Electrochemical and computational study of gum exudates from *Canarium schweinfurthii* as green corrosion inhibitor for mild steel in HCl solution

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**ABSTRACT**

The inhibition efficiency of the corrosion on mild steel in acid by Canarium schweinfurthii (CS) gum as inhibitor has been studied using chemical techniques (thermometric, gasometric, spectrophotometric and weight loss) and electrochemical techniques (electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PDP)). Scanning Electron Spectroscopy (SEM) was used to examine the surface morphology of mild steel samples both in the presence and absence of inhibitors at optimum conditions. Results obtained indicated that CS inhibited the corrosion of mild steel in HCl. The inhibition efficiency increases with increasing the inhibitor concentration but decreases with increasing the temperature. CS behaved as mixed type inhibitor retarding both cathodic and anodic reaction with high inhibition efficiency. Gas chromatography mass spectrometer (GCMS) spectrum of CS gum showed the presence of Nonacosane; 1-piperoylp: Stearic acid, 2-(hydroxymethyl)-2-nitropropane-1,3-diol, Stigmasta-5,22-dien-3-ol, dihex-5-en-2-yl phthalate, 9-octadecenoic acid, methyl ester, 1-pentadecarboxylic and Oleic acid. Inhibition action of the gum gave a strong correlation with these phytochemicals in the gum. Phenomenon of physical adsorption was proposed for the inhibition and the process followed the Langmuir adsorption isotherm with very high negative values of the free energy of adsorption ($\Delta G_{ads}$). Both the chemical and electrochemical results are in good agreement with each other while the surface studies reveal the formation of a smooth, dense protective layer in the presence of inhibitor. Quantum chemical computations of parameters associated with the electronic structures of specific components of the gum supported their inhibiting potentials.

**1. Introduction**

Corrosion is a serious economic and environmental problem and because of this, several researches have been carried out with the aimed of controlling or preventing the loss of valuable metals [1–3]. Corrosion of metals such as mild steel has been given considerable attention because during industrial processes such as acid wash, etching, descaling and prickling, corrosion cannot be avoided. Hence, the need to prevent such metal from corrosion has economic and environmental benefits. Several methods have been adopted to protect metals against corrosion in industries [2–4]. The use of inhibitors has been found to be one of the best options available for the protection of metals against corrosion [5]. Generally, characteristics of corrosion inhibitors include, possession of hetero atoms (N, O,S,P), triple bonds, suitable functional groups and aromatic or long carbon chain systems [8]. Based on these properties, organic products of plants origin have been successfully selected and used in the inhibition of the corrosion of some metals [6,7]. The use of gum exudates (a natural polymer from plant) to inhibit the corrosion of metals in acidic and alkaline environment is on the increase as they have been found to be cheap, biodegradable and comply with required environmental standards that vitiated the abolition of chromates and other non-eco-friendly inhibitors [8,9]. The inhibitive properties of plant gum exudates from Acacia trees [10], Guar [11], oleo-gum [12] *Raphia* [13], *Khaya* [14] and *Ficus* [5], have been reported.

One technique that has been used to evaluate organic corrosion inhibitors is molecular modeling [12]. Through this technique, it is possible to obtain structural parameters, such as the energies of the frontier molecular orbitals, the HOMO (highest occupied molecular orbital) and LUMO (lowest unoccupied molecular orbital), and the dipole moment which can be used to predict the action of corrosion inhibitors. In spite of the broad spectrum of information available for some gums and their corrosion inhibition potentials, literature is scanty on the use of quantum chemical study to calculate the molecular properties most relevant to their action as corrosion inhibitors. Also, explicit knowledge of the chemical structures of the phytochemicals

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of plant gums is required in order to identify the active constituents involved in corrosion inhibition. Therefore, the present study is aimed at

(i) elucidating the chemical structures of the constituents of \textit{Canarium schweinfurthii} (CS)

(ii) investigating their corrosion inhibition potentials for mild steel in solutions of HCl using gravimetric, thermosteric, gasometric, spectrophotometric, potentiodynamic polarization (PDP), electrochemical impedance spectroscopy (EIS), Scanning electron microscopy (SEM)

(iii) and evaluating their quantum chemical properties

\textit{Canarium schweinfurthii} (family-Burseraceae) also called African Elemi tree, is a perennial giant rocky plant grown which is widely distributed in the East, Central and West Africa [15]. In Nigeria, the plant is found in parts of Middle-Belt, South-East and South-West regions of the country [16]. The local names for the plant include “atile” (Hausa), “pet” (angas) “ako” (Yoruba), “agba” (Igbo) and “oda” (Idoma). It has been reported that when the bark of this plant is cut, it yields resinous aromatic gum (Figure 1) which have been used for different applications [17–19].

2. Experimental details

2.1. Material preparation

The sheets of mild steel used for this study were obtained from the Novara group Limited England. For the electrochemical measurement, the coupons were cut into 2 by 1.5 by 0.05 cm dimension while the coupons were mechanically pressed cut to a dimension of 5 by 4 by 0.05 cm for weight loss and gasometric measurements. The coupons were polished with series of emery paper of variable grades starting with the coarsest (400 grade) and then proceeding in steps to the finest (1200) grade, washed with distilled water, rinsed with absolute ethanol, cleaned in acetone and allowed to dry in the air before being preserved in a desiccator prior to corrosion testing. Samples of \textit{Canarium schweinfurthii} (CS) exudate gum were collected from matured stem of the plant located in Dutse Local Government Area of Jigawa State, Nigeria in January 2016. The exudates were purified following the method of Ameh [20]. All reagents used for the study were analar grade from BDH supplies chemicals, England and doubled distilled water was used for their preparation.

