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Electrochemical Study on the Effect of Schiff Base Compounds as Effective Corrosion Inhibitors for Aluminum in Acid Media

¹Ghulamullah khan, ¹Kazi MD. Salim Newaz, ²Wan Jeffrey Basirun, ³Ghulam Mustafa khan and ⁴Pervaiz Ahmed

¹Department of Mechanical Engineering, Faculty of Engineering Building, University of Malaya, 50603 Kuala Lumpur, MALAYSIA.

²Department of Chemistry, Faculty of Science Building, University of Malaya, 50603 Kuala Lumpur, MALAYSIA.

³Department of Chemistry, Faculty of Science, University of Balochistan, 87300 Quetta, PAKISTAN.

⁴Department of Physics, Faculty of Science, University of Malaya, 50603 Kuala Lumpur, MALAYSIA.

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ABSTRACT

The corrosive inhibition and adsorption effects of two newly synthesized Schiff bases namely, 3-(5-methoxy-2-hydroxybenzylideneamino)-2-(5-methoxy-2-hydroxyphenyl)-2,3-dihydroquinazoline-4(1H)-one, and 2-(3,3-Dimethyl-2,3-dihydro-indol-2-ylidene)-3-[(2-hydroxyphenyl)-imino] propanal (abbreviated as SBCI-1 and SBCI-2, respectively) on the corrosion of aluminum in 1M HCl were investigated by numerous electrochemical approach including weightloss method, electrochemical measurements, and scanning electron microscopy (SEM). The achieved results point out that the synthesized Schiff base compounds are competent corrosion inhibitors for aluminum in 1M HCl solution. Polarization outcome recommend that these compounds perform anodic type inhibitor and results obtained from electrochemical analysis were in a good compliance with each other. EIS results exhibits that charge transfer resistance increased as a result of increasing inhibitor concentration. The adsorption of inhibitor molecules on aluminum surface comply Langmuir adsorption isotherm. The results obtained from free energy of adsorption recommend that the adsorption mechanism of synthesized Schiff base compounds was spontaneous.

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INTRODUCTION

Aluminum and its alloys are extensively used in domestic and industrial applications such as tank interiors, household appliance, automobiles, ship hulls, off shore structure, submerged pipelines and electrical wirings (Markley *et al.*, 2007, Musa *et al.*, 2011 and Ashassi-Sorkhabi *et al.*, 2006). The anti-corrosion properties of aluminum is due to the development of a strongly adherent, compact, non-porous and continuous oxide film on the surface when exposed to atmospheric or aqueous environments, however, in some cases, this oxide film may be damaged or dissolved when exposed in the presence of high concentrations of acids or bases.

It is generally accepted that acids solutions are widely used pickling, descaling, cleaning and electrochemical etching of aluminum (Agrawal and Namboodhiri, 1990). 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 (Khan *et al.*, 2015

and El-Etre, 2003). Literature proposed that organic compounds containing heterocyclic organic compounds containing oxygen, nitrogen, phosphorus, and sulphur atoms along with multiple bonds in their molecular structure have the ability to donate lone pair electrons, are effective corrosion inhibitors for aluminum and steel (Ebenso *et al.*, 2008, Quraishi *et al.*, 2001, Kumar, 2002 and Safak *et al.*, 2012). Generally, the strong anti-corrosion properties of organic compounds in acidic medium is due to the adsorption on the metal surface via bond formation between the lone pair of electrons and/or the pi-electron cloud with the metal (Emregül and Atakol, 2004). Researchers have reported several Schiff base compounds as excellent corrosion inhibitors for the corrosion protection of copper, steel and aluminum (Shokry *et al.*, 1998, Bilgic & Caliskan, 2001 and Desai *et al.*, 1986). Literature review reveals that the presence of $-N=C-$ group in Schiff bases enhance their corrosion inhibition efficiency and adsorption ability as compared to corresponding aldehydes and amines (Obot and Obi-Egbedi, 2011).

