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The Effect of MethyleCarbazodithoate" Corrosion inhibitor on Corrosion Behaviour of Low Carbon Steels in Acid Solution at Different Temperatures

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Abstract The purpose of this study is to investigate the effect of an organic compound inhibitor, namely "Methyl Carbazodithoate ", on the corrosion of low carbon steel (mild steels) in 2.0 M Sulphuric acid (H₂SO₄) solution by weight loss measurements and over the temperature range 30 - 50 °C. The effects of organic compound inhibitor concentration on the corrosion rate of low carbon steel have been found to increase with inhibitor concentration (7.5x10⁻⁴ - 4.0x10⁻³) M at both temperature 30 °C and 50 °C. A detailed analysis of the experimental data was performed to determine the best models of isothermal equilibrium data sets for the used inhibitor. The results were fit to all used adsorption isotherms including; Langmuir isotherm and Temkin isotherm, while a slight improvements were observed when using an organic inhibitor at temperature of 50 °C. The standard free energies of adsorption ΔG_{ads} are calculated. Finally, the inhibition efficiencies of "Methyl Carbazodithoate" obtained from the all various measurements were in good agreement.

Keywords: Corrosion, Inhibition, mild Steels, Methyl Carbazodithoate, Sulphuric acid.

دراسة تأثير المركب العضوي "المثبط ميثايلكربوزوديت" على تـآكل الفولاذ الصلب منخفض الكربون في الأوساط الحمضية وعند درجات حرارة مختلفة محمد سليمان¹ و *محمد الكيلاني² و طه عبد الله² و مسعودة علي² محمد سليمان¹ و *محمد الكيلاني² و طه عبد الله² و مسعودة علي² ¹ قسم الهندسة الكيميائية- كلية هندسة الطاقة والتعدين- جامعة سبها، ليبيا ² قسم هندسة المواد و التأكل- كلية هندسة الطاقة والتعدين- جامعة سبها، ليبيا ² قسم هندسة المواد و التأكل- كلية هندسة الطاقة والتعدين- جامعة سبها، ليبيا المراسلة: moh.ibrahim@sebhau.edu.ly المنص الهـدف من هذا المشروع هو دراسة تأثير المركب العضوي "المثبط ميثابلكربوزوديت" على تـآكل الفولاذ الصلب منخفض الكربون في الأوساط الحمضية. في هذه الدراسة استعملت طريقة "قدان الونن" في درجتي حرارة قالمثبط (30 و 20 °) درجة مئوبة.

الكربون في الأوساط الحمضية. في هذه الدراسة استعملت طريقة "ققدان الوزن" في درجتي حرارة المثبط (30 و 50 °) درجة مئوية. بدراسة تأثير زيادة تركيز المثبط تبين أن معدل التآكل يقل نسبيا. لدراسة مدى امتزاز المثبط على سطح عينات الفولاذ الصلب، تم استخدام عدة معاملات امتزاز عند ثبوت درجة الحرارة. وقد أشارت النتائج أن امتزاز المثبط على سطح الفولاذ يتم بصورة جيدة، مع ملاحظة أنه في درجة حرارة المثبط 50 درجة مئوية يكون الامتزاز أكثر كفاءة مقرنة بدرجة الحرارة 30 درجة مئوية. تم أيضا دراسة ظاهرة الامتزاز الفيزيائي، وكانت النتائج سالبة مما يدل على أن امتزاز المثبط على سطح القولاذ يتم بصورة جيدة، مع ملاحظة أنه أن المركب العضوي المستعمل في هذه الدراسة يكون فعالا في مقاومة تآكل الفولاذ الصلب منخفض الكربون في الأوساط الحمضية. الكلمات المفتاحية: التآكل، التثبيط، الفولاذ منخفض الكربون، ميثيل كارباز وديثوات، حاصل منخفض الكربون في الأوساط الحمضية.

1. Introduction

Organic corrosion inhibitors are an attractive field of research, due to their usefulness in various industries. Organic molecules can form a barrier through adsorption on the metal surface to reduce the corrosion of metal in acidic solution [1].

