

Inhibition of Mild Steel Corrosion in HCl Solution by Gongronemalatifolium Methanol Extract

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Abstract: Corrosion inhibition effect of methanol extract of *Gongronemalatifolium* on mild steel in HCl solution was studied using gasometric methods at 303K, 313K and 323K. The inhibition efficiency was found to increase as the concentration of extracts increased from 0.1%w/v to 0.5%w/v and decreased with increase in temperature. The maximum efficiency of inhibition was found to be 77.17% at 303K. Values of activation energy for the inhibited system were greater than the values obtained for the uninhibited system. Physical adsorption mechanism was proposed for the adsorption of the inhibitor from the trend of the inhibition efficiency with temperature and the values of E_a , Q_{ads} and ΔG_{ads} obtained. The adsorption of *Gongronemalatifolium* extract on the surface of the mild steel followed Langmuir, Freundlich and El-Awardy adsorption isotherms.

Key Words: Corrosion, inhibition, mild steel, *Gongronemalatifolium*

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I. Introduction

Corrosion is one of the major problems in several technical installations involving metals and alloys hence; prevention mechanism for corrosion of metals is of paramount importance, to increase their lifespan especially those in aggressive environments. Corrosion is the deterioration of metals in chemicals or reaction with its environment (Rani and Basu, 2012). Corrosion of mild steel is a major problem in some industries including the oil and fertilizer industries (Bentisset al., 2007). This is because in these industries, there are some processes (such as scaling, etching, etc.) that necessitate contact between mild steel and aggressive medium such as acid, alkaline and salt solutions (Ashassi-Sorkhabiet al., 2005). In view of the problems created by mild steel corrosion, several researches on the methods of inhibition for its corrosion have been reported and it has been established that the use of inhibitors is one of the best methods of prevention of the corrosion of mild steel in acidic medium (Odiogenyiet al., 2009; Gopiet al., 2009). However, because of the sequel to toxic nature, high cost and increasing awareness and strict environmental regulations of some inorganic inhibitor compounds, the use of natural product of plant origin as corrosion inhibitor is receiving attention. The search for an efficient inhibitor for metal corrosion in different aggressive media has, in recent times, taken a new dimension owing to the clarion call for green chemistry. Recently, corrosion control using plant extract has become an interesting research because it can be easily extracted, environmentally friendly, easily affordable, and renewable. These plants extract contained O, N and S which are the active centre for adsorption process on the metal surface (Peteret al., 2009; Umoren et al., 2007; 2008). The mechanism of this process is that O, N, and S, have lone pair and π electron which facilitate electron transfer from inhibitor to the metal surface thereby forming a compact barrier reducing corrosive attack. Extracts from different parts of plant have been widely reported as effective and good metal corrosion inhibitors in various corrosive environments (Oguzie, 2008; Ebensoet al., 2009; Ebensoet al., 2004; Abiola et al., 2007; Chauhan and Gunasekaran, 2007; Kumar et al., 2010; Chauhan et al., 2010; Lebriniet al., 2010; Dahmaniet al., 2010; Sharmila et al., 2010; Deng and Li, 2012; Iloamae et al., 2012; Iloamae et al., 2015; Eduoket al., 2012). *Gongronemalatifolium* commonly called utazi leaf is a non-toxic plant available in every part of Nigeria. As contribution to the current interest on environmentally friendly, non-toxic, green corrosion inhibitors, the present study investigates the inhibiting and adsorption effects of *Gongronemalatifolium* leaf extract on mild steel corrosion in HCl solution using gasometric method at 303K, 313K and 323K.

II. Experimental Methods

2.1 Preparation of *Gongronemalatifolium* leaf extract:

Samples of *Gongronemalatifolium* leaves were air dried at ambient temperature under a shade after which it was crushed into powdery form using an electric blender and stored in a closed container for analysis. These were Soxhlet-extracted using methanol. The solutions were further subjected to evaporation at 65°C in order to leave the sample free of the methanol. The crude extract was collected and stored in a reagent bottle for

analysis. The stock solution was diluted with appropriate quantity of 3.0M HCl to obtain inhibitor test solutions of 0.1–0.5 % w/v concentrations. This leaf extract was subjected to FTIR and phytochemical analysis.

2.2 Specimen preparation

The sheet of mild steel used for this study was obtained commercially. It was mechanically cut into coupons (4 x 3 x 0.12cm). A small hole was drilled at one end of the coupons for easy hooking. During the study the samples were polished using sand paper, degreased in absolute ethanol, dried using acetone and stored in desiccator for further use.

