



Electrochemical study of *Aspilia africana* leaf extract for combating corrosion of mild steel corrosion in acid solutions

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Abstract

The work studied the performance of *Aspilia africana* leaf (AAL) extract in retarding the corrosion of mild steel substrates in 0.5 M H₂SO₄ and 1.0 M HCl acid solutions. The inhibition performance and efficiency of the extract was studied using electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PDP) measurements. The results of the study indicated that AAL extract effectively inhibited the dissolution of mild steel in the said acid solutions. The inhibition efficiencies of the plant extract increased as the concentration of the leaf extract increased. The leaf extract exhibited the maximum inhibition efficiency $\eta(\%)$ of 98.30% and 60.82% at 700mg/L in 0.5 M H₂SO₄ and 1.0 M HCl respectively using electrochemical impedance spectroscopy, while potentiodynamic polarization measurements reveal maximum inhibition efficiency $\eta(\%)$ of 81.25% and 54.55% at 700 mg/L in 0.5 M H₂SO₄ and 1.0 M HCl acid media respectively. The Tafel plots of the PDP showed that the inhibitor functioned as a mix-type inhibitor in which a decreased corrosion current density resulted as extract concentration was increased, suggesting an inhibiting effect. The AAL extract was found to retard corrosion via adsorption of the extract organic molecules on the mild steel and the blocking of the active sites by hindering dissolution of metal at the anode and evolution of hydrogen at the cathode.

Keywords: Corrosion inhibition, inhibitor efficiency, corrosion rate, *Aspilia africana*, desorption.

Introduction

Among the metals and alloy metals, mild steel appears to be the most widely used in industrial applications, apparently because of its relative cheapness and weld-ability when compared to stainless steel¹⁻³. Consequent upon its wide range of applications, mild steel is highly affected by corrosion when it comes in contact with corrosive acid media especially during industrial routine processes such as cleaning or pickling with acid, removal of scales and petrochemical processes⁴⁻⁶. Corrosion is a natural phenomenon whereby a metal or its alloy product deteriorates when the material reacts electrochemically with its surroundings⁷⁻⁹. The problem of corrosion has adverse effects on the economy of any nation as it causes loss of efficiency of industrial plants, breakdown of equipment and machineries, loss of lives, oil spillage from petroleum pipelines with its attendant environmental pollution, as well as huge cost of control¹⁰. One of the best and ecofriendly ways to suppress corrosion of mild steel is to effectively separate it from aggressive (corrosive) environments by the use of corrosion inhibitors harnessed from naturally occurring substances. Extensive research activities in recent times have revealed that extracts from leaves, flowers, seeds, roots, nuts and barks of certain plants have excellent inhibiting effects that protect mild steel as well as other metals and their alloy products from acid attack^{4,6,11-17}. The success of plant extracts in retarding the corrosion of metallic substrates is accounted for by the presence in them of complex bioactive organic molecules present in them

^{6,18,19}. These organic molecules contain heteroatoms such as O, S, and N and conjugated π -bond systems in their chemical structures with active sites that enable the adsorption of these molecules on to the substrates thereby inhibiting corrosion^{6,18,19}. The present study seeks to investigate the performance and effectiveness of AAL extract in retarding the corrosion and dissolution of mild steel substrates in two different acidic solutions namely, 0.5 M H₂SO₄ and 1.0 M HCl acid solutions using electrochemical experiments. *Aspilia africana* is a semi woody herb growing in the savannah and tropical regions of Africa²⁰.

It is known by different local names by different indigenous Nigerian population. It is known in Igbo as *Orangila*, in Hausa as *Tozalin* and in Yoruba as *Yunyun*. A review of literature has revealed that *Aspilia africana* possesses some medicinal and pharmacological properties^{21,22}. Reports by research scholars have equally shown that the plant possesses wound healing effect on account of its n-hexane and methanolic extracts^{21,23}. Phytochemical profiling of the plant has shown that it is sufficient in saponins, tannins, glycoside and alkaloids^{24,25}.

