

## CORROSION INHIBITION OF CARBON STEEL ALLOY IN HYDROCHLORIC ACID SOLUTIONS USING THIOUREA DERIVATIVES

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### ABSTRACT

Corrosion is the most serious problem that opposes alloys and metals, and it leads to many industrial issues, such as the deterioration of structures and equipment and corrosion in factory tanks and pipelines in petroleum refineries, which shortens the lifespan and reduces the efficiency of such systems. Inhibitors are therefore applied to corrosive media via effective ways in order to improve the corrosion inhibition of metals and alloys. In the present work, the present work prepared a compound N-((2-aminoethyl) carbamothioyl)benzamide by green chemistry procedure then evaluate the prepared compound as corrosion inhibitor by electrochemical methodologies Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic Polarisation (PP) techniques and found that the prepared inhibitor has the adsorption ability over the carbon steel (CS) and it has the double layer capacitance ( $C_{dl}$ ) value was often less than in its absence, owing to the synthesized inhibitor's effective adsorption. So the prepared compound N-((2-aminoethyl) carbamothioyl)benzamide has binary effect first prepared by green chemistry and the second one is the prepared compound has good efficiency as corrosion inhibitor so make long life time for the surface of Carbon Steel Alloy.

**Key words:** green chemistry; thiourea derivatives; corrosion inhibitor; EIS; potentiodynamic polarization.

### INTRODUCTION

Corrosion, which is the degradation of a substance brought on by its contact with its environment, can happen at any stage or time throughout the production of petroleum and natural gas. Although any type of material can be covered by this

description, metallic alloys are usually the only ones allowed (Speight, 2014). Corrosion is a problem that cannot be completely eradicated, only partially prevented. Monitoring the corrosion process can be done in many different ways to slow down the deterioration of metal. One of the best ways to reduce corrosion is to use corrosion inhibitors (Pakiet *et al.*, 2020). Corrosion inhibitors are substances that, when given in very small quantities to corrosive conditions, slow down the pace at which metals corrode. The concentration of the inhibitor is the major factor affecting its efficiency. Its impact is caused by the kinetics and mechanism of reactions of the inhibitor on the surface in the corrosive medium (Pletnev, 2013; Kaczerewska *et al.*, 2017; Hart, 2016). Thiourea derivatives show a good effect as anticorrosion effect (Kamal *et al.*, 2022a).

The extent to which inhibitor molecules cover the metal surface affects the effectiveness of inhibitor adsorption often. Up until a specific concentration limit, it is generally believed that the inhibitor's protective effect will decrease with falling concentration and vice versa. The electrochemical method known as electrochemical impedance spectroscopy (EIS) measures a system's impedance in relation to the frequency of an AC voltage. It is fairly well-liked for a variety of reasons (Atrens *et al.*, 2017). The ability of EIS to separate the effects of various components, including the contribution of electron transfer resistance, double layer capacity, etc., is one explanation (Benavente, 2005). Another is that electrochemical impedance spectroscopy is highly surface sensitive, making numerous changes observable that other methods miss, such as changes in polymer layers caused by swelling, surface changes caused by protein adsorption, or corrosion protective layer penetration. Because molecules may be recognised without a redox active marker, electrochemical impedance spectroscopy is intriguing for analytical electrochemistry (Arya and Joseph, 2021). Through the passage of a current through the electrolyte, a process known as potentiodynamic polarisation allows the potential of the electrode to be changed at a predetermined pace. Utilizing this method, specimens are

polarised for corrosion testing. A metal sample of interest and an inert electrode are connected by a current during potentiodynamic polarisation, an electrochemical research technique, to change the test sample's electrochemical potential (V). Then, the current density that passed for a certain potential is noted (Turcotte and Little 2016; Baran *et al.*, 2019; Abboud *et al.*, 2009). The potential at which there is no current is known as the open circuit potential, also known as the equilibrium potential, the rest potential, or the corrosion potential; investigations based on the measurement of the open circuit potential are known as potentiometric experiments (Choudhary *et al.*, 2016). The voltage between the working (WE) and reference (RE) electrodes is the electrochemical cell's OCP. When measuring open circuit potential, you need a voltmeter with a high input impedance, similar to an electrometer, to measure the voltage without applying any current or voltage to the cell (Usubelli *et al.*, 2020). In the present work the prepared compound based on thiourea derivatives is prepared and evaluate the efficiency as corrosion inhibitor by the previous techniques.

