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# Synthesis, Characterization and Study of a New Heterocyclic Compound as Corrosion Inhibitor in 15%HCl Solution

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**ABSTRACT:** One-pot synthesis of 6,12,14-trithia-1,4,8,10-tetraaza-tricyclo  $[9.4.0.0^{3,9}]$  pentadeca-3(9),10-dien-2-one via domino reactions of 4,5- diamino -6- hydroxy -2-mercapto pyrimidine with formaldehyde and H<sub>2</sub>S gas. It characterized by Liquid chromatography- Mass spectroscopy (LC-MS), <sup>1</sup>H – NMR, and 13C-NMR spectroscopy, then the compound applied as corrosion inhibitor for carbon steel type C38 in 15% HCl as a corrosive medium, at 25°C by weight loss method, gasometrical method, electrochemical impedance spectroscopy (EIS), and X-ray diffraction (XRD) spectroscopy techniques. Maximum inhibition efficiency of heterocyclic compound to attain a value 92.33%.

**KEYWORDS:** Corrosion, Inhibitors, Heterocyclic compounds, Domino reactions, EIS, Weight loss method, XRD, and Gasometrical method.

### I. INTRODUCTION

Corrosion is the degradation of materials properties due to chemical or electrochemical reaction with environments [1]. Metallic corrosion is one of the problems often encountered in industrialized society, hence it has been studied widely since the industrial revolution in the late eighteenth century [2]. Most organic compounds contain heterocyclic atoms such as nitrogen, sulfur, oxygen, phosphor and multiple bonds in the molecules are adsorbed on the metal surface, are called heterocyclic compounds [3-4]. Modern domino reactions include the formation of O- atom heterocycles, N- atom heterocycles, S- atom heterocycles, and formation of heterocycles containing Nitrogen, Oxygen, Sulfur, and phosphor atoms together [5]. The adsorption process, inhibition mechanism, and inhibition efficiency depend on the electronic and structural properties of the inhibitor, the temperature and pressure of the reaction, the nature of the metallic surface, and the flow velocity of corrosive medium [6-7]. Organic inhibitors can be adsorb onto the surface metal by four distinct mechanisms are, (a) electrostatic attraction between the charged inhibitor molecule and the charged metallic surface, (b) interaction between the electron pairs in the inhibitor molecule and vacant d-orbital of metal surface atom, (c)  $\pi$  electron interaction with the metal, and (d) a combination of mechanism such as, (a,b,c) are occur tandem [8-11]. To avoid minimize the cost and damage, the inhibitors are used to secure degradation of materials. Used of inhibitors and their application, does not require any skill it only depends on adding of inhibitor in proper proportion [12-14]. Large numbers of the organic inhibitors used are, contain hetero atoms such as oxygen ,nitrogen ,sulfur, and multiple bonds in their molecules through which they are adsorbed on metal surface, and therefore are a good corrosion inhibitors [15]. Carbon steel (C-steel), also called plain carbon steel, means iron which contains carbon. The properties of carbon steel depend primarily on the amount of carbon it contains. C- steel is a type of steel having a maximum carbon content of 1.5%. Generally, with an increase in the carbon content from 0.01% to 1.5% in the alloy, its strength and hardness increases but still such an increase beyond 1.5% causes appreciable reduction in the ductility and malleability of the steel, and found several types of carbon steel are, C- steel SAE 1045, C- steel SAE 1035, C- steel IS226, C-steel C1018, C- steel APIX60, and C- steel C38[16-23].

In view of the continued interest on the application of inhibitors, for metal corrosion control, was the aim of this work is investigate and study the heterocyclic compound as inhibitor on the corrosion behavior of the carbon steel in 15% HCl solution by using several techniques.



