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Experimental and Quantum Chemical Studies on the Corrosion Inhibition of Mild Steel By 2-((Thiophen-2-Ylmethylene) Amino)Benzenethio in 1M HCl

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Abstract:

The impact of a Schiff base namely 2-((thiophen-2-ylmethylene)amino)benzenethiol to corrode mild steel in 1 M HCl resolved was evaluated using different weight loss technique and scanning electron microscopy (SEM).different weight measurements to expand that the 2-((thiophen-2-ylmethylene) amino) benzenethiol inhibits the corrosion of mild steel through adsorbing of top for mild steel and block the active locality. The inhibitive impacts of 2-((thiophen-2-ylmethylene)amino)benzenethiol increase with increasing concentration and decrease with increasing temperature. SEM to checking revealed that the alloy surface was quite unaffected and formed protective film on its surface. The investigated inhibitor become as a shield for the mild steel surface from corrosive solution. Quantum chemical investigations corroborate experimental results well. The synthesized inhibitor was characterized employing NMR (nuclear magnetic resonance), FT-IR (Fourier-transform infrared) spectroscopies and CHN elemental analysis.

Key words: Corrosion steel, Inhibiter, Thiophene-2-carbaldehyde.

Introduction:

Mild steel is the most widely employ alloy in industries such as gas and oil (1). Corrosion of alloys are of considerable economically relevance and destroy many industries and their applications (2, 3). The protection of alloys from corrosion caused by environment, aggressive electrolytes (4) using organic molecules might permeate the layer or contact the surface of alloy directly after failure of coating under stress such as stone chipping of automotive coatings (5). Corrosive media have been utilized in the activities of industries naming acid pickling, descaling and cleaning. Employing of natural organic inhibitors become the most active and cost successful process of reduce alloys corrosion (6-8). Synthetic organic molecules that have heteroatoms (such as nitrogen, sulfur or oxygen) in addition to pi-electron systems were approved to exhibit excellent inhibition efficiencies (9-11). The inhibition impact such organic molecules depend on capability to adsorb on alloy surface. The inhibition impact of the synthesized organic molecules are commonly revolve on the replacement of H_2O molecules by organic inhibitor and form protective film on the alloy surface (12).

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In this study, new Schiff base molecule namely 2-((thiophen-2-ylmethylene) amino) benzenethiol has been synthesized, characterized and evaluated for it inhibition impacts towards mild steel corrosion reaction in corrosive solution of hydrochloric acid employing different weight and scanning electron microscope technicality. Aquality, chemical activities and structure relationship of the corrosion inhibition were also investigated.

Martials and Methods

Synthesis of corrosion inhibitor, 2-((thiophen-2-ylmethylene) amino) benzenethiol

All the dissolvent solution and priority materials have been acquired in "Sigma-Aldrich Malaysia". FT-IR "Fourier-transform infrared spectroscopy" spectrum for the corrosion inhibitor has been recorded through "Shimadzu 8300 spectrometer". A Carbon, Hydrogen & Nitrogen Analyzer could be utilized for CHN analysis via Carlo Erba 5500 CHN elemental analysis". The spectrum of NMR "Nuclear magnetic resonance spectroscopy" was recorded through Bruker Spectro-spin instrument with 300 MHz UltraShield using the solvent Hexadeuterodimethyl sulfoxide and Tetramethylsilane "TMS" as internal standard for calibrating chemical shift. The preperation of 2-((thiophen-2-ylmethylene) amino) benzenethiol has been done as in Fig. 1, by the mixed of an equimolar of 2-aminobenzenethol , thiophene-2-

carbaldehyde in ethanol as a solvent than reflux. After 5 h., filtered and recrystallized from ethanol than dried to produce yellow precipitate with the yield of 66%. M.P.=180-182.

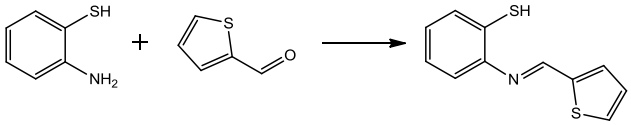

Figure 1. The synthesis of the corrosion inhibitor.

