

NOVEL ECO-FRIENDLY EXTRACT FOR CORROSION PROTECTION OF MATERIALS IN 1 M H₂SO₄ ENVIRONMENT DURING TRANSPORTATION AND STORAGE OF OIL SECTOR

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ABSTRACT

In this study, the corrosion inhibition effect of *Dioscorea dumentorum* leaf extract on mild steel was evaluated in a 1 M H₂SO₄ solution at various concentrations, using weight loss measurements and Tafel polarization techniques. This study represents an effort to curb metallic corrosion in the transportation and storage sectors of the oil industry. The highest inhibition efficiency recorded was 90.8% in 3 g of inhibitor concentration with a corrosion rate of 0.0140 mpy and weight loss of 0.2567 g, respectively. Also, the inhibition efficiency increased with higher concentrations of the *Dioscorea dumentorum* leaf extract. The presence of phytochemical constituents such as saponins, phenols, steroids, and flavonoids in the extract was found to be responsible for the effective inhibition, as these compounds adsorbed onto the metal surface. The adsorption of the inhibitor on the mild steel surface followed both Langmuir and Temkin adsorption isotherms, as plots for both isotherms showed good regression that were all near unity ($\cong 1$), respectively, for samples with 0.5 to 3 g/0.25 L extract concentrations, while the computed Gibbs free energy of the adsorption process (ΔG_{ads}^0) for both isotherm models showed values less than -20 kJ mol^{-1} . This suggests that adsorption of the extract onto the metal surfaces was by physisorption. The results confirm that the leaf extract acts as a corrosion inhibitor by enhancing surface protection on the metal.

Keywords: polarization curves; mild steel coupons; sulfuric solution; physisorption; *Dioscorea dumentorum*; corrosion inhibition.

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1. Introduction

Corrosion, the degradation of materials due to chemical reactions with their environment, remains a significant challenge across various industries, including manufacturing, energy production, and transportation [1, 2]. The economic and safety implications of corrosion are profound, as it leads to equipment failure, increased maintenance costs, and shortened service life for infrastructure [1, 3]. One of the most effective strategies to mitigate corrosion is the use of corrosion inhibitors—chemicals that either slow down or prevent the electrochemical reactions that lead to material degradation [4, 5].

Among the growing range of corrosion inhibitors, plant-based inhibitors have gained attention due to their eco-friendly nature, availability, and effectiveness [4, 6]. These natural inhibitors, derived from various plant species, contain organic compounds that possess corrosion-resistant properties, often through mechanisms such as the formation of protective films on metal surfaces or the adsorption of active molecules onto the material [7-9]. Furthermore, plant-based inhibitors are biodegradable, non-toxic, and less harmful to the environment

compared to traditional synthetic chemicals [10, 11].

Numerous studies have explored the effectiveness of plant extracts. These compounds often act by adsorbing onto the metal surface, creating a physical barrier that shields the metal from aggressive ions in the acidic solution [12-14]. The inhibition efficiency of plant extracts is influenced by several factors, including the concentration of the extract, the type of plant, and the nature of the acidic medium [7, 15].

Research has demonstrated that a variety of plant species, such as *Azadirachta indica* (neem) [16, 17], *Cymbopogon citratus* (lemongrass) [18], *Paederia foetida* (Chinese fever vine) [19], *Coriandrum sativum* (coriander) [20], and *Cinnamomum verum* (cinnamon) [21], possess significant corrosion inhibition properties in acidic environments. For example, neem extract, rich in flavonoids and tannins, has been shown to provide effective protection against corrosion in hydrochloric acid solutions [22]. Similarly, extracts from coriander and cinnamon have demonstrated promising inhibition efficiency, with mechanisms attributed to the formation of complexes with metal ions or the deposition of protective organic films on the metal surface [20, 21].

