Uzoma S. Nwigwe[1,2] *, *Obinna D. Ikwechegh*[1], *Chinonso S. Ezeanyaeji*[1]

[1]*Department of Mechanical Engineering, Faculty of Engineering and Technology, Alex Ekwueme Federal University Ndufu-Alike, Ikwo, Ebonyi State, 840001, Nigeria,* [2]*Department of Materials and Metallurgical Engineering, Federal University of Technology, Owerri, P.M.B. 1526, Owerri, Imo State, Nigeria*

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Development of novel leaf extract of *Dioscorea dumetorum*: A corrosion-resistant material for steel in HCl medium

ABSTRACT

The use of green corrosion inhibitors is one of the most efficient control methods for protecting metals from electrochemical reactions. In this study, Discorea dumetorum was explored as a potential corrosion inhibitor for mild steel in 1 M HCl medium using the weight loss technique, phytochemical screening, and optical microscopy. This novel inhibitor of plant origin is analyzed for anticorrosive characteristics in an acidic medium. The quantitative result of the phytochemical elements in Dioscorea dumetorum showed that it contains steroids, phenols, saponins, and tannins. The results obtained from weight loss measurement showed that the maximum inhibition efficiencies were 71.3%, 80%, and 76.9% from 1 g, 2 g, and 3 g concentrations, respectively, and that of the optical microscopy indicated pitting and intergranular corrosion on the sample surface, while the degree of surface coverage established the formation of protective film on the sample surface, which implies that the inhibitor is adsorbed at the interface between the mild steel and the acid medium. The theoretical fittings of the two adsorption models for the extract were found to obey the Langmuir's isotherm, with its adsorption-desorption equilibrium constant (K_{ads}) values between 0.6030 and 0.7858 g/l and R^2 of 0.3252 to 0.4555, while values of Gibb's energy of adsorption (ΔG°_{ads})are from -11.424 down to -7.3730 kJ mol^{-1} indicate electrostatic interaction between charged molecules and charged metal, indicating physisorption. The extract has proven its potential as an eco-friendly alternative to synthetic corrosion inhibitors.

Keywords*: Corrosion; mild steel; Dioscorea dumetorum; corrosion inhibition; physisorption*

1.0. INTRODUTION

Mild steel is a type of carbon steel with low carbon content [1,2], which is the most utilized type of steel due to its traits such as impressive mechanical and magnetic properties, not excluding affordability [3,4]. It has been employed for structural applications and for transportation of chemicals between different locations [4,5]. Acid solutions of varying concentrations are widely used in a variety of manufacturing procedures, such as acid pickling, chemical cleaning, and acid descaling, as well as oil well acidification, which causes corrosion [6,7,8].

*Corresponding author: Uzoma S. Nwigwe
E-mail: nwigweuzoma@gmail.com
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Corrosion entails the worsening of metal structure because of interaction with the environment [9,10]. This process results in the dilapidation of material's properties as a result of oxidation [9,11]. Figure 1 [6] shows the fundamental forms of corrosion.

Organic inhibitors have shown excellent efficiency in preventing corrosion of mild steel and have been widely researched by many authors. Table 1 shows a brief review of inhibitors of plant origin that has been used in combating this menace:

The aim of this research is to apply a novel extract of *Dioscorea dumetorum (D. dumetorum)* leaves, known as cluster yam which is native to West Africa [24, 25] to prevent corrosion of mild steel in HCl using weight loss measurement, phytochemical screening, and optical microscopy.

