

Article

Jatropha gossypifolia Ethanol Extract as Corrosion Inhibitor for Galvanized Steel in 0.5M of Hydrochloric Acid

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Abstract

The study investigated the corrosion inhibition properties of *Jatropha gossypifolia* extract in 0.5 M HCl solution on galvanised steel with a view to understanding the reaction mechanisms as well as kinetics and thermodynamics of adsorption step in the corrosion reaction. The experimental investigation was conducted using weight-loss, gasometric techniques and Polarization curves. The surface morphology of the exposed steel was studied using scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS). The results of this study revealed that the acidic medium increased the corrosion rate of galvanised steel. It was deduced that the adsorption of *Jatropha gossypifolia* extract on galvanised steel surface obeyed the Langmuir adsorption isotherm. The negative value of Standard Change in Gibbs Free Energy of adsorption (ΔG_{ads}) showed that the reaction was thermodynamically feasible and spontaneous. Data obtained from weight loss and polarisation measurements show that *Jatropha gossypifolia* extract has fairly good inhibiting properties for galvanized steel corrosion in acidic medium, with inhibition efficiency of 89% at optimum concentration of 0.4 g/L. Addition of the extract to corrosive medium reduces current densities at both cathode and anode of the electrochemical cell thereby act as a mixed inhibitor. The SEM images confirmed that galvanised steel corroded in acidic environment and *Jatropha gossypifolia* extract inhibited the rate of corrosion in galvanised steel in 0.5 M HCl solution. All the studied techniques revealed that galvanised steel could corrode in 0.5 M HCl and that corrosion progress could be in hindered by adding *Jatropha gossypifolia* extract as an inhibitor.

Keywords: *Jatropha gossypifolia*, Corrosion, Weight Loss, Gasometric, polarization, Inhibition, Adsorption.

1. Introduction

Metals have a wide applications in human's day to day activities ranging from mechanical to electrical applications [1–4]. In the course of their usage, metals are exposed to various chemicals and different environmental conditions. Exposure of metals to chemicals causes deterioration of their properties [5]. Among metals, galvanized steel is commonly used in construction, energy, food and chemical industries due to its desirable mechanical properties and relatively low cost of production[6]. Additionally, galvanized steels are coated with zinc to increase their corrosion resistance. Nonetheless, galvanized steels corrode when expose to corrosion agents. Corrosion is caused by chemical or electrochemical reaction between a metal and its environment. It is undesired phenomenon that leads to loss of mechanical strength and chemical composition of the metals [7,8]. Consequences, of metal

corrosion is not limited to economy [9] but also to environmental risk in term of pollution in product leakage and structural collapse[10,11]. In order to maintain the desired properties of metals, corrosion control is a priority. Common techniques used to prevent corrosion include cathodic and anodic protection, coating and green corrosion inhibitor [12]. To avoid environmental issues, green corrosion inhibitor method is favoured due to its eco-friendly technology [13].

Plant extracts have been suggested to be suitable replacement for the expensive and toxic synthetic corrosion inhibitors due to their reduced environmental risk, biodegradable, low cost and high corrosion inhibition efficiency[4]. Corrosion inhibition effect of plant extract has been attributed to the adsorption of inhibitor molecules via heteroatoms or π -bonds onto the metal surface thereby forming a protective layer [14,15]. *Jatropha gossypifolia* L. (Euphorbiaceae) is widely distributed in countries of tropical, subtropical, and dry tropical weather[16]. Leaves of *Jatropha gossypifolia* are used in traditional medicine [17] and its seed oil is raw material in biofuel industry [18]. Leaf and stem extracts of *Jatropha curcas* have been reported as green and efficient corrosion inhibitors for mild steel in hydrochloric as well as in sulphuric acid medium. Optimum inhibition efficiency of 92.1% was recorded at 1.5 g/L for *J. curcas* leaf extract [19]. Similarly, inhibition performance of *Jatropha Curcas* [20,21] and *Jatropha gossypifolia* leaf extracts [22] on carbon steel have been investigated. Extracts from different parts of *Jatropha gossypifolia* have been reported to contain high concentration alkaloid [23,24], Coumarin-lignoids [25,26] and terpenes [27]. The presence of these heteroatoms phytochemicals together with eco-friendly nature of *Jatropha gossypifolia* extract inspires the present study. Hence, the aim of the present work is to investigate corrosion inhibitory efficiency of *Jatropha gossypifolia* ethanol extract on galvanized steel in acidic medium.

2. Materials and Methods

2.1 Collection and preparation of plant extract

Jatropha gossypifolia leaves were collected from a farm land at Boroboro area Oyo, Oyo state. The leaves were identified at the Department of Botany, University of Ibadan, Ibadan. The leaves were air-dried, pulverized and soaked in ethanol for 72 hours. The extract obtained was filtered and concentrated to form a slurry residue. This was then air dried to drive away remaining solvent. Different concentrations of inhibitor solutions (0.025, 0.05, 0.1, 0.2 and 0.4g/L) were prepared from dried extract using 0.5 M HCl as solvent.

2.2 Preparation of Galvanized Steel Specimen

Galvanized steel rod of diameter 2.0cm and thickness 0.02 cm was obtained from local supplier and cut into 1.80 cm in length. The specimens were cleaned using absolute ethanol, rinsed with distilled water, dried with acetone and kept in desiccator until they were ready for use.

