Corrosion Inhibition Efficiency of *Terminalia Catappa* Leaves Extracts on Stainless Steel in Hydrochloric Acid

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Abstract-

Corrosion inhibition potential of *Terminalia catappa* leaves extract was investigated using Grade 304 austenitic stainless steel in 1.0 M HCl solution with a view to finding natural, eco-friendly, low-cost and readily available corrosion inhibitor. The corrosion inhibitory characteristics of the extract were investigated by utilizing gravimetric measurements and microstructural surface changes obtained from scanning electron microscopy (SEM). This study was carried out in the absence and presence of *Terminalia catappa* inhibitor (0.01-0.09 g/ml) and temperatures (30-50 °C). Data obtained from weight loss experiments and adsorption isotherms (El-Awady, Temkin, and Freundlich) indicated that *Terminalia catappa* leaf extract repressed the corrosion of stainless steel in acidic media by 96.8 % at 30 °C and conforms to physisorption adsorption mechanism. Inhibition efficiency (% IE) of the extract improved with increasing extract concentration but declined with rising temperature.

Keywords: Adsorption; corrosion inhibition; eco-friendly inhibitor; stainless steel; *Terminalia catappa*.

1. Introduction

Many industries use equipment made from metals under varied conditions ranging from mild to harsh chemical environments, making their surfaces susceptible to corrosion [1,2]. Studies have shown that corrosion cannot be completely eliminated from metal surfaces because of the varied environments in which metals are used [3]. Corrosion affects numerous industries in different sections of the economy and is usually given a multi-dimensional assessment, ranging from economic view (cost, loss, downtime of machinery and profits) to the protection of employees and environmental safety in the event of its occurrence [4,5]. Thus, corrosion is a ubiquitous problem that continues to cause apprehension in a wide range of application in industries. Numerous efforts are being made to have a long and trouble-free operation by minimizing the rate of corrosion of metals used for industrial applications.

Numerous economic and practical strategies are used to decrease the corrosion rate of metals yet, mitigating corrosion remains an enormous and perplexing task that industries confront as costs are incurred in the form of designing machines that are resistant to corrosive media and create an
The environment that is safe to human life by minimizing breakage and explosion resulting from corrosion [6,7]. Strategies currently utilized in mitigating corrosion ranges from the use of inorganic and organic materials as inhibitors, polymeric compounds, synthetic materials, coatings by electro-deposition, anodizing, electrophoresis to naturally occurring corrosion inhibitors such as those present in plants [8-10]. Among these methods of corrosion mitigation, the use of corrosion inhibitors has been described as the most efficient and feasible method of reducing the effect of corrosive agents on metal surfaces [9,11].

Corrosion inhibitors can be synthetic or natural chemicals which when present even in minuscule amounts in a corrosive medium decreases the corrosion rates of metals. However, the use of synthetic inhibitors in several applications has raised sundry questions regarding safety, toxicity, public health (injury to organs like kidneys and liver, and disturbances of biochemical processes including enzymes have been reported) and environmental friendliness among other concerns [12]. The parlous aftermaths of using many synthetic inhibitors have become one of the stimuli driving the research and development of natural corrosion inhibitors. Thus, the use of synthetic corrosion inhibitors has come under stringent regulation making most researchers to favor the exploration of natural corrosion inhibitors. Hence, there is a renewed effort to discovering natural products especially plant extracts with potentials as green anticorrosive agents [10,12].

Globally, scientists have reported several extracts from plants as promising corrosion inhibiting agents [13,14]. This class of corrosion inhibitors possesses abundant advantages in that they are usually innocuous substances, inexpensive, environmental-friendly, efficient inhibitors and are from sustainable sources [15]. Some plants extracts are endowed with valuable corrosion inhibitory properties. These anticorrosive properties have been attributed to some phytochemicals present in these plants. However, the vast majority of plants in our environment have not yet been investigated for their anti-corrosive activity. Several works have demonstrated the corrosion repressing activities of plant extracts in acid, salt and alkaline solutions [16,17].

Although stainless steels are corrosion resistant, they can be susceptible to corrosion in the presence of halide compounds such as NaCl and HCl [18]. Therefore, several researchers have investigated corrosion inhibition capabilities of plant extracts on stainless steels in halide containing media [19]. The corrosion inhibition of Salvia officinalis leaves extract on 304 stainless steel in HCl solution was investigated by Soltani et al., [20] by means of weight loss, potentiodynamic polarization, and electrochemical impedance spectroscopic techniques. They varied the inhibitor concentrations from 0.5 to 2.5 g/L and temperatures from 25 to 65 °C and obtained a maximum inhibition efficiency of 96.6% with 2.5g/L concentration, and a physisorption inhibition mechanism was observed.

