N. Elemental Analyses calcd. For C₈H₉N₃S₃O : C 37.06; H 3.47; N 16.21; S 37.06; Found C 37.08; H 3.33; N 16.15; S 36.92. Scheme (1) show the one pot reactions to 4-amino -6- hydroxy -2-mercapto pyrimidine monohydrate with formaldehyde, and H₂S gas.



-2- mercapto pyrimidine monohydrate with formaldehyde, and H₂S gas

III. RESULTS AND DISCUSSION

Study of compound (A) (2-oxa-4, 8, 10 - trithia-6, 12, 15 - triaza - tricyclo [9.3.1] pentadeca-1 (15), 11, 13 - triene) as corrosion inhibitor.

A. Potential dynamic Polarization Technique (Tafel Plot)

Corrosion rate for carbon steel alloy was measured in the brine water at room temperature, in the presence of inhibitor A with concentrations between (0.001 - 0.005) M by using potential dynamic polarization (Tafel plot). The corrosion parameters such as corrosion potential (E_{corr}), corrosion current density (I_{corr}), corrosion rate (C_R), and polarization resistance (R_p), were calculated from this technique. The percentage of inhibition efficiency was calculated by the following equation [9]:

$$\%IE = \frac{I_{corr}^0 - I_{corr}^i}{I_{corr}^0} \times 100 \dots \dots \dots (1)$$

Where:

I_{corr}^0 : Corrosion current density in the absence of inhibitor.

I_{corr}^i : Corrosion current density in the presence of inhibitor.

From table (1), and figure (1) show, tafel curve for corrosion of carbon steel alloy in brine water without inhibitor was compared with tafel curve in presence of inhibitor (A) in concentration (0.005 M). We observed a decrease in corrosion current density from (4.229 mA/cm²) in the presence brine water to (0.954 mA/cm²) with inhibitor, and a decrease in the rate of corrosion from (290.11 mpy) in the presence brine water to (37.03 mpy) with inhibitor, while polarization resistance with inhibitor was significantly higher than the polarization resistance in the inhibitor absence, growing from (187.81 Ω) in the presence of brine water to (377.92 Ω) with inhibitor. Whereas corrosion potential increased from (- 0.364 V) in the presence brine water to (- 0.340 V) with inhibitor. Therefore, positive shift occurred. Also note corrosion potential at concentration (0.002 M) decreased to (- 0.405 V), therefore negative shift occurred, it is clear that there was no definite trend in the shift of E_{corr} values (slightly shift). This result indicates that positive shift could be classified as mixed type of inhibitor for corrosion of carbon steel alloy, and could be classified as cathodic inhibitor at negative shift [10], [11]. Polarization resistance inversely proportional with the rate of corrosion, and corrosion current density [10], [12], reaching the highest efficiency of inhibition (77.44 %) in the concentration (0.005 M).

Table (1) The corrosion parameters for carbon steel alloy in different concentrations of inhibitor (A) in brine water

Inhibitor Concentration (M)	I_{corr} mA/cm ²	E_{corr} (V)	CR (mpy)	R_p (Ω)	%IE
Blank	4.229	-0.364	290.11	187.81	-----
0.001	1.444	-0.315	66.23	293.12	65.85
0.002	1.295	0.405	61.34	301.58	69.37
0.003	1.199	-0.353	56.95	321.41	71.64
0.004	1.101	-0.318	47.39	360.10	73.96
0.005	0.954	-0.340	37.03	377.92	77.44

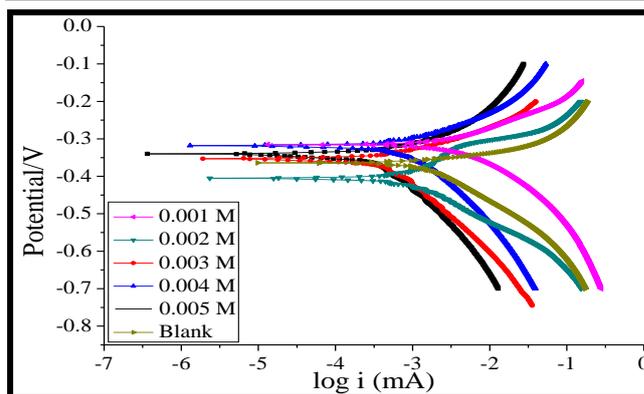


Figure (1) Tafel plots for carbon steel alloy in the different concentration of inhibitor (A) in brine water

B. Electrochemical Impedance Spectroscopy technique (EIS)

Corrosion rate for carbon steel is measured in the brine water as corrosive medium at room temperature, with using inhibitor A at concentrations between (0.001 - 0.005) M by

using electrochemical impedance spectroscopy (EIS). The impedance parameters, are namely, the maximum frequency (f_{max}), double layer capacitance (C_{dl}), and charge transfer resistance (R_{ct}) are calculated.