## MATERIALS AND METHODS

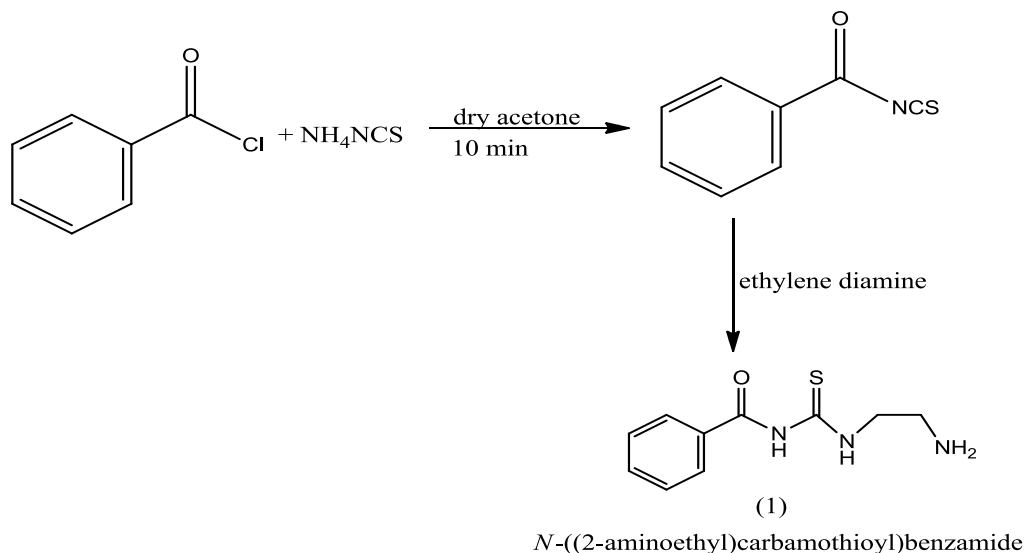
### Methodology of synthesis

All chemicals were used without further purification unless mentioned in the procedure and were purchased from Sigma-Aldrich.

Preparation of N-((2-aminoethyl) carbamothioyl) benzamide (1)

The materials [compound 1] was prepared In dry acetone we react benzoyl chloride (10 mmole, 1.4 gm) with ammonium thiocyanate (10 mmole, 0.76 gm) stirred for 10 minutes, the ammonium chloride product was filtered to remove it from the reaction medium then we add ethylene diamine, to the reaction medium with stirring for 20 minutes to obtain the products , in addition, we evaporate the acetone to obtain an yellow oily product (1) with yield 92%, the structure was

proved with IR (KBr,  $\nu$  / $\text{cm}^{-1}$ ): 3342.2, 3354.2, 3331.20  $\text{cm}^{-1}$  ( $\nu\text{NH}$ ); 1682.42  $\text{cm}^{-1}$  ( $\nu\text{C=O}$ ). (Abd-elsatar *et al.*, 2021; Guo *et al.*, 2010; Abd-elsatar *et al.*, 2022)



Scheme (1)

**Specimens:** The ingredients of CS samples (wt%) are: 0.014% Ni, 0.19% C, 0.003% Ti, 0.022% Cu, 0.016% V, 0.009% Cr, 0.01% Si, 0.94% Mn, 0.009% P, 0.034% Al, 0.004% S, 0.034% Fe and the remainder Fe. Emery papers in varying grades (600–2000) were used to abrasion them and they were then washed in  $\text{CH}_3\text{COCH}_3$  & purified  $\text{H}_2\text{O}$ . (Kamal *et al.*, 2022a; Kamal *et al.*, 2022b)

**Electrochemical methodologies:** Electrochemical impedance spectroscopy (EIS) and potentiodynamic polarisation (PP) techniques were used with a Potentiostat/Galvanostat Origaflix to monitor the electrochemical behaviour of a carbon steel (CS) alloy in 1 M HCl. The PP and EIS tests for CS were run under static conditions and at room temperature after 30 minutes of submersion in the testing solution. A minor alternating voltage perturbation (10 mV) at open circuit potential was applied to the exposed working electrode surface area in the frequency range of 100 kHz to 30 mHz. The electrode potential was then polarized by first going to -900 mV in the cathodic direction and subsequently to -200 mV in the

anodic direction at a rate of 1 mV/s scan. Saturated calomel electrode (SCE) is utilized as a reference electrode.