The objective of the current study is to evaluate the corrosion inhibition efficiencies of two newly synthesized Schiff bases for the corrosion protection of aluminum at various concentrations in 1 M HCl solution at room temperature. The approaches used in this study were weight loss measurements, electrochemical techniques and scanning electron microscopy (SEM).

2. Experimental methodology:

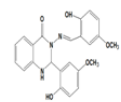
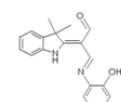
2.1 Electrode fabrication:

The working electrode was a disc cut from a commercial aluminum sheet and mounted in polyester so that only 2.56 cm² surface area was exposed to the aggressive solution. The working electrode were mechanically abraded by emery papers in successive grades from 800, 1000, 1200

and 1500, followed by rinsing double-distilled water, CCl₄, and finally degreased with acetone respectively. For each test, a freshly abraded electrode and a clean set of electrode was used. The HCl solution was prepared with dilution of an analytical grade of concentrated acid with deionized water. All the tests were performed in a concentration range of 25-100 ppm in 1 M HCl at room temperature without stirring.

2.2 Inhibitors:

Both Schiff bases were characterized and synthesized from the previous published report (Faraj, 2014). The molecular structure, abbreviations and the corresponding IUPAC names of the synthesized Schiff base compounds are given in Table 1.

Inhibitor	Abbreviation	Structure
3-(5-methoxy-2-hydroxybenzylideneamino)-2-(5-methoxy-2-hydroxyphenyl)-2,3-dihydroquinazoline-4(1H)-one	SBCl-1	
2-(3,3-Dimethyl-2,3-dihydro-indol-2-ylidene)-3-[(2-hydroxyphenyl) imino] propanal	SBCl-2	

2.3 Weight loss measurements:

Aluminum specimens of 2.0 cm x 2.0 cm x 0.001 cm after abrasion, washing and degreasing were dried and kept in a desiccator. The specimens after weighting were immersed in 1M HCl solution with and without the Schiff bases. After 24 h of exposure, the specimens were taken out, rinsed thoroughly with deionized water, dried and weighted accurately.

The corrosion rate was calculated using the following equation:

$$C_R = \frac{W_1 - W_2}{A \cdot t} \quad (1)$$

Where, W_1 and W_2 represents average weight of specimens before and after exposure. A is the total available surface area of specimens in cm² and t is immersion time in h.

The values of surface coverage (θ) and inhibition efficiency $\eta_{WL}(\%)$ were calculated using the following equations:

$$\theta = \frac{C_R^0 - C_R}{C_R^0} \quad (2)$$

$$\eta_{WL}(\%) = \frac{C_R^0 - C_R}{C_R^0} \times 100 \quad (3)$$

Where θ is the surface coverage, $\eta(\%)$ and C_R^0

and C_R are the corrosion rates in the absence and presence of the inhibitors.

2.4 Electrochemical measurements:

The electrochemical behavior of the aluminum samples in the absence and presence of the inhibitors was investigated by a potentiostat/galvanostat (AUTOLAB PGSTAT30, Netherlands). The experimental data were analyzed with a Pentium IV computer with GPES software, by the recording anodic and cathodic Tafel curves in 1 M HCl solutions with various inhibitor concentrations. A conventional three electrode cell was utilized in these experiments, with the aluminum (2.56 cm²) as the working electrode, a platinum wire and Ag/AgCl as the auxiliary and reference electrodes, respectively. The working electrode was first immersed into the test solution for 30 seconds to establish a steady state open circuit potential (E_{ocp}).

Potentiodynamic polarization curves were obtained with a scan rate in the potential range between -1000 to -200 mV at 5 m Vs⁻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 relation:

$$\eta(\%) = 1 - \frac{i_{corr}}{i_{corr}^0} \times 100 \quad (4)$$

Where, i_{corr}^0 and i_{corr} denotes the corrosion current densities (mA/cm^2), in the absence and presence of the inhibitors, respectively.

