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Ethanol Extract of *Vernonia amygdalina* Leaf as a Green Corrosion Inhibitor for Carbon Steel in Solution of HCl

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Abstract: *To investigate the potential of ethanol extract from the bitter leaf as a green corrosion inhibitor for the corrosion of carbon steel, gravimetric, FTIR and scanning electron microscopy analytical techniques were adopted for monitoring the corrosion. Results obtained from gravimetric analysis indicated that the inhibition efficiency of the extract was 98.86, 94.33, 94.81, 95.28 and 96.22 % for 0.1, 0.2, 0.3, 0.4 and 0.5 g/L of the extract. The corrosion and the corrosion inhibition processes were found to follow pseudo-first-order kinetics and at various concentrations, the inhibitor demonstrated the potential to extend the half-life of the metal in the acid solution. The adsorption of the inhibitor suited the Langmuir and Frumkin adsorption models. The inference drawn from the isotherms revealed that there exists an interaction between the inhibitor's molecules and that the lateral interaction parameter pointed to the attractive behaviour of the inhibitor. The adsorption was spontaneous and agreed with the mechanism of physical adsorption. The scanning electron micrograph of the metal surface after inhibition revealed the formation of a protective layer on the metal surface. Some functional groups that were native to the leaf extract were found to be missing in the FTIR spectrum of corrosion product which suggested that they were used for adsorption while some were shifted, an indication that there was interaction between the adsorbed species.*

Keywords: *Metal degradation, electrochemical attack, retardation, bitter leaf extract*

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1.0 Introduction

Corrosion attacks metals and metallic alloys more than other materials. Metals are valuable materials that form the bulk components of most industrial installations. The involvement of metals in fertilizers, oil, metallurgical and other industries can not be economically viable if adequate provisions for the protection of these metals against corrosion are not made. Corrosion of metals is an electrochemical process that tends to return the metal to its natural state. It requires an aggressive medium to create an electrochemical cell (Abod, *et al*., 2019).

Given its adverse environmental impact, several measures have been adopted and applied to protect metals against corrosion including oiling/greasing, galvanization, cathodic/anodic protecting, electroplating, etc (Saxena *et al*., 2018). The application of corrosion inhibitors is an essential process that is aimed at reducing the rate of corrosion of metals by adding a substance (called an inhibitor) into the corrosion environment (Eddy *et al*., 2022). The option of using corrosion inhibitors is preferred against some other methods because it is economical, can easily be implemented on eco-friendly bases and requires less technology (Eddy *et al*., 2023). The first set of corrosion inhibitors were chromates and some metals. These inhibitors were very efficient but were discontinued because of their toxicity and consequence impact on the environment. Consequently, the current global challenge in the field of corrosion science is searching for corrosion inhibitors that are cost-effective, eco-friendly, bridgeable and natural (Muthukrishnan *et al*., 2019). These properties are satisfied by a class of compounds called green corrosion inhibitors (Lukovits *et al*., 2001; Maij *et* al., 2019; Matos *et al*., 2019). Success in green corrosion inhibition or at least near green corrosion inhibition has been recorded through the use of extracts of plants and animals such as exudate gum and other natural polymers, carbohydrates, and some drugs (Ameh and Eddy, 2014a-b; Eddy *et al*., 2014; Momoh-Yahaya *et al*., 2014)