### **Scanning Electron Microscope (SEM)**

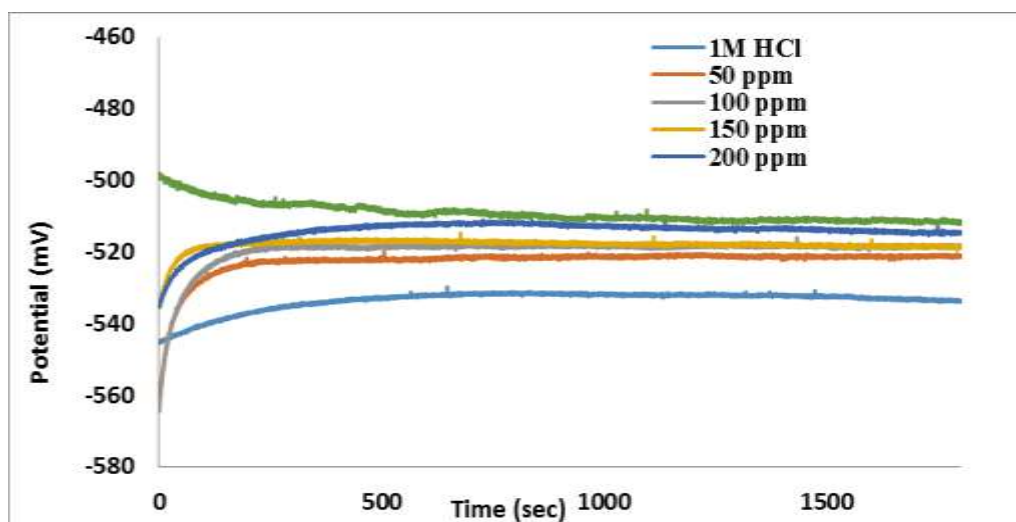
(JEOL JSM-53000, Japan) was the model of the used instrument for the surface inspection. The beam energy of acceleration used was 30 kV. At a magnification power (X 750), all the surface morphologies were provided.

## RESULTS AND DISCUSSION

**Chemistry discussion:** The prepared compound has at least 2 NH as well as CO and CS as minted in scheme (1) due to reaction of benzoyl chloride with ammonium thiocyanate in dry acetone the we remove the ammoniochloride as a byproduct after that we add ethylene diamine to form the minted compound with stirring, this method considered as green chemistry synthesis, furthermore, we proved the structure using IR which show presence of NH and CO at  $\nu$  3342.2, 3354.2, 3331.20, 1682.42  $\text{cm}^{-1}$  respectively, this is a new application for this compound, we expected to show a good corrosion inhibitor effect (Kamal et al., 2022a).

### Open Circuit Potential Assessment (OCP)

Fig. (1) depicts the changes of C- steel's OCP in the 1M HCl with time. Two remarkable features can be observed in this Figure. First, the electrode potential shifts over time towards more negative values at the time of immersion for the free and the inhibited solutions. Such behavior suggests that the pre-immersion air-formed oxide film has to be resolved before achieving the steady state. In another word, the anodic processes are more prevalent in contrast to cathodic processes. Therefore, oxide film formation's rate is ergo lesser than that of dissolution. Secondly, with inhibiting solution, the inhibitors give more positive OCP than the free 1M HCl. Moreover, through rising inhibitors concentrations, the steady-state electrode's potential is shifted towards more positive values. Ergo, the inhibitors' inhibition extraordinary effect can thus be due to inhibitors constituents' adsorption on C-steel surface, creating the inhibitors shielding layer on the C-steel electrode surface (Han et al., 2018a).



**Fig. (1):** potential-time after immersion in Steel in 1.0 M HCl in absence and presence of different concentrations of compounds at 25°C.