2.3 Test Solution Preparation

All the chemicals used for the study were of analytical grade. Solutions were prepared by using distilled water, 3.0M HCl was **prepared**.

2.4 Gasometric Method

Gasometric method was carried out at 303, 313 and 323K as described in the literature (Eddy et al., 2009; Ebensoet al., 2008; Umoren et al., 2008). From the volume of hydrogen gas evolved per minute, inhibition efficiencies (%I), surface coverage (θ), and corrosion rate were calculated using Equations (1), (2) and (3):

$$I.E\% = \frac{V_0 - V_i}{V_0} \times 100 \dots\dots\dots(1)$$

$$\theta = \frac{V_0 - V_i}{V_0} \dots\dots\dots(2)$$

Where V_0 and V_i are the volume of hydrogen evolved in uninhibited and inhibited solutions respectively.

Rate of hydrogen evolution was computed using equation 3 (Umoren et al., 2011).

$$CR_H = \frac{V_t - V_i}{T_t - T_i} \dots\dots\dots(3)$$

Where V_t and V_i are the volumes of hydrogen evolved at time T_t and T_i respectively, while CR_H is the rate of hydrogen evolution.

III. Results & Discussion

3.1. Phytochemical Analysis

Table 1: Phytochemicals of Gongronema latifolium.

Parameters	Presence
Alkaloids	+
Flavanoid	++
Reducing Sugars	-
Tannins	+
Phlobatannins	+
Proteins	+
Amino acid	+
Resins	++
Saponins	++
Glycosides	-
Cardiac glycosides	++
Carbohydrates	++
Terpenoid	++
Triterpenes	++
Steroids	++
Quinones	+++
Anthraquinones	-
Phenols	+
Anthracene	-

Key: + = trace amount; ++=moderate amount; +++= appreciable amount; - =complete absence

The phytochemical constituents of methanol extract of Gongronemalatifolium are shown in Table 1. The result revealed that the inhibition efficiency of the extract was due to the presence of the phytochemical constituents which retards corrosion (Eddy et al., 2010).

3.2 FTIR Analysis of Extract

The IR spectrum of Gongronema latifolium leaf extract is shown in fig 1. The different peaks and the corresponding functional groups are shown in table 2.

