

CORROSION INHIBITION EFFICIENCY AND ADSORPTION MECHANISM OF SOME SCHIFF BASES AT MILD STEEL/HCL INTERFACE

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ABSTRACT

In this study, the inhibition effect of two Quinazolinone-Based compounds, namely, 2-(3, 3-Dimethyl-2, 3-dihydro-indol-2-ylidene)-3-[(2-hydroxyphenyl)-imino] propylidene and 2-(3, 3-Dimethyl-2, 3-dihydro-indol-2-ylidene) -3-[(2-hydroxyphenyl)-imino] propanal (LH₁ and LH₂, respectively),were investigated as potential corrosion inhibitors for mild steel in 1.0 M HCl solution. Various corrosion analysis methods were used, such as the gravimetric method, potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) and scanning electron microscope (SEM). The results revealed that the inhibition efficiencies increased with an increase in HLs concentration having a mean efficiency of 92 % at 50 ppm in 1.0 M HCl. The obtained results showed that the inhibition efficiencies from methods employed were in a good agreement. The polarization results revealed that the inhibitor decreased the corrosion current densities by means of a mixed mode mechanism. The SEM results indicated that the adsorption of the protective layer of the inhibitor on the metal/solution interface and obeyed Langmuir adsorption isotherm.

KEYWORDS: Metal, Corrosion, Electrochemical Methods, Schiff Bases, Surface Characteristics

INTRODUCTION

Organic compounds as corrosion inhibitors of iron, iron alloys, and steel have great significance due to the important applications of the metal [1-3]. Due to its low cost and excellent mechanical properties, mild steel is widely used as a construction material in automobiles, pipings, structures, fencing and in different applications in the chemical industry [4]. As the pipe performance of the equipment is severely affected by the presence of both scale and carrion products, researchers have focused on the corrosion inhibition of mild steel in acid solutions. Acids, particularly hydrochloric and sulphuric acids solutions are widely used for the pickling, descaling and cleaning of mild steel [5].

In order to retard the degree of metal dissolution and acid consumption, corrosion inhibitors are one of the most economical, practical and effective methods for the protection of metals against electrochemical corrosion [6,7]. The literature reveals that various organic compounds have been investigated in the terms of their effectiveness as corrosion inhibitors, including surfactants [8], bipyrazole derivatives [9], triazole derivatives [10], quinoxaline derivatives [11], organic dyes and drugs [12,13], aromatic hydrazides[14]. The inhibition results from the adsorption on the metal surface which blocks the active sites, thereby resulting in a decrease in the corrosion rate [15]. Research work reveals that the most well-known effective corrosion inhibitors for aluminium and steel are heterocyclic organic compounds. These

mainly comprises nitrogen, oxygen, phosphorous, sulphur atoms and multiple bonds in their molecular structure, which hare usually effective inhibitors they provide important features for the adsorption of inhibitor molecules on the metal substrate [16-19].

Among several organic compounds, some of the Schiff base compounds are extensively employed as excellent corrosion inhibitors for minimizing the corrosion of steel in acidic environments [20-22]. Some researchers reported that the presence of the –CN– group in Schiff base compounds enhances their adsorption activity and inhibition ability much greater than those containing amines and aldehydes [23]. A literature survey reveals that the Schiff base compounds that contain both nitrogen and sulphurare more effective corrosion inhibitors compared to those with only nitrogen or sulphur [26-27]. It is generally assumed that the inhibitor molecules are usually adsorbed on the metal surface by the electrostatic interaction between the charged centers of molecules and the charged metal surface. This results in a dipole interaction of the molecules and metal surface (physical adsorption) or by the transfer or shearing of electrons from the inhibitor to the metallic surface resulting in the formation of a co-ordinate covalent bond (chemical adsorption) in which the bonding strength is much larger than physical adsorption[28].Several parameters, such as, the structural and electronic characteristics of the inhibitor, surface morphology, temperature, pressure, the flow velocity as well as the pH and composition of the aggressive environment depends on the adsorption process, and consequently, the inhibition efficiency and inhibition mechanism[29-32].