**Potentiodynamic polarization (PP) results:**

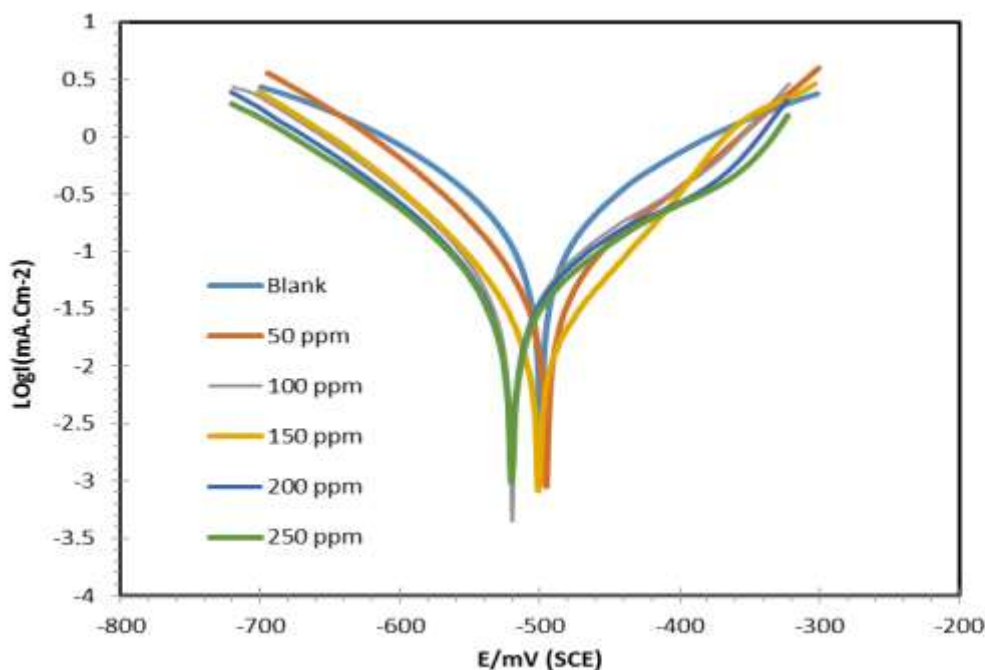
Fig. (2) depicts a PP plot of CS in 1M HCl before and after applying various concentrations of the compound Electrochemical variables like corrosion current density ( $i_{corr}$ ), cathodic & anodic slopes ( $\beta_c$  &  $\beta_a$  respectively), corrosion potential ( $E_{corr}$ ) & inhibition performance are tabulated in **Table (1)** Inhibition efficiency ( $\eta_p$ ) is calculated from formula (1) (Hart, 2016; Baran et al., 2019):

$$\eta_p = \frac{i_{corr} - i_{corr}(inh)}{i_{corr}} \times 100 \quad (1)$$

where  $i_{corr}$  &  $i_{corr}(inh)$  are the uninhibited and inhibited corrosion current densities, respectively.

Fig. (2) apparently, after applying the three inhibitors to HCl solution, both cathodic and anodic reactions of CS electrode are hindered. The data in **Tables (1)** reveal that the existence of inhibitor in HCl solution induces a slight shift in  $E_{corr}$  toward the -ve direction relative to the absence of inhibitors. The difference in  $E_{corr}$  in the absence and presence of inhibitors is still in  $\pm 85$ mV range. These findings imply that three inhibitors work as a mixed-type inhibitor. In addition,  $\beta_a$  &  $\beta_c$  values are slightly changed implying that the corrosion mechanism has not altered.

With increasing inhibitors (1,2,3) concentration,  $\eta_p$  rises while  $i_{corr}$  declines meaning the inhibitor reduces corrosion surface area. This is attributable to the active centers in the molecular structure of inhibitors, which have a high adsorption capacity on CS surface.



**Fig. (2):** Potentiodynamic polarization curves for the corrosion of Steel in 1.0 M HCl in absence and presence of different concentrations of compound at 25°C.

