



# Unveiling the adsorption characteristics and corrosion inhibitory effect of *Justicia carnea* leaf extract on carbon steel in a simulated acidic medium – Mass Loss Approach and AFM Characterizations

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**Abstract:** This study focused on the adsorptive and corrosion inhibitory behaviours of *Justicia carnea* (JC) leaf extract on low-carbon steel immersed in a 0.5 M H<sub>2</sub>SO<sub>4</sub> solution. Hot extraction by reflux distillation was deployed using methanol. Solvent was removed after filtration using a rotary evaporator under vacuum conditions. Phytochemical analyses were performed on the crude extract using the Trease-Evans and Pandey-Tripathi methods. Corrosion inhibitory effect of the extract was investigated using mass loss method. Phytochemicals detected are alkaloids, glycosides, flavonoids, steroids and terpenoids. Corrosion inhibitory efficiency of JC crude extract was 91 – over 99 % at 300 K and 87 – over 96 % at 333 K for 0.4 – 2.0 g L<sup>-1</sup> concentration range at both temperatures, and the efficacy of the extract was found to be a function of extract dosage/concentration and temperature to the effect that at a fixed temperature, inhibitor efficiency was directly proportional to extract dosage/concentration, while at constant extract dosage/concentration, the inhibitor efficiency depreciated with rise in temperature. Adsorption studies and isotherm model fittings revealed that best approximation matched with Langmuir adsorption isotherm with R<sup>2</sup> = 0.99991 and 0.99894 at 300 K and 333 K respectively. The fitting isotherm parameters.

## 1. Introduction

Adsorption is a phenomenon through which a substance in a liquid phase accumulates and adheres on a solid surface and is then removed from the liquid phase. In this phenomenon, the substance that is removed from the fluid bulk is called the adsorbate and the solid substrate on which the substance settles or adheres (adsorbs) is known as the adsorbent (Abin-Bazzaine *et al.*, 2022; Tonk *et al.*, 2022; Mihit *et al.*, 2006). Adsorption as surface phenomenon arises as a result of unbalanced forces on the surface of solids and liquids and this makes the surface to be under tension. The surface of the solid or liquid, when brought in contact with other substances, tends to satisfy their residual forces by attracting the molecules of those substances and retaining them (Chakrabarty, 2001; Davis, 2012; Erhayem1 *et al.*, 2015). Two modes of adsorption are known, namely physical adsorption (or physisorption) and chemical adsorption (or chemisorption). In physisorption, the adsorbate molecules are accumulated on

the solid (substrate) surface due to weak forces, known as Van der Waals forces. In chemisorption, the adsorbate molecules accumulate on the substrate and are held by chemical bonds (Chakrabarty, 2001; Davis, 2012; Erhayem *et al.*, 2015).

The adsorption characteristics of most dyes, pigments and secondary metabolites extracted from plant materials have been harnessed in the making of organic inhibitors to impede the impact of corrosion on metallic and alloy-metal substrates (Madkour *et al.*, 2016; Peme, *et al.*, 2015; Fouda *et al.*, 2014; Hmamou *et al.*, 2012; Kabanda *et al.*, 2012; Abboud *et al.*, 2012), and also in textile industries for the dyeing of fabrics (Akpuaka *et al.*, 1998). Lately, the use of chemical inhibitors such as chromates, silicates and organic amines has come under severe criticism due to the health hazards and environmental pitfalls associated with their use as a result of the presence in them of heavy metals and other toxic compounds. Attention, therefore, has been turned towards the use of natural products (secondary metabolites from botanical extracts) because they are not only non-toxic, benign and biodegradable, but are also abundantly available, renewable and cost effective. Plant extracts have been found to be rich in naturally synthesized chemical compounds, some with complex molecular structures and having different functional groups containing the heteroatoms (N, O or S), aromatic rings and conjugated  $\pi$ -electronic systems which make these natural products excellent candidates for corrosion inhibition (Elmsellem *et al.*, 2014; Madkour *et al.*, 2016; Peme *et al.*, 2016; Ahanotu *et al.*, 2022). Most of these natural products have been used by many researchers as corrosion inhibitors for different metals in various hostile environments and optimum performance were reported for all of them (Onukwube *et al.*, 2016).

Some prominent research scholars have reported the successful use of plant extracts as potent and sustainable corrosion suppressants to inhibit the dissolution of metals and different grades of alloy metals in aggressive environments such as acid, alkaline and salt solutions (Ahanotu *et al.*, 2022; Ahanotu *et al.*, 2020; Obot *et al.*, 2020; Obot *et al.*, 2011; Zheng *et al.*, 2017; Sivakumar *et al.*, 2018; Njoku *et al.*, 2016., Fouda *et al.*, 2014; Akalezi *et al.*, 2012; Laaroussi *et al.*, 2022; Bouammali *et al.*, 2013). Palumbo *et al.* (2023), for instance, studied the corrosion inhibitory effect of a natural polysaccharide guar gum on N80 carbon steel under CO<sub>2</sub>-saturated saline solution at different temperatures and immersion time. Their study was conducted using mass loss method and electrochemical approaches, in addition to surface characterization and spectroscopic methods. The results obtained revealed that guar gum exhibited good inhibition performance at lower and higher temperatures, with efficiency of 76.16% at 25 °C and 63.19% at 50 °C at 0.4 g/L dosage of the inhibitor through mass loss measurements. The results from the electrochemical measurements revealed that guar gum was able to sustain its efficacy even at longer immersion time. The adsorption insights and thermodynamic considerations showed that guar gum exhibited a mixed-type of adsorption involving physical and chemical modes (Palumbo *et al.*, 2023). Considerable research studies are being undertaken around the world on the extraction and applications of novel plant extracts as promising and sustainable alternatives to synthetic compounds for fighting against the deterioration of metallic substrates. A host of other similar studies exist in literature where plant extracts are employed as corrosion inhibitors for protecting different grades of steel and metals from attack by acidic, alkaline and salt solutions. Till date, there is no documented scientific information or publication on the application of crude leaf extract of *Justicia carnea* against the corrosion of carbon steel or any other substrate in a corrosive medium. *Justicia carnea* (commonly known as Brazilian plume flower, flamingo flower or jacobinia) is an important medicinal plant species in the acanthaceae family (USDA, 2015; Correa & Alcântara, 2012; Wasshausen & Wood, 2004) which have continually been used in traditional medicine by local residents in the Southeastern region of Nigeria for boosting blood level

and ameliorating other health conditions (Otuokere *et al.*, 2016; Bajpai *et al.*, 2005). This study seeks to explore the leaf extract of this plant to unveil its adsorption characteristics and corrosion inhibitory effect for preserving carbon steel substrates from corrosive attacks.