Eddy *et al*. (2009b) investigated the inhibitive role of *Gnetum africana* against the corrosion of mild steel in a solution of H_2SO_4 and reported excellent inhibition efficiency that was observed to increase with an increase in the extract concentration but decreased with a temperature rise and with increasing period of contact. The inhibition was attributed to the presence of phytochemicals such as alkaloid, saponin, tannin, terpene, anthraquinone and cardiac glycoside. Eddy *et al*. (2009b) also investigated the inhibitive and adsorption properties of ethanol extract of *Terminalia catappa* for the corrosion of mild steel in H_2SO_4 using weight loss, hydrogen evolution, and infrared methods. They obtained an average inhibition efficiency approaching 90% and also attributed the phytochemicals in the plants as the major cause of the inhibition. Some research groups have also reported good inhibition efficiency for *Heinsia crinata* leaf (Eddy and Odiongenyi, 2010), leaf extract of *Hibiscus sabdariffa calyx* (Eddy *et al*. (2011). *Piper guinensis* (Ebenso *et al.*, 2010), *Vernonia amygdalina* (Odiongenyi *et al*., 2009), *Lasianthera Africana* (Eddy *et al*., 2009c), *Aloe vera* (Eddy and Odoemelam, 2009), *Azadirachta indica* (Eddy and Mamza, 2009), *Phyllanthus amarus* (Eddy, 2009; Eddy and Awe, 2018), *Colocasia esculenta* (Eddy, 2009), *Gongronema latifolium* (Eddy and Ebenso, 2010), *Gnetum Africana* (Eddy *et al*., 2009d), *Anogessus leocarpus (*Ameh *et al*., 2012), *Terminalia atappa* (Eddy *et al*., 2009b), *Occimium gratissimum* (Eddy *et al*., 2010a), *Solanum melongena (*Eddy *et al*., 2010b), *Andrographis paniculate (*Uwah *et al*., 2013*)*, *Gloriosa superba* (Eddy *et al*., 2014), *Saraca ashoka extract* (Saxena *et al*., 2018) and *Glycyrrhiza glabra leaves extract* (Alibakhshi *et al*., 2018). All the listed applications of plant leaves were commendable in producing excellent results concerning the inhibition of corrosion.

2.0 Materials and Methods

2.1 Sample and sample preparation

The bitter leaf samples were purchased from Ikot Ekpene main market and transported to the laboratory. They were thoroughly washed and allowed to dry to constant weight. The dried leaf samples were grounded and soaked in ethanol for 48 hours. The extracts of the plant were recovered from the ethanol using a cold extractor. Stock solution of the extract was prepared in 3 M of HCl and through serial dilutions, 0.1- 0.5 g/L were obtained.

The carbon steel sheet was obtained from a dealer in Nsukka, Enugu state. Nigeria. Each sheet was used to produce metallic coupons measuring 5 × 4 × 1.0 cm. The coupons were washed in distilled water containing 20% NaOH and 200 g/L of zinc dust and then with ethanol before rinsing with acetone.

2.2 Gravimetric experiment

The weight loss measurement was implemented according to the standard weight loss measurement protocol as reported elsewhere (Ferigita *et al*., 2023). A known weight of each coupon was completely immersed in different beakers containing 150 ml of the respective test solutions and allowed to stand for 168 hours in a thermostated water bath (whose temperature was fixed). After every 24 hours, each coupon was retrieved from the solution. They were thoroughly washed to remove corrosion products from the surfaces. The washed coupons were rinsed with acetone, allowed to dry and re-weighed. The difference in weight was recorded as weight loss. The experiments were repeated for different test solutions containing different concentrations of the extract and at 303 K. Weight loss was initiated as the parameter for calculating inhibition efficiency, corrosion rate and surface coverage according to equations 2.1 respectively,

$$\%I = \left(1 - \frac{W_2}{W_1}\right) \times \frac{100}{1} \quad (1)$$

$$CR\ (gm^{-1}h^{-1}) = \frac{(W_1 - W_2)}{At} \quad (2)$$

$$\theta = \left(1 - \frac{W_2}{W_1}\right) \quad (3)$$

W_1 and W_2 represent the weight of the metal before and after immersion respectively, D is the density of the metal, t is the period of contact and A is the surface area of the metal.

3,0 Results and Methods

3. 1 Weight loss

Fig. 1 shows the variation of weight loss with time for the corrosion of carbon steel in 3 M HCl in the absence and presence of various concentrations of ethanol extract of bitter leaf (weight loss for the blank is inserted into the plot). The graph reveals that the weight loss of carbon steel increased with the increase in the period of contact but decreased with an increase in the concentration of the extract. This indicates that various concentrations of ethanol extract from bitter leaves inhibited the corrosion of carbon steel and that the inhibition efficiency increases with an increase in concentration (Eddy *et al*., 2009). The observed increase in inhibition efficiency with concentration suggests that the inhibitor is an adsorption inhibitor. The decrease in inhibition efficiency with the period of contact is an indication that the rate of corrosion of carbon steel in 3 M HCl increases with an increase in the period of contact (Eddy, 2011).
Corrosion rates for the blank and in the presence of various concentrations of the inhibitor were calculated in the unit of gm/day and the calculated values are recorded in Table 1.