**Table (1):** The Polarization parameters for different concentrations of compound at ambient temperature in 1 M HCl

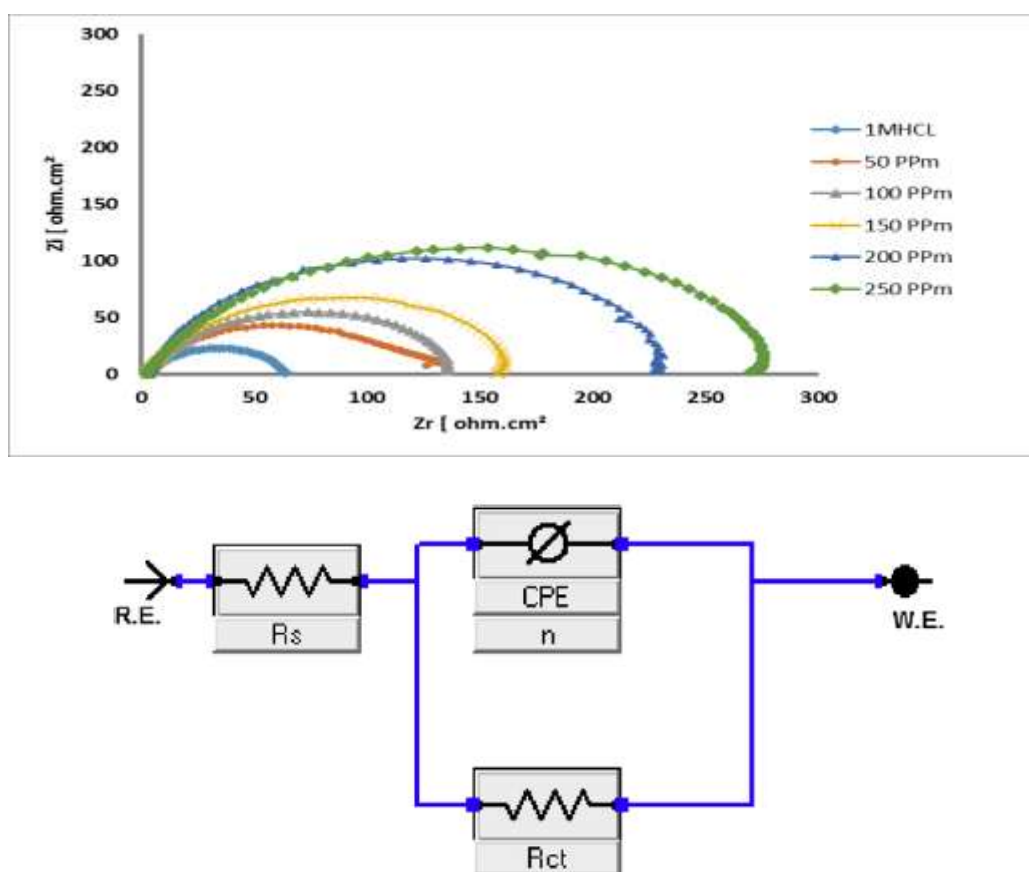
CONC. (PPM)	$E_{CORR}$ , MV	$I_{CORR}$ , MA/CM	$B_A$ , MV/DEC	$B_C$ , MV/DEC	$\Theta$	I%
0	500	0.25	190	-152	-	-
50	-495	0.11	129	-131	0.56	56



<b>100</b>	-518	0.097	208	-133	0.61	61
<b>150</b>	-502	0.08	120	-135	0.68	68
<b>200</b>	-520	0.07	211	-127	0.72	72
<b>250</b>	-530	0.06	302	-134	0.76	76

#### EIS results

At 20°C, the EIS Fig. (3) was utilized to investigate the efficiency of synthesized 1,2,3 inhibition. As a consequence of the frequency dispersion phenomenon results from steel roughness, inhomogeneity, and active site distribution, the Nyquist curve Fig. (3) revealed an imperfect semicircle. Fig. (3) also displays that the Nyquist diameter increases at an increasing inhibitors dosage, which is caused by the creation of a preventative layer on CS surface which effectively impedes the corrosion process. The inclusion of the inhibitor clearly leads to an improvement in real as well as imaginary impedance; the creation of prepared inhibitor film on CS surface is responsible for it. The irregularities and heterogeneities of CS surface result in frequency scattering in Nyquist plot (Hegazy and Atlam, 2016; Hegazy *et al.*, 2015a). It depicts the Bode and Phase angle plots for CS in 1 M HCl without and with the prepared inhibitor. As can be seen in this graph, the impedance value improves as the concentration of inhibitor rises. This implies that inhibitor (1)S delay the processes of Fe dissolution and H<sub>2</sub> evolution over CS surface. When plotting phase angle versus frequency, indicates only one time constant (Bode plot). This assumes only one circuit is the equivalent circuit for this system. This circuit consists of a resistance solution