This study investigates the novel eco-friendly extract of *Dioscorea dumentorum* for corrosion protection of materials in a 1 M H₂SO₄ environment relevant to the transportation

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and storage of the oil sector using weight loss measurements and Tafel polarization techniques. The research will provide a comprehensive understanding of the interactions between the plant compounds and metallic surfaces, particularly in acidic environments, which are common settings for corrosion processes, and through which it will contribute to the development of sustainable corrosion protection methods that align with the growing demand for green and environmentally conscious technologies.

2. Experimental

2.1. Materials

Sulfuric acid (H_2SO_4) was used as the corrosive medium and was of 99% analytical grade obtained in Abakaliki, Ebonyi State, Nigeria. The samples used were mild steel cut into coupons with dimensions of 2.6 cm × 2.5 cm × 0.1 cm. All the sample coupons were cut with hand shears so as not to cause significant distortion of their grain structures. Distilled water was used to prepare 1 M of H_2SO_4 .

2.2. Extract preparation

The *Dioscorea dumentorum* leaves were collected from Abakaliki city and identified at the Botany Department of Alex Ekwueme Federal University in Ndufu-Alike, Ikwo, Nigeria. The leaves were thoroughly washed with distilled water and then sun-dried for ten days until crisp. Afterward, the leaves were ground into a fine powder and sieved. A 30 g sample of the powdered leaves was extracted using 300 ml of methanol. The methanol-extract mixture was left to stand for 4 days, followed by evaporation of the methanol to obtain the extract. Weights of 0.5, 1, 1.5, 2, and 3 g of the dried extract were dissolved in separate containers containing 0.25 L of 1 M H_2SO_4 . A control solution was prepared without the leaf extract.

2.3. Phytochemical analysis

Qualitative analysis of the methanolic extract of *Dioscorea dumentorum*, was carried out to determine the presence of alkaloids, flavonoids, steroids, phenols, saponins, and tannins using methods reported by Ejikeme et al [23]

2.4. Weight-loss procedure

This study employed weight-loss measurements to evaluate corrosion. The mild steel coupons were pickled in hydrochloric acid (HCl), rinsed with distilled water, and dried using acetone. The initial and final weights of the coupons were recorded with a digital balance before and after immersion. Five coupons were placed in each of six containers, each containing 0.25 L of 1 M H_2SO_4 solution with varying concentrations of the plant extract, excluding the control group. The setup was left undisturbed for 2 days, and the coupons were then removed at 3-day intervals. After removal, the coupons were dried and weighed again.

3. Results and discussion

3.1 Phytochemical analysis

Results of the analysis of the phytochemical constitution of *Dioscorea dumentorum* are shown in table 1 below.

3.2 Weight-loss measurements

Weight-loss measurements were carried out in the presence of the plant extract and a blank which served as the control. These measurements show the effectiveness of the

Phytochemicals	Result
Alkaloids	Absent
Flavonoids	Present
Steroids	Present
Phenols	Present
Saponins	Present
Tannins	Present
Terpenoids	Absent

plant extract in the corrosive environment as it affects the test samples and also show the corrosion rate in the different test solutions at different time intervals.

The results obtained using weight loss method to measure the weight loss parameters are shown in table 2 below.

Corrosion rate of the mild steel coupons was computed using the formula below:

$$CR = \frac{534\Delta W}{\rho AT} \quad (1)$$

where CR = Corrosion rate (mpy); ΔW = weight-loss loss (g); ρ = density of mild steel (kgm^{-3}); A = Total surface Area of the specimen (cm^2); T = time of immersion (days)

$$A = 2[(LB + BT + LT)] - 2(\pi(D^2)/4) \quad (2)$$

where A = total surface area ($13.6 cm^2$); L = length (2.6 cm); B = breadth (2.5 cm); T = thickness (0.5 mm); D = diameter of the hole on the coupon (0.5 cm)