Acid solutions are generally used for the removal of rust and scale in several industrial processes. Sulphuric acid is often used as a pickling acid for steel and its alloys [2]. Mild steel is employed widely in most industries due to its low cost, availability and ease of fabrication of various reaction vessels such as cooling tower tanks and pipelines, etc. [3]. Inhibitors are [4] substances which retard the cathodic and/or the anodic processes. Moreover, inhibitors can function in one or more ways to control corrosion; by adsorption of a thin film onto the surface of a corroding material, by inducing the formation of a thick corrosion product, or by resulting in reduced aggressiveness. Inhibitors are generally used in these processes to control metal dissolution. Acid inhibitors are essentially used in metal finishing industries, acidizing of oil wells, cleaning of boilers and heat exchangers [5]. Corrosion of metals is a serious environmental problem that has been given adequate attention in the oil and gas industries because, during industrial processes such as acid cleaning and etching, metal surfaces are often made to come in contact with acidic medium, indicating that the use of inhibitors is necessary [6]. Although there are numerous options for controlling the corrosion of metals, the use of inhibitors is one of the best methods for protecting metals against corrosion. Steel is the most important engineering and construction material in the world. It is used in

changing the characteristic of the environment

every aspect of our lives (automotive manufactures, construction products, steel toecaps, protective footwear, refrigerators, washing machines, cargo ships and so on) [7].

Oxidation occurs at anodic site and reduction occurs at cathodic site. In acidic medium, hydrogen evolution reaction predominates. Corrosion inhibitors reduce or prevent these reactions; they are adsorbed onto the metal surface and act by a forming barrier to oxygen and moisture, and some of the inhibitors facilitate the formation of a passive film on the metal surface. Figure (1) illustrates hydrogen grooving in sulfuric acid piping system.

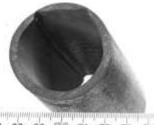


Figure 1 - Hydrogen Grooving in Sulfuric Acid of Piping System [12].

Several works have studied the influence of organic compounds containing nitrogen on the corrosion of steel in acidic media [8-10]. Most organic inhibitors act by adsorption on the metal surfaces [4]. The effect of surfactants has been studied and it was shown that the protection efficiency increases with number of carbon atoms in the molecule [11]. an important tool Thermodynamic model is to study the mechanism of the inhibitor on the corrosion of metal. A thermodynamic model for the adsorption process has been suggested [10-11]. In the present work, the protection effect of sulphur containing organic compounds on low carbon steel surface in aggressive 2.0 M H₂SO₄ solution is presented.

Experimental Work Steel Materials preparation

The commercial low carbon steel rods were kindly provided by Musirata Steel Factory. The steel were selected as a test samples for corrosion studies. The chemical composition data for the low carbon steel rods were provided by the manufacture, which are listed in the table (1).

Table 1 - Chemical compositions ofLowCarbon Steel Used in this Work

С	Si	Mn	Р	S	Cr	Ni	Cu	Al
0.32%	0.75%	0.014%	0.004%	0.014%	0.002%	0.001%	0.001%	0.002%

The provided steel rods were cut into cylinder shapes using a diamond wheel cutter. The dimensions of samples were taken as 40 mm length "and 10 mm diameter. After the specimens were cut, they were cleaned and polished using emery papers with different grades "60, 100, 120, 180, 220, 320, 400, and 1200 grade ". These activities

onthen dried. The prepared samples were then
weighted using electronic balance with accuracy
about 0.0001 g. The cleaned specimens were kept
in desiccators in order to protect them against any
weather variables, until using them in the
experiment.ve2.2. Laboratory Preparation of "Methyl
Carbazodithioate"
The organic compound under investigation was

The organic compound under investigation was prepared by reaction 1:1 carbon disulphide and diamine in presence of potassium hydroxide "KOH". The obtained potassium carbazodithioate was reacted with idomethane to produce the final product of methyle carbazodithioate which was extracted by eather, filtered and dried from the solvent.

were done in order to get a smooth specimen

surface, free of rust, scale or dust. Following the

polishing procedure, the samples were washed

using methanol, acetone and distilled water and

All used chemical are in reagent grade. Freshly distillated deionized water was used in all preparations. Organic compound as an inhibitor in "2 M sulphuric acid (H₂SO₄) medium was prepared in dimethylformanide " D. M. F". All tested solutions contained "10 volume percent of "D. M. F" to maintain complete solubility.

