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Corrosion Inhibitive Effect of Psidium Guajava Leaves on Mild Steel in an Induced Alkaline Solution

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Abstract Corrosion inhibition of mild steel by Guava Leaves (*Psidium guajava*) Extract in varying Irish Potato (*Solanum tuberosum*) Extract induced NaOH solution was studied using gravimetric and electrochemical (potentiodynamic polarization) methods. Material characterization was carried out using Fourier Transform infrared spectroscopy (FTIR), and Scanning Electron Microscope (SEM). The inhibition was found to increase with an increase in Guava Leaves Extract (GLE). The effect of immersion time, temperature, and the concentration of Sodium Hydroxide (NaOH) solution on the corrosion behaviour on mild steel were also studied. The inhibition occurred by adsorption of the inhibitors molecules on the surface of the mild steel samples, which obeys the Langmuir adsorption isotherm. It was observed that the adsorption of the extracts on the mild steel samples was a spontaneous process. Potentiodynamic polarization (PDP) showed that GLE acts as a mixed-type inhibitor while FTIR revealed O-H and N-H as the main functional groups responsible for the inhibition exhibited by the extracts. SEM showed the layer of film formation which was due to physical adsorption of the extracts molecules on the surface of the samples. The PDP and gravimetric analysis of inhibition efficiency show 64.16% and 89.0% respectively. GLE is a good corrosion inhibitor in IPE induced NaOH solution medium.

Keywords Induced alkaline solution, Guava leaves extract, Irish potato extract, Corrosion current density, Tafel plot, polarization

Introduction

According to Smith and Hashemi [1], corrosion is the deterioration of a material; mostly metal, due to its reaction with the environment. The rate at which metal corrode depends upon the environment and the type of metal. The corrosion of metal can be very rapid in a highly corrosive environment or takes years in a slightly corrosive environment. Almost all metallic corrosion is electrochemical in nature. However, a corrosion cell consisting of an anode, a cathode, and an electrolyte must exist [2].

Aesthetical values and mechanical strength of metallic materials are always compromised during their interaction with different environments such as atmosphere, aqueous solution, solids, acids and bases, inorganic solvents, molten salts, liquid metals, a human body. This leads to material degradation and has resulted in the shutdown of many industrial plants, power plants, refineries and cumulatively caused excessive economic loss [3-4]. Most finite elemental analysis of failed metallic storage tanks revealed that the mechanical integrity of the tanks is compromised and the load carrying capacity of the tank had been drastically reduced [5].

Corrosion products formed on degrading material surfaces have become deadly contaminants in pharmaceutical, dye and packaged food industries and at the long run jeopardize the health of the consumers of these products [6]. As such, due to the life-threatening consequences that corrosion brings, the protection of metallic materials has attracted the attention of researchers. Although there are evolving methods to control corrosion, the use of inhibitors is one of the best methods for protecting metals against corrosion [7]. Most of the corrosion inhibitors are synthetic chemicals which are expensive and very hazardous to environments. As a result of the toxicity of some corrosion inhibitors, there has been an increasing search for green corrosion inhibitors [8].

The use of plant extracts as corrosion inhibitor has become important because they are environmentally acceptable, readily available and are a renewable source for a wide range of green inhibitors. Works of literature revealed that plant leaves extract such as those from *Moringa oleifera*, *Carica papaya, Dodonaea viscose, Carica papaya* and *Camellia sinensis, Baphia nitida, Psidium guajava, Musa paradisiacal, Azadirachta indica, Centella asiatica,* have been studied for corrosion inhibition of mild steel in acidic media [9-17]. Oil extracts from *Arachis hypogeae*, *Cyperus esculentus*, *Sesamum indicum* had been proven to exhibit good corrosion inhibition on mild steel in acidic media [18-20]. Also, the use of peels from some plants such as *Musa sapientum,* Aqueous extract of *Mangifera indica* and *Citrus aurantium* peel, *Theobroma cacao,* was identified as a good corrosion inhibitor on mild steel. Seed husk and roots of some plant were also studied and the result showed good corrosion inhibitor property. For instance, the corrosion inhibition study of *Jathrpha curcas* seed husk and *Vernonia amygdalina* root showed that both were good corrosion resistance in mild steel in acidic media [21-22].

However, the advantages of green corrosion inhibitor have not been translated into effective usage, which could be as a result in the research gaps in terms of inappropriate usage of medium concentration against industrial concentration, and need to synergize the adsorption rate. The study examined the corrosion inhibitive effect of *Psidium guajava* leaves extract on mild steel in an induced NaOH solution.