2.3 Extract Analysis

Phytochemical profiling of the ethanol extract was done using gas chromatography–mass spectrometry (GS-MS). The gas chromatography system 7890 was equipped with polar fused silica capillary DB-1 column (30m length, 0.32 mm internal diameter, and thickness 0.25 μ m). The oven temperature was set at 80°C to 240°C, programmed to increase at the rate of 10°C/min and finally held isothermally for 10 min. The injector and detector temperatures were kept at 200°C and 250°C, respectively. The carrier gas used was helium at a flowrate of 1 mL/min, and the splitting ratio was set at 100:1. Agilent mass spectrometer 5975 set at 230°C was used to fragment and analyze ion peaks. Molecules were ionized using the electron impact ionization mode at a voltage of 70 eV, and the mass spectra were taken over the m/z range of 30-700 amu. The components of the plant extract were identified by WILEY and NIST database matching and by comparison of mass spectra with published data [28].

2.4 Gravimetric method (Weight-loss)

Weight-loss studies were carried out in triplicate by complete immersion of pre-weighed galvanized steel rod of 2.0 cm diameter and 1.80 cm length into inhibitor solution of different concentrations at room temperature. Immersion of galvanized steel was also done using 0.5 M HCl as corrodent solution. The samples were retrieved every 24 hours and soaked in cleaning reagent (50 g of NaOH, 200 g of Zn dust in 1000 mL H₂O) maintained at 90 °C for 40 minutes. The samples were washed with distilled water, dried and reweighed after treating with cleaning reagent. The difference in weight before and after 24 hours of soaking in corrodent solution was taken as weight lost. These procedures were repeated for six days for all the samples. The weight loss values were used to determine inhibition efficiency (IE) of the galvanized steel rod using equations 1

$$IE = \left(\frac{W_u - W_i}{W_u} \right) \times 100 \dots\dots 1$$

IE = inhibition efficiency, W_u and W_i uninhibited and inhibited weight respectively

2.5 Gasometric Method

Corrosion of metal in acidic medium always generate hydrogen gas with the rate of corrosion directly proportional to the amount of gas generated [29]. The experimental procedure designed by Aisha et al., 2012 [30] was adopted with little modification. A Büchner flask containing corrodent solution was connected to the burette through a rubber tubing to form u-tube shape with the burette. Paraffin oil was then introduced into the burette through the open end until its levels in both rubber tubing and burette are the same. Initial volume of paraffin oil in the burette was recorded and metal sample gently introduced into the corrodent and Büchner flask quickly closed. Rise in the volume of paraffin oil was recorded at every one minute for ten minutes. The volume of paraffin oil displaced was taken as volume of hydrogen gas generated during corrosion process. This procedure was repeated at three different temperatures 30, 60, and 90 °C for three different corrodent solutions; 0.025, 0.1 and 0.4 g/L. The control experiment (blank solution) was performed under the same conditions and each experiment was replicated twice for validation of the results.

2.6 Electrochemical Measurement

Three-electrode system consisting of galvanized steel as a working electrode, a saturated calomel electrode (SCE) as a reference, and a graphite rod as a counter electrode was used to determine electrochemical parameters. A Hokuto Denko potentiostat (HA151) was used to make potentiodynamic polarization. Open circuit potential (OCP) was measured for 50 minutes at an interval of 1 minute before each potentiodynamic polarization of the galvanized steel in 0.5 M HCl and various concentrations of inhibitor solutions to attain a steady-state condition. Then, the galvanized steel sample was subjected to potentiodynamic polarization from -1.6 V to 0.8 V vs. SCE. From the polarization curves, the electrochemical parameters, corrosion potential (E_{corr}) and corrosion current (I_{corr}) were determined.

2.7 Scanning Electron Microscopy and Energy dispersive X-ray (EDS)

Surface morphologies of the galvanized rod specimens immersed in 0.5 M HCl with and without *Jatropha gossypifolia* leaves extract for 24 hours were carried out using a ZEISS scanning electron microscope (SEM) and a Bruker energy dispersive X-ray spectroscopy (EDS) system. After retrieval of the metal specimens from the corrodent, their surfaces were rinsed thoroughly with distilled water and dried at room temperature. Imaging was performed at an accelerating voltage of 20kV using a secondary electron detector (SEI), and magnifications of 250, 500 and 1000x.

3. Results and Discussion

3.1 Phytochemical screening

The phytochemical profile of *Jatropha gossypifolia* ethanol extract was carried out to identify and quantify the phytochemicals present in the extract. The screening revealed 38 components as presented in Table 1. The two major components, 2, 2 dimethoxyl propane (64.65%) and toluene (21.33%) contained heteroatom and pi electrons respectively. Since corrosion is an electrochemical process that involves transfer of electron from electron rich centre to vacant d-orbital of metal, thus heteroatom and pi-bond present on phytochemicals serve as electron rich centre[31,32]. Consequently, the corrosion inhibitive potential of *Jatropha gossypifolia* extract observed in this study could be attributed to the adsorption of lone pair of electrons on the oxygen atoms of 2, 2 dimethoxyl propane or pi electrons in the aromatic ring of toluene onto the metal surface.

3.2 Weight loss

The loss in weight of the galvanized steel specimens in the absence and presence of inhibitor were evaluated and the results obtained were then used to determine corrosion inhibition efficiency of the plant extract. The information obtained from the weight loss-time measurement for galvanized steel is presented in fig. 1. The plot revealed that the weight loss decreased linearly and significantly in the presence of inhibitor compared to the blank solution, (0.5 M HCl) and it was also established that the loss in weight was concentration dependent. This shows that the extract inhibits the corrosion of galvanized steel in 0.5 M HCl solution.