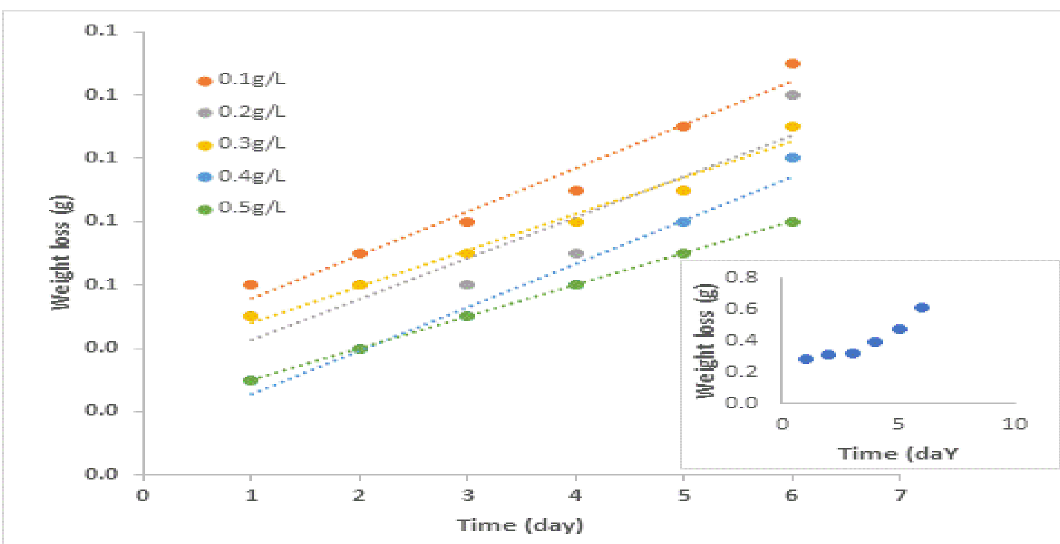

Fig. 1: Variation of the corrosion rate with time for the corrosion of carbon steel in the absence and presence of various concentrations of ethanol extract of bitter leaf

The information recorded in Table 1 was used to develop the plots in Fig. 2 and it is evident from the plots that the corrosion rate is proportional to weight loss since the graph followed the same pattern (see Fig. 1). Values of the corrosion rate were used to calculate the inhibition efficiency of the ethanol extract of bitter leaf and the results obtained are shown in Table 2. The results clearly show that the inhibition efficiency increases with an increase in the concentration of the extract and ranges from 93.86 to 96.28 %. A similar trend was followed by the degree of surface coverage (Table 2) which implies that the surface coverage increases with increasing concentration and may be due to an increase in the number of inhibitor molecules that diffuse to the metal surfaces.

The inhibition efficiencies recorded in Table 2 are average inhibition efficiency which were obtained after six days of immersion. Instantaneous inhibition calculated after every 24 hours of immersion is shown in Table 3 and depicted graphically in Fig. 3. From the figure, it can be seen that the inhibition efficiency first decreased before it increased with time. Therefore, there is a period required for the passivation or development of the protective film on the surface of the metal (Daoud *et al* ., 2023).

Table 1: Corrosion rate ($gm^{-2}h^{-1}$) of carbon steel in 3 M HCl in the absence and presence of various concentrations of ethanol extract of bitter leaf.

Day	Blank	0.1g/L	0.2g/L	0.3g/L	0.4g/L	0.5g/L
1	5.8333	1.2500	1.0417	1.0417	0.6250	0.6250
2	6.4583	1.4583	1.2500	1.2500	0.8333	0.8333
3	6.6667	1.6667	1.2500	1.4583	1.0417	1.0417
4	8.1250	1.8750	1.4583	1.6667	1.2500	1.2500
5	9.7917	2.2917	1.8750	1.8750	1.6667	1.4583
6	12.7083	2.7083	2.5000	2.2917	2.0833	1.6667