## 2. Experimental Methodology

### 2.1 Collection and preparation of plant samples and carbon steel substrates

Matured leaves of *Justicia carnea* were locally harvested from the live plants at Ihitte Village in Afara Autonomous Community in Mbaitoli L.G.A of Imo State, Nigeria. The leaves were botanically identified by a Plant Scientist, washed thoroughly, air-dried and ground to fine powder of 2,620 g weight which was preserved in an air-tight opaque container. Low carbon steel sheets of known chemical composition were procured from the Materials and Metallurgical Engineering Workshop, Federal University of Technology, Owerri, Imo State and mechanically cut into test coupons of dimensions 30 mm (length) × 20 mm (breadth) × 1.5 mm (thickness). An open cylindrical hole of diameter 3 mm was drilled in each coupon as a provision for free suspension of the coupons inside the hostile corrosive solution using a polymeric thread. With iron as the chief element, the elemental composition of the carbon steel has been reported elsewhere (Puspitasari *et al.*, 2017).

### 2.2 Determination of moisture content

The leaf powder sample (2.0 g) was subjected to moisture content determination by heating in a vacuum oven (Servewell Instruments Vacuum oven, Model: VO124) to a temperature of 105 °C for 3 hours and weighing repeatedly until it becomes completely dry and no more weight loss was observed. The loss-on-drying was used to calculate the percentage of moisture in the sample according to the following formula:

$$\%LOD = \frac{\text{Initial weight of sample} - \text{Final weight of sample}}{\text{Initial weight of sample}} \times 100 \quad (1)$$

### 2.3 Extraction

The finely ground leaf sample (2,400 g) was subjected to hot extraction under reflux in a multi-necked round bottom flask equipped with water bath in two batches. In the first batch, the finely ground *Justicia carnea* leaves of mass 600 g was put into the three-necked 5-litre round bottom flask and 3 litres of methanol was added. The content was then refluxed at temperature of 60–65°C for 3 hours. Thereafter, the refluxed mixture was cooled and filtered by using Whatman filter paper under vacuum condition. Further, the leaf residue was extracted twice with methanol under the same condition to ensure optimum extraction of the molecules. In the second batch of the extraction process, finely ground *Justicia carnea* leaves of mass 1800 g was put into a three-necked 20-litre round bottom flask equipped with water bath and 9 litres of methanol was added. The content was again refluxed at temperature of 60–65°C for 3 hours. Thereafter, the refluxed mixture was cooled and filtered by using Whatman filter paper under vacuum condition. Again, the leaf residue was extracted twice with methanol under the same condition to ensure optimum extraction of the molecules. All six filtrates were combined and on concentration under reduced pressure in a rotatory evaporator (Buchi, Switzerland; Model no: R-220 Pro equipped with vacuum pump V-600) at 50°C, a completely dried green paste (slurry) was obtained and percentage yield was determined.

### 2.4 Preliminary phytochemical screening of the crude extract

The preliminary qualitative phytochemical screening of the crude methanol extract was done in accordance to standard methods adopted by Trease & Evans (1989) and Pandey & Tripathi (2014).

#### *Detection of alkaloids*

*Mayer's test:* The slurry extract (1.0 g) was dissolved in 10 ml dilute hydrochloric acid and filtered. A 1 ml aliquot of the solution of the extract was added in separate test tube containing 1 ml of potassium mercuric iodide solution (Mayer's reagent). A cream (yellow) precipitate formation was observed which indicated the presence of alkaloids.

*Dragendorff's test:* About 1 mL of the extract solution was taken and placed into a test tube. Then 1 mL of Dragendorff's reagent (potassium bismuth iodide solution) was added and the solution shaken. Appearance of orange red precipitate indicated the presence of alkaloids.

*Wagner's test:* About 1 mL of extract solution was put into a test tube and 1 mL of potassium iodide (Wagner's reagent) was added and the solution shaken. Appearance of reddish brown precipitate signified the existence of alkaloids.

#### *Detection of flavonoids*

The test for flavonoids was carried out using sulfuric acid test. A few drops of concentrated sulfuric acid was added to 1 ml of the extract solution, and the formation of orange colour was observed which indicates the presence of flavonoids.

#### *Detection of steroids*

The Liebermann-Burchard test was adopted in the screening for steroids. About 0.5 g of extract was dissolved in 2 ml chloroform and filtered. Then 3 drops of acetic anhydride and 2 mL of concentrated sulfuric acid were added to 1 ml solution of the extract in chloroform. The development of blue-green coloration indicated the presence of steroids.

#### *Detection of terpenoids*

The test for terpenoids was carried out using the Salkowski's test. Exactly 5 mg extract was mixed with 2 ml chloroform in a test tube and 3 ml concentrated sulfuric acid was added carefully to form a layer. A reddish brown colour was observed which indicated the presence of terpenoids.

#### *Detection of saponins*

The screening for saponins was carried out using the frothing test. Exactly 1 mg of extract was mixed with 10 mL of distilled water and shaken vigorously. There was no formation of frothing and this was an indication that saponins were absent.

#### *Detection of glycosides*

Exactly 5 mg of extract was dissolved in 5 mL of methanol, put on TLC plate, dipped in methanol-sulphuric acid reagent and kept on hot plate. Charring was observed which indicated the presence of glycosides.

