Green Corrosion Inhibitors for Mild Steel in H_2SO_4 Solution: Flavonoids of Gongronema latifolium

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Abstract

The inhibitive properties of flavonoids extracted from *Gongronema latifolium* (FEGL) and the crude extracts from *Gongronema latifolium* (EEGL) on the corrosion of mild steel in H_2SO_4 solutions was studied using hydrogen evolution method at 30, 40, 50, and 60^oC. The results revealed that the extracts act as good inhibitors for the corrosion of mild steel in 5.0 M H_2SO_4 solutions. The inhibition efficiencies were found to increase with increase in the extract concentration and decreased with increase in temperature. The order of efficiency was EEGL > FEGL. The experimental data fitted well into the Langmuir adsorption isotherm. Physical adsorption mechanism was proposed for the adsorption of the extracts on the mild steel surface from the data obtained for the activation energies of the inhibited and uninhibited systems.

Keywords: Adsorption; Corrosion; Flavonoids; Inhibitor; Mild Steel

1.Introduction

Metals have numerous applications in industrial installations and equipment; however these metals are often attacked by corrosion. Metals and alloys are prone to corrosion either by man-made fluids or by one or more natural fluids. These reactions may damage the metal simultaneously with consequent loss of cohesive strength. Corrosion can be defined as the gradual deterioration of materials as a result of reaction with its environment (Shashi, 2003) It is basically the result of interaction between materials and their environment. Hence suitable inhibitors must be used if the life span of this valuable metal must be guaranteed. This has brought about the use of inhibitors which can counteract the corrosive action of such aggressive environments. Corrosion inhibitors are chemical substances that when added in small concentration to an environment, effectively decreases the corrosion rate. The chemical which can act as corrosion inhibitor may be inorganic or organic, but due to environmental toxicity of inorganic corrosion inhibitors, plant extracts have become important because they are environmentally acceptable, inexpensive, readily available and renewable sources of materials (Okafor *et al.,* 2006; Abeng *et al.,* 2013). Due to the usefulness of organic inhibitor in various industries, the study of the organic inhibition is now an alternative field of research. Most of the well-known acid inhibitors are organic compounds containing nitrogen, sulphur, oxygen and multiple bonds in the molecules. The lone pair of electron available on the atoms allows the easy adsorption into the metal surface (Amadi *et al*., 2004; Umoren *et al* 2006; 2008; Okafor *et al*., 2004; 2007; 2008; 2010; 2012; Ikeuba *et al*., 2013; Uwah *et al*., 2013; Ekanem *et al*., 2010; Odiongenyi *et al*., 2008; Solmaz *et al*., 2008; Alaneme *et al*., 2012; Singh *et al*.,2010; Ebenso *et al*., 2004).

Gongronema latifolium is a tropical rainforest plant belonging to the Ascepiadaceae family .*Gongronema latifolium* is a perennial edible plant with soft and pliable stem. It is widely used in the West African sub region for a number of medicinal and nutritional purposes. *Gongronema latifolium* has a very bitter taste and its phytochemical composition indicates that it contains saponins and flavonoid (Morebise *et al*., 2002). In some African cultures it is used as a spice to support the pancreas. *Gongronema latifolium* have been used by herbal medicine practitioners in combination with other herbs in the treatment of Staphylococcal and Salmonellal infections. In southern Nigeria the Igbo people call the leaves 'utazi' and the Yoruba people 'arokeke'. They are sharp-bitter and sweet and widely used as a leafy vegetable and as a spice for sauces, soups and salads. The leaves are used to spice locally brewed beer. The bark contains much latex and has been tested for exploitation (Ejoh *et al*., 2007). The extract of this plant which contains many environmental friendly compounds may be utilized as eco-friendly corrosion inhibitors. Several researches have been carried out on this plant as medicinally and nutritionally potent, traditional anti-diabetic agent and corrosion inhibitor (Adeleye *et al.*, 2011)

The present study seeks toinvestigate the effect of flavonoids and ethanolic extracts of *Gongronema latifolium* on the corrosion of mild steel in H_2SO_4 solutions at different temperatures and to investigate the adsorption mechanisms of the extracts on the corrosion of mild steel in H_2SO_4 solutions using gasometric technique.