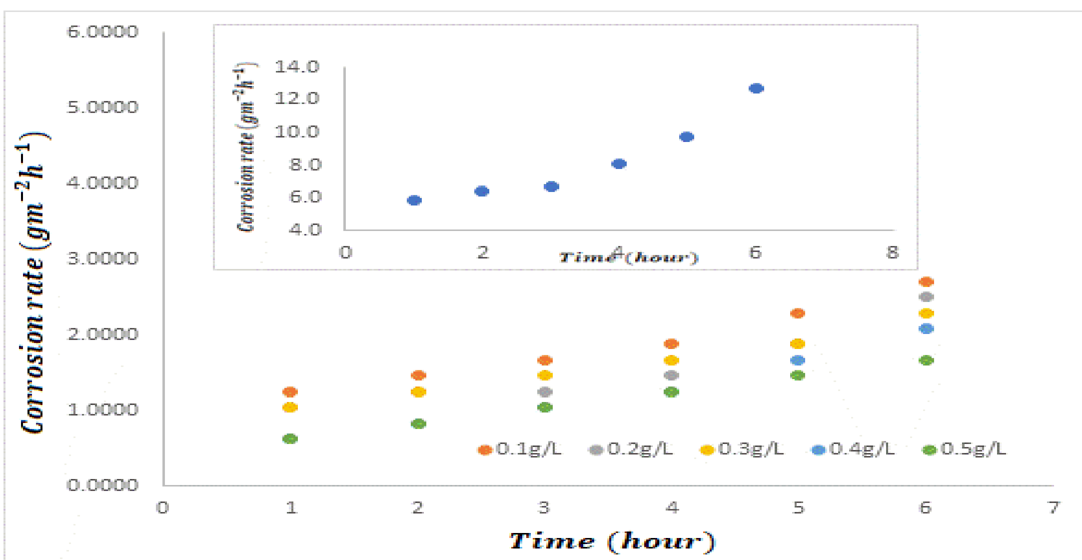

Fig. 2: Variation of the corrosion rate with time for the corrosion of carbon steel in 3 M HCl in the absence and presence of various concentration of ethanol extract of bitter leaf

Table 4: Inhibition efficiencies of various concentrations of ethanol extract of bitter leaf for carbon steel in solution of HCl

C (g/L)	Inhibition efficiency (%IE)	Degree of surface coverage (θ)
0.1	93.86	0.9386
0.2	94.33	0.9433
0.3	94.81	0.9481
0.4	95.28	0.9528
0.5	96.22	0.9622

3.2 Kinetic study

Most corrosion reactions have been reported to be pseudo-first order, which implies that equation 1 can be used to explain the kinetic of corrosion and corrosion inhibition (Hosny *et al*., 2024)

$$-\log(weight\ loss) = k_1 t + constant \quad (4)$$

Based on equation 1, a plot of -log(weight loss) versus time was found to be linear as shown in Fig. 4. The plots have an excellent degree of linearity (R^2 ranged from 0.9238 to 0.9967). Calculated rate constants are recorded in Table 4. The rate constant (k_1) is related to the half-life according to equation 5.

$$t_{1/2} = \frac{0.693}{k_1} \quad (5)$$

The half-lives of the metal in the presence of various concentrations of the inhibitor were higher compared to the half-life of the metal in the blank solution (3 M HCl) which was 9 days. Therefore, ethanol extract of bitter leaf extended the half-life of carbon steel in 3 M HCl.

3.3 Adsorption isotherm

Adsorption isotherm is useful in explaining the adsorption characteristics of a corrosion inhibitor, the mechanism of inhibition and other processes occurring in the corrosion inhibition process (Betti *et* al., 2023; Eddy and Odoemelam, 2008).

Experimental data were tested for the fitness of various adsorption isotherms using values of surface coverage at various concentrations of the inhibitor. The test revealed that the adsorption of the inhibitor obeyed the Langmuir and the Frumkin adsorption isotherms. The assumptions establishing the Langmuir adsorption isotherm can be expressed according to equation 6 (Ahmadi & Khormali, *et al*., 2024),

$$ln\left(\frac{C}{\theta}\right) = lnC - lnk_{ad} \quad (6)$$

where C is the concentration of the inhibitor in the bulk solution, θ is the degree of surface coverage and k_{ads} is the Langmuir equilibrium constant of adsorption which is related to the standard free energy of adsorption as follows (Eddy and Ebenso, 2009):

$$\Delta G^0_{ads} = -RTln(55.5k_{ads}) \quad (7)$$

where R is the gas constant, T is the temperature and 55.5 is the molar heat of the adsorption of water. The Langmuir isotherm for the adsorption of ethanol extract of bitter leaf on the surface of carbon steel is shown in Fig. 5 while parameters calculated from the plot are shown in Table 5. This included the equilibrium constant of adsorption, the free energy change, values of R^2, slope and intercept of the plot. The fitness of the Langmuir isotherm is affirmed by a perfect degree of fitness ($R^2 = 1$). However, the slope value is slightly less than unity, which is a deviation from the ideal Langmuir isotherm. According to Eddy and Ita (2011), the ideal Langmuir isotherm should have a slope value of unity and that slope value greater or less than unity indicates that there is interaction between the adsorbed species. Therefore, there is some level of interaction between the adsorbed species. Also, the calculated value of the standard free energy of adsorption was -10.03

kJ/mol which is negatively less than the threshold value (- 40 kJ/mol) required for the mechanism of chemical adsorption (Lin *et al*., 2024)

