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SYNTHESIS OF NEW BIOACTIVE LEAF EXTRACT FOR CORROSION INHIBITION OF STEEL PIPELINES IN THE ACIDIC MEDIUM OF OIL AND GAS INDUSTRY

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ABSTRACT

This manuscript investigated the corrosion inhibition and adsorption behavior of Dioscorea alata leaf extract on mild steel to explore their bioactive anti-corrosion potentials in 1M H_2SO_4 environment. It used chemical and electrochemical methods and phytochemical screening. The weight loss measurement revealed that samples with 1 to 5 g/300 ml extract recorded maximum inhibitory efficiency of 72.4, 78.4, 81.1, 81.5, and 83.3 %, respectively, within their first 5 days. Generally, their inhibitory efficacy has been observed to decrease with greater exposure time, while the computed Gibbs free energy of the adsorption process ($\Delta G°_{ads}$) values from the weight loss data ranged from -9.5491 to -9.7165 kJ mol^{-1}, and the adsorption isotherm measurements corresponds well with the Temkin adsorption isotherm, demonstrating a physisorption reaction between the extract molecules and the surface of the coupons. Phytochemical screening showed steroids, saponins, and terpenes were active ingredients in the extract. On the other hand, the potentiodynamic test produced maximum percentage inhibition efficiency of 97.99, 31.51, 97.30, and 98.17 %, respectively, for 1 to 5 g/300 ml extract concentrations. The highest corrosion rate of $9.905e^{+003}$ mpy was obtained from the sample without extract. Based on the corrosion inhibition effectiveness of the extract obtained through both methods, it is suggested that the extract be added in the production of paints that are used to cover pipelines in the oil and gas industry. This is because it has chemicals in it like saponins, terpenes, and steroids that can stick to the metal's surface and inhibit corrosion in acidic media.

Keywords: sulfuric acid; adsorption isotherm; phytochemical compounds; Dioscorea alata; mild steel.

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1. Introduction

The importance of mild steel to man cannot be overstated, owing to its wide range of applications in the manufacturing, automobile, processing, and production industries, among others. As an alloy of iron [Fe] and carbon [C], mild steels popularity and favorability in use above many other metals and alloys are due to their outstanding mechanical properties, affordability, high quality, and availability [1-3]. Notwithstanding, mild steel has a particularly obvious flaw which is its weak resistance to corrosion attacks [2, 4, 5], which results in structural defects, deterioration in mechanical properties, and aesthetic depreciation of the mild steel. Metal surfaces deteriorate and wear away due to corrosion, which is caused by chemical reactions with the environment that produce more stable compounds like hydroxides, sulphides, oxides, etc. [6-10]. Metallic corrosion is a serious worldwide concern that can lead to monetary losses, hazards to one's health and safety, and even death. [11-13]. The service life of mild steel can be characterized by acidic, salty or alkaline environments, polluted atmospheres, and high temperatures among others. All these contributes to the corrosion of the mild steel equipment [2, 14, 15].

The practice of inhibition is an appealing method used to reduce the corrosive attack on metallic materials [4, 5]. Chemicals known as corrosion inhibitors slow down the cathodic and/or anodic reaction or provide a barrier by forming an adsorbed coating when applied in small quantity into a corrosive medium. [1, 5, 16-18]. The toxicity and high cost of most currently used inorganic inhibitors make the development of affordable and ecologically friendly substitutes essential [1, 3-5, 19-21]. Therefore, plant extracts are increasingly viewed as sources of green corrosion inhibitors, due to environmental considerations. They replace hazardous and environmentally unfriendly chemicals in shielding metals and alloys from hostile conditions [1]. Leaf products are naturally occurring and contain certain photochemical compounds that can be extracted using easy and inexpensive methods, including flavonoids, tannins, saponins, organic and amino acids, pigments, and alkaloids [5, 22, 23]. Some

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Table 1
Review on some plant extracts efficiency

Plant extract	Corrosion medium	Highest efficiency (%)	Reference
Irvingia Gabonensis	HCl	67.14	[2]
Vernonia Amygdalina	HCl	80.85	[24]
Corchorus olitorius	KOH	99.93	[1]
Dioscorea spp	NaOH, NaCl, H_2SO_4	92	[4]
Azadirachta Indica	H_2SO_4	72.03	[5]
Alchornea laxiflora	HCl	96	[25]
Chromolaena Odorata	HCl, H_2SO_4	69. 82	[6]
Murraya Koenigii	H_2SO_4	82.01	[26]
Paederia foetida	HCl	73.77	[27]
Azadirachita indica	H_2SO_4	85.1	[26]
Carica papaya	H_2SO_4	82.3	[26]

studies have shown that extracts from different leaves have been widely reported as effective and good metal corrosion inhibitors in various corrosive media. They are included in table 1 below.