The AC frequency range was between 10^5 to 3×10^2 Hz, with a sine wave of 5 mV around the E_{OCP} . The inhibition efficiency $\eta_{RP}(\%)$ was calculated from the polarization resistance as shown in equation (5):

$$\eta_{RP}(\%) = \frac{R_p - R_p^0}{R_p} \times 100 \quad (5)$$

Where, R_p and R_p^0 are the polarization resistances of the inhibited and uninhibited solutions, respectively.

2.5 Scanning Electron Microscope (SEM):

The aluminum specimens with dimensions of 2 cm x 2 cm x 0.001 cm were immersed in 1.0 M H_2SO_4 solution for 24 h in the presence and absence of Schiff base inhibitor, at room temperature. The surface morphological changes of the specimens

were investigated by scanning electron microscopy (SEM), with Carl Zeiss LEO SUPRA 50 VP, at an accelerating voltage of 10 KeV.

3.1 Weight loss measurements:

The weight loss experiments were carried out in a glass cell in 1.0 M H_2SO_4 with the absence and presence of different concentration of the Schiff base inhibitors (25-100 ppm) at room temperature for 24 h immersion. Table 2 summarizes the values of corrosion rate (CR), surface coverage (θ) and inhibition efficiency $\eta_{WL}(\%)$, obtained from gravimetric method. Table 2 suggests that, the increase in inhibitor concentration in the aggressive solution decreases the corrosion rate of aluminium efficiently, while the surface coverage and inhibition efficiency increases. The decrease in weight loss (i.e CR) with the increase of the Schiff base inhibitor indicates that the corrosion inhibition process occurs due to the adsorption of the inhibitor molecules on the metal surface (Obot and Obi-Egbedi, 2011).

Table 2: Corrosion parameters obtained from gravimetric measurements on aluminium in 1 M HCl solution with various concentrations of Schiff bases at room temperature.

Concentration (mM)	SBCH-1			SBCH-2		
	CR ($\text{mg cm}^{-2} \text{h}^{-1}$)	θ	η_{WL} (%)	CR ($\text{mg cm}^{-2} \text{h}^{-1}$)	θ	η_{WL} (%)
Blank	9.3	-	-	9.3	-	-
0.01	6.4	0.45	45	6	0.55	55
0.03	5.6	0.66	66	5.5	0.69	69
0.05	5.4	0.72	72	5.2	0.78	78
0.07	5.1	0.82	82	5	0.86	86

3.2 Potentiodynamic polarization:

In Fig.1, the anodic and cathodic plots were recorded on aluminum electrode in 1M HCl with the presence and absence of different concentration of Schiff bases at room temperature. The

electrochemical parameters such as corrosion potential (E_{corr}), anodic and cathodic Tafel slopes (b_a and b_c), corrosion current density (i_{corr}) and corrosion inhibition efficiency $\eta_p(\%)$ were calculated and listed in table 3.

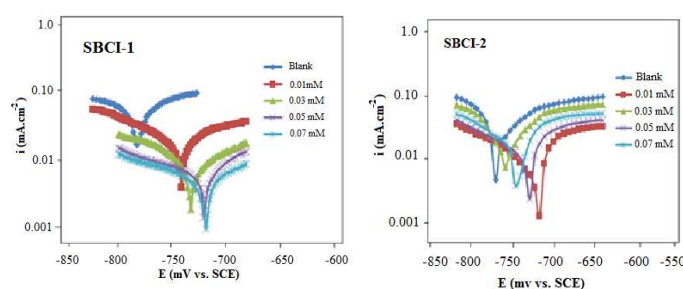


Fig. 1: Tafel polarization curves for aluminum in 1 M HCl solution with and without the optimum concentration of Schiff bases at room temperature.

From Fig 1, it can be seen that the addition of Schiff base to the aggressive solution decreases both anodic metal dissolution and cathodic hydrogen evolution reactions with the increase of the inhibitor concentration. This result suggests that the inhibitor molecules adsorbed on the active sites of the aluminum surface (solmaz, 2011). Also from table 3, the corrosion current densities decreases with the

increase in inhibitor concentration, while the inhibition efficiencies increases as would be expected. The data from the table 3 shows that the b_a values are more or less the same in the presence and the absence of the Schiff base inhibitors, whereas the value of b_c is nearly the same in the presence of SBs, indicating that the addition of the Schiff base

inhibitors does not modify the mechanism of the corrosion process.