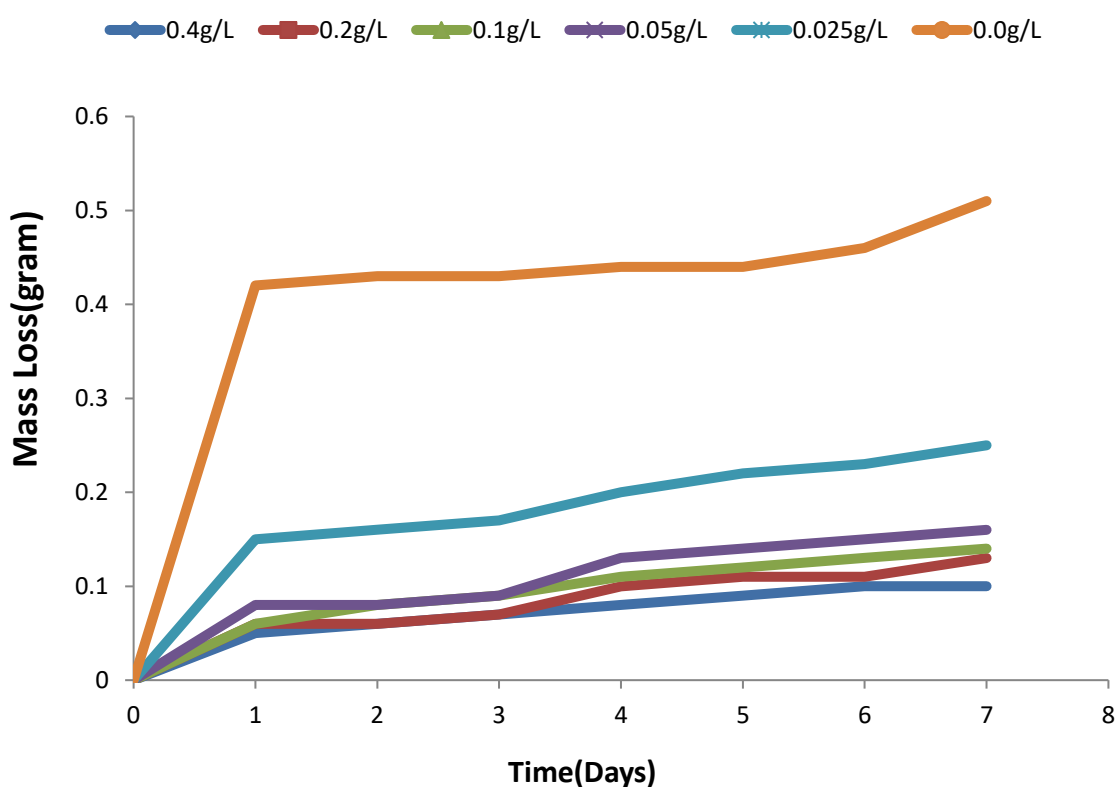


Figure 1: Galvanized steel weight loss due to HCl corrosion

Table 1: Chemical composition of *Jatropha gossypifolia* ethanol extract

Peak No.	Retention Time	Compound	Molecular Formula	Percentage composition
1	2.244	2,2-dimethoxy- Propane	C ₅ H ₁₂ O ₂	64.658%
2	2.996	N,N-dimethyl-2-aminoethanol	C ₄ H ₁₁ NO	0.619%
3	3.286	2,2-Dimethoxybutane	C ₆ H ₁₄ O ₂	0.198%
4	3.375	Toluene	C ₇ H ₈	21.328%
5	3.872	3-Penten-2-one, 4-methyl-	C ₆ H ₁₀ O	0.711%
6	4.527	2-Pentanone, 4-hydroxy-4-methyl-	C ₆ H ₁₂ O	1.265%
7	11.176	Caprolactam	C ₆ H ₁₁ NO	0.230%
8	18.183	Neophytadiene	C ₂₀ H ₃₈	0.206%
9	18.625	Neophytadiene	C ₂₀ H ₃₈	0.067%
10	19.382	n-Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	0.624%
11	19.723	Hexadecanoic acid, ethyl ester	C ₁₈ H ₃₆ O ₂	0.222%
12	19.799	trans-2-Hexadecenoic acid	C ₁₆ H ₃₀ O ₂	0.207%
13	20.872	Phytol	C ₂₀ H ₄₀ O	1.948%
14	21.036	9,12-Octadecadienoic acid	C ₁₈ H ₃₂ O ₂	0.155%
15	21.099	9,12,15-Octadecatrienoic acid	C ₁₈ H ₃₀ O ₂	0.759%
16	21.377	9,12,15-Octadecatrienoic acid	C ₁₈ H ₃₀ O ₂	0.632%
17	21.572	Octadecanoic acid, ethyl ester	C ₂₀ H ₄₀ O ₂	0.075%
18	21.667	Decyl oleate	C ₂₈ H ₅₄ O ₂	0.179%
19	23.010	4,8,12,16-Tetramethylheptadecane	C ₂₁ H ₄₆	0.119%
20	23.870	1H-Indole, 1-methyl-2-phenyl	C ₁₅ H ₁₃ N	0.044%
21	24.224	5-Methylthio-7,8-dihydro-6H-benzene	C ₁₉ H ₂₃ N ₅ O ₃	0.152%
22	24.374	1H-1,3-Benzimidazole-1-acetonitrile	C ₁₀ H ₉ N ₃	0.052%
23	24.553	1,2-Benzenedicarboxylic acid	C ₈ H ₆ O ₄	0.110%
24	24.694	1-(Ethoxy-2,2,2-D3)-9,10-Anthraquinone	C ₇ H ₁₆ O ₃	0.171%
25	24.869	1-(5'-Chloro-2'-methylaminobenzonate	C ₉ H ₁₀ ClNO ₂	0.166%
26	25.475	2-Ethylacridine	C ₁₅ H ₁₃ N	0.844%
27	25.547	(S)-N-Benzyl-2-hydroxy-3-phenyl	C ₁₆ H ₁₆ O ₃	0.323%
28	25.651	N-Methyl-1-adamantaneacetamide	C ₁₃ H ₂₁ NO	0.188%
29	25.718	5,6-Dimethoxy-1,3-diphenylindan	C ₁₂ H ₁₄ O ₄	0.224%
30	25.850	3 2-Ethyl-6-(2,4,6-trimethylphenyl)	C ₂₂ H ₂₇ N ₅	0.252%
31	26.033	2-Amino-1-methyl-9,10-anthraquinone	C ₂₁ H ₁₅ NO ₂	0.856%
32	26.397	2,7-Dichloro-3-methoxy-dibenzo-p-dioxin	C ₁₃ H ₈ Cl ₂ O ₃	0.157%
33	26.908	2,6,10,14,18,22-Tetracosahexaene	C ₂₄ H ₃₈	0.283%
34	27.470	4-(2-Methoxyphenyl)-2-quinolene	C ₁₈ H ₁₅ NO	0.352%
35	28.097	10-Phenyl-2,3-dihydroimidazo[1,2-a]pyrazino[2,3-d]pyrimidin-5(10H)-one	C ₁₅ H ₁₄ N ₂ O ₃	0.611%
36	29.770	γ-Tocopherol	C ₂₈ H ₄₈ O ₂	0.386%
37	30.078	Benzamide, N-(3-methylphenyl)-2,	C ₇ H ₇ NO	0.117%
38	33.720	Stigmasta-5,22-dien-3-ol	C ₂₉ H ₄₈ O	0.509%