2. Experimental

2.1 Mild steel coupon

The mild used for the study was obtained from Physics mechanical workshop in the University of Calabar,

Calabar. The mild steel coupons of dimension $0.8 \times 2.0 \times 5.0$ cm were mechanically polished with series of Emery paper of variable grades starting with the most coarse and proceeding in steps to the finest (600) grade. These polished coupons were degreased with ethanol, dried with acetone and weighed. The weighed coupons were then used for analyses. All the experimental solutions were prepared with distilled water. All the chemicals used were Analar grade and all weighing were done with AE ADAM PGW 253e digital analytical balance.

2.2 Preparation of ethanolic extract of Gongronema latifolium corrosion test.

The leaves of *Gongronema latifolium* were obtained from a local garden in Opu-oko in Khana Local Government Area, Rivers State, Nigeria. They were identified in the Department of Botany, University of Calabar. The leaves were dried in an N53 C-Genlab laboratory oven at $60^{o}C$ and grounded to powder form. 50 g of the powder was extracted continually with 250 cm^3 of absolute ethanol in a Soxhlet extractor for 24 hours. The extract was heated in a water bath at $50^{o}C$ until almost all of the ethanol evaporated to form a gel. 12 g of the gel was dissolved in 500 mL of 5.0 M H_2SO_4 solution, agitated vigorously and kept overnight (24 hours). The resultant solution was filtered and stored. This stock solution (24 g/L) was used to prepare test solutions of different concentrations of the inhibitor; 0.1, 1.0, 2.5, 5.0 and 10.0 g/L. The test solutions for the inhibitor were prepared by serial dilution of the stock solution.

2.3 Preparation of flavonoids extract of Gongronema latifolium stock solution

5 g of the dried powdered sample was weighed into a beaker and extracted with 50 cm^3 of 80% methanol at room temperature for one hour. The solution was filtered through filter paper. The filtrate was evaporated to dryness over water bath. The weight of the dried extract was taken and the amount of flavonoid present was calculated (Amadi *et al.*, 2004) 4.0 g of flavonoids extract of *Gongronema latifolium* was digested in 250 mL of 5.0 M H_2SO_4 solution. The resultant solution was kept for 24 hours and filtered. The stock solution (16g/L) obtained was used in preparing test solution of different concentrations of the inhibitor; 0.1, 1.0, 2.5, 5.0 and 10.0 g/L. The test solutions for the inhibitor were prepared by serial dilution of the stock solution.
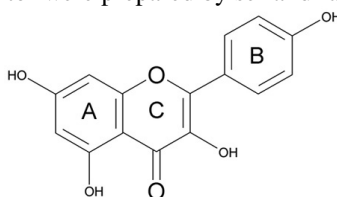

Figure 1: Typical Structure of a Flavone

2.4 Gasometric method

The gasometric measurements were carried out as previously described (Ekpe *et al.,* 1995).100 mL of the corrodent was introduced into the reaction chamber, connected to a burette through a delivery tube. A weighed mild steel coupon was dropped into the solution in the chamber and the reaction vessel was quickly closed to avoid any escape of hydrogen gas. The volume of the hydrogen gas evolved from the reaction was monitored by the depression (in mL) in the level of paraffin oil in the gasometric equipment. This depression in the paraffin oil level was recorded every minute for 30 minutes at ambient temperature. The same experiment was repeated in the presence of the inhibitor with concentrations of 0.1, 1.0, 2.5, 5.0, and 10.0 g/L. The surface coverage (θ) and inhibition efficiency were calculated using Equation 1 and 2. The rate for the hydrogen evolution was obtained from the slope of the trend line of the graph of volume of hydrogen evolved against time.

$$\theta = 1 - \frac{R_{inh}}{R_{blank}} \qquad\qquad 1$$

$$IE \ = \ \theta \times 100 \qquad\qquad 2$$

where θ is the surface coverage, R_{inh} is the corrosion rate in the presence inhibitor, R_{blank} is the corrosion rate in the absence of the inhibitor, IE is the inhibition efficiency (%).