**Fig. (3)** Nyquist plots and equivalent circuit of EIS measurements for inhibitor in 1 M HCl, at ambient temperature.

( $R_s$ ), charge transfer resistance ( $R_{ct}$ ) and non-ideal capacitance constant phase element ( $CPE$ ) as an electrical capacitor. Since Nyquist plots are imperfect semicircle, the  $C_{dl}$  was substituted by  $CPE$ .

The following equation is used to estimate  $CPE$  impedance ( $Z_{CPE}$ ) (Hegazy et al., 2015a; Wang *et al.*, 2010):

$$Z_{CPE} = Q_{dl}^{-1} (i\omega_{max})^{-n} \quad (2)$$

$$\omega = 2\pi f_{max} \quad (3)$$

where  $Q$ ,  $\omega_{max}$ ,  $f_{max}$ ,  $i$ ,  $n$  are the constant phase element, the angular frequency, the frequency at the maximum imaginary element of the impedance, the imaginary number, and a coefficient of surface inhomogeneity, respectively.

where  $Q$  = constant phase element,  $\omega_{max}$  = the angular frequency,  $f_{max}$  = the frequency at the maximum imaginary element of the impedance and  $i$ ,  $n$  are the imaginary number and a coefficient of surface inhomogeneity, respectively.

Equation (22) is employed to evaluate inhibition effectiveness ( $\eta_1$ ) (Hegazy et al., 2012; Han et al., 2018c):

$$\eta_1 = \frac{R_{ct}^o - R_{ct}}{R_{ct}^o} \times 100 \quad (4)$$

where  $R_{ct}^o$  &  $R_{ct}$  are the charge-transfer resistance with and without the inhibitor, respectively.

The following formula described  $C_{dl}$  values (Hegazy et al., 2015a; Ma et al., 2017):

$$C_{dl} = 2\pi f_{max} R_{ct} \quad (5)$$

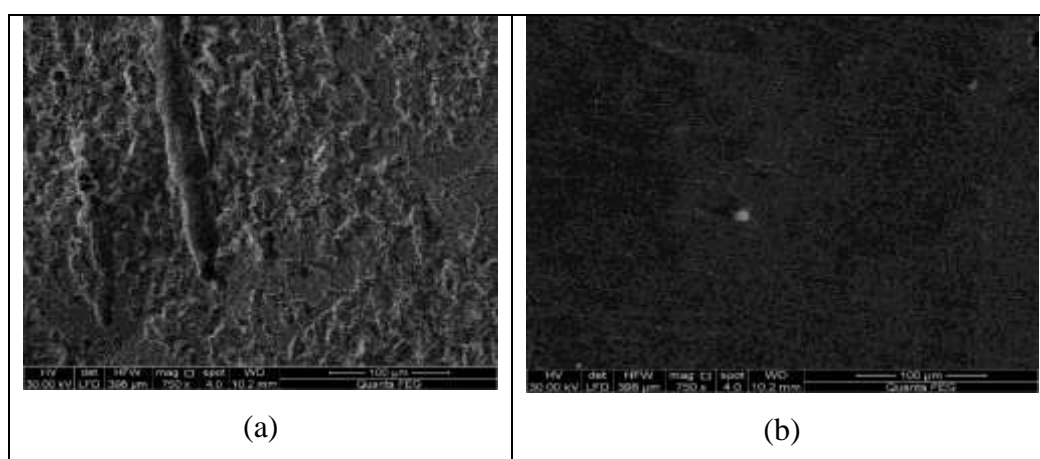
Table (2) summarizes the findings of the EIS. Table (2) displays  $n$  values in the range (0.70 to 0.95), which illustrative non-ideal capacitive behavior. The increment in the inhibitor concentration results in an increment in charge transfer resistance ( $R_{ct}$ ) with decline in  $C_{dl}$ . As  $C_{dl}$  decreases, water molecules are replaced on a regular basis at the CS/solution interface through prepared inhibitors adsorption, and the metal was shielded against corrosion (Hegazy *et al.*, 2016; Han *et al.*, 2018b). According to the EIS findings,  $\eta_1$  rises as inhibitor concentration raises (Hegazy et al., 2015c; El-Tabei *et al.*, 2022a; Abbas *et al.*, 2022; El-Tabei *et al.*, 2022b).