Methodology

Preparation of Test Coupons: Samples of mild steel were prepared in form of coupons dimensioned 1.5cmx1.5cm. The coupons were mechanically cleaned and polished with emery paper, and further covered with epoxy resin seal in a manner

that only a surface area of 1.0cm² was left exposed. Oil and grease particles were removed from the exposed surface using acetone, rinsed with double-distilled water and dried in warm air.

Preparation of Plant Extract: Matured leaves of *Aspilia africana* were harvested from the live plant from the bush in the premises of Federal University of Technology, Owerri. The leaves were subjected to thorough washing with clean water, dried in a natural circulation solar dryer at the Physics Department of the University and crushed into fine powder. Weighed quantities of the powdered leaf sample were boiled in dilute acid solutions of required molarity in a reflux apparatus for 3 hours. The resulting stock solutions were cooled to room temperature and then filtered.

The filtrate was weighed and the volume of stock solution measured to determine the weight of the plant extract contained therein. It is with the stock solutions that the inhibitor test solutions containing serial concentrations of AAL extract were prepared.

Electrochemical Experiments: Electrochemical measurements were conducted with a PARC Parsat-2273 Advanced Electrochemical System in a conventional three-electrode glass cell of capacity 400 ml. A foil of platinum served as courier electrode while a saturated calomel electrode (SCE) served as reference electrode, which was connected via a Luggin's Capillary. Electrochemical impedance measurements were taken after 2 hours of immersion and at corrosion potential (E_{corr}) over a frequency range of 100 kHz – 10 mHz, with a signal amplified perturbation of 5 mV. The data were collected using power sine software, also supplied by PARC. Potentiodynamic polarization measurements were taken in the potential range ± 250 mV relative to corrosion potential (E_{corr}) at a scan rate of 0.333mVs⁻¹.

The corrosion current densities (I_{corr}) were obtained by extrapolating the linear Tafel segments of anodic and cathodic curves to corrosion potential. The experiments were carried out in each of the aggressive test solutions (0.5 M H₂SO₄ and 1.0 M HCl) containing the metal coupons at room temperature with and without the plant extracts. The concentration of the plant extracts used varied from 100 mg to 700 mg in the aggressive test solutions.

Results and discussion

Electrochemical Impedance Spectroscopy in 0.5 M H₂SO₄ solution: The results obtained from the electrochemical impedance spectroscopy (EIS) measurements for the mild steel substrates in 0.5 M H₂SO₄ solutions without and with AAL extract for an exposure period of 2 hours are displayed in the form of complex Nyquist impedance plots, Bode plots and phase angle plots (Figure-1, 2, 3).

Table-1: Electrochemical parameters deduced from EIS measurements for dissolution of mild steel in 0.5 M H₂SO₄ without and with AAL extract for exposure time of 2 hours.

AAL extract concentration (mg/L)	R_s (Ωcm^2)	R_{ct} (Ωcm^2)	$\frac{1}{R_{ct}}$ ($\times 10^{-3}$) ($\Omega^{-1}\text{cm}^{-2}$)	C_{dl} (μFcm^{-2})	N	n%
Blank	1.73	90	11.11	25	0.90	-
50	1.64	400	2.50	6	0.92	77.50
700	1.38	680	1.47	3	0.87	86.77

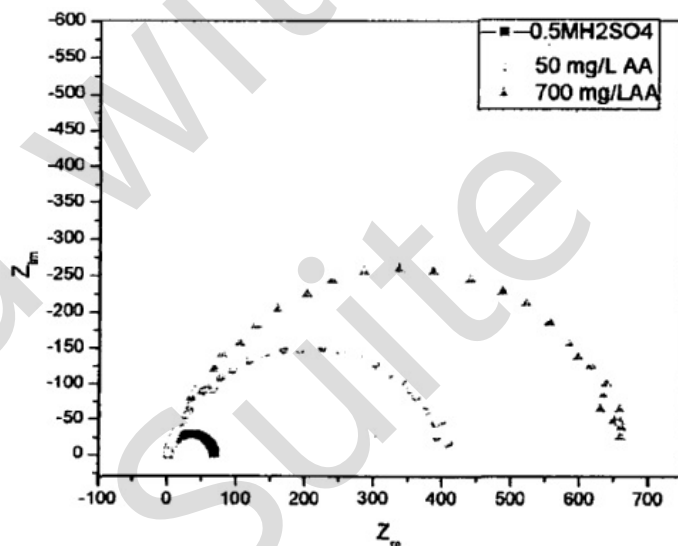


Figure-1: Nyquist plots for dissolution of mild steel in 0.5 M H₂SO₄ solution without and with AAL extract for exposure time of 2 hours.