2.2. Weight loss method

The corrosion rates of mild steel were calculated by considering the total affected sample area and immersion time. The test samples were weighed using Scaltec high precision balance (Model SPB31) and immersed in 250 mL of 0.1 M HCl in the absence and presence of varying concentrations of CS gum myrrh extract (0.1–0.5 g/L), taken in a beaker at temperatures 303–333 K maintained in a thermostat water bath. After every 24 h, the corrosion product was removed by washing each coupon (withdrawn from the test solution) in a solution containing 50% NaOH and 100 g l$^{-1}$ of zinc dust. The washed coupon was rinsed in acetone and dried in the air before re-weighing. The difference in weight for a period of 168 h was taken as total weight loss. In order to get good results, the experiments were carried out in triplicate. From the average weight loss results, the percentage of inhibition efficiency (\% IE) and the degree of surface coverage ($\theta$) were calculated using the following equations: [20, 21].

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

\[
\theta = \left(1 - \frac{W_1}{W_2}\right) \quad (2)
\]

Where $W_1$ and $W_2$ are the average weight losses (g/dm$^3$) for mild steel in the presence and absence of inhibitor in HCl solution respectively.

The corrosion rates (in g m$^{-2}$ h$^{-1}$) for mild steel corrosion in different concentrations of the CS gum and other media have been determined for 168 h immersion period from weight loss using equation 3 [5].

\[
\text{Corrosion rate} = \frac{534W}{DAT} \quad (3)
\]

where $W =$ weight loss (mg); $D =$ density of specimen (g/cm$^3$), $A =$ area of specimen (square inches) and $T =$ period of immersion (hour).

2.3. Spectrophotometric analysis

The results of weight loss method were confirmed by estimating total iron ions (Fe$^{2+}$/Fe$^{3+}$) entered into the test solution during the course of immersion following the method of [21]. The analysis was performed spectrophotometrically using UV/Visible spectrophotometer (Jenway, Model 6405). The inhibition efficiency (\% I)
and corrosion rate were calculated using equation 4 and 5 respectively.

\[ CR = \frac{m}{A \times t} \quad (4) \]

\[ \%I = \left(1 - \frac{CR_0}{CR_1}\right) \times 100 \quad (5) \]

where, CR is the Corrosion rate in g m\(^{-2}\) h\(^{-1}\), m is the mass of corroded mild steel (calculated from the total iron content determined in the test solution); A is the area of the test specimen in m\(^2\); t is the exposure time in h, CR\(_0\) and CR\(_1\) are the corrosion rates of mild steel in presence and absence of CS gum iminium surfactant, respectively.

2.4. Gasometric method

The gasometric measurement was determined as previously described elsewhere [20] using a gasometric assembly. The progress of corrosion reaction was monitored by careful measurement of the volume of hydrogen gas evolved at fixed time intervals. From the volume of hydrogen evolved during the corrosion reaction, the corrosion rate (CR) was determined using equation 6 [22].

\[ \%IE = \frac{V_b - V_t}{V_b} \times 100 \quad (6) \]

where \(V_b\) is the volume of hydrogen gas evolved by the blank and \(V_t\) is the volume of hydrogen gas evolved in the presence of the inhibitor, after time, \(t\).

2.5. Thermometric method

Thermometric experiments were carried out as reported elsewhere [22]. The volume of the corrodent (HCl) used was 50 ml while the initial temperature in all the experiments was maintained at 303 K. The reaction number (RN) of each system was calculated by dividing the difference between the highest and lowest temperature attained by the time interval as expressed in equation 7.

\[ \text{RN}(\circ C \text{ min}^{-1}) = \frac{T_f - T_i}{t} \quad (7) \]

where \(T_f\) and \(T_i\) are final and initial temperatures and \(t\) is time interval.

From the reaction number, the inhibition efficiency (\% I) of the inhibitor was calculated using the following equation [22].

\[ \%I = \frac{RN_{aq} - RN_{wi}}{RN_{aq}} \times 100 \quad (8) \]

where \(RN_{aq}\) is the reaction number in the absence of inhibitors (blank solution) and \(RN_{wi}\) is the reaction number of 0.1 M HCl containing the studied inhibitor.

2.6. Polarization study

Measurements were obtained using a Voltalab 40 Potentiostat PGZ 301 combined with easy corrosion programme (Voltamaster 4). The test solution contained 0.1 M HCl and various concentration of CS gum. The electrical circuit was used for determining the variation of electrode potential with the electrical current. The potentiodynamic current-potential curves were recorded by changing the electrode potential automatically with a scan rate 0.33 mV s\(^{-1}\) from a low potential of \(-800\) to \(-300\) mV (SCE). Before each run, the working electrode was immersed in the test solution for 30 min to reach steady state. All potentials were measured against saturated calomel electrode (SCE).

The inhibition efficiency was calculated from the charge transfer resistance values as follow [23].

\[ \%I = \frac{i_0^{Corr} - i^{Corr}}{i_0^{Corr}} \times 100 \quad (9) \]

where \(i_0^{Corr}\) and \(i^{Corr}\) are the uninhibited and inhibited corrosion current densities, respectively.