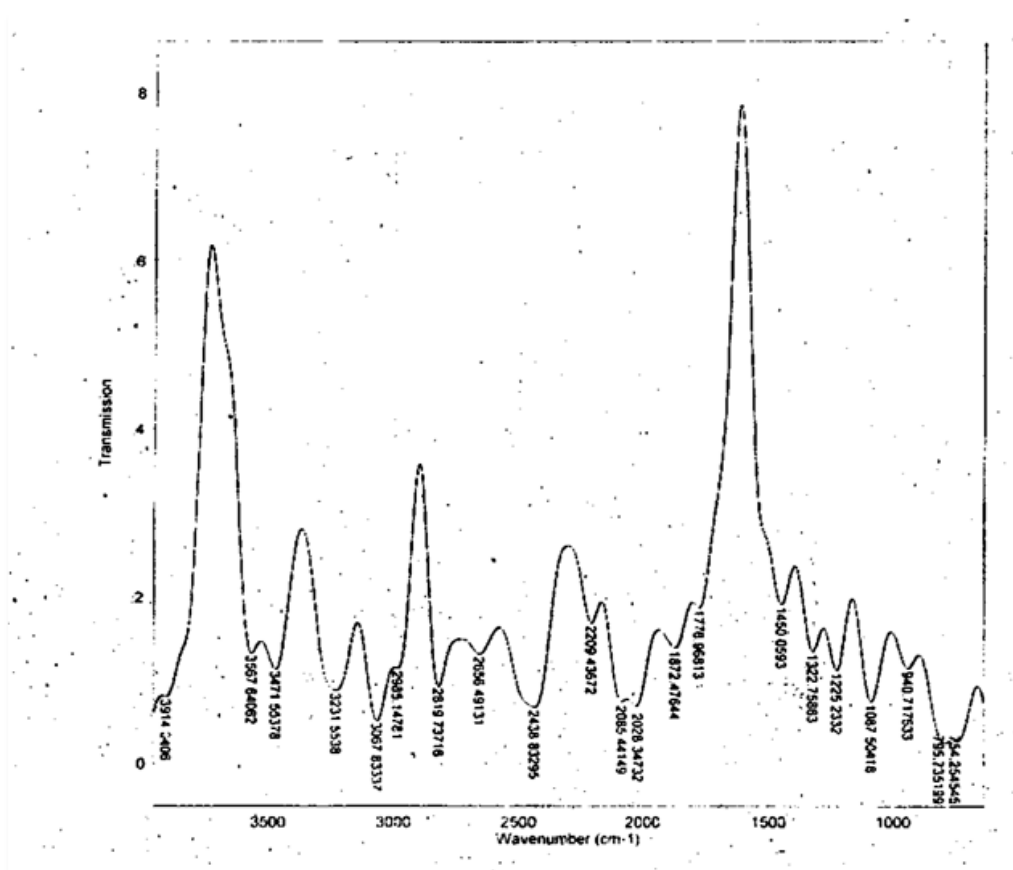


Fig.1: FTIR spectrum of methanolic extract of *Gongronemalatifolium*

Table 2: FTIR results for methanol extract of *Gongronema latifolium*

Wavelength of Peak (cm ⁻¹)	Assigned Functional Groups
754.254	C-Cl of aromatic chloro compound
940.717	C-H of alkene
1087.504	C-O of alcohol, ether
1225.233	C-O of carboxylic acid, ester, anhydride
1322.759	C-O of carboxylic acid
1450.059	C=C of aromatic groups
1776.968	C=O of anhydrides, ester, carbonyl
1872.476	R-N=C=S of isothiocyanates
2028.347- 2085.441	C-O of Carbonyl
2209.437	C≡C of alkynes
2438.833	C≡N of nitrile groups
2656.491	S-H of thiol
2819.737	C-H of alkyl groups
2985.148	C-H of vinylidene
3067.833	C-H of alkene groups
3231.554-	N-H of Primary amine
3471.554-3914.340	O-H of alcohols and phenolic groups

Figure 1 and Table 2 show the FTIR spectrum and the assigned functional groups for the methanol extract of *Gongronema latifolium* leaves. It was observed that the extract contains different functional groups. Organic compounds which have different heteroatoms such as N, O, S, P and aromatic rings in their structure are reported as effective inhibitors (Shukla and Quraishi, 2009). The results obtained showed the presence of these functional groups in the extract structures, which are responsible for the inhibition efficiency.

3.3 Gasometric experiment

Fig.1, 2 and 3 show the variation in volume of hydrogen evolved with time at 303, 313 and 323K with different concentrations of the Gongronematifoliumextract.It could be observed from the plots that volume of hydrogen evolved increased with time for all the temperatures with different concentrations of extract. It was also revealed that the volume of hydrogen gas evolved for the control sample was the highest at all temperatures studied.

It can be clearly observed from the plots that the volume of hydrogen gas evolved decreases as the concentration of extract increases because of the shielding effects of the extract on the surface of the mild steel (Umoren et al., 2006). This infers that the Gongronematifolium extract in the solution had a retarding effect on the corrosion of mild steel in HCl. Thus, the degree of inhibition can be said to be governed by the amount of extract present (James et al., 2007).

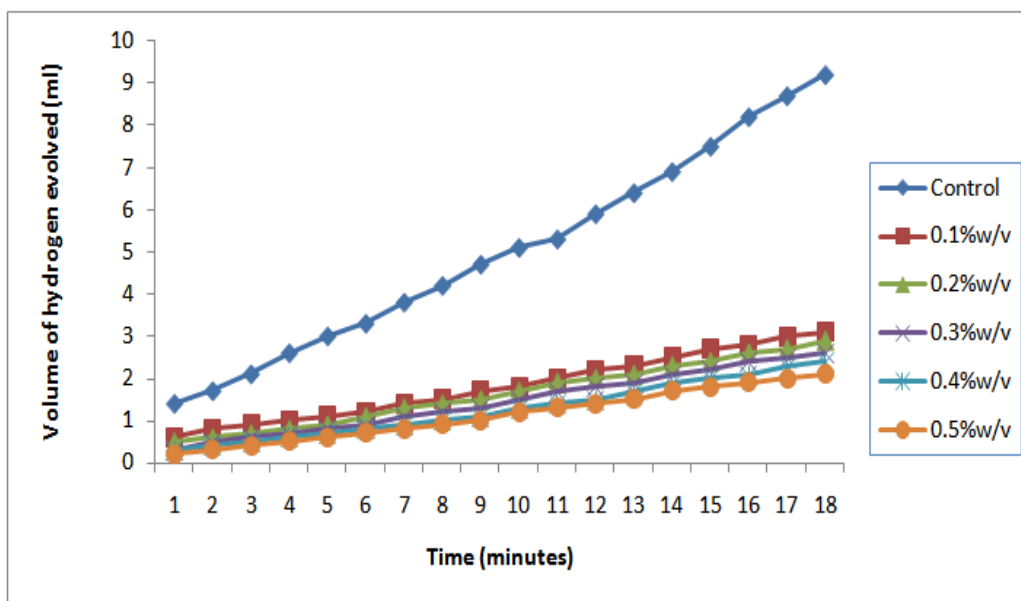


Fig.2: Volume of hydrogen evolved with time for corrosion of mild steel in 3.0M HCl in the presence and absence of extract of Gongronematifolium at 303K.