Table 3: Instantaneous inhibition efficiency of ethanol extract of bitter leaf for carbon steel corrosion

Time (hours)	0.1g/L	0.2g/L	0.3g/L	0.4g/L	0.5g/L
24	78.57	82.14	82.14	89.29	89.29
48	77.42	80.65	80.65	87.10	87.10
72	75.00	81.25	78.13	84.38	84.38
96	76.92	82.05	79.49	84.62	84.62
120	76.60	80.85	80.85	82.98	85.11
144	78.72	80.36	82.00	83.64	86.92

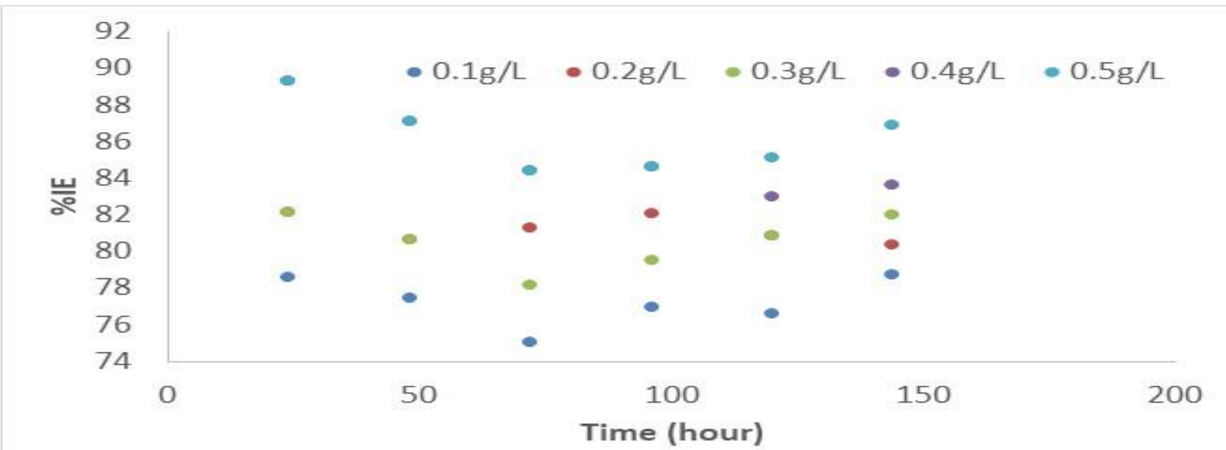

Fig. 3: Variation of instantaneous inhibition efficiency of ethanol extract of bitter leaf with time