The new leaf extract Dioscorea alata has seldom been investigated for its potential to prevent mild steel from corroding in acidic medium. This research work aims to study to the efficiency of this leave extract in the corrosion protection of mild steel in sulfuric medium. Methods such as phytochemical screening, weight loss analysis, potentiodynamic polarization test, and visual analysis were used in this study. This research hopes to add to the existing knowledge on plant extracts as green corrosion inhibitors and contribute to the field of corrosion studies at large.

2. Experiment

2.1. Materials

The composition of the mild steel and the experimental procedure used for this study were as stated on [5]. The mild steel was pressed cut with hand shearer to produce numerous replicas of the coupons with the same dimensions of 2.6×2.5×0.1 cm with a hole drilled in the center of each coupon and the chemicals used were of Analytical grade (fig. 1).

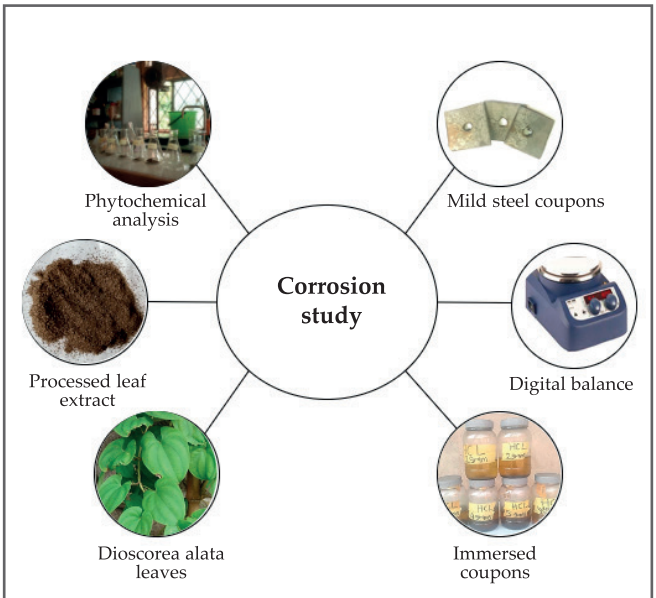

Fig. 1. Experimental setup for corrosion studies

2.2. The preparation of Dioscorea alata leave extract

Fresh Dioscorea alata leaves were procured from a farm in Rivers state, Nigeria. and identified at Alex Ekwueme Federal University's Botany Department in Ndufu-Alike, Ikwo, Nigeria. They were dried for 10 days under the scorching sun to remove their moisture content, then they were grounded into powdered form and stored. The powdered leaf extract was soaked in methanol for 7 days to expose its secondary metabolites after which it was spread and allowed to dry by the evaporation of methanol and then utilized as the pure form corrosion inhibitor. The Dioscorea alata extracts was measured and dissolved into five different containers with 300 ml of 1M of H_2SO_4 solution at respective concentrations of 1, 2, 3, 4 and 5 g.

2.3. Qualitative analysis of phytochemicals

To look for secondary metabolites, such as steroids, terpenes, saponins, alkaloids, flavonoids, tannins, and phenols, qualitative analysis was done at Food Science Technology Laboratory, Ebonyi State University, Abakaliki, Ebonyi State, Nigeria to determine the presence of these phytochemicals on the powdered Dioscorea alata leaves, according to the standard procedure [3, 29].