The percentage of inhibition efficiency (IE) is calculated using the following equation [13]:

$$\%IE = 1 - \left\{ \frac{R_{ct}^0}{R_{ct}^i} \right\} \times 100 \dots \dots \dots (2)$$

Where R_{ct}^0 , and R_{ct}^i , are the charge transfer resistance in absence, and presence of inhibitor respectively.

Also the area of surface metal covered by molecules of inhibitor (Surface Coverage θ) can be calculated from the equation [14]:

$$\theta = 1 - \left\{ \frac{R_{ct}^0}{R_{ct}^i} \right\} \dots \dots \dots (3)$$

The double layer capacitance C_{dl} values for different inhibitor concentrations can calculate by using the equation [15]:

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

Where f_{max} is the frequency at which the impedance is maximum, and charge transfer resistance R_{ct} .

From the results shown in table (2), and figure (2), Nyquist plot for corrosion of carbon steel alloy in brine water without inhibitor was compared with Nyquist plots in presence inhibitor (A) in concentration (0.005 M). An observed increase in charge transfer resistance (R_{ct}) was observed from (32.12 Ω) in the case of presence brine water to (133.20 Ω) with inhibitor, and decrease the value of double layer capacitance (C_{dl}) from (86.60 $\mu F/cm^2$) in blank to (40.74 $\mu F/cm^2$) with inhibitor. An increase (R_{ct}) is observed from (86.80 Ω) in concentration (0.001M) to (133.20 Ω) in (0.005 M), and a decrease in the (C_{dl}) from(48.06 $\mu F/cm^2$) in concentration (0.001M) to (40.74 $\mu F/cm^2$) in concentration (0.005M). As well, both values of surface coverage, and inhibition efficiency increase from (0.6300 to 0.7590), and from(% 63.00 to % 75.90),respectively when changing the concentration from (0.001 to 0.005)M . It is clear from changes in impedance parameters are indicative of the adsorption of this inhibitor on the carbon steel surface [16]. The adsorption on the metal surface decreases the double layer capacitance (C_{dl}) [17]. It is clear from interpretation of the results, by increasing the inhibitor concentration, the double layer capacitance (C_{dl}) values tend to decrease, and the inhibition efficiency increases. The increase in charge transfer resistance (R_{ct}) value is ascribed to the formation of protective film on the metal [16]. This film may be formed from oxide or oxide with adsorbed inhibitor, therefore, suggesting the compound formed protective film on the metal by adsorption that occurs through functional groups of molecules [18].

Table (2) Results of Nyquist plots for carbon steel alloy in different concentrations of inhibitor (A) in the brine water

Inhibitor Concentration (M)	R_{ct} (Ω)	C_{dl} ($\mu F/cm^2$)	f_{max} (Hz)	Surface Coverage (θ)	%IE
Blank	32.12	86.60	57.19	-----	-----
0.001	86.80	48.06	38.13	0.6300	63.00
0.002	90.20	47.66	37.01	0.6440	64.40
0.003	100.05	44.65	35.61	0.6790	67.90
0.004	112.20	43.05	32.93	0.7140	71.40
0.005	133.20	40.74	29.31	0.7590	75.90

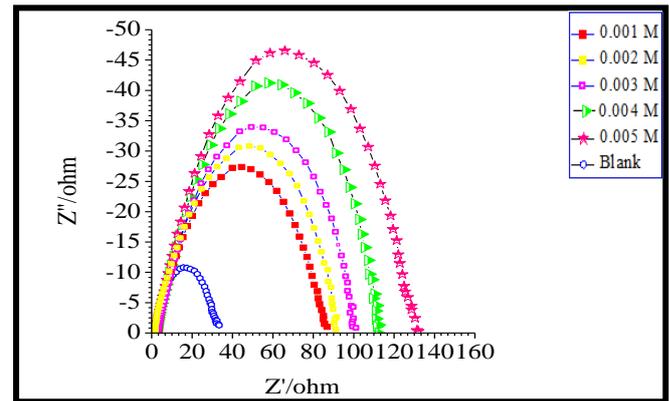


Figure (2) Nyquist plots for carbon steel alloy in the different concentration of inhibitor (A) in the brine water