### **2.5 Preparation of hostile solution and corrosion inhibitor test solutions**

Sulfuric acid solution (0.5 M) was used as the corrosive medium and was prepared from concentrated acid of 98% certified percent purity, specific gravity of 1.84 and molar mass of 98.097 gmol<sup>-1</sup> in accordance to the procedure reported elsewhere (Ahanotu *et al.*, 2022). Five inhibitor test solutions were prepared from the methanol (crude) extract of *Justicia carnea*. The inhibitor test solutions were prepared to have increasing order of concentration in the range 0.4 – 2.0 g L<sup>-1</sup> with increment of 0.4 g L<sup>-1</sup> per step, using the 0.5 M solution of sulfuric acid as the solvent in each case.

## 2.6 Classical mass loss experiments

The determination of corrosion inhibitory tendencies of the two crude plant extracts were conducted *via* the classical mass loss experiments as described elsewhere (Ahanotu *et al.*, 2022) using different concentrations (dosages) of the *Justicia carnea*-based inhibitor (crude leaf extract) and at two different temperatures. With the use of silicon carbide abrasive papers (from grade #400 - #1000), the carbon steel specimens were polished by abrasion, washed in absolute ethanol to get rid of oil and grease particles, dried with acetone and warm air, weighed and saved in a moisture-free desiccator prior to use. To achieve aerated condition in the corrosion cells, six plastic containers labeled A – F with air-tight lids were used as corrosion cells. The hostile acid corrodent (150 ml) were poured into the containers, followed by the introduction of the serial inhibitor test solutions, starting with Cell B which was allowed to contain the least inhibitor dosage (0.4 g L<sup>-1</sup> of extract) while Cell F contains highest inhibitor dosage (2.0 g L<sup>-1</sup>). Cell A contained no inhibitor additive and therefore served as the control (blank). The carbon steel specimens were then fully immersed into the contents of each cell and the temperature was maintained at 27 °C (300 K) using a six-hole thermostat water bath (Wincom thermostat water bath, Model no. HH-6). All the cells were corked and allowed to stay undisturbed for 9 hours, with periodic retrieval of the specimens from the hostile solution at 3-hour intervals. At each retrieval, the specimens were cleaned by washing and rinsing in absolute ethanol, and drying with acetone and warm air. This was done to remove corrosion particles. The mass loss ( $\Delta m$ ) was taken to be the difference between the initial mass before total immersion and the final constant mass after washing and rinsing. The experiments were repeated by setting and maintaining temperature at 60 °C (333 K) using a six-hole thermostat water bath. This was to evaluate the effect of temperature variation (Ekpe *et al.*, 1995; Umoren *et al.* 2008). The mass loss ( $\Delta m$ ) by each carbon steel specimen after 9 hours of total immersion in the sulfuric acid solution without and with the crude plant extracts (inhibitors) in different concentration was converted to corrosion rate ( $R$ ), degree of surface coverage ( $\theta$ ) and inhibitor efficiency ( $\eta\%$ ) of the molecules (plant extracts) using Equations (2), (3) and (4) respectively (Ahanotu *et al.*, 2022).

$$R = \frac{K\Delta m}{At\rho} \quad (2)$$

$$\theta = \left[ \frac{R_b - R_i}{R_b} \right] \quad (3)$$

$$\eta\% = \theta \times 100 \quad (4)$$

where  $K$  is a constant with value  $8.76 \times 10^4$ . ( $K = 87,658.1277$  millimeters per year);  $\Delta m$  is the mass loss in  $g$  of the carbon steel specimen at the end of test,  $A$  is the total surface area of one test specimen to the nearest  $0.01 \text{ cm}^2$ ,  $t$  is the time or duration of immersion in hour,  $\rho$  is the density of the carbon steel specimen in  $g \text{ cm}^{-3}$ ,  $R_b$  is the corrosion rate in the blank cell (absence of the inhibiting molecules) and  $R_i$  is the corrosion rate in the inhibited cell (presence of the inhibiting molecules).

## 2.7 Adsorption insights into the corrosion inhibitory effect of extract

The mode of adsorption of the crude leaf extracts of *Justicia carnea* crude leaf extract on the surface of the carbon steels was investigated by approximating the experimental data with adsorption isotherm models. Four different adsorption isotherm models (Equations 5–8) were tested for approximation.

Langmuir: 
$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (5)$$

$$\text{Freundlich:} \quad \ln \theta = \ln K_{ads} + \ln C_{inh} \quad (6)$$

$$\text{Temkin:} \quad \theta = \frac{\ln K_{ads}}{2a} + \frac{\ln C_{inh}}{2a} \quad (7)$$

$$\text{Frumkin:} \quad \ln \left[ \frac{\theta}{(1-\theta)C_{inh}} \right] = \ln(b) - \frac{2\omega}{RT} \theta \quad (8)$$

where  $K_{ads}$  is a constant known as the adsorption equilibrium constant and it is deduced as the reciprocal of the intercept ( $I$ ) from the isotherm plots,  $C_{inh}$  is the concentration (dosage) of the inhibitor,  $\theta$  is equilibrium adsorbate phase concentration which is equal to the degree of surface coverage, and  $a$  is adsorbate interaction factor,  $R$  is the universal gas constant and  $T$  is the absolute temperature (Solomon *et al.*, 2010; Singh *et al.*, 2023). For Langmuir isotherm model,  $C/\theta$  was plotted against  $C$ , while  $\ln \theta$  vs.  $\ln C$  was plotted for Freundlich isotherm model,  $\theta$  vs.  $\ln C$  for Temkin isotherm model and  $\ln \left[ \frac{\theta}{(1-\theta)C} \right]$  vs.  $\theta$  for Frumkin isotherm model.

## 2.8 Surface observation of carbon steel specimens

The Stromlinet nano atomic force microscope (AFM) was deployed to capture the images of the surfaces of the carbon steel substrates in order to support the gravimetric (mass loss) findings. Each carbon steel specimen was installed into the sample stage of the AFM instrument using a pair of forceps. The scanning probe (with a sharp tip) attached on the free-swinging end of a cantilever was aligned and the laser beam was focused on the probe with a photodetector. The specimen was then scanned. The interaction between the surface of the specimen and the tip of the scanning probe causes the cantilever to deflect and the microscope measures this interaction at many points on the specimen's surface. The detector and feedback electronics of the instrument makes a spatial map of the interaction and generates an image of the surface with resolution in the order of fractions of a nanometer.