The inhibitor solution used in this work was prepared with different concentrations, a nine different concentration samples in 2.0 M H₂SO₄. The concentration of the first inhibitor sample was carried out as 7.50×10^{-4} M and the concentration of the last sample was 4.00×10^{-3} M for both inhibitor solution temperature 30 and 50 °C. Figure 2 shows the formula structure of "Methyl Carbazodithioate" organic inhibitor [13].

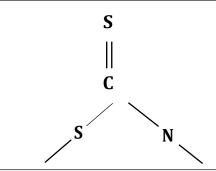


Figure 2 - Formula Structure of "Methyl Carbazodithioate " Organic Inhibitor [13].

2.3. Corrosion Measurements

The corrosion rates for inhibited and a noninhibited steel samples were measured using a weight loss method. The weight loss measurements were carried out at 30 and 50 °C. The loss in weight per area in mg/cm² (W_t), the corrosion rate (R_{corr}), and the percentage of protection efficiency percent protection IE%, were calculated over different inhibitor concentrations according to the following equations:

$$W_t = \frac{W_o - W_i}{A} \tag{1}$$

Where W_o is the original weight (mg) and W_i the weight after immersion in the test electrolyte, t the immersion time (min), and R'_{corr} and R_{corr} are the corrosion rates with and without an inhibitor, respectively.

3. Results and Discussion

3.1. Corrosion Rate and Protection efficiency

The data for area of specimens and inhibitor concentration of the research are presented in table 2. Moreover, the data for weight loss and corrosion rate calculations are given table 3.

Table 2 - Area of Specimens and InhibitorConcentration of the Research

S. No.	S. Area cm²	Expos. Time min	(I) Inhibitor Concentration in 2.0 M H_2SO_4
1	7.58	90	Blank
2	7.94	90	7.50×10^{-4}
3	8.40	90	1.00×10^{-3}
4	7.90	90	1.25×10^{-3}
5	7.64	90	1.75×10^{-3}
6	7.67	90	2.00×10^{-3}
7	8.32	90	2.50×10^{-3}
8	8.12	90	3.00×10^{-3}
9	8.11	90	3.50×10^{-3}
10	8.12	90	4.00×10^{-3}

 Table 3 - Weight Loss and Corrosion Rate

 Calculations.

S. No.	W _t mg 30°C	W _t mg 50 °C	$ CR mg cm^2 \min_{x \ 1^{-3}} 30^{\circ}C $	$\frac{CR}{mg}}{cm^{2}\min}_{x 1^{-3}}$ 50°C
1	80.40	217.6	0.1178	0.3190
2	54.20	165.2	0.0758	0.2312
3	54.30	154.3	0.0718	0.2041
4	56.96	143.9	0.0801	0.2024
5	42.20	132.9	0.0614	0.1933
6	29.60	106.4	0.0429	0.1541
7	19.60	83.7	0.0262	0.1118
8	17.80	71.1	0.0244	0.0973
9	16.80	67.1	0.0230	0.0919
10	15.70	59.6	0.0215	0.0815

According to the data in previous table it is clear that the corrosion rate decreases gradually with increasing the inhibitor concentration at a temperature 30 and 50 °C. It seen that the sulphur containing compound inhibits the corrosion of low carbon steels in 2.0 M H_2SO_4 at all concentrations. It was observed that the corrosion inhibition progressively with increase increases in concentration of the added inhibitor as shown on table (3). Maximum inhibition efficiency of the compound at 30 °C was 80.47 % and 72.61 % which was achieved at concentration 4.00×10^{-3} M, as shown on table (4). According to the results a "Methyl Carbazodithioate" inhibitor has given an

acceptable level of inhibition efficiency against low carbons steel corrosion in acid solution, at both of inhibitor temperatures.

Table	4 -	Inhibitor	Efficiency	Calculations	at
tempe	ratu	res 30 - 5	o°C.		

