

Short report of mild steel corrosion in 0.5 M H₂SO₄ by 4-ethyl-1-(4-oxo-4-phenylbutanoyl)thiosemicarbazide

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KEYWORDS	ABSTRACT
Thiosemicarbazide Langmuir Inhibition efficiency Corrosion inhibitor H ₂ SO ₄	Thiosemicarbazide derivative namely 4-ethyl-1-(4-oxo-4-phenylbutanoyl)thiosemicarbazide which is indicated as EOPT was synthesized and assessed as an inhibitor for the corrosion mild steel (MS) in 0.5 M sulfuric acid, utilizing mass loss measurements. The inhibitive efficacy was found to be dependent on the concentration of EOPT and immersion time, the most significant inhibitive efficacy values 88.7% by gravimetric techniques were reported in the presence of an optimized concentration of EOPT (500 ppm) at 303 K. The adsorption of EOPT molecules on the surface of MS has been explored to understand the mechanism for corrosion prevention, the outcomes showed that the process of adsorption follows Langmuir isotherm, the determined free energy ΔG_{ads}^o value was found to be $-36.86 \ kJ \ mol^{-1}$ which signifies that EOPT molecules are absorbed by through both chemisorption and physisorption mechanism on the MS surface.

1.0 INTRODUCTION

Acidic environments were utilized for the chemical purification, descaling, and pickling process of various alloy compositions and materials in manufacturing (Al-Amiery et al., 2020)(Dhafer S. Zinad et al., 2020)(Al-Taweel et al., 2020)(Al-Amiery, et al., 2020)(Sheet et al., 2020)(Al-Amiery, Salman, et al., 2020)(Al-Amiery, 2021). The MS pickling process is a fundamental technical level to eliminate metal-oxides produced through the annealing process (Salman et al., 2020)(Al-Baghdadi et al., 2020)(Salim et al., 2020)(Hanoon et al., 2020) (Abdul Mutalib 2020) (Yamin et al., 2020)(Jawad et al., 2020)(Zinad et al., 2020)(Jawad, et al., 2020). Using corrosion inhibitors through the pickling MS process appears to be higher and more

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important because there is an extreme and unwanted acidic attack (Al-Baghdadi et al., 2021)(Kadhim et al., 2021)(Al-Baghdadi et al., 2021)(Alamiery et al., 2021), which is considered as one of the common effective techniques to protect the alloy surface from corrosion (Shaker et al., 2021) (Eltmimi et al., 2021) (Pauzi et al. 2020) (Hanoon et al., 2021). Corresponded to other reported technologies on the protection of corrosion, utilizing corrosion inhibitors is an efficient and encouraging approach with many benefits of no specific material needed, non-expensive and simple technique. Several inhibitors have been discovered in the past decade. Organic molecules with electronegative negative atoms including phosphorous, sulfur, oxygen and nitrogen, as well as pi bonds in their molecular structures are effective corrosive inhibitor compounds (Alamiery et al., 2021)(Kadhim et al., 2021)(Resen et al., 2021) (Sulaiman et al. 2021). The corrosion inhibitory characteristics of the organic molecules are generally due to physisorption and/or chemisorption resulting from the inhibitor molecules' active sites interactions with on metallic surface (Al-Amiery & Shaker, 2020)(Salman et al., 2020)(Salman et al., 2019). To improve the efficiently of synthesized inhibitor, herein, the present study is to examine the MS corrosion inhibition in the sulfuric acid environment by utilizing gravimetric techniques. The use of 4-ethyl-1-(4- oxo-4-phenylbutanoyl)thiosemicarbazid as corrosion inhibitor was due to containing two oxygen atoms, a sulfur atom, and especially three nitrogen atoms to reduce corrosion attack on mild steel has been investigated in some detail. While the inhibitor concentration effect and immersion time was studied in particular. Moreover, the inhibition adsorption isotherms of EOPT particles on examined metallic surface, and the free adsorption energy, were also determined, and it was noticed that the adsorption of EOPT particles on metallic surface was followed both chemisorption and physisorption inhibition mechanism.

2.0 EXPERIMENTAL PART

2.1 Material Preparation

Mild steel samples were used for this investigation and were regularly cut into small pieces with dimensions $4.5 \times 2.5 \times 0.2$ cm³. Analytical grade sulfuric acid was utilized to prepare the corrosive solution of sulfuric acid (0.5 M). The chemical structure of the tested inhibitor is presented in Figure 1.