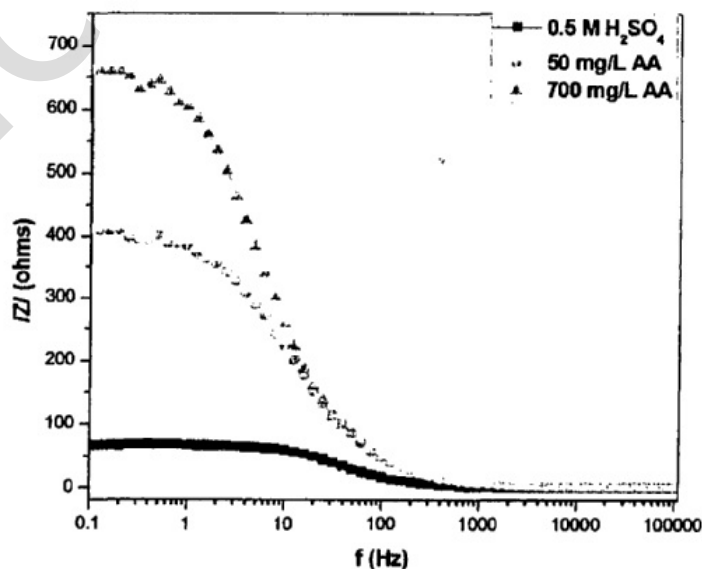


Figure-2: Bode plots for dissolution of mild steel in 0.5 M H₂SO₄ solution without and with AAL extract for exposure time of 2 hours.

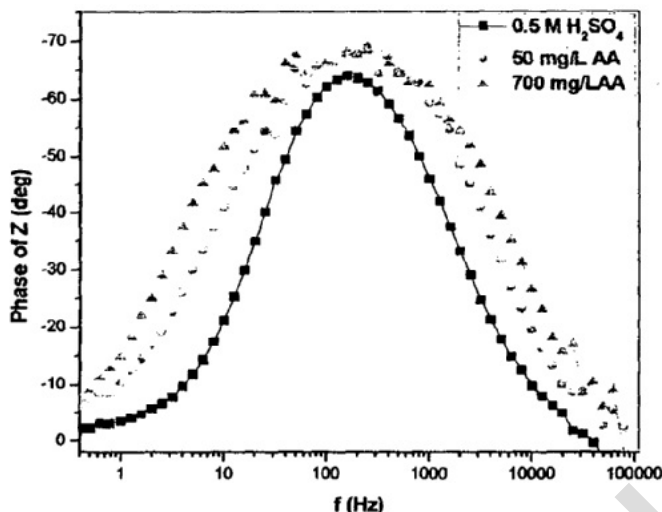


Figure-3: Phase angle plots for dissolution of mild steel in 0.5 M H₂SO₄ solution without and with AAL extract for exposure time of 2 hours.

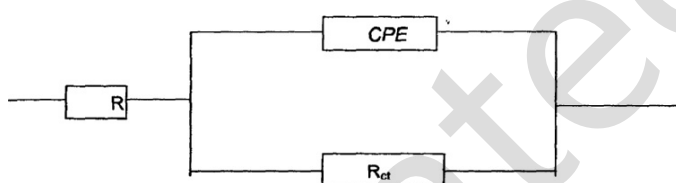


Figure-4: The electrochemical equivalent circuit suitable for modeling the impedance spectra.