3. Results and Discussion

3.1 Phytochemical screening of Gongronema latifolium leaves

Phytochemical results obtained for the aqueous extract of *Gongronema latifolium* is shown in Table 1. It is observed that saponins and flavonoids are present in a large quantity; oxalate is moderately present; alkaloid and glycosides are present in a negligible quantity and tannin is absent. This suggests the potency of the extract as a good inhibitor as it contains a considerable amount of photochemical with basic heteroatom. This work focuses on the use of the flavonoids fraction of the extract for corrosion studies. The structure of a typical flavonoid is shown in Figure 1 with oxygen as the major heteroatom present. The result of the quantitative phytochemical analysis of the plant extracts presented in Table 1 reveal the presence of flavonoids and saponins, alkaloids,

tannins, oxalates and glycosides. These compounds have been shown to possess significant anti-corrosion activity which is based principally on their structural characteristics such as lone pair of electrons on their nitrogen, sulphur and oxygen groups. These compounds adsorb on the metal surface via their lone pair of electrons on their oxygen, sulphur and nitrogen atoms, thus creating a protective film of metal–inhibitor complex leading to reduced metal-acid contact and reduced corrosion rate (Uwah et al., 2013).

Table 1. Phytochemical screening results for aqueous extract of *Gongronema latifolium* leaves

Phytochemicals	Inference	Quantitative results (%)
Alkaloids	+	4.00
Saponins	+++	22.00
Tannins	-	0.10
Flavonoids	+++	28.00
Glycosides	+	2.16
Oxalates	++	12.80

+++ = highly present; ++ = moderately present; + = present in negligible quantity and - = absent

3.2 Hydrogen evolution results for the extracts

The corrosion of mild steel in acid medium is characterized by hydrogen evolution and the corrosion rate is proportional to the amount of hydrogen gas evolved per surface area (Okafor *et al*., 2014). In 5.0 M H_2SO_4 solutions, the volume of hydrogen evolved per surface area during mild steel corrosion in the absence and presence of FEGL and EEGL at 30, 40, 50, and 60°C was measured as a function of time. Figure 2 shows the variation at 30°C, similar trends were obtained for 40, 50, and 60°C. It was observed that the hydrogen gas evolved per surface area increased with time.
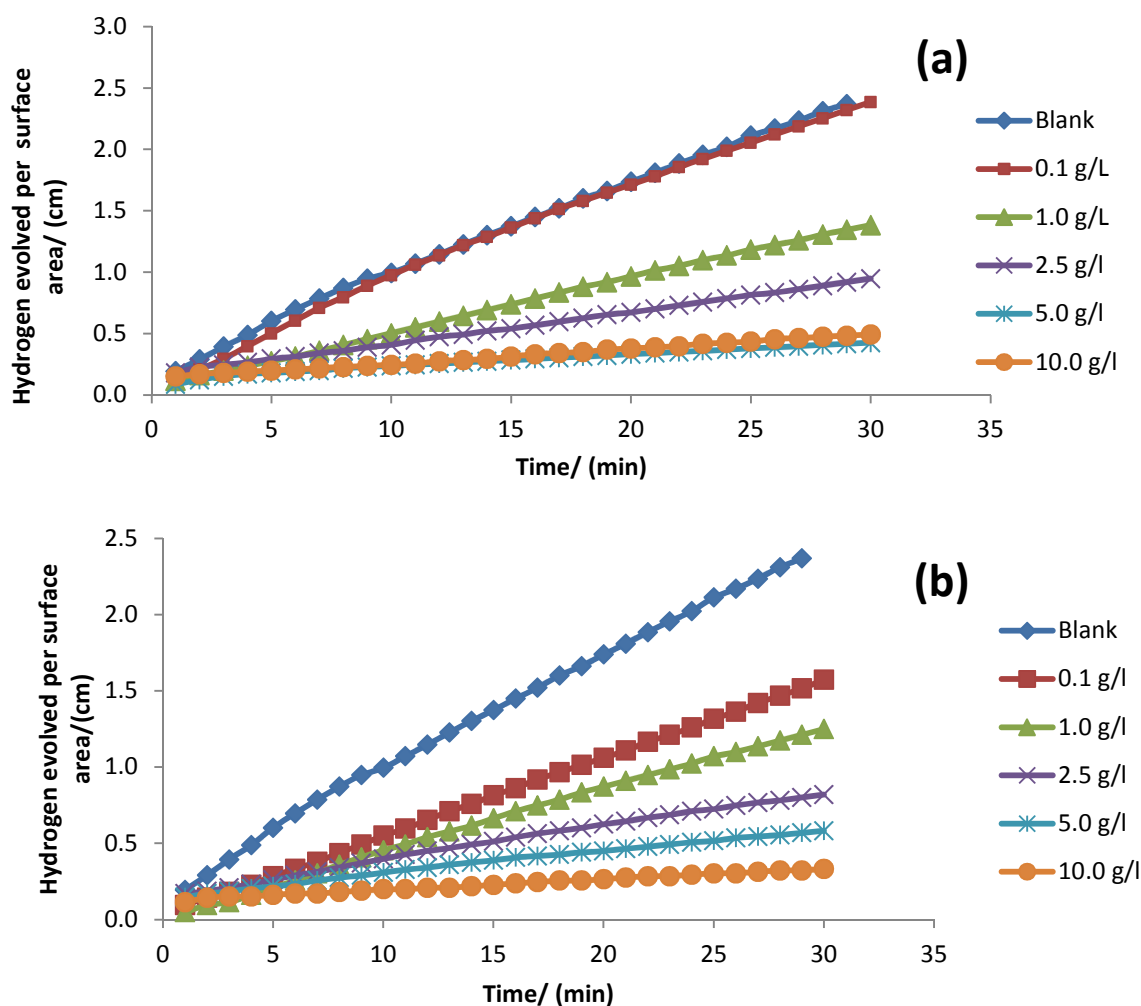