Table 3: Potentiodynamic polarization parameters and the corresponding corrosion inhibition efficiency on aluminum in 1 M HCl in the absence and presence of various concentration of Schiff bases at room temperature.

Inhibitors	Concentration (mM)	- E _{corr} (mV vs. SCE)	ba (V/dec)	bc (V/dec)	i _{corr} (μAcm ⁻²)	η _P (%)
	Blank	0.767	0.126	0.121	9.5	-
SBCI-1	0.01	0.745	0.092	0.113	3.9	58
	0.03	0.731	0.076	0.098	2.7	71
	0.05	0.718	0.063	0.086	2.2	76
	0.07	0.711	0.054	0.071	1.1	88
SBCI-2	0.01	0.755	0.109	0.114	7.3	23
	0.03	0.741	0.092	0.091	5.2	45
	0.05	0.733	0.084	0.079	2.1	77
	0.07	0.724	0.071	0.065	1.4	85

Table 3 shows that the corrosion potential values are shifted toward anodic region in the range of 56 - 43 mV. If the displaced value of corrosion potential is higher than 85 mV to the corresponding values in the blank solution, then the inhibitor can be classified as anodic or cathodic type of inhibitor (Noor, 2009). In the present work, the maximum displacement observed was 56 mV and 43 mV for SBCI-1 and SBCI-2 respectively, which strongly suggests that the

Schiff bases may be classified as mixed type inhibitor in HCl solution, with a predominantly anodic formation of a protective surface film.

3.3 Electrochemical Impedance Spectroscopy:

The Nyquist plots on aluminum in the presence and absence of various concentration of Schiff base inhibitors in 1 M HCl are given in Fig 2.

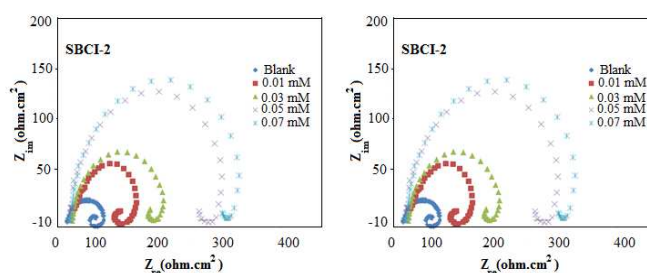


Fig. 2: Nyquist plots for aluminum in 1 M HCl at room temperature at various concentration of Schiff bases.

It can be seen clearly from Fig. 2, that the Nyquist plot shows an apparent increase in the total impedance with the increase of the inhibitor concentration. The Nyquist plot of aluminum shows the existence of a large depressed semicircle capacitive loop at higher frequency region, followed by a small inductive loop at lower frequencies. The presence of a high frequency capacitive loop is attributed to the relaxation process in the aluminum oxide film, and the low frequency inductive loop is attributed to the initially passivated surface, which starts to re-dissolve at lower

frequency range (Benali *et al.*, 2007). From Fig. 2, it is clear that, the deviation from the ideal semicircle is due to the frequency dispersion, which is attributed to surface roughness, impurities, adsorption of inhibitors and inhomogeneities on the aluminum surface (Aljourani *et al.*, 2009 and Oguzie *et al.*, 2007). Figure 2 shows that the increase in inhibitor concentration increases the diameter of the depressed semi circle, which may be attributed to the adsorption of the Schiff base inhibitor on the aluminum surface.

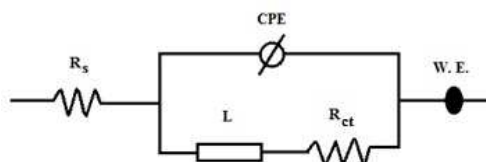


Fig. 3. Equivalent circuit diagram to fit EIS data for aluminum in 1 M HCl solution.