The inhibition efficiency (I.E %) and surface coverage (θ) of the specimen were calculated using the equations shown below:

$$IE (\%) = \frac{W_1 - W_2}{W_1} \times 100 \quad (3)$$

$$\theta = 1 - \frac{W_1}{W_2} \quad (4)$$

where W_1 = the steel loss in weight in the presence of the inhibitor; W_2 is the mild steel loss in weight in the absence of the inhibitor

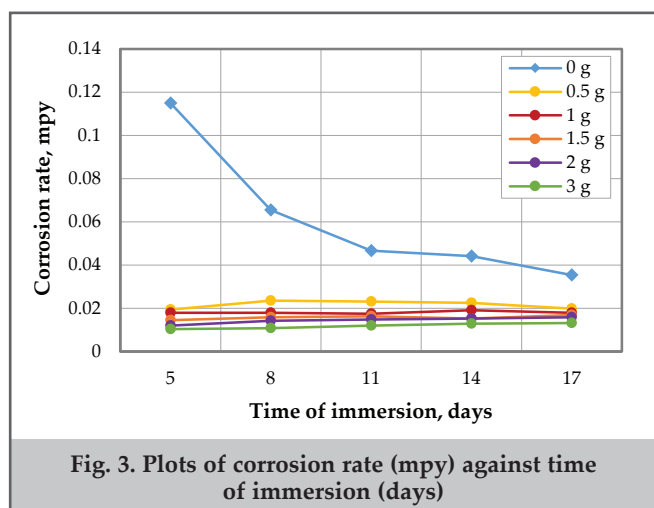
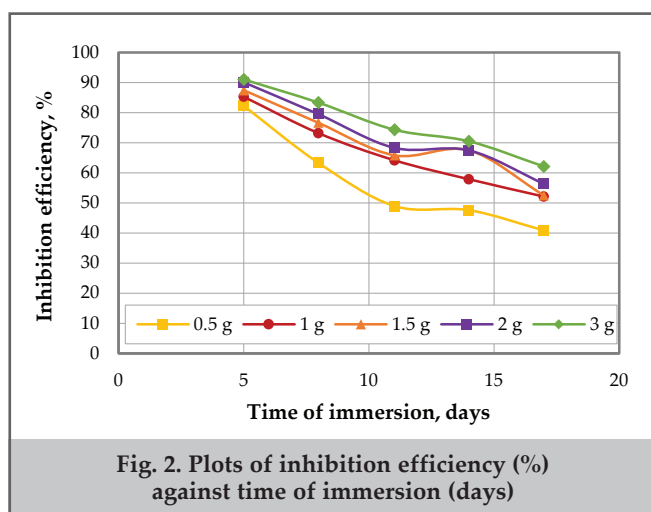
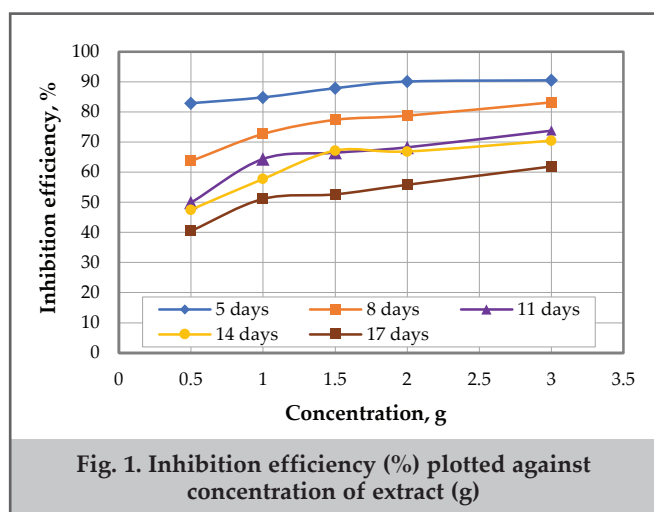
3.3. Effect of extract concentrations on inhibition efficiency

The inhibition efficiency of the extract increases as the concentration of the extract increases, as shown in figure 1. This is due to the increase in the surface area of the mild steel covered by the adsorbed molecules of the extracts at higher concentrations as was reported by. The maximum efficiencies recorded by the extract are 88, 90 and 90.8 % in concentrations of 1.5, 2 and 3 g respectively.