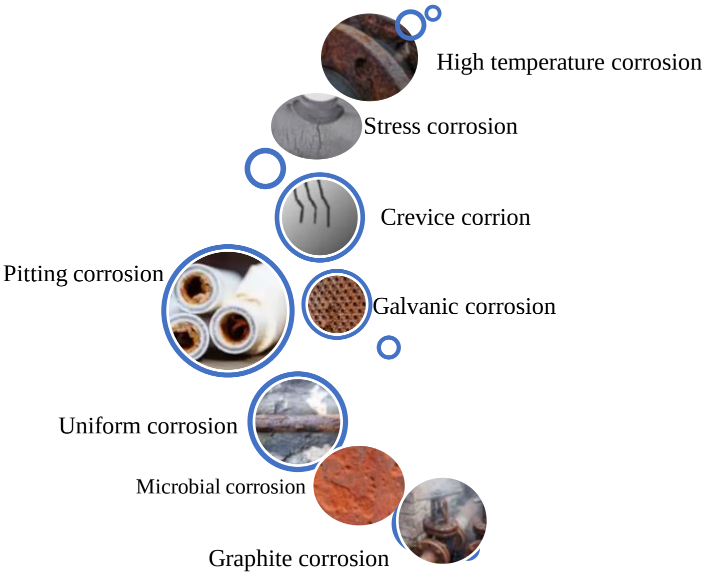


Figure 1. Types of corrosion

Table 1. Organic plant inhibitors for mild steel in various corrosive media

S/n	Plant	Plant part used	Corrosion medium	Maximum IE (%)	Reference
1.	Nauclea latifolia	leaves	1 M H_2SO_4	91.58	[7]
2.	Musa paradisiaca	peel	1 M HCl	71	[9]
3.	Azadirachta indica	leaves	1.5 H_2SO_4	71.89	[12]
4.	Irvingia gabonensis	leaves	1 M HCl	67.14	[13]
5.	Murraya koenigii	leaves	1 M H_2SO_4	82.01	[14]
6.	Carica papaya	leaves	1 M H_2SO_4	82.3	[14]
7.	Vernonia amygdalina	leaves	1 M HCl	88.5	[15]
8.	Jatroph acurcas	leaves	1 M H_2SO_4	92.05	[16]
9.	Eichhornia crassipes	leaves	0.5 M NaOH	99.53	[17]
10.	Alchornea laxiflora leaves	leaves	1 M HCl	96	[18]
11.	Cymbopogon citratus	leaves	sea water	34	[19]
12.	Olive	leaves	2 M HCl	53	[20]
13.	Neolamarckia cadamba	leaves	1 M HCl	84	[21]
14.	Tabernaemontana divaricata	leaves	1 M HCl	95.24	[22]
15.	Fenugreek	seed	0.5 M H_2SO_4	94.2	[23]

2. EXPERIMENTAL

2.1. Materials

The primary raw material required for this project is a mild steel sheet bought from the local market in Abakaliki, Ebonyi State, Nigeria, of composition manganese (0.6), carbon (0.15), silicon (0.03), phosphorus (0.36), and Fe (balance), according to the spark analysis at the National Metallurgical Development Centre, Jos, Nigeria. The mild steel was carefully marked and then cut using a hand cutter to create multiple coupon samples of the same size of 2.6 cm × 2.5 cm × 0.1 cm. Analytical-grade chemicals of HCl, methanol, and acetone were used. The purity levels of the reagents were above 99%, and the entire reagents were used without further purification. Other materials used are beakers, plastic containers, measuring cylinders, and weighing balances [26], [27], [12].The resulting amounts of 1, 1.5, 2, and 3 g were then weighed with a weighing scale and dissolved into five different 250 ml containers of 1 M HCl test solution, with the exception of the control, which did not include the extract.

2.2. Preparation of the extract

D. dumetorum leaves were obtained from Abakaliki city and identified at Alex Ekwueme Federal University's Botany Department in Ndufu-Alike, Ikwo, Nigeria. The leaf samples of *Dioscoreadumentorum* were first washed with distilled water to remove impurities and then dried under the sun for ten days to ensure complete removal of water. The leaves were later ground into powdery form using a mortar and pestle and sieved using an 18-mesh sieve that produced particles below 1.00 mm size. 30 g of the leaf sample was transferred into a container, and extraction was carried out by pouring 300 ml of methanol into the container and allowing the mixture to stand for 4 days for fermentation of the secondary metabolites. The extract was allowed to dry in order to expel the methanol [26], [27], [12]. The resulting amounts of 1, 1.5, 2, and 3 g were then weighed with a weighing scale and dissolved into five different 250 ml containers of 1 M HCl test solution, with the exception of the control, which did not include the extract.

2.3. Characterization of extracts from D. dumetorum leaves

The phytochemical analysis, which included qualitative analysis of the methanolic extract of *D. dumetorum*, was carried out to determine the presence of alkaloids, flavonoids, steroids, phenols, saponins, tannins, and tannin constituents. The phytochemical determination techniques employed in this study were modified from previously published reported by Ejikeme et al [28].