Corrosion tests

Mild steel coupons texture of C (0.21%), Si (0.38%), Mn (0.05%), P (0.09%), Al (0.01%), and Fe (99.21%) were utilized for weight loss investigations. The coupons were cleaned according to the standard procedure to be specific ASTM G1-03 (13-22). In a typical methodology, coupons were suspended volume 200 mL of a corrosive solution (in duplicate) to try or not of 2-((thiophen-2-ylmethylene) amino) benzenethiol. Various concentrations of 2-((thiophen-2-ylmethylene) amino)benzenethiol (0.1, 0.2, 0.3, 0.4 and 0.5 mM) in 1M HCl as corrosive solution were utilized at 303, 313, 323 and 333 K. After 5h as reaction time the mild steel to check coupons then to rinse with distilled water , ethanol and to drain then weighted intire.

The inhibition competence were calculated by Equation 1(23).

$$IE\% = \frac{W_o - W_1}{W_o} \times 100 \qquad 1$$

"where w_o is the weight loss in absence of the tested inhibitor and w_1 is the weight loss in presence of the tested inhibitor".

Corrosion quickness calculated by the Equation 2(24).

$$C_R = \frac{mg}{cm^2 h^1} \qquad 2$$

Mild Steel Surface Characterization

Mild steel coupons surfaces were investigated by scanning electron microscope (SEM) that immersed in HCl solution before and after added of the corrosion inhibitor at the concentration 0.5 mM for 5 h at 303 K

Computation Methods

Density function theorem (DFT) was employed to elucidate optimization texture tested inhibitor and quantum parameters "EHOMO and ELUMO (Energy of highest occupied molecular orbital and Energy of lowest unoccupied molecular orbital), dipole moment/μ, chemical hardness/η, chemical softness/σ and Electrophilicity index/ω" at the function DFT-B3LYP with the basis set 6-31G. The mechanism of inhibiton of the investigated inhibitor depending on quantum parameters were figured as in Equations 3-7 (14).

$$\Delta E = E_{LUMO} - E_{HOMO} \qquad 3$$

$$\eta = \frac{1}{2}(E_{HOMO} - E_{LUMO}) \qquad 4$$

$$\sigma = \frac{1}{\eta} \qquad 5$$

$$\mu = \frac{1}{2}(E_{HOMO} + E_{LUMO}) \qquad 6$$

$$\omega = \frac{\mu^2}{2\eta} \qquad 7$$

Results and Discussion

Structure

The inhibitor 2-((thiophen-2-ylmethylene) amino)benzenethiol was facilely prepared in superior yield through the reaction of 2-aminobenzenethol and thiophene-2-carbaldehyde under reflux conditions. Molecular weight of 2-((thiophen-2-ylmethylene)amino)benzenethiol was 219 regarding to chemical formula C11H9NS2 that was confirmed by elemental analysis. Elemental analysis (C.H.N): C, 60.24 (59.82); H, 4.14 (4.48) and N, 6.39(7.01). Fourier-transform infrared spectroscopy (FTIR) is a method applied infrared spectra in order to characterized the chemical formula of the tested compound. This technique was employee to investigated the functional groups of the synthesized inhibitor to elucidated and confirmed it structure. No amino or carbonyl absorption bands were observed for 2-((thiophen-2-ylmethylene)amino)benzenethiol in FTIR spectrum. New absorption band was observed at 1651.6 cm -1, for the azomethine group. Another absorption bands were observed at thiol group at 2363.73 cm-1; carbon-sulfur bond has asym and sym absorption bands at 1053.68 and 744.94 cm-1 respectively. Nuclear magnetic resonance spectroscopy, usually recognized as1H- NMR spectroscopy or MRS "magnetic resonance spectroscopy", is a spectroscopic method to realize, the magnetic fields around nuclei.1H- NMR is the firm technique to elucidate organic molecules. The general kind of 1H- NMR were H-1and13 C- NMR spectroscopy. The 1H- NMR spectrum showed singlets at δ 3.39 ppm, due to the SH proton and multplate was observed at δ 7.03-7.81 ppm due to the aromatic and azomethine protons.