Figure 3 shows the electrical equivalent circuit diagram to model aluminum/HCl interface which

consists of a constant phase element (CPE), charge transfer resistance (R_{ct}), solution resistance (R_s) and

an inductor (L) due to the presence of an inductive loop. In general, the CPE is required for the distribution of the relaxation time, as a result of surface inhomogeneities and (n) represents the degree

of surface inhomogeneity (Navarro-Flores *et al.*, 2005 and Okafor *et al.*, 2010).

Table 4: EIS parameters and inhibition efficiencies on aluminum in 1 M HCl solution, with the presence and absence of various concentration of Schiff bases at room temperature.

Inhibitors	Concentration (mM)	CPE (10^{-4}) (F)	n	Rct (Ω)	$\eta\%$ (%)
	Blank	2.007	0.87942	107.282	
SBCI-1	0.01	1.957	0.93578	164.129	35
	0.03	1.454	1.03174	244.769	56
	0.05	1.309	1.03518	362.261	70
SBCI-2	0.07	0.995	0.96935	670.057	84
	25	1.556	0.93971	124.429	14
	50	0.851	1.05247	172.971	37
	75	0.813	1.02136	335.887	68
	100	0.735	1.03315	591.221	82

The values are tabulated in Table 4, which suggest that the Rct and inhibition efficiency increase with the decrease of CPE, which increases with the Schiff base concentration, due to the formation of a protective film at the aluminum surface, as a barrier against the charge and mass transfer process. The increase of Rct is attributed to the adsorption of the inhibitor molecules on the aluminum/HCl interface, which decrease the corrosion rate of the aluminum surface.

3.4 Adsorption isotherms:

Adsorption isotherms can provide important information on the interaction of inhibitors and corresponding active sites on the metal surface. For this purpose, the surface coverage values (θ) for different inhibitor concentration were calculated to present the best adsorption isotherm. Several attempts were made to fit the (θ) values to the standard isotherms including Langmuir, Temkin, Freundlich Frumkin and Floy-Huggins (Okafor *et al.*, 2009, Obot & Obi-Egbedi, 2010, Abdel-Gaber *et al.*, 2006 and Torres *et al.*, 2011). A plot of C versus C/θ (Fig. 4) gives a straight line which suggests a Langmuir adsorption isotherm. This isotherm can be expressed as (Ashassi-Sorkhabi *et al.*, 2004):

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (6)$$

Where C is inhibitor concentration, K_{ads} is the adsorption equilibrium constant, relative to the interaction of the inhibitor with the metal surface. Larger adsorption equilibrium constant represents a

higher adsorption tendency of the Schiff base inhibitors on the aluminum surface. The standard free energy of adsorption of the Schiff base inhibitor on the aluminum surface can be calculated from the following equation (Ashassi-Sorkhabi *et al.*, 2004):

$$\Delta G_{ads}^{\circ} = -RT \ln(55.5 K_{ads}) \quad (7)$$

The calculated values of ΔG_{ads} for the investigated inhibitors are -43.6 kJmol^{-1} (SBCI-1) and -42.3 kJmol^{-1} (SBCI-2) respectively shown in table 5. These results reveal that the adsorption process of the inhibitors on the aluminum surface is chemisorption, indicating a strong adsorption due to the charge sharing between the SB_s molecules and the aluminum surface. The negative value of the free energy of adsorption indicates a spontaneous adsorption process and strong interaction between the Schiff base molecules and the aluminum surface (Daoud *et al.*, 2014 and Avci, 2008). It is generally accepted that the values of the Free Energy around -20 kJ mol^{-1} or less are consistent with a physical adsorption process (an electrostatic interaction between the charged inhibitor and the charged metal surface), while those around -40 kJ mol^{-1} or higher, is associated with a chemical adsorption process (involves the transfer or sharing of electrons between the inhibitor molecules and the metal surface, resulting in a coordinated covalent bond) (Bayol *et al.*, 2008). The calculated values of the free energy of adsorption indicates that the adsorption process of the Schiff base compounds on the aluminum surface in acid media at room temperature are both physical and chemical adsorption processes.