Methodology

materials such as mild steel plate (3 mm thickness), guava leaves, ethanol, acetone, sodium hydroxide pellet, Irish Potato Tubers, reagents (distilled water, Mayer's reagent, Aqueous NaOH, $FeCl_3$ reagent, acetic anhydride, copper sulphate solution) were used and the equipment used are; Gallenkamph hotbox oven, Desiccator, Test Tubes (50 ml), Measuring cylinder (100 ml), Volumetric flask (2000 ml), Soxhlet extractor, Water bath, Sortorius analytical balance, AutoLab Potentiostat electrochemical system workstation, filter paper, venier caliper, paper sieve, masking tape, Cary 630 FTIR spectroscopy, Phenom Prox Scanning Electron Microscope

Preparation of Samples

The elemental chemical composition analysis of the Mild steel sample was carried out as shown in table 1 at National Metallurgical Development Centre, Jos, Nigeria. The mild steel was mechanically press cut into twenty-nine (24 mm x 14 mm x 3 mm) coupons and a smooth surface was obtained using 600-1200 grades of emery paper and then de-greased with acetone and air dried.

Table 1: Chemical Composition of Mild Steel Sample

Elements	C	Si	P	Cu	Pb	Fe
% composition	0.08	0.05	1.00	0.02	0.02	98.83

Preparation of *Guava Leaves Extracts (GLE)*

Leaves of *Psidium guajava* were collected rinsed and air dried. The dried guava leaves were crushed into smaller particles. 50 g of the crushed leaves was refluxed using Soxhlet extractor in 500 ml of ethanol, the extract and the solvent were separated using the distillation process. The solution was evaporated inside a ceramic bowl at 50^{o}C.

Preparation *Irish Potato Extract (IPE)*

Irish potato (*Solanum tuberosum*) tubers show strong antioxidant capacity and also a rich source of phenolic acids, flavonoids, folates, phytates and carotenoids [23]. Irish Potato extract (from potato starch processing waste) contains about 5% dry matters [24]. Protein (patatin, protease inhibitors, other protein) accounts for approximately 25-30% of the dry matter [25]. Protein in Potato juice extract contains both hydrophobic and hydrophilic components owing to their amino acids when added to two immiscible medium, decreasing the interfacial tension between the two medium [26].

500 g of Irish potato (*Solanum tuberosum*) tubers were collected, peeled, rinsed and allowed to drain. The drained peeled potatoes were then cut into smaller sizes and mechanically compressed using a pressurized liquid extractor to extract the juice. The turbid extract was allowed to settle for 15 minutes. Then, the liquid was decanted and centrifuged for 15 minutes at room temperature, the supernatant filtered through a paper filter.

Phytochemical Analysis of the Extracts

The extracts were subjected to phytochemical screening to detect the presence of secondary metabolites such as alkaloids, flavonoids, tannins, saponin and protein using reagent such as; Meyer's, aqueous NaOH, acetic anhydride, 1% $FeCl_3$ and copper sulphate solution.

Gravimetric Corrosion Measurement

Gravimetric analysis was carried out on pre-weighed mild steel samples which were immersed in 20ml of blank and induced NaOH solution in the presence and absence of inhibitor, maintained at 30^{o}C, 50^{o}C and 70^{o}C for 3hours in a thermostat controlled water bath. The weight loss of each coupon was determined at every 1hr intervals by retrieving the samples from the solution, clean with acetone and then reweighed; the difference in weight was taken as weight loss. From the weight loss data collated, Corrosion Rate (CR), the inhibition efficiency (I.E %) of the inhibitor, and the degree of surface coverage (θ) were calculated using equations 1 to 3 respectively (ASTM G1 standard) ;

$$\mathrm{CR(mm/y)} = \frac{87{,}600\,\mathrm{w}}{\mathrm{A\rho t}} \quad \ldots \quad (1)$$

Where, CR is corrosion rate, w is weight loss (g), A is the area of the coupon in cm^2 and t is time (hr), ρ is the density (g/cm^3).

$$\mathrm{I.E\,\%} = \left(1 - \frac{\mathrm{CR_{inh}}}{\mathrm{CR_{blank}}}\right) \mathrm{x}\ 100 \quad \ldots \quad (2)$$

Where I. E is the Inhibition Efficiency, $\mathrm{CR_{inh}}$ and $\mathrm{CR_{blank}}$ correspond to the corrosion rates in the presence and absence of inhibitor respectively.

$$\theta = \left(1 - \frac{\mathrm{CR_{inh}}}{\mathrm{CR_{blank}}}\right) \quad \ldots \quad (3)$$

Where θ is the surface coverage, $\mathrm{CR_{inh}}$ and $\mathrm{CR_{blank}}$ correspond to the corrosion rates in the presence and absence of inhibitor respectively.

Electrochemical Corrosion Measurement

Potentiodynamic polarization examination was carried out on four representative samples in 12.5 M concentration of NaOH solution in the presence and absence of inhibitor at room temperature using a potentiostat. The electrochemical test was conducted using the AutoLab Potentiostat electrochemical system workstation with a three-electrode corrosion cell. A Platinum rod and a saturated Ag/Ag electrode were used as a counter and reference electrodes, respectively. The working electrode (samples) were positioned at the glass corrosion cell kit fixed in epoxy resin with a surface area of 100 mm^2 exposed to the test solution. The polarization curves were determined by stepping the potential at a scan rate of 0.003 V/sec. The polarization curves were plotted using the Autolab data acquisition system and both the corrosion rate and potential were estimated by the Tafel extrapolation method.