C. Dispersive X-ray Fluorescence Spectroscopy technique (EDX)

The percentage of corroded metals for carbon steel alloy was measured in the brine water at 25 °C after immersion for (5hrs) in the absence, and presence of the inhibitor. The percentage of the fresh carbon steel alloy was also measured by using energy dispersive X-ray fluorescence spectrometer (EDX). Analysis in this technique is quantitative and qualitative at the same time. The X-ray parameters are the percentage of: all elements in the carbon steel alloy, all elements corroded, the percentage, and type of iron corrosion product. The percentage of corroded element (E loss %) was calculated by using the equation:

$$\%E\ loss = \frac{f - f^0}{f} \times 100 \dots \dots \dots (5)$$

Where, f and f^0 are the percentage of element in the fresh carbon steel, and percentage of element after corrosion, respectively, then the percentage of inhibition efficiency (IE %) was calculated by using equation:

$$\%IE = \frac{L^0 - L^i}{L^0} \times 100 \dots \dots \dots (6)$$

Where, L^0 and L^i are the percentage of corroded element without inhibitor, and with inhibitors, respectively.

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Table (3) the percentages of all elements in the carbon steel alloy before, and after corrosion determined by X-ray-fluorescence spectroscopy

Samples	% Fe	% O	% C	% Mn	% Si	% Cu	% Cr	% S
Fresh carbon Steel	98.227	-----	0.2	0.841	0.366	0.19	0.152	0.024
without inhibitor (Blank)	82.32	15.62	1.39	0.27	0.16	0.12	0.098	0.022
with inhibitor (A)	94.675	1.771	2.116	0.79	0.3	0.13	0.102	0.116

Using inhibitor (A) with concentration (0.005 M) was observed to decline the percentage of iron from 98.227% in the case of fresh carbon steel to 94.675 % in presence inhibitor (A), with height rates of both oxygen, and carbon to (1.771 %, and 2.116 %), respectively as shown in table (3). It can be concluded that the percentage of iron corroded in the event of inhibitor (A) is (3.552%), and hence the inhibitor (A) reduces the rate of corrosion by (12.355 %). Table (4) shows the percentage of corroded iron, and inhibition efficiency.

Table (4) The percentages of the corrosion of iron and inhibition efficiency in brine water

Sample	Percentage of Iron corroded (%)	Inhibition efficiency (%)
Brine water(Blank)	15.907	-----
Blank with 0.005 M inhibitor A	3.552	77.67

D. Atomic Force Microscopy (AFM)

In order to establish whether inhibition is due to the formation of a film on the carbon steel surface through adsorption, AFM were studied. Surface roughness analysis for carbon steel immersed for (5hrs) in the brine water (blank), and in the presence of the inhibitor at concentration 0.005M, and surface roughness analysis for polished carbon steel metal surface (reference) was studied by using atomic force microscopy. Surface roughness analysis by AFM was performed to obtain the average roughness Sa, the maximum peak Sp, the maximum depth Sv, and the maximum peak to depth Sz. calculated Sz by using equation:

$$S_z = (S_p + S_v) \dots \dots \dots (6)$$

The table (5) shows a summary of the Sa, Sp, Sv, and Sz for carbon steel in different environments.

Table (5) AFM parameters for surface of carbon steel in brine water with and without inhibitor

Samples	Values (µm)			
	Maximum peak Sp	Maximum depth Sv	Average roughness Sa	Maximum peak to depth Sz
Polished carbon steel (reference)	0.098	0.102	0.026	0.2
Carbon steel immersed in brine water (Blank)	0.919	0.795	0.119	1.714
Carbon steel immersed in brine water containing inhibitor A	0.717	0.533	0.0935	1.25

The presence of 0.005 M of inhibitor A in brine water leads to reduce the average roughness value from 0.1190 µm to 0.0935 µm when compared with blank. The maximum peak-to- depth Sz value decrease from 1.714 µm in brine water to 1.25 µm with inhibitor. These parameters confirm that the surface does not appear significantly smooth. But the smoothness of the surface is owing to the formation of a protective film. Also from parameters in table (5) the (Sa), and (Sz) values are greater than the atomic force microscope data of polished metal surface. Figure (3) shows the surface of carbon steel alloy is not homogeneous significantly with the presence of defects, and distortions on the carbon steel surface [19] - [22].