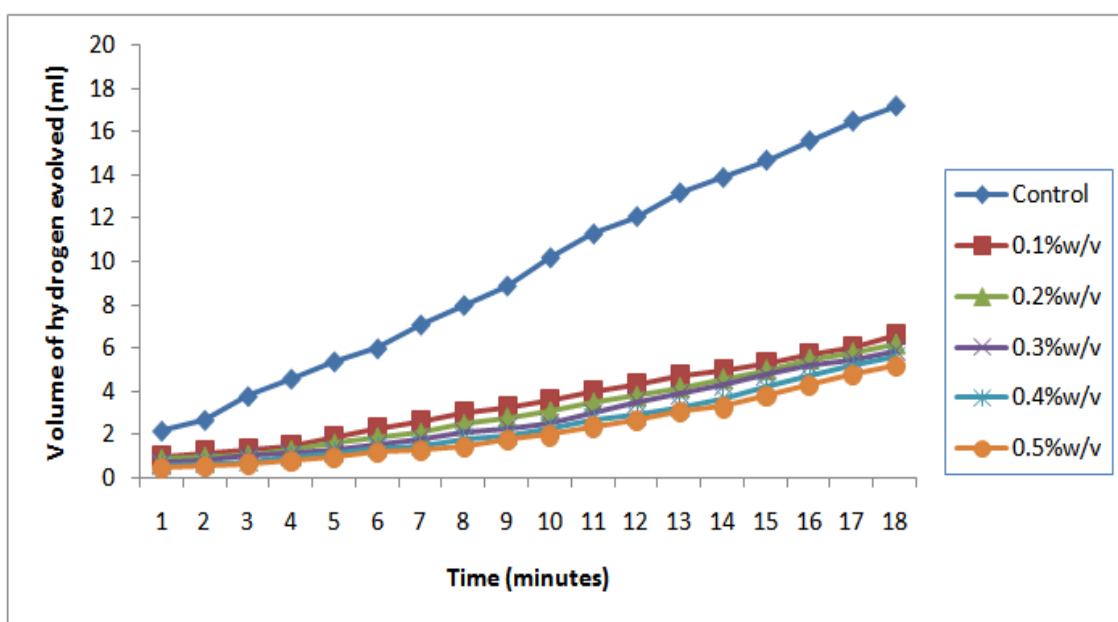


Fig. 3: Volume of hydrogen evolved with time for corrosion of mild steel in 3.0M HCl in the presence and absence of extract of Gongronematifolium at 313K.

