Protecting Mild Steel from Acid Corrosion Using Extract from Ocimum gratissimum Leaves

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Abstract. The anti-corrosion properties of the ethanol extract of Ocimum gratissimum (OG) leaves on the corrosion of mild steel in both 1 M HCl and 0.5 M H\textsubscript{2}SO\textsubscript{4} was studied using gravimetric, electrochemical, Fourier transform Infrared spectroscopy and scanning electron microscopy methods of corrosion monitoring. The gravimetric results revealed that the inhibitor maintained good inhibition efficiency even at 120 hours of immersion. The electrochemical results showed that OG extract functioned as a mixed type corrosion inhibitor for mild steel in both 1 M HCl and 0.5 M H\textsubscript{2}SO\textsubscript{4} inhibiting both the anodic and cathodic partial reactions via adsorption of the extract constituents on the metal/solution interface. Fourier Transform Infrared Spectroscopy and surface examination results confirmed the adsorption of the extract, organic constituents on the mild steel surface. The results showed that OG ethanol extract functioned as a good corrosion inhibitor for mild steel in both 1 M HCl and 0.5 M H\textsubscript{2}SO\textsubscript{4} solutions.

Introduction

Mild steel corrosion has become a source of concern to many researchers for the past few decades. The reason for this is that mild steel finds applications with many industries where acids are used especially in the petro-chemical, oil and gas, nuclear, pulp and paper industries. The best method to protect materials from corrosion attack is to isolate the material from the environment where the corrosion effect takes place. Most effective corrosion inhibitors that are available today are costly, poisonous, and constitute environmental hazards, hence the need for testing materials of plant origin for their anti-corrosion properties [1-10]. These materials are preferred considering the fact that they are save for human consumption, cheap, and are less hazardous to the environment [11-20]. Also these materials of plant origin are known to contain phytochemicals constituents whose molecular and electronic structures resemble those of conventional inhibitors and have been found to possess the ability to reduce metal corrosion attack [21-25]. These inhibitors usually act by preventing close contact between the metal and the corrodent solution by allowing the inhibitor constituents to adsorb themselves on the surface of the metal [26].

Many plant materials have been reported to reduce metal corrosion. Extracts of Brassica oleracea [27], Pyllanthus amarus [28], Carica papaya [29], Tryptamine [30], black pepper [31], khillah [32], Piper guineense [33-35], orphandrine [36], Cassia Auriculata [37] and Vernonia amygdalina [38] have been investigated for their inhibitive effects on different metals and they were found to be effective corrosion inhibitors for the metals studied.

The present study focuses on the study of the anti-corrosion effects of the ethanol extract of Ocimum gratissimum on mild steel corrosion in both 1 M HCl and 0.5 M H\textsubscript{2}SO\textsubscript{4} using gravimetric and electrochemical methods of corrosion monitoring. The adsorption characterization was done with FTIR spectroscopy study and surface examination.
Experimental

Materials preparation

The mild steel used for the experiment contained (weight percent): C – 0.05; Mn – 0.6; p – 0.36; Si – 0.3 and the balance iron. 1 M HCl and 0.5 M H$_2$SO$_4$ solutions were used as the corrodents. Stock solutions of the plant material were made by heating weighed amount of the previously grounded leaves of OG in absolute ethanol for three hours. The solutions were cooled and then filtered with filter paper. The stock solution of the plant extract was quantified by subtracting the weight of the dried residue on the filter paper from the weight of the dried leaves before extracting with absolute ethanol. The desired OG concentrations were prepared in the range of (200-900 mg/L) by diluting the resulting solution with 1 M HCl and 0.5 M H$_2$SO$_4$ respectively.

Weight loss measurements

The dimension of the text coupons used for the gravimetric experiments was 3 cm x 3 cm x 0.14 cm. These metal coupons were polished while wet using silicon carbide abrasive paper (from grade #400 to #1000) [34], washed with distilled water, dried using acetone and exposed to dry air, weighed and kept for future use. The coupons were tired with ropes and suspended in beakers that contained the test solutions. The gravimetric experiments were performed by dipping the prepared mild steel into 300ml of the test solutions at a temperature of 30 ± 1 °C. To determine the weight loss after a given time, the coupons were removed every 24-h continuously for 120 h. When removed, the coupons were placed in 20% NaOH solution containing 200 g/L of zinc dust [34], scrubbed with a brush, washed, dried in acetone, re-weighed and re-immersed into the test solutions.

