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Corrosion Inhibition Effects of *Ficus exasperatae* and *Costus afer* in Acidic Medium at Different Temperatures

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ABSTRACT

One of the most effective ways of mitigating the corrosion of metals in various media is by using of corrosion inhibitors. Due to the harmful effects of chemical-based corrosion inhibitors, attention is now shifting to plant materials which are environmentally friendly. Therefore, this paper investigates the corrosion inhibition potentials of *Ficus exasperata* and *Costus afer* leaves extracts on the corrosion of mild steel in 0.5 M hydrochloric acid medium at different temperatures (303 – 343 K). Secondly, to determine the effect of temperature on the corrosion inhibition performance of *Ficus exasperata* and *Costus afer*. The weight loss method of corrosion measurement was used to evaluate the corrosion inhibition potentials of the plant extracts. The results showed that *Ficus exasperata* and *Costus afer* inhibited the corrosion of mild steel in hydrochloric acid medium at all temperatures. Corrosion rates decreased with increase in concentration of the extracts. Concentration of the extracts used (0.125 g/L, 0.25 g/L, 0.5 g/L, 1.25 g/L and 2.5 g/L) did not significantly ($p > 0.05$) affect the corrosion rate of mild steel in the acidic media. Corrosion rates increased with increase in temperature. Inhibition efficiencies of the extracts increased with increase in concentration of the extracts and decreased with increasing temperatures. The adsorption of the extracts on the metal surface occurred by physical adsorption. *Ficus exasperata* and *Costus afer* could serve as effective inhibitors for mild steel in acidic media and their efficiencies are affected by high temperatures.

Keywords: Corrosion rate, Inhibition efficiency, Temperature, *Ficus exasperata, Costus afer*

INTRODUCTION

Mild steel, a ferrous alloy is used in the design of numerous accessories in petrochemical, chemical, sewage, water and food industries exposing them to different concentration of corrosive solutions (Alaneme *et al*., 2016). The ease of fabrication, low cost, good weldability and great combination of mechanical properties are some of the qualities which account for the choice of mild steel for construction purposes (Yaro *et al*., 2013). However, mild steel has little or no resistance to corrosion particularly in acidic environment making them vulnerable to corrosion. Corrosion is of high economic, environmental, industrial and domestic safety importance and as such needs to be mitigated (American Society for Materials, 2000). Among several methods that have been employed for curbing corrosion, the use of inhibitors is the most common (Ostovai *et al*., 2009). Most organic compounds containing heteroatoms like oxygen (O), nitrogen (N), sulphur (S) or multiple bonds have the ability to mitigate corrosion of mild steel in various media (Hussin and Kassim, 2010; Hussin *et al*., 2016). However, the high cost of production and toxicity of these compounds have necessitated the search for less toxic and eco – friendly corrosion inhibitors (Ostovai et al., 2009; Hussin and Kassim, 2011; Hussin *et al*., 2016; Alaneme *et al*., 2016; Umoren *et al*., 2016; Chaubey *et al*., 2017).

Plants are now being used as alternatives to chemical-based corrosion inhibitors because they are cheap, ecologically friendly and renewable sources (Umoren *et al*., 2008). Several researchers have successfully used extracts from green plants to mitigate the corrosion of metals in various media (Molina – Ocampo *et al*., 2015; Fadare *et al.,* 2016; Godwin-Nwakwasi *et al*., 2017; Khadom *et al*., 2018; Agi *et al*., 2018; Waidi *et al.,* 2020; Izionworu *et al.,* 2021; Oruene *et al*., 2021). Though many plants have been investigated for their anticorrosion inhibition capabilities, there are still many that have not been studied for their corrosion inhibition potentials. In order to increase the number of plants that possess anticorrosion potentials, we report the corrosion inhibition potentials of *Ficus exasperata* and *Costus afer* leaves extracts. Phytochemical analyses of *Ficus exasperata* and *Costus afer leaves* extracts revealed the presence of phytochemical compounds in the extracts (Anyasor *et al*., 2010; Adebayo *et al*., 2009). Phytochemical screening of methanol extract of *Ficus exasperata* revealed the presence of flavonoids, saponins, tannins, steroids and phlobatannins with no traces of alkaloids and anthraquinones (Adebayo *et al*., 2009). The phytochemical constituents and antioxidant activities of aqueous and methanol extracts of *Costus afer* were evaluated by Anyasor *et al*. (2010). The results showed that *costus afer* contained flavonoids, phenols, anthraquinones, cardiac glycosides, terpenoids, alkaloids and tannins. These phytochemical constituents are mostly responsible for the corrosion inhibition potentials of plants (Eddy, 2009; Okafor *et al*., 2010; Al-Otaibi *et al*., 2014; Fetouh *et al*., 2014); therefore, these plants (Ficus exasperata and *Costus afer*) have good corrosion inhibition potentials. The objectives of the study are to evaluate the corrosion inhibition potentials of Ficus exasperata and *Costus afer leaves* in acidic medium and to compare the corrosion inhibition performance of Ficus exasperata and *Costus afer* leaves extracts at various temperatures (303 K, 323 K and 343 K) in hydrochloric acid medium.