S. No.	log (I)	IE % 30 °C	IЕ % 50 °С
1	0.00	00.00	00.00
2	- 3.125	32.59	24.08
3	- 3.000	32.46	29.09
4	- 2.903	29.15	33.87
5	- 2.757	47.51	38.92
6	- 2.699	63.18	51.10
7	- 2.602	75.62	61.53
8	- 2.523	77.86	67.33
9	- 2.456	79.10	69.16
10	- 2.398	80.47	72.61

As shown on figure 3, the increasing in inhibitor concentration causes decreasing in corrosion rate in the both temperatures, this indicates that the presence of "Methyle carbazodioate" compound retards the corrosion rate of low carbon steels in 2.0 M H₂SO₄. The ability degree and of inhibition depends on the concentration of the sulphur containing inhibitor compound.

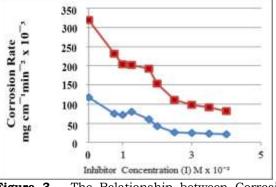


Figure 3 – The Relationship between Corrosion Rate and Inhibitor Concentration

For most chemical reactions, the reaction rate with increasing temperature. increases Temperature affects the corrosion rate of metals in electrolytes primary through its effect on factors which control the diffusion rate of oxygen. The corrosion of iron and steel is an example of this because temperature affects the corrosion rate by virtue of its effect on the oxygen solubility and oxygen diffusion coefficient. As temperature increases, the diffusion coefficient of oxygen also increases which tends to increase the corrosion rate. However as temperature is increased, oxygen solubility in aqueous solutions decreases until at the boiling point all oxygen is removed; this factor tends to decrease the corrosion rate [14]. According to the results, the net effect of low carbon steels is that the corrosion rate in acid solution approximately doubles when the temperature rises from 30 to 50°C. Even though chemical adsorption is an exothermic process, it does not occur slowly at lower temperature due to high kinetic energy barrier. Hence, like most chemical changes, the

extent of chemisorptions increases with increase in temperature up to certain limit and then after that it starts decreasing. As shown on figure 4. (a) and (b), the resulted curve at both temperatures 30 and 50 °C respectively range have characteristics of S-shape adsorption isotherm indicative of adsorption mechanism for inhibition process [14].

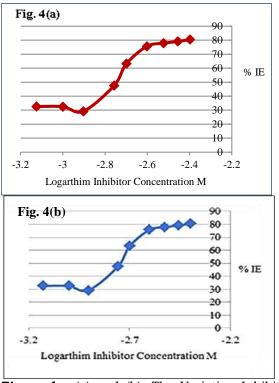


Figure 4 - (a) and (b), The Variation Inhibition Percentage With the Logarithm of the Concentration of "Methyl Carbazodithioate" Sulphur Containing Inhibitor Compound.

3.2. Adsorption Isotherm 3.2.1. Introduction

In general, adsorption is governed by a number of forces such as covalent bonding, electrostatic attraction, hydrogen bonding and non-polar interactions between the adsorbed species, lateral associative interaction, solvation and desolvation [15].

The relationship between inhibition efficiency and the bulk concentration of the inhibitor at constant temperature, which is known as isotherm, gives an insight into the adsorption process. Several adsorption isotherms were attempted to fit surface coverage values to classical isotherms of Langmuir, Temkin, Frumkin, Flory-Huggins. It is generally assumed that the adsorption of the inhibitors on the metal surface is essential step in the inhibition mechanism [16].

3.2.2. Lungmir Adsorption Isotherm

A correlation between coverage (θ) defined by (IE %) and the concentration of inhibitor (I) in electrolyte can be represented by the Langmuir adsorption isotherm [17]:

$$\Theta/(1 - \Theta) = K.[I]$$
(4)

the relationship between surface coverage and the inhibitor concentration. Large K values mean better and stronger interaction between the inhibitor molecules and the metal surface. Table 5 and table 6 provide estimated inhibitor surface coverage and adsorption energy at temperatures 30 and 50 °C. According to the obtained K values, it is concluded that the interaction between the inhibitor molecules and the metal was improved [17].