Electrochemical measurements were carried out in aerated and unstirred solution within 2 to 7 minutes of immersion, which allowed the open circuit potential (OCP) to attain a steady state. The temperature was maintained at room temperature. The inhibition efficiency was determined using equation (4).

$$IE\% = \frac{i_{cu} - i_{ci}}{icu} * \frac{100}{1} \quad \ldots \quad (4)$$

Where i_{cu} and i_{ci} are the corrosion current density values without and with corrosion inhibitor respectively.

Material Characterization

The nature of the film formed on the surface of the metal specimen was analyzed by Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). FT-IR spectra were recorded with a frequency ranging from 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1} for the inhibitor as well as the inhibitor adsorbed on the mild steel in aqueous solution in KBr matrix using Cary 630 FTIR spectroscopy by Agilent Technologies. The polished mild steel specimens were immersed in NaOH solutions in the absence and presence of the inhibitors. After 12hrs, the specimens were taken out and dried. The SEM photographs of the surfaces of the specimens were investigated using Phenom Prox Ultra-high resolution field emission Scanning electron microscope. The energy of the acceleration beam employed was 15 kV.

Adsorption Isotherm

The degree of surface coverage (θ) obtained from gravimetric techniques was used to evaluate the best isotherm that fits into the data obtained. Correlation coefficient obtained from the plots of various isotherms such as Langmuir, Freundlich, Temkin were used to determine the isotherms most applicable to experimental data.

Results and Discussion

Phytochemical Test

The phytochemical composition of the Guava Leave Extract (GLE) and Irish Potato Extract (IPE) showed it contains alkaloids, flavonoids, saponins, tannins etc as shown in Table 1.

Table 1: Phytochemical Constituents analysis of the extracts using ethanol as extraction medium

Extract medium	Tannins	protein	Saponins	alkaloid	Flavonoids
GLE	+	_	_	+	+
IPE	_	+	+	+	+

+ …………… Presence

- ……………. Absence

This indicates that the inhibitive property of the extract is due to the presence of some or all of the above listed phytochemical constituents[7].

Gravimetric Technique

The results generated through equation 1-3 were tabulated in table 2 and 3 and, the effect of inhibition concentration, immersion time, and the temperature was analyzed.

Effect of Inhibitor Concentration

Table 2 shows the corrosion rate and inhibition efficiency within 1 -3 hours of immersion. The result revealed that the corrosion rate decreases with an increase in inhibition concentration and also, inhibition efficiency increases with an increase in GLE concentration. This indicates that more molecules of the inhibitor were adsorbed on the mild steel surface at higher concentration leading to the formation of protective film [27]. The result also shows that the higher the surface coverage, the higher is the inhibition efficiency.

Table 2: Inhibition Efficiency of Mild Steel in 12.5 M of Induced NaOH Solution with and without GLE

Inh. Conc. (ppm)	1h			2h			3h		
	CR (mm/y)	IE (%)	(θ)	CR (mm/y)	IE (%)	(θ)	CR (mm/y)	IE (%)	(θ)
blank	43.3112	31.16	0.3116	42.4891	34.70	0.3470	37.7613	41.02	0.4102
1000	12.0041	49.35	0.4935	11.6713	53.21	0.5321	9.2331	62.46	0.6246
2000	4.0727	72.37	0.7237	3.9801	74.01	0.7401	3.1267	74.44	0.7444
3000	2.3140	80.11	0.8011	2.2159	83.05	0.8305	2.0141	84.61	0.8461

Effect of Immersion Time

The result obtained from Table 5 showing the relationship of inhibition efficiency and the immersion time in 12.5 M of an induced NaOH solution with and without GLE varying concentration. The inhibition was found to gradually increase with an increase in time due to persistence formation of protective film on the surface of the mild steel samples. The highest inhibition efficiency of 84.61% was obtained at 3 h immersion time. The plot of inhibition efficiency variation is shown in figure 1.