MATERIALS AND METHODS

Preparation of Acid Standard Solution

The required concentration of the corrodent (0.5 M hydrochloric acid) was prepared by diluting 11.29 M hydrochloric stock solution in 500 mL volumetric flask.

Processing of Plant Materials

The leaves of two plants *Ficus exasperata* and *Costus afer* were collected from a farm in Ugwuorji in Owerri - North local government area of Imo State, Nigeria. The leaves were identified and authenticated in the Department of Plant Science and Biotechnology, Imo State University, Owerri, Nigeria. The leaves were washed under running water to remove dust and sand particles. The leaves were further rinsed in distilled water. The leaves were cut into pieces, shade dried for five days and then grounded into fine powder using an electric blender. The powdered samples were separately stored in airtight containers.

Preparation of Plant Extracts

To extract the active ingredients in the plant materials, 500 mL each of 0.5 M hydrochloric acid was measured and poured into two flasks. The required quantity (25 grammes) of the finely powdered leaves were measured and placed in each of the flasks containing 500 mL of 0.5 M hydrochloric acid solution. The mixtures were allowed to stay overnight. The mixture in each flask was filtered by suction and the filtrate collected in a conical flask. The filtrate obtained from each flask was taken as the stock solution. The required concentrations of the extracts (0.125 g/L, 0.25 g/L, 0.5 g/L, 1.25 g/L and 2.5 g/L) were separately obtained from the stock solutions of Ficus exasperata and *Costus afer.*

Preparation of Mild Steel Coupons

Mild steel with chemical composition 0.07 % C, 0.01 % Si, 1.27 % Mn, 0.02 % P and the rest Fe was used for the preparation of the corrosion test specimens. The sheet was mechanically press cut into different test specimens each of dimension 1.5 cm × 3 cm × 0.1 cm. The coupons were polished with abrasive papers using water as a lubricant, to produce smooth surfaces and to remove any trace of contaminants. The coupons were degreased by soaking in ethanol (100 ml of 99 % pure ethanol BDH). The coupons were further immersed in 70 % acetone for three minutes, washed thoroughly in deionized water and rinsed. The washed specimens were cleaned with cotton wool, sun dried for five minutes and then stored in an airtight container.

Weight Loss Measurements

Weight loss measurements were conducted at temperatures of 303 K, 323 K and 343 K respectively. The mild steel specimens were each suspended and totally immersed in 0.5 M hydrochloric acid without and with different concentrations (0.125 g/L, 0.25 g/L, 0.5 g/L, 1.25 g/L and 2.5 g/L) of Ficus exasperata and *Costus afer* leaves extracts with the aid of strings and rods for two hours. The samples were retrieved from the corrodent, washed thoroughly in ethanol, rinsed in distilled water, dried and weighed to obtain the final weight. The weight loss was obtained by computing the difference between the initial weight and final weight. From the weight loss results, the corrosion rate (CR) was computed using Equation (1) (Okafor *et al*., 2010).

$$\text{CR } (\text{gcm}^{-2}\text{hr}^{-1}) = \frac{W_L}{At} \qquad (1)$$

Where W_L = Weight loss in grammes, A = Total surface area of test specimen in square centimeters and t = Immersion time in hours

The inhibition efficiencies (%) of the extracts were computed using Equation (2) (Okafor *et al*., 2010).

$$\eta = \frac{CRo - CRi}{CRo} \times 100\% \qquad (2)$$

Where CRo and CRi are the corrosion rates in the absence and presence of the extracts respectively.