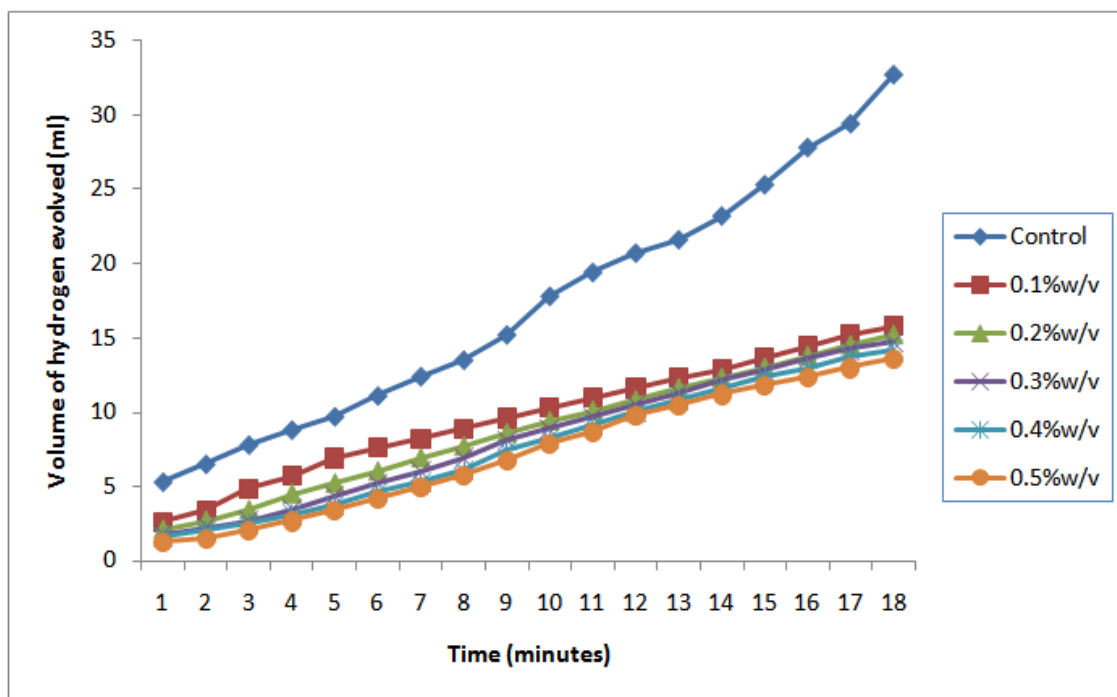


Fig. 4: Volume of hydrogen evolved with time for corrosion of mild steel in 3.0M HCl in the presence and absence of extract of Gongronemalatifolium at 323K.

Table 3: Inhibition efficiency (I%) of mild steel in different concentration of methanol extract of Gongronema latifolium leaf.

Concentration of GL inhibitor in (%w/v)	Inhibition efficiency (%)		
	303K	313K	323K
0.1	66.30	61.63	51.68
0.2	68.49	63.95	53.52
0.3	71.74	65.70	55.05
0.4	73.91	67.44	56.57
0.5	77.17	69.77	58.41

Table 4: Rate of hydrogen evolution.

Concentration of GL inhibitor in (%w/v)	Rate (cm ³ /mins)		
	303K	313K	323K
Blank	0.511	0.956	1.817
0.1	0.172	0.367	0.878
0.2	0.161	0.344	0.844
0.3	0.144	0.328	0.817
0.4	0.133	0.311	0.789
0.5	0.117	0.289	0.756

Table 3 showed values of inhibition efficiency of different concentrations of Gongronemalatifolium extract. It could be further illustrated using figure 5. From the results, it was seen that values of inhibition efficiency of Gongronema latifolium for the corrosion of mild steel increases as the concentration of the extract increases indicating that the extract acts as an adsorption inhibitor. The significant difference between the values of inhibition efficiency of Gongronema latifolium obtained at 303, 313 and 323K suggests that the mechanism of adsorption of the inhibitor on the mild steel surface was by physical adsorption. For a physical adsorption mechanism, inhibition efficiency of an inhibitor decreases with temperature while for a chemical adsorption mechanism, values of inhibition efficiency increase with temperature (Umoren et al., 2006; Ebenso et al., 2008).

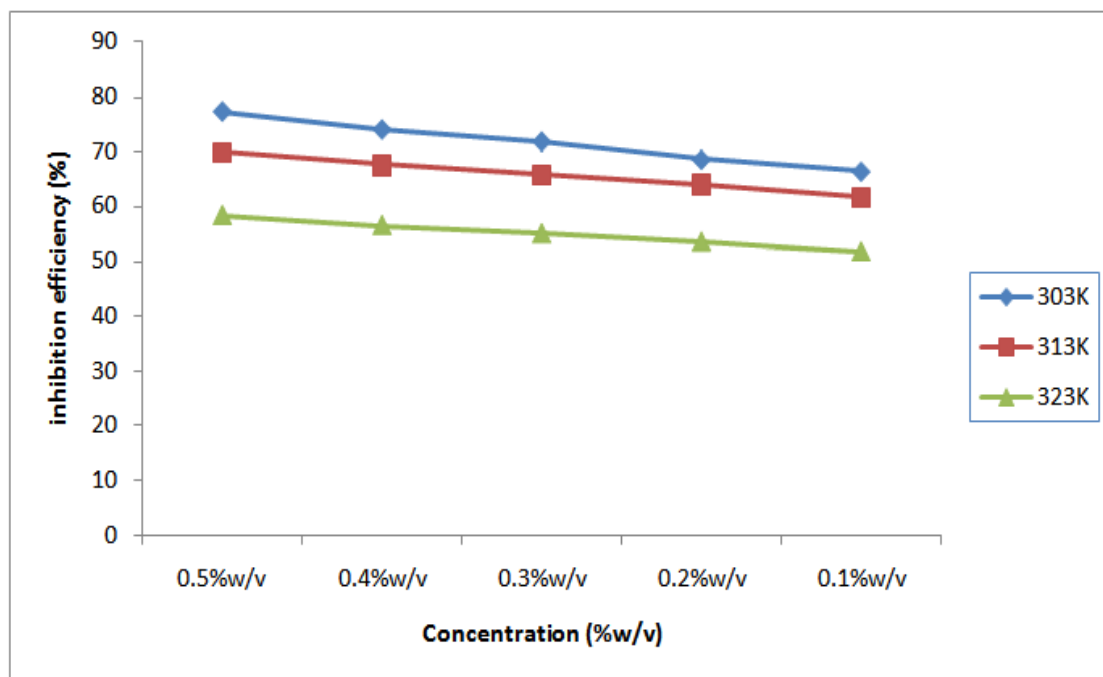


Fig.5: Inhibition efficiency (% I.E.) of Gongronemalatifolium obtained at different temperatures.