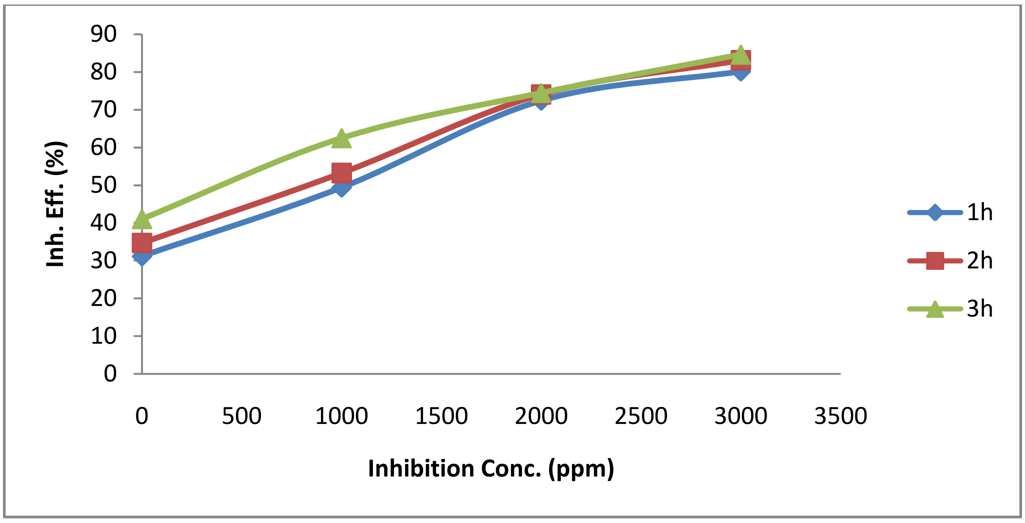

Figure 1: Inhibition Efficiency Variation with Inhibition Concentration

Effect of Temperature

Table 3 shows the effect of varying temperature on the Inhibition Efficiency of GLE in 12.5 M of induced NaOH solution. There are remarkable decreases in the corrosion rate of mild steel in the presence of GLE compared with the solution without GLE (blank) at each temperature. The inhibition efficiency also decreases with an increase in temperature which is in agreement with [28]. The plot of the effect of temperature on the inhibition efficiency of GLE is shown in figure 2.

Table 3: Inhibition Efficiency of Mild Steel in 12.5 M induced NaOH Solution in the Presence and Absence of GLE at Different Temperature

Inh. Conc.	303K		323K		343K	
(ppm)	CR (mm/y)	IE (%)	CR (mm/y)	IE (%)	CR (mm/y)	IE (%)
0	42.297	21.30	50.4089	16.7	68.1034	12.30
1000	18.8100	47.80	15.3261	39.60	14.0021	35.01
2000	7.9801	74.03	8.5159	73.05	12.8547	70.21
3000	2.8990	89.0	2.9159	83.05	2.6031	79.46

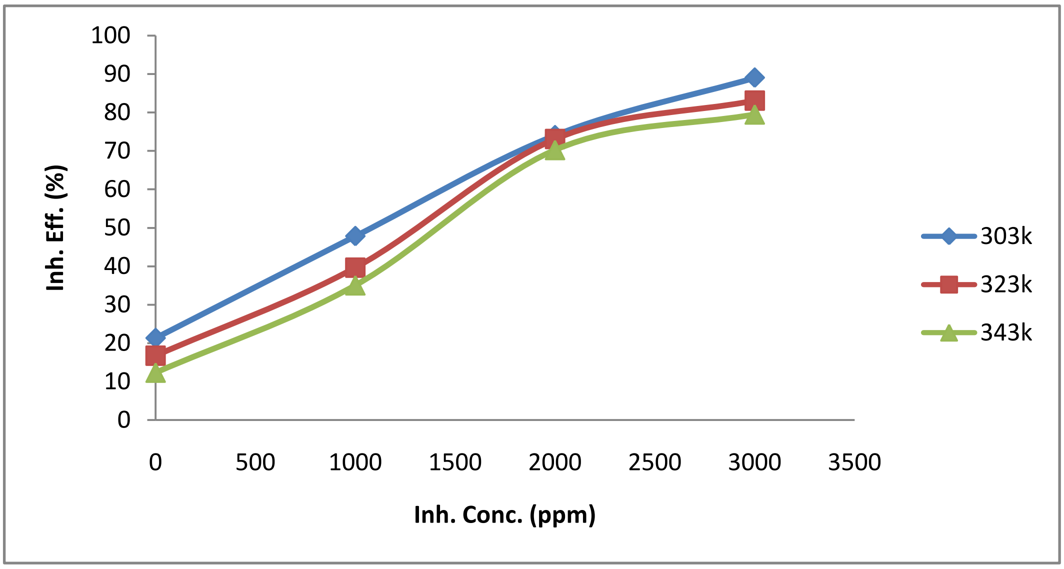

Figure 2: Variation of Inhibition Efficiency with Temperature

Potentiodynamic Polarization Techniques

The potentiodynamic polarization curves of the mild steel electrode in solution containing 12.5 M of NaOH in the absence and or presence of inhibitors combinations are shown in Figure 3. The electrochemical parameters, namely, corrosion potential (E_{corr}), corrosion current density (I_{corr}), anodic Tafel slope (β_a), cathodic Tafel slope (β_c), and percentage inhibition efficiency (IE), determined from the polarization curves were summarized in Table 4.

At a given temperature, the addition of GLE to the NaOH solution increases both the anodic and cathodic polarization resistance (R_P) and decreases the corrosion current density (I_{corr}). The shift in anodic Tafel slope (β_a) is greater than the cathodic Tafel slope (β_c), which indicates that the extracts affect the anodic reaction predominantly. The polarization curves data extrapolation showed that the corrosion current density decreases significantly with the addition of GLE, and inclusion of IPE compare with a blank solution. Thus, it is evident that GLE with or without IPE act as an effective mixed-type inhibitor.

