Corrosion inhibition potential of *Terminalia avicennioides* extract on mild steel in 1.0 M HCl: kinetics, thermodynamics and synergestic studies

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**ABSTRACT**

The corrosion inhibition effect of *Terminalia avicennioides* (TA) stem extract in 1.0 M HCl solution was investigated using weight loss and electrochemical method. The operation variables considered include temperature (303-333 K) and concentration of the inhibitor (0.2 - 1.0 g/L). Inhibition efficiency increased from 21.88% to 88.71% with concentration but decreased with rise in temperature which is suggestive of physisorption. This was supported by the values of activation energies (E_a) obtained ranging from 22.67 KJ mol^{-1} to 60.67 KJ mol^{-1} which was lower than 80 KJ mol^{-1}. The values of Gibb’s free energy of adsorption (ΔG_{ads}) indicated that the adsorption of the inhibitor on mild steel surface was a spontaneous process. The Kinetic treatment of the data followed a pseudo-first order reaction while the positive values obtained for enthalpy and entropy of activation is reflective of endothermic reaction nature and associative activation complex respectively. The adsorption mechanism followed the Langmuir adsorption isotherm model. Electrochemical analysis showed that the inhibitor functioned as a mixed type inhibitor. The inhibition efficiency of the inhibitor was found to be enhanced by the presence of KI. Therefore the extract functioned as good corrosion inhibitor for mild steel in 1.0 M HCl.

**Keywords:** *Terminalia avicennioides*, inhibition efficiency, physisorption, synergestic effect
1. INTRODUCTION

The tendency of a metal to corrode depends on factors such as the composition of the metal as formed during alloying, the temperature for deformation of a single metal surface developed during fabrication or grain structure of the metal. Corrosion prevention would be more practical than trying to eliminate it completely [1].

Corrosion related costs for most developed countries in the world represent up to a few percent of the gross domestic product (GDP) which can sometimes be close to what is spent on, for example, public education or healthcare. This has attracted interest of the scientific community to corrosion and its related phenomena [2].

There are several methods of corrosion control and prevention such as material selection, coating, cathodic protection and the use of corrosion inhibitors among others. Apart from the expensiveness of inorganic inhibitors, most of the chemical substance used as corrosion inhibitors contaminate the environment after use and cause a lot of problems and destruction of plant and animal life, therefore restrictions have been placed on the use of some of these chemical inhibitors because of their toxic nature. This is why attention has shifted to the use of environmentally friendly and ecologically acceptable, inexpensive, readily available and renewable plant products as green inhibitor. Synthetic organic inhibitors have also been extensively applied but their use is now being marred by their toxicity and high cost of manufacturing [3].

Inhibitors are commonly used to prevent metal dissolution as well as acid consumption and retard the corrosive attack on metallic materials. Corrosion inhibitors are substances which when added in small concentrations to corrosive media decrease or prevent the reaction of the metal with the media. Inhibitors are added to many systems. Most of the effective inhibitors contain hetero-atom (such as O, N, and S) and multiple bonds in their molecules through which they are adsorbed on the metal surface. It has been observed that adsorption depends mainly on certain physicochemical properties of the inhibitor group, such as functional groups, electron density at the donor atom, π-orbital character, and the electronic structure of the molecule [4].

2. EXPERIMENTAL METHODS

2.1. Materials

1.0 M analytical grade HCl solution was used as aggressive acid environment, methanol, acetone as drying agent, potassium hydroxide was used for the synergistic effect, sodium hydroxide and distilled water, all obtained from Emole Scientific in Makurdi. Analytical digital balance (Acculab Startorius Group) was used for weighing. Linear polarization measurement was carried out using Potentiostat/Galvanostat (NOVA AUTOLAB – PGSTAT 302N VERSION – 1.10.1.9) at Ahmedu Bello University, Zaria, Nigeria.

2.2. Material Preparation

The mild steel specimens were polished successively with sand paper, washed in distilled water, degreased with acetone and finally dried in hot air blower. Weight loss experiments were conducted on mild steel specimens of dimension 3.0 cm × 2.0 cm × 0.14 cm and electrochemical measurements were conducted in 1.0 cm long stem (isolated with
epoxy resin) to provide an exposed surface area 1.0 cm² of working electrode (WE). 5 mM of KI was used for the synergistic studies.