Corrosion measurements

Weight loss measurements have been done in coupons top of mild steel in 1 M HCl in absence and presence of 2-((thiophen-2-ylmethylene) amino)benzenethiol for reaction time 5h. Fig.2

represents the inhibition efficiencies for various concentrations for added compound 2-((thiophen-2-ylmethylene)amino)benzenethiol at 303, 313, 323 and 333 K, respectively.

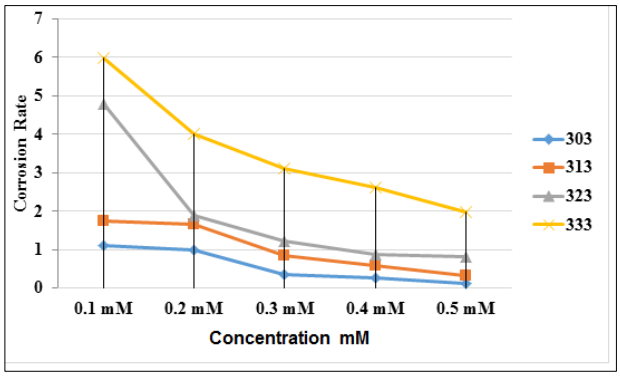

Figure.2 Inhibitor concentration effect on corrosion rate at 303-333K.

Figure 2, explain the relation CR with different focusing of inhibitor at versus temperature degrees. The CR reduce regarding to increasing of inhibitor concentration in corrosive resolved. CR decrease to swear together with concentration increasing at the 313 K to reach 0.32 mg·cm−2·h−1 at 0.5 mM as inhibitor concentration. As in Figure 2, the curves at 323 K and 333 analogous forms in the curves at 303 K and 313 K.

Figure 3 displayed the locality of the IE% to reflect the impact of various concentrations started from 0.1 to 0.5 mM with different degrees of temperature |303, 313, 323 and 333 K". It is obvious that the IE% was not enough influenced by studied temperature degrees to begin from 303 K to 313, exceptionally of concentrations 0.2-0.4 mM; whilst at the highest investigated concentration the inhibition quite increased. At the highest studied temperature degree, it can be seen that the inhibitor concentration has not considerable effects IE% and this result might be due to waste of the scholar ship inhibitor.

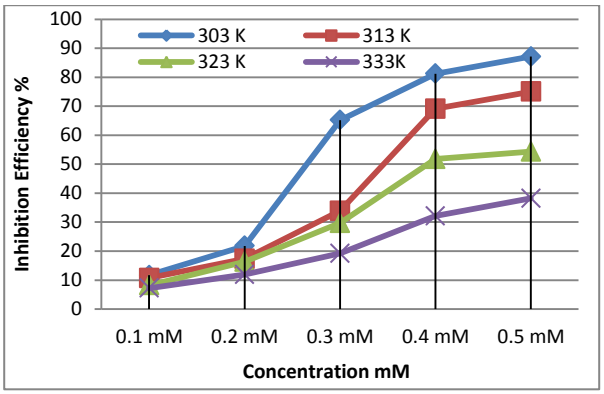

Figure 3. Effects different kinds concentrations of 2-((thiophen-2-ylmethylene) amino) benzenethiol on inhibition performance at different temperature degrees

Evaluation of the product demonstrated IE in target corrosion inhibitor increased to make parallel concentration increased. Most active value of IE was 87.1% that at the highest studied concentration for 2-((thiophen-2-ylmethylene) amino) benzenethiol. Also, the ideal concentration for 2-((thiophen-2-ylmethylene)amino)benzenethiol was 0.5 mM, this was due to no considerable changes in IE at more than this concentration. The Arrhenius equation (as in equation 8) was employed to displayed the temperature impacts on the IE of 2-((thiophen-2-ylmethylene)amino)benzenethiol as investigated inhibitor compound(25,26).

$$lnC_R = \frac{-E_a(\text{activation energy})}{R(\text{gas constant})T(\text{temperature})} + lnA\ (\text{Arrhenius factor}) \quad 8$$

The energy in absence and presence of 2-((thiophen-2-ylmethylene)amino)benzenethiol have been calculated depending on equation 9 and Arrhenius plots which demonstrated in fig. 4.

$$Slope = -\frac{\Delta E_a}{2.303R(\text{gas constant})} \quad 9$$

The Ea value was 88.4 kJmol−1 for 2-((thiophen-2-ylmethylene) amino) benzenethiol. This refer to importance of presence of 2-((thiophen-2-ylmethylene) amino) benzenethiol in corrosive solution which can be compare with Ea value in absence of 2-((thiophen-2-ylmethylene) amino) benzenethiol and hence this result indicate that 2-((thiophen-2-ylmethylene) amino) benzenethiol molecules forming barrier as protection film on the top of mild steel (25).