2.7. Electrochemical impedance spectroscopy (EIS) analysis

Electrochemical impedance was obtained using a Vol talab 40 for all EIS measurements with a frequency range of 100 kHz to 50 mHz with a 4 mV sine wave as the excitation signal at open circuit potential. The real part was plotted on the X-axis and the imaginary part is plotted on the Y-axis of a chart to develop a Nyquist Plot. The charge transfer resistance values (R\(_{ct}\)) were calculated from the difference in impedance at Lower and higher frequencies [24,25]. The inhibition efficiency was calculated from the charge transfer resistance values using equation 10 [24].

\[ \%I = \frac{R_{ct(\text{Inh})} - R_{ct}}{R_{ct(\text{Inh})}} \times 100 \quad (10) \]

where \(R_{ct(\text{Inh})}\) and \(R_{ct}\) are values of the charge transfer in the presence and absence of the inhibitor.

2.8. Scanning electron microscope (SEM) analysis

A scanning electron microscope (SEM) model JSM-5600 LV, were used to analyze the morphology of the mild steel surface in the presence and absence of inhibitor. The images were taken at an accelerating voltage of 10 kV using different magnifications.

2.9. GC-MS analysis

GC-MS analysis was carried out on a GC Clarus 500 Perkin Elmer system comprising of a AOC-20i autosampler and gas chromatograph interfaced to a mass
spectrometer (GC-MS) instrument employing the following conditions: column Elite-1 fused silica capillary column (30 × 0.25 mm ID × 1 µm df, composed of 100% dimethylpolysiloxane), operating in electron impact mode at 70 eV; helium (99.999%) was used as carrier gas at a constant flow of 1 ml/min and an injection volume of 0.5 μl was employed (split ratio of 10:1) injector temperature 250°C; ion-source temperature of 280°C. The oven temperature was programmed from 110°C (isothermal for 2 min), with an increase of 10°C/min, to 200°C, then 5°C/min to 280°C, ending with a 9 min isothermal at 280°C. Mass spectra were taken at 70 eV; a scan interval of 0.5 s and fragments from 40 to 450 Da. Total GC running time was 36 min.

Interpretation on mass spectrum GC-MS was conducted using the database of National Institute Standard and Technology (NIST) Abuja, having more than 62,000 patterns. The spectrum of the unknown component was compared with the spectrum of the known components stored in the NIST library. The name, molecular weight and structure of the components of the test materials were ascertained. The concentrations of the identified compounds were determined through area and height normalization.

### 2.10. Phytochemical screening

The gum exudates were screened for the presence / absence of secondary phytochemical metabolites using the standard qualitative phytochemical methods reported by Trease and Evans [17].

### 3. Results and Discussion

#### 3.1. Gravimetric study

Figure 2 shows plots for the variation of weight loss with time during the corrosion of mild steel in 0.1 M HCl containing various concentrations of CS gum at 303 K. The plot generally revealed that weight loss of mild steel increases with time but decreases with increase in the concentration of the inhibitor indicating that the corrosion rate of mild steel increases with increase in the period of contact of the metal with the acid. At higher temperatures (313, 323 and 333 K; plots not shown), the weight loss of mild steel was found to increase with increase in temperature indicating that the corrosion rate of mild steel also increases with increase in temperature.

Table 1 presents values of corrosion rates of mild steel at various temperatures in the absence and presence of various concentrations of CS gum. The results indicated that in the presence of CS gum, the corrosion rate is decreased, even as the concentration of the inhibitor increases. The trend for the decrease is presented graphically in Figure 3. The plots generally reveal that the decrease in corrosion rate varies with the concentration of CS gum.

The values of inhibition efficiency of the inhibitor obtained using equation 1 are also presented in Table 1 and Figure 4. It can be seen that the inhibition efficiency seems to increase with concentration but decreases with temperature indicating that the mechanism of

| C (g/L) | 303 K | 313 K | 323 K | 333 K | 303 K | 313 K | 323 K | 333 K |
|--------|-------|-------|-------|-------|-------|-------|-------|-------|
| Blank  | 12.02 | 26.27 | 25.80 | 33.39 |      |      |       |       |
| 0.1    | 1.93  | 4.97  | 4.88  | 7.53  | 43.91 | 41.09 | 39.08 | 37.45 |
| 0.2    | 1.76  | 4.64  | 4.55  | 7.32  | 48.41 | 44.33 | 42.32 | 40.08 |
| 0.3    | 1.64  | 4.55  | 4.46  | 7.08  | 56.40 | 53.70 | 50.64 | 48.80 |
| 0.4    | 1.49  | 4.52  | 4.43  | 6.88  | 67.60 | 62.81 | 58.76 | 54.44 |
| 0.5    | 1.31  | 3.94  | 3.87  | 6.79  | 79.10 | 70.01 | 64.91 | 60.66 |
inhibition of the corrosion of mild steel in solution of HCl is physical adsorption. Physical adsorption mechanism according to Momoh – Yahaya et al., [25] is characterized with a decrease in inhibition efficiency with temperature as opposed to chemical adsorption mechanism, where inhibition efficiency is expected to increase with increase in temperature. The highest inhibition efficiency of 81.15% was obtained at 0.5 g / L of inhibition concentration at 303 K. This value was higher than those reported for gums of Ficus Platyphylla [26], Daniella oliverri [27], Ficus glumosa [28], Pachylubus edulis [29], Rapha hookeri [29] and lower than that reported for Khaya senegalensis [14].