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### **II. EXPERIMENTAL**

### 2.1. Materials preparation.

Carbon steel sheet (C38) of composition Mn 0.439%, C 0.179%, S 0.034%, Si 0.165%, Cu 0.203 %, and 98.98% Fe [21]. Each sheet cut into coupons 7cm, 2.5cm, 0.3cm for length, width, and thickness, respectively for weight loss study, 4cm, 2cm, 0.3cm for length, width, and thickness, respectively for hydrogen evolution study and 1.5cm, 0.5cm both diameter and thickness respectively for electrochemical study. The coupons were polished by silicon carbon papers SiC (120, 600 grit size) then cleaned by distilled water, absolute ethanol, acetone, and stored in a moisture-free desicator prior to use in corrosion studies. The corrosive medium was 15% HCl solution prepared from 37% analytical grade supplied by Sigma-Aldrich. All material were procured from Sigma –Aldrich and Merck company. General structure of heterocyclic compound used in this study, show in Figure 1.



Fig. 1: General structure of inhibitor 6,12,14-trithia-1,4,8,10-tetraaza-tricyclo [9.4.0.0<sup>3,9</sup>] pentadeca-3(9),10-dien-2-one.

### 2.2. Instruments

<sup>1</sup>H and <sup>13</sup>C NMR spectra where recorded on a Bruker AM 500 MHz spectroscopy, with CDCl<sub>3</sub> as solvent. LC-mass spectra recorded on QP900 at scan rate time 2.220 ->2.627 min. Electrochemical impedance analyzer (EIS) model 6310 was used record AC impedance measurements in a frequency range of  $10^{-1}$  Hz to $10^{4}$  Hz under potentiodynamic conditions, with amplitude of 10 mV peak-to-peak. The X-ray diffraction (XRD) analyzer were performed on a Philips PW-1840 with Cu K $\alpha$  radiation ( $\lambda = 1.5008$  Å) at a scanning speed of 0.05° /sec over the 2 $\theta$  range 20 –75°.

### 2.3. Determination of Corrosion rate.

#### 2.3.1. Weight Loss Method.

The weight loss experiments were carried out in water bath for 3 hours at 25 °C. Carbon steel was immersed once in aqueous solution of hydrochloric acid (15% HCl), and it once again with presence inhibitor in size beaker 250 ml, the samples were immersed in triplicate and average for weight loss, and corrosion rate was calculated. The corrosion rate was calculated using equation [24].

Corrosion rate (mpy) = 
$$\frac{534W}{DAT}$$
 ----- (1)

Where, W is weight loss in (mg); D is the density of carbon steel 7.86 g/cm<sup>3</sup>; A is the area of specimen in square inch (area is 40.7cm<sup>2</sup> = 6.3 inch<sup>2</sup>); T is exposure time in hrs; and mpy mils per year. The percentage of inhibition efficiency (%IE) was calculated the following equation [25].

% IE = 
$$\frac{CR_{blank} - CR_{inhi}}{CR_{blank}} \times 100 \quad \dots \quad (2)$$

Where,  $CR_{blank}$  corrosion rate in the absence of inhibitor, and  $CR_{inh}$  corrosion rate in the presence of inhibitor. Degree of surface coverage ( $\theta$ ), was calculated the following equation [26]

$$\theta = \frac{CR^{o} - CR^{i}}{CR^{o}} - - - - - (3)$$

Where, CR<sup>o</sup> and CR<sup>i</sup> the corrosion rates of the carbon steel in absence and presence of inhibitor, respectively.



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### 2.3.2. Gasometric Method.

In this method the resulted gas volume is calculated in the absence, and presence of inhibitor. From the volume of gas the resulting, was calculated inhibition efficiency (%IE), and degree of surface coverage ( $\theta$ ), using equations 4, and 5 respectively [27-29].

$$\% IE = \left(1 - \frac{V_{Ht}^{i}}{V_{Ht}^{o}}\right) X 100 - - - - (4),$$
  
$$\theta = \frac{\% IE}{100} = \left(1 - \frac{V_{Ht}^{i}}{V_{Ht}^{o}}\right) - - - - (5)$$

Where  $V_{Ht}^{i}$  is the volume of gas at time t in presence inhibitor and  $V_{Ht}^{o}$  is the volume of gas evolved at time t in absence inhibitor.