The objective of the present work is to investigate the corrosion behaviour and corrosion mechanism of mild steel in1 M HCl solution at in the absence and presences of two newely synthesized Schiff base compounds (HL_S). Gravimetric measurements, electrochemical and scanning electron microscope (SEM) techniques were utilized to evaluate the corrosion inhibition efficiency of the investigated Schiff base compounds systemically.

EXPERIMENTAL METHODS

Preparation of Electrode

The working electrode wasa commercial mild steel sheet with the following chemical composition of (wt %): C = 0.103, Si = 0.03, P = 0.021, Mn = 0.230, Al = 0.012, with the rest being Fe. The mild steelspecimen was purchased from Power Hardware and Trading Sdn. Bhd, Kuala Lumpur, Malaysia. Steel samples of size 1 cm x 1.5 cm x 0.005 cm were mounted on a polyester base so that only 2.56 cm² of their cross-section alarea was exposed to the corrosive solution. The surfaces of the working electrodes were mechanically abraded using different grades of sand papers, 400, 600, 800, 1000, 1200 and 1500, prior to use. The electrodes were cleaned by washing with deionized water and degreased with ethanol respectively, and finally, dried with filter papers. For each test, a freshly abraded electrode was used. The HCl solution was prepared from analytical grade concentrated acid and deionized distilled water.All the tests were performed in a concentration range of 25–100 ppmat room temperature.

Inhibitors

The Schiff base inhibitors were synthesized by the condensation reaction according to the published method [33]. The obtained products were purified by recrystillzation from DMF solvent. The structural formula, abbreviations and IUPAC names of the synthesized Schiff base compounds are given in Table 1.

Inhibitor	Abbreviation	Structure	
2-(3,3-Dimethyl-2,3-dihydro-indol-2-ylidene)-3 -[(2-hydroxyphenyl) imino]propylidene	LH_1	ОН ОН	
2-(3,3-Dimethyl-2,3-dihydro-indol-2-ylidene)-3-[(2-hydroxyphenyl) imino] propanal	LH ₂	OF NH	

Table 1: The Chemical Structure and Abbreviation of HLs Schiff Base Inhibitors

Weight Loss Measurements

Weight loss experiments were carried out using steel specimens with 1 cm x 1.5 cm x 0.005 cm dimensions. The series of steel specimens were abraded with different grades of emery paper, washed with distilled water, degreased with acetone, dried and kept in a desiccator. The specimens were accurately weighed and then immersed in 1 MHCl solution with and without the Schiff base. After 6 h exposure, the specimens were taken out, rinsed thoroughly with distilled water, dried and weighted accurately. The corrosion rate, surface coverage and inhibition efficiencywere calculated using equations 1, 2 and 3, respectively.

$$C_{\rm R} = \frac{\Delta W}{A \, X \, t} \tag{1}$$

$$\Theta = \frac{C_R^0 - C_R}{C_R} \tag{2}$$

$$\dot{\eta}_{\rm WL}(\%) = \frac{C_R^0 - C_R}{C_R} \ge 100$$
(3)

Where ΔW is the average weight loss (mg), A is the available surface area of the specimen (cm²), t is the immersion time (h), Θ is surface coverage, $\eta_{WL}(\%)$ is the weight loss efficiency and C_R^0 and $C_R(mg/cm^2.h)$ are the corrosion rates in the absence and presence of the inhibitors, respectively.

Potentiodynamic Polarization Measurements

The electrochemical behavior of the MS sample in the absence and presence of an inhibitor was investigated using AUTOLAB (version 4.99) potentiostat/galvanostat. The experimental data were analysedutilizing Pentium IV computer with GPESsoftware, by recording the anodic and cathodic Tafel curves in 1.0 MHCl solutions with various inhibitor concentrations. A conventional three-electrode cell was employed, consisting of MS as the working electrode with an area of 2.56 cm², a platinum auxiliary electrode and an Ag/AgCl reference. The working electrode was first immersed in the test solution for 30 seconds to establish a steady state open circuit potential (E_{ocp}).

