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BIOMASS EXTRACT FROM *VIGNA SUBTERRANEA* LEAF IMMERSED IN HCL FOR CORROSION MITIGATION OF STEEL IN AN OIL WELL ENVIRONMENT

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ABSTRACT

In an effort to reduce the corrosion impact on mild steel within the oil well industry, Vigna subterranea leaf extract (VSLE), utilized as a green biomass and immersed in HCl, was employed as a corrosion inhibitor. An analysis of the phytochemicals present in the extract was conducted, and the elemental makeup of the mild steel was determined. The weight loss method was evaluated through gravimetric analysis. Kinetic and thermodynamic parameters were assessed, and the adsorption isotherm was analyzed using the Langmuir isotherm model. The results indicate that the steel's composition was predominantly iron (Fe), accounting for 97.26%. The phytochemical analysis of the extract revealed the presence of flavonoids, phenols, saponins, alkaloids, tannins, steroids, and terpenoids within the organic biomass extract. A low rate of corrosion (ROC) and a high efficiency of inhibition (EOI) were recorded. The Langmuir isotherm model was determined to be the best fit, effectively describing the corrosion inhibition mechanism of mild steel. According to the thermodynamic data, the negative ΔG_ads suggests that the adsorption process is chemisorption. The study concluded that the VSLE extract, when treated with hydrochloric acid, could function as an effective inhibitor for steel corrosion in an oil well environment.

KEYWORDS: *Vigna subterranea* leaf extract, Corrosion, Green Biomass, Rate of corrosion, Efficiency of Inhibition, Langmuir isotherm, phytochemical analysis.

INTRODUCTION

Corrosion is defined as the damaging process through which a material interacts with its environment, representing a natural risk in the production and transportation of oil and gas. This phenomenon can take place in various aqueous settings and is affected by intricate conditions present in the oil and gas production and pipeline systems. The process consists of three elements: an anode, an electrolyte, and a cathode (Oghenerukevwe et al., 2023). The oil and gas production sector faces substantial financial burdens due to corrosion. On a global scale, the yearly costs associated with pipelines and facilities are estimated to be $1.40 billion, while expenses for down hole tubing amount to $460 million, in addition to corrosion-related capital expenditures (Benziane *et al.*, 2021). Hence, the urgent needs to reduce the lost due to corrosion.

The used of acid concentration “reducers” otherwise known as inhibitors/additives have been reportedly researched and found to help reduced the initial acid effects on oil plants equipment in central process units (CPU) of refineries during corrosion control (Oghenerukevwe et al., 2024). These reducers comes in form of green biomass leaves extract, and it has been reported that a strong acid (such as HCl) at greater than or equal to 15% concentration when mixed with organic biomass leaves extract, can basically control corrosion rate (Al-Moubaraki *et al*., 2021).

This study therefore employed *Vigna subterranea* leaves extract in hydrochloric acid for the corrosion control of tubing steel in an oil well.

Materials and Methods

Materials

Freshly harvested *Vigna subterranea* leaf (VSL) was obtained farmer in Isoko North Local government, Ozoro, Delta State, Nigeria. The leaf was made cleaned by washing with ionized water and dried in the oven at 40 ^oC until the moisture content reduced to <0.02% . The dried leave leaf was milled into powder form, and was allowed to pass through a mesh sieve size 0.35 µm to improve its surface area during extraction.

The corroded steel tubing pipe (CSTP) identified to be SAE 4140 graded was obtained from steel company located in Asaba, Delta State, Nigeria. The CSTP was made cleaned by screed with sand papers, washed with acetone, washed, and then dried. The composition of the cleaned CSTP was made known by characterized the cleaned CSTP by AMSA (atomic mass spectrometry analyzer).

Figure 1: *Vigna subterranea* leaves. (VSL)

Methods

Acetone extraction VSL

The extract was obtained from the milled VSL powder using acetone as organic solvent due to its low boiling point (56 ^oC), this process was carried out using Soxhlet extractor placed in the heating mantle. The excess acetone in the extract was recovered using a rotary evaporator, and the pure *VSL* extract (VSLE) was obtained by filtration. Phytochemical analyses VSLE were examined before the extract was acidified by immersion in 0.5 - 1.5 g/L of 15% HCl acid at temperature range of 333 -353 K. The efficiency of inhibition (EOI) and the rate of corrosion (ROC) were obtained via the weight loss measurements (Hazzan *et al*., 2016).