2.3. Inhibitor

The *Terminalia avicennioides* stem barks were collected from Ipinu-Oju in Oju L.G.A., Benue State, Nigeria. They were air dried and mechanical grinder was used to grind them into powdery form, sieved and the fine powder was stored in an air tight container and was extracted with methanol at room temperature using maceration process. The resultant solution was filtered after 24hrs using Whatman filter paper No 1. The filtrate was subjected to evaporation (to leave the sample free of methanol). The stock solution of the extract so obtained was used to prepare different concentrations (0.2 g/L, 0.4g/L, 0.6 g/L, 0.8g/L and 1.0 g/L) of inhibitor for the corrosion inhibition test by diluting with the respective aggressive solutions.

2.4. Test Solution

The aggressive solution 1.0 M HCl was used to carry out all weight loss and electrochemical experiments which was prepared by dilution of analytical grade 37% HCl with distilled water [22-29].

2.5. Weight Loss Measurements

The effect of time, concentration, temperature and synergistic studies were carried out using weight loss measurement. The effect of temperature was carried out at 303, 313, 323 and 333 K. Gravimetric experiments was conducted on test coupons of dimension 2 cm × 3 cm × 0.14 cm. The pre-cleaned and weighed coupons were suspended in beaker containing the test solutions using glass hooks and rods. Tests were conducted under total immersion conditions in 200 mL of the aerated and unstirred test solutions. To determine weight loss with respect to time, the coupons were retrieved at 24 hrs intervals and were repeated for 168 hrs, immersed in 20% NaOH solution, scrubbed with bristle brush, washed, dried and weighed. The weight loss was taken to be the difference between the weight of the coupons at a given time and its initial weight. All tests were run in duplicate to obtain good reproducible data. Average values for each experiment was obtained and used in subsequent calculations. A mild steel specimen was immersed in duplicate in 200 mL of the test solutions (1.0 M HCl) with and without addition of inhibitors of different concentrations at room temperature (27±2 °C). The cleaned specimens were weighed before and after 3 hours of immersion in the test solution for effect of temperature. The same procedure was followed for synergistic effect but with addition of 5 mM of KI to the test solution. The percentage inhibition efficiency (IE), the degree of surface coverage (θ) and the corrosion rate (CR) of mild steel and synergism (S) was calculated using equations below [5].

\[
IE_{exp} = \left(1 - \frac{W_{(1)}}{W_{(0)}}\right) \times 100
\]

(1)

where: \(W_{(0)}\) is the weight loss of the mild steel without inhibitor and \(W_{(1)}\) is the weight loss of mild steel with inhibitor.
\[
\theta = \frac{IE_{\text{exp}}}{100}
\]  
\[
CR\left(gh^{-1}cm^{-2}\right) = \frac{\Delta W}{At}
\]  
\[
S = \frac{1 - I_a - I_b + I_aI_b}{1 - I_{ab}}
\]

2.6. Electrochemical Linear Polarization Measurements

The mild steel was sealed with epoxy resin in such a way that only one square surface area will be left uncovered. The exposed surface was degreased in acetone, rinsed with distilled water and dried in warm air. A conventional three-electrode system consisting of mild steel as working electrode, platinum (Pt) as an auxiliary electrode and saturated calomel electrode (SCE) as reference electrode was used for the measurements. The experiments was conducted at room temperature (27±2 °C) using 200 mL of test solution. The % IE was calculated from the charge transfer resistance (Rct) values by using the equation,

\[
IE = \frac{R_{ct(0)} - R_{ct(1)}}{R_{ct(1)}} \times 100\%
\]

where: Rct(0) is the charge transfer resistance of MS without inhibitor and Rct(1) is the charge transfer resistance of MS with inhibitor.