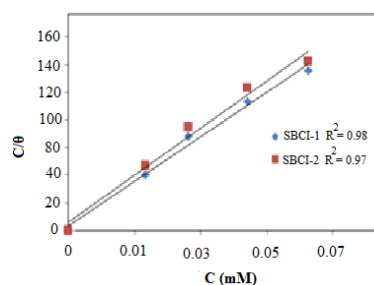


Fig. 4: Langmuir adsorption isotherm of Schiff base inhibitors on aluminum in 1M HCl at room temperature.

Table 5: Thermodynamic parameters for the adsorption of Schiff bases on the aluminum surface immersed in 1 M HCl at optimum concentration at room temperature.

Inhibitors	K_{ads} (M^{-1})	ΔG_{ads} ($kJmol^{-1}$)	R^2
SBCI-1	17.54×10^3	-43.6	0.98
SBCI-2	13.08×10^3	-42.3	0.97

3.5 SEM analysis of electrode surface:

SEM imaging was performed to establish a link between the experimental parameters and the morphology of the aluminum surface. Figure 5(a, b) represents the surface image of the abraded sample before and after immersion in 1M acid solution for 24 h, whereas Fig. 5(c, d) are the images of the aluminum surface in the presence of optimum concentration of Schiff bases. It can be observed from Fig. 5(b) that the abrasion and scratches are clearly visible on the surface, due to the attack of the

aggressive solution in the absence of inhibitors. The photomicrograph of the aluminum surface in the presence of Schiff bases is shown in Fig. 5(c, d), which represents a smooth surface due to the formation of a protective layer by the inhibitors. These results indicate that the presence of Schiff base inhibitors hinders the dissolution of the aluminum due to the formation of a protective film formed by inhibitor molecules on the surface, therefore decreases the corrosion rate of aluminum in 1M HCl solution.

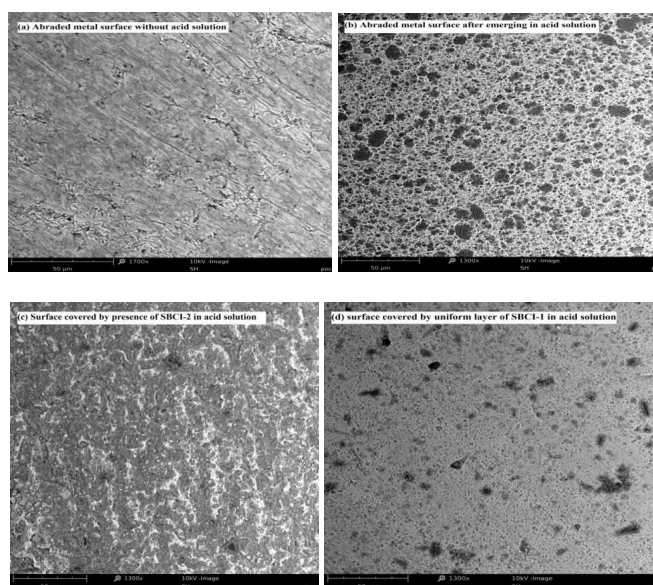


Fig. 5: SEM micrographs for aluminum samples at room temperature; (a) after immersion in 1M HCl without immersion in acid solution, (b) after 24 h immersion in HCl solution without inhibitor, (c) after 24 h immersion in 1 M HCl containing 0.03 mM of SBCI-2, (d) after 24 h immersion in 1 M HCl containing 0.07 mM of SBCI-1.

Conclusions:

Schiff base corrosion inhibitors are efficient corrosion inhibitors for aluminum in 1M HCl and the inhibition efficiency of SBCI-1 is greater than SBCI-2. The polarization curves obtained spectacle that the Schiff base inhibitors are mixed-type inhibitors with a predominantly anodic effect. The adsorption of Schiff base compounds on aluminum surface is spontaneous and follows the Langmuir adsorption isotherm. The achieved values of free adsorption energy, acknowledge that the adsorption mechanism of Schiff base compounds is chemical adsorption. Surface morphology photographs confirm the formation of a dense and uniform protective film on aluminum surface in the presence of Schiff base compounds.

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