## 3. Results and Discussion

### 3.1 Results of moisture content

The moisture content of the samples based on loss-on-drying are presented in Table 1.

**Table 1.** Moisture content of sample

Weight Before Drying (g)	Weight After Drying (g)	Loss-on-Drying (g)	Moisture Content (% w/w)
2.0	1.89	0.110	5.5

### 3.2 Percentage yield of extract

The masses and percentage yield of crude methanol extract after complete drying are presented in Table 2 below.

**Table 2.** Mass and percentage yield of crude methanol leaf extract of *Justicia carnea*

Weight of Raw Leaf Powder (g)	Methanol Extract	
	Yield (g)	Percent Yield (% w/w)
2400	209.00	9.0 (green colour paste)

### 3.3 Phytochemical constituents of the crude methanol extract

The results of the preliminary phytochemical screening of the crude extracts are presented in Table 3.

**Table 3.** Phytochemical constituents of crude methanol extracts from leaves of *Justicia carnea*

Phytoconstituents	Status	Inferred Prevalent Functional Groups
Alkaloids	++	NH <sub>2</sub> , NHR, NR <sub>2</sub>
Glycosides	++	OH, -O-, aromatic rings
Flavonoids	+	OH
Steroids	+++	OH, C=C
Terpenoids	+	OH, C=C
Total	<b>5</b>	

**Key:** (+++) = highly present, (++) = moderately present, (+) = present in traces, (-) = absent

The table reveals the presence of alkaloids, glycosides, flavonoids, steroids and terpenoids in the leaves of *Justicia carnea*. Other phytochemicals were not detected in the extract. A plethora of similar chemical compounds have also been found in other *Justicia* species, especially alkaloids, flavonoid, steroids, terpenoids and lignans (Corrêa & Alcântara, 2012). A study by Rattan (2023) also showed that the prevalent phytochemicals in Acanthaceae plants are flavonoids, alkaloids, lignans, saponins, triterpenoids, steroids and fatty acids. This can be further buttressed with the reports by Aziz *et al.* (2022) which reveal that *Justicia carnea* contain the same active compounds like other acanthaceae plants among which are lupeol,  $\beta$ -amyrin, stigmaterol,  $\gamma$ -sitosterol, 9,12-octadecadienoic acid (linoleic acid), n-hexadecanoic acid, It is, therefore, evident from the results in Table 3 that *Justicia carnea* exhibit very similar phytochemical profiles with other plants in the acanthaceae family and this transcends to the same active chemical compounds. The phytochemicals found in the extracts have been reported to possess many therapeutic, nutraceutical and cosmetic values (Khoo *et al.*, 2017; Rizwanullah *et al.*, 2017; Fatima *et al.*, 2021; Zhao *et al.*, 2022; Ahmed *et al.*, 2022; Yusuf *et al.*, 2017; Bommakanti *et al.*, 2023; Sut *et al.*, 2022; Hassan *et al.*, 2021).

### 3.4 Variation of mass of carbon steel with time of immersion

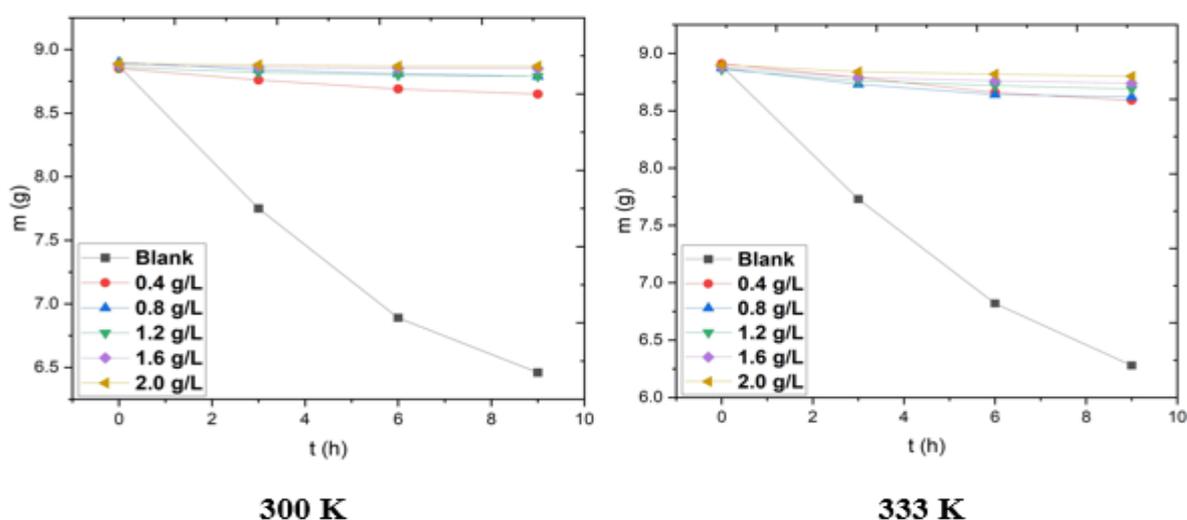
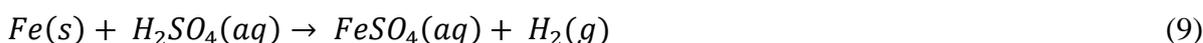
The variation of the mass of the carbon steel test coupons with time of immersion in the hostile corrodent without and with different dosages of crude methanol extract of *Justicia carnea* at 300 K and 333 K for 9 hour immersion is presented in Table 4.