The Tafel polarization curves was recorded by scanning the electrode potential from -300 mV to 300 mV (vs SCE) with a scanning rate of 1 mV/s. The corrosion current densities (Icorr) was extrapolated from the anodic and cathodic curves to corrosion potential. The % IE was obtained from the equation

3. RESULTS AND DISCUSSION

3.1. Weight Loss Measurements

3.1.1. Effect of Temperature

Inhibition efficiency increased with increase in concentration of the inhibitors but decreased with increase in temperature. Corrosion rates of mild steel, in the absence and presence of inhibitor in the acid solution increase with increase in temperature, but the corrosion rate decreased with increase in the concentration of the inhibitor than in the absence of it. This indicates that TA is effective corrosion inhibitor for mild steel in hydrochloric acid. This result suggests a physical adsorption of the extract compounds on the mild steel surface. That is, the inhibitor is physically adsorbed (physicosorption) at lower temperature while chemisorption is favoured at higher temperature [6]. Inhibition efficiency and Corrosion rates ($\times 10^{-3} \ gh^{-1}cm^{-2}$) of mild steel in various concentrations of TA are summarized in Table 1. Variation of weight loss as a function of temperature, inhibition efficiency and corrosion rate are represented in Figures 1-3.
Figure 1. Variation in weight loss for the effect of temperature on the corrosion of mild steel in 1.0 M HCl solution in the absence and presence of TA.

Figure 2. Effect of temperature on corrosion inhibition efficiency of mild steel in 1.0 M HCl in the absence and presence of TA extract.
Figure 3. Effect of temperature on corrosion rate of mild steel in 1.0 M HCl in the absence and presence of TA

Table 1. Inhibition efficiency and Corrosion rates ($\times 10^{-3}$ g h$^{-1}$ cm$^{-2}$) of mild steel in various concentrations of TA

<table>
<thead>
<tr>
<th>Concentration (g/L)</th>
<th>Inhibition Efficiency (%IE)</th>
<th>Corrosion Rate ($\times 10^{-3}$ g h$^{-1}$ cm$^{-2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>303K</td>
<td>313K</td>
</tr>
<tr>
<td>Blank</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>0.2</td>
<td>28.57</td>
<td>25.00</td>
</tr>
<tr>
<td>0.4</td>
<td>42.85</td>
<td>37.00</td>
</tr>
<tr>
<td>0.6</td>
<td>64.28</td>
<td>50.00</td>
</tr>
<tr>
<td>0.8</td>
<td>71.42</td>
<td>62.50</td>
</tr>
<tr>
<td>1.0</td>
<td>85.71</td>
<td>75.00</td>
</tr>
</tbody>
</table>

3.1.2. Electrochemical Linear Polarization

The values of change in current as a result of applied potential obtained from electrochemical measurements were used in the linear polarization resistance (LPR) technique. After measuring the currents and potentials, a plot of the parameters measured for mild steel as the working electrode immersed in 1.0 M HCl containing different concentrations of TA as inhibitor is presented in Figure 4.
Figure 4. Electrochemical linear polarization plot for mild steel corrosion in 1.0 M HCl in the absence and presence of TA

Table 2. Electrochemical Linear Polarization parameters for mild steel corrosion in 1.0 M HCl in the absence and presence of TA

<table>
<thead>
<tr>
<th>System</th>
<th>$E_{corr}$ (mV)</th>
<th>$j_{corr}$ (μA/cm$^2$)</th>
<th>$\beta_a$ (V/dec)</th>
<th>$\beta_c$ (V/dec)</th>
<th>CR (mm/year)</th>
<th>%IE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank</td>
<td>-1577.20</td>
<td>1404.80</td>
<td>-163.33</td>
<td>63.46</td>
<td>16.3240</td>
<td></td>
</tr>
<tr>
<td>0.2g/L</td>
<td>-8317.60</td>
<td>62.56</td>
<td>162.57</td>
<td>755.23</td>
<td>6.1159</td>
<td>24.50</td>
</tr>
<tr>
<td>0.6g/L</td>
<td>-855.04</td>
<td>66.76</td>
<td>368.84</td>
<td>735.59</td>
<td>5.4297</td>
<td>43.71</td>
</tr>
<tr>
<td>1.0g/L</td>
<td>-843.85</td>
<td>95.17</td>
<td>716.35</td>
<td>484.21</td>
<td>7.8808</td>
<td>88.95</td>
</tr>
</tbody>
</table>

