Mild Steel Corrosion Inhibition by *Eremomastax polysperma* Leaf Extract in Acidic Medium

Okon U. Abakedi† and James E. Asuquo†

†Department of Chemistry, University of Uyo, P.M.B.1017, Uyo, Nigeria.

Authors’ contributions

This work was carried out in collaboration between both authors. Author OUA designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Author JEA managed the analyses of the study. Authors OUA and JEA managed the literature searches. Both authors read and approved the final manuscript.

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ABSTRACT

The inhibition of mild steel corrosion in 1 M H₂SO₄ solution by *Eremomastax polysperma* leaf extract has been studied using weight loss and hydrogen evolution techniques. The results obtained show that *Eremomastax polysperma* leaf extract is a good inhibitor of mild steel corrosion in H₂SO₄ solution. The inhibition efficiency increased with increase in extract concentration but decreased with increase in temperature. Physical adsorption has been proposed for the adsorption of the leaf extract on mild steel surface. Thermodynamic parameters reveal that the corrosion inhibition process in the presence of the extract was endothermic and occurred spontaneously. The adsorption of *Eremomastax polysperma* leaf extract on mild steel surface best fitted the Langmuir adsorption isotherm.

Keywords: Corrosion inhibition; *Eremomastax polysperma*; mild steel; physisorption; weight loss; hydrogen evolution; Langmuir isotherm.

*Corresponding author: E-mail: ouabakedi@yahoo.com*
1. INTRODUCTION

The present day high operational cost of extracting metals from their ores has made it imperative to protect existing metallic structures from corrosion. Corrosion weakens the mechanical strength of metals leading to equipment breakdown or failure [1]. The use of corrosion inhibitors for the protection of metals in aggressive media is a standard practice globally. Compounds containing nitrogen, sulphur, oxygen and/or phosphorous have been reported as good inhibitors of mild steel corrosion in acidic media [2-6]. The use of traditional inhibitors (i.e. synthesised organic and inorganic compounds) is now limited due to environmental safety concerns, as some of them are toxic and environmentally - unfriendly. The desire for biodegradable, cheap, renewable and environmentally - friendly corrosion inhibitors has led to the extraction of inhibitors from natural products, especially of plant origin. Plant extracts contain phytochemicals such as saponins, alkaloids, terpenes, tannins, etc, which are rich sources of organic nitrogen, sulphur and oxygen. Many workers have reported high inhibition efficiencies of mild steel corrosion by leaves extracts in acidic medium [1,7-10]. Unfortunately, some of the reported plants are known to grow only in certain regions of the world. Importation of extracts of such plants for use in other regions of the world is not only expensive but time consuming. Hence, the need for the local sourcing of efficient eco-friendly corrosion inhibitors cannot be over-emphasised.

Eremomastax polysperma is a medicinal plant in the family Acanthaceae. It is locally called Edem iduduot by the Efik and Ibibio speaking people of Nigeria. The traditional medicinal uses of the plant in eastern Nigeria have been documented [11–13]. The phytochemical screening of Eremomastax polysperma leaf extract showed the presence of tannins, phenol, flavonoid, alkaloid, saponin and sterol [14].

The aim of this work was to evaluate the inhibitory effect of Eremomastax polysperma leaf extract on mild steel corrosion in H₂SO₄ solution. H₂SO₄ solution is widely used industrially in the pickling of iron and steel because it is less fumy than other mineral acids. Additionally, the choice of 30°C – 60°C for this work was to simulate the temperature for the pickling process.

2. MATERIALS AND METHODS

2.1 Test Materials

Mild steel sheet used for this work was obtained in Calabar, Nigeria. It had the following chemical composition (weight %): C (0.12), Mn (0.85), S (0.06), P (0.05), Si (0.09) and Fe (98.83). The sheet was mechanically press-cut into 4 cm x 5 cm coupons, and polished to mirror finish using different grades of silicon carbide papers. The coupons were degreased in absolute ethanol, dipped in acetone before air-drying. They were then stored in a moisture - free desiccator before use in corrosion studies.

2.2 Preparation of Eremomastax polysperma Leaf Extract

Fresh leaves of Eremomastax polysperma were collected from a farm in Nung Oku Ibesikpo, Akwa Ibom State, Nigeria and authenticated by a plant taxonomist in the Department of Botany and Ecological Studies, University of Uyo, Nigeria. They were washed and air-dried at 30°C for seven days. They were then ground to powder. The dried ground sample of Eremomastax polysperma leaves was macerated with 90% ethanol for seven days at room temperature in a glass trough with cover. The mixture was then filtered. The filtrate was evaporated at 40°C in a water bath to constant weight, leaving a dark green extract (paste) in the beaker.