Table 4: Tafel Parameters, Corrosion Rate and Inhibition Efficiency for Mild Steel in 12.5M NaOH Solution

Inhibitor	βa (v/dec)	βc (v/dec)	E_{corr} cal (V)	E_{corr}ob (V)	j_{corr} (A/cm^2)	i_{corr} (A)	CR (mm/y)	R_P (Ω)	IE (%)
Blank	0.5334	0.3951	-1.5923	-1.4887	0.0258	0.0257	299.25	3.8279	-
GLE	0.4177	0.1878	-0.9100	-0.8530	0.0081	0.0081	94.42	6.9249	68.45
GLE/IPE	0.3876	0.2059	-0.8585	-0.8295	0.0092	0.0092	107.25	6.3268	64.16

*blank (no inhibitor), *GLE (2000ppm of Guava Leave Extract), *GLE/IPE (2000ppm of Guava Leave Extract with 1.5ml of Irish Potato Extract)

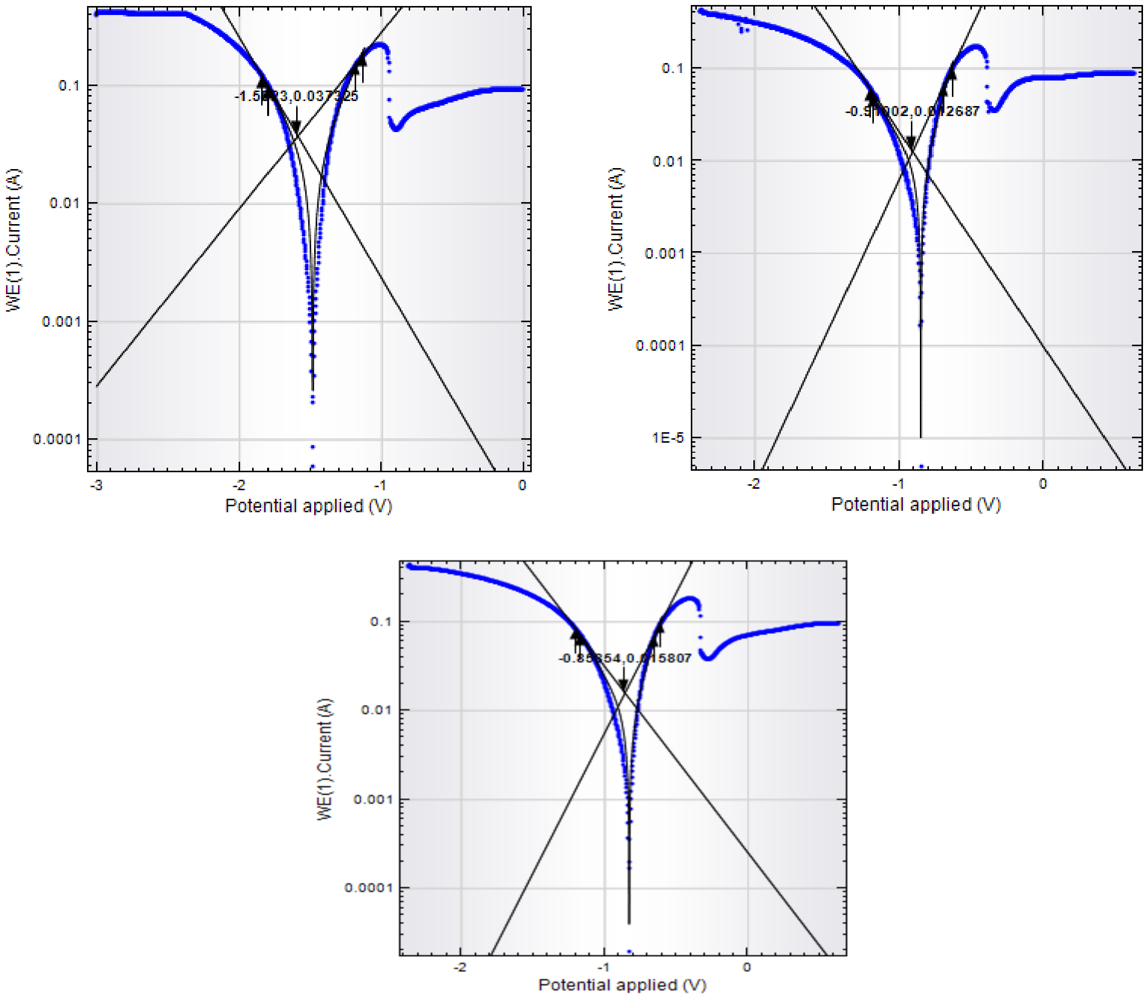

Figure 3: Potentiodynamic Polarization Plot for Mild Steel in 12.5M NaOH Solution in (a) blank solution (b) 2000ppm GLE (c)2000ppm GLE/IPE

Fourier transform infra-red

The FTIR spectra of the pure extract and corrosion product are presented in Figure 4. The peaks and their corresponding intensities represent the functional groups of the Guava leaves and Irish Potato extract. The analysis of the extracts as shown in Table 5 and 6, revealed the presence of O-H and C-H stretch in GLE while IPE revealed N-H stretch and 0-H stretch. Comparing the spectra, there is a downshift of O-H stretch from 3239.1 cm^{-1} (GLE) to 3056.4 cm^{-1} (corrosion product). Also, C-H stretch (GLE) wavelength 2922.2 cm^{-1} remain the same with the wavelength stretch of the corrosion product. N-H stretch of IPE downshifts from 1636.3 cm^{-1} to 1617.7cm^{-1} in corrosion product. The presence and the shift in the frequencies of the wavelength of O-H, and N-H functional groups of the extracts during the experiment were predominantly responsible for the corrosion inhibition process; hence, the inhibition is due to adsorption of hydroxyl and amide groups of phenolic molecules of the extracts.