Calculated values of the inhibition efficiency (% I) and corrosion rate obtained from the spectrophotometric analysis are presented in Table 2. It is evident from Table 2 that the inhibition efficiency of CS gum increases with increasing concentration and decreases with increasing temperature. It can be seen that 0.5 g/L of the inhibitor gave the highest inhibition efficiency as also obtained in the case of weight loss measurements. Also, the corrosion rate as expected was highest in the blank at 303 K. Similar trend was observed at 313, 323 and 333 K but with higher value of corrosion rate.

### 3.2. Gasometric method

The plots obtained for the variation of the volume of hydrogen gas evolved with time for the corrosion of mild steel in the absence and presence of different concentrations of CS gum at 303 and 333 K are presented in Figures 5 and 6 respectively. It can be seen from both plots that the volume of hydrogen gas evolved by the blank exceeded the volumes evolved by systems containing various concentrations of the inhibitor indicating that CS gum retarded the corrosion of mild steel in solutions of HCl. It is also evident from the plots that the volume of hydrogen gas evolution (which is related to corrosion rate) is concentration dependent, in that the corrosion rate decreases with increase in the concentration of the inhibitor. The volume of hydrogen gas evolved increases with time and increase in temperature.

Values of inhibition efficiency, calculated from equation 6 are presented in Table 3. Inspection of the Table reveals that inhibition efficiency increases with increase in the concentration of CS gum and decrease with increase in temperature. This also suggest that the adsorption of CS gum follows the mechanism of physical adsorption and similar with the trend in Figures 5 and 6 respectively. It can be seen from both plots that the volume of hydrogen gas evolved by the blank exceeded the volumes evolved by systems containing various concentrations of the inhibitor indicating that CS gum retarded the corrosion of mild steel in solutions of HCl. It is also evident from the plots that the volume of hydrogen gas evolution (which is related to corrosion rate) is concentration dependent, in that the corrosion rate decreases with increase in the concentration of the inhibitor. The volume of hydrogen gas evolved increases with time and increase in temperature.

| Table 2. Corrosion rate and inhibition efficiency of various concentrations of Canarium schweinfurthii (CS) for mild steel from Spectrophotometric measurement. |
|-----------------|-----------------|-----------------|-----------------|--------------|-----------------|
| C (g/L)         | 303 K           | 313 K           | 323 K           | 333 K        |
|                 | 303 K           | 313 K           | 323 K           | 333 K        |
|空白             | 0.000366        | 0.0005222       | 0.0009443       | 0.001686     |
| 0.1             | 0.000141        | 0.000244        | 0.000475        | 0.000939     |
| 0.2             | 0.000131        | 0.000224        | 0.000443        | 0.000847     |
| 0.3             | 0.000103        | 0.000203        | 0.000406        | 0.000788     |
| 0.4             | 8.75E-05        | 0.00017         | 0.000377        | 0.000705     |
| 0.5             | 6.9E-05         | 0.00015         | 0.000327        | 0.000614     |
| inhibition efficiency (%) | 61.41 | 53.25 | 49.72 | 44.31 |
|                  | 64.21           | 57.12           | 53.12           | 49.74        |
|                  | 71.80           | 61.11           | 57.02           | 53.26        |
|                  | 76.11           | 67.52           | 60.11           | 58.2         |
|                  | 81.15           | 71.23           | 65.34           | 63.60        |
Table 3. Reaction Number and Inhibition efficiencies of Canarium schweinfurthii (CS) CS gum calculated from hydrogen evolution and thermometric measurements.

| Conc. of CS gum (g/L) | Reaction Number | % Ithermometric (303 K) | % Igasometric (303 K) | % Igasometric (333 K) |
|-----------------------|-----------------|------------------------|-----------------------|-----------------------|
| Blank                 | 1.95            | 78.56                  | 57.22                 | 49.41                 |
| 0.1                   | 0.97            | 67.69                  | 69.44                 | 50.32                 |
| 0.2                   | 0.63            | 73.85                  | 77.24                 | 60.11                 |
| 0.3                   | 0.51            | 81.54                  | 82.39                 | 64.32                 |
| 0.4                   | 0.36            | 89.23                  | 89.58                 | 77.54                 |

Figure 7. Plots of correlation of inhibition efficiencies obtained from thermometric and gasometric methods at 303 K.

3.3. Thermometric method

The calculated values of reaction number (RN) and the percentage inhibition efficiency in the present and absent of the inhibitor obtained from thermometric study are also presented in Table 3. It is seen clearly from the values presented that reaction number decreased in the presence of CS gum compared to the blank solution. The inhibition efficiency as expected increased with increase in concentration of the inhibitor. This supports our findings from the previous thermometric and gasometric studies. Results obtained from thermometric and gasometric methods at 303 K were in excellent agreement as can be seen in high values of R² shown in Figure 7. The observed difference in the values of inhibition efficiencies obtained from the gasometric and thermometric method may be attributed to the difference in time required to form an adsorbed layer of the inhibitor on the mild steel surface that can inhibit corrosion.

3.4. Potentiodynamic polarization studies

Polarization curves obtained for mild steel carried out in 0.1 M HCl at 303 K in the absence and presence of different concentrations of CS gum are shown in Figure 8. The plot clearly show that the addition of CS gum is seen to affect the anodic as well as the cathodic partial reactions, shifting the corrosion potential (Ecorr) slightly toward more positive (anodic) values. Also corresponding values of corrosion current density (Icorr) obtained as presented in Table 4 were also found to decrease with increase in inhibitor concentration. This according to Tribak et al., [30] is an indication of a mixed-type corrosion inhibition mechanism.