Adsorption Isotherm

The adsorption characteristics of Ficus exasperata and *Costus afer* extracts on mild steel surface were investigated using the Langmuir isotherm model. The Langmuir adsorption isotherm model is based on the assumption that the adsorption takes place at specific sites within the adsorbent (Noor, 2009). The Langmuir adsorption isotherm is represented by Equation (3) (Noor, 2009; Chaubey *et al*., *2017*).

$$\frac{Cinh}{\theta} = \frac{1}{Kads} + C_{inh} \qquad (3)$$

Where K_{ads} (Lg^{-1}) is the adsorption equilibrium constant, C_{inh} is the concentration of the inhibitor in g/L and Ɵ is the degree of surface coverage. The degree of surface coverage was computed using the expression in Equation (4) (Umoren *et al*., 2008).

$$\theta = \frac{CRo - CRi}{CRo} \qquad (4).$$

Where CRo and CRi are the corrosion rates in the absence and presence of the extracts. The Langmuir adsorption isotherm model was applied to the degree of surface coverage of each of the extracts by calculating the values of $\frac{C_{inh}}{\theta}$ for each of the extracts. After that, the graph of $\frac{C_{inh}}{\theta}$ against C_{inh} for each of the extracts were plotted using excel spreadsheet. The coefficient of correlation (R^2) and the adsorption equilibrium constant (K_{ads}) for each of the extracts were determined from each of the graphs. The free energy of adsorption (ΔG^o_{ads}) was computed using the expression in Equation (5) (Eddy, 2009; Noor, 2009)

$$\text{Log } K_{ads} = -\text{Log}C_{H2O} - \frac{\Delta Gads}{2.303RT} \qquad (5)$$

where C_{H2O} is the concentration of water in solution expressed in gL^{-1}, R is the universal gas constant in $KJmol^{-1}K^{-1}$ and T is absolute temperature in Kelvin (K).

Thermodynamics Studies

The apparent activation energy (E_{app}) for the corrosion of mild steel in hydrochloric acid was computed using Arrhenius Equation in (6) (Noor, 2009).

$$\text{Log CR} = \text{Log A} - \frac{Eapp}{2.303RT} \qquad (6)$$

Where CR is the corrosion rate, R is the gas constant and T is the temperature. The logarithm of corrosion rate (Log CR) was plotted against the inverse of temperature ($\frac{1}{T}$) for each of the extracts using excel spreadsheet. The values of the apparent activation energy (E_{app}) for each extract were computed from the slopes of the plots. The enthalpy of activation ($\Delta H^{\neq}$) and the entropy of activation ($\Delta S^{\neq}$) were computed using Eyring's Equation in (7) (Hassan and Zaafarany, 2013).

$$-\text{In}\frac{Rh}{NT}\text{ Rc} = \frac{\Delta H^{\neq}}{RT} - \frac{\Delta S^{\neq}}{R} \qquad (7)$$

where h is the Plank's constant, N is the Avogadro's number, T is the absolute temperature, R is the gas constant and Rc is the corrosion rate. The graph of -In$\frac{hRc}{KBT}$ was plotted against the inverse of temperature ($\frac{1}{T}$). The activation parameters were evaluated from the slopes and intercepts of the plots for each extract (Hassan and Zaafarany, 2013).

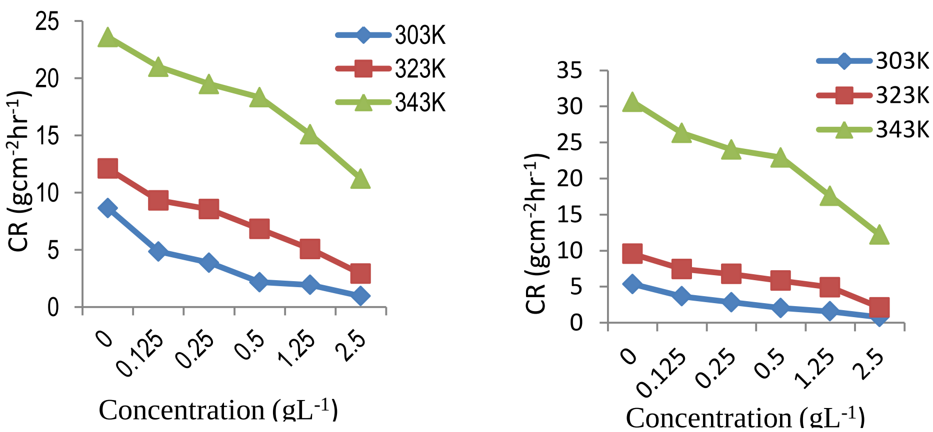
Data Analysis

Experimental data were presented graphically using Microsoft excel spread sheet. Descriptive statistics was used to compare the corrosion rates in environment without any extract (control) and in environments with different concentrations of the extracts. The values were considered statistically significant at $p < 0.05$.