Figure 1: The chemical stricter of 4-ethyl-1-(4-oxo-4-phenylbutanoyl)thiosemicarbazide (EOPT).

2.2 Weight Loss Measurements

All the tests were conducted in 250 ml of 0.5 M H_2SO_4 environment and open to air, at 303 K with different EOPT concentrations for the immersion periods 1, 5, 10, and 24 h. The coupons were sandpaper polished, washed with distilled water, dried in oven and weighed accurately on an electronic balance. In gravimetric techniques the coupons were exposed to the sulfuric acid environment with addition and without addition of a EOPT at various concentrations with the solution Temperature 303K for the above immersions periods (1, 5, 10, and 24 h) (Salman et al., 2019)(Jawad et al., 2019). After exposure, the analysis samples were rinsed with water then

acetone and dried in the oven, and finally reweighed. The weight loss was evaluated as the variations in coupon weight before and after exposure to the acidic environment.

From the gravimetric measurements, the rate of corrosion (C_R), inhibitive efficacy (IE%) and surface coverage (θ) were determined by the relations (1), (2), and (3) respectively (Al-Majedy et al., 2019)(Salman et al., 2019)(Ahmed et al., 2018).

$$C_R = \frac{m_1 - m_2}{at} \tag{1}$$

$$IE(\%) = \frac{C_{Ro} - C_{Ri}}{C_{Ro}} \times 100$$
(2)

$$\theta = \frac{C_{Ro} - C_{Ri}}{C_{Ro}} \times 100 \tag{3}$$

where m_1 and m_2 represent the coupons masses before and after corrosion, *a* is refers to the exposed coupon area, t is the exposure time, C_{Ro} and C_{Ri} are the rates of corrosion without and with the addition of EOPT respectively.

3.0 RESULTS AND DISCUSSION

3.1 Gravimetric Measurements

From gravimetric analysis, the identical inhibitive efficacy of EOPT for coupon surface in 0.5 M H₂SO₄ at 303 K is demonstrated in Figure 2. From Figure 2, it can be recognized that the inhibitive efficacy increases with the inhibitor concentration increasing, when the inhibitor concentration increases to 500 ppm, the inhibitive efficacy varies lightly with the further addition of the EOPT concentration. The C_R reduces with the EOPT concentration increasing. The MS corrosion inhibition is imputed to the adsorption of EOPT particles on the coupon surface. These molecules have heteroatoms such as nitrogen, oxygen, and sulfur and have the pi-bonds. The experimental findings reveal that the inhibitory activity of EOPT increase to 500 ppm, the inhibition efficiency is 88.7 %, which further demonstrates that the tested inhibitor may acts as an active corrosion inhibitor for the metallic surface in $0.5 \text{ M H}_2\text{SO}_4$ environment. The effect of exposure times was also investigated. The experimental results found that the inhibitive efficacy increases with increasing immersions time starting from 1 to 5 hours, and is no significant changes from 5 to 10 hours of exposure period, on the other hand the inhibitive efficacy decreases after 24 hours (Habeeb et al., 2018)(Al-Baghdadi et al., 2018). The values of corrosion rate reduce from 20.77 mmy⁻¹ to 6.91 mmy⁻¹ on the increase of 100 ppm to 500 ppm of EOPT. The increased inhibitory activity and decrease corrosion rate may owing to the increased adsorption of EOPT particles at the surface of MS with increasing EOPT concentration (Abdullah et al., 2018)(Al-Azawi et al., 2018). The variety of inhibitory activity with various immersions time from 1 h to 24 h at various inhibitor concentrations is demonstrated in Figure 2.



Figure 2: Difference of corrosion rate and inhibitive efficacy in $0.5 \text{ M H}_2\text{SO}_4$ on the surface of MS with various exposure times.

The inhibitory activity increased from 55.8% to 88.7% when the immersions time increases from 1 h to 5 hours. These findings are attributed to the reduced the rate of corrosion of coupon surface with exposure period and the increasing the number of absorbed inhibitor molecules by the surface. EOPT exhibited significant inhibitive performance for the corrosive of the surface of coupon in H_2SO_4 environment (Jamil et al., 2018)(Kadhim et al., 2017)(Kadhum et al., 2013).