Test for saponin: A 0.3 g powder sample of the extract was mixed with 30 ml of distilled water, heated for 10 minutes in a water bath, and then filtered through Whatman filter paper. For a steady, long-lasting foam, 10 ml of the filtrate and 5 ml of distilled water were vigorously stirred. To see if an emulsion was forming, three drops of olive oil were added. When three drops of olive oil are added, an emulsion forms, which indicates a positive result.

Test for Tannin: A 0.3 g powder sample of the extract was mixed with 30 ml of distilled water and boiled for 10 minutes in a water bath. Whatman filter paper was used for the filtration process. 5 ml of the filtrate was measured into a test tube, and 3 drops of 0.1% ferric chloride were added. A brownish green or a blue-black colouration shows a positive test.

Test for steroids: 0.3 g of the sample was measured into a beaker and mixed with ethanol; the component was extracted for 2 hours. 5 ml of the ethanoic extract was poured into a test tube, and 2 ml of acetic anhydride, followed by 2 ml of concentrated tetraoxosulphate (VI) acid, was added. A violet to blue or green color change in the sample indicates the presence of steroids.

Test for flavonoids: 0.3 g of the extract was weighed into a beaker and 30 ml of water added. It was allowed to stand for 2 hours and filtered with Whatman paper. 10 ml of the filtrate was added 5 ml of 1.0 M dilute ammonia solution, followed by the addition of 5 ml of concentrated tetraoxosulphate (VI) acid. The appearance of yellow colouration that disappeared on standing shows the presence of flavonoid.

Test for phenols: 0.3 g of the extract was weighed into a beaker and 30 ml of water added. It was allowed to stand for 2 hours. A few drops of ferric chloride ($FeCl_3$) solution were added to a 5 ml aqueous solution of the leaf extract. The appearance of a greenish colouration indicates a positive result for the phenolic group.

Test for terpenoids: 0.3 g of the extract was weighed into a beaker and 30 ml of water added. It was allowed to stand for 2 hours. A mixture of 2 ml of chloroform and 3 ml of concentrated tetraoxosulphate (VI) acid was added to 5 ml of extract to form a layer. The presence of reddish-brown colouration at the interface shows a positive result for the presence of terpenoids.

Test for alkanoids: 5% tetraoxosulphate (VI) acid in 50% ethanol was added to 2 g of the extract and boiled in a water bath for 2 minutes. The mixture was filtered using Whatman filter paper. The filtrate was made alkaline using 5 ml of 28% ammonia solution (NH_3) in a separating funnel. 5 ml of chloroform was used in further solution extraction, in which chloroform solution was extracted with two 5 ml portions of 1.0 M dilute tetraoxosulphate (VI) acid. This final acid extract was then used to carry out the following test: 0.5 ml of Dragendorff's reagent was mixed with 2 ml of acid extract, and the precipitated orange colour indicates the presence of alkaloid.

2.4. Corrosion inhibition experiment and weight loss measurement

The experiments were conducted under total immersion of the metallic steel using containers containing 250 ml of prepared solution. Samples of mild steel were weighed and inserted into different concentrations of HCl with the help of threads. The mild steel samples were retrieved every 5 days. At the end of the exposure time, the mild steels were removed, dipped in acetone, and allowed to dry. The mild steel was re-weighed to determine the weight loss, in grammes (g) by the difference of mild steel weight before and after immersion.

Weight loss is the difference between weight of each coupon before and after immersion shown in equation (1) as reported [12].

$$\Delta W = W_o - W_f \qquad (1)$$

W_o is the initial weight of the metal (g) and W_f is the final weight of the metal (g).

The corrosion rate was calculated using equation (2):

$$CR = \frac{534\Delta W}{\rho AT} \quad (2)$$

where ΔW is the weight loss (g) after exposure time T (hours), A is the area (cm^2) of the specimen, ρ is the metal density and CR is the corrosion rate. The corrosion rate was used to calculate inhibition efficiency (IE %) in the presence of inhibitor using equations (3):

$$IE\ (\%) = \frac{C_{R1} - C_{R2}}{C_{R1}} \times 100 \quad (3)$$

where IE (%) is inhibition efficiency, C_{R1} is the corrosion rate of the mild steel absence of inhibitors and C_{R2} is the corrosion rate of the mild steel coupons in presence of concentration of inhibitors.