**Table (2):** The impedance parameters for different concentrations of (DHBAE and DHBADEA) compounds at ambient temperature in 1 M HCl

CONC. (PPM)	R1 (OHM.CM <sup>2</sup> )	R2 (OHM.CM <sup>2</sup> )	C (μF/CM)	E%
0	3.299	60.92	653.0	
50	3.589	129.3	194.3	53.5
100	<b>3.422</b>	<b>135.3</b>	<b>235.2</b>	55.6
150	3.875	157.8	112.8	61.9
200	4.203	228.1	278.9	73.6
250	3.437	277.4	90.62	78.4

**SEM measurements:** SEM examination was used to investigate the shape of the substrate's outer surface after it had already been corroded in a corrosive medium both with and without inhibitor. Fig. (4) illustrates surface morphologies of the face

of a C-st object after 24 hours of exposure in uninhibited solution with 1 M HCl Fig. (4 a) and after 24 hours of treatment in aggressive solution with 250 ppm of inhibitor. Fig. (a) polished surface while (b), illustrates several large holes caused by the acidic media's severe impact along with the chloride ions. Also Fig. (c) demonstrates that the outside surface is fully coated with an inhibitor film and/or corrosion byproducts, with very low pits. (c) clearly show the high coverage efficiency and protection for inhibitor. The SEM characteristic agrees well with the electrochemical estimates.



**Fig. (4)** SEM images of C-steel surfaces (a) in the absence of inhibitor, (b) in the presence of inhibitor.

## CONCLUSION

- 1- The inhibitor has been synthesized and characterized.
- 2- The corrosion monitoring techniques used suggest the prepared inhibitor has the adsorption ability over the CS.
- 3- The formation of a protective film explained the higher in  $\eta$ % and  $R_{ct}$  values.
- 4- The inhibitor adsorption is compliant with the Langmuir isotherm.
- 5- Inhibitor concerns an inhibitor of a mixed sort according to Tafel data.

6- In the presence of the inhibitor, the  $C_{dl}$  value was often less than in its absence, owing to the synthesized inhibitor's effective adsorption.

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## منع تآكل سبائك الفولاذ الكربوني في محاليل حمض الميكرولوجلوريك باستخدام مشتقات ثيوريا

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### المستخلص

يعد التآكل من أخطر التحديات التي تواجه قطاع السبائك والمعادن مما ويؤدي إلى العديد من القضايا الصناعية، مثل تدهور الهياكل والمعدات والتآكل في خزانات المصنع وخطوط الأنابيب في مصافي البترول، وبالتالي يقصر من العمر الافتراضي ويقلل من كفاءة هذه الأنظمة. لذلك يتم تطبيق الموانع على الوسائط المسببة للتآكل من خلال طرق فعالة لتحسين منع تآكل المعادن والسبائك في العمل الحالي. قام العمل الحالي بتحضير مركب ((2-aminoethyl) carbamothioyl) benzamide - N عن طريق تطبيق منهج الكيمياء الخضراء ثم تقييم المركب المحضر كمثبط للتآكل بواسطة المنهجات الكهروكيميائية المطيافية (EIS) وتقنيات الاستقطاب الديناميكي (PP) ووجد أن المركب المحضر المانع لديه القدرة على الامتزاز على الفولاذ الكربوني. (CS) وله سعة طبقة مزدوجة (C\_dl) كانت في كثير من الأحيان المركب له تأثير ثنائي تم تحضيره أولاً بواسطة الكيمياء الخضراء والثاني هو المركب المحضر له كفاءة جيدة كمثبط للتآكل، مما تسبب في إضافة عمراً طويلاً لسطح سبائك الصلب الكربوني.