RESULTS AND DISCUSSION

Effect of Concentration of Extracts on Corrosion Rate

Extracts of two plants (*Ficus exasperata* and *Costus afer)* were investigated for their corrosion inhibition potentials in 0.5 M hydrochloric acid solution at different temperatures. Figures 1a and b shows the effect of concentration of Ficus exasperata and *Costus afer* extracts on the corrosion of mild steel in 0.5 M hydrochloric acid solution at different temperatures. It was also observed that the corrosion rates at 0.0 g/L



(a) (b)

Figure 1: Effect of concentration of *Ficus exasperata* (a) and *Costus afer* (b) extracts on the corrosion of mild steel in Hydrochloric acid solution at different temperatures.

were higher compared to the corrosion rates obtained at other concentrations (0.125 g/L, 0.25 g/L, 0.5 g/L, 1.25 g/L and 2.5 g/L) of the extracts. This implies that the extracts (*Ficus exasperata* and *Costus afer)* inhibited the corrosion of mild steel in hydrochloric acid medium (Abeng *et al.*, 2013; Babatunde *et al.*, 2019). The low corrosion rates obtained in the presence of the extracts could be attributed to the adsorption of the phytochemical compounds present in the extracts on the metal surface(s) (Waidi *et al.*, 2020). These adsorbed compounds reduce corrosion reactions at the metal – solution interface, thereby decreasing the rate of corrosion (Prithiba *et al.*, 2014). Therefore, there is a reduction in the deterioration of the metal (s) in environment (s) containing the extracts. Also, corrosion rates were observed to decrease as the concentration of the extracts increased from 0.0 g/L to 2.5 g/L at all temperatures. Similar results were previously reported (Ogukwe *et al*., 2012; Uwah *et al*., 2013; Oruene *et al*., 2021). The decrease in corrosion rate with concentration of the extracts could be attributed to the increase in the degree of surface coverage and also an increase in the adsorption of the inhibiting species on the mild steel surface (Oguzie, 2006; Fadare *et al*., 2016 and Shuaib – Babata *et al*., 2018). The adsorption of the extracts created a barrier between the metal (s) and the acid solution, thereby reducing the rate of corrosion reactions (Fadare *et al*., 2016 and Oruene *et al*., 2021). Table 1 shows the effect of concentration of the extracts on the corrosion rate of mild steel in hydrochloric acid solution using descriptive statistics test. The result showed that the corrosion rates decreased with increase in the concentration of the extracts. Corrosion rates were maximum at 0.000 g/L (control) and minimum at 2.500 g/L of the extracts. There was no significant difference ($p > 0.05$) in the corrosion rate of mild steel in hydrochloric acid medium without any extract (control) compared to the corrosion rate in hydrochloric acid medium with different concentrations (0.125 g/L, 0.250 g/L, 0.500 g/L, 1.250 g/L and 2.500 g/L) of the extracts. This could be attributed to the concentrations of the extracts used.

Table 1: Effect of concentration of extracts on the corrosion rate of mild steel in hydrochloric acid medium

Concentration (g/L)	Corrosion rate (g$Cm^{-2}hr^{-1}$)	
	Ficus exasperata	*Costus afer*
0.000 (control)	14.79 ± 4.52^a	15.17 ± 7.81^b
0.125	11.73 ± 4.81^a	12.47 ± 7.00^b
0.250	10.64 ± 4.63^a	11.20 ± 6.51^b
0.500	9.12 ± 4.80^a	10.26 ± 6.42^b
1.250	7.37 ± 3.97^a	8.02 ± 4.87^b
2.500	5.03 ± 3.14^a	5.03 ± 3.61^b

Values are expressed as Mean$\pm$ Standard error of the mean. Values with the same alphabetic superscript on the same column are not significantly different ($p > 0.05$)