Where, K is the adsorption constant, it represents

Table 5 - Estimated Inhibitor Surface Coverage	
and Adsorption Energy at Temperature of 30 °C.	

$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$
2 0.3259 644.667 - 26.24 3 0.3246 480.600 - 25.50
3 0.3246 480.600 - 25.50
4 0.2015 320.120 - 24.57
T 0.4910 549.120 - 24.07
5 0.4751 517.200 - 25.69
6 0.6318 857.950 - 26.95
7 0.7562 1260.668 - 27.92
8 0.7786 1172.323 - 27.73
9 0.7910 1081.343 - 27.53
10 0.8047 1030.075 - 27.41

Table 6 - Estimated Inhibitor Surface Coverage and Adsorption Energy at Temperature of 50 $^\circ C.$

S. No.	Surface overage (θ) IE /100	Adsorption Constant (K) M^{-1} $\left(\frac{\theta}{1-\theta}\right) / I$	Adsorption Energy (ΔG _{ads}) KJ mol
1	0.0000	000.000	00.00
2	0.2408	353.000	- 26.54
3	0.2909	359.000	- 26.58
4	0.3387	296.000	- 26.07
5	0.3892	308.000	- 26.18
6	0.5110	104.000	- 23.26
7	0.6153	547.000	- 27.72
8	0.6733	649.000	- 28.17
9	0.6916	568.000	- 27.82
10	0.7261	337.000	- 26.42

The value of the correlation " R² " is used to determine the best fit isotherm which was obtained for Langmuir. If the value of " R² " is very close to unity, that is indicated the strong adherence to Langmuir adsorption isotherm. Figure 5 (a) and (b) shows a plot of $(\theta / 1-\theta)$ versus the inhibitor concentration (I) yield a straight line with correlation value $\ensuremath{\mathbb{R}}^2$ ". The plot obeys Langmuir adsorption isotherm as the plot has linearity and good correlation coefficient. According to results The " R² " value in case of temperature 50 is "0.9509 ", and for a temperature 30 is "0.9148". Both of the values are not very close to unity, but the " R² " of 50 is higher than that of 30 indicating that the inhibitor adsorption at 50°C has a adherence to Langmuir relatively stronger adsorption isotherm.

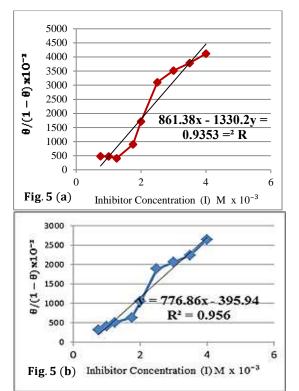


Figure 5 - (a) and (b)Langmuir AdsorptionIsotherm of "MethylCarbozodithoate"Inhibitor on LowCarbonSteel in 2.0 M H2SO4for 30 and 50 °C.

The value of adsorption constant " K " at each inhibitor concentration were calculated for a two temperatures as shown on tables 5 (a) and (b). The obtained values of adsorption constant were used to determine the Gibb's free adsorption energy (ΔG_{ads}) using the following equation [18]:

 $K=1/55.5 e^{-\Delta G_{ads}/RT}$ (5)

where R is the universal constant "8.314 J mol⁻¹ $K^{{\scriptscriptstyle -1}{\scriptscriptstyle "}}~$ and " T " is the absolute temperature of " 30 and 50 °C " and the constant value of " 55.5 " is the concentration of water in solution in mol L⁻¹. According to the obtained values of " ΔG_{ads} ", it can be observed that all values using both which temperatures have negative signs, thermodynamically indicate that the spontaneous adsorption of inhibitor on the surface of low carbon steel (indicates the occurrence of exothermic process). The negative value of also suggest the strong interaction of inhibitor molecules with the low carbon steel surface.