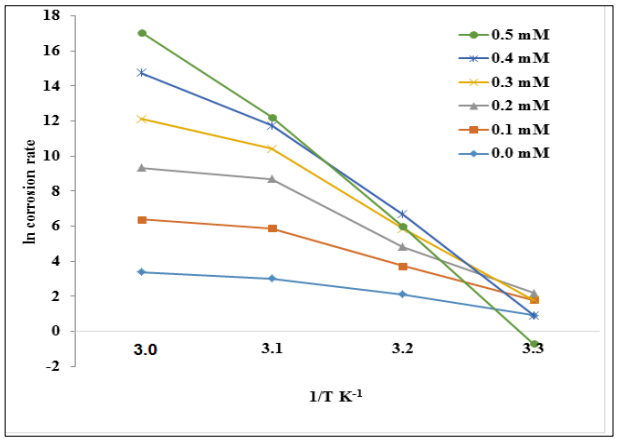

Figure 4.The effects of degrees of temperature on the CR "corrosion rate" based on Arrhenius plot of for mild steel in corrosive solution.

2.1 Surface Morphology Investigations

From Fig 5, it can be shown that the SEM images of the of MS top samples which to dip in corrosive resolvent for five hours, when absence (Figure 5a) and presence (Fig. 5b) of 2-((thiophen-

2-ylmethylene) amino) benzenethiol as corrosion inhibitor stabilized destruction in MS surface (Fig. 5a) of samples. As well, the SEM image for samples which immersed in corrosive solution for five hours, in presence of corrosion inhibitor 2-((thiophen-2-ylmethylene) amino) benzenethiol at concentration of 0.5 mM was smooth and coated by protective film. This indicate that the inhibitor 2-((thiophen-2-ylmethylene) amino) benzenethiol adsorbed and block the surface of MS from acidic impact.

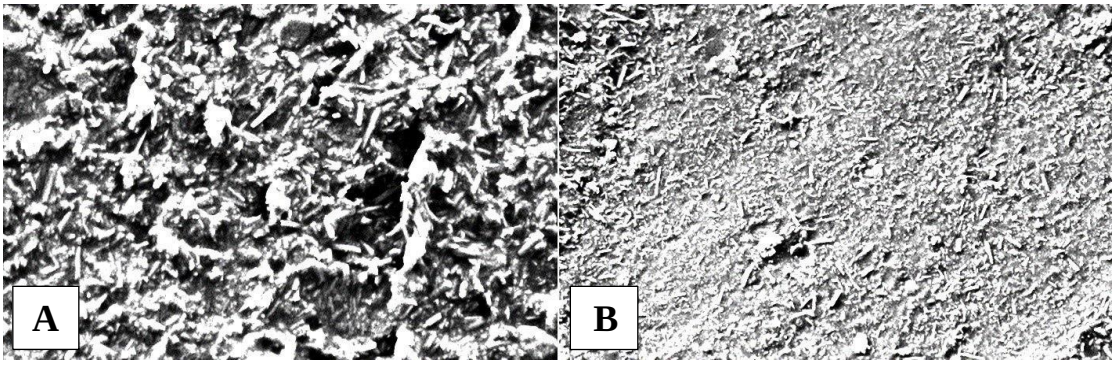

Figure 5. The SEM pictures of samples of MS surface in corrosive acid in the absence (a) and presence (b) of inhibitor.