Effect of Temperature on Corrosion Rate

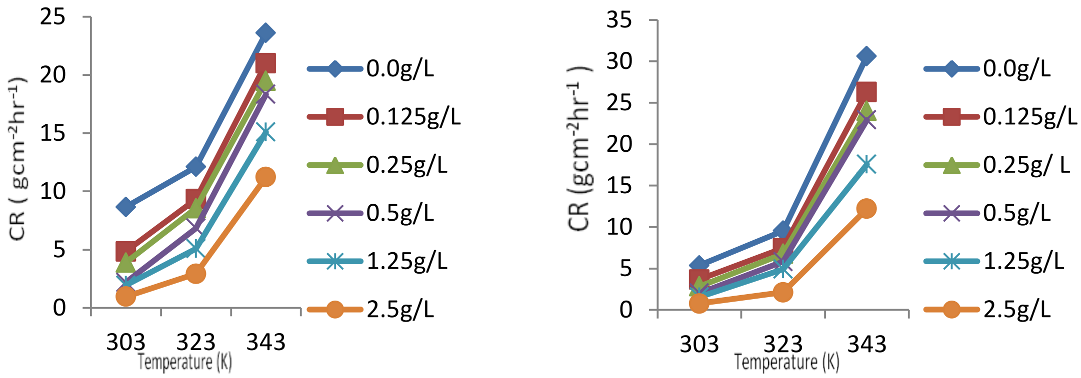
The effects of temperature on the corrosion rate of mild steel in the acidic medium at various concentrations of the extracts are presented in Figures 2a and b. An inspection of the figures revealed that the corrosion rates increased with temperature. Similar trends were also obtained by Loto *et al*. (2011) and Godwin - Nwakwasi *et al*. (2017). The corrosion rate of mild steel in the acidic medium increased with temperature due to increase in corrosion reactions at elevated temperatures (Loto *et al*., 2011) and the partial desorption of the inhibiting species (Fadare *et al*., 2016) at elevated temperatures. It was also observed that the corrosion rates obtained at 303 K were lower compared to the corrosion rates obtained at 323 K and 343 K. Maximum values of corrosion rates were obtained at a temperature of 343 K in environments containing *Ficus exasperata* and *Costus afer* extracts. This could probably be due to the reasons already stated above.

Effect of Concentration on the Inhibition Efficiencies of the Extracts

Presented in Figures 3a and b are the calculated values of the inhibition efficiencies of Ficus exasperata and *Costus afer* at temperatures of 303 K, 323 K and 343 K. It was observed from all the figures that the inhibition efficiencies of all the extracts increased with increase in the concentration of the extracts. Similar observations were made by Uwah *et al.* (2010); Babatunde *et al.* (2019) and Izionworu *et al*. (2020). Optimum inhibition efficiencies of the extracts were observed at concentration of 2.5 g/L

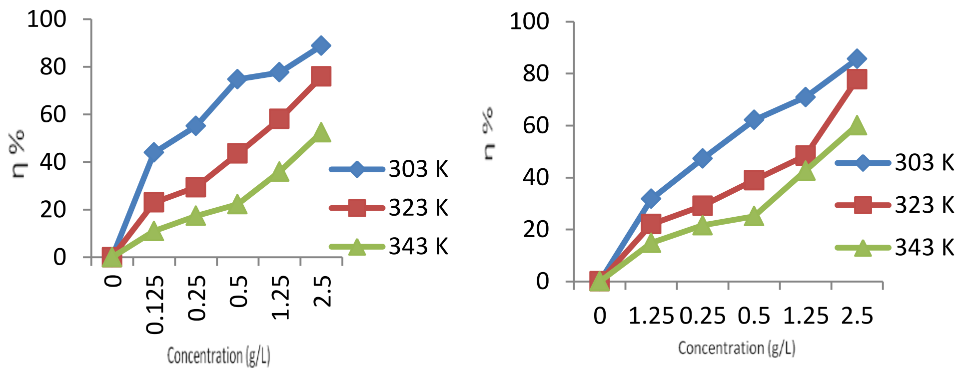
Effect of temperature on the Inhibition Efficiencies of the Extracts

The effect of temperature on the inhibition efficiencies of the extracts are presented graphically in Figures 4a and b. It was observed that the inhibition efficiencies of the extracts decreased with increase in temperature, most likely due to the weakening or desorption of the inhibiting species on the metal surface(s) with increase in temperature (Al-Amiery *et al*., 2014; Fadare *et al*., 2016). Consequently, a greater area of the metal surface is available for corrosion reactions as the temperature increases. Maximum and minimum efficiencies were obtained at 303 K and 343 K respectively. Decrease in inhibition efficiencies of the extracts with temperature is suggestive of physical adsorption mechanism (Ating *et al*., 2010).