Gravimetric method

To evaluate the weight loss, surface coverage, EOI, and ROC, the method of gravimetric method was conducted prior to immersion. The procedures were as reported by Oghenerukevwe et al. (2024). The equations employed to determine the weight loss, surface coverage, EOI, and ROC were indicated in Equation 1-4.

$$\text{Weight lost } (\varphi) = W_{bf} - W_{af} \quad (1)$$

$$\text{ROC (mm/yr)} = \frac{676\ \varphi}{10\ A \times T \times \rho} \quad (2)$$

$$\text{Surface coverage } (\gamma) = \frac{ROC_{ainh} - ROC_{pinh}}{ROC_{pinh}} \quad (3)$$

$$\text{EOI } (\%) = \frac{ROC_0 - ROC_1}{ROC_0} \times 100 \quad (4)$$

Where: W_{bf} and W_{af} are the weight before and after immersion, ROC_{ainh} is the rate of corrosion in the presence, while and ROC_{pinh} is the rate of corrosion in the absence of inhibitor. The total surface area is represented by A steel, ρ is the density g/cm^3, 676/10 is the rate of corrosion constant, and T is the immersion time.

Kine-thermo parameters analysis

The kine-thermo parameter analysis was evaluated by considering the kinetic of reaction and the thermodynamics properties. The activation energy (E_a) was determined via kinetic study employing Arrhenius equation (Eqn. 5). The enthalpy (ΔH_a^o) and the entropy (ΔS_a^o) were obtained using the modified Eyring Polanyi Eqn. (6), while the Gibb's free energy (ΔG_{ads}) at temperatures of 333 K was also evaluated using Eqn. (7).

$$ln\ ROC = -\frac{E_a}{2.303RT} + ln\ A \quad (5)$$

$$\log\left(\frac{ROC}{T}\right) = \left[\log\left(\frac{R}{Nh}\right) + \frac{\Delta S_a^o}{2.303R}\right] - \frac{\Delta H_a^o}{2.303RT} \quad (6)$$

$$\Delta G_{ads} = -RT\ln(C_{solvent}K_{ads}) \quad (7)$$

Where; R = gas constant (8.3145J/mol.k), h = Planck's constant, N = Avogadro's number, T = Absolute temperature (K), ΔS_a^o = Apparent entropy of activation (J/mol. k), ΔH_a^o =

Apparent enthalpy of activation (KJ/mol), K_{ads} = the equilibrium constant of adsorption (l/g), $C_{solvent}$ = concentration of water in solution (g/L).

Adsorption isotherm study

The nature of adsorption was examined using Langmuir isotherm. Reports revealed that almost 99% adsorption process obey Langmuir isotherm equation, hence its application (Oghenerukevwe *et al*., 2023; Oghenerukevwe *et al*., 2024; Udensi et al., 2020; Benziane et al., 2021). The Langmuir (Eqn. 8) isotherm was examined, the intercepts and the slopes were evaluated from the plots, the value of adsorption equilibrium constant (K) deduced from the isotherm was employed to compute the "ΔG_{ads}" of the adsorption.

$$\frac{I_C}{\gamma} = \frac{1}{K_{ads}} + I_C \qquad (8)$$

Where; I_C = concentration of inhibitor, K_{ads}= adsorption equilibrium constant, (γ) = surface coverage

RESULTS AND DISCUSSIONS

Phytochemical properties of *Vigna subterranea* leaf

The phytochemical characteristics of the Vigna subterranea leaf extract were investigated to determine its potential as an inhibitor. Table 1 presents the qualitative, quantitative, and phytochemical compounds identified in the extract. The extract contained flavonoids, phenols, saponins, alkaloids, tannins, steroids, and terpenoids. The detection of flavonoids signifies the presence of active polyphenolic substances in the plant, which play crucial roles in biological and environmental processes, particularly in oil well environments (Shen et al., 2022). The identification of phenolic compounds suggests the occurrence of secondary metabolic activity in the plant leaf, as well as its oxidative capabilities (Cosme et al., 2020). The presence of saponins indicates the extract's absorption capacity and the production of surface glycosides by the plant (Liwa et al., 2017). The alkaloids found in the extract point to the existence of complex organic molecules characterized by heterolytic nitrogen rings (Murphy, 2016). Conversely, tannins contribute to regulating plant growth and serve as a protective agent against corrosion rates (Das et al., 2020). Their presence in the plant suggests that the control process unit can be safeguarded from corrosion. Additionally, the presence of steroids and terpenoids confirms the existence of metabolites in plants, indicating that VSLE can effectively function as a green inhibitor on the metal medium's surface.

Table 1: Results of phytochemical, qualitative and quantitative analysis VSLE.