The Nyquist plot shows relationship between the real component Z'_{real} and imaginary component Z''_{im} of the systems impedance over a wide frequency range (in this case 100 kHz – 10 mHz). In the Nyquist plots of Figure-1, each impedance spectrum appears as a depressed semi-circle which represents a capacitive loop at high and intermediate frequencies. It corresponds to one time constant in the Bode plots (Figure-2). The diameter of the capacitive loop depends on the value of the charge transfer resistance, R_{ct} , at the mild steel-solution interface. The charge transfer resistance, R_{ct} , is described as the limiting zero frequency value of the real part of the complex impedance of the electrochemical system during the alternating current impedance measurements of the substrate corrosion in the acid medium with or without an inhibitor. According to electrochemical theory, the reciprocal of R_{ct} is proportional to the corrosion rate²⁶. The Nyquist plot shows that the diameter of a capacitive loop increases when an AAL extract is added to the aggressive medium and that the greater the extract concentration the greater the diameter of the semi-circular loop. This means that the charge transfer resistance, R_{ct} , increased when the AAL extract was added to the aggressive solution and also implies that, R_{ct} increases as the dosage of the AAL extract increases.

Hence, the addition of the AAL extract decreases the corrosion rate. The impedance spectra shown in Figure-1 were further analyzed by seeking a fit between a model electrochemical equivalent circuit shown in Figure-4 with the impedance spectra. The model equivalent circuit consists of a solution resistance, R_s , in series with a parallel connection of the charge transfer resistance, R_{ct} , and a constant phase element (CPE). The latter is introduced in the model since the metal/solution interface is far from ideal and is capacitive in nature. It is characterized by a double-layer capacitance, C_{dl} , and a parameter, n , which indicates the level of surface in homogeneity and is indicative of the degree of non-ideality in its capacitive behaviours. The value of C_{dl} was calculated at a frequency at which the imaginary component of impedance is a maximum¹⁵, that is;

$$F(-Z_{max}^*) = \frac{1}{2\pi C_{dl} R_{ct}}$$

So that,

$$C_{dl} = \frac{1}{2\pi F R_{ct}}$$

The values of the electrochemical parameters and the inhibition efficiency deduced from the Nyquist impedance plots from the electrochemical impedance spectroscopy measurements are shown in Table-1. The inhibition efficiency, $n\%$, of the AAL extract mitigating the corrosion of the steel substrate in the acid solution was calculated using the equation;

$$n\% = \frac{R_{ct(inh)} - R_{ct}}{R_{ct(inh)}} \times 100$$

The table reveals that the addition of AAL extract in 0.5 M H₂SO₄ solution brought about an increased charge transfer resistance and hence, a decreased corrosion rate. It also leads to a decreased double layer capacitance, C_{dl} . Both corrosion rate and double layer capacitance decrease at higher extract concentration. The table also shows that the inhibition efficiency of AAL extract was improved as its dosage increases.

Electrochemical Impedance Spectroscopy in 1.0 M HCl solution: With mild steel substrate in 1.0 M HCl without and with the AAL extract, the electrochemical impedance spectroscopy measurements were also taken after 2 hour exposure time. Figure-5, 6, 7 show the Nyquist plots, Bode plots and phase angle plots respectively.

Table-2: Electrochemical parameters deduced from EIS measurements for dissolution of mild steel in 1.0 M solution of HCl without and with AAL extract for exposure time of 2 hours.

AAL extract concentration (mg/L)	R_s (Ωcm^2)	R_{ct} (Ωcm^2)	$\frac{1}{R_{ct}}$ ($\times 10^{-3}$) ($\Omega^{-1}\text{cm}^{-2}$)	C_{dl} (μFcm^{-2})	N	$n\%$

Blank	1.15	250.3	4.00	10	0.89	-
50	1.04	339.3	2.95	7	0.87	26.23
700	1.19	638.9	1.57	4	0.85	60.82