2.4. Corrosion medium preparation for gravimetric analysis

A 1M H_2SO_4 solution was prepared as the corrosion medium for carter for both inhibited and uninhibited cases. For the inhibited corrosion medium, the solid residue of the Dioscorea alata leaf extract was added in different concentrations (1, 2, 3, 4 and 5 g) in five containers, while the uninhibited medium has the sixth container as the control. The coupons were immersed in the different media by means of a cotton thread that was suspended in the container using tapes (which recorded each coupon's original weight and immersion number), and sufficient anaerobic conditions were ensured by tightly covering the containers with their restraining lid. The mild steel coupons submerged in the

H_2SO_4 solution containers were left for two days to allow the inhibitor to fully soak onto the coupons. After then, coupons were taken out every three days because of the medium's high acidity level. The first set was taken out after five days, while the succeeding sets were taken out every three days (72 hours). None of the coupons were allowed to touch each other to avoid galvanic and crevice corrosion [4]. The mild steels were taken out after the exposure period, thoroughly rinsed to get rid of the corrosion stain product, and then dried in acetone [3, 28].

2.5. Weight loss calculation

The weight loss was determined by comparing the pre-immersion and post-immersion weights of each coupon according to [26] given as:

$$\text{Weight loss } (\Delta W) = W_{initial} - W_{final} \quad (1)$$

Corrosion rate was calculated by the formula:

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

where, CR is the corrosion rate in miles per year (mpy), 534 is a constant, ΔW is the weight loss in grams (g), is the density of mild steel (7.87 g/cm^3), A=the total surface area (cm^2), and T is the exposure time in hours (hrs).

$$\text{Inhibition efficiency} = \left[\frac{CR_{0g} - CR_{inh}}{CR_{0g}}\right] \times 100\% \quad (3)$$

where CR_{0g} and CR_{inh} are the corrosion rates in the absence and presence of plant extracts. The calculated results are displayed in table 2.

$$\theta = \frac{W_{out} - W_{in}}{W_{out}} \quad (4)$$

where W_{out} and W_{in} are weight loss with and without inhibitor respectively, and θ is the degree of surface coverage.

2.6. Potentiodynamic polarization test

The potentiodynamic polarization technique was carried performed using the method as described in reference [5]. The Dioscorea alata leaf extract at concentrations of 1, 2, 3, 4 and 5 g was used in 1M H_2SO_4 test solution.

2.7. Calculation of corrosion inhibition by photodynamic polarization test

The percentage inhibition efficiency (%IE) using photodynamic polarization test was obtained in accordance to reference [1]

$$\%IE = \left[\frac{I_{corr(out)} - I_{corr}(in)}{I_{corr}(out)}\right] \times 100 \quad (5)$$

where, $I_{corr(in)}$=corrosion current with inhibitor, and $I_{corr(out)}$= =corrosion current without inhibitor.

2.8. The degree of surface coverage by photodynamic polarization test

The degree of surface coverage,

$$\theta = (\%IE/100) \quad (6)$$

where %IE=percentage inhibition efficiency of the extract (%IE) in the solution (1M of H_2SO_4), [1].

Table 2

Phytochemicals constituents						
Alkaloids	Saponins	Flavonoids	Phenols	Tannins	Steroids	Terpenes
–	+	–	–	–	+	+

3. Results and discussions

3.1. Results of phytochemical analysis

The results of the phytochemical tests carried out on the Dioscorea alata leaf extract is shown in the table below:

In table 2 are the present secondary metabolites, which include saponins, steroids, and terpenes, while flavonoids, alkaloids, tannins, and phenols were absent in the Dioscorea alata leaf extract.

3.2. Gravimetric analysis result

Results from gravimetric analysis showed that all specimens experienced varying degrees of weight loss in their respective concentrations in H_2SO_4 medium, with a uniform attack on the coupons, that were uniformly spread across the surface area of the coupons, thus resulting in the thickness reduction of the coupons. In this medium, 0 g concentration specimens experienced minimal pitting corrosion as well, which was likewise assumed to be due to the absence of inhibition [11]. The highest weight loss in this medium was 4.4125 g for a 0 g concentration. Samples in 1, 2, 3, 4 and 5 g concentrations indicated their best inhibition efficiencies (74.4, 78.4, 81.1, 81.5 and 83.8 %) after the first 5 days. The least inhibition efficiency of 28.4% was recorded by 1 g concentration on the last day (day 17). It was also observed that inhibition efficiency generally decreased with exposure time.

Figure 2 to 7 shows the graphs of multiple variables on mild steel surface when exposed to 1M of H_2SO_4 medium without and with Dioscorea alata extract at different concentrations.