The weight loss was calculated by subtracting the weight of the coupon at a particular time from the weight of the mild steel before initial immersion [33]. All the tests were done in quadruplicate and average value of the results were used.

Electrochemical experiments

The dimension of the mild steel coupons that were used for the electrochemical experiments was 1 x 1 cm, they were fixed in polytetrafluoroethylene (PTFE) rods by using epoxy resin in a way that just one surface, of area 1 cm$^2$ was exposed [6], and the area that was left open was cleaned as described earlier in the gravimetric section. Potentiodynamic polarization experiments were performed in the potential range of ±250 mV versus corrosion potential at scan rate of 0.33 mV/s. Each test was also run in quadruplicate and the data was reproducible. The electrochemical measurements were performed in a conventional three electrode glass cell of capacity 400 mL using a VERSASTAT 400 Complete DC Voltammetry and corrosion system, with a V$^3$ studio software. The counter electrode that was used in this experiment was a graphite rod and a saturated calomel electrode (SCE) was used as the reference electrode. Electrochemical impedance measurements were performed at corrosion potentials (E$_{corr}$) over a frequency range of 100 KHz to 10 mHz, with a signal amplitude perturbation of 5 mV.

Fourier Transform Infrared Spectroscopy

FTIR spectra (KBr pellet) were carried out by using a Nicolet Magna-IR 560 FTIR spectrophotometer. The spectra of OG powder as well as that of the adsorbed constituents scraped from the mild steel surface after 3 h of immersion in 1 M HCl and 0.5 M H$_2$SO$_4$ solutions containing 900 mg/L OG extract were recorded by scraping the adsorbed constituents, mixing it with KBr, and making the pellets.

Surface Examination

Scanning electron microscopy examination was undertaken on the mild steel surfaces using XL-30FEG scanning electron microscope. Mild steel specimens of dimensions 15 x 15 x 2 mm were cleaned as previously described in the gravimetric section and immersed for 24 h in the blank solutions (1 M HCl and 0.5 M H$_2$SO$_4$) and in solutions containing 900 mg/L OG extract at 30 ±1 °C, cleaned with distilled water, dried using acetone and then subjected to SEM examination.
Results and Discussion

Gravimetric Results: To ascertain the effects of exposure time and concentration on the corrosion inhibition of the test coupons in the presence of ethanol extract of OG immersed in 1 M HCl and 0.5 M H₂SO₄ solutions, weight loss tests were undertaken. Fig. 1 shows the plot of weight loss and time for mild steel corrosion in the absence and presence of OG extract in 1 M HCl (Fig. 1a) and 0.5 M H₂SO₄ (Fig. 1b) solutions. Fig. 2 shows the variation of inhibition efficiency with time for mild steel corrosion in 2(a) 1 M HCl and 2 (b) 0.5 M H₂SO₄ solutions respectively.

Fig. 3 shows the variation of inhibition efficiency with the concentrations of OG extract in (a) 1 M HCl and (b) 0.5 M H₂SO₄ solutions respectively.

Figure 1. Variation of weight loss with time for mild steel corrosion in (a) 1 M HCl and (b) 0.5 M H₂SO₄ without and with OG extract.

Figure 2. Inhibition efficiency vs Time for mild steel corrosion in (a) 1 M HCl and (b) 0.5 M H₂SO₄
From Fig. 1, it is clear that OG extract retarded the corrosion of the test coupons in both acid environments. The result shows that weight loss increased steadily with increase in immersion time indicating that shorter immersion time is needed to achieve good results. This result is in good agreement with that reported by Oguzie et al. [34]. Fig. 2 indicates that inhibition efficiency decreased with exposure time and that efficiency did not fall below 40% even at 120 h of immersion in 1 M HCl and 0.5 M H₂SO₄. Fig. 3 revealed that inhibition efficiency increased steadily with increase in concentration of OG extract. The trend of efficiency reasonable agrees with that reported by Ikpi et al. [35].