The values of corrosion rate for the corrosion of mild steel in the absence and presence of various concentrations of Gongronema latifolium are recorded in Table 4. From Table 4, it was observed that the corrosion rates for the corrosion reaction of mild steel in the presence of Gongronema latifolium were lower than values obtained for the blank indicating that various concentrations of Gongronema latifolium inhibits the corrosion of mild steel in HCl. The corrosion rate was also observed to decrease as the concentration of Gongronema latifolium increases indicating that the rate of corrosion of mild steel in HCl decreases as the concentration of Gongronema latifolium increases. The increase in corrosion rate with increasing temperature may be due to faster reaction kinetics at both the anodic and the cathodic sites (Eddy et al., 2010).

3.4 Mechanism of Inhibition using Thermodynamic Studies

Activation energy was evaluated using Arrhenius relationship (Abdallah, 2004).

$$\log \frac{CR_2}{CR_1} = \frac{E_a}{2.303R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right) \dots\dots\dots 4$$

Where CR_1 and CR_2 = corrosion rates at temperatures T_1 and T_2 respectively, E_a = activation energy and R = molar gas constant ($8.314 \text{ JK}^{-1} \text{ mol}^{-1}$)

The values of E_a calculated are recorded in Table 5 and was found to range from 63.02 to 75.19 kJ/mol. These values are larger than the value for the blank (52.09 kJ/mol) confirming that Gongronema latifolium retards the corrosion of mild steel in HCl. Ebenso et al., (2008) reported that values of $E_a < 80 \text{ kJ/mol}$ is indicative of physical adsorption while $E_a > 80 \text{ kJ/mol}$ is indicative of chemical adsorption. Thus the activation energies values supported the fact that methanol extract of Gongronemalatifolium was physically adsorbed on the mild steel surface.

The values of heat of adsorption were calculated using equation 5 (Bilgic and Sahin, 2001; Bhajiwala and Vashi, 2001).

$$Q_{ads} = 2.303R \left[\log \left(\frac{\theta_2}{1-\theta_2} \right) - \log \left(\frac{\theta_1}{1-\theta_1} \right) \right] \frac{T_1 \times T_2}{T_2 - T_1} \dots\dots\dots 5$$

Where θ_1 and θ_2 are degrees of surface coverage of the inhibitor at temperature T_1 and T_2 respectively, R is the molar gas constant ($8.314 \text{ JK}^{-1} \text{ mol}^{-1}$).

Values of Q_{ads} calculated through equation 5 are recorded in Table 5. These values were found to be negative in all concentrations showing that the adsorption of the extract was exothermic (Bhajiwala and Vashi, 2001; Iloamae et al., 2015).

Table 5: Calculated values of E_a and Q_{ads} for Gongronematifoliumextract at different concentrations

Blank	52.0	-
0.1	63.02	-13.71
0.2	63.13	-14.07
0.3	68.45	-22.17
0.4	70.64	-26.64
0.5	75.19	-28.47

3.5 Adsorption Consideration

The effectiveness of organic compounds as corrosion inhibitors can be ascribed to the adsorption of molecules of the inhibitors through their polar functions on the metal surface (Umoren et al., 2011). Adsorption isotherms provide information about the interaction among adsorbed molecules themselves as well as their interactions with the metal surface (Umoren et al., 2011).

The adsorption behaviour of methanol extract of Gongronematifolium was also studied by fitting data obtained from degree of surface coverage to different adsorption isotherms such as Langmuir, Freundlich and El-Awardy. The test revealed that adsorption of GL extract on the surface of the mild steel is consistent with the assumption of Langmuir, Freundlich and El-Awardy Isotherms.

Langmuir adsorption models can be represented as follows (Umoren et al., 2011; Ayuba et al., 2014).

$$C/\theta = C + 1/K_{ads} \dots\dots\dots 6$$

Taking the logarithm of equation 6, equation 7 was obtained

$$\log C/\theta = \log C - \log K \dots\dots\dots (7)$$

Where C is the concentration of the inhibitor in the electrolyte, θ is the degree of surface coverage of the inhibitor and k is the equilibrium constant of adsorption.