Figure 2 shows the relationship between weight loss and time. It was discovered that weight loss increased with increasing exposure time, which also agrees with studies carried out by U. S. Nwigwe et al. and Ekeke et al., respectively [5, 29]. The uninhibited coupons experienced the highest weight loss by the 14th day but dropped in the subsequent days after, which was attributed to the possible formation of passivation on the metal surface. Figure 3 shows the variation of inhibition efficiency against time. Inhibition efficiency was highest in all samples at 1, 2, 3, 4 and 5 g concentrations, indicating the best inhibition efficiencies (74.4, 78.4, 81.1, 81.5, and 83.3 %) on their first 5 days, respectively. More so, the 3 g concentration still maintained its high efficiency (81.1%) on the 8th day. On the final day (day 17), extract concentrations of 1 and 3 g had the lowest inhibitory efficiencies of 28.4 and 42.6 %, respectively. Figure 4 showed the variation of weight loss against concentration. It was discovered that different concentrations experienced varying degrees of weight loss on different days. The weight loss was discovered to increase over longer time intervals. Figure 5 shows the variation of corrosion rate against time of the mild steel in the absence and presence of the Dioscorea alata leaf extract. Inhibited coupons showed a greatly reduced corrosion rate across their

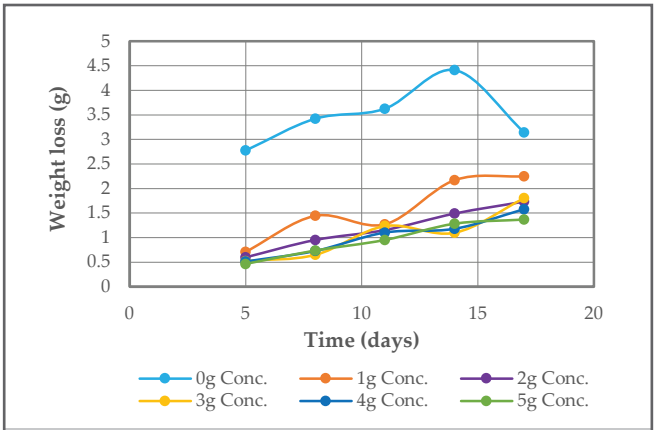

Fig. 2. Plot of weight loss (g) vs. time of immersion

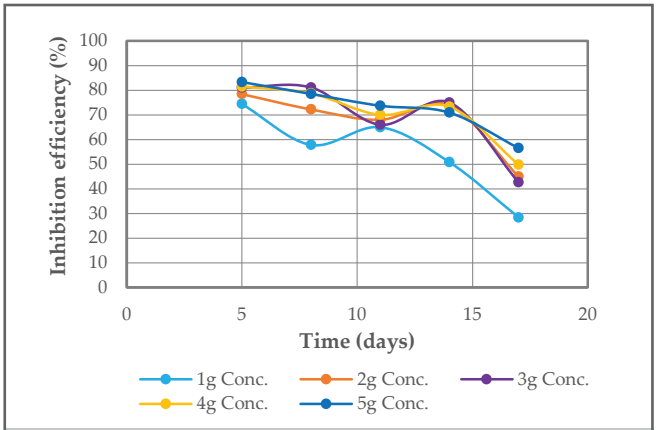

Fig. 3. Plot of inhibition efficiency vs. time (days)

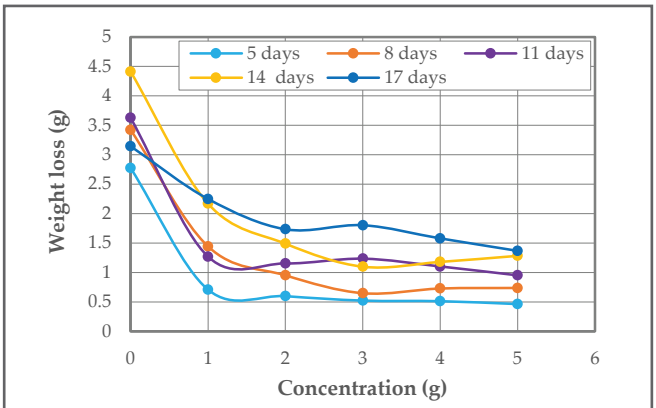

Fig. 4. Plot of weight loss vs. concentration (g)