### 2.3.3. Electrochemical Impedance Spectroscopy (EIS).

The corrosion cell used at 25 °C, consist had three electrodes are, platinum as counter electrode (auxiliary electrode), Ag/AgCl as reference electrode, and carbon steel as working electrode. Working electrode surface, the exposed to aggressive solution in absence, and presence inhibitor is  $1 \text{ cm}^2$ . The percentage of inhibition efficiency (IE) is calculated using the following equation [30].

% IE = 
$$1 - \left(\frac{R_{ct}^{0}}{R_{ct}^{i}}\right) \times 100$$
 ----- (6)

Where  $R_{ct}^{o}$ , and  $R_{ct}^{i}$ , are the charge transfer resistance in absence, and presence of inhibitor respectively. The area of surface metal covered by molecules of inhibitor (Surface coverage  $\theta$ ) can be calculated from the equation (7) [31]. The double layer capacitance  $C_{dl}$  values for different concentrations of inhibitor can calculate by using the equation (8)[32].

$$\Theta = 1 - \left(\frac{R_{ct}^{0}}{R_{ct}^{1}}\right) - \dots - (7)$$

$$C_{dl} = \frac{1}{2\sqrt{7} f_{max} R_{ct}} - \dots - (8)$$

### **III. RESULTS AND DISCUSSION**

## 3.1. Synthesis of heterocyclic compound 6,12,14-trithia-1,4,8,10-tetraaza-tricyclo [9.4.0.0<sup>3,9</sup>] pentadeca-3(9),10-dien-2-one.

In the three – neck flask fitted with a stirrer blending mixture consisting of (20 mmol) 4,5- diamino -6hydroxy -2-mercapto pyrimidine (**I**), 50 ml water, 25 ml methanol, 5 ml calcium oxide solution at concentration 0.1M, and 5 mmol triethyl amine at temperature 0°C in ice bath for 4 hours, then added 80 mmol aqueous solution 35 % formaldehyde as a dropwise to the mixture. After that added 80 mmol bubbled hydrogen sulfide for 1.5 hour. The mixture was extracted with chloroform, aqueous layer neglected, and organic layer was dried by MgSO<sub>4</sub> for 5 hours, then evaporated to produce oily orange. Its yield were 65 %. Scheme (1) shows the mechanism for domino reactions of 4,5 diamino -6- hydroxy -2-mercapto pyrimidine (**I**) for produce inhibitor 6,12,14-trithia-1,4,8,10tetraaza-tricyclo [9.4.0.0<sup>3,9</sup>] pentadeca-3(9),10-dien-2-one (**II**).



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### 3.2 Characterization of Heterocyclic Compound