**Table 4.** Variation of mass of low carbon steel with time of immersion in 0.5 M H<sub>2</sub>SO<sub>4</sub> solution

Temperature (K)	Cell ID	Extract Conc. (g L <sup>-1</sup> )	Varying Mass of Specimens				$\Delta m$
			$m_0$	$m_1$	$m_2$	$m_3$	
300	A	0	8.87	7.75	6.89	6.46	2.41
	B	0.4	8.85	8.76	8.69	8.65	0.20
	C	0.8	8.90	8.84	8.81	8.79	0.11
	D	1.2	8.86	8.82	8.80	8.79	0.07
	E	1.6	8.88	8.86	8.85	8.85	0.03
	F	2.0	8.89	8.88	8.87	8.87	0.02
333	A	0	8.89	7.73	6.82	6.28	2.61
	B	0.4	8.91	8.79	8.66	8.59	0.32
	C	0.8	8.87	8.73	8.64	8.62	0.25
	D	1.2	8.86	8.76	8.72	8.69	0.17
	E	1.6	8.88	8.79	8.76	8.73	0.15
	F	2.0	8.90	8.84	8.82	8.80	0.10

**Key:**  $m_0$  = original mass,  $m_1$  = mass after 3 hrs,  $m_2$  = mass after 6 hrs and  $m_3$  = mass after 9 hrs

In Table 4,  $\Delta m = m_0 - m_3$  and it represents the mass lost by the corroding carbon steel specimens after 9 hours of immersion of the specimens in the acid solution. It can be deduced from the table that the crude methanol extracts of the *Justicia carnea* leaves (JCL) deployed as anticorrosion additives (corrosion inhibitors) for protection of the carbon steel specimens experienced gradual loss in mass as the immersion time progressed. As the carbon steel test specimens were corroding (dissolving) in the hostile solution, gas bubbles were observed being generated on the surface of the specimens and accumulating in the solution of the acid solution. An explanation to this observation can be offered from the knowledge of basic chemistry, to the effect that when a reactive metal reacts with an acid solution, hydrogen gas is evolved. Iron (Fe) as the chief element in carbon steel (Puspitasari *et al.*, 2017) was evidently reacting with sulfuric acid solution (corrodent) and by so doing, hydrogen gas was being liberated as bubbles according to the following equation.



**Figure 1.** Variation of mass of low carbon steel with time of immersion

The data in Table 4 have been presented graphically in Figure 1. A cursory look at the curves for inhibited systems will reveal that the curves are almost horizontally flat after 6 hour immersion time. The flattening of the curves is a clear indication that the mass change in the carbon steel specimens has become so negligible that their masses are becoming constant as the immersion time progresses. This could be due to the stability of the inhibitor layer adsorbed on the steel surfaces. A simple extrapolation of the curves will give an insight to the maximum length of immersion time of immersion at which no mass lost is recorded in the carbon steel specimens. Such extrapolation could lead to the prediction of the optimum concentration of the inhibitor at which no appreciable reduction of mass of the test coupons is recorded.

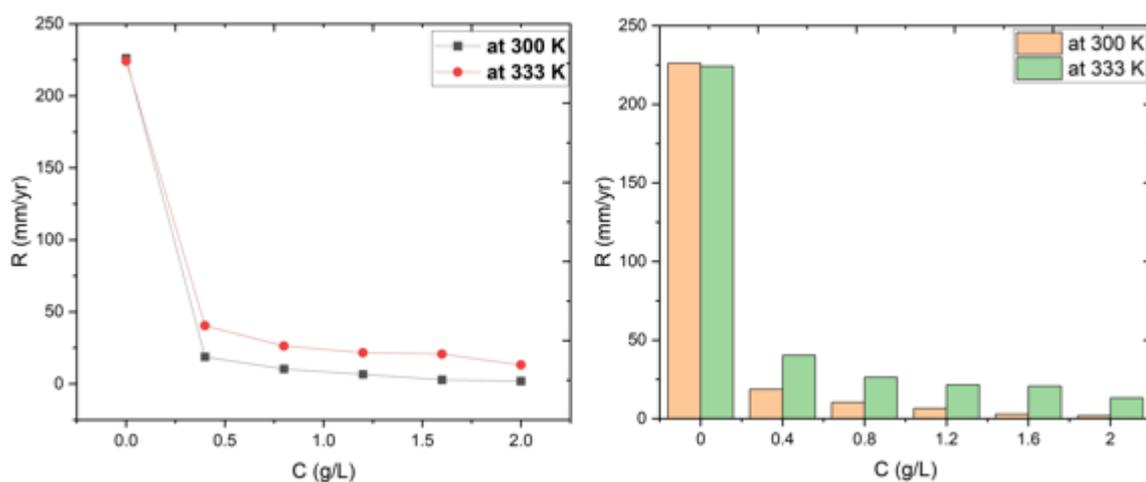
### 3.5 Effect of extract dosage/concentration on corrosion rate and inhibitor efficiency

The overall mass loss, corrosion rate, inhibitor efficiency and degree of surface coverage for low carbon steel immersed in the acid corrodent without and with different dosages of the JCL crude methanol extract at 300 K and 333 K for 9 hour immersion are presented in Table 5. The degree of surface coverage ( $\theta$ ) is an indication of the fraction of the exposed total surface area of the carbon steel that is covered by the inhibitor molecules, while the inhibitor efficiency ( $\eta\%$ ) is a measure of the protective performance of the inhibitor system for the metallic substrates.

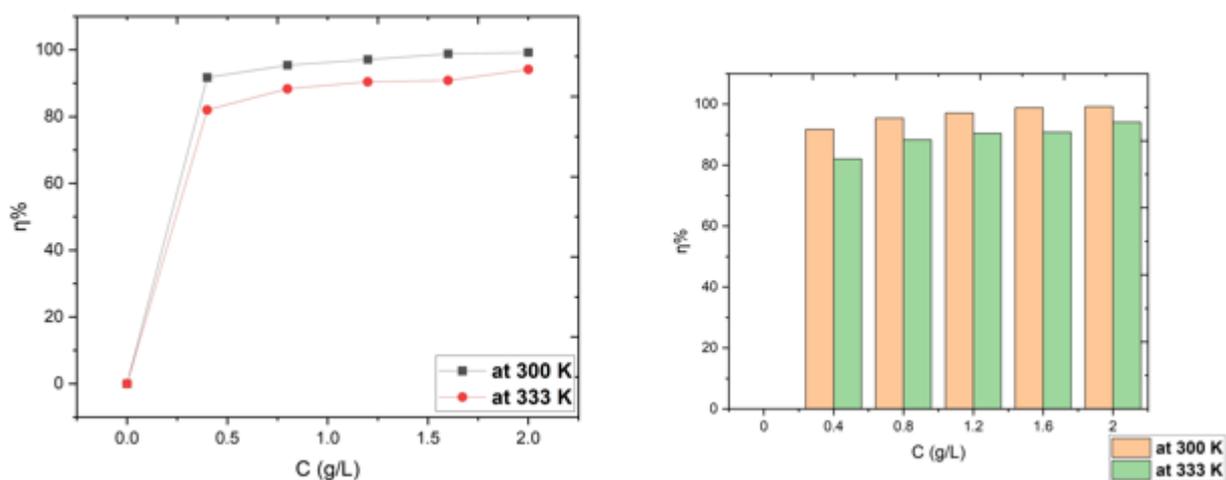
**Table 5.** Overall mass loss, corrosion rate, inhibitor efficiency and degree of surface coverage for low carbon steel immersed in 0.5 M H<sub>2</sub>SO<sub>4</sub> solution