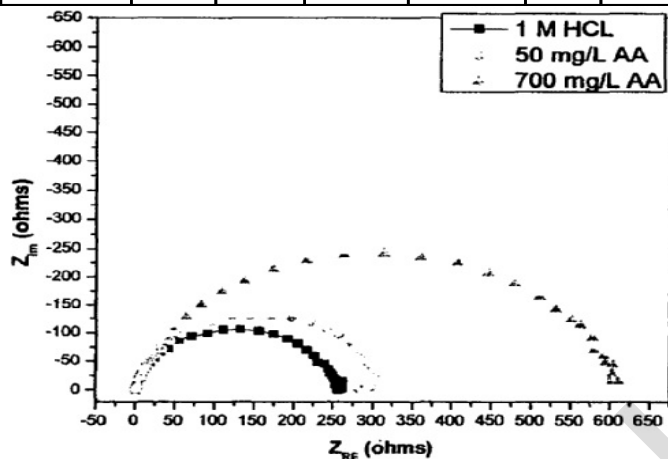


Figure-5: Nyquist plots for dissolution of mild steel in 1.0 M HCl solution without and with AAL extract for exposure time of 2 hours.

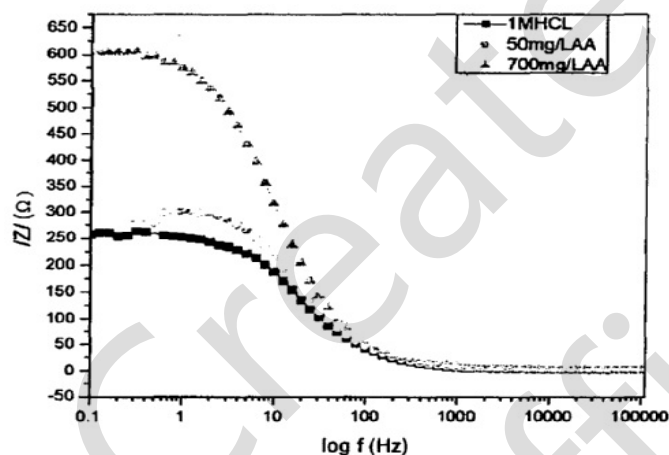


Figure-6: Bode plots for dissolution of mild steel in 1.0 M HCl solution without and with AAL extract for exposure time of 2 hours.

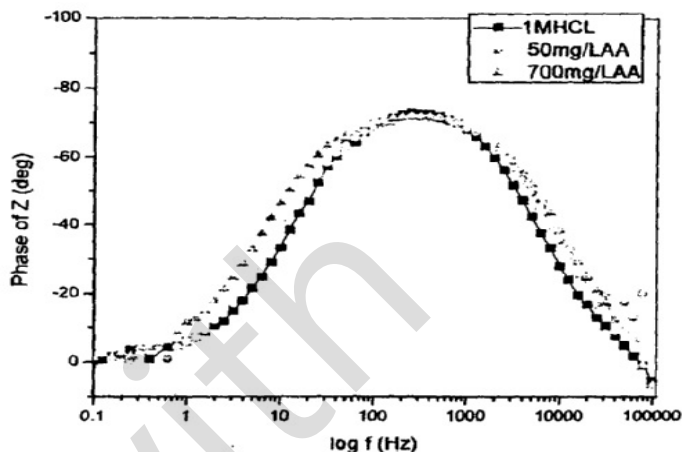


Figure-7: Phase angle plots for dissolution of mild steel in 1.0 M HCl solution without and with AAL extract for exposure time of 2 hours.

The electrochemical parameters denoted R_s , R_{ct} , C_{dl} , and n as well as the protection efficiency ($\eta\%$) of the AAL extract deduced from the Nyquist impedance spectra are shown in Table-2. The results show that the charge transfer resistance, R_{ct} , increases with the introduction of the AAL extract and the increase in the extract concentration leads to a further increase in R_{ct} . This is interpreted to mean that the presence of AAL extract resulted to a decreased corrosion rate of the medium and that increasing the AAL extract concentration further decreases the corrosion rate. In addition, the addition of AAL extract decreases the capacitance of the double layer at the substrate/acid solution interface. Table-2 also indicates that the protection efficiency $n\%$, of the AAL extract increases as the extract concentration increases.