Potentiodynamicpolarization curves were obtained with a scan rate in the potential range between -800to -250 mV at5 mVs⁻¹. The linear Tafel segments of the anodic and cathodic curves were extrapolated to obtain the corrosion potential

 (E_{corr}) , and corrosion current density (i_{corr}) . The $\eta_P(\%)$ at different inhibitors concentrations were calculated from the following Eq.

$$\hat{\eta}_{P}(\%) = \frac{i_{corr}^{0} - i_{corr}}{i_{corr}^{0}}$$

$$\tag{4}$$

Where i_{corr}^0 and i_{corr} are uninhibited and inhibited corrosion current densities (μ A/cm²), respectively

Electrochemical Impedance Spectroscopy (EIS)

The impedance measurements were carried out at the open circuit potential (Eocp), using a computercontrolled potentiostat (autolab). The impedance data were analysed using a Pentium IV computer and FRA software. The alternating current frequency range was between 10^5 and 3 x 10^{-2} Hz, with a 5 mV sine wave as the excitation. The inhibition efficiency $\hat{\eta}_{RP}(\%)$ was calculated from the polarization resistance, as shown in equation (5);

$$\hat{\eta}_{\rm RP}(\%) = \frac{R_P - R_P^0}{R_P} x \ 100 \tag{5}$$

Where R_P and R⁰_P represents the polarization resistance for the inhibited and uninhibited solution, respectively

Surface Characterization

The steel specimens with 1 cm x 1.5 cm x 0.005 cm dimensions were immersed in 1.0 M HCl solution for 6 h in the absence and presence of the LHs inhibitor at 298 K. The specimens were removed and the surface morphological changes of the samples were investigated with a scanning electron microscope (SEM), using model Carl Zeiss LEO SUPRA 50 VP at an accelerating voltage of 15 KeV.

RESULTS AND DISCUSSIONS

Weight Loss Measurements

The values of corrosion rate (C_R), surface coverage (Θ), and $\eta_{WL}(\%)$ obtained from the weight loss measurements of mild steel after 6 h immersion in 1.0 M HCl solutions with and without the various concentrations of the Schiffbase are summarized in table 2.The lone pair of electrons in the nitrogen atom and the presence of higher electron density in the inhibitor molecules co-ordinate with the active sites of the metal, and hence inhibit the corrosion process [25]. The data in Table 2reveals that the corrosion rate of mild steel decreases with the addition of the Schiff base of the HLs, while the inhibition efficiency η_{WL} (%) and surface coverage (Θ) increase with the increase in inhibitor concentration. At a concentration of 100 ppm, the HL₁ and HL₂Schiff bases exhibit maximum inhibition efficiency (94 % and 91%),respectively,at room temperature, which is due to efficient inhibition performance. This suggests that the inhibitor molecules act by the adsorption on the metal surface [18].

 Table 2: Corrosion Inhibition Effect of LH1and LH2 Obtained from the Weight Loss Measurements of Mild Steel

 after 6 h Immersion in 1 M HCl, Solution in the Absence and the Presence of the Schiff Bases at 298 K

	LH_1			LH_2		
Concentration (ppm)	A ($\mu g \ cm^{-2} \ h^{-1}$)	θ	ή _{WL} (%)	A ($\mu g \ cm^{-2} \ h^{-1}$)	θ	ή _{WL} (%)
Blank	5.66			5.5		
25	1.06	0.82	82	3.6	0.34	34
50	0.93	0.84	84	2.9	0.41	41
75	0.53	0.9	90	2.1	0.61	61
100	0.3	0.94	94	0.5	0.91	91

Potentiodynamic Polarization Measurements

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Potentiodynamic polarization curves were recorded to investigate the corrosion inhibition effects of the green inhibitor on the electrochemical behaviour of MS in 1.0 M HCl. Figure 1 show the anodic and cathodic polarization plots recorded on MS in 1 M HCl in the absence and presence of the LH₁ and LH₂Schiff bases at different concentrations. The corrosion parameters, including corrosion potential (Ecorr), corrosion current density (icorr), anodic and cathodic Tafel slopes (ba and bc, respectively) and corrosion efficiency $\dot{\eta}_P(\%)$ were derived from the polarization curves and summarized in table 3.