The synthesized heterocyclic compound were characterized using <sup>1</sup>H,<sup>13</sup>C-NMR, and LC mass spectroscopy techniques. The spectra of heterocyclic compound are shown in Figures from 2 to 5 respectively, and the pertinent details of all spectra are as follows: <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$  ppm : 4.4 (s, 2H, S-CH<sub>2</sub>-S); 5.1 (s, 2H, N-CH<sub>2</sub>-S); 3.0-3.2 (d, 4H, NH-CH<sub>2</sub>-S); 9.3 (br. s, 2H, NH); 7.3 (s,CDCl<sub>3</sub>); 0.007 (s,TMS). <sup>13</sup>C- NMR (CDCl<sub>3</sub>)  $\delta$  ppm: 33.9 (1C, S-CH<sub>2</sub>-S); 50.67 (1C, N-CH<sub>2</sub>-S); 85.75 (1C, N-C-NH); 170.5 (C=O); 160.4 (N-C=N); 87.30 (2C, NH-CH<sub>2</sub>-S); 51.32 (1C, NH-C-C=O); and triplet signal at shift 76.67 - 77.51 (CDCl<sub>3</sub>). MS, m / z (Irel,%) : 274 [M] <sup>+</sup> (100); 260 [M-CH<sub>2</sub>] <sup>+</sup> (19); 246 [M-C<sub>2</sub>H<sub>4</sub>] <sup>+</sup> (38); 230 [M-C<sub>3</sub>H<sub>8</sub>]<sup>+</sup> (19); 195 [M-CH<sub>3</sub>S<sub>2</sub>]<sup>+</sup> (48); 185 [C<sub>6</sub>H<sub>9</sub>N<sub>4</sub>OS] <sup>+</sup> (20); 183 [M-SCH<sub>2</sub>SCH] <sup>+</sup> (54); 98 [C<sub>5</sub>H <sub>10</sub>N<sub>2</sub>]<sup>+</sup> (45); 95[C<sub>4</sub>H<sub>3</sub>N<sub>2</sub>O] <sup>+</sup> (35); 90 [C<sub>2</sub>H<sub>6</sub>N<sub>2</sub>S] <sup>+</sup> (17); 60 [C<sub>2</sub>H<sub>4</sub>S] <sup>+</sup> (38) [33-35].



Fig. 2: <sup>1</sup>H-NMR Spectrum of inhibitor.



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Fig. 4: LC Spectrum of inhibitor.



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Fig. 5: Mass spectrum of inhibitor.

## 3.3. Application .3.3.1. Weight Loss Measurements.

Corrosion rate of carbon steel in 15% HCl solution at 25 °C in absence and presence of inhibitor was studied using weight loss method, and the data obtained after 3 hours of immersion have been evaluated and values are given in the Table 1. It can be concluded from these results that the decrease of both weight loss, and corrosion rate when increasing concentration of inhibitor, and increase of surface coverage ( $\theta$ ), this may be due to the adsorption of inhibitor onto the carbon steel surface. Corrosion rate decreased from 2.74 mpy in blank to 0.21 mpy in 500ppm, and therefore increase inhibition efficiency to attain a value 92.33 [36-37].

Table 1.	<b>Corrosion parameter</b>	s of carbon steel in absen	ce and presence of the inhibitor
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Inhibitor concentration	Weight loss (mg)	Surface coverage	Corrosion rate (mpy)	Inhibition efficiency IE%
15% HCl Blank	0.763		2.74	
100 ppm	0.339	0.5583	1.21	55.83
200 ppm	0.246	0.6788	0.88	67.88
300 ppm	0.103	0.8650	0.37	86.50
400 ppm	0.092	0.8795	0.33	87.95
500 ppm	0.061	0.9233	0.21	92.33



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### 3.3. 2. Gasometric Measurements.

The inhibition efficiency of heterocyclic compound on corrosion of carbon steel (C38) in 15% HCl solution was investigated at 25 °C using gasometric method. The results obtained from this method are presented in Table 2, and Figure 6, it was noted from these results that the volume of hydrogen gas evolved increased with increase in the period of immersion in 15% HCl, indicating that the increasing rate of corrosion of carbon steel in acidic solution, but volume of hydrogen gas evolved decreasing with increase the concentration of inhibitor at constant time. It was also observed that the volume of hydrogen gas evolved by the blank (15% HCl) was greater than the volume of hydrogen gas evolved by solutions containing various concentrations of inhibitor as shows in Figure 6, indicating that the inhibitor inhibited the corrosion of carbon steel in acidic solution. The volume of hydrogen gas evolved, is directly proportional with exposure duration in the corrosive medium (approximately, linearly with time) as shows in Figure 6 [ 38-39].