3.3 Percentage efficiency

The percentage efficiency was determined according to Abiola *et al.* (2008)[29], the percentage inhibition efficiency (I%) was calculated using Equation (1).

$$I\% = \left(\frac{W_u - W_i}{W_u} \right) \times 100 \quad \dots \dots \dots \quad (1)$$

Where I is the inhibition efficiency of *Jatropha gossypifolia* in %, W_u and W_i are the uninhibited and inhibited weight losses respectively.

The percentage inhibition efficiency (IE%) of the extract at various concentration as determined from weight loss is presented in fig. 2. It was evident from the plot that (IE%) increases with increase in extract concentration but declines over time. It was observed that as the concentration of the extract increased and time of exposure elongated more inhibitor molecules got adsorbed on the metal. Thus, the degree of surface cover increased until the surface of metal was saturated with inhibitor molecules [33,34].

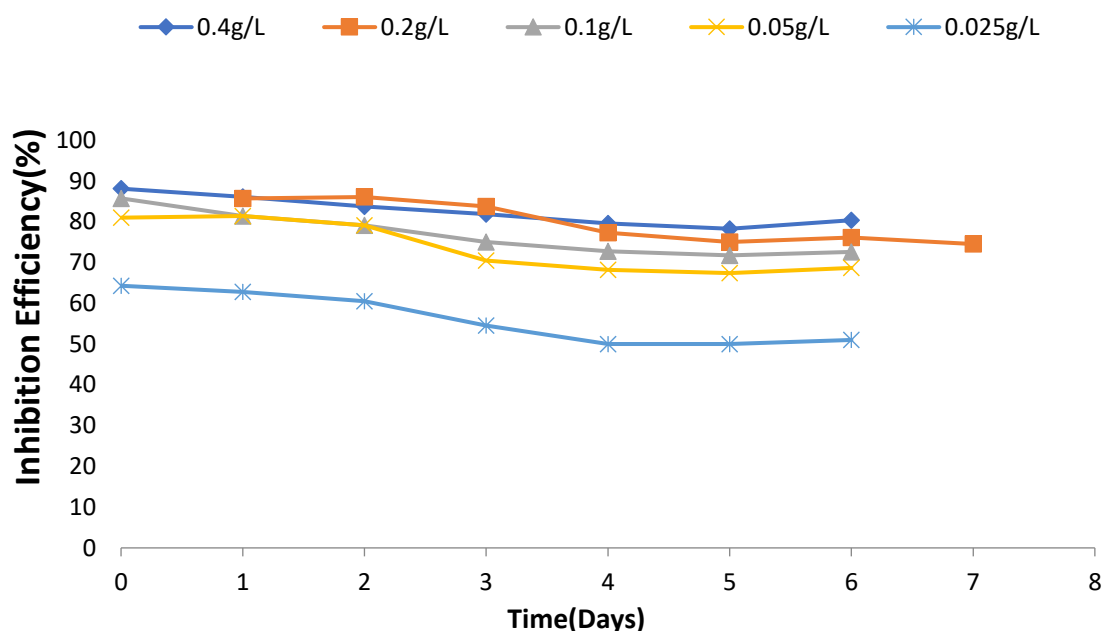


Figure 2: Corrosion inhibition efficiency of *Jatropha gossypifolia* for a period of 6 days at different extract concentrations

3.4 Adsorption isotherm

The nature of interaction between metal and inhibitor could be explained by adsorption isotherm. Fraction of surface coverage (θ) was determined according to equation (2) using the inhibition efficiency (IE%) data obtained through equation 2.