The linear polarization parameters obtained from the measurement are presented in Table 2. The results in the table indicates that the introduction of the various concentrations of TA extracts remarkably shift $E_{corr}$/SCE. For instance, the difference between the $E_{corr}$/SCE of the blank solution and that of the highest concentration of TA is 733.35 mV. It could be
inferred that TA acted as a mixed-type inhibitor and the inhibition is due to simple geometric blocking mechanism [7]. Also, the corrosion current densities of the additives decreased very noticeably compared to the blank. It is clear also from the result (Table 2) that the $\beta_a$ and $\beta_c$ values for the various concentrations of the TA reduced remarkably compared to the blank indicating that the additive simultaneously modified both the anodic and cathodic reactions thus supporting that the extract is a mixed-type inhibitor [8]. The inhibition efficiencies were calculated from the linear polarization data using the relation in equation 5 above.

3. 1. 3. Kinetics/Thermodynamics of the Corrosion Rate constant and half-life

Figure 5 shows the plot of $-\log$ (weight loss) against temperature (K) for mild steel in 1.0 M HCl in the absence and presence of TA. The rate constant parameters; rate constant and half-life obtained from the graph are also recorded in Tables 3. The plots showed a linear variation and slope k, which confirms a pseudo-first order reaction kinetics with respect to the corrosion of mild steel in 1.0 M HCl solution in the absence and Presence of TA. It is evidenced from the result that the half-life decreased with temperature. This further supports the fact that the interaction between the mild steel and the inhibitors is physisorptive [9].

![Figure 5. Variation of -log (weight loss) with temperature (K) for mild steel in 1.0 M HCl in the absence and presence of TA.](image)

The energy of activation ($E_a$), the enthalpy of adsorption ($\Delta H$) and the entropy of adsorption ($\Delta S$) were calculated from the plots of Log CR/T vs. 1/T using equation 8 and 9 respectively [10-11].
The calculated values of activation energy \( (E_a) \), enthalpy of adsorption \( (\Delta H) \), and entropy of adsorption \( (\Delta S) \) are shown in Table 4.

\[
\log \frac{CR_2}{CR_1} = \frac{E_a}{2.303R} \left( \frac{1}{T_1} - \frac{1}{T_2} \right) \quad (6)
\]

\[
CR = \frac{RT}{Nh} \exp \left( \frac{\Delta S_{ads}}{R} \right) \exp \left( \frac{-\Delta H_{ads}}{RT} \right) \quad (7)
\]

where: \( CR_1 \) and \( CR_2 \) are the corrosion rates of mild steel at the temperature \( T_1 \) and \( T_2 \), \( E_a \) is the activation energy, \( R \) is the gas constant, \( N \) is the Avogadro’s number, \( h \) is the plank’s constant, \( T \) is temperature, \( \Delta S_{ads} \) and \( \Delta H_{ads} \) are the entropy and enthalpy of adsorption of the inhibitor on a metal respectively.

**Table 3.** Rate constant and half-life parameter at different concentration

<table>
<thead>
<tr>
<th>System</th>
<th>Rate constant K (day(^{-1}))</th>
<th>Half-life ( t_{1/2} ) (days)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank</td>
<td>0.011</td>
<td>63.00</td>
</tr>
<tr>
<td>0.2g/L</td>
<td>0.013</td>
<td>53.31</td>
</tr>
<tr>
<td>0.4g/L</td>
<td>0.016</td>
<td>39.38</td>
</tr>
<tr>
<td>0.6g/L</td>
<td>0.021</td>
<td>33.00</td>
</tr>
<tr>
<td>0.8g/L</td>
<td>0.023</td>
<td>30.13</td>
</tr>
<tr>
<td>1.0g/L</td>
<td>0.030</td>
<td>23.10</td>
</tr>
</tbody>
</table>

**Table 4.** Thermodynamic activation parameters for the dissolution of mild steel in 1.0 M HCl in the absence and presence of TA at 303-333K