2.3 Weight Loss Method

Previously weighed mild steel coupons were suspended with the aid of glass hooks and rods and immersed in 100 ml of 1 M H₂SO₄ solution (blank) and in 1 M H₂SO₄ solution containing 1.0 g/L – 4.0 g/L Eremomastax polysperma leaf extract (inhibitor) in open beakers. In each experiment, one mild steel coupon per beaker was used. The beakers were then placed in a thermostatic water bath maintained at 30°C, 40°C, 50°C, and 60°C, respectively. The mild steel coupons were retrieved from the test solutions after four (4) hours and scrubbed with bristle brush under running water. They were then reweighed and used to compute the inhibition efficiency, I(%) [15]:
where \( W_0 \) and \( W_1 \) are the weight losses of the mild steel coupons in the absence and presence of extract, respectively, in 1 M H\(_2\)SO\(_4\) at the same temperature.

The corrosion rate (CR) was calculated using the formula [16]:

\[
CR \ (mg \ cm^{-2} hr^{-1}) = \left(\frac{W}{A}\right)
\]

where \( W \) is the weight loss (mg), \( A \) is the total surface area (cm\(^2\)) while \( t \) is the exposure time (hours).

### 2.4 Hydrogen Evolution Method

The reaction vessel and procedure for measuring the corrosion process by this method are as described in literature [17]. A 100 cm\(^3\) of 1 M H\(_2\)SO\(_4\) solution was introduced into the reaction vessel connected to a burette through a delivery tube. The initial volume of air in the burette was recorded. Two mild steel coupons weighing 8.0 g were dropped into the 1 M H\(_2\)SO\(_4\) solution and the reaction vessel quickly closed to prevent any escape of hydrogen gas. The volume of H\(_2\) gas evolved from the corrosion reaction was monitored by the depression (in cm\(^3\)) in the paraffin oil level. The depression in paraffin oil level was monitored every 60 seconds for 100 minutes. The same experiment was repeated in the presence of 1.0 g/L – 4.0 g/L *Eremomastax polysperma* leaf extract in 1 M H\(_2\)SO\(_4\) solution.

The inhibition efficiency \( I(\%) \) was calculated using the equation [18]:

\[
I(\%) = \left(\frac{V_0 - V_1}{V_0}\right) \times 100
\]

where \( V_0 \) and \( V_1 \) are the volumes of H\(_2\) gas evolved in the absence and presence of inhibitor, respectively, at a specified time.

### 3. RESULTS AND DISCUSSION

#### 3.1 Effect of *Eremomastax polysperma* Leaf Extract Concentration on Inhibition Efficiency

Fig. 1 shows that at a particular temperature the inhibition efficiency increased with increase in the concentration of *Eremomastax polysperma* leaf extract. The maximum inhibition efficiency of the leaf extract was 90.42% at extract concentration of 4.0 g/L at 30°C. An increase in inhibition efficiency with increase in inhibitor concentration indicates a strong interaction between the mild steel surface and the inhibitor [19].

![Fig. 1. Effect of *Eremomastax polysperma* leaf extract concentration on the inhibition efficiency of mild steel corrosion in 1 M H\(_2\)SO\(_4\) at different temperatures](image-url)
3.2 Hydrogen Evolution Measurements

Fig. 2 illustrates the effect of *Eremomastax polysperma* leaf extract on the volume of H\(_2\) gas evolved in the corrosion of mild steel in 1 M H\(_2\)SO\(_4\). It is observed that, at a given time, as the leaf extract concentration increases, the volume of H\(_2\) gas evolved decreases. This indicates that the extract inhibits the corrosion process by adsorbing on the metal surface, forming protective thin films which reduce/stop the electron transfer process on the metal surface [20]. Table 1 contains the calculated values of inhibition efficiency for the inhibition process containing *Eremomastax polysperma* leaf extract. Table 1 reveals that the inhibition efficiency increased with increase in the concentration of *Eremomastax polysperma* leaf extract. The inhibition efficiencies obtained by both the weight loss and hydrogen evolution methods followed a similar trend.

3.3 Effect of Temperature on Inhibition Efficiency

Table 2 reveals an increase in the corrosion rate of mild steel in H\(_2\)SO\(_4\) solution with increase in temperature. This shows that the reactants acquired more energy and overcame the activation energy barrier more easily with an increase in temperature than at lower temperatures. Additionally, Table 2 shows that the inhibition efficiency decreased with increase in temperature at the extract concentrations studied. This indicates weakening of adsorption bonds between mild steel surface and inhibitor as well as a physical adsorption process [21].