Temperature (K)	Cell ID	Extract Conc. (g L <sup>-1</sup> )	$\Delta m$	R (mm/yr)	$\theta$	$\eta\%$
300	A	0	2.41	226.09	–	–
	B	0.4	0.20	18.76	0.917	91.7
	C	0.8	0.11	10.32	0.954	95.4
	D	1.2	0.07	6.57	0.971	97.1
	E	1.6	0.03	2.81	0.988	98.8
	F	2.0	0.02	1.88	0.992	99.2
333	A	0	2.61	244.85	–	–
	B	0.4	0.32	30.02	0.877	87.7
	C	0.8	0.25	23.45	0.904	90.4
	D	1.2	0.17	15.95	0.935	93.5
	E	1.6	0.15	14.07	0.943	94.3
	F	2.0	0.10	9.38	0.962	96.2

From Table 5, it is clearly observed that both the corrosion rate of the test specimens and efficacy of the inhibitor packages are dependent on the dosage/concentration of the extracts. The corrosion rate depreciated at higher dosage of the extract while the inhibitor efficiency appreciated at higher extract dosage/concentration. Data in Table 5 show that a minimum 0.4 g L<sup>-1</sup> of JCL-based inhibitor clamped down the corrosion from 226.09 mm/yr to 18.76 mm/y at 300 K and from 244.85 mm/yr to 30.02 mm/y at 333 K, thereby producing efficiency of 91.7% and 87.7% respectively at the two temperatures tested. The inhibitor efficiency appreciated with increasing dosage/concentration of the extract and at 2.0 g L<sup>-1</sup>, inhibitor efficiency of 99.2% and 96.2% respectively was recorded at 300 K and 333 K respectively. The implication of this result is that the phytochemical constituents of the extract constituting the inhibiting molecules got adsorbed on the surface of the test specimens thereby creating a protective layer that hinders further attack of the acid solution on the carbon steel (Ahanotu *et al.*, 2022). The variation of corrosion rate with extract concentration is graphically illustrated using the data from Table 5 and presented in Figure 2 while the variation of inhibitor efficiency with extract concentration is illustrated in Figure 3.



**Figure 2.** Variation of corrosion rate (R) with extract concentration (C) at 300 K and 333 K



**Figure 3.** Variation of inhibitor efficiency ( $\eta\%$ ) with extract concentration (C) at 300 K and 333 K

A very steep negative slope can be observed in the graph in Figure 2 which corresponds to the tallest pair of bars in the corresponding bar chart. This represents a sharp decline in the rate of corrosion from 226.09 mm/yr to 18.76 mm/yr and 244.85 mm/yr to 30.02 mm/yr after addition of only 0.4 g/L dosage of JCL-based inhibitor at 300 K and 333 K respectively. Thus, it can be inferred that a minimum 0.4 g/L of each extract was effective in suppressing the dissolution (corrosion) of the carbon steel specimens in the acid medium. The corrosion rate continued to decline steadily with increasing extract concentration but increased at higher temperature of the hostile solution for a given inhibitor dosage. These trends also explain the shape of the curve in Figure 3 where the steep positive slope represents a sharp rise in inhibitor efficiency after the addition of 0.4 g/L of the extract. The infinitesimally small slopes along the near-horizontal portions of the graphs in Figures 2 indicate that the corrosion rate is approaching the minimal value and will eventually become negligible if the concentration of the extract is progressively increased. This also transcends to the inhibitor efficiency approaching its optimal value as observed in Figure 3. The results from these experiments are in sufficient agreement with what previous research scholars have reported among whom are Ahanotu *et al.* (2020), Ahanotu *et al.* (2022), Obot *et al.* (2020), Mabrouk *et al.* (2017, Njoku *et al.*, 2016) and a host of others.

### 3.6 Effect of temperature elevation on corrosion rate and inhibitor efficiency

A careful look at the data in Table 5 could reveal that the efficacy of the JCL-based inhibitor is temperature-dependent for a given extract concentration to the effect that as the temperature was raised from 27°C (300 K) to 60°C (333 K), the inhibitor efficacy ( $\eta\%$ ) depreciated at each specific extract concentration and this is indicative that the JCL inhibiting molecules physically adsorbed on the surface of the test specimens (Ahanotu *et al.*, 2022; Messali *et al.*, 2017; Palumbo *et al.*, 2019). It is pertinent to note that after addition of a minimum 0.4 g L<sup>-1</sup> dosage at 300 K, the inhibiting molecules of JCL have already attained stability and produced efficiency of 91.7% and even at the higher temperature, the inhibitor efficiency of the extract continued to appreciate with higher extract concentration. It can therefore be stated that at a constant temperature, the inhibitor performance appreciated with increasing inhibitor concentration, while for a constant concentration value, higher temperature depreciates the inhibitor performance. Recall, that corrosion is an electrochemical reaction. At elevated temperatures, electrochemical reactions occur at a faster rate and as a result, the corrosion rate increases, and *vice versa*. Temperature has been found to control the rate of diffusion reactants to and from the substrate surface, and hence diffusion in turn affects corrosion rate (Wasim *et al.*, 2018). A bare steel surface

freshly exposed to a hostile medium has the tendency to corrode at a faster rate than those covered with a compact protective layer. The corrosion rate is also heavily controlled by the diffusion of oxygen through the solution to the steel surface to the effect that corrosion rate is higher in areas where oxygen diffusion is prevalent. For these reasons, the corrosion of metals and alloy metals such as steel occur at faster rates at elevated temperatures than at lower temperatures (Wasim *et al.*, 2018). This accounts for the loss in efficiency of the inhibitor packages at elevated temperature for each given extract concentration.