<table>
<thead>
<tr>
<th>System</th>
<th>( E_a ) (KJmol(^{-1}))</th>
<th>( \Delta H ) (Jmol(^{-1}))</th>
<th>( \Delta S ) (KJmol(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank</td>
<td>22.67</td>
<td>0.247</td>
<td>0.285</td>
</tr>
<tr>
<td>0.2g/L</td>
<td>25.96</td>
<td>1.034</td>
<td>0.286</td>
</tr>
<tr>
<td>0.4g/L</td>
<td>30.59</td>
<td>2.815</td>
<td>0.291</td>
</tr>
<tr>
<td>0.6g/L</td>
<td>40.70</td>
<td>3.81</td>
<td>0.292</td>
</tr>
<tr>
<td>0.8g/L</td>
<td>45.85</td>
<td>4.385</td>
<td>0.298</td>
</tr>
<tr>
<td>1.0g/L</td>
<td>60.67</td>
<td>6.165</td>
<td>0.307</td>
</tr>
</tbody>
</table>
The activation energy $E_a$ for the blank was found to be 22.67 KJ mol$^{-1}$ and increased to 60.67 KJ mol$^{-1}$ in the presence of the TA extract which shows that the adsorbed organic matter has provided a physical barrier to charge and mass transfer, leading to reduction in corrosion rate. This value confirmed the assertion that the inhibition of mild steel corrosion by the extract is by physical adsorption mechanism [12]. The activation energy for physical adsorption mechanism should be less than 80 kJ mol$^{-1}$ whereas, the activation energy for chemical adsorption should be greater than 80 KJmol$^{-1}$. The higher value of $E_a$ in the presence of the inhibitor compared to that in the absence of the inhibitor was attributed to physisorption [13].

The positive value of enthalpy of adsorption ($\Delta H$) suggests that the reaction was endothermic and the adsorption of the inhibitor on the metal surface has taken place. The Positive value of entropy of adsorption ($\Delta S$) indicates that the reaction was spontaneous and feasible. Earlier work [14] supports this. Therefore from the above results, it is clear that the inhibitor were physically adsorbed on the mild steel.

### 3.2. Adsorption Isotherm

The inhibition of the corrosion of mild steel in 1.0 M HCl medium with addition of different concentrations of the extract can be explained by the adsorption of the components of the plant extracts on the metal surface [15]. Inhibition efficiency (IE) is directly proportional to the fraction of the surface covered by the adsorbed molecules ($\theta$). Therefore, ($\theta$) with the extract concentration specifies the adsorption isotherm that describes the system and gives the relationship between the coverage of an interface with the adsorbed species and

![Transition state plots for the corrosion of mild steel in the absence and presence of TA](image)
the concentration of species in solution [16]. The Values of the degree of surface coverage (θ) were evaluated at different concentrations of the inhibitors in 1.0 M HCl solution and were fit to various adsorption isotherms. Different adsorption isotherms were tested in order to obtain more information about the interaction between the inhibitors and the mild steel surface. The various isotherms tested includes Temkin, Frumkin, Freundlich and Langmuir adsorption isotherms and linear regression coefficients (r²) were used to determine the best fit. Langmuir adsorption isotherms were found to be best fit in which case all the linear regression coefficients (r²) were close to unity as shown in Figure 7 below.

![Figure 7. Langmuir Isotherm for the Adsorption of TA Stem Extract on Mild steel Surface in 1.0 M HCl at 303 – 333 K.](image)

Gibb’s free energy of adsorption was calculated from the equation 8 and the results obtained for ΔG and K_{ads} are shown in Table 5. The values obtained for ΔG were negative indicating that the adsorption process proceeded spontaneously

\[
\Delta G_{ads} = -2.303RT\log(12.19K_{ads})
\]  