Quantum Calculations

The inhibitor 2-((thiophen-2-ylmethylene) amino) benzenethiol has different chemical compounds by having nitrogen and sulfur atoms in addition to aazomethan group with resonance impact, through the linkage between thiophene groups, azomethine and phenyl group jointly yield compound with unique characteristics due to resonance and inductive impress. It is obvious from ideal texture for 2-((thiophen-2-ylmethylene) amino) benzenethiol, the geometrical structure is planar because of the cycliozation of the molecule. Electronic texture for 2-((thiophen-2-ylmethylene) amino) benzenethiol was considered utilizing of DFT-B3LYP/6−311G. HOMO "highest occupied molecular orbital" and LUMO "lowest unoccupied molecular orbital" to check for 2-((thiophen-2-ylmethylene) amino) benzenethiol and to present as in Figure 6. Parameters such as ΔE "energy gap = ELUMO − EHOMO", μ"dipole moment" and χ "electronegativity" have been demonstrated as in Table 1. EHOMO displayed electron capability of 2-((thiophen-2-ylmethylene)amino)benzenethiol with electron susceptibility as comparing to other investigated inhibitors(27-32). The releasing electronic group namely thiophene linkage to phenyl group via azomethane in 2-((thiophen-2-ylmethylene)amino)benzenethiol increase the electron donating capabilities of 2-((thiophen-2-ylmethylene)amino)benzenethiol molecules as in Fig. 6. EHOMO and ELUMO could be considered as the affinity of 2-((thiophen-2-ylmethylene) amino) benzenethiol molecules. ΔE is major parameter which depict the linkage of MS surface and inhibitor molecules. The investigated inhibitor 2-((thiophen-2-ylmethylene) amino) benzenethiol have different of energy hole and that of 3.898 eV as displayed in Fig. 6 and table 1. Generally, investigated compound with minimum χ refer to higher electron propensity showed higher inhibition efficiency. In this search, χ completely to lean to close results. The dipole moment for the inhibitor that the dipole moment with high value indicating to excellent corrosion inhibitor. Regarding to this investigation, the value dipole moment of 2-((thiophen-2-ylmethylene) amino) benzenethiol was 4.599 that indicate to 2-((thiophen-2-ylmethylene) amino) benzenethiol molecules have excellent achievement inhibition activity. From table 1 we can say that μ of 2-((thiophen-2-ylmethylene) amino) benzenethiol molecules have higher value comparing to μ of water molecules and this refer to that of 2-((thiophen-2-ylmethylene)amino)benzenethiol molecules have higher activities to react either physically or chemically with the MS surface. From this it can be conclude that, 2-((thiophen-2-ylmethylene) amino) benzenethiol molecules may be taking up on the MS surface through removing of water molecules(33-35).

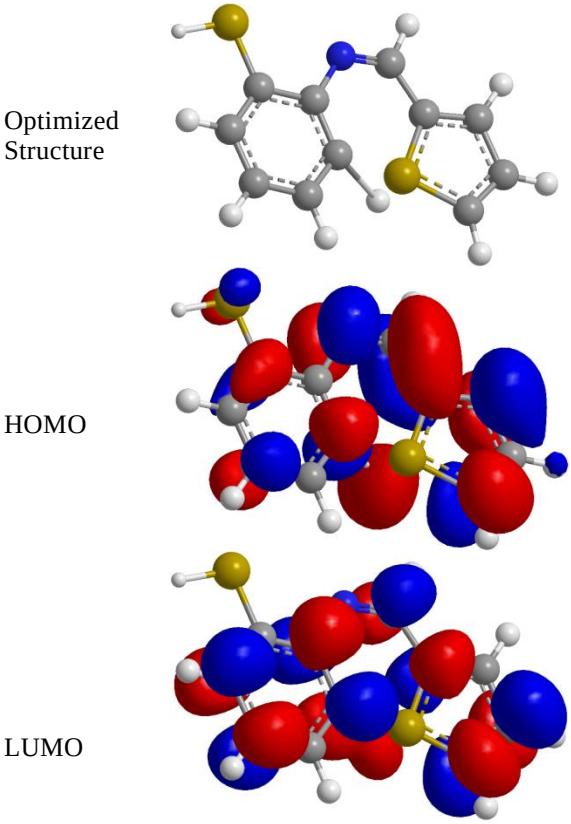

Figure 6. Electronic structures of 2-((thiophen-2-ylmethylene)amino)benzenethiol molecule