The degree of surface coverage can be calculated using equation (4):

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

where θ is the surface coverage, W_1 is the steel loss in weight in the presence of the inhibitor and W_2 is the mild steel loss in weight in the absence of inhibitor.

The overall area (A) of the rectangular steel samples used was estimated using the mathematical relation provided in reference [12].

3.0. RESULTS AND DISCUSSION

3.1. Phytochemical analysis

Result of the phytochemical analysis on *D. dumetorum* leaf extract is shown in the Table 2.

Table 2. Results of phytochemical analysis

Phytochemicals	Result
Alkaloids	-
Flavonoids	+
Steroids	+
Phenols	+
Saponins	+
Tannins	+
Terpenoids	-

Present +, absent -

The quantitative characterisation of the phytochemical constitutes of *D. dumetorum* showed it contains steroids, phenols, saponins, flavonoids, and tannins. Figure 2 shows the chemical structures of some phytochemicals tested, labeled a, b, c, and d, respectively.

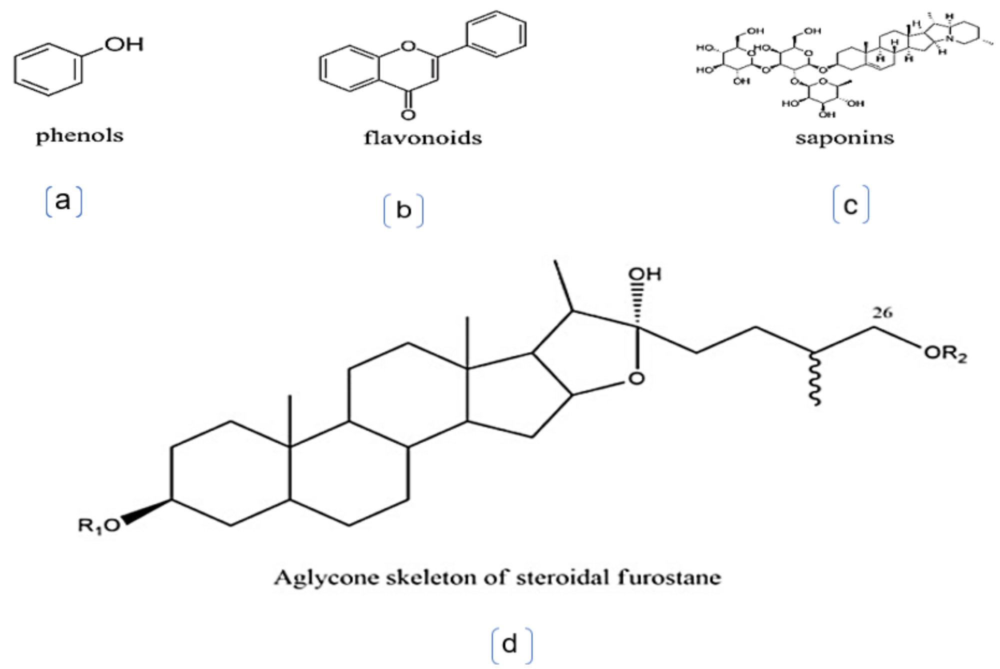


Figure 2 [a – d]. Chemical structures of some phytochemicals

3.2. The effect of extract concentrations on corrosion rate

Figure 3 shows how different *D. dumetorum* leaf extract concentrations affect the corrosion rate of mild steel in 1 M HCl. The weight loss measurements were used to track the corrosion rate. The findings reveal that the corrosion rate of mild steel in 1 M HCl medium reduces with increasing concentrations of the leaf extract. This is expected because as the concentration of the extract increases, there is an increase in the rate at which the phytochemical constituents are adsorbed on the surface of the mild steel, thereby creating a barrier for charge and mass transfer as is also suggested by Okoronkwo et al [29], this results in a decrease in the interaction between the metal and the corrosive medium and hence reduces the corrosion.