![Fig. 2. Variation of volume of H\(_2\) gas evolved (cm\(^3\)) with time (min) for mild steel corrosion in 1 M H\(_2\)SO\(_4\) in the absence and presence of *Eremomastax polysperma* leaf extract at 30°C](image)

Table 1. Effect of *Eremomastax polysperma* leaf extract concentration on inhibition efficiency of mild steel in 1 M H\(_2\)SO\(_4\) solution at 30°C (Hydrogen evolution measurements)

| Extract concentration (g/L) | Volume of H\(_2\) evolved (cm\(^3\)) | Time taken (min) | Inhibition efficiency (%) |
|-----------------------------|-------------------------------------|------------------|--------------------------|
| Blank                       | 48.00                               | 100              | -                        |
| 1.0                         | 17.70                               | 100              | 63.1                     |
| 2.0                         | 12.50                               | 100              | 74.0                     |
| 3.0                         | 10.20                               | 100              | 79.0                     |
| 4.0                         | 9.00                                | 100              | 81.0                     |
Table 2. Calculated values of corrosion rate and inhibition efficiency for mild steel corrosion in 1 M H₂SO₄ in the absence and presence of different concentrations of *Eremomastax polysperma* leaf extract

| Extract conc. (g/L) | Weight loss (g) | Corrosion rate (mg cm⁻² hr⁻¹) | Inhibition efficiency (%) |
|--------------------|----------------|-------------------------------|--------------------------|
|                    | 30°C | 40°C | 50°C | 60°C | 30°C | 40°C | 50°C | 60°C | 30°C | 40°C | 50°C | 60°C |
| 1M H₂SO₄           | 0.355 | 0.454 | 0.853 | 1.604 | 2.219 | 2.838 | 5.331 | 10.025 | -   | -   | -   | -   |
| 1.0                | 0.071 | 0.094 | 0.394 | 0.764 | 0.445 | 0.588 | 2.463 | 4.775 | 80.0 | 79.3 | 53.8 | 52.4 |
| 2.0                | 0.054 | 0.086 | 0.258 | 0.672 | 0.338 | 0.538 | 1.613 | 4.200 | 84.8 | 81.1 | 69.8 | 58.1 |
| 3.0                | 0.043 | 0.065 | 0.235 | 0.513 | 0.269 | 0.406 | 1.469 | 3.206 | 87.9 | 85.7 | 72.5 | 68.0 |
| 4.0                | 0.034 | 0.047 | 0.194 | 0.443 | 0.213 | 0.294 | 1.213 | 2.769 | 90.4 | 89.6 | 77.3 | 72.4 |

The values of the activation energy (Eₐ) for mild steel corrosion in 1 M H₂SO₄ solution in the presence and absence of *Eremomastax polysperma* leaf extract, respectively, were obtained using the alternative formulation of Arrhenius equation [20]:

\[
\ln CR = \frac{-E_a}{RT} + \ln A
\]

(4)

where CR is the corrosion rate, R is the universal gas constant, T is the absolute temperature while A is the pre-exponential factor.

The activation energies (Eₐ) of mild steel corrosion in 1M H₂SO₄ solution, with and without inhibitors, were obtained from the gradients of ln CR vs. 1/T plots (Fig. 3) and the results are presented in Table 3. Table 3 shows that the Eₐ values in the presence of the leaf extract were higher than the Eₐ value of the blank (42.8578 kJ mol⁻¹). The increase in the Eₐ values in the presence of the extract indicates physical adsorption while the reverse signifies chemical adsorption [22,23].

The values of enthalpy of activation (ΔH°ₐds) and entropy of activation (ΔS°ₐds) were obtained from an alternative formulation of the transition state equation [24]:

\[
\ln \left( \frac{CR}{T} \right) = \ln \left( \frac{R}{NhN} \right) + \frac{\Delta S°ₐds}{R} - \frac{\Delta H°ₐds}{RT}
\]

(5)

where CR is the corrosion rate, T is the absolute temperature, R is the universal gas constant, h is the Planck’s constant, and N is the Avogadro’s number. Fig. 4 shows linear plots of ln (CR/T) vs. 1/T with gradients of (-ΔH°ₐds/R) and intercepts of [ln (R/Nh) + ΔS°ₐds/R] from which the values of ΔH°ₐds and ΔS°ₐds were calculated and listed in Table 3. The positive values of ΔH°ₐds indicate that mild steel corrosion inhibition was an endothermic process. Additionally, the negative values of ΔS°ₐds show a decrease in the disorderliness of the system, indicating an ordered layer of extract on mild steel surface.

### 3.4 Adsorption Isotherm

The best fit for the adsorption of *Eremomastax polysperma* leaf extract on mild steel surface was obtained by the modified Langmuir isotherm defined as:

\[
\frac{C}{\theta} = nC + \frac{n}{K_{ads}}
\]

(6)

where C is the inhibitor concentration, θ is the degree of surface coverage while K_ads is the equilibrium adsorption constant. The degree of surface coverage of mild steel by *Eremomastax polysperma* leaf extract is presented in Table 4. It is noticed that the degree of surface coverage increased with increase in extract concentration but decreased with increase in temperature. Linear plot of C/θ vs. C (Fig. 5) shows that the adsorption of *Eremomastax polysperma* leaf extract on mild steel surface in 1 M H₂SO₄ solution obeyed the Langmuir adsorption isotherm. The values of K_ads were evaluated from the intercept of the graph and presented in Table 5. K_ads is related to the standard free energy of adsorption (ΔG°ₐds) by the formula [25]:

\[
\Delta G°ₐds = -RT \ln (55.5K_{ads})
\]

(7)

where 55.5 is the molar concentration of water in the solution in mol dm⁻³.