Inhibitors	Concentration (ppm)	- Ecorr (mV vs. SCE)	ba (V/dec)	bc (V/dec)	icorr (µAcm ⁻²)	ή _P (%)
	Blank	0.634	0.161	0.192	543	-
	25	0.722	0.134	0.142	74	86
HL1	50	0.736	0.121	0.114	67	88
	75	0.758	0.118	0.112	51	90
	100	0.762	0.068	0.061	37	93
HL2	25	0.701	0.221	0.159	102	81
	50	0.623	0.196	0.139	70	87
	75	0.644	0.167	0.981	56	89
	100	0.656	0.121	0.251	47	91

Table 3: Electrochemical Polarization Parameters and the Corresponding Inhibition Efficiency for Mild Steel in 1.0M HCl Solution with and without Addition of the Schiff Base HLs at Different Concentrations

Figure 1 shows that the presence of the LHs Schiff base inhibitors decreases both the anodic and cathodic slopes with the increase inhibitor concentration. Generally, in acidic solutions the metal dissolution is the anodic process and hydrogen evolution or reduction of oxygen is the principal cathodic process. The cathodicTafelcurves suggests that the addition of LH_s does not modify the hydrogen evolution mechanism and the reduction of H⁺at the MS surface which mainly occurs through a charge transfer mechanism. Firstly, the inhibitor molecules are adsorbed on the MS surface and block all the available reaction sites. Hence, the available surface for the reaction of H⁺ ions decreases, and as a result, the actual reaction mechanism stands unaffected[34].

It must be noted from table 3 that, when the concentration of the corrosion inhibitor increases the corrosion current density decreases, while the inhibition efficiency increases, and the corrosion potential with the LHs inhibitors shifts towardsacathodic potential. These results suggestthat the LHs are a mixed type inhibitor, with predominant cathodiceffectiveness in the HCl medium. The obtained results are a in a good agreement with those obtained from impedance measurements.



Figure 1: Potentiodynamic Polarization Curves for MS in 1 M HCl Solution Containing Different Concentrations of (a) HL₁ and (b) HL₂

Electrochemical Impedance Spectroscopy Measurements

Electrochemical Impedance Spectroscopy is a powerful tool for a detailed investigation of the electrode kinetics and surface properties of the system under study. The analysis of the different shapes of the impedance diagram provides a basis for the mechanistic approach [35]. Figure 3 presents the Nyquist plots of MS in uninhibited and inhibited acidic solution with various concentrations of Schiff base compounds of LH₁ and LH₂, respectively.

The existence of a single semicircle shows a single charge transfer process during the dissolution, and it is important to note that the changes in the concentration of Schiff base inhibitors did not alter the impedance mechanism, which suggests a similar mechanism for the corrosion inhibition of mild steel by the Schiff base compounds. Figure4 and Fig 5 shows that the Nyquist plots do not yield a perfect semicircle as expected but rather a depressed semicircle due to the frequency dispersion of interfacial impedance, which arises due to the surface roughness, impurities, dislocations, grain boundaries, adsorption of inhibitors, and the formation of the porous layers and homogenous electrode surface [36,37]. The fitted values for polarization resistance (R_P), constant phase element (CPE) and the inhibitor efficiency η_{RP} (%), are illustrated in table 4.

Inhibitor	Concentration (ppm)	CPE (10 ⁻⁴) (F)	n	R p (Ω)	ήz (%)
	Blank	7.89	0.87	11.63	-
HL_1	25	6.29	0.82	55.14	78
	50	5.81	0.66	58.07	80

5.35

4.73

5.8

3.31

3.11

2.95

75

100

25

50

75

100

 Table 4: Electrochemical Impedance Parameters of Mild Steel in 1 M HCl Solution in the Absence and Presence of Different Concentrations of HLs at Room Temperature

0.79

0.67

0.68

0.66

0.65

0.64

75.04

153.67

31.94

50.08

61.17

122.22

84

92

67

76

80

90

 HL_2

The results obtained from EIS were fitted according to the electrical equivalent circuit diagram shown in Figure 2, inwhichCPE is the constant phase element and *n* is the phase shift which can be explained as a degree of the surface inhomogeneity [38,39]. CPE is commonly used instead of double layer capacitance (C_{dl}) to achieve a more accurate fit to the experimental results [40].