Polarization parameters (i.e. corrosion current density (icorr), and corrosion potential (βa and βc) evaluated using the Tafel extrapolation method and inhibition efficiency (I %) calculated from the related polarization curves, are presented in Table 4. The Tafel constants (βa and βc) showed significant changes in the presence of CS gum as expected signifying that the inhibitor adsorbed on the surface of the metal. Also, the observed changes in the values of both βa and βc further indicates that the corrosion of the mild steel in presence of inhibitor is under both anodic and cathodic control. The maximum displacement in corrosion potentials values (Ecorr) in millivolts observed on addition of the inhibitor was found to be less than 85 mV which further supports the fact that CS gum is acting as a mixed type inhibitor [31]. The inhibition efficiency values obtained increases with increasing inhibitor concentrations. The values were also found to be in good agreement from that of weight loss measurements, thermometric and gasometric measurements.

Table 4. Electrochemical data for the corrosion of mild steel in 0.1 M HCl in the absence and presence of CS gum at 303 K.

| C (g/L) | βa (mVdec⁻¹) | βc (mVdec⁻¹) | Ecorr (mV) | Icorr (μA) | I % |
|---------|---------------|---------------|------------|------------|-----|
| Blank   | 135.1         | 139.3         | −863       | 189.26     | −   |
| 0.02    | 117.2         | 123.0         | −917       | 30.15      | 84.07 |
| 0.04    | 113.5         | 118.2         | −923       | 25.18      | 86.70 |
| 0.06    | 111.2         | 115.5         | −937       | 22.28      | 87.70 |
| 0.08    | 108.6         | 111.6         | −941       | 21.89      | 88.43 |
| 0.10    | 104.7         | 107.8         | −947       | 18.13      | 90.42 |

3.5. Electrochemical impedance spectroscopy (EIS) study

The behaviour of the corrosion of mild steel in hydrochloric acid in the present and absence of CS gum
Figure 9. Nyquist plots for mild steel immersed in 0.1 M HCl in absence and presence of different concentrations of the inhibitor

Table 5. EIS parameters for corrosion of mild steel in 0.1 M HCl in the absence and presence of different concentrations of CS gum at 303 K.

| C (g/l) | Rct (Ω cm²) | Cdl (μF cm⁻²) | %|
|--------|-------------|---------------|---|
| Blank  | 100.42      | 281           | - |
| 0.01   | 700.12      | 232           | 85.66 |
| 0.05   | 718.22      | 174           | 86.02 |
| 0.10   | 851.18      | 160           | 88.20 |

was studied at 303 K using electrochemical impedance spectroscopy (EIS) in order to complete and compare the results from the other techniques. The obtained impedance responses are given in Figure 9 as Nyquist plots. The electrochemical parameters deduced are shown on Table 5.

It can be seen that the plots in absence and with varying concentrations of CS gum are almost identical implying that addition of inhibitor does not cause any significant change in the mechanism of the corrosion reaction but inhibits the corrosion by increasing the surface coverage by the adsorbed inhibitor.

From the results presented, it can be seen that the charge transfer resistance (Rct) increased with increasing concentration of the CS gum. Also, the capacitance values of double-layer Cdl was found to decrease with the concentration of inhibitor. The decrease in Cdl values with increase in concentration is due to the increase in the surface coverage by CS gum which resulted in an increase in the inhibition efficiency. The observed decrease in Cdl values, which normally corresponds to alteration of the double-layer thickness can be attributed to the adsorption of the gum (with lower dielectric constant compared to the displaced adsorbed water molecules) on the metal/acid interface, thereby protecting the metals from the corrosive effect of the aggressive acids [30].

The inhibition efficiency obtained by this method was found to be in good agreement with those obtained from the previous techniques.

Figure 10. Arrhenius plot for the corrosion of mild steel in the presence of Canarium schweinfurthii (CS) as an inhibitor at various temperatures.

3.6. Effect of temperature

The apparent activation energy (Ea) for dissolution of mild steel in 0.1 M HCl was calculated from the slope of plots by using modified Arrhenius equation [31–33].

\[
\ln(CR) = \ln(A) - \frac{E_a}{RT} \quad (11)
\]

where CR is the corrosion rate of mild steel in solution of 0.1 M HCl, A is the Arrhenius constant or pre-exponential factor, R is the universal gas constant and T is the temperature.

By plotting ln (CR) against 1/T (See Figure 10), the values of activation energy (Ea) has been calculated (Ea = (slope)·R) and presented in Table 6. From the results obtained, it is evident that excellent degree of linearity (R² values are very close to unity) were obtained in all cases indicating the application of the Arrhenius model to the inhibited and uninhibited corrosion reaction of mild steel. Value of the activation energy for the blank was 28.01 J/mol. In the presence of the inhibitor, Ea values ranged from 36.85 to 44.52 J/mol and were found to increase with increase in concentration of the inhibitor. The high values of activation energy obtained in the presence of the inhibitor as compared to the blank further indicate that the corrosion of mild steel is retarded by the presence of CS gum. The Ea values increasing with increase in concentration of CS gum indicates that the ease of adsorption of the inhibitor on the surface of the metal increases with increasing concentration. According to Obot et al [34], most corrosion reaction that have values of activation energy less than 80 kJ/mol is associated with the mechanism of physical adsorption while those more than 80 kJ/mol points toward the mechanism of chemical adsorption. Hence the mechanism of adsorption of CS gum on the surface of mild steel is consistent with charge transfer from charged inhibitor to charged metal surface, which favours physical adsorption [14, 22].