3.4. Effect of immersion time on the inhibition efficiency and corrosion rate

Weight loss measurement in the absence and presence of the extract at various concentrations after an immersion time of 17 days was used to study the stability of the inhibitive behavior and the rate of corrosion attack on the mild steel coupons with respect to the time of immersion. It was shown in figure 2 and 3 that inhibition efficiency decreased as time of immersion increased and the corrosion rate increased in

Weight loss of coupons in 1.0 M H ₂ SO ₄ environment												Table 2
Time (days)	Initial weight (g)	Final weight (g)	Weight loss (g)	Corrosion rate (mpy)	Inhibition efficiency (%)	Time (days)	Initial weight (g)	Final weight (g)	Weight loss (g)	Corrosion rate (mpy)	Inhibition efficiency (%)	
0 g concentration of extract						0.5 g concentration of extract						
5	8.6559	5.8494	2.8065	0.1167		5	7.8852	7.4151	0.4701	0.0195	8.3	
8	7.9313	5.2385	2.6928	0.0670		8	7.8395	6.9030	0.9365	0.0243	63.7	
11	7.7914	5.2747	2.5167	0.0476		11	8.3577	7.0907	1.2670	0.0239	49.8	
14	8.3163	5.2996	3.0167	0.0448		14	8.3676	6.7889	1.5786	0.0234	47.8	
17	8.2104	5.2940	2.9164	0.0357		17	8.3046	6.5692	1.7354	0.0212	40.6	
1 g concentration of extract						1.5 g concentration of extract						
5	7.4180	6.9937	0.4243	0.0176	84.9	5	7.8157	7.4801	0.3356	0.0140	88	
8	7.5155	6.8224	0.6931	0.0180	73.1	8	7.7594	7.1699	0.5895	0.0153	77.2	
11	7.9834	7.0960	0.8874	0.0168	64.7	11	7.7324	6.8936	0.8388	0.0159	66.6	
14	8.0254	6.7465	1.2789	0.0190	57.6	14	7.4229	6.4325	0.9904	0.0147	67.2	
17	7.6330	6.2152	1.4178	0.0173	51.5	17	7.7058	6.3204	1.3854	0.0169	52.7	
2 g concentration of extract						3 g concentration of extract						
5	6.9160	6.6355	0.2805	0.0117	90	5	8.3194	8.0627	0.2567	0.0107	90.8	
8	7.8720	7.3346	0.5374	0.0140	79.1	8	7.4860	7.0609	0.4251	0.0110	83.6	
11	7.9407	7.1437	0.797	0.0151	68.3	11	7.4766	6.8265	0.6501	0.0123	74.2	
14	7.7168	6.7354	0.9814	0.0146	67.4	14	8.1060	7.2150	0.891	0.0132	70.5	
17	8.1751	6.8853	1.2898	0.0158	55.7	17	8.2891	7.1805	1.1086	0.0136	61.9	



the inhibited solutions with increase in time of immersion. This could be due to the desorption of the extract from the surfaces of the mild steel coupons which exposes more areas of the coupons to corrosion attack.

3.5. Effect of extract concentration on the corrosion rate

The plots of corrosion against concentration as can be seen in figure 4, were used to study the effect of extract concentration on the corrosion rate of mild steel in 1 M H₂SO₄. The corrosion rate was monitored using the weight loss measurement. It was observed that the corrosion rate of decreases with an increase in the concentrations. The samples from the blank test solution showed the highest corrosion rates while that containing 3g of the extract showed the lowest corrosion rates.