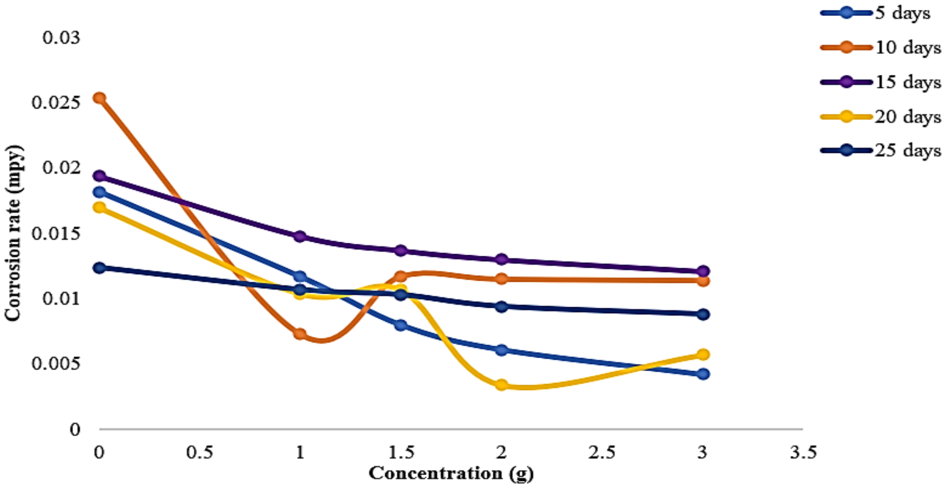
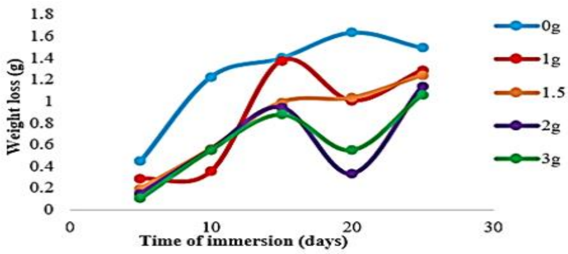


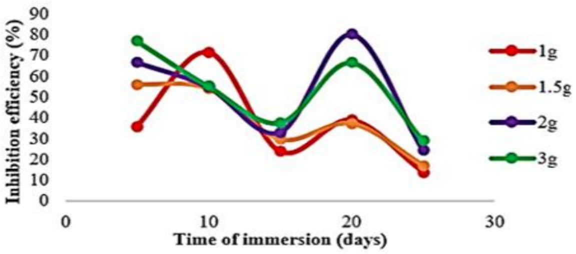
Figure 3. Corrosion rate (mpy) plotted against concentration of extract (g)

3.3. The effect of immersion time on weight loss

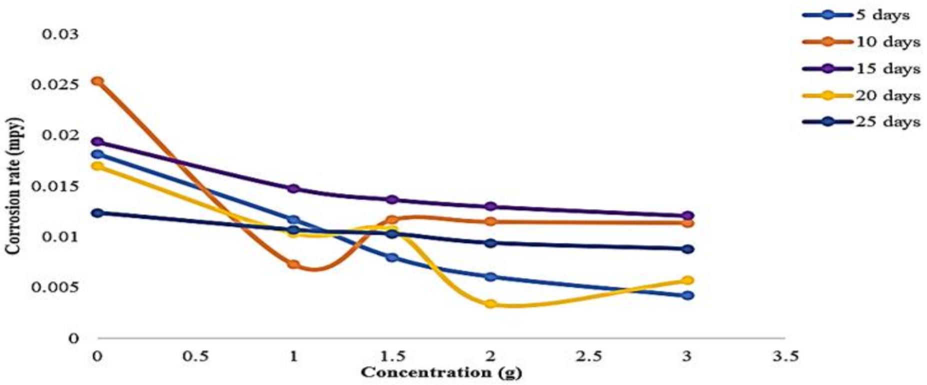
The coupons' loss of weight was measured in 1 M HCl in the absence and presence of the *D. dumetorum* leaf extract at various quantities to determine the efficacy of the extract's inhibitive action over time. Figure 4a shows that weight loss rises with increase in the immersion time. Nevertheless, the weight loss is substantially lower in the presence of the inhibitor than in the blank solution. The reduction in weight loss in the presence of the green inhibitor is due to the adsorption of the phytochemical elements in the leaf extract on the outer layer of the mild steel. Figure 4b shows that the inhibition efficiency decreased at day 15 and increased sharply at day 20. The corrosion rate showed an increase in the first 10 days of immersion and steadily decreased in the 15 days of immersion, as can be seen in Figure 4c.