Figure 2 show that the impedance behaviour of mild steel in 1 M HClpresents a marked change in the impedance response for the concentrations of LHs. It is important to note that the impedance profiles did not alter with the change in concentration, which suggests a similar corrosion inhibition mechanism on the mild steel surface. The addition of Schiff base to the aggressive solution results in an increase in the diameter of the semicircle. This enhancementbecomes more and more altered with the increase in the inhibitor concentration.



Figure 2: An Electrical Equivalent Circuit Diagram for the Modeling of Mild Steel/Solution Interface



Figure 3: EIS-Fitted Data for HLs in 1 M HCl Solution Using the Equivalent Circuit Model Diagram in Figure 2

From table 4, it was found that the Rp values increases and the CPE values decreases with the increase in the inhibitor concentration. The increase in Rp values suggests the formation of aprotective film at the metal/solution interface. Moreover, the decrease in CPE is due to the decrease in the local dielectric constant and/or an increase in the thickness of the electrical double layer at electrode surface, therefore enhancing corrosion resistance of mild steel. The decreasein theCPE values isrelated to the gradual replacement of water molecules by the inhibitor molecules on the mild steel surface, and decreases the number of active sites necessary for the corrosion reaction [41]. The increase in the inhibitor

concentration increaseRpbut decreases CPE, which suggests that the LHs Schiff base acts as a primary interface inhibitor and controls the corrosion charge transfer of mild steel under open circuit conditions. Figure 2 suggests that the Nyquistplotshows depressed semicircleinstead perfect semicircles. This deviation may be attributed to the frequency dispersion of the interfacial impedance. This anomalous phenomenon is due to the inhomogeneous electrode surface arising from the surface roughness or interfacial phenomena [27-29]. The impedance results show a good correlation as found in both Tafel polarization and weight loss methods.



Figure 4: The Nyquistand (Log Freq) – (Log z) Plots for Mild Steel in 1.0 M HCl Solution with the Presence of the Corrosion Inhibitors for HL₁ at Room Temperature



Figure 5: The Nyquistand (Log Freq) – (Log z) plots for Mild Steel in 1.0 M HCl Solution with the Presence of the Corrosion Inhibitors for HL₂ at Room Temperature

ADSORPTION ISOTHERM

The interaction of the inhibitor molecule with the active sites on the metal surface can be explained from the adsorption isotherms[42].Various isotherms, such as the Funkin, Temkin, Freundlich, Foly-Huggins and Langmuir [43-45], have been tested but the best fit was achieved with the Langmuir isotherm for the Schiff base on mild steel. The Langmuir isotherm is presented by equation (7),[42]:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + c \tag{7}$$

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Where K_{ads} is the equilibrium constant of the adsorption process, denoting the interaction of the inhibitor with the metal surface, θ is the surface coverage and *C* is the inhibitor concentration. The linear relationship of *C*/ θ versus θ in Figure 6, having the values of correlation coefficient of (0.99 and 0.97) which are close to unity, suggests that the adsorption of HLs on MS surface in 1.0 M HCl acid solution obeys Langmuir adsorption This model assumes that the solid surface contains a fixed number of adsorption sites and that each site holds one adsorbed species [3]. The constant K_{ads} is related to the standard free energy of adsorption ΔG_{ads}^0 by the following equation(8):