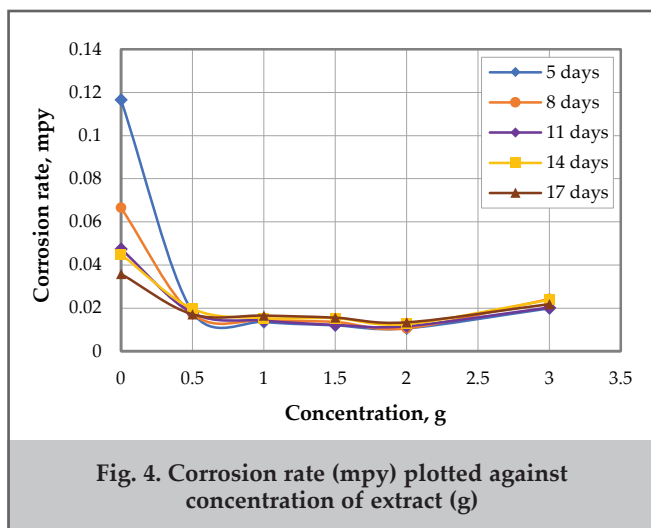


Fig. 4. Corrosion rate (mpy) plotted against concentration of extract (g)

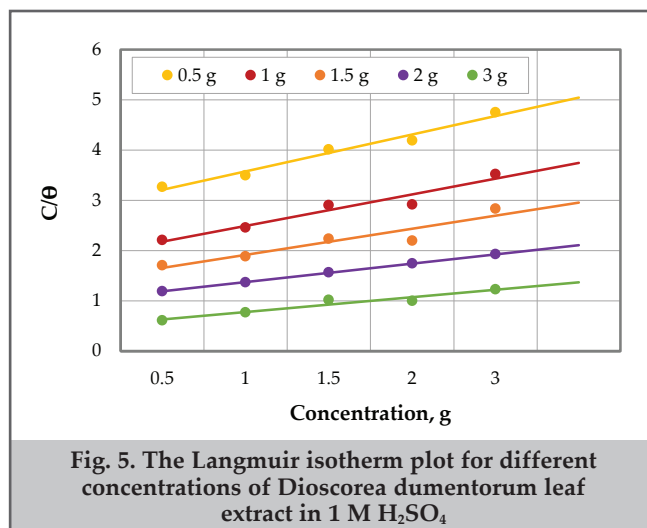


Fig. 5. The Langmuir isotherm plot for different concentrations of *Dioscorea dumetorum* leaf extract in 1 M H₂SO₄

3.6. Adsorption isotherm and free energy of adsorption (ΔG_{ads}^0)

The effectiveness of a corrosion inhibitor is influenced by the extent to which its molecules adsorb onto the steel surface [24]. An adsorption isotherm illustrates the relationship between the corrosion inhibition efficiency and the degree of surface coverage at varying inhibitor concentrations [25]. The isotherm also provides insight into the nature of the interaction between the inhibitor molecules and the mild steel surface [26]. The degree of surface coverage (θ) for different inhibitor concentrations on the mild steel coupons was calculated to identify the best-fitting isotherm. Various isotherms have been proposed to describe the adsorption of corrosion inhibitor molecules on metal surfaces. Langmuir, Frumkin, Temkin, and Flory-Huggins are some of the most commonly utilized adsorption isotherms [27]. From these isotherms, linear relationship between surface coverage (θ) and inhibitor concentration can be established.

3.6.1. Langmuir adsorption isotherm

The Langmuir adsorption isotherm describes the adsorption of the extract on the metal surface. In this adsorption model, values of the adsorption constant k suggest the strength of interaction between the mild steel coupon surface and the molecules of the inhibitor, with values of $k > 0$ indicating a stronger interaction [28]. The following equation according to the Langmuir isotherm relates the surface coverage (θ) and the inhibitor concentration, C:

$$\frac{C}{\theta} = \frac{1}{K} + C \tag{5}$$

The plots of Langmuir adsorption isotherm, C/θ vs. C at different concentrations is shown in (fig. 5) for all the studied inhibitors at different temperatures.