Inhibitor Con.	Volume of hydrogen evolved (mL)					Inhibition efficiency (IE%)						
	5min.	10min	15min	20min	25min	30min	5min.	10min	15min	20min	25min	30min
15% HCl	1.85	3.31	4.36	5.21	6.21	7.15						
100 ppm	1.34	1.67	1.98	2.45	2.98	3.12	27.6	49.6	54.6	53.0	52.1	56.4
200 ppm	1.11	1.23	1.45	1.79	1.99	2.16	40.0	62.9	66.8	65.7	68.0	69.8
300 ppm	0.89	0.99	1.23	1.44	1.72	1.89	51.9	70.1	71.8	72.4	72.4	73.6
400 ppm	0.74	0.85	1.12	1.32	1.41	1.52	60.0	74.4	74.4	74.7	77.3	78.8
500 ppm	0.61	0.84	0.93	0.99	1.19	1.26	67.1	74.7	78.7	81.0	80.9	82.4

Table 2: Hydrogen evolution rate, inhibition efficiency of carbon steel in with and without inhibitor



Fig. 6: Variation of volume of H<sub>2</sub> evolved with time for carbon steel corrosion in the absence and presence of inhibitor at 25°C

### 3.3.3. Electrochemical Impedance Spectroscopy (AC Impedance).

In order to determination of formation of protective film on the metal surface, electrochemical impedance were studied. When a protective film is formed on the metal surface, charge transfer resistance (Rct) increases, and double layer capacitance value (Cdl) decrease [40-41]. The adsorption on the carbon steel surface decreases the double layer capacitance  $C_{dl}$  [42]. It is clear from changes in AC- impedance parameters occurrence of adsorption of inhibitor on the carbon steel



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surface as shown in Table 3 [17]. The Figures, **7a,7b**, and **7c** shows the AC impedance spectra (Nyquist plots), AC impedance Bode- phase angle plots, and AC impedance Bode plots respectively of carbon steel immersed in acidic solution 15% HCl in the absence and presence of inhibitor. The decrease in  $C_{dl}$  values can be attributed (i) to a decrease in local dielectric constant (ii) increase in the thickness of the electrical double layer (iii) a combination of (i) and (ii) together [43-45], which suggests the substitution of H<sub>2</sub>O molecules with inhibitor molecules leading to a protective film on the carbon steel surface [30]. The increase in charge transfer resistance  $R_{ct}$  values are ascribed to the formation of protective film from inhibitor moleculars on the metal surface, and adsorption of inhibitor molecules on metal surface causing the decrease in  $C_{dl}$  values [17, 46]. It is observed that in the presence of inhibitor at 500 ppm,  $R_{ct}$  value increases from 31.41 ohm cm<sup>2</sup> in 15% HCl to 240.11 ohm cm<sup>2</sup> with inhibitor, and  $C_{dl}$  value decreases from 51.66 µF /cm<sup>2</sup> to 25.95 µF /cm<sup>2</sup>, these observations suggest that the formation of protective film adsorbed on the metal surface, as shows in Table 3 [47- 49].

The diameter of the capacitive loop in the presence of inhibitor is larger than in the absence of inhibitor (blank solution), and increases with the inhibitor concentration, this suggests that the impedance of inhibitor increases with the inhibitor concentration. Furthermore, some figures show that there is a little difference at diameter of the capacitive loop, but at another concentrations the difference will be large as shows in Figure **7a**. Such behavior may be considered that the physisorption at a little difference, and chemisorption at a large difference. [50]. Depression of phase angle, at relaxation frequency with decreasing the inhibitor concentration. Such a phenomenon could be attributed to higher corrosion activity at low concentrations of inhibitor. In the Bode- phase angle, the maximum phase angle shifts to lower frequency after inhibitor addition, indicating an increase in inhibition and time constant of the system. AC impedance Bode plots increase with increasing inhibitor as shown in figure **7c**. The presence of low frequency may be attributed to the relaxation process obtained by the adsorption, like Cl<sup>-</sup>, H<sup>+</sup> ions on the carbon steel surface [8, 42, 51-52].





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Table 3. Corrosion parameters of carbon steel immersed in 15% HCl solution in the absence and presence of inhibitor obtained from AC impedance .