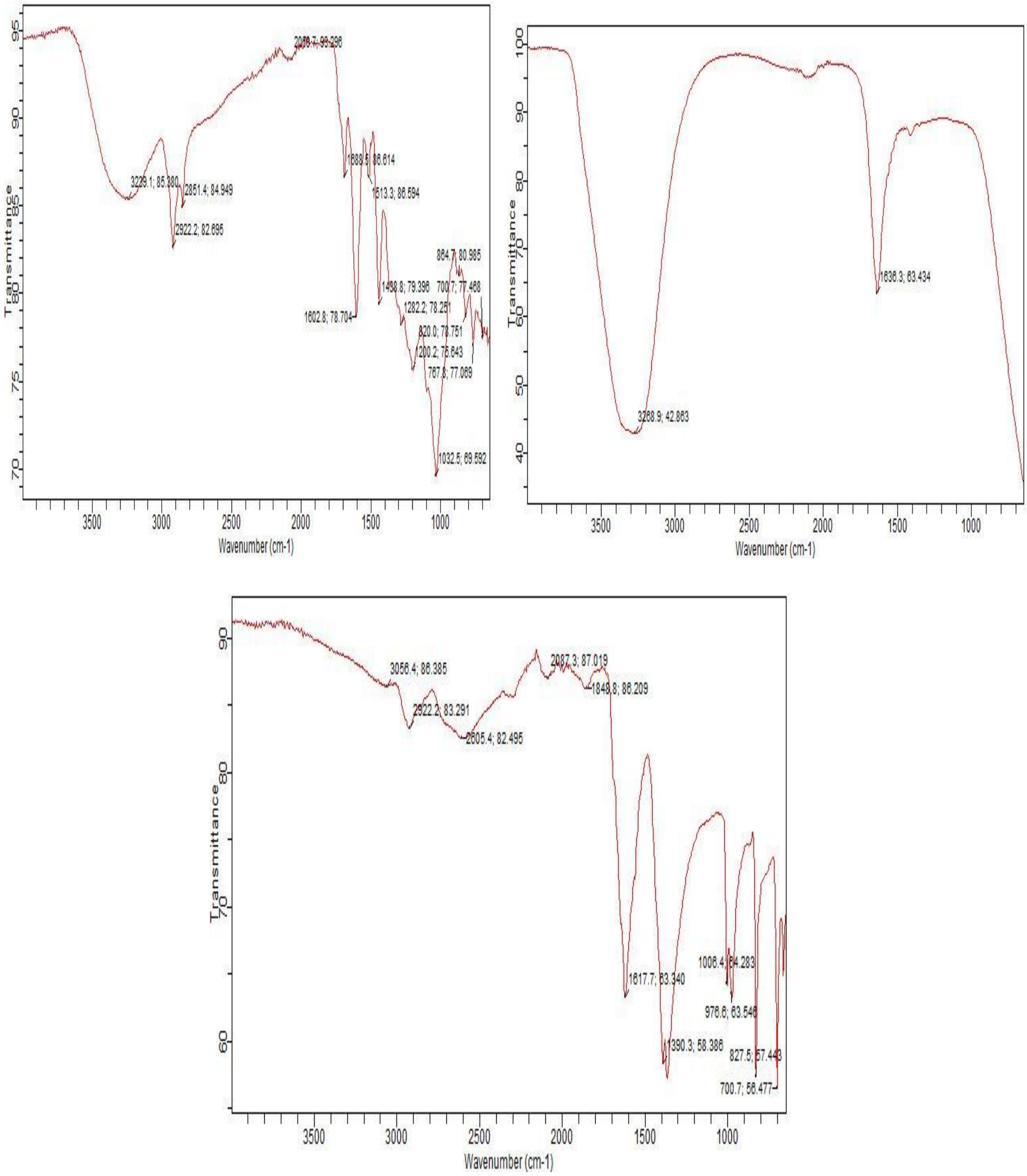

Figure 4: FTIR spectrum of the (a) Guava Leaves Extract (b) the Irish Potato Extract (c) Corrosion Product of Guava Leave and Irish Potato Extract, Transmittance versus wave number (cm^{-1}).