The values of adsorption parameters obtained from Langmuir adsorption isotherm listed in table 3. The negative value of ΔG_{ads}^0 indicates the spontaneity of the adsorption process and the stability of the adsorbed layer on the steel surface. The values of ΔG_{ads}^0 up to -20 kJmol^{-1} indicates physisorption. The theoretical fittings of the adsorption model indicated obedience to Langmuir isotherm with R^2 values from 0.9359 to 0.9884. The ΔG_{ads}^0 is calculated using the equation:

$$\Delta G_{ads}^0 = 2.303RT \log (K_{ads} \times 55.55) \tag{6}$$

where R is the gas constant ($8.314 \text{ J. K}^{-1}\text{mol}^{-1}$), T is absolute temperature (K), and 55.5 is the water concentration in solution expressed in mol L^{-1} .

From table 3 above it can be seen that the values of the adsorption constant, k , are low, indicating a weak interaction between the extract and the mild steel surface.

3.6.2. Temkin Adsorption Isotherm

The Temkin adsorption isotherm assumes that the inhibitor molecules are evenly distributed over the surface of the mild steel coupon [29]. This model suggests that the adsorption energy decreases in a linear manner as the surface coverage (θ) increases [30]. The surface coverage values (θ) at various inhibitor concentrations in 1.0 M H₂SO₄ solution, determined from weight-loss measurements, were analyzed and fitted to the Temkin adsorption isotherm presented in figure 6 below, using the following equation:

$$\exp(-2\alpha\theta) = K_{ads}C \tag{7}$$

The values of adsorption parameters including: intercept, slope, R^2 , and ΔG_{ads}^0 were computed from Temkin isotherm plots, which are shown in table 4. The negative ΔG_{ads}^0 values for all inhibitors at various concentrations suggest that there is repulsion between the molecules in the adsorbed layer on the metal surface. The theoretical Temkin's isotherm model fitting showed good regression with R^2 values ranging from 0.9080 to 0.9872.

3.7. Electrochemical methods

Potentiodynamic polarization method

The polarization studies were carried out from a potential of -1.5 V to 1.5 V with respect to the steady-state potential

Conc. of the extract, g	Slope	Intercept	K value	Regression	ΔG_{ads}^0 , KJmol^{-1}
0.5	0.2504	0.5308	1.8839	0.9359	-11.5221
1	0.3136	1.0485	0.9537	0.9808	-9.8353
1.5	0.4437	1.4829	0.6744	0.9592	-8.9763
2	0.5324	1.9877	0.5031	0.9730	-8.2503
3	0.6227	3.005	0.3328	0.9884	-7.2262

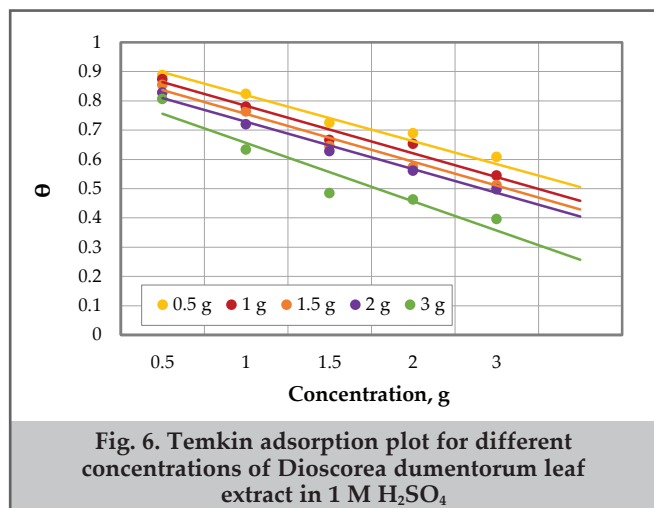


Fig. 6. Temkin adsorption plot for different concentrations of Dioscorea dumentorum leaf extract in 1 M H₂SO₄