$$\theta = \frac{I\%}{100} \dots\dots 2$$

The linear regression between C/θ and C as presented in figure 3 gave regression coefficient of 0.9997 (almost unity). This indicates that the adsorption behavior follows the Langmuir Adsorption Isotherm, which is expressed as:

$$\frac{C}{\theta} = \frac{1}{K} + C \dots\dots\dots 3$$

Where C is the inhibitor concentration and K the equilibrium constant for the adsorption/desorption process of the inhibitor molecules on the metal surface. Large value of K_{ads} , $50.46 \text{ dm}^3\text{mol}^{-1}$, determined from the plot suggested efficient adsorption and better inhibition efficiency [35]. The value of K_{ads} obtained from the plot was used to determine spontaneity and type of the adsorption using equation (4).

$$\Delta G^{\circ}_{ads} = - RT \ln (55.5K_{ads}) \dots\dots 4$$

The value of ΔG_{ads} closer to -20 kJmol^{-1} is attributed to Physisorption while ΔG_{ads} closer to -40 kJmol^{-1} is attributed to chemisorption [36]. Since ΔG_{ads} obtained through equation (4) was -20.23 kJ/mol , the

adsorption of *Jatropha gossypifolia* extract onto the surface of the metal was spontaneous and due to electrostatic interactions between the charged metal and charged molecules of the extract, physisorption [37].

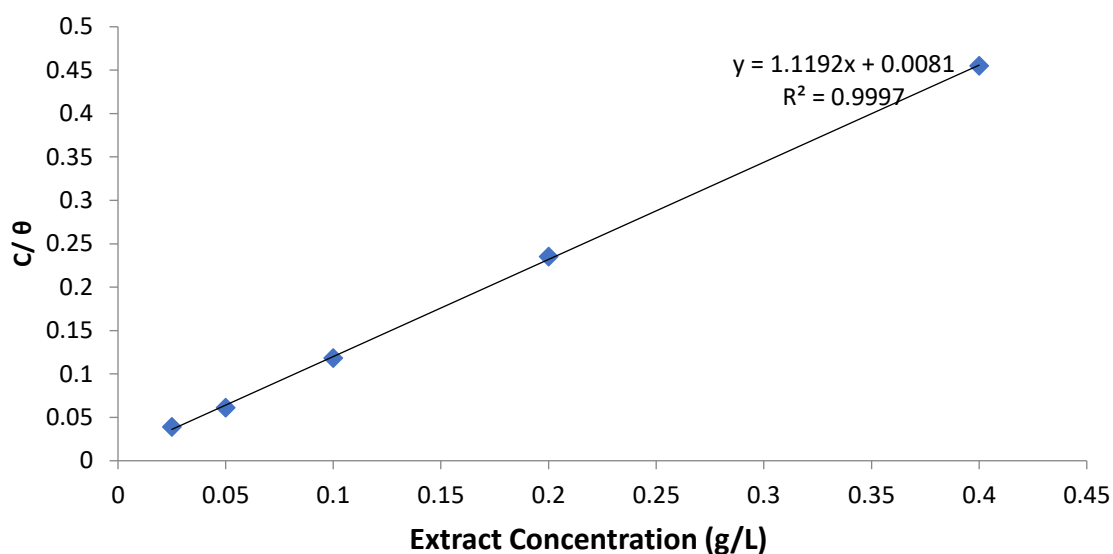


Figure 3: Langmuir adsorption isotherm (C/θ vs. C) at room temperature

3.5 Gasometric analysis

The mean volume of hydrogen evolved with time during corrosion of galvanized steel in 0.5 M HCl at different concentrations of inhibitor is presented in fig.4. This study was conducted at room temperature, from the plot it was evident that the time taken to evolve the same volume of H_2 (40 mL) gas at different concentrations of inhibitor increased as the concentration of the inhibitor increased. Figure 4, clearly showed that *Jatropha gossypifolia* extract greatly hindered the evolution of the H_2 gas at higher concentration as compared to the control. This implies that inhibitor formed a protective film over the metal surface thereby reducing the interaction between metal and corrodent. The same observation was reported by Ogunrinde and his co-workers, 2020[38].