The negative values of ΔG°ₐds reveal that the mild steel corrosion inhibition process by *Eremomastax polysperma* leaf extract occurred spontaneously. Generally, values of ΔG°ₐds less negative than -20 kJ mol⁻¹ indicate physical adsorption while those more negative than -40 kJ...
mol⁻¹ indicate chemical adsorption [26-28]. Consequently, the values of ΔG°_{ads} obtained in this work being less negative than -20 kJ mol⁻¹ coupled with a decrease in the inhibition efficiency with increase in temperature indicates a physical adsorption process.

![Fig. 3. Plot of ln CR vs. 1/T (Arrhenius plot) for mild steel corrosion in 1 M H₂SO₄ in the absence and presence of Eremomastax polysperma leaf extract](image)

![Fig. 4. Plot of ln (CR/T) vs. 1/T (transition state plot) for mild steel corrosion in 1 M H₂SO₄ solution in the absence and presence of Eremomastax polysperma leaf extract](image)

Table 3. Calculated values of thermodynamic parameters for mild steel corrosion in 1 M H₂SO₄ solution in the absence and presence of Eremomastax polysperma leaf extract

| Extract concentration | Eₐ (kJ mol⁻¹) | ΔH°_{ads} (kJ mol⁻¹) | ΔS°_{ads} (J K⁻¹ mol⁻¹) |
|-----------------------|--------------|---------------------|------------------------|
| 1M H₂SO₄ (Blank)      | 42.9         | 40.2                | -106.6                 |
| 1.0 g/L               | 71.1         | 68.5                | -27.4                  |
| 2.0 g/L               | 72.0         | 69.4                | -26.4                  |
| 3.0 g/L               | 72.5         | 69.9                | -26.6                  |
| 4.0 g/L               | 75.8         | 73.2                | -18.1                  |
Table 4. Calculated values of degree of surface coverage of mild steel by *Eremomastax polysperma* leaf extract in 1 M H$_2$SO$_4$ solution

| Extract concentration | Degree of surface coverage (θ) |
|-----------------------|--------------------------------|
|                       | 30°C | 40°C | 50°C | 60°C |
| 1.0 g/L               | 0.80 | 0.79 | 0.54 | 0.52 |
| 2.0 g/L               | 0.85 | 0.81 | 0.70 | 0.58 |
| 3.0 g/L               | 0.88 | 0.86 | 0.72 | 0.68 |
| 4.0 g/L               | 0.90 | 0.90 | 0.77 | 0.72 |

Fig. 5. Plot of C/θ vs. C (Langmuir isotherm) for mild steel corrosion in 1 M H$_2$SO$_4$ solution containing *Eremomastax polysperma* leaf extract

Table 5. Some parameters of the linear regression of Langmuir adsorption isotherm for mild steel corrosion in 1 M H$_2$SO$_4$ solution containing *Eremomastax polysperma* leaf extract

| Temperature | $R^2$  | n     | 1/$K_{ads}$ (g L$^{-1}$) | $K_{ads}$ (g$^{-1}$L) | $ΔG^{°}_{ads}$ (kJ mol$^{-1}$) |
|-------------|--------|-------|--------------------------|-----------------------|-------------------------------|
| 303K        | 0.9996 | 1.06  | 0.22                     | 4.60                  | -14.0                         |
| 313K        | 0.9973 | 1.06  | 0.26                     | 3.79                  | -13.9                         |
| 323K        | 0.9980 | 1.12  | 0.70                     | 1.42                  | -11.7                         |
| 333K        | 0.9902 | 1.18  | 0.88                     | 1.15                  | -11.5                         |

4. CONCLUSION

On the basis of this study, the following conclusions could be drawn: *Eremomastax polysperma* leaf extract appreciably inhibited the corrosion of mild steel in H$_2$SO$_4$ solution. The inhibition efficiency increased with increase in *Eremomastax polysperma* leaf extract concentration but decreased with increase in temperature. Based on a decrease in inhibition efficiency with temperature, higher $E_a$ values in the extract compared to the blank and the $ΔG^{°}_{ads}$ values being less negative than -20 kJ mol$^{-1}$, physically adsorption has been proposed for the adsorption of *Eremomastax polysperma* leaf extract onto mild steel surface. The adsorption of the extract obeyed the Langmuir adsorption isotherm.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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