Conc. of the extrac, g	Slope	Intercept	Regression	$\Delta G_{ads}, KJmol^{-1}$
0.5	-0.1031	0.8818	0.9080	-9.6410
1	-0.0836	0.9167	0.9872	-9.7371
1.5	-0.0820	0.9510	0.9405	-9.8282
2	-0.0810	0.9664	0.9577	-9.8680
3	-0.0715	0.9778	0.9867	-9.8970

Extract conc., g	Corrosion rate, mpy	Corrosion current, A	Corrosion current density, A/cm ²
0	3.232×10^{-3}	2.492×10^{-2}	7.059×10^{-3}
0.5	1.270×10^{-3}	9.791×10^{-3}	2.774×10^{-3}
1	5.570×10^{-3}	4.294×10^{-2}	1.216×10^{-2}
1.5	3.434×10^{-3}	2.649×10^{-2}	7.504×10^{-3}
2	2.932×10^{-3}	2.260×10^{-2}	6.402×10^{-3}
3	1.659×10^{-3}	1.279×10^{-2}	3.623×10^{-3}

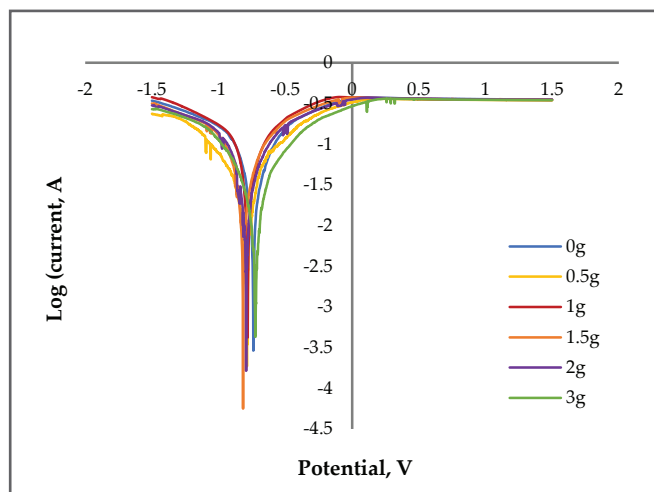


Fig. 7. Tafel plots of potentials (V) versus log (currents/A) obtained from potentiodynamic polarization test of mild steel coupons in 1 M of H₂SO₄

at a scan rate of 0.01 v/s. The Tafel plots, obtained from the potentiodynamic polarization test for the inhibition efficacy of Dioscorea dumentorum of mild steel in 1.0 M H₂SO₄.

Note: hold time at Ef (S)=0 and quiet time (S)=2.

Corrosion current density

The corrosion current density (I_{corr}) values were obtained from the polarization experiments in accordance to ASTM G 102- 89 [31]. The Corrosion current density was obtained using the relation [31]:

$$i_{corr} = \frac{I_{corr}}{A} \tag{8}$$

where i_{corr} is the corrosion current density (A/cm²), I_{corr} is the total anodic current (A), and A is the exposed specimen area (cm²).

Corrosion rate

The corrosion rate (CR) with unit in mm/year was calculated using the Faraday’s law [31-33]:

$$CR = k \times \frac{I_{corr}}{\rho} \times EW \tag{9}$$

where $k=3.273 \times 10^{-3}$ mm g/ μ A cm yr., ρ =density in g/cm³, and EW=equivalent weight of the metal (27.92).