### 3.7 Adsorption insights into the anticorrosion effect of JCL crude extract

A graph of the relevant coordinates plotted for the adsorption isotherm approximations reveal that the best approximation was obtained with the Langmuir isotherm at both temperatures and this is illustrated in Figure 4. The deduced isotherm parameters from the Langmuir isotherm model for the 9-hour immersion of carbon steel substrates in 0.5 M sulfuric acid are presented in Table 6.

**Table 6.** Adsorption and thermodynamic parameters for carbon steel using JCL-based inhibitor at 300 K and 333 K

Temp. (K)	$R^2$	Slope	Intercept	$K_{ads}$
300	0.99991	0.985	0.0472	21.186
333	0.99954	1.0145	0.0626	15.974

The free energy of adsorption ( $\Delta G_{ads}^0$ ) was deduced from the equilibrium constant of adsorption,  $K_{ads}$ , according to the following mathematical expression.

$$\Delta G_{ads}^0 = -RT \ln (55.5 K_{ads}) \quad (10)$$

where  $R$  is the universal gas constant ( $8.314 \text{ J K}^{-1} \text{ mol}^{-1}$ ),  $T$  is the absolute temperature,  $K_{ads}$  is the adsorption equilibrium constant, and 55.5 is a constant equivalent to the concentration of water in solution in mol/L.

It's safe that the use of more than one compounds as in synergistic effect of two compounds or various components at different contents which is the case of extract of natural plant, to not evaluate free enthalpy as discussed in various studies (Lrhoul *et al.*, 2023, Ahanotu *et al.*, 2022; Bouknana *et al.*, 2014; Umoren *et al.*, 2006).

The calculation of apparent activation energy ( $E_a$ ) and heat of adsorption ( $Q_{ads}$ ) in the temperature range of 300 K – 333 K for JCL-based inhibitory additive relied on the dependence of corrosion rate on temperature as expressed by Equation 11 (the condensed Arrhenius equation) and Equation 12.

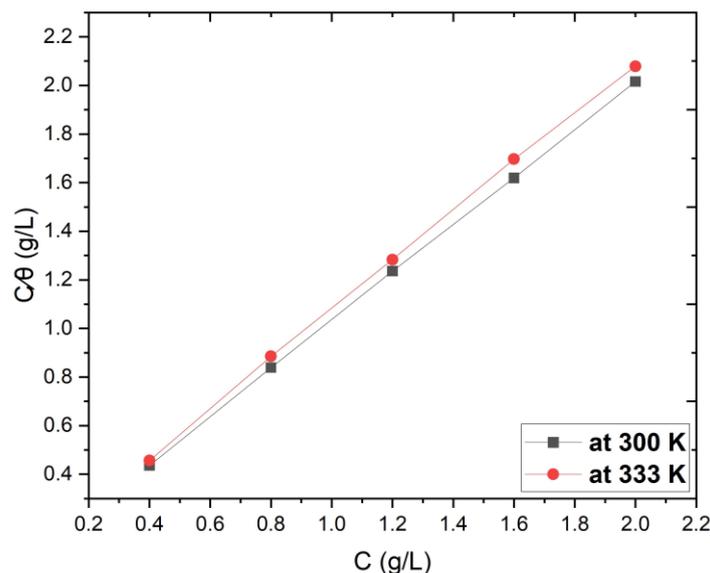
$$\text{Log} \frac{R_2}{R_1} = \frac{E_a}{2.303 R} \left[ \frac{T_2 - T_1}{T_1 T_2} \right] \quad (11)$$

where  $R_1$  and  $R_2$  are the corrosion rates at absolute temperatures  $T_1$  (300 K) and  $T_2$  (333 K) respectively, and  $R$  is the universal gas constant.

$$Q_{ads} = 2.303R \left[ \text{Log} \left( \frac{\theta_2}{1-\theta_2} \right) - \text{Log} \left( \frac{\theta_1}{1-\theta_1} \right) \right] \times \left( \frac{T_1 \times T_2}{T_2 - T_1} \right) \quad (12)$$

where  $\theta_1$  and  $\theta_2$  are the values of the degree of surface coverage at the tested temperatures  $T_1$  (300 K) and  $T_2$  (333 K) respectively.

The values of the activation energy of adsorption ( $E_a$ ) and the deduced values of  $Q_{ads}$  for this study are presented in Table 7.