### Table 5. Langmuir adsorption isotherm parameters obtained from the corrosion data for mild steel in 1.0 M HCl containing TA extract.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Intercept</th>
<th>Slope</th>
<th>K_{ads}</th>
<th>R²</th>
<th>ΔG_{ads} (kJ mol⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>313 K</td>
<td>0.6167</td>
<td>2.3214</td>
<td>1.67</td>
<td>0.9796</td>
<td>-7.843</td>
</tr>
<tr>
<td>333 K</td>
<td>0.4933</td>
<td>2.425</td>
<td>1.40</td>
<td>0.9438</td>
<td>-7.855</td>
</tr>
</tbody>
</table>
The negative values of $\Delta G_{\text{ads}}$ indicate spontaneity of the adsorption process and $\Delta G_{\text{ads}}$ values with magnitude $<-40$ kJ mol$^{-1}$ have typically been correlated with electrostatic interactions between inhibitor molecules and charged metal surface (physisorption) [17-19]. Whilst those of magnitude in the order of 40 kJ mol$^{-1}$ and above are associated with charge sharing or transfer from the inhibitor molecules to the metal surface (chemisorption) [20]. From Table 5, it can be observed that evaluated values of $\Delta G_{\text{ads}}$ were negative and $<-20$ kJ mol$^{-1}$ indicating that the adsorption of the extract is spontaneous and that the mechanism of adsorption is physisorption.

3. 3. Synergistic Effect

**Figure 8.** Effect of the addition of 5 mM KI on the corrosion inhibition efficiency of mild steel in the absence and presence of TA extract at 303-333 K.

**Table 6.** Comparison between the inhibition efficiency of the inhibitor in the absence and presence of KI

<table>
<thead>
<tr>
<th>Temp K</th>
<th>TA</th>
<th>TA+ 5 mM KI</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.2</td>
<td>0.4</td>
</tr>
<tr>
<td>303</td>
<td>28.57</td>
<td>42.85</td>
</tr>
<tr>
<td>313</td>
<td>25.00</td>
<td>37.00</td>
</tr>
<tr>
<td>323</td>
<td>22.22</td>
<td>27.77</td>
</tr>
<tr>
<td>333</td>
<td>21.88</td>
<td>25.00</td>
</tr>
</tbody>
</table>
Synergism refers to combined total action of a compound greater than the sum of its individual effects. Synergistic inhibition is an effective means to improve the inhibitive force of inhibitor, to decrease the amount of usage of the inhibitor in acidic media. Synergism (S) of corrosion inhibitors is either due to interaction between components of the inhibitors or due to interaction between the inhibitor and one of the ions present in aqueous solution. Figure 8 shows the effect of potassium iodide (KI) on corrosion inhibition efficiency in 1.0 M HCl in the absence and presence of TA extract at 303, 313, 323, and 333 K respectively. Table 6 compares the inhibition efficiency of the inhibitor in the presence and absence of KI. The highest inhibition efficiency of 85.71% was obtained for only TA while it increased to 98.30% in the presence of TA + KI. The observations clearly established that the addition of KI improved the corrosion inhibition power of TA extract [21].

4. CONCLUSIONS

From the results obtained, it can be concluded that the methanol extract of TA acted as good and efficient inhibitor for the corrosion of mild steel in hydrochloric acid medium. Inhibition efficiency increased with the concentration of the extracts but decreased with rise in temperature. Kinetic treatment showed that the adsorption of the plant extracts to the surface of the mild steel followed pseudo first order rate. The positive values of enthalpy of adsorption suggests that the reaction of the adsorption of the inhibitors on the surface of the metal is an endothermic reaction hence increase in the temperature of the medium will decrease the inhibition efficiency of TA. The positive values of entropy of adsorption indicates that the reaction followed dissociation step and that the reaction was spontaneous and feasible. Increase in activation energy ($E_a$) from 22.67 KJmol$^{-1}$ of the blank to 60.67 KJmol$^{-1}$ of 1.0 g/L inhibitor shows that the adsorbed organic matter has provided a physical barrier to change and mass transfer, leading to reduction in the rate of corrosion. The extracts of TA acted as mixed inhibitors as obtained from the linear polarization result. The adsorption of different concentrations of the plant extract on the surface of the mild steel in 1.0 M HCl followed Langmuir adsorption isotherm. The effect of temperature revealed physical adsorption for the inhibition action of the plant extracts. The negative values of the free energy of adsorption indicates that the adsorption of the inhibitors on the surface of the mild steel was a spontaneous process and was found to be physisorption. The presence of iodide ion synergistically enhanced the inhibition efficiency of TA in 1.0 M HCl.

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References


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