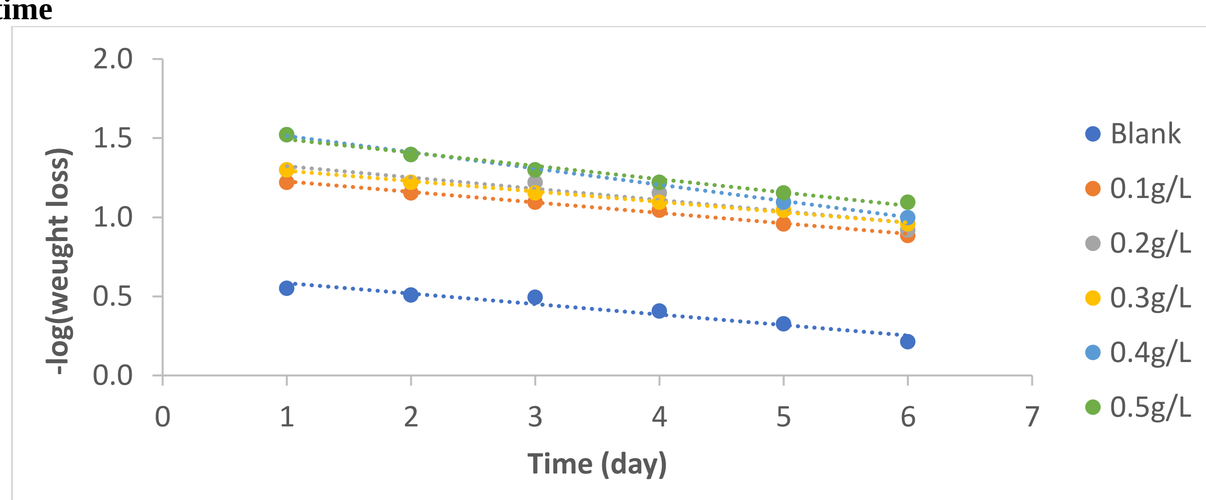

Fig. 4: Variation of -log(weight loss) with time for the corrosion of carbon steel in 3 M HCl containing various concentrations of ethanol extract of bitter leaf.

Table 4: Kinetic data for the corrosion of carbon steel in 3 M HCl containing various concentrations of ethanol extract of bitter leaf at 303 K

C (g/L)	Half-life (day)
Blank	9
0.1	10
0.2	11
0.3	11
0.4	13
0.5	13

Therefore, the adsorption of ethanol extract from bitter leaf occurred through the mechanism of physical adsorption (Eddy *et* al., 2010c). Generally, when the calculated free energy is negatively less than – 20 kJ/mol, the mechanism of physical adsorption is sustained but free energy values between -20 to -40 kJ/mol point toward both mechanisms while those above -40 kJ/mol are consistent with the mechanism of chemical adsorption.

The model of the Frumkin adsorption isotherm is expressed by equation 8 (Abd El Rehim *et al*., 2016; Marzorati, *et al*., 2019; Ukpe, 20191-b),

$$\boldsymbol{ln}\left[\boldsymbol{C}.\left(\frac{\boldsymbol{\theta}}{\mathbf{1}-\boldsymbol{\theta}}\right)\right] = lnk_{ads} + 2\alpha\boldsymbol{\theta} \quad (8)$$

α is the lateral interaction parameter and describes the interaction in the adsorbed layer. Fig. 4.6 shows the Frumkin isotherm for the adsorption of ethanol extract of bitter leaf on the surface of the carbon steel. Fig. 6 shows a linear plot (R^2 = 0.9322) revealing the fitness of the Frumkin isotherm to the adsorption of ethanol extract of bitter leaf on the surface of the carbon steel. The lateral interaction parameter (18.89) was positive which indicated that there was an attraction between the corrosion inhibitor's molecules (Momoh-Yahaya *et al*., 2015)