3.2 Adsorption Isotherms

The adsorption isotherms can explain and studied the interactions of the inhibitor molecules with metallic surfaces. Adsorption performance of inhibitor molecules can be explained utilizing two interaction classes: the first one is physical adsorption (Physisorption is the physical bonding

between EOPT particles and the coupon surface that occurs due to Van der Waals forces) and chemical adsorption (Chemisorption is an adsorption type which involves a chemical reaction between the EOPT particles and coupon surface and also involves generated new chemical bonds) (Jamil et al., 2018) (Kadhim et al., 2017) (Kadhum et al., 2013). The process of adsorption depends on the molecular structures of the investigated molecules, the Temperature of the solution, charges, and metal characteristics. The adsorption of organic molecules is recognized as a process of quasi-substitution between the organic molecules [Org_(sol)] and water correlated with the metal surface [H₂O_(ads)] according to Equation (4) (Al-Azawi et al., 2016) (Obayes et al., 2017):

$$Org_{(sol)} + xH_2O \to Org_{(ads)} + xH_2O_{(sol)}$$
⁽⁴⁾

where *x* is the water molecules number.

To achieve the objective adsorption isotherm for tested inhibibitor molecules on steel surface, several adsorption models such as Langmuir and Temkin types are utilized. In current investigation, the Langmuir model is exhibited according to Equation (5) (Al-Amiery et al., 2014):

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \tag{5}$$

where C_{inh} represents the EOPT concentration and K_{ads} refers to the constant of the adsorption. According to Equations (3) and (5), the plots of C_{inh}/θ aginst C_{inh} give the straight line as presented in Figure 3. Figure 3. The coefficient of linear regression relationship (*R2 is almost equal to unity*) recommends that the adsorption of EOPT on surface of tested coupon in 0.5 M H₂SO₄ obeys the Langmuir isotherms.

On the other hand, the *K*ads, associated to ΔG_{ads}^o , value may be determined from the intercept of the straight line of the utilizing the following relation, Equation (6)

$$\Delta G_{ads}^o = -RT \ln \left(55.5 \, K_{ads} \right) \tag{7}$$

where *R* refers to constant of gas, and *T* represents the solution temperature (K) whereas, the value 55.5 refers to water concentration (molar).



Figure 3: Langmuir isotherms plot of EOPT molecules which adsorbed on the surface metallic coupon corrosive environment.

Generally, the ΔG_{ads}^{o} value around $-20 kJ mol^{-1}$ or less negative is correlated to physisorption, and the ΔG_{ads}^{o} around $-40 kJ mol^{-1}$ or more negative is related to chemisorption (Junaedi et al., 2013)(Al-Amiery et al., 2014)(Junaedi et al., 2012)(Al-Amiery et al., 2014)(Junaedi et al., 2012)(Al-Amiery et al., 2014)(Junaedi et al., 2013). According to equation (5) the value of ΔG_{ads}^{o} for the tested inhibitor is equal to $-36.86 kJ mol^{-1}$. The Δ Gads0 for tested inhibitor between $-20 kJ mol^{-1}$ and $-40 kJ mol^{-1}$, it is proposing that the adsorption of tested EOPT particles on coupon surface isn't chemisorption or physisorption only, but involves that a the two type of interactions, physisorption and chemisorption.

3.3 Suggested Mechanism

EOPT is adsorbed on a mild steel surface via a chemical reaction and physical interactions. The unpaired electrons (on the N, S, and O atoms) are transferred from inhibitor molecules (EOPT) to the vacant d-orbital of iron atoms at the mild steel surface and formed coordination bonds. Physical interactions were done because of the benzene ring and the functional groups in the inhibitor molecule which is a charged molecule that interact physically with a metal surface. Figure 4, represent the suggested mechanism of inhibition through chemisorption and physical interactions.



Figure 4: The suggested inhibition mechanism of EOPT molecules.

4.0 CONCLUSIONS

In conclusion, the organic compound namely 4-ethyl-1-(4-oxo-4-phenylbutanoyl)thiosemicarbazide (EOPT) was studied as corrosive inhibitor for MS in H_2SO_4 environment. With rising EOPT concentration, inhibitive effectiveness increases. The rate of corrosion decreases with an increase in EOPT concentration. EOPT molecules are adsorbed on the MS surface in accordance with the isothermal model for Langmuir adsorption. The adsorption of EOPT molecules on the surface of MS involving both chemisorption, and physisorption.

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