The inhibition efficiency from the gravimetric data was calculated with the formula below, where (∆W_blank) is the weight loss without OG extract and (∆W_inh) is the weight loss in the presence of OG extract.

\[ IE\% = \left( 1 - \frac{\Delta W_{inh}}{\Delta W_{blank}} \right) \times 100 \]  

**Potentiodynamic Polarization Results**

Potentiodynamic polarization experiments were undertaken to give insight into the effect of OG extract on the anodic as well as the cathodic half reactions. Typical potentiodynamic polarization plots for mild steel corrosion in 1 M HCl and 0.5 M H₂SO₄ in the absence and presence of OG extract are given in Fig. 4 (a) 1 M HCl and (b) 0.5 M H₂SO₄ respectively while the parameters from the potentiodynamic polarization curves are shown in Table 1.

From Table 1, it is clear that increase in OG concentration affected the anodic as well as the cathodic partial reactions. OG extract can be seen to have shifted the corrosion potential (E_corr) slightly towards more positive (anodic) region in both 1 M HCl and 0.5 M H₂SO₄ solutions. OG extract can also be seen to reduce the anodic as well as cathodic current densities and the corresponding corrosion current density (i_corr) indicating that the inhibitor modified the cathodic reduction of hydrogen ion [39, 40] and also the anodic dissolution of the metal hence OG extract functioned as a mixed-type corrosion inhibitor in both acids.

The values of the inhibition efficiencies from the polarization data were calculated with the formula below:

\[ IE\% = \left( \frac{i_{corr(bi)} - i_{corr(inh)}}{i_{corr(bi)}} \right) \times 100 \]  

where \(i_{corr(bi)}\) and \(i_{corr(inh)}\) represent the corrosion current density when no OG was added and when different concentrations of OG were added respectively.
Figure 4. Potentiodynamic polarization curves for mild steel corrosion in (a) 1 M HCl and (b) 0.5 M H$_2$SO$_4$ solutions without and with OG extract.

Table 1. Potentiodynamic polarization parameters for mild steel corrosion in 1 M HCl and 0.5 M H$_2$SO$_4$ with and without OG extract.

| System (mg/L) | E mV (vs SCE) | $i_{corr}$ (µA/cm$^2$) | IE% |
|---------------|---------------|------------------------|-----|
| 1 M HCl       | -508.3        | 456.8                  |     |
| 900 OG        | -463.7        | 34.7                   | 92.4|
| 0.5 M H$_2$SO$_4$ | -639.2      | 3570                   |     |
| 900 OG        | -470.8        | 813.7                  | 77.2|

Electrochemical Impedance Spectroscopy Results

The behavior of the test coupons in 1 M HCl and 0.5 M H$_2$SO$_4$ in the absence and presence of OG extract was investigated using the EIS method. The electrochemical impedance tests were performed to give information on the kinetic behavior of the corrosion processes. The Nyquist plots for mild steel corrosion are presented in Fig. 5(a) in 1 M HCl without and with OG extract and Fig. 5(b) in 0.5 M H$_2$SO$_4$ in the absence and presence of OG extract.

From Fig. 5 it is clear that there was a gradual increase in the diameter of the semicircles of the Nyquist plots on the addition of OG extract. The high frequency intercept with the real axis that was found in the Nyquist plots is called the solution resistance (Rs) and the low frequency intercept with the real axis is the charge transfer resistance (Rct). The impedance spectra were fitted into an equivalent circuit model $R_s(Q_{dl}R_{ct})$ using the ZSimpWin software, this circuit has been successfully used to model impedance spectra by other researchers [41, 42]. In this equivalent circuit, the solution resistance (Rs) is connected with a constant phase element (CPE) which was placed in parallel with the charge transfer resistance (Rct). The CPE was used instead of a capacitor to give room for shifts from ideal dielectric activity that may arise due to the differences in the nature of the electrode surfaces.
The electrochemical parameters were determined using the ZSimpWin software and are presented in Table 2. The results revealed that increase in OG concentration increased the value of $R_{ct}$ while decreasing the double layer capacitance ($C_{dl}$). This increase in the $R_{ct}$ values as the concentration increases resulted in the increase of the diameter of the Nyquist semicircle. This confirms that OG extract reduced mild steel corrosion in 1 M HCl and 0.5 M H$_2$SO$_4$. The decrease in $C_{dl}$ values which usually comes from a reduction in the dielectric constant and/or a rise in the double layer thickness shows that OG extract constituents (which has a dielectric constant smaller that of the water molecule that was displaced) are attached on the test coupon surface hence protecting it from corrosion attack. This can be accounted for using the Helmholtz equation below:

$$C_{dl} = \varepsilon \varepsilon_0 A / d \quad (3)$$

Here, $\varepsilon_0$ represents the vacuum permittivity, $\varepsilon$ stands for the dielectric constant of the system and $C_{dl}$ is the double-layer capacitance, $A$ stands for the electrode area and $d$ is the thickness of the protective layer. The value of the ($C_{dl}$) was calculated using the formula below:

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}} \quad (4)$$

where $f_{max}$ stands for the frequency at which the imaginary component is maximum and $R_{ct}$ stands for the charge transfer resistance.

Inhibition efficiency from the impedance data was calculated using the formula below:

$$IE\% = \left(\frac{R_{ct,bl} - R_{ct,inh}}{R_{ct,bl}} \times 100\right) \quad (5)$$

where $R_{ct,bl}$ and $R_{ct,inh}$ represent the charge transfer resistance values in the uninhibited and inhibited systems respectively.

Table 2. Impedance Results for mild steel Corrosion in 1 M HCl and 0.5 M H$_2$SO$_4$ without and with OG 1 M HCl extract.

| System (mg/L) | $R_{ct}$ ($\Omega \text{cm}^2$) | $C_{dl}$ ($\mu \Omega^{-1}\text{S} \text{cm}^{-2} \times 10^{-6}$) | IE% |
|---------------|------------------|-----------------|-----|
| 1 M HCl       | 23.46            | 13.5            |     |
| 900 (HCl)     | 470.2            | 2.69            | 95.0|
| 0.5 M H$_2$SO$_4$ | 9.788          | 12.9            |     |
| 900 (H$_2$SO$_4$) | 257.8           | 1.95            | 96.2|
Adsorption consideration

The adsorption behavior of the 1 M HCl extract of OG was studied by fitting the experimental data into different adsorption isotherms; it was found that the best fit isotherm was the Langmuir adsorption isotherm as determined from the gravimetric measurement after 24 h of immersion.

The equation for the Langmuir adsorption isotherm is given as follows:

\[
\frac{C}{\theta} = \frac{1}{b} + C,
\]

where \( C \) is the inhibitor concentration, \( b \) stands for the equilibrium constant and \( \theta \) is the fraction of the surface covered by adsorbed molecules called the degree of surface coverage.

The value of \( \theta \) was calculated from the inhibition efficiency (IE) with the equation below:

\[
\theta = \frac{IE}{100}\quad(7)
\]

The plot of \( \frac{C}{\theta} \) versus \( C \) is shown in Fig. 6 to be linear for OG extract in (a) 1 M HCl and (b) 0.5 M H\(_2\)SO\(_4\) solutions respectively.

**Figure 6.** Langmuir adsorption isotherms for OG extract on mild steel in (a) 1 M HCl and (b) 0.5 M H\(_2\)SO\(_4\) solutions.

Table 3 gives the adsorption parameters from the Langmuir adsorption isotherm plots. The values of the slopes and \( R^2 \) suggest that the adsorption of OG extracts followed the Langmuir adsorption isotherm in both acids showing that the extract constituents actually adsorbed themselves on the mild steel surface. The deviation of the resultant slopes from the expected value of 1 may be as a result of the interaction between OG constituents as well as changes in the heat of adsorption with increasing surface coverage which may have been ignored while deriving the Langmuir isotherm [44-45].