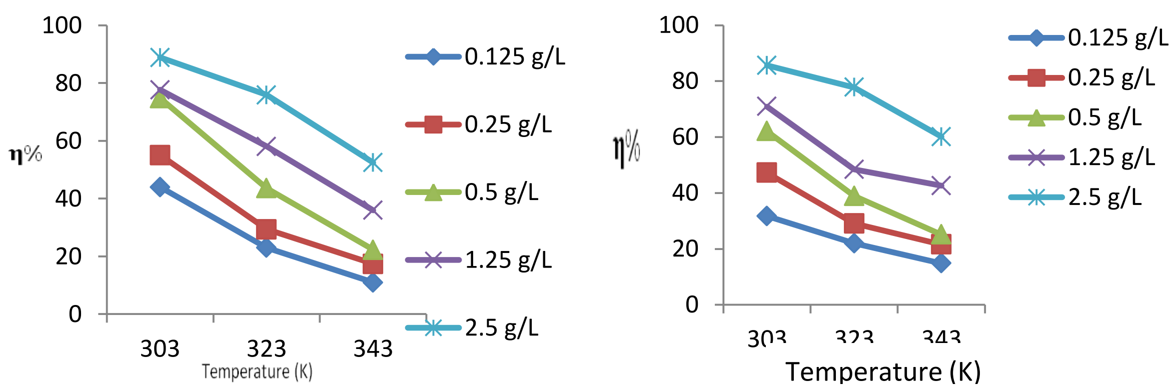
(a) (b)

Figure 2: Effect of temperature on the corrosion rate of mild steel in HCl acid solution with *Ficus exasperata* (a) and *Costus afer* (b) as inhibitor.



(a) (b)

Figure 3: Effect of concentration on the inhibition efficiency of *Ficus exasperata* (a) and *Costus afer* (b) extracts in 0.5 M hydrochloric acid solution at different temperatures.

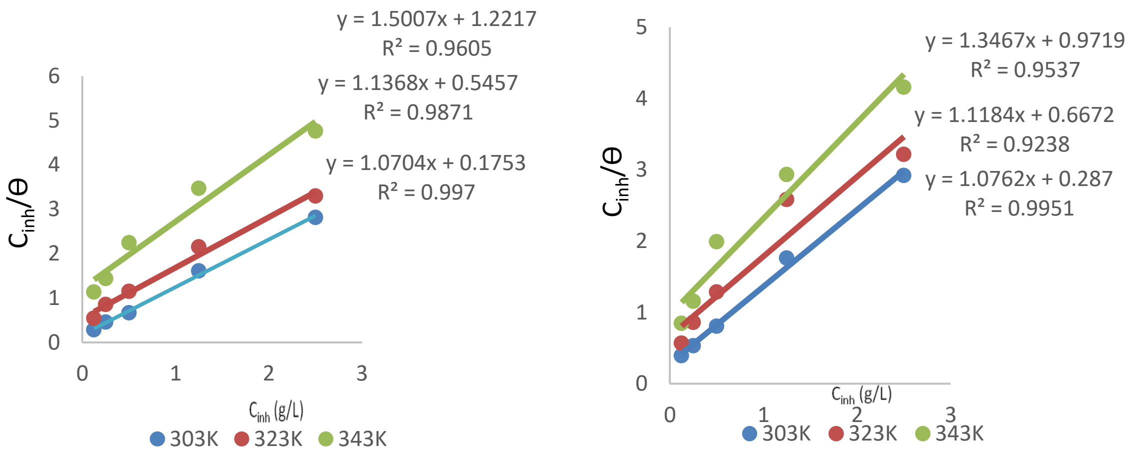


(a) (b)

Figure 4: Effect of temperature on the inhibition efficiency of *Ficus exasperata* (a) and *Costus afer* (b) in the acidic medium

Interpretation of adsorption isotherm plots and parameters

Figures 5a and b are the Langmuir adsorption isotherm plots for the adsorption of the extracts (*Ficus exasperata* and *Costus afer)* at various temperatures (303 K, 323 K and 343 K) in the acidic medium. The linear plots obtained is an indication that the adsorption of the extracts on the metal surface is consistent



(a) (b)