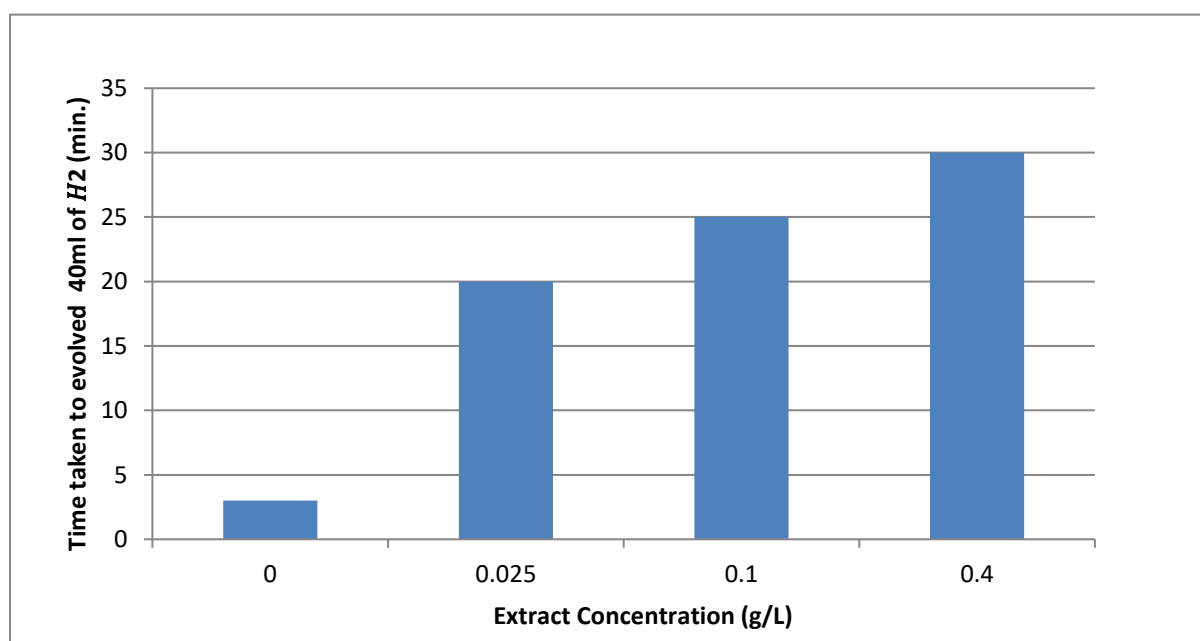


Figure 4: Volume of H_2 evolved at different concentrations of inhibitor

Effect of temperature on the rate of corrosion in the absence and presence of inhibitor was investigated at 303, 333, and 363 K. The time taken to evolved 40 mL of hydrogen gas at different temperatures was recorded for various concentrations of inhibitor and the control. The result is presented in fig. 5. As indicated in fig. 5, the rate of hydrogen gas evolution decreased as concentration of the inhibitor increased. However, at high temperature the rate of gas evolution was slightly increased but not as observed in the control. At high temperature the desorption of the adsorbed inhibitor occurs thereby exposing the surface of the metal to the corrosion[39]. Hence, the rate of hydrogen gas generation serves as an indicator for corrosion rate.

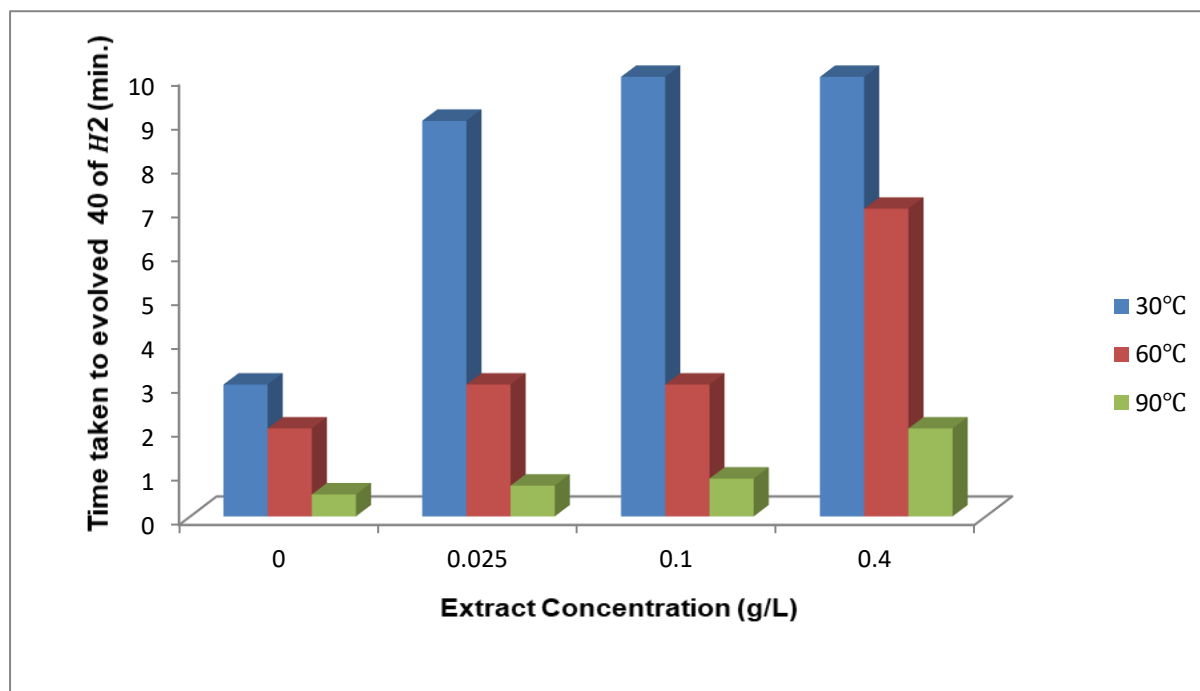


Figure 5: Effects of temperature on the rate of corrosion using *Jatropha gossypifolia* extract as inhibitor

In addition, inhibition efficiency of *Jatropha gossypifolia* extract increases as the concentration of the extract increases from 0.05 to 0.4 g/L as indicated in fig. 6. However, the inhibition efficiency of the extract at higher temperature slightly increases as the concentration of the inhibitor increases while at low temperature inhibition efficiency increases steadily with increase in concentration.