The plot of $\log C/\theta$ versus $\log C$ (fig 6) gave linear plots indicating that Langmuir adsorption isotherm is applicable to the adsorption of methanol extract of Gongronema latifoilum on the surface of the mild steel.

Freundlich adsorption isotherm of Gongronematifoilum extracts on the surface of the mild steel and is given by the equations 8 and 9 (Sharma and Sharma, 1999; Adejo et al., 2013).

$$\frac{x}{m} = kc^{1/n} \dots\dots\dots 8$$

Taking the logarithm of equation 8, equation 9 was obtained

$$\log \frac{x}{m} = \log K + \frac{1}{n} \log C \dots\dots\dots 9$$

The fraction $\frac{x}{m}$ in equation 9 has been found to be approximate to the inhibition efficiency of the inhibitor, k and n are constant. Slope is equal to $\frac{1}{n}$ and intercept = $\log K$.

Freundlich adsorption isotherm parameters were obtained from the plots of \log inhibition efficiency (I.E %) versus $\log C$ in Figure 7 which produced a straight line that obeyed Freundlich adsorption isotherm (Umoren et al., 2006). The degrees of linearity (R^2) were also close to unity indicating strong adherence of the adsorption of Gongronematifolium extract on the surface of the mild steel.

The strength of adsorption of Gongronematifolium on a mild steel surface and the possible formation of a multi-molecular layer of adsorption were investigated using the El-Awardy isotherm, which can be written as equation 10 (Shukla and Ebenso, 2011; Adejo et al., 2013).

$$\log \left(\frac{\theta}{1-\theta} \right) = \log k + y \log C \dots\dots\dots 10$$

Where, C is molar concentration of inhibitor in the bulk solution, θ is the degree of surface coverage, K is the equilibrium constant of adsorption process; $K_{ads} = K^{1/y}$ and y represent the number of inhibitor molecules occupying a given active site.

El-Awardy adsorption parameters were deduced from the plots of $\log (\theta/1-\theta)$ against $\log C$ in Figure 8 which gave straight lines and a good correlation coefficient which showed that the experimental data fits the isotherm (Shukla and Ebenso, 2011; Adejo et al., 2012). Values of $1/y$ less than unity imply the formation of multilayers of the inhibitor on the surface of the metal while values of $1/y$ greater than unity indicate that a given inhibitor will occupy more than one active site (Ebenso et al., 2010; Adejo et al., 2013).

The results obtained revealed that the values of $1/y$ are greater than unity, indicating that the inhibitor occupies more than one active site on the metal surface.

The equilibrium constant of adsorption of Gongronematifolium extract on the surface of the mild steel are related to the free energy of adsorption ΔG_{ads} according to equation 10 (Adejo et al., 2012; Odiongenyiet al., 2015).

$$\Delta G_{ads} = -2.303RT \log (55.5K) \dots\dots\dots 10$$

Where R is the gas constant, T is the temperature, K is the equilibrium constant of adsorption, 55.5 is the molar heat of adsorption of water. Values of K obtained from intercept of Langumir, Freundlich and El-Awardy isotherms were used to compute for ΔG_{ads} according to equation 10 and the result in Table 6.

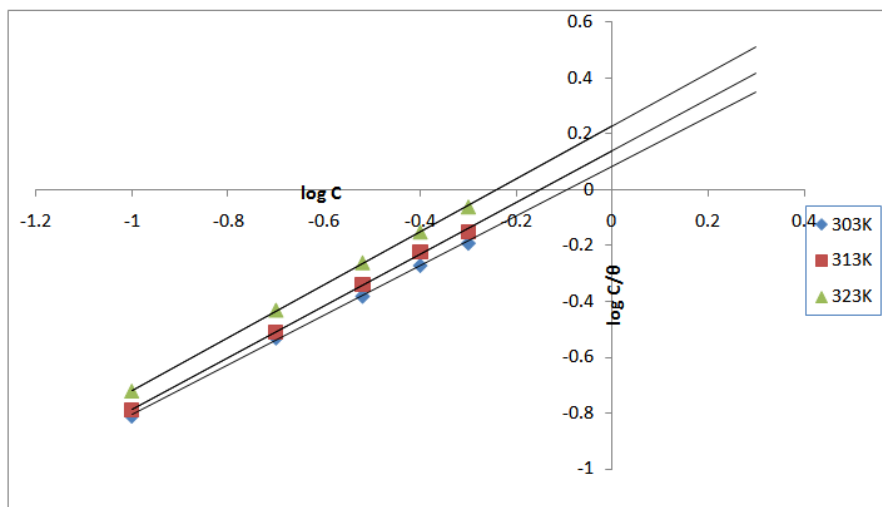


Figure 6: Langmuir isotherm for the adsorption of Gongronemalatifolium on the surface of mild steel.