**Figure 4.** Langmuir adsorption isotherms for JCL at 300 K and 333 K

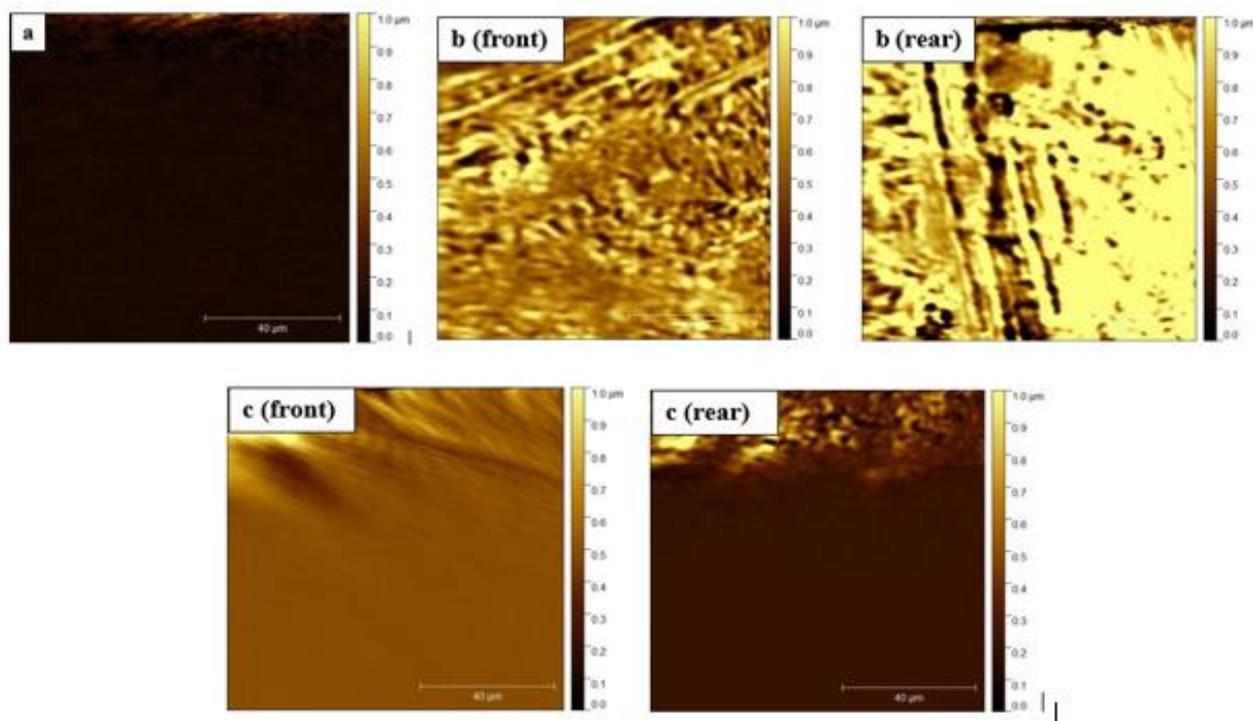
**Table 7.** Values of apparent activation energy and heat of adsorption calculated in the temperature range of 300 K – 333 K for JCL crude extract

$C$ (g/L)	$E_a$ ( $\text{kJ mol}^{-1}$ )	$Q_{ads}$ ( $\text{kJ mol}^{-1}$ )
0	2	–
0.4	11.8	–11.01
0.8	20.7	–19.9
1.2	22.3	–21.3
1.6	40.6	–40.4
2.0	40.5	–40.0

It is a general belief that activation energy of adsorption with a value less than or equal to 40 kJ/mol suggests physical adsorption process, while values higher than 80 kJ/mol suggest chemical adsorption process. Again, when the value of the heat of adsorption ( $Q_{ads}$ ) is between  $-20$  kJ/mol and  $0$  kJ/mol, the adsorption process is considered to be a physical one, but when the value is in the neighborhood of  $-400$  kJ/mol and  $-80$  kJ/mol, then the adsorption is regarded as a chemical process (Palumbo *et al.*, 2023, Messali *et al.*, 2017; Palumbo *et al.*, 2019; Umoren *et al.*, 2006; Bayol *et al.*, 2008). The values of activation energy of adsorption ( $E_a$ ) deduced in this work satisfied this condition and therefore indicate that the adsorption occurred by physical mechanism (physisorption). This result is further supported by the values of the heat of adsorption obtained, although JCL extract inhibiting molecules slightly exhibited a mixed-type of adsorption at  $1.6 - 2.0$  g/L at 300 K. These trends also agree with the trend of the depreciation of inhibitor efficiency with rise in temperature which is an indication of physical adsorption (Al-Amiery, 2023). Hence, physisorption mechanism is proposed for the interaction between the inhibitor molecules and the substrate surface.

### 3.8 Results of AFM surface observation of low carbon steel substrates

The surface morphology of the test carbon steel specimens after exposure to 0.5 M sulfuric acid solution without and with 2.0 g/L JCL-based inhibitor system for 9 h immersion at room temperature and that of the unexposed/unimmersed specimen were scanned using the atomic force microscopy (AFM) and the 2D images taken of the surfaces are presented in Figure 5. The images depict the surface roughness of the test carbon steel specimens and clearly, it can be observed that the surfaces of the steel substrates in Figure 4c are smoother than the surfaces shown in Figure 5b. Again, there are more generated deep pits in the surface profile depicted in Figure 5b than those shown in Figure 4a and 4c. These observations show that corrosion was severe on the unprotected substrate (Figure 5b) than on the protected substrates (Figure 5c). These results not only support the findings from the mass loss experiments but also agrees with existing literature reports (Ahanotu *et al.*, 2020; Onyeachu *et al.*, 2021; Onyeachu *et al.*, 2020).



**Figure 5(a-c).** AFM images of test carbon steel specimens (a) cleaned specimen not immersed in corrodent (b) specimen immersed in corrodent without any inhibitor for 9 hours at 27 °C (c) specimen immersed in corrodent with 2.0 g L<sup>-1</sup> *Justicia carnea*-based inhibitor for 9 hours at 27 °C

## 4. Conclusions

From the results, the following inferences/conclusions can be drawn;

1. *Justicia carnea* crude leaf extract functioned as an effective inhibitor for preservation of carbon steel from corrosion in a sulphuric acid medium.
2. The corrosion inhibitory effect of the extract is accounted for by the phytochemical compounds (alkaloids, glycosides, flavonoids, steroids and terpenoids) contained in the crude methanol extract of *Justicia carnea* leaves which reveal the presence of NH<sub>2</sub>, NHR, OH, -O-, C=C functional groups in the extract.
3. JCL-based inhibitor exhibited protective efficiency of 91 – over 99 % at 300 K and 87 – over 96 % at 333 K for a 0.4 – 2.0 g L<sup>-1</sup> dosage at both temperatures for carbon steel and this efficiency is a function of concentration and temperature.

4. The best approximation in isotherm fitting was obtained with the Langmuir adsorption isotherm with  $R^2 = 0.99991$  and  $0.99954$  at 300 K and 333 K respectively.
5. The values of free energy of adsorption,  $\Delta G_{ads}^0$ , the activation energy of adsorption ( $E_a$ ) and the heat of adsorption ( $Q_{ads}$ ) all satisfied the condition of physical adsorption.
6. The inhibitor exhibited a slight mixed-type of adsorption (physisorption and chemisorption) at higher inhibitor concentration  $> 1.5$  g/L.

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