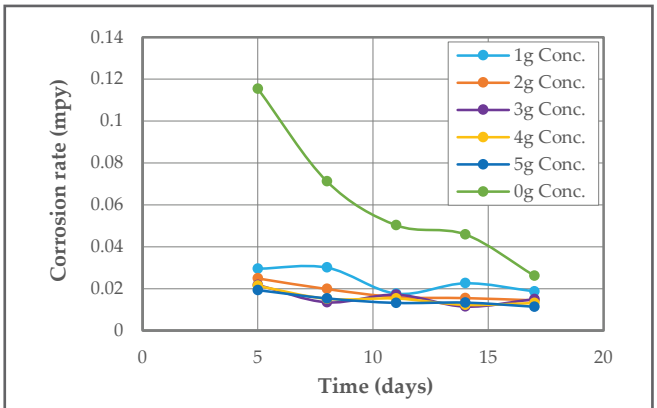

Fig. 5. Plot of corrosion rate vs. time (days)

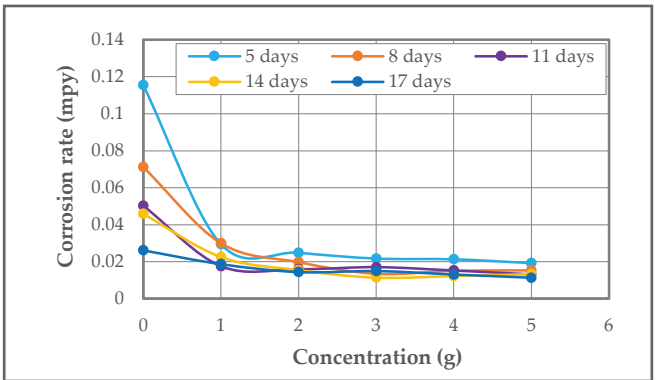

Fig. 6. Plot of corrosion rate vs. concentration (g)

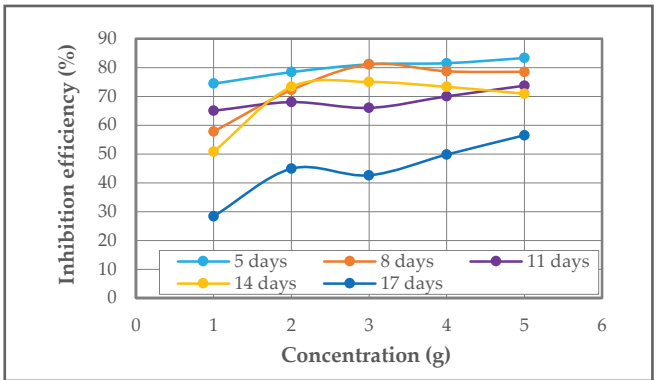

Fig. 7. Plot of inhibition efficiency vs. concentration (g)

different concentrations as compared to their uninhibited neighbors, which had a substantially higher corrosion rate of about 0.11539 mm/yr. It was also discovered that uninhibited coupons recorded a decrease in corrosion rate by day 14, and more so by day 17. This could be attributed to the formation of passivation [13, 16] on the mild steel surface. Figure 6 shows the relationship between corrosion rate and concentration. For the uninhibited medium, it was discovered that at day 5 the corrosion rate was highest and at day 17 it was least. Generally, it was discovered that the corrosion rate almost uniformly reduced with an increase in extract concentration. Figure 7 summarizes the relationship between the inhibition efficiency and the concentration of extract. It was observed that inhibition efficiency gradually dropped over time, albeit not uniformly (day 11).

3.3 Potentiodynamic polarization measurement

The figure 8 shows the image of the polarization curves.