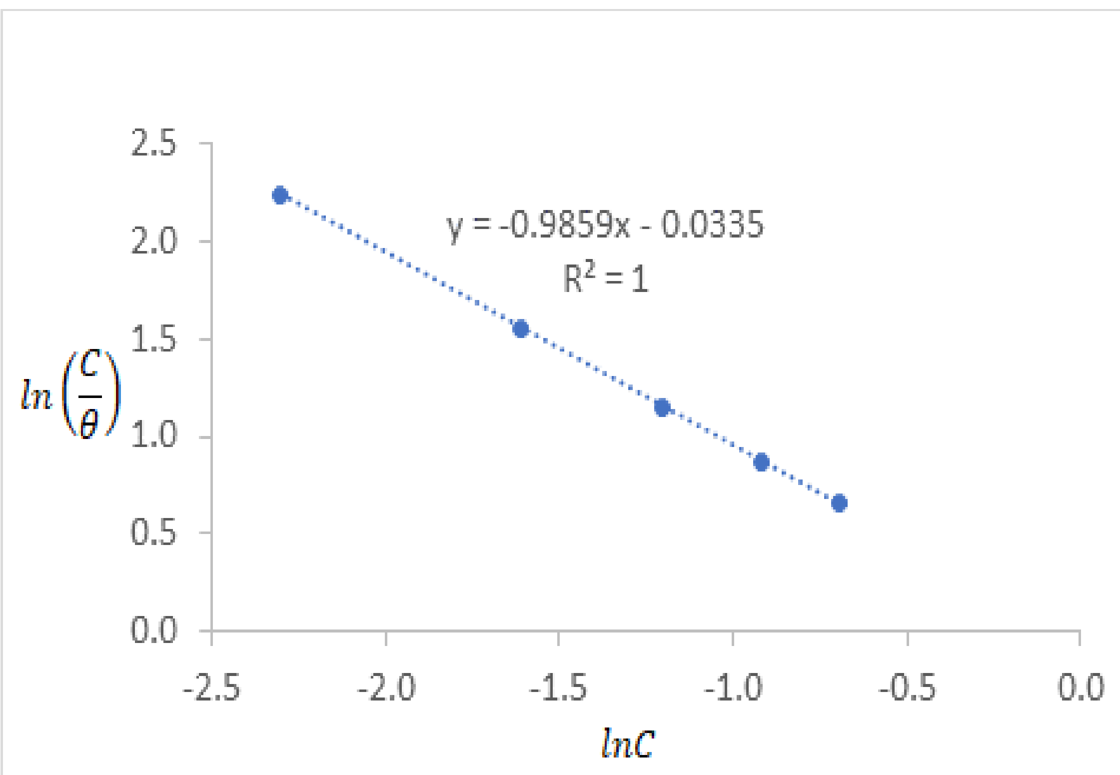

Fig. 5: Langmuir isotherm for the adsorption of ethanol extract of bitter leaf on the surface of carbon steel

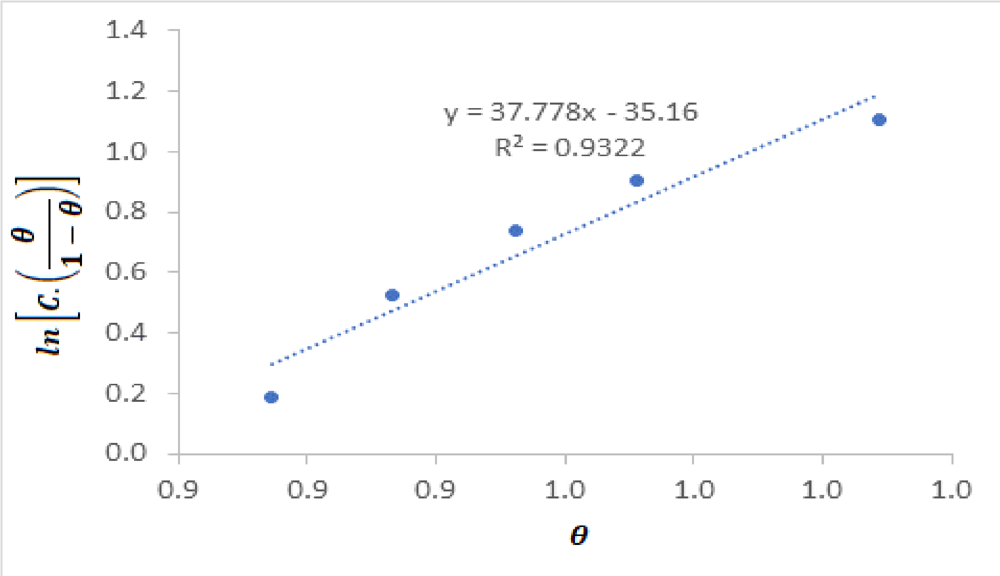

Fig. 6: Frumkin isotherm for the adsorption of ethanol extract of bitter leaf on the surface of carbon steel

4.0 Conclusion

The present study led to the findings that supports the following conclusions

(i) Ethanol extract of bitter leaf is a good adsorption inhibitor for the corrosion of carbon steel in a solution of HCl.

(ii) The inhibition efficiency of the extract increases with an increase in the extract concentration but decreases with increasing periods of contact

(iii) The average inhibition efficiency mechanism of the extract supports physical adsorption

(iv) The adsorption of the extract on the metal surface is spontaneous and is best described by the Langmuir and Frumkin adsorption isotherms

(v) The corrosion of carbon steel in 3 M HCl in the absence and presence of various concentrations of ethanol extract of bitter leaf followed a pseudo-first-order kinetics

(vi) The extract has the potential to extend the half-life of carbon steel in a solution of HCl.

Given the above conclusions, the a need for continuous research on the extract of bitter leaf for the inhibition of the corrosion of different metals in different environments.

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Compliance with Ethical Standards Declarations

The authors declare that they have no conflict of interest.

Data availability

All data used in this study will be readily available to the public.

Consent for publication

Not Applicable

Availability of data and materials

The publisher has the right to make the data public.

Competing interests

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Authors' contribution

The work was jointly designed by CA E and GCU and both authors participated in experimental, and reporting.