(a)



(b)



(c)

Figure 4. Shows plots of weight loss against time (a), inhibition efficiency against time of immersion (b), and corrosion rate against time of immersion (c)

3.4. 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 5. This is attributed to an increase in the fraction of the mild steel surface covered by the extracts' absorbed molecules as concentrations grow. The maximum percentage inhibition of 71.3%, 80%% and 76.9% were recorded at inhibitor concentrations of 1 g, 2 g and 3 g, respectively.

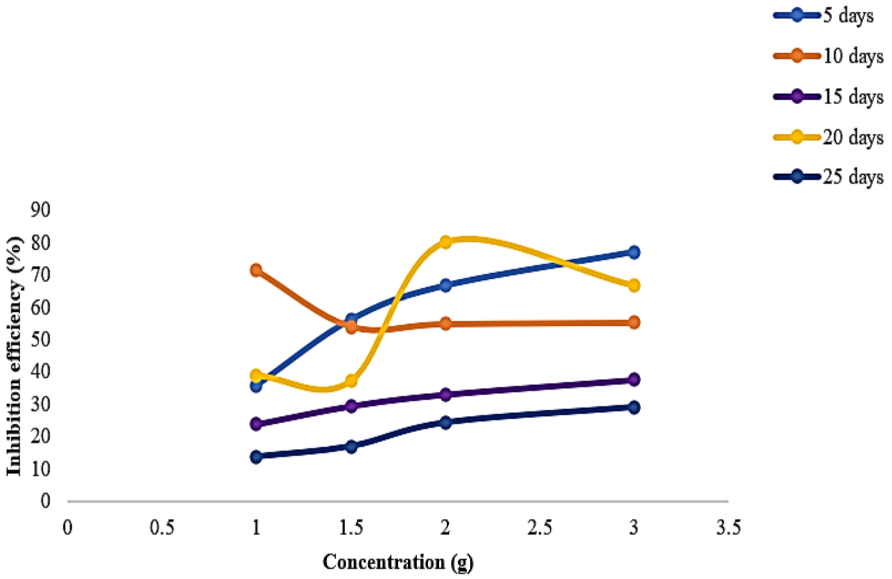
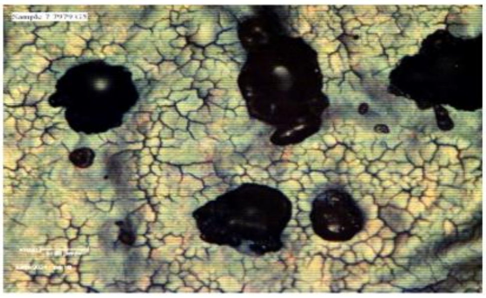


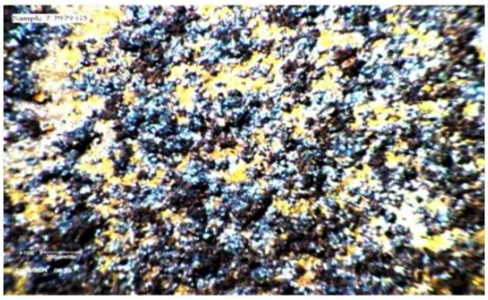
Figure 5. Inhibition efficiency (%) plotted against concentration of extract (g)

3.5. Optical microscopy

Optical microscopy of the mild steel coupons was carried out according to [30]. The submerged samples were first inspected without etching to avoid removing the extract's thin film protective effect on the specimens' surfaces. The samples were then polished, etched out with nital, and the surfaces were immediately examined under a microscope. Results are as shown in Figure 6 below.



(a) 2g after 20 days of immersion (etched)



(b) 2g after 20 days of immersion (unetched)

(c) 0g (uninhibited) after 20 days (etched)

(d) 0g (uninhibited) after 20 days (unetched)

Figure 6. Optical microscopy of inhibited and uninhibited samples

Figures 6a and 6c show the grains and boundaries of the mild steel coupon samples both in the inhibited and uninhibited environments. The pits on the samples seen in Figures 6b and 6d are believed to be formed by pitting corrosion, which is a localized attack that causes metal penetration at specific locations [31].