Figure2: Variation of hydrogen evolved per surface area with time for mild steel in 5.0 M H_2SO_4 solutions in the presence and absence of FEGL (a) and EEGL(b) at 30°C.

Table 2: Calculated values of corrosion rate, surface coverage, inhibition efficiency and activation energy for mild steel in 5.0 M H$_2$SO$_4$ solutions containing FEGLand EEGL

| Inhibitor	Conc g/L	Corrosion rate (cm/min)				Surface Coverage, θ				Inhibition Efficiency (% IE)			
		30^{0}C	40^{0}C	50^{0}C	60^{0}C	30^{0}C	40^{0}C	50^{0}C	60^{0}C	30^{0}C	40^{0}C	50^{0}C	60^{0}C
	Blank	0.075	0.158	0.23	0.385	-	-	-	-	-	-	-	-
	0.1	0.077	0.134	0.213	0.365	0.027	0.152	0.074	0.052	2.7	15.2	7.4	5.2
	1.0	0.044	0.086	0.147	0.256	0.413	0.456	0.361	0.335	41.3	45.6	36.1	33.5
FEGL	2.5	0.026	0.058	0.103	0.202	0.653	0.633	0.552	0.475	65.3	63.3	55.2	47.5
	5.0	0.01	0.026	0.068	0.108	0.867	0.835	0.704	0.719	86.7	83.5	70.4	71.9
	10.0	0.012	0.027	0.051	0.088	0.84	0.829	0.778	0.771	84.0	82.9	77.8	77.1
	0.1	0.051	0.099	0.211	0.344	0.32	0.687	0.541	0.554	32.0	68.7	54.1	55.4
	1.0	0.042	0.08	0.152	0.29	0.44	0.747	0.67	0.624	44.0	74.7	67.0	62.4
EEGL	2.5	0.022	0.045	0.053	0.152	0.707	0.858	0.885	0.803	70.7	85.8	88.5	80.3
	5.0	0.014	0.025	0.045	0.054	0.813	0.921	0.902	0.930	81.3	92.1	90.2	93.0
	10.0	0.007	0.017	0.031	0.045	0.907	0.946	0.933	0.942	90.7	94.6	93.2	94.2
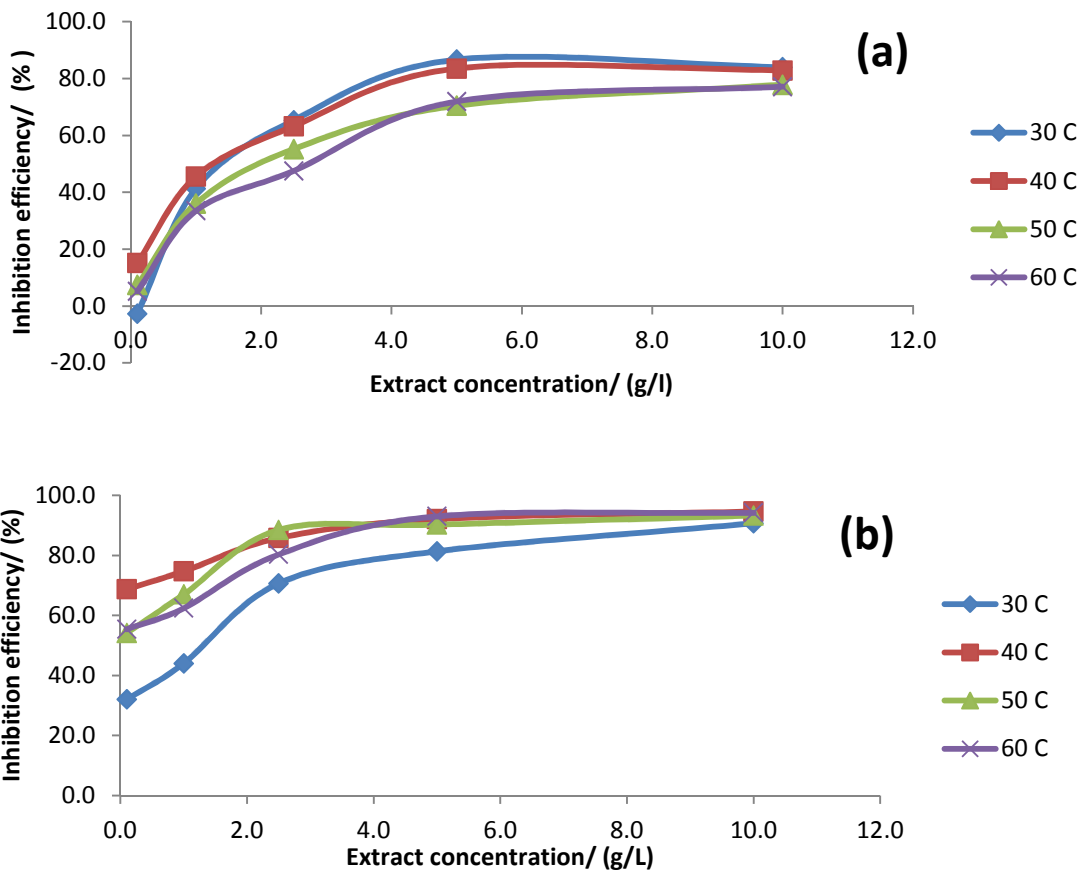
Figure 3:   Variation of inhibition efficiency with extract concentration for mild steel corrosion in 5.0 M H$_2$SO$_4$ solutions containing FEGL (a) and EEGL (b) at different temperatures.