The result of the polarization curves indicates that the inhibitor functions as a mixed-type inhibitor, spreading the curves to both the anodic and cathodic reactions, as shown in figure 8. This agrees with the mode of function of organic inhibitors of plant origins [30]

The result of the polarization curve for the coupons reported corrosion rates of $9.905e^{+003}$, $1.989e^{+004}$, $6.783e^{+003}$, $2.665e^{+004}$ and $1.811e^{+004}$ in (mil/year), for the various concentrations

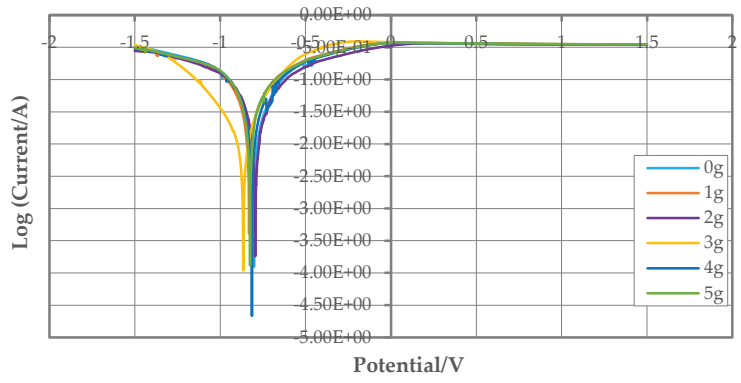

Fig. 8. Tafel graphs of corrosion potentials versus corrosion currents as were obtained from potentiodynamic polarization analysis of the coupons in 1M of H_2SO_4

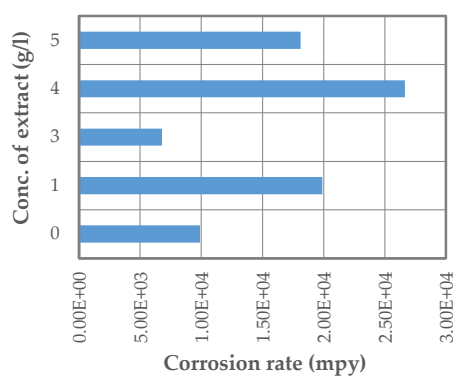

Fig. 9. Graph of concentration of extract against corrosion rate by polarization test

of the extract from 0 to 5g respectively for 1M of H_2SO_4 at room temperature as shown in the plot above (fig. 9).

3.4 Adsorption isotherm

The adsorption isotherm shows the direct relationship between the degrees of surface coverage with the corrosion inhibition efficiency. It provides the basic information about the nature of interaction between the mild steel surface and inhibitor molecular constituents [5, 16].

From the gravimetric test, the degree of surface coverage (θ) at different extract concentrations in 1M of H_2SO_4 medium was applied to evaluate the best isotherm that fits the experimental parameters using the mathematical expression provided in reference [5].

$$\exp(-2\alpha\theta) = KC_{inh} \quad \text{(Temkin isotherm)} \qquad (7)$$

where θ=degree of surface coverage of the extract, C_{inh}=concentration of the extract, and K=adsorption equilibrium constant.

The graphs of θ against C_{inh} are shown in the figure 10.

The linear plots obtained, followed Temkin isotherm shown by its good regression coefficients (R^2), The graphs of θ against C_{inh} has the best fitted lines and their linear correlation coefficients were almost equal to unity, just like that of the slopes, hence the extract adsorption on the mild steel surface conformed with the Temkin adsorption isotherm.

Concentrations 1 g/300 ml to 5 g/300 ml, respectively. The equilibrium constant value of adsorption (K) has a relationship with the standard free energy of adsorption (ΔG°_{ads}), expressed as below [5]:

$$\Delta G^{\circ}_{ads} = -2.303RT \log\left(55.5K\right) \qquad (8)$$

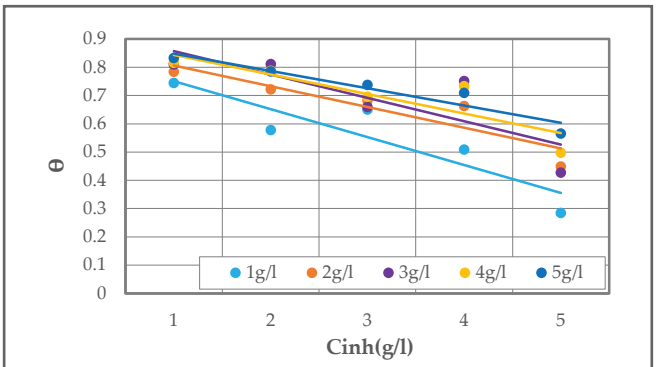

Fig. 10. Adsorption plot for Temkin isotherm on the coupons with Dioscorea alata extract in 1M H_2S0_4 medium

where R is the gas constant value (8.314 $J.K^{-1}mol^{-1}$), T is the measurement of temperature on the Kelvin scale (K), and 55.5 is the water concentration in solution given in mol L^{-1}.