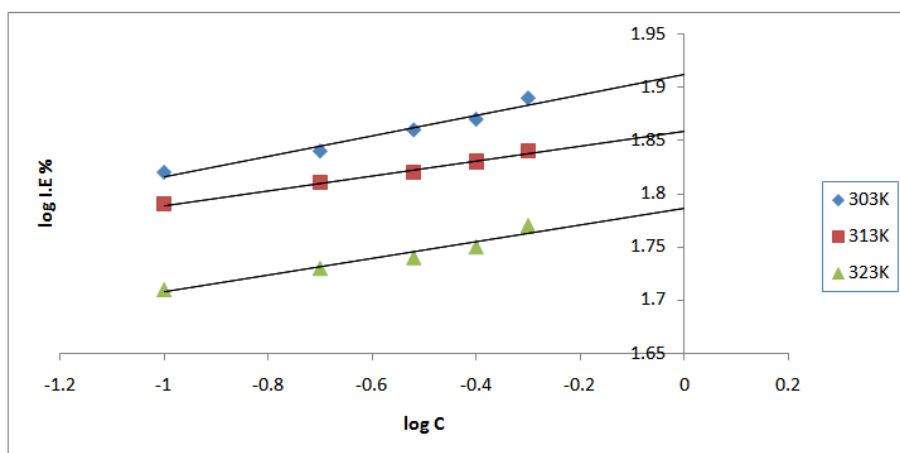


Figure 7: Freundlich isotherm for adsorption of the extract of Gongronemalatifolium on the surface of the mild steel.

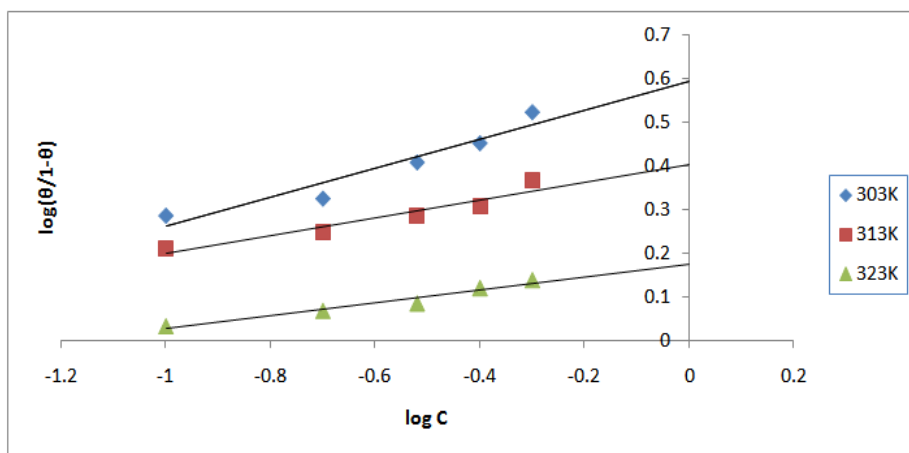


Figure 8: El-Awardy isotherm for adsorption of the extract of Gongronemalatifolium on the surface of the mild steel.

Table 6: Isotherm parameters for adsorption of Gongronema latifolium extract on the surface of the mild steel

Isotherm	Temperature(K)	1/y	Slope	R ²	K _{ads}	ΔG _{ads}
Langmuir	303	-	0.8865	0.9994	0.83	-9.65
	313	-	0.9274	0.9990	0.73	-9.63
	323	-	0.9443	0.9998	0.59	-9.37
Freundlich	303	-	0.0960	0.9649	81.68	-21.21.
	313	-	0.0690	0.9915	72.19	-21.59
	323	-	0.0790	0.9439	61.08	-21.83
El-Awardy	303	3.02	0.3315	0.9182	3.93	-13.57
	313	4.92	0.2032	0.9108	2.54	-12.88
	323	6.77	0.1478	0.9583	1.50	-11.88

IV. Conclusion

Gongronemalatifolium extract showed inhibitive effect on corrosion of mild steel in acidic environment. Inhibition efficiency increases with an increase in inhibitor concentration of the extract. The adsorption of the extract on the surface of the mild steel followed Langmuir, Freundlich and El-Awardy adsorption isotherms. The mechanism of physical adsorption was proposed from the trend of the inhibition efficiency with temperature and the values of E_a , Q_{ads} and ΔG_{ads} obtained.

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