Phytochemicals	Qualitative	Quantitative (%)
Flavonoids	+	1.410
Phenols	+	8.250
Saponins	+	3.500
Alkaloids	+	0.120
Tanins	+	0.984
Terpenoids	+	0.110
Steroids	–	–

+ indicates presence, - indicates absence

3.2 Elemental compositions of cleaned CSTP by AMSA

Table 2 displayed the elemental compositions analysis of the steel. Observations indicated that Iron (Fe) was found to be chief dominant element in the corroded steel; other elements were also found to be present in the CSTP. This indicated that the sample used in this study was truly a corroded sample and suitable for this study.

Table 2: Elemental compositions of CSTP by AMSA.

Elemental compositions		Percentage compositions
Name	**Symbol**	
Iron	Fe	97.26
Chromium	Cr	0.84
Manganese	Mn	0.59
Carbon	C	0.37
Copper	Cu	0.25
Silicon	Si	0.14
Others		0.55
Total		**100**

Gravimetric assessment of weight loss

Table 3 displayed the assessment of weight lost, EOI, and ROC in relation with the temperature, inhibition concentration, and time. It was observed that as the concentration increases, the weight loss and the ROC decreases, and the EOI increases in the presence of varied inhibition concentrations, and vice-versa.These observation showed that VSLE in the acid medium (HCl) proved to be a good green inhibitor for corrosion control of steel in an oil well industry.

Table 3: Gravimetric weight loss estimate.

Time (h)	Temperature (K)	Inhibition concentration (g/L)	Weight Loss (g)	ROC (mm/yr)	EOI (%)
6	313	0.5	0.042	5.04	66.40
		1.0	0.039	4.68	68.80
		1.5	0.035	4.20	72.00
	333	0.5	0.120	19.39	73.64
		1.0	0.102	17.18	76.64
		1.5	0.095	15.44	79.01
	353	0.5	1.046	125.51	46.05
		1.0	0.808	96.95	58.33
		1.5	0.632	75.83	67.35

3.4 Study of kinetic and thermodynamic parameters

To estimate the parameters, plots of log ROC against the reciprocal of temperature (1/T) were plotted at various concentrations (Fig. 2a & 2b), the values of E_a and the Arrhenius pre-exponential factor (log A) were obtained via the slopes and intercepts of the plots. The enthalpy (ΔH_a^o) and entropy (ΔS_a^o) of activation of the corrosion inhibition of the steel at different concentration was obtained from the plots and the results are presented in Table 4. The lines obey Arrhenius theory which shows that consistence in the corrosion process of steel with inhibitor. The correlation coefficient (R_1^2) of the regression plot revealed high mutual interactions between ROC and temperature. Furthermore, the estimated values of E_a increase linearly from 0.5 to 1.5 g/L of VSLE concentration, this can be likening to a slower corrosion reaction rate and reduction in EOI with an increase in temperature.

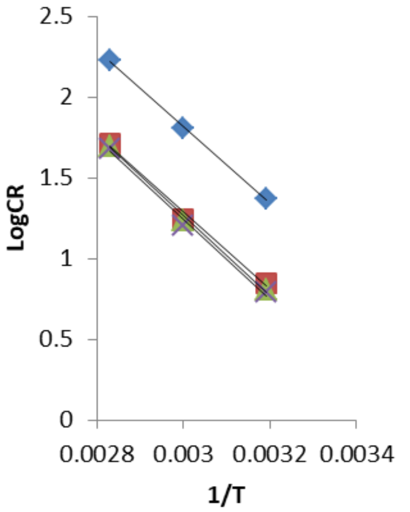

Fig. 2a: Plots of log ROC against 1/T

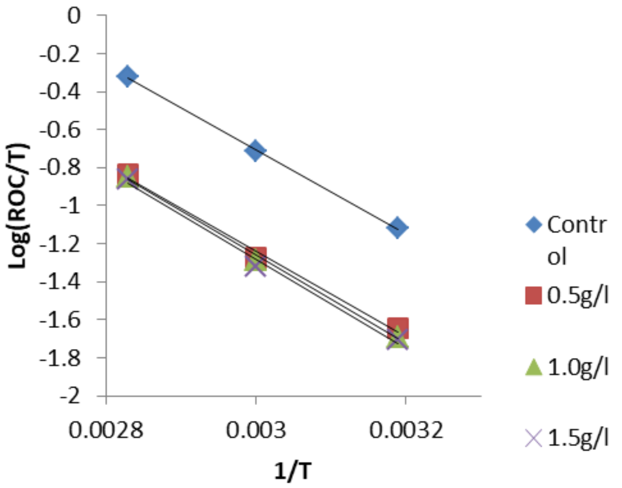

Fig. 2b: Plots of log ROC against 1/T

Table 4: Corrosion-based thermodynamic parameters.