3.3 The effect of concentration on inhibition efficiency
From Table 2, it is observed that corrosion rate decreases as inhibitor concentration increases; surface area increases with increase in extract concentration; inhibition efficiency increases with increase in extract concentration as well as decrease in temperature. This suggests that EEGL retards the corrosion of mild steel (Ikeuba *et al.,* 2013). Figure2 shows the variation of rate of evolution of hydrogen with extract concentration for mild steel in 5.0 M H$_2$SO$_4$ solutions containing EEGL and FEGL. It is observed that the corrosion rate decreases with increasing concentration of extract. This indicates that the plant extracts mitigate the corrosion of mild steel in 5.0 M H$_2$SO$_4$solutions and that the extent of corrosion inhibition depends on the amount of the extract present. Figure 3 gives the trend of inhibition efficiency with concentration. There is an initial sharp increase in inhibition efficiency which later levels up after 6 g/L of the inhibitor. This is an indication that the efficiency of the

inhibitor lowers with concentrations.

The results obtained for corrosion rate, surface coverage, inhibition efficiency and activation energy is shown in Table 2. From Table 2, it is observed that inhibition efficiency increases with increase in EEGL and FEGL concentration from 1.0 g/L to 10 g/L attaining a maximum of 86.7 and 94.2 at 10.0 g/L for FEGL and EEGL respectively. This suggests that FEGL are adsorbed on the metal surface thereby protecting metal from the action of the corrodent. The adsorption of phytochemicals present in the plant on the metal surface is believed to be via the basic hetero atoms present in the molecular structure of the phytochemicals. This is in line with the previous reports by Okafor *et al*., 2014; 2012

3.4 Effect of temperature

From the data in Table2 the inhibition efficiency was found to generally decrease with rise in temperature for FEGL and increase with rise in temperature for EEGL. This suggests a physical interaction between the metal and the inhibitor for FEGL and a chemical interaction for EEGL. The results obtained are as displayed in Table 2 from which it is observed that the inhibition efficiency is temperature dependent; this is evident in the general decrease in efficiency with increase in temperature for FEGL and the reverse for EEGL. The activation energy (E_a) of the corrosion/corrosion inhibition process was determined using the Arrhenius equation given in Equation 3.

$$Log\ Rate = Log\ A - \frac{Ea}{2.303RT}$$ 3

Where E_a is the activation energy, R is gas constant, A is Arrhenius constant and T is temperature. Figure 4 shows the Arrhenius plot for mild steel corrosion in 5.0 M H_2SO_4 solutions containing EEGL and FEGL at different temperatures. From the plot, it is observed that the logarithm of corrosion rate decreases with increase in temperature.