The enthalpy of activation (ΔH_{ads}^0) and entropy of activation (ΔS_{ads}^0) for the corrosion of mild steel in HCl were obtained by applying the transition-state
Table 6. Arrhenius parameters for the inhibition of the corrosion of mild steel by various concentrations of *Canarium schweinfurthii* (CS).

| C (g/L) | Slope | ln(A) | E_a (J/mol) | R^2     |
|---------|-------|-------|-------------|---------|
| Blank   | 3.464 | 13.938| 28.01       | 0.9922  |
| 0.1     | 4.552 | 15.684| 36.85       | 0.9997  |
| 0.2     | 4.730 | 16.243| 39.33       | 0.9996  |
| 0.3     | 4.892 | 16.648| 40.68       | 0.9996  |
| 0.4     | 5.149 | 17.406| 42.81       | 0.9971  |
| 0.5     | 5.479 | 18.334| 44.52       | 0.9987  |

Figure 11. A plot of log (ln CR / T) versus 1 / T for mild steel in 0.1 M HCl in the absence and presence of different concentrations of CS gum.

Figure 12. Langmuir isotherm for the adsorption of *Canarium schweinfurthii* (CS) gum on mild steel surface.

Table 7. Transition state parameters for the inhibition of the corrosion of mild steel by various concentrations of *Canarium schweinfurthii* (CS).

| C (g/L) | Slope | Intercept | ΔS^0_{ads} (J/mol) | ΔH^0_{ads} (J/mol) | R^2     |
|---------|-------|-----------|--------------------|--------------------|---------|
| Blank   | 3.149 | 7.1829    | 257.26             | −26.22             | 0.9908  |
| 0.1     | 4.236 | 8.9294    | 271.78             | −35.18             | 0.9997  |
| 0.2     | 4.435 | 9.4870    | 276.42             | −36.88             | 1.0000  |
| 0.3     | 4.578 | 9.8938    | 279.80             | −38.06             | 1.0000  |
| 0.4     | 4.833 | 10.6520   | 286.10             | −40.19             | 0.9972  |
| 0.5     | 5.159 | 11.5800   | 293.82             | −42.46             | 0.9999  |

The adsorption characteristics of CS gum for mild steel was investigated by fitting the degree of surface coverage values calculated from weight loss measurements into different adsorption isotherm models including Langmuir, Freundlich, Flory-Huggins, El awardy et al., Bockris-Swinkel and Temkin isotherms. The best results judged by the correlation coefficient (R^2) was obtained with Langmuir adsorption isotherm.

Langmuir isotherm is given by the expression [35]:

\[ \log \left( \frac{C}{θ} \right) = \log b_{ads} - \log C \]  

(13)

where C is the concentration of CS gum in the bulk electrolyte, θ is the degree of surface coverage of the inhibitor and b_{ads} is the equilibrium constant of adsorption.

Table 8. Langmuir parameters for the adsorption of *Canarium schweinfurthii* (CS). 

| T (K) | Slope | log b_{ads} | ΔG^0_{ads} (kJ/mol) | R^2     |
|-------|-------|-------------|---------------------|---------|
| 303   | 0.964 | 0.0425      | −10.36              | 0.9992  |
| 313   | 0.976 | 0.0681      | −10.51              | 0.9998  |
| 323   | 0.976 | 0.0686      | −10.52              | 0.9987  |
| 333   | 0.981 | 0.0936      | −10.66              | 0.9989  |

into different adsorption isotherm models including Langmuir, Freundlich, Flory-Huggins, El awardy et al., Bockris-Swinkel and Temkin isotherms. The best results judged by the correlation coefficient (R^2) was obtained with Langmuir adsorption isotherm.

Langmuir isotherm is given by the expression [35]:

\[ \log \left( \frac{C}{θ} \right) = \log b_{ads} - \log C \]  

(13)

where C is the concentration of CS gum in the bulk electrolyte, θ is the degree of surface coverage of the inhibitor and b_{ads} is the equilibrium constant of adsorption.

Figure 12 shows the Langmuir plots developed through equation 13, while adsorption parameters deduced from the plots are presented in Table 8. Slope and R^2 values are seen to proximate unity, which suggest that there is little or no interaction between the inhibitor and the metal.

Standard free energy (ΔG^0_{ads}) was obtained according to the following equation [25]:

\[ ΔG^0_{ads} = −RT \ln (55.5 b_{ads}) \]  

(14)

where 55.5 is the molar concentration of water in solution expressed in g\(^{-1}\)L. Using this equation, the standard Gibbs free energy of adsorption of CS gum...
on the mild steel surface at the different temperature studied was found to be within the range of $-10.66$ to $-10.36$ kJ mol$^{-1}$. These are within the range of values expected for the mechanism of physical adsorption. According to literature, values of standard Gibbs free energy of adsorption with magnitude much less than 40 kJ mol$^{-1}$ have typically been correlated with the electrostatic interactions between inhibitor molecules and charged metal surface (physisorption), whilst those of magnitude in the order of 40 kJ mol$^{-1}$ and above are associated with charge sharing or transfer from the inhibitor to the metal surface (chemisorption) [29]. The negative values of standard free energy of adsorption obtained from this study is an indication that adsorption of the inhibitor on the mild steel surface is spontaneous [5]. Also, the increasing negative values of the Gibbs free energy of adsorption with increase in temperature is an indication that the adsorption process is more feasible at lower temperatures [20].
Table 9. Phytochemical Screening of CS gum.