**Table 3.** Adsorption parameters from the Langmuir adsorption isotherm plots for mild steel corrosion in 1 M HCl and 0.5 M H\(_2\)SO\(_4\).
Fourier Transform Infrared Spectroscopy

The FTIR spectra for OG powder was recorded, also recorded was that of the adsorbed films removed from the surface of the metal after 3 h of immersion in 1 M HCl and 0.5 M H₂SO₄ solutions containing 900 mg/L OG extract. Fig. 7 represents the FTIR spectra.

![FTIR spectra](image)

**Figure 7.** FTIR spectra of (a) OG powder and that of surface film on mild steel specimen immersed in (b) 1 M HCl and (c) 0.5 M H₂SO₄ solution containing OG extract.
The peaks for OG can be seen to have multiplied showing that the extract contained a complex mixture of constituents. Also some of the peaks found in the spectra of OG powder were seen to have appeared in the spectra of the adsorbed constituents scraped from the surface of the mild steel after 3 h of immersion in both 1 M HCl and 0.5 M H₂SO₄. The major peaks observed in the spectra of OG powder are those around 3389.5 cm⁻¹, 1177.8 cm⁻¹, 1036.2 cm⁻¹, and 719.4 cm⁻¹ which correspond to O-H, C-N and C-H functional groups. The C-H functional group around 2855.1-2922.2 cm⁻¹ can be seen to have reappeared in both 1 M HCl and 0.5 M H₂SO₄ showing that they are directly involved in the adsorption of the OG constituents on the mild steel surface. Some peak can be seen to have shifted. For instance the peak for C–N around 1177.8 shifted to 1174.1 cm⁻¹ in both 1 M HCl and 0.5 M H₂SO₄ while peaks around 887.1-719.4 cm⁻¹ disappeared showing that they were not involved in the adsorption process.

**Scanning Electron Microscopy**

The surfaces of the mild steel specimens before corrosion and after 3-h of immersion in 1 M HCl and 0.5 M H₂SO₄ blank solutions were studied by SEM examination followed by SEM study after 3-h immersion in 1 M HCl and 0.5 M H₂SO₄ solutions containing 900 mg/L OG extract. Fig. 8 shows the SEM image of the mild steel specimen before corrosion in the acidic solutions. Fig. 9 shows the corresponding images after 3 h immersion in 1 M HCl without OG extract (Fig. 9a) and with 900 mg/L OG extract (Fig. 9b) while Fig. 10a and 10b show similar images for mild steel in 0.5 M H₂SO₄ solution without and with OG extract.

**Figure 8.** SEM image of mild steel surface before corrosion in acidic solutions.

**Figure 9.** SEM images of mild steel surface after 3 h in 0.5 M 1 M HCl (a) without OG extract and (b) with 900 mg/L OG extract.
Figure 10. SEM images of mild steel surface after 3 h in 0.5 M H$_2$SO$_4$ (a) without OG extract and (b) with 900 mg/L OG extract.

The SEM results showed a rough surface morphology after immersion in the uninhibited acids, this may be because the mild steel surface has dissolved in the uninhibited acids (Fig. 9a and 10a). With the addition of OG extract, the corrosion damage is seen to have reduced, and there is also evident of adsorbent species on mild steel surface in both acid environments, which confirms the adsorption of OG constituents.

Conclusions

The results of this study have revealed that ethanol extract of *Ocimum gratissimum* (OG) exhibited corrosion inhibiting effect on the corrosion of mild steel in both 1 M HCl and 0.5 M H$_2$SO$_4$ solutions.

The gravimetric results revealed that OG extract is a good corrosion inhibitor for mild steel corrosion in both 1 M HCl and 0.5 M H$_2$SO$_4$ even at longer exposure time.

The polarization results showed that OG extract is a mixed type corrosion inhibitor for mild steel corrosion in both 1 M HCl and 0.5 M H$_2$SO$_4$ inhibiting both the anodic and cathodic partial reactions.

Impedance data, Fourier transform infrared spectroscopy and scanning electron microscopy results revealed that OG extract functioned by the adsorption of the extract constituents on the mild steel surface.

Conflict of Interest

The authors declare no conflict of interest regarding the submission of this paper

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