Potentiodynamic Polarization Measurements in 0.5 M H_2SO_4 solution: The results of the potentiodynamic polarization (PDP) measurements taken for the test substrates in 0.5 M H_2SO_4 solutions without and with AAL extract are displayed in the form of polarization curves or Tafel plots as shown in Figure-8.

Table-3: Electrochemical parameters deduced from PDP measurements for the corrosion of mild steel in 0.5 M solution of H_2SO_4 without and with AAL extract for exposure time of 2 hours.

AAL extract concentration (mg/L)	E_{corr} (mV)	I_{corr} (μAcm^{-2})	$n\%$
Blank	-491	320	-
50	-480	110	65.62
700	-481	60	81.25

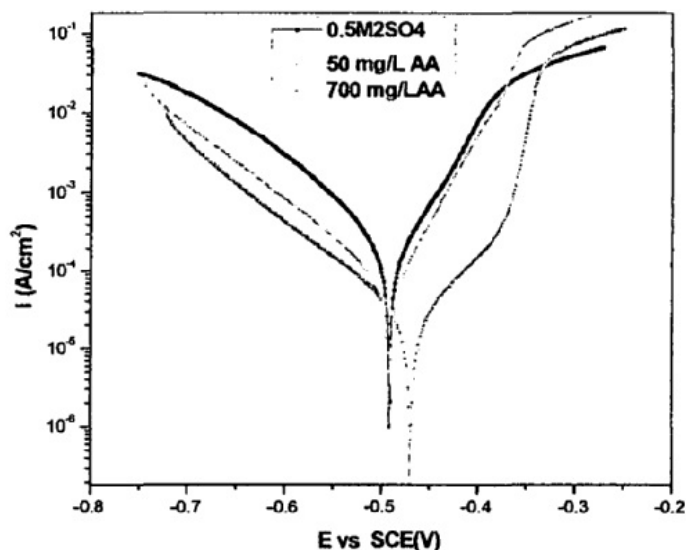


Figure-8: Potentiodynamic polarization curves obtained for mild steel dissolution in 0.5 M solution of H₂SO₄ without and with AAL extract for exposure time of 2 hours.

The plots show anodic and cathodic current densities versus electrode potential relative to the standard calomel electrode (SCE). Figure-8 shows that the introduction of AAL extract into the corrosive test solutions produced a reduction in both the anodic and cathodic current densities, the decrease becoming more pronounced with the increase in the extract concentration. This suggests that the introduction of the AAL extract reduces both anodic dissolution and gas evolution at the cathode. Moe of the corrosion potential, E_{corr} , can be observed to shift slightly to the positive, that is, it becomes less negative relative to the SCE. Table-3 shows the electrochemical parameters determined from the potentiodynamic polarization measurements. They include the corrosion potential (E_{corr}) relative to SCE and the corrosion density (I_{corr}). The corrosion inhibition efficiency, $n\%$, of the AAL extract is also deduced in the table according to the equation;

$$n\% = \frac{I_{corr} - I_{corr(inh)}}{I_{corr}} \times 100$$

Clearly, the current density decreases as the concentration of the AAL extract increases, implying a corrosion resistive effect. Moreover, the efficiency of inhibition by the AAL extract increased with higher extract dosage.

Potentiodynamic Polarization Measurements in 1.0 M HCl solution: Figure-9 shows the polarization curves obtained for mild steel dissolution in 1.0 solution of HCl without and with AAL extract.

Table-4: Electrochemical parameters deduced from PDP measurements for the corrosion of mild steel in 1.0 M solution of HCl without and with AAL extract for exposure time of 2 hours.

AAL extract concentration (mg/L)	E_{corr} (mV)	I_{corr} (μAcm^{-2})	$n\%$
Blank	-496	110	-
50	-487	74	32.72
700	-478	50	54.55

The addition of the AAL extract affects both the anodic and cathodic polarization curves and causes the corrosion potential to be slightly more positive. Table-4 shows that the current density is decreased when a small amount of AAL extract is added. It further decreases with increase in AAL extract concentration. These results suggest that the introduction of the AAL extract reduces the corrosion rate since reduction in current density implies decrease in corrosion rate. In effect, AAL extract exhibits a corrosion inhibition effect on mild steel in 1.0 M solution of HCl.