Table 1. Electronic factors for 2-((thiophen-2-ylmethylene)amino)benzenethiol molecule

Factors	Inhibitor
HOMO [eV]	-4.832
LUMO [eV]	-0.934
ΔE = ELUMO − EHOMO [eV]	3.898
Dipole moment μ	4.599
Electron affinity X	4.832
Ionization potential (I)	0.934
Electronegativity χ $[\chi = \frac{I+A}{2}]$	2.883
Global hardness $[\eta = \frac{I-A}{2}]$	1.949
Chemical softness $[S = \frac{1}{\eta}]$	0.513

The factors hardness and softness were employee to assess the reactivity with stability for the studied molecules. Hardness demonstrate the resistance towards the electron polarization of molecules. To harden molecules have big energy gap and soft molecules have fine energy gap (31). In this work, the 2-((thiophen-2-ylmethylene) amino)benzenethiol molecules to harden value 1.949, they were proved that 2-((thiophen-2-ylmethylene)amino)benzenethiol molecules that to harden with small value was possible to be as well inhibitor. To post of electron in easy way need maximum value chemical softness (32). 2-((thiophen-2-ylmethylene) amino) benzenethiol molecules have chemical softness equel to 0.5131eV so, it has the maximum inhibition achievement.

Conclusion:

The effect of inhibition of the inhibitor 2-((thiophen-2-ylmethylene) amino) benzenethiol on MS surface in corrosive solution has been investigated employing different weight and SEM "scanning electron microscopy" device. Quality chemistry has been employee to prove trial results. The different weight findings displayed 2-((thiophen-2-ylmethylene)amino)benzenethiol inhibit the corrosive solution and the inhibition performance increase regarding to increasing of concentration. The inhibition activity was 87.1% which displayed the high protection activity against hydrochloric acid. The molecules of 2-((thiophen-2-ylmethylene) amino) benzenethiol inhibit the corrosion at MS in 1 M hydrochloric acid through adsorbing on the top of MS and form protection film by physisorption or chemisorption SEM pictures to indicat that 2-((thiophen-2-ylmethylene) amino) benzenethiol adsorbed on MS the top and shield the MS surface from corrosive solution. Quantum chemistry displayed that active sites in the molecules of 2-((thiophen-2-ylmethylene) amino) benzenethiol have sulphur and nitrogen atoms with pi-electron.

Acknowledgment

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Conflicts of Interest: None.

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دراسات كيميائية كمية وعملية لتثبيط تأكل الفولاذ الطري بواسطة 2-((ثايوفين -2- يل مثيلين) أمينو) بنزين ثايول

ايمان مهدي محمد حسن

قسم الكيمياء، كلية العلوم للبنات، جامعة بغداد، بغداد، العراق

الخلاصة:

تم تقييم تأثير قاعدة شيف والمسماة2 -((ثايوفين-2- يل مثيلين)امينو)بنزين ثايول على تأكل الفولاذ الطري في محلول حامض بتركيز (1 مولاري) وباستخدام تقنية فقدان الوزن والمسح الضوئي للمجهر الالكترونيHCl حيث اظهرت القياسات فقدان الوزن 2-((ثايوفين-2- يل مثيلين) امينو) بنزين ثايول كمثبط لتأكلSEM.

الفولاذ الطري من خلال التكثيف على سطح الفولاذ الطري ومنع المواقع النشطة من خلال البحث وجد ان التاثيرات المثبطة لل2-((ثايوفين-2- يل مثيلين) امينو) بنزين ثايول تزداد طرديا مع التركيز وعكسيا مع درجة الحرارة واظهرت قياسات المسح الضوئي للمجهرالالكتروني ان سطح السبيكة يتأثر تماما وبشكل فيلم واقي على سطحه.

ان الدراسات الكيميائية الكمية للبحث ونتائجها العملية كانت جيدة وتم تشخيص مانع التأكل باستخدام تقنيات الرنين النووي المغناطيسي والاشعة ما تحت الحمراء وتحليل العناصر..

الكلمات المفتاحية: المثبط، تآكل الستيل، ثيوفين-2-كربالديهيد.