3.8. Comparative discussion of the results of gravimetric vs polarization methods

Looking at the overall results, the results of gravimetric analysis (weight loss) generally looks more consistent, convincing, and indicates a decrease in the rate of corrosion in the presence of the extract. However, it could be observed that there are discrepancies between the results obtained by weight loss measurement and polarization methods. While the weight loss measurements showed a systematic decrease in the rate of corrosion as the concentration increases, the results of polarization test in some cases seem non-systematic. Usually, gravimetric analysis (weight loss) and electrochemical polarization methods frequently show discrepancies in corrosion inhibitor studies, stemming from fundamental differences in what they measure and the conditions under which they operate. While both aim to determine corrosion rate and inhibitor efficiency, discrepancies arise because weight loss measures the total, time-averaged mass loss, whereas polarization measures instantaneous corrosion kinetics – which are heavily influenced by the nature of the inhibitor film and experimental conditions [34, 35].

The gravimetric technique is widely acknowledged as the most common methodology for evaluating metal corrosion, and many believe it to be the benchmark standard. In addition, weight loss tests have the benefit of displaying an exposed surface, allowing for the measurement of corrosion rates over extended periods of time. It also enables the examination of corrosion morphology, such as general or localized attacks, etc. This technique represents overall corrosion rates in the system, as opposed to electrochemical techniques, which allow one to track changes in the kinetic process, like the polarization test, which is a fast technique, providing an instantaneous corrosion rate at a specific moment. If the inhibitor film forms slowly or breaks down over time, the two methods will yield different results [34, 35], as observed in tables 2 and 5 respectively.

3.9. Mechanism of Inhibition

The adsorption of the leaf extract occurs due to the phytochemical compounds in the extract binding to the metal surface, thereby protecting the metal from acid attack and preventing corrosion [36]. In general, if the adsorbed molecule/ion on the surface complex is stable, the corrosion of steel is inhibited. Therefore, it can be concluded that the adsorption of the phytochemical components of the plant extract leads to the formation of stable surface complex, which blocks the active sites on the surface of the metal thereby reducing the corrosion rate. Increase in the concentration of the phytochemicals increases the amount of the surface complex resulting in greater inhibition of the corrosion.

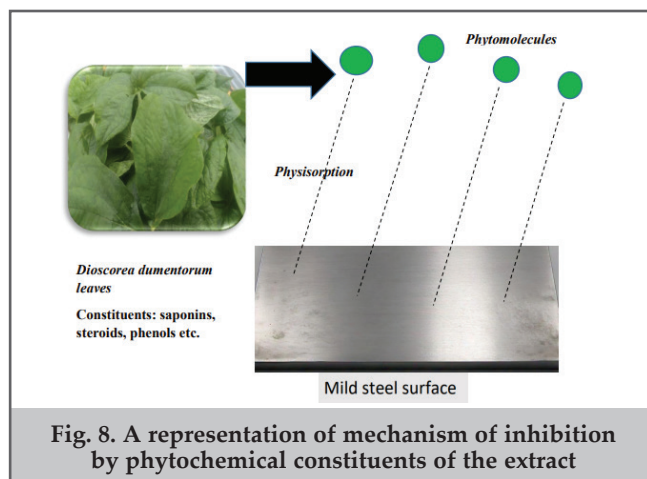


Fig. 8. A representation of mechanism of inhibition by phytochemical constituents of the extract

Conclusions

Dioscorea dumentorum leaf extract was studied as a potential eco-friendly inhibitor for mild steel in 1 M H_2SO_4 . It was determined from the study that:

1. *Dioscorea dumentorum* leaf extract proved to be a potent inhibitor of the corrosion of mild steel in 1 M H_2SO_4 . It exhibited higher inhibition efficiencies as the concentration of the extract increased.
2. The ΔG_{ads}^0 from the adsorption isotherm models showed values less than -20 kJ mol^{-1} which indicates the physical adsorption of the extract on the surfaces of the mild steel samples. The corrosion efficacy of the extract can be traced to this physical interaction between the extract and the mild steel steel surfaces.
3. The polarization curves showed that the extract was more potent as a corrosion inhibitor at 3g concentration.

The results of this study have proven that *Dioscorea dumentorum* is a viable eco-friendly option for the inhibition of the corrosion of mild steel in 1 M H_2SO_4 .

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