| S/N | Chemical constituent | Observation / inference |
|-----|----------------------|-------------------------|
| 1   | Tannins              | +                       |
| 2   | Flavonoids           | +                       |
| 3   | Alkaloid             | −                       |
| 4   | Saponins             | +                       |
| 5   | Carbohydrates        | +                       |
| 6   | Anthraquinone        | +                       |
| 7   | Carotenoids          | +                       |
| 8   | Steroid              | −                       |

3.8. Scanning electron microscopy (SEM) studies

The SEM images of (a) mild steel alone, (b) mild steel in 0.1 M HCl and (c) mild steel in 0.1 M HCl plus 0.5 g/L CS gum are given in Figure 13. The results show that the images obtained for the surface of mild steel immersed in 0.1 M HCl in the presence of CS gum (Figure 14c) were smoother than that obtained when the inhibitor was absent (Figure 14b). The smooth surfaces may have appeared as a result of the formation of a protective film on the mild surface by the CS gum.

3.9. Phytochemical study

The results obtained from the phytochemical analysis of the CS gum is as presented in Table 9. CS gum as seen from the table contains saponins, carotenoids, tannins, carbohydrates, anthraquinone and flavonoids. These compounds are rich in conjugated aromatic structures and contain heteroatoms with free electron pairs that are available to form bonds with the metal surface [20]. It is therefore, pertinent to say that the adsorption of these compounds onto mild steel surface is responsible for the corrosion inhibition effect.

3.10. GC-MS study

The GC-MS spectrum obtained for the CS gum is presented in Figure 14. Six prominent peaks were obtained from the GC-MS spectrum of CS gum. The fragmentation pattern was searched in National Institute Standard and Technology (NIST) Mass Spectral Library database. The values of retention time, mass peak, area under

Table 10. Properties of compounds identified from the GCMS spectrum of CS gum.

| Line no | IUPAC name                        | Chemical Formula | Molar mass | RT (min) | Mass peak | %C   | Fragmentation peaks |
|--------|-----------------------------------|------------------|------------|----------|-----------|------|-------------------|
| 1      | Nonacosane                        | C_{19}H_{38}O_{2} | 298        | 17.142   | 16        | 0.11 | 25(5),43(30),43(45),57(20),74(100),87(60),101(5),115(1),129(5),143(15),157(1),187(1),199(4),213(1),253(5),267(2),298(10) |
| 2      | 1-piperoyl piperidine             | C_{12}H_{19}NO_{3} | 285        | 20.180   | 59        | 25.84 | 38(16%),51(20%),53(20%),61(12%),73(30%),103(10%),110(100%),133(21%),153(20%),211(40%),275(50%) |
| 3      | Stearic acid                      | C_{18}H_{36}O_{2} | 284        | 29.725   | 63        | 0.63  | 41(72%),41(100%),56(69%),73(72%),85(30%),98(20%),116(10%),129(18%),171(8%),185(12%),199(10%),213(6%),227(100%),241(20%),284(29%) |
| 4      | 2-(hydroxymethyl)-2-nitropropane-1,3-diol | C_{6}H_{9}NO_{4} | 151        | 14.321   | 32        | 29.95 | 41(10%),43(10%),44(30%),46(95%),66(100%),76(10%) |
| 5      | Stigma-stearic acid               | C_{31}H_{50}O_{2} | 454        | 25.268   | 58        | 18.28 | 43(70%),66(45%),81(50%),93(35%),105(45%),119(25%),136(50%),145(45%),197(10%),239(15%),253(55%),283(45%),355(2%),394(100%),421(100%),508(50%),521(100%),630(48%),765(50%),933(38%),105(40%),121(31%),149(100%),177(50%) |
| 6      | dihex-5-en-2-yl phthalate          | C_{20}H_{26}O_{4} | 330        | 24.495   | 31        | 24.87 | 42(10%),50(28%),63(48%),76(50%),93(38%),105(40%),121(31%),149(100%),177(50%) |
| 7      | 9-octadecenoic acid, methyl ester | C_{19}H_{36}O_{2} | 296        | 17.744   | 42        | 0.09  | 27(15),41(85),55(100),69(70),74(68),87(45),98(35),123(15),137(10),180(13),222(20),264(35),298(5) |
| 8      | 1-penta-decanecarboxylic acid     | C_{16}H_{32}O_{2} | 256        | 21.545   | 82        | 0.13  | 40(10%),41(80%),60(90%),73(90%),85(30%),98(20%),115(15%),129(25%),143(5%),157(6%),171(6%),185(7%),199(6%),213(20%),227(5%),239(3%),256(20%),285(2%),300(2%) |
| 9      | Oleic acid                        | C_{18}H_{34}O_{2} | 282        | 29.118   | 70        | 0.10  | 41(90%),559(100%),69(70%),83(52%),97(40%),98(22%),112(18%),127(5%),264(18%) |
Figure 15. Optimized structures, HOMO and LUMO molecular orbitals (Using Huckel approximation) of dihex-5-en-2-yl phthalate; 2-(hydroxymethyl)-2-nitropropane-1,3-diol; stigmasta-5,22-dien-3-ol and 1-piperoylp.