Conc. (g/L)	**E_a (KJ/mol)**	**log A**	**ΔH_a^o** (KJ/mol)	**ΔS_a^o** (KJ/mol. K)	R^2
0.00	45.23	8.91	45.45	-35.50	0.999
0.50	46.11	8.54	46.64	-26.25	1.000
1.00	46.09	8.51	46.67	-27.84	0.999
1.50	47.52	8.71	47.26	-33.58	0.999

The values of ΔH_a^o possesses +ve values that are greater than the uninhibited state, this shows that the steel suspension in hydrochloric acid solution exhibited endothermic reaction processes. Furthermore, the values of the entropy of reaction (ΔS_a^o) in the inhibited cases were found -ve as the VSLE concentration increases, this can be attributed to a model controlled and tolerant array of VSLE inhibitor molecules on the surface of steel. A phenomenon that indicated the profile activated complex of the reaction to be association step (Singh *et al*., 2016).

3.5 Isotherms absorption analysis

The plots representing the Langmuir isotherms was plotted and presented in Fig. 3. The R^2 of 100%, 99.90%, and 99.90%, respectively, observed from Langmuir isotherm fitted well to the prediction, and this can be said to describes the interaction of the adsorbate molecules and the surface of the adsorbent (Table 5). Moreover, the K_{ads} values, which account for the ratio of the amount of adsorbate molecules absorbed by the surface of the adsorbent was high. The higher value represents the strength of the bonds that holds the VSLE and the steel surfaces. The -ve value (Table 6) of the ΔG_{ads} signified the adsorption of VSLE molecules was spontaneous and stable on steel surfaces (Hazzan *et al*., 2016).

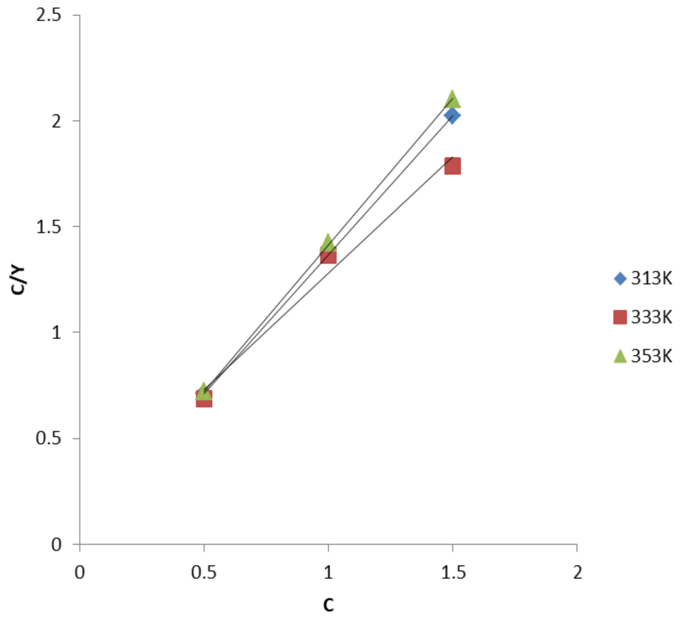

Fig. 3: Langmuir isotherm plot.

Table 5: Langmuir parameters for the adsorption of VSLE on the surface of steel in HCl.

Temp (K)	**K (l/g)**	**ΔG (KJ/mol)**	**R^2**
313	18.52	-25.57	1.00
333	21.74	-27.65	0.9990
353	30.30	-30.28	0.9990

Table 6: Thermodynamic parameters on corrosion inhibition of steel in HCl using VSLE at different inhibitor concentration.

Conc. (g/l)	**Ea (KJ/mol)**	**Pre-exponential factor: log(A)**	**R^2**	**Q_{ads} (KJ/mol)**
Blank	45.23	8.91	0.999	-
0.5	45.84	8.47	0.994	-0.95
1.0	47.41	8.69	0.997	-3.17
1.5	47.52	8.69	0.994	-3.28

CONCLUSIONS

The study adopted the organic green biomass of VSLE inhibitor for corrosion study of steel in hydrochloric acid medium. Low COR and high EOI were obtained. The extract possesses phytochemical properties responsible for effective VSLE to act as inhibitor. The absorption study shows chemisorptions owing to negative Gibb's free energy. The adsorption isotherm model that best described the corrosion inhibition mechanism of was Langmuir isotherm. The study demonstrated that VSLE proved to be a green biomass extract for corrosion control of steel in an oil well environment.

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