The plot above shows the adsorption parameters fitting well, thus obeying the Temkin isotherm. The reported standard free energy of adsorption (ΔG°_{ads}) were all negative. Physisorption was confirmed for the medium, which was associated with the interaction of the charged molecules of the extract and that of the mild steel. Thus, in this study, the Dioscorea alata extract values for ΔG°_{ads} were found to range from –9.5491 to –9.7983 $KJmol^{-1}$, indicating that the adsorption model of the Dioscorea alata extract on the mild steel surface aligned with a physical adsorption process [5]. The linear plots obtained for this medium (H_2SO_4) followed the Temkin isotherm, as shown by its also good regression coefficients (R^2). The graphs of the degree of surface cov-

Table 3

Adsorption isotherm thermodynamic data

	Conc. of extract	Slope	Intercept	R^2	ΔG°_{ads} ($KJmol^{-1}$)
Temkin isotherm	1	-0.0989	0.8497	0.8112	-9.5491
	2	-0.0731	0.8791	0.829	-9.6334
	3	-0.0827	0.9396	0.6634	-9.7983
	4	-0.069	0.9126	0.758	-9.7260
	5	-0.0611	0.9091	0.9066	-9.7165

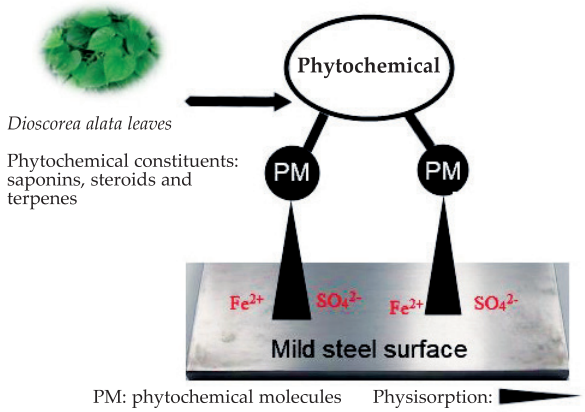

Fig. 11. Plot of corrosion rate vs. concentration (g)

erage versus extract concentrations show the best-fitting straight lines since all of the linear regression coefficients are almost equal to unity, and the slope values are also very close to one, demonstrating that extract adsorption on the mild steel surface follows the Temkin adsorption isotherm (table 3).

3.5 Mechanism of corrosion inhibition of Dioscorea alata leaf extract on mild steel in in 1M H_2SO_4

The phytochemical constituents present in Dioscorea alata leaf extract, as shown in table 2, were able to adsorb onto the surface of the mild steel by forming a protective film, which prevented the corrosive acid from coming into contact with the metal. Physisorption was confirmed in accordance with [5] as the chemical mechanism by which the extract safeguards the steel surface in the acid medium (fig. 11).

Conclusions

The study concludes as follows:

1. The samples with 1 to 5 g/300 ml extract had the highest inhibitory efficiency of 72.4, 78.4, 81.1, 81.5, and 83.3 %, respectively, after 5 days, as obtained using the weight loss test.
2. The extract inhibited the corrosion of mild steel in H_2SO_4 even as its inhibitory efficiency increased with increasing extract concentration but declined with increasing exposure time.
3. The computed Gibbs free energy of adsorption process values by weight loss data ranged from –9.5491 to –9.7165 KJ mol^{-1}, with the adsorption isotherm parameters fitting well into the Temkin adsorption isotherm.
4. The potentiodynamic polarization test produced maximum percentage inhibition efficiency of 97.99, 31.51, 97.30, and 98.17n%, respectively, for 1 to 5 g/300 ml extract concentrations. And the highest corrosion rate of 9.905e^{+003} mpy was obtained from the sample without extract.
5. Based on the corrosion inhibition efficacy of the extract obtained using both procedures, it is believed that the Dioscorea alata leaf extract can be utilized to prevent corrosion assaults of metallic materials in acidic environments.

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