| Optimized structure | HOMO | LUMO |
|---------------------|------|------|
| dihex-5-en-2-yl phthalate |
| 2-(hydroxymethyl)-2-nitropropane-1,3-diol |
| Stigmasta-5,22-dien-3-ol |
| 1-piperoylp |

the curve and concentrations of likely chemical compounds (obtained through area and height normalizations) in each peak are presented in Table 10. Phytoconstituents identified from the gum included Nonacosane, 1-piperoylpiperidine (or 1-piperoylp), Stearic acid, 2-(hydroxymethyl)-2-nitropropane-1,3-diol, Stigmasta-5,22-dien-3-ol, dihex-5-en-2-yl phthalate, 9-octadecenoic acid, methyl ester, 1-pentadecanecarboxylic, Oleic acid, with % concentration of 0.11, 25.84, 0.63, 29.95, 18.28, 24.87, 0.09, 0.13 and 0.10 respectively.

3.11. Quantum chemical studies

One of the aspects of recent corrosion inhibition studies is the use of quantum chemical methods to calculate electronic properties possibly relevant to explain the inhibiting action [36]. With this method, the capability of inhibitor molecules to donate or accept electrons can be predicted with analysis of global reactivity parameters, such as energy gap ($\Delta E$) between Highest Occupied Molecular Orbital (HOMO) and Lowest Unoccupied Molecular Orbital (LUMO), chemical potential, hardness, softness, dipole moment etc. The reactive ability of the inhibitor is considered to be closely related to their frontier molecular orbitals, the HOMO and LUMO. $E_{\text{HOMO}}$ of the molecule means a higher electron donating ability to appropriate acceptor molecules with low-energy empty molecular orbital and thus explains the adsorption on metallic surfaces by way of delocalized pairs of $\pi$ – electrons. $E_{\text{LUMO}}$, signifies the electron receiving tendency of a molecule [36].
Table 11. Frontier molecular orbital energies of chemical constituents of CS gum.

| S/N | Molecule                                      | $E_{\text{HOMO}}$ (eV) | $E_{\text{LUMO}}$ (eV) | $\Delta E$ (eV) |
|-----|-----------------------------------------------|-------------------------|-------------------------|---------------|
| 1   | dihex-5-en-2-yl phthalate                     | −10.129                 | −0.885                  | 9.244         |
| 2   | stigmasta-5,22-dien-3-ol                      | −10.778                 | 1.3936                  | 12.172        |
| 3   | 1-piperoylp                                   | −11.24                  | 3.80                    | 15.04         |
| 4   | 2-(hydroxymethyl)-2-nitropropane-1,3-diol     | −10.27                  | −0.986                  | 9.284         |

The optimized molecular structure obtained with Gaussian 03 code of programmes using the B3LYP hybrid functional and the 6-31G (d) basis set for 1-piperoylp piperidine, 2-(hydroxymethyl)-2-nitropropane-1,3-diol, Stigmasta-5,22-dien-3-ol, dihex-5-en-2-yl phthalate identified as the major constituents in the GC – MS study are depicted in Figure 15. Quantum chemical parameters such as $E_{\text{HOMO}}$, $E_{\text{LUMO}}$ and $\Delta E$ calculated for these common phytochemical constituents are presented in Table 11. The HOMO and LUMO diagrams (red indicate positive and blue, negative) of the four compounds indicated that in dihex-5-en-2-yl phthalate, the lobes are concentrated in the radical nitro group while the lobes are concentrated in the aromatic ring, the amide and hydroxyl bonds for 2-(hydroxymethyl)-2-nitropropane-1,3-diol, stigmasta-5,22-dien-3-ol and 1-piperoylp respectively, indicating that adsorption of the inhibitor might have proceeded through these three groups.

Analysis of Table 11, indicated that the values of $E_{\text{HOMO}}$ of dihex-5-en-2-yl phthalate, stigmasta-5,22-dien-3-ol, 1-piperoylp, 2-(hydroxymethyl)-2-nitropropane-1,3-diol increased in the following order: dihex-5-en-2-yl phthalate > 2-(hydroxymethyl)-2-nitropropane-1,3-diol > stigmasta-5,22-dien-3-ol > 1-piperoylp. The values of $\Delta E$ decreased in the following order: dihex-5-en-2-yl phthalate < 2-(hydroxymethyl)-2-nitropropane-1,3-diol < stigmasta-5,22-dien-3-ol < 1-piperoylp. The values of HOMO obtained also indicate the greater tendency of these phytochemical constituents to donate the electron to the vacant d-orbital of the iron atom. The CS gum can be said to have the tendency to bind with the mild steel surface.

4. Conclusions

The present study revealed that *Canarium schweinfurthii* (CS) gum exudate acts as a very good inhibitor for the corrosion of mild steel in HCl. The inhibition efficiency was found to increase with the inhibitor concentration. The gum acted as a mixed-type inhibitor in 1.0 M HCl, while the adsorption on the mild steel surface obeys Langmuir adsorption isotherm. The values of $\Delta G_{\text{ads}}$ obtained are low and negative, which reveals the spontaneity of the adsorption process. SEM reveals the formation of a smooth surface on the mild steel in presence of CS gum which may be due to the formation of an adsorptive film of electrostatic character.

Disclosure statement

No potential conflict of interest was reported by the author.

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