It is one of the most harmful and sneaky types of corrosion that causes equipment failures owing to perforation, while the microscopy view of the polished samples on Figures 6a and 6c depicts attack by intergranular corrosion, characterized by localized attack at or near the grain boundaries [32].

3.6. Adsorption Isotherm

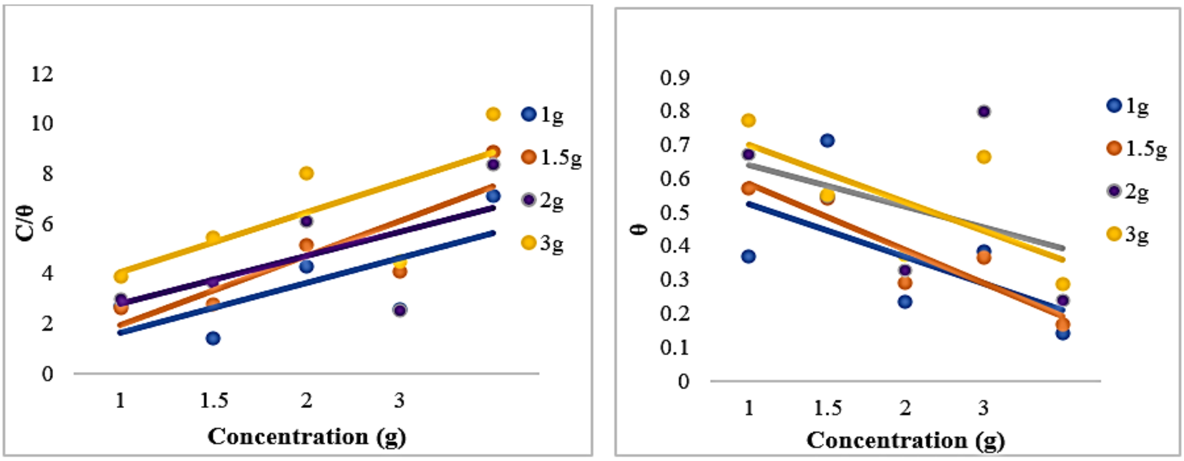
The adsorption mechanism of phytochemical constituents on a metal surface is described using the adsorption isotherm. The surface coverage (ϴ) that was used for the plot of the isotherms was calculated from the corrosion rate values using equation (4).

The obtained data were best fitted into Langmuir and Temkin adsorption isotherm using the relation shown in equations (7) and (8) [12].

Langmuir isotherm: $\frac{C}{\theta} = \frac{1}{K} + C$ *(7)*

Temkin isotherm: $exp(-2a\Theta) = KC$ *(8)*

Where C is the inhibitor concentration, θ is the surface coverage and K is the adsorption equilibrium constant.



(a) Langmuir plot (b) Temkin plot

Figure 7. Adsorption isotherm plots

Table 3. Adsorption parameters

Adsorption isotherm	Conc. of the extract (g)	Slope	Intercept	Regression	ΔG_{ads}(KJmol^{-1})
Langmuir	1	0.9981	0.6248	0.5140	-11.118
	1.5	1.3838	0.5522	0.7310	-11.424
	2	0.9665	1.8198	0.3832	-8.4690
	3	1.2056	2.8324	0.4967	-7.3730
Temkin	1	-0.0783	0.6030	0.3252	-8.6991
	1.5	-0.0979	0.6817	0.8322	-9.0032
	2	-0.0618	0.7026	0.1758	-9.0780
	3	-0.0852	0.7858	0.4555	-9.3553

3.7. Determination of free energy ΔG

Adsorption constant (K_{ads}) for Temkin and Langmuir isotherms were applied to compute the Gibbs free energy change of adsorption (ΔG°_{ads}) for the different inhibitor concentrations. The equilibrium constant of adsorption (K_{ads}) is related to ΔG°_{ads} as given by equation (9) [12]:

ΔG°_{ads} = *-2.303RT log (55.5K_{ads})* *(9)*

where R is the gas constant with its value given as (8.314 J. $K^{-1}mol^{-1}$), T stands for absolute temperature (K), and 55.5 is the water concentration in solution in mol L^{-1}.