The results of the electrochemical experiments revealed that the introduction of AAL extract led to an increase in the charge transfer resistance and a decrease in the double layer capacitance which became more pronounced upon the increased concentrations of the inhibitors. This implies that an increase in the dosage of the inhibitor gave rise to a corresponding reduction in the corrosion rate of the mild steel specimens *via* adsorption on to the mild steel interface thereby saving the substrates from the attack of the corrodents. It is also observed from the findings, that increasing the concentration of AAL extract resulted to an increase in the size of the semi-circle in Figure-1 and 5 in the impedance of the interface and in the maximum phase angle in Figure-3 and 7 which reveals a mitigation in the corrosion reaction. Also, from the Tafel plots in Figure-8 and 9, the AAL extract added to the acid solutions functioned as mix-type inhibitor in which there is a drop in corrosion current density at higher concentration of AAL extract, suggesting an inhibiting effect.

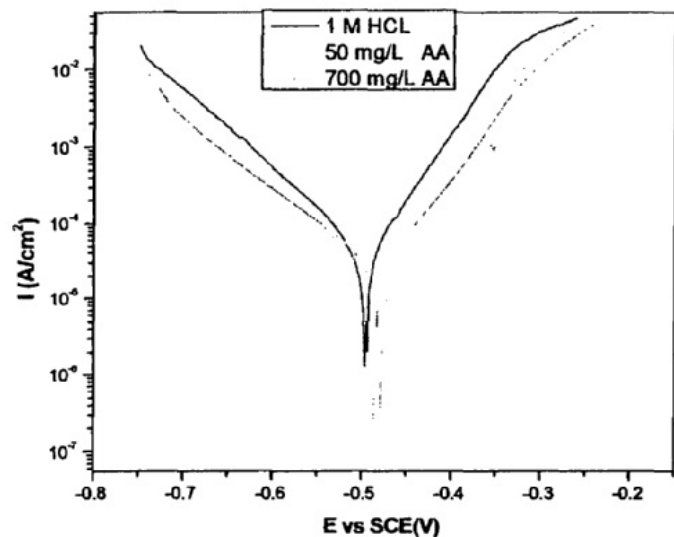


Figure-9: Potentiodynamic polarization curves for mild steel dissolution in 1.0 M HCl solution without and with AAL extract for exposure time of 2 hours.

In addition to the findings, it was discovered that AAL extract contains a mixture of complex organic compounds of plant origin. Phytochemical analysis of the plant extract performed in the previous studies revealed that the AAL contains alkaloids, saponins, flavonoids, phenols, essential oil and tannins²¹, as well as mineral constituents including calcium, magnesium, potassium, iron, sodium and phosphorus. Many of the phytochemical constituents of the leaf extract are among the organic compounds of plant origin which have proven capability to inhibit metal corrosion as mentioned in the introduction. It is therefore not an aberration to ascribe the observed suppression of the corrosion of the mild steel to the presence of these complex organic compounds in the aggressive test solutions. Moreover, it has been shown that some of the metal ions contained in the AAL extract synergistically enhance the inhibition of metal corrosion²⁷. However, it is not possible to identify which of the phytochemical compounds contained in the AAL extract is actually contributing the molecules that were adsorbed on to the coupon's surface.

Conclusion

With the results of this study, it has been established that AAL extract effectively retarded the dissolution of mild steel in 0.5M and 1.0 M solutions of H₂SO₄ and HCl respectively. The inhibition efficiency of the plant extract determined from the various tests increased with increased as the dosage of the extract increased. The AAL extract was found to retard corrosion *via* adsorptive interaction of the organic molecules in the extract with the mild steel and the blocking of the active sites by hindering dissolution of metal at anode and liberation of hydrogen at cathode. The Tafel plots revealed that the AAL extract functioned as a mix-type inhibitor in which there is a

decreased corrosion current density as the extract concentration increased, suggesting an inhibiting effect.

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