Table 5: FTIR spectrum analysis of the Guava leaves extract and Irish Potato extract

Guava Leaves Extract (GLE)			Irish Potato Extract (IPE)		
Peak (cm^{-1})	**Intensity**	**Assignment**	**Peak ($^{cm-1}$)**	**Intensity**	**Assignment**
3239.1	85.380	O-H stretch	3268.9	42.863	O-H stretch, N-H symmetric, ≡C-H stretch
2922.2	82.695	C-H stretch	1636.3	63.434	N-H bend, C-O stretch
2851.4	84.949	C-H stretch, O-H stretch			
1688.5	86.614	C-O stretch			
1602.8	78.704	N-H bend			

Table 6: FTIR spectrum analysis of the corrosion product (GLE/IPE)

Corrosion Product of GLE and IPE		
Peak (cm^{-1})	**Intensity**	**Assignment**
3056.4	86.385	=C-H stretch, O-H stretch, Ar C-H stretch
2922.2	83.291	C-H stretch, O-H stretch
2605.4	82.495	O-H stretch
1617.7	63.340	N-H bend

Scanning electron microscope (SEM) examinations

The SEM micrographs analysis of the representative mild steel samples at 3 hours immersion time at 70°C in 12.5 M NaOH solution in the presence and absence of GLE are shown in Plates I-III.

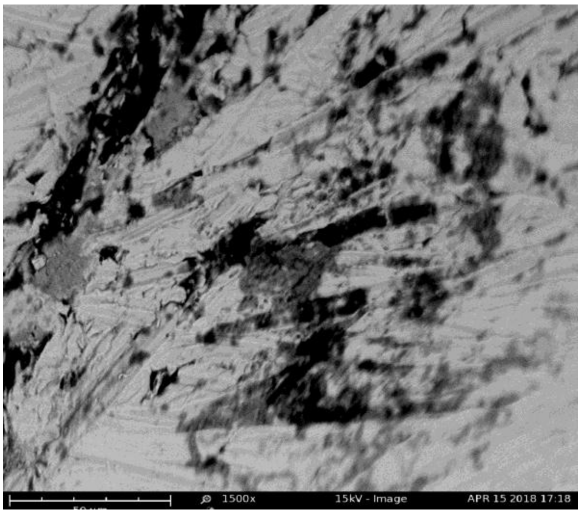

Plate I: SEM Micrograph of Mild Steel in NaOH Solution (blank)

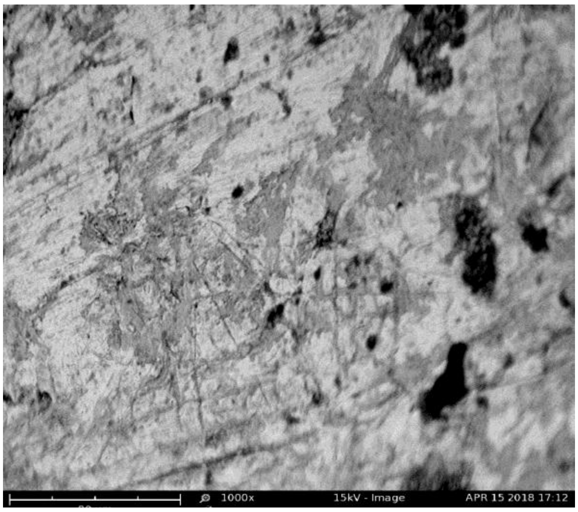

Plate II: SEM Micrograph of Mild Steel in NaOH+GLE+IPE Solution

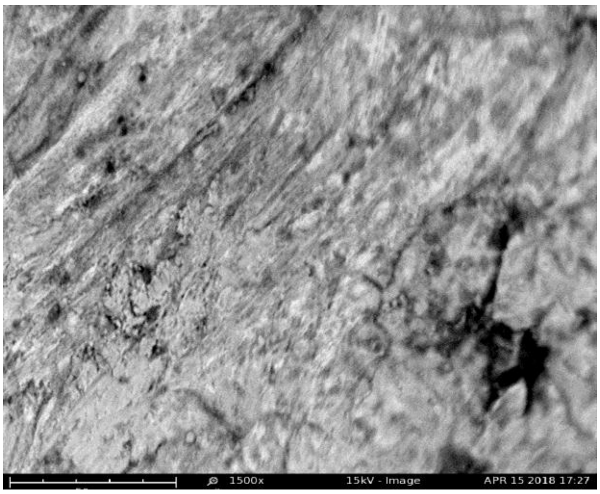

Plate III: SEM Micrograph of Mild Steel in NaOH+GLE Solution

In the comparison of SEM micrograms of the representative samples, plate I shows a rough surface of the sample which was due to the aggressive attack of NaOH solution on the mild steel. Plate II shows a smoother surface compare to plate I, this is as a result of GLE/IPE molecules reaction with the active site of the sample which formed a protective film on the sample's surface. For plate III, the presence of GLE resulted in a smooth surface with almost zero corrosion active site due to a protective layer formed on the sample surface. This infers that the inhibition of corrosion was as a result of the formation of an insoluble stable protective film by adsorption process.

Adsorption Isotherm of Mild Steel

Figure 5-7 represents the plot of Langmuir, Temkin and Freundlich adsorption. The result obtained as shown in Table 7.

The plot of $\frac{C}{\theta}$ against C yielded straight lines as shown in Figure 14 with slope a correlation coefficient (R^2) at 0.987, and K_{ads} (9.3458) was obtained. The plot obeys Langmuir adsorption isotherm as the plot has linearity and good correlation coefficient at different exposure time. Each inhibitor molecule occupies one active site on the metal surface, which implies strong adherence to Langmuir adsorption isotherm [29].