The results from Table 3 shows that all values of ΔG°_{ads} are negative, this shows that the process is spontaneous, and that the *D. dumetorum* leaf extracts is effectively absorbed on the surface of the mild steel employing a physical absorption mechanism according to [18]. The values of ΔG°_{ads} up to -20 $kJmol^{-1}$ indicates electrostatic interaction between charged molecules and charged metal indicating physisorption, while those more negative than -40 $kJmol^{-1}$ involve charge sharing from the inhibitor molecules to the metal surface to form a co-ordinate type of bond otherwise known as chemisorption [18]. The theoretical fittings of the two adsorption models for the extract were found to obey the Temkin's isotherm, with its K_{ads} values between 0.6030 and 0.7858 g/l and R^2 of 0.3252 to 0.4555.

4. CONCLUSION AND FUTURE OUTLOOK

The development of a novel leaf extract of *D. dumetorum* as a corrosion-resistant material for steel in HCl medium has been duly explored. The study showed that:

1. The extract inhibited the corrosion of mild steel in 1 M HCl medium with maximum inhibition efficiencies of 71.3%, 80%, and 76.9% at 1 g, 2 g and 3 g concentrations, respectively. As a result, the extract's inhibitory efficiency increased with increasing concentration but declined with increasing exposure time.
2. The Temkin adsorption isotherm was confirmed with the computed Gibbs free energy change of adsorption values for the inhibitor recorded between the range of -8.6991 and -9.3553 kJ mol^{-1}.
3. *D. dumetorum* is a natural and biodegradable product that can serve as a cost-effective alternative to synthetic corrosion inhibitors.

Future experiments using various new leaf extracts other than *D. dumetorum* for corrosion inhibition of steel are suggested since the anticorrosion capability of a large number of plants remains undetermined as only a small percentage (<1%) of the world's 300,000 plant species have been studied for anticorrosive characteristics [33].

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IZVOD

RAZVOJ NOVOG EKSTRAKTA LISTA *DIOSCOREA DUMENTORUM:* MATERIJAL OTPORAN NA KOROZIJU ZA ČELIK U HCl MEDIJUMU

Upotreba zelenih inhibitora korozije jedna je od najefikasnijih metoda kontrole zaštite metala od elektrohemijskih reakcija. U ovoj studiji, dioskoreja (Dioscorea dumentorum) je istražena kao potencijalni inhibitor korozije za meki čelik u 1 M HCl medijumu korišćenjem tehnike gubitka težine, fitohemijskog skrininga i optičke mikroskopije. Ovaj novi inhibitor biljnog porekla analiziran je na antikorozivne karakteristike u kiseloj sredini. Kvantitativni rezultat fitohemijskih elemenata u dioskoreji (Dioscorea dumentorum) pokazao je da sadrži steroide, fenole, saponine i tanine. Rezultati dobijeni merenjem gubitka težine pokazali su da je maksimalna efikasnost inhibicije bila 71,3%, 80% i 76,9% za koncentracije od 1 g, 2 g i 3 g, respektivno, a optička mikroskopija je ukazala na tačkastu i interkristalnu koroziju na površini uzorka, dok je stepen pokrivenosti površine utvrdio formiranje zaštitnog filma na površini uzorka, što implicira da je inhibitor adsorbovan na granici između mekog čelika i kisele sredine. Teorijsko podešavanje dva modela adsorpcije za ekstrakt pokazalo se kao da poštuje Langmirovu izotermu, sa vrednostima konstante ravnoteže adsorpcije-desorpcije (Kads) između 0,6030 i 0,7858 g/l i R2 od 0,3252 do 0,4555, dok su vrednosti Gibove energije adsorpcije (〖∆G〗_ads^°) od -11,424 do -7,3730 kJ mol-1, što ukazuje na elektrostatičku interakciju između naelektrisanih molekula i naelektrisanog metala, što ukazuje na fizisorpciju. Ekstrakt je dokazao svoj potencijal kao ekološki prihvatljiva alternativa sintetičkim inhibitorima korozije.

Ključne reči: *Korozija; meki čelik; Dioscorea dumentorum; inhibicija korozije; fizisorpcija*

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Uzoma S. Nwigwe http://orcid.org/0000-0002-5712-9812
Obinna D. Ikwechegh https://orcid.org/0009-0003-8421-0223
Chinonso S. Ezeanyaeji https://orcid.org/0009-0007-4691-6670