3.2.3 Temkin Adsorption Isotherm

The Temkin model of isotherm adsorption contains a factor which explicitly takes into account the interactions of ions of the aqueous solution and the membrane (adsorbent - adsorbate). The surface coverage values for "Methyle carbozodioate" were fitted into the Temkin adsorption isotherm model, which has the form [19]:

 $Exp(-2a\theta) = K \cdot I$ (6)

Thus, in figure 6 (a) and (b) the Temkin isotherm was represented by a linear plot " θ " versus the logarithm of inhibitor concentration "log I". According to Temkin equation (6), the obtained values of the correlation "R2" for inhibitor temperatures 30 and 50 were (0.8941) and (0.9614) respectively. As " R² " for inhibitor temperature where the value is more close to unity, this indicate that it has a strongest adherence to Temkin adsorption isotherm comparing with the same inhibitor applied at temperature of 30 °C.

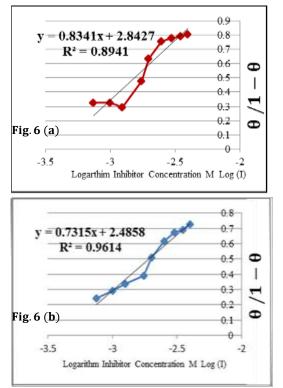


Figure 6 - (a) and (b) Temkin Adsorption Isotherm for "Methyle carbozodithoate" Inhibitor on Low Carbon Steel in "2.0 M H_2 SO₄" at 30 and 50 °C.

3.2.4. Freundlich Adsorption Isotherm

The Freundlich model of isotherm adsorption has been chosen fourthly to evaluate adsorption potential of the adsorbent and adsorbed solution. The Freundlich isotherm is given by [19]:

$$Log \theta = \log K_{ads} + n \log I$$
 (7)

Where K_{ads} is the adsorption equilibrium constant, n is the interaction parameter and I is the inhibitor concentration.

The plot of $(\log \theta)$ vs. $(\log I)$ is typical of Freundlich adsorption isotherm at 30 and 50°C. As shown on figure 7 (a) and (b) a perfectly linear plot was obtained with regression constant (R²) (0.8523) at inhibitor temperature 30°C and (0.9546) at temperature of 50 °C. According to the results of "R²", it is clear that the adsorption of inhibitor is stronger at temperature of 50°C comparing with that of 30°C.

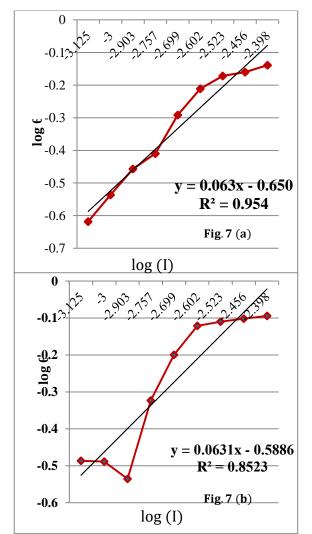


Figure 7 - (a) and (b) Freundlish Adsorption Isotherm for "Methylecarbozodithoate" Inhibitor on Low Carbon Steel in "2.0 M H₂ SO₄" at 30 and 50 $^{\circ}$ C.

3.3. Adsorption Mechanism

In 2.0 M H_2SO_4 at a temperature 30 and 50 °C, the "methyl carbazodithoate" inhibit the corrosion by controlling both the anodic and cathodic reactions. In H_2SO_4 solution this compound exists as protonated species. These protonated species may be absorbed on the cathodic sites of the low carbon steel and decreases the evaluation of hydrogen. The better inhibition efficiency attributed to the difference of adsorption of SO₄ ions. The low adsorption of SO₄ ions on the metal surface permits more space for adsorption of inhibitor molecules and enhance inhibition of corrosion [20].

4. Conclusions

In the present work, the effect of an organic compound inhibitor, "Methyl Carbazodithoate", on the corrosion of low carbon steel in 2.0 M Sulphuric acid (H_2SO_4) solution was investigated over the temperature range of 30 and 50 °C. The main findings in this work are:

1. Inhibition efficiency increases with increase of inhibitor concentration at temperatures 30 and 50 $^\circ$ C.

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- isotherms models.
 3. ΔG_{ads} values are slightly less negative than -40 kJ/mol, indicating that the adsorption of inhibitor molecules obeying physical and chemical adsorption.
- 4. The overall conclusion is that the inhibition of corrosion using "methyl carbazodithoate" will be more effective at temperature of 50 °C than at temperature of 30 °C.

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