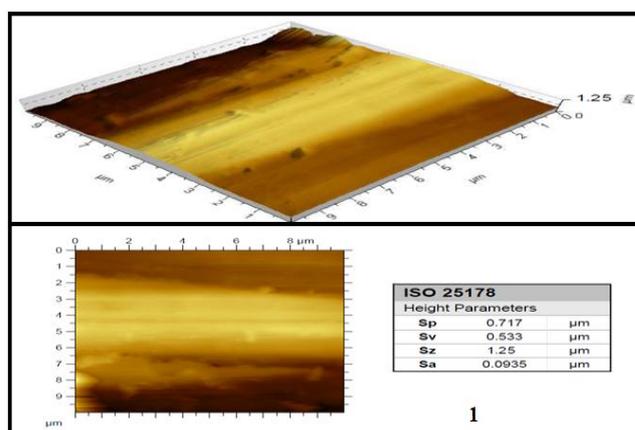


Figure (3) 2D, and 3D AFM images for carbon steel surface with inhibitor A (1)

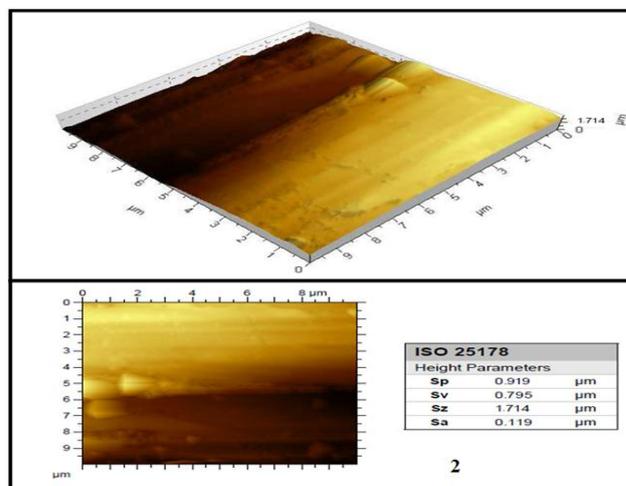


Figure (4) 2D, and 3D AFM images for carbon steel surface in brine water (Blank) (2)

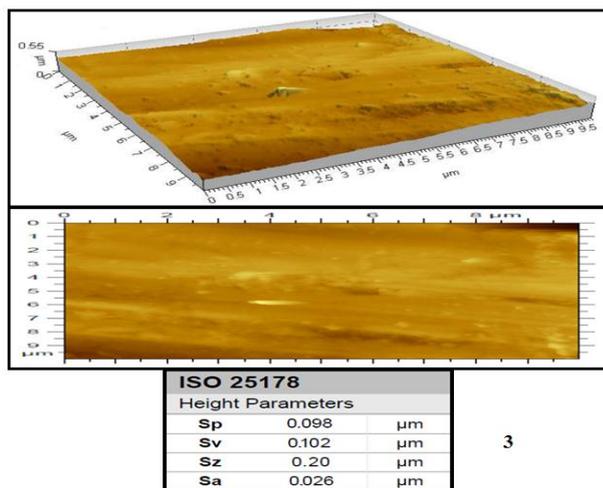


Figure (5) 2D, and 3D AFM images for Polished carbon steel surface (reference) (3)

IV. CONCLUSION

The compound was prepared by domino reaction is novel. The inhibition efficiency increases with increasing inhibitor concentrations and time to achieve a maximum value of 88.89%. Polarization results show that compound act as mixed-type inhibitor in brine water. Impedance results, indicates that compound adsorbed on carbon steel surface with increasing charge transfer resistance, and decreasing the double-layer capacitance. Adsorption process accrue on the carbon steel surface due to presence hetero atoms (oxygen, nitrogen and sulfur) in the molecule.

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