Inhibitor		Bode plot				
concentration	f <sub>enae</sub> (Hz)	<b>C</b> <sub>cte</sub> (μF/cm <sup>2</sup> )	$(\Omega \text{ cm}^2)$	%IE	Surface coverage	Impedance value $(\Omega \text{ cm}^2)$
15% HCl Blank	98.12	51.66	31.41			39.89
100 ppm	79.03	25.15	80.10	60.80	0.6080	106.71
200 ppm	66.87	26.78	88.91	64.70	0.6470	175.51
300 ppm	43.91	32.95	110.04	71.50	0.7150	187.51
400 ppm	32.11	38.79	127.83	75.50	0.7550	236.41
500 ppm	25.55	25.95	240.11	87.00	0.8700	308.10

### 3.3.4. X-ray Diffraction Spectroscopy (XRD).

X-ray diffraction is one of the most powerful techniques for surface analysis, and this technique is very important to the analysis of corrosion product , and determining the action of corrosion inhibitors, as it is the only analysis method that readily provides information about the phase-composition of solid materials as a corrosion products. An investigation was undertaken to study the X-ray diffractometry for the identification and characterization of corrosion products that formed on carbon steel surface in absence and presence of inhibitor. In the presence of oxygen, metallic iron is oxidized to form iron hydroxides Fe(OH)<sub>2</sub> and Fe(OH)<sub>3</sub> then iron hydroxides change gradually to iron oxides, hematite Fe<sub>2</sub>O<sub>3</sub> and magnetite Fe<sub>3</sub>O<sub>4</sub>, resulting in the formation of a passive film on the carbon steel, it is very thin it may breakdown in the presence of aggressive ions [53-54]. The corrosion products without inhibitor is shown in Figure 8a, the peak at  $2\theta = 35.5^{\circ}$  suggested the presence iron oxide (hematite Fe<sub>2</sub>O<sub>3</sub>). The peaks at  $2\theta = 59.5^{\circ}$ , and  $71.1^{\circ}$  suggested the presence siderite FeCO<sub>3</sub>. Figure 8b shows the X-ray diffraction pattern of carbon steel immersed in the test solution containing 500ppm from inhibitor. Peaks at  $2\theta = 44^{\circ}$  and  $66.0^{\circ}$  in figure 8b back to the iron ,and absence both of oxides of iron Fe<sub>3</sub>O<sub>4</sub>, FeO(OH) and siderite FeCO<sub>3</sub>, with traces of hematite Fe<sub>2</sub>O<sub>3</sub> (small amount) at  $2\theta = 35.5^{\circ}$ , this is indicate the formation of adsorbed protective film on the surface of metal in the presence of inhibitor, finally this is indicate high inhibition efficiency for heterocyclic compound [29, 55-58].



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Fig. 8: (a)XRD spectrum of carbon steel after corrosion in15%HCl solution, (b) XRD spectrum after corrosion with inhibitor.



Fig. 9: Shows 20 degree in XRD spectrum

### **IV. CONCLUSIONS**

This study investigated the properties of inhibition heterocyclic compound on carbon steel type C38 in 15% HCl. From this investigation can be concluded are, impedance results indicate that compound adsorbs on carbon steel surface by formation of a dense and protective film with increasing transfer resistance and decreasing of the double-layer capacitance, and also indicate adsorption mechanism for inhibitor are a well dependent on chemisorptions, and physical adsorption. Weight loss, gasometric, and impedance studies reveals that the concentration of 500 ppm offers best efficiency to carbon steel immersed in 15% HCl solution, and the analyses of the results showed that the inhibition efficiency increased with increase in concentration of the inhibitor. X-ray diffraction results, show that products of



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corrosion are, hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>), goethite  $\alpha$ -FeO(OH), magnetite (Fe<sub>3</sub>O<sub>4</sub>), and siderite (FeCO<sub>3</sub>) in the absence of inhibitor, but in presence inhibitor, products of corrosion, are small amount from hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>)only.

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