Figure 5: Langmuir adsorption isotherm plots for corrosion of mild steel in Hydrochloric acid solution in the presence of *Ficus exasperata* (a) and *Costus afer* (b) *extracts*

with the Langmuir adsorption isotherm model (Fadare *et al*., 2016). Values of Langmuir adsorption parameters are presented in Table 2 for each of the extracts (Ficus exasperata and *Costus afer)*. The coefficient of correlation (R^2) and the slopes obtained at each temperature for the extracts were close to unity. Therefore, the adsorption of the extracts (*Ficus exasperata* and *Costus afer*) on the metal surface strongly adheres to Langmuir adsorption isotherm model (Eddy *et al*., 2011). The adsorption equilibrium constant (K_{ads}) decreased with increase in temperature for each of the extracts. The value of the adsorption equilibrium constant (K_{ads}) obtained at 303 K for each extract is higher compared to the values obtained at 323 K and 343 K. This is an indication that the extracts were more efficient in corrosion inhibition at 303 K compared to the other temperatures (323 K and 343 K) *(*El-Awady *et al., 1993)*. The free energy of adsorption (ΔG^o_{ads}) obtained from Equation (5) are presented in Table 9. From the Table, the results were all negative. This implies that the adsorption of the extracts (Ficus exasperata and *Costus afer)* on the metal surface is a spontaneous process (Umoren *et al*., 2008; Noor, 2009; Ali *et al*., 2021). The calculated values of ΔG^o_{ads} are less than - 40 KJ/mol which is suggestive of physical adsorption (Fadare *et al*., 2015).

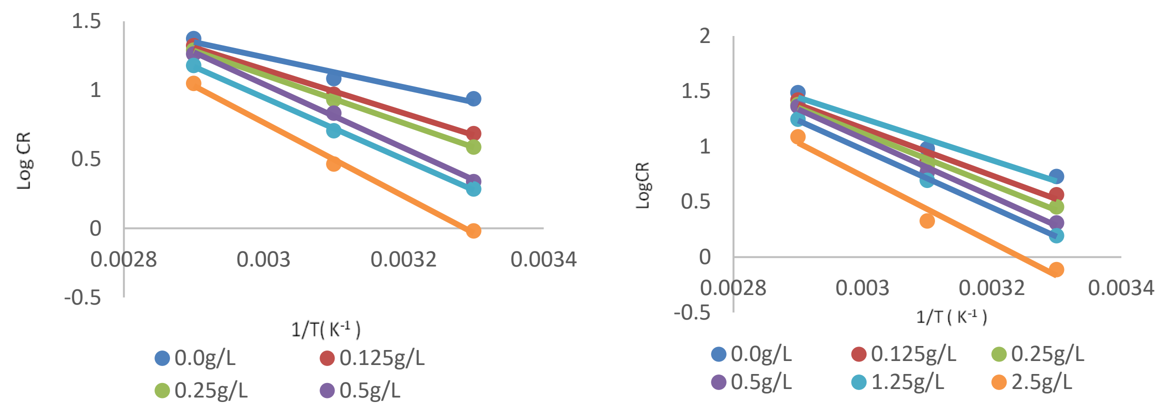
Interpretation of Thermodynamic Parameters

Presented in Figures 6a and b are the Arrhenius plots for the corrosion of mild steel in the absence and presence of the extracts (*Ficus exasperata* and *Costus afer*) in the acidic medium. Straight lines were obtained at all concentrations of the extracts. The values of the apparent activation energy (E_{app}) computed from the slopes of the plots are presentd in Table 3. The values of the apparent energy of activation (E_{app}) in the presence of various concentrations of the extracts (*Ficus exasperata* and *Costus afer*) were higher compared to the values obtained without extracts (0.0 g/L).

Table 2: Langmuir adsorption isotherm parameters for the adsorption of the extracts on the metal surface

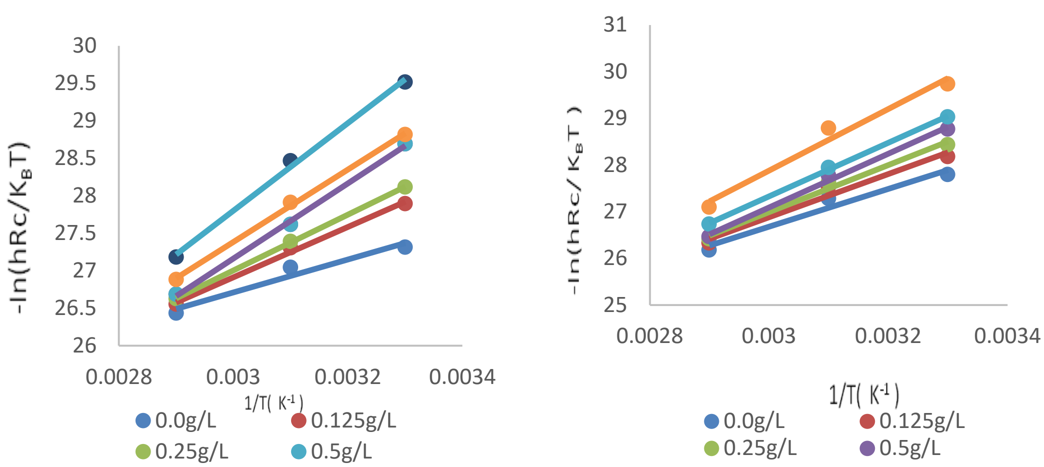
Extract	Temp (K)	Slope	K_{ads}	ΔG^o_{ads} (KJmol)	R^2
	303	1.0704	5.71	-21.79	0.997
Ficus exasperata	323	1.1368	1.83	-20.18	0.9871
	343	1.5007	0.82	-19.14	0.9605
	303	1.0762	3.48	-20.55	0.9951
Costus afer	323	1.1184	1.50	-19.64	0.9238
	343	1.3467	1.03	-19.79	0.9537