Table 3 shows the calculated values of activation energy obtained in the study. From Table 3, the activation energy for the corrosion of mild steel in 5.0 M H_2SO_4solution in the presence of EEGL and FEGL was found to be < 80.0 kJ/mol. This suggests that the adsorption phenomenon is a physical adsorption for both extracts. The above observation agree with that of Ikeuba *et al*. 2013 which indicates that the activation energy will be less than 80.0 kJ/mol for physical adsorption mechanism and more than 80.0 kJ/mol for chemical adsorption mechanism. The increase in the apparent activation energies in the inhibited solution may be attributed to an appreciable decrease in the adsorption of the inhibitor on the mild steel surface with increase in temperature Uwah *et al.*, 2013. The activation energy for FEGL increased gradually from 42.09 to 54.33 kJ/mol with increase in concentration indicating an increased tendency toward chemical adsorption at higher concentrations. However an opposite tendency was observed in the presence of EEGL, the general fall in the activation energy from 60.9 to 51.0 kJ/mol for EEGL suggesting that an increased tendency towards physical adsorption of the extracts unto the mild steel surface at higher concentrations.
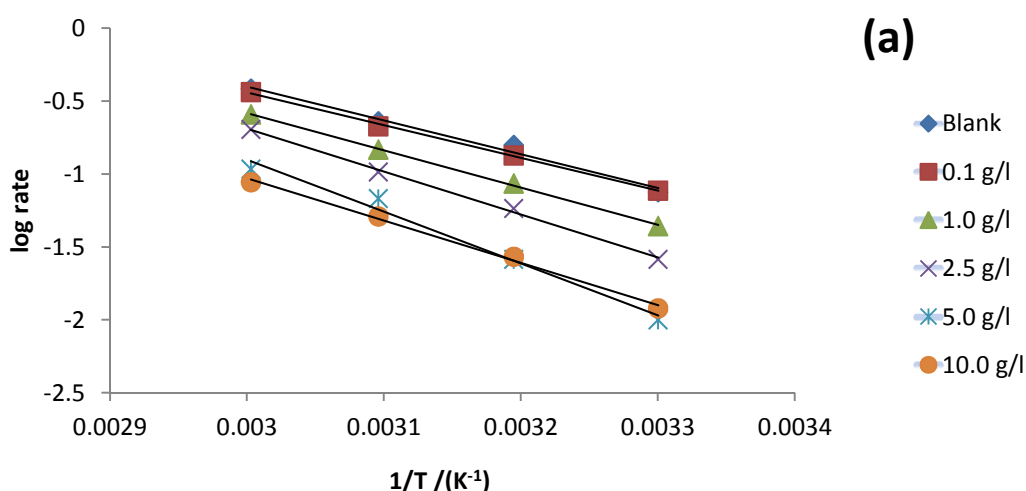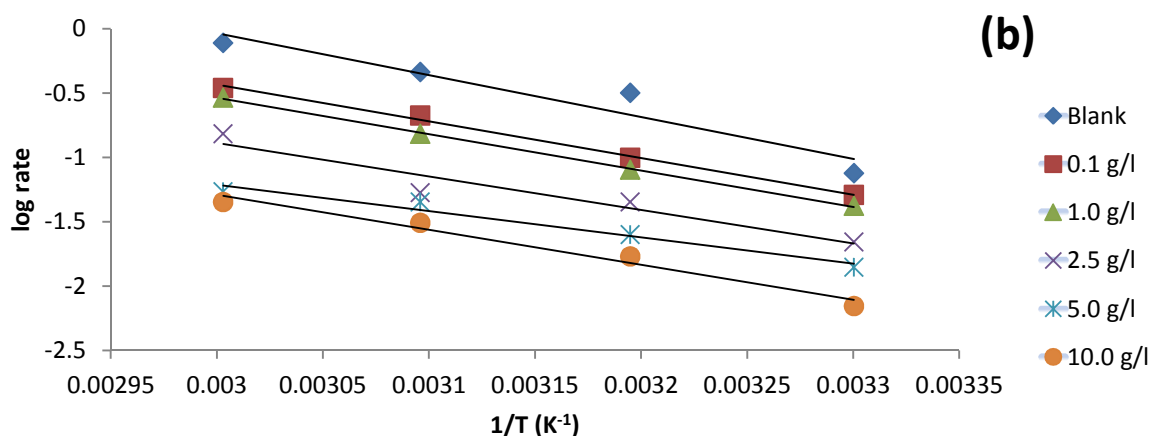
Figure. 4: Arrhenius plot for mild steel corrosion in 5.0 M H_2SO_4 solutions containing (a) EEGL and (b) FEGL