$$\Delta G^o_{ads} = -RT \ln(55.5 K_{ads}) \tag{8}$$

Where R is the universal gas constant8.3142 JK⁻¹mol⁻¹, and T is the temperature in Kelvin. The value of 55.5 is the concentration of the water in the solution in moles per liter [46].The large negative values of ΔG^{o}_{ads} indicatea spontaneous adsorption process, as well as the stability of the adsorbed inhibitor layer on the solid surface [47]. In addition the negative value of ΔG^{o}_{ads} represents a strong interaction between the HLs molecules and mild steel surface. [48]. Literature review suggests that the values of ΔG^{o}_{ads} around -20 kJ mol⁻¹ or lower (more positive) are consistent with a physisorption (electrostatic interaction between inhibitor molecules and metal) phenomenon, while those around or higher (more negative) than -40 kJ mol⁻¹ demonstrates the charge shearing between the adsorbent and adsorbate (chemisorption) [49,50]. The calculated values of ΔG^{o}_{ads} for investigated inhibitor, HL₁ and HL₂ are -34.03 and 33.32 kJ/mol, respectively as listed in table 5, which suggests that the adsorption mechanism of these inhibitors on mild steel surface in 1.0 M HCl solution involve a complex combination of both physical and chemical adsorption [51].



Figure 6: Langmuir Adsorption Plots of Mild Steel in 1 M HClwith Various HLs Concentrations

Generally, it is expected that both protonated and protonated species of HLs could exists in acidic medium. As a result of electrostatic attraction, there may be the adsorption of the cationic species on the cathodic sites of the MS and reducing hydrogen evolution, hence shielding the cathodic sites of steel surface. Increasing the inhibitor concentration may increase the protonated species on metallic surface [52], thereby a decrease in the cathodic currents and limits the decrease in the anodic currents as well.

Inhibitors K _{ads} (M ⁻¹)		$ \begin{array}{c c} \Delta \mathbf{G}_{ads} & \Delta \mathbf{G}_{ads} \\ \mathbf{M}^{-1} & (\mathbf{kJmol}^{-1}) \end{array} $	
HL1	16.66 x 103	-34.03	0.99
HL2	12.50 x 103	-33.32	0.99

 Table 5: Langmuir Adsorption Isotherm Parameters of Inhibitors for MS in 1 M HCl at 298 K

Scanning Electron Microscope (SEM) Measurements

The SEM analysis of the electrode surfaceprovides the effect of the Schiff base on the corrosion process of MS.Figure7 represents an array of SEM images of the abraded mild steel surfaces exposed for 6 h in 1 M HCl solutions with and without inhibitors at the maximum concentration of 50 ppm at room temperature. Figure 7 (b) represents the SEM examination of MS sample in uninhibitedHCl solution. This imagestrongly suggests that the metal surface wasseverely damaged in the absence of inhibitor, and resulted in the metal dissolution in acidic solution. While Figure 7(c-d) shows that the presence of the inhibitor decreased the damaged of metal surface, where the corrosion rate was suppressed due to the formation of a more compact protective film by the adsorption of the HLs compounds on the surface of the sample.



Figure 7: SEM Micrographs of Mild Steel Surfaces (a) Abraded Mild Steel (b) un Inhibited 1.0 M HCl (c) 50 ppm of LH₁(d) 50 ppm of LH₂

CONCLUSIONS

The corrosion inhibition efficiency of the LHs Schiff base on MS in 1 M HClwas studied by polarization, impedance and SEM analysis. Both LHs showedgood inhibition properties with the formation of acompact protective film,

and adsorption on the metal surface, there by obeying the Langmuir adsorption isotherm. The inhibition efficiency values of LH₁are higher than that of LH₂, and increases with the increase in the inhibitor concentration. The polarization curves results indicate that both inhibitors act as a mixed type inhibitor by inhibiting the anodic metal dissolution and cathodic hydrogen evolution reactions by simply blocking the active metal sites, i.e. the addition of the inhibitor did not change the corrosion mechanism of mild steel in 1 M HCl medium. The EIS results reveal that the double layer capacitance decreased and the charge transfer resistance when the inhibitor concentration was increased. All results obtained from the polarization, EIS and weight loss experiments are in good agreement, with a slight deviation in numerical values. Surface morphology reveals the formation of a protective and dense film on the mild steel surface as a result of the addition of inhibitor in the aggressive solution.

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