This is an indication that the extracts acted by decreasing the energy barrier for the corrosion of mild steel in hydrochloric acid (Hassan and Zaafarany, 2013). The increase in activation energy in the presence of the extracts signifies physical adsorption (Umoren *et al*., 2008; Undiandeye *et al*., 2011; Al-Amiery *et al*., 2014). Presented in Figures 7a and b are the Eyrings plots for the corrosion of mild steel in the absence and presence of the extracts.



(a) (b)

Figure 6: Arrhenius plots for the corrosion of mild steel in hydrochloric acid without and with different concentrations of *Ficus exasperata* (a) and *Costus afer* (b) extracts



(a) (b)

Figure 7: Eyrings plot for the corrosion of mild steel in 0.5 M hydrochloric acid solution in the absence and presences of Ficus exasperata (a) and *Costus afer* (b) extracts

Table 4 shows the values of enthalpy of activation ($\Delta H^{\neq}$) and entropy of activation ($\Delta S^{\neq}$) obtained from the graphs at various concentrations of the extracts. From the table, the values of ($\Delta H^{\neq}$) increased with increase in the concentration of the extracts. This is an indication that the energy barrier of the corrosion process increases with increase in the concentration of the extracts (Undiandeye *et al*., 2011). The positive values of ($\Delta H^{\neq}$) in the presence and absence of the extracts implies that the metal dissolution process is endothermic (Undiandeye *et al*., 2011; Hassan and Zaafarany, 2013). The negative values of $\Delta S^{\neq}$ in the presence and absence of the inhibitors means that the activated complex in the rate determining step represents an association instead of dissociation (Undiandeye *et al*., 2011; Fadare *et al*., 2015) meaning that there is more disorderliness in the solution without any extract (Undiandeye *et al*., 2011).

Table 3: Activation energy parameters for the corrosion of mild steel in hydrochloric acid in the absence and presence of the extracts

Extract	Concentration (g/L)	E_{app} (KJ/mol)
Ficus exasperata	0.0	20.84
	0.125	30.47
	0.25	33.57
	0.5	44.27
	1.25	42.77
	2.5	51.11
Costus afer	0.0	36.23
	0.125	41
	0.25	44.45
	0.5	50.39
	1.25	50.34
	2.5	57.45

Table 4: Activation parameters for the corrosion of mild steel in hydrochloric acid in the absence and presence of the extracts

Extract	Concentration (g/L)	$\Delta H^{\neq}$ (KJ/mol)	$\Delta S^{\neq}$ (J/mol)
Ficus exasperata	0.0	18.27	-167.27
	0.125	27.89	-140.1
	0.25	30.99	-131.56
	0.5	41.67	-100.83
	1.25	40.18	-107.17
	2.5	48.53	-85.56
Costus afer	0.0	33.65	-120.86
	0.125	38.41	-108.27
	0.25	41.86	-98.81
	0.5	47.81	-81.84
	1.25	47.74	-83.97
	2.5	54.87	-67.17

CONCLUSION

Ficus exasperata (FE) and Costus afer (CA) extracts could serve as effective green corrosion inhibitors for mild steel in hydrochloric acid medium. The inhibition efficiencies of the plant materials decreased with increased temperature. The extracts acted by physically adsorbing onto the metal surface. The adsorption of the extracts (*Ficus exasperata (FE) and Costus afer (CA*)) on the metal surface obeyed Langmuir adsorption isotherm.

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