Table 3.   Calculated values of activation energy for mild steel corrosion in 5.0 M H_2SO_4 solutions in the absence   (blank)and presence of different concentrations of EEGL and FEGL

Concentration / (g/L)	E_a (kJ/mol)	
	EEGL	FEGL
Blank	60.9	42.09
0.1	53.3	43.46
1.0	52.8	47,79
2.5	48.7	55.22
5.0	38.2	66.69
10.0	51.0	54.38

3.5 Adsorption behaviour of EEGL and FEGL
The possible adsorption mode was investigated by testing the experimental data obtained with several adsorption isotherms. Such investigations will greatly throw more light to understanding of the corrosion inhibition mechanism. It is suspected that the inhibition of metal corrosion occurred as a result of the adsorption of the active principles of the extracts onto the metal surface. The experimental data for EEGL and FEGL fitted the Langmuir adsorption isotherm model given as:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad\qquad 4$$

where, θ is the surface coverage, C is the concentration, K_{ads} is the equilibrium constant of adsorption process. The plot of $\frac{C}{\theta}$ against C is shown in Figure. 5. Linear plot were obtained across the temperatures studied. The values of the adsorption rate constant (K_{ads}) from the isotherm and the correlation factors (R) were estimated from Figure 4 and are presented in Table 4. The correlation factors obtained from the plots were close to unity (0.990 to 0.999and 0.991 to 0.999 for FEGL and EEGL, respectively). This indicates strict adherence to the principles underlying the derivation of Langmuir isotherm. However these values were not equal to 1, this indicates a slight deviation from Langmuir theory of adsorption (Okafor *et al.,* 2007). The slopes obtained from the plots were observed to be close to unity; and the isotherm was observed to be best fitted. Adherence to Langmuir suggests formation of monolayer of adsorption and no interaction between the adsorbed species. From Table 4, it is observed that FEGL shows greater deviation from Langmuir adsorption isotherm than that of EEGL. From the K_{ads} values, the standard adsorption free energy ΔG values (shown in Table 2) were obtained using Equation 5.