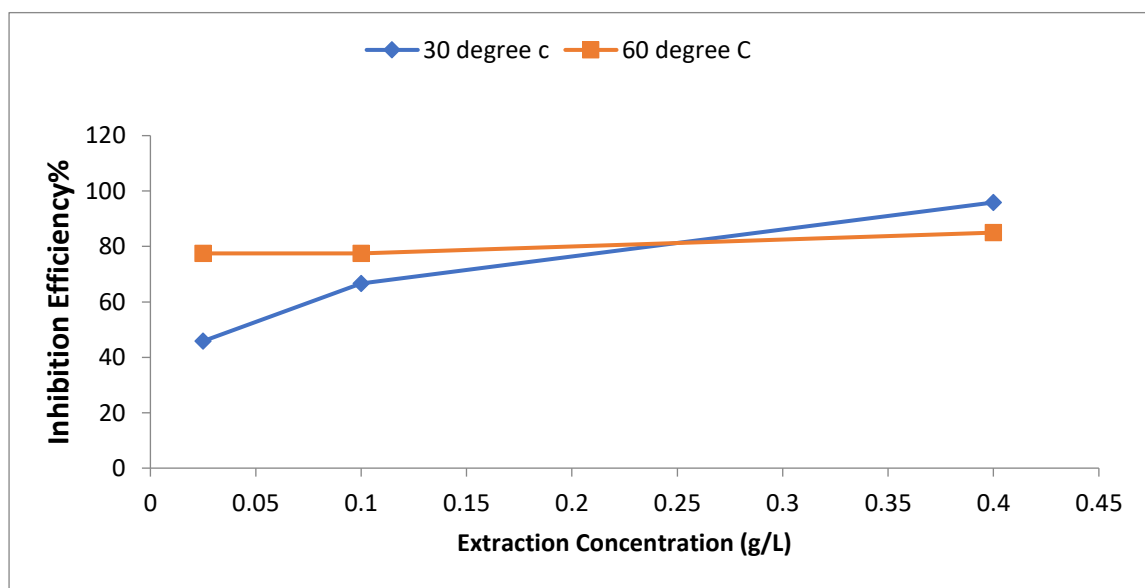


Figure 6: Inhibition Efficiency of *Jatropha gossypifolia* at different temperatures

3.6 Adsorption Isotherm

Equation (5) was used to evaluate inhibition efficiency values from were fraction of the surface cover (θ) was determined. This was carried out at 303 K. The adsorption isotherm was established by plotting (C/θ vs C) has shown in figure 7. It was concluded that the adsorption relationship follows a pattern resembling that of figure 3 (gravimetric analysis) or the Langmuir Adsorption model.

$$I\% = \left(\frac{V_u - V_t}{V_u} \right) \times 100 \dots\dots 5$$

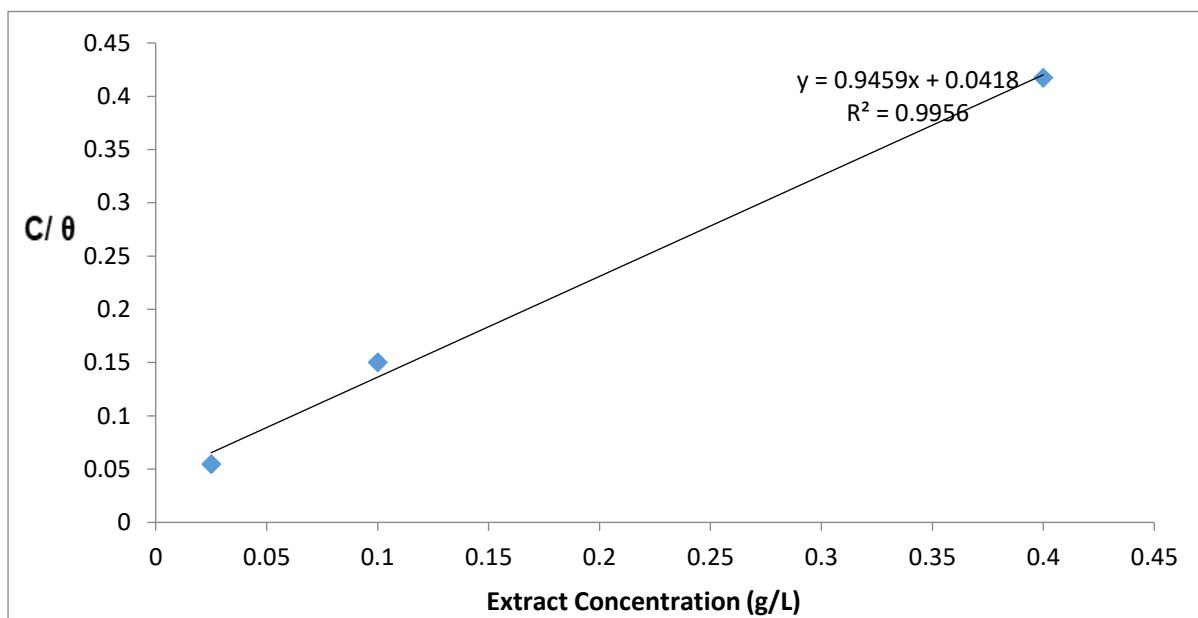


Figure 7: Langmuir adsorption model from gasometric analysis at 303 K

3.7 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy micrograph (magnification 500×) of un-inhibited and inhibited galvanized metals are presented in fig. 8 and 9 respectively. From fig. 9 it is evident that *Jatropha gossypifolia* extract formed a protective layer on the surface of inhibited steel. Hence, corrosion activity on the surface of inhibited metal is minimized. The surface of un-inhibited metal was highly damage because its surface was not protected from corrosion environment.