From Table 7, the negative value of the adsorption Gibb's free energy ΔG^o_{ads} indicates spontaneous adsorption of GLE on the mild steel surface which corroborates that the adsorption process is physiosorption [30].

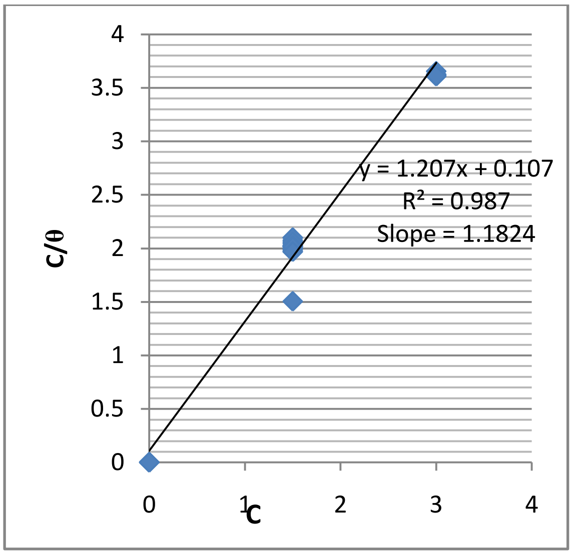

Figure 5: Langmuir Adsorption Isotherm

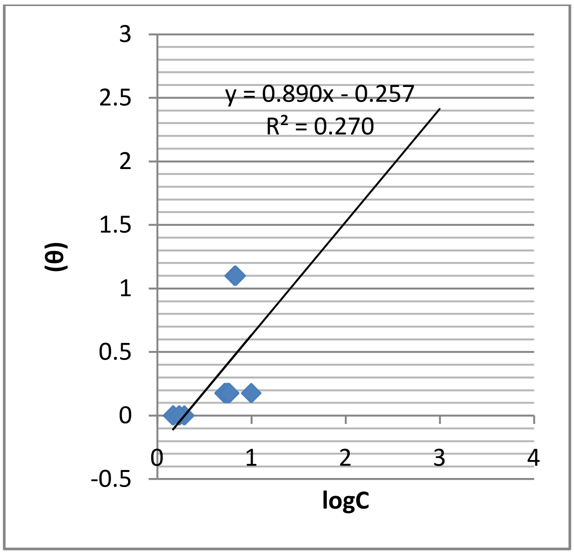

Figure 6: Temkin Adsorption Isotherm

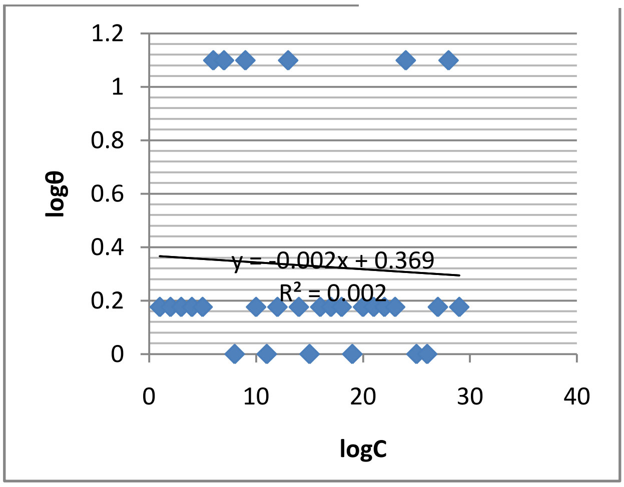

Figure 7: Freunlich Adsorption Isotherm

Table 7: Adsorption isotherm parameters obtained from the corrosion data of mild steel in *GLE*

Isotherm	Intercept	R^2	K_{ads}	ΔG^o_{ads} (kJ/mol)
Langmuir	0.107	0.987	9.3458	-15.7479
Temkin	-0.257	0.270	-3.8911	13.5405
Freundlich	0.369	0.002	2.7100	-12.6293

Conclusion

i. The phenolic agents from guava leaf and Irish potato tubers were extracted and the phytochemical analysis showed that the inhibitive property of the extracts was due to the presence of the phenolic agents which resulted in the formation of protective film on the surface of the mild steel samples

ii. The inhibition efficiency and the corrosion resistance of the mild steel increased with the increased in inhibitor concentration and time but decrease with an increase in temperature. Potentiodynamic polarization result showed 64.2% corrosion inhibition efficiency and that the extracts act as a mixed-type inhibitor. The adsorption mechanism of the extracts conformed to Langmuir adsorption isotherm and the negative value of adsorption free energy ΔG^o_{ads} indicates spontaneous adsorption of GLE on the Mild Steel. Gravimetric technique result shows 89.0% inhibition efficiency at 303K with 3000ppm inhibition concentration.

iii. Fourier Transform Infrared (FTIR) Spectroscopy showed that functional group O-H and N-H were actively responsible for the inhibition exhibited by the extract.

iv. Scanning Electron Microscopy (SEM) examination showed that there was the formation of protective film on the surface of the mild steel samples which was due to the adsorption of the extract phenolic agents molecules on the mild steel surface.

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