$$\Delta G = -2.303RT \, Log \, (55.5K_{ads}) \qquad\qquad 5$$

where ΔG is the standard adsorption free energy, R is the gas constant, T is the absolute temperature and K_{ads} is the adsorption rate constant. Negative values of ΔG; -9.88 to -9.12 for FEGL and -10.69 to -13.97 for EEGL indicate spontaneity and stability of the adsorption layer. The values of the free energy obtained which were below -40kJ/mol also suggests a physical adsorption mechanism for the adsorption of the inhibitor on the mild steel surface in 5.0 M H_2SO_4 solution (Singh *et al.,* 2010).
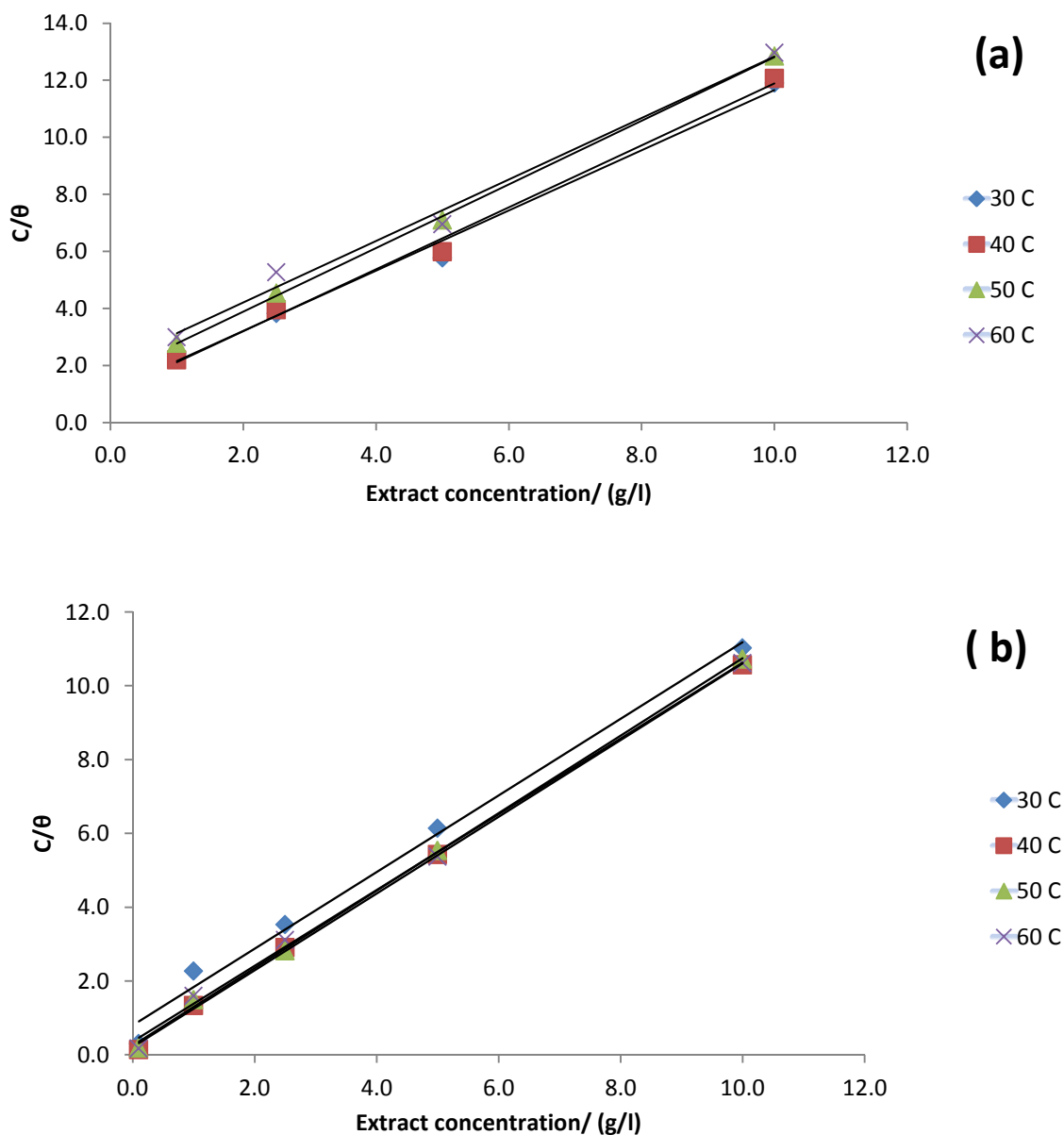
Figure. 5. Langmuir adsorption isotherm for FEGL (a) and EEGL (b) at 30, 40, 50 and 60°C

Table 4. Adsorption parameters for the corrosion of mild steel in 5.0 M H_2SO_4 solutions containing FEGL and EEGL at different temperatures

	Temperature (^{o}C)	K_{ads}	ΔG (KJ/mol)	R^2	Slope
FEGL	30	0.908	-9.88	0.990	1.054
	40	0.978	-10.4	0.994	1.086
	50	0.605	-9.44	0.999	1.115
	60	0.486	-9.12	0.990	1.077
	30	1.26	-10.69	0.991	1.038
EEGL	40	4.76	-14.52	0.999	1.043
	50	4.08	-14.57	0.999	1.05
	60	2.79	-13.97	0.997	1.026

4. Conclusion

The following conclusions have been drawn from the results of the study:

- EEGL and FEGL are good corrosion inhibitors for mild steel in 5.0 M H_2SO_4 solutions.
- The inhibition efficiency of EEGL and FEGL increases with increase in extract concentration.
- The adsorption characteristics of EEGL and FEGL on mild steel in 5.0 M H_2SO_4 solutions follow Langmuir adsorption isotherm and physical adsorption mechanism has been proposed for the adsorption process based on the values of E_a and ΔG.
- The inhibition properties of EEGL and FEGL on mild steel in 5.0 M H_2SO_4 solutions are due to the phytochemicals present in the extract which adsorb on the metal surface via their hetero atoms.
- The trend of the inhibitive performance of the extract is EEGL > FEGL.

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