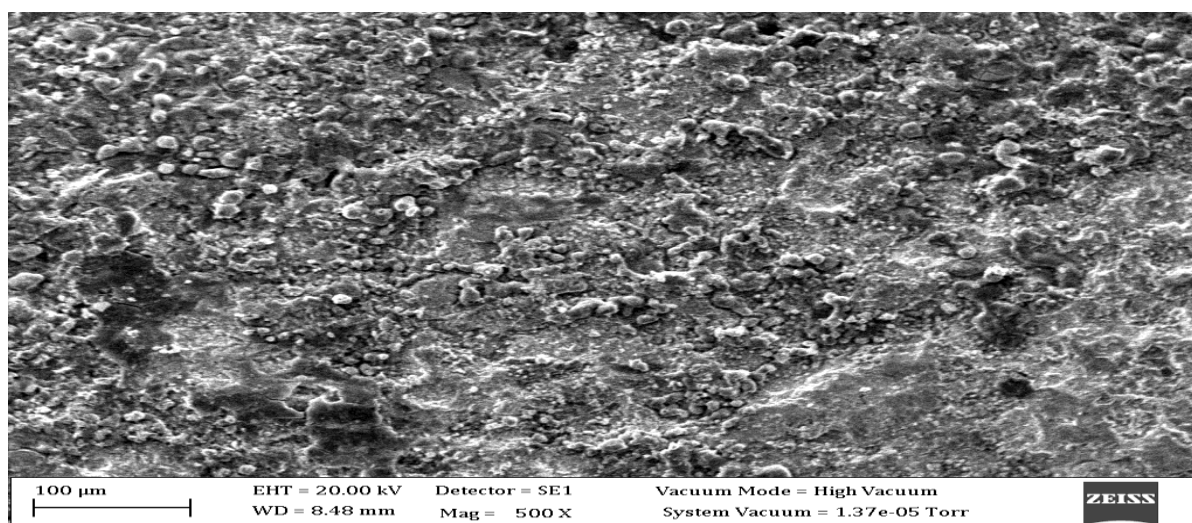


Figure 8: SEM micrograph of uninhibited galvanized steel in 0.5M HCL solution

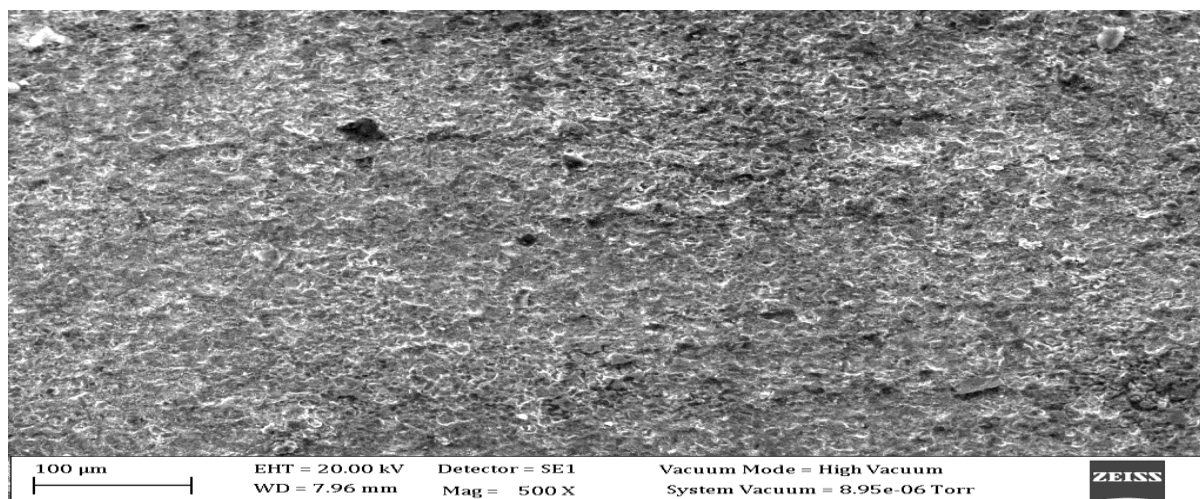


Figure 9: SEM micrograph of inhibited galvanized steel in 0.4g/L of *Jatropha gossypifolia* extract solution

3.8 Electrochemical measurement

Further investigation on the effect of *Jatropha gossypifolia* leaf extract on corrosion was conducted using electrochemical method. The parameters associated with electrochemical polarization measurement such as corrosion rate, corrosion potential (E_{corr}), current density (I_{corr}) and polarization resistant were determined from polarization plots and presented in table 2.

Table 2: Electrochemical parameters for galvanized steel of inhibited and un-inhibited.

Conc g/L	E_{corr} (v)	i_{corr} ($\mu\text{A}/\text{cm}^2$)	Corr rate (mm/yr)	Polarization resistant (Ω)	[ba](v/dec)	[bc](v/dec)
0.4	-1.224	0.0048	5.5598E-05	6937628.312	0.055601	0.033542
0.0	-0.488	0.0028	0.000326627	1180912.63	0.061206	0.043709

Table 2 shows that the addition of *Jatropha gossypifolia* extract leads to a significant decrease in the corrosion current i_{corr} as it decreases from 0.0281 $\mu\text{A}/\text{cm}^2$ in the absence of the extract to 0.0048 $\mu\text{A}/\text{cm}^2$ at 0.4 g/L of the extract. Corrosion potential E_{corr} shifts to more negative value, -1.224 V, in the presence of the inhibitor as compare to -0.488 V in the absence of inhibitor. Addition of *Jatropha gossypifolia* extract reduces corrosion rate from 0.0003 mm/yr to 5.5598 mm/yr. Moreover, addition of inhibitor reduces both cathodic and anodic current density thus *Jatropha gossypifolia* extract acts as a mixed corrosion inhibitor [40,41].

5. Conclusion

Phytochemical profiling of the extract revealed several heteroatoms molecules which are related to anticorrosion properties of the extract. Chemical and electrochemical methods used confirmed that methanol extract of *Jatropha gossypifolia* extract was an excellent corrosion inhibitor for the protection of metal in acidic medium. Additionally, the adsorption of the extract to the surface of the metal followed Langmuir adsorption isotherm model with negative value of Gibbs free energy of adsorption indicating chemisorption process and spontaneity of the adsorption. The inhibition efficiency of the extract using different methods showed a good agreement with each other.

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