Tropical almond (Terminalia catappa), a plant that is abundant in Sub-Saharan Africa and other subtropical areas. It is grown mainly for ornamental purposes and previous research have established that the leaves and seeds possess high antioxidant activities [21,22]. Antioxidant activity increases the likelihood that the plant will be a potent corrosion inhibitor. Omotosho et al., [23], studied the corrosion inhibition of stainless steels in 0.5 M H₂SO₄ using Cassia fistula leaves extract. An efficiency of 98.59 % was recorded from a 4 g/l concentration of inhibitor and the Langmuir isotherm confirmed the inhibition mechanism to be by physical interaction (physisorption). Terminalia catappa has been utilized as a corrosion inhibitor for different
metals [24-26]. However, it is yet to be studied as a potential inhibitor for stainless steels in a highly corrosive environment such as 1 M HCl. This study, therefore, investigated the corrosion inhibitory potential of *Terminalia catappa* leaves extract on stainless steel in 1M HCl under varied conditions.

2. Materials and Methods

2.1 Materials

Grade 304 austenitic stainless steel with a dimension of 1.8 cm x 0.9 cm x 0.3 cm and compositions shown in Table 1 were obtained from South Africa. The leaves of *Terminalia catappa* were obtained from the American University of Nigeria, Yola. Julabo water baths with temperature stability of ± 0.2 °C and an Adam PW254 analytical balance with 0.0001 g precision were used for the varied temperature, and weighing of samples, respectively. Absolute ethanol, hydrochloric acid (HCl), potassium iodide, ammonia, ferric chloride, and sulphuric acid used for this study were obtained from Fisher Scientific UK.

| Composition | Cr | Ni | Si | Mn | C | P | S | Fe |
|-------------|----|----|----|----|---|---|---|----|
| %           | 19.00 | 9.25 | 1.00 | 2.00 | 0.080 | 0.045 | 0.030 | Balance |

2.1.1 Preparation of tropical almond extracts

The leaves of *Terminalia catappa* were washed and air dried at room temperature (28±3 °C) for 96 hours. The dried leaves were then pulverized using a commercial grinder and stored in an airtight container. The plant extract was obtained by soaking 300 g of pulverized dry leaves in one liter of ethanol for 48 hours. The mixture was first sieved using a muslin cloth and the liquid obtained was subsequently filtered using Whatman No 1 filter paper, the filtrate was then concentrated using a rotatory evaporator until a semi-solid extract was left. The semi-solid extract obtained was oven dried to a solid residue at 45 °C. The phytochemicals (Alkaloids, tannin, flavonoids, Cardiac glycosides, and Saponin) in the extract were determined the method reported by Tiwari et al. [27].

2.2 Corrosion study

Varying concentrations (0.01, 0.03, 0.06, 0.07 and 0.09 g/ml) of the solid plant extracts which contain alkaloids, tannins, saponins, and phenolic compounds were prepared in 1M HCl. The study was executed at varying temperatures of 30, 40 and 50 °C. The weight loss was measured at intervals of four days for 20 days. During the measurements, the metals were removed from the solutions, cleaned with deionized water, rinsed with acetone and mopped dried using paper towels. The weights were obtained and used to estimate the changes that occurred during the experiment. All experiment was carried out in triplicates.
The following equations were used in evaluating the effect of the extract: weight loss (Equation 1), corrosion rate (Equation 2), surface coverage (Equation 3) and percent inhibition efficiency (% IE) of the inhibitors (Equation 4).

\[
\Delta W = w_2 - w_1 \quad (1)
\]

\[
CR = \frac{K\Delta W}{\rho At} \quad (2)
\]

\[
\theta = \left(1 - \frac{w_1}{w_2}\right) \quad (3)
\]

\[
%IE = \theta \times 100 \quad (4)
\]

Where \(\Delta W\) is the change in mass in mg, \(w_1\) and \(w_2\) are initial and final mass, respectively. In Equation 2, \(CR\) represents the corrosion rate in mm/y, \(\rho\) is the density of stainless steel in g/cm\(^3\), \(A\) is the area of the stainless steel bars in cm\(^2\) and \(t\) is the time in hours. The weight loss of the coupon in the electrolyte with the \textit{Terminalia catappa} inhibitor is \(w_1\) and \(w_2\) is the weight loss of the coupon in electrolyte without inhibitor. The surface coverage of the inhibitor on the surface of the stainless steel is \(\theta\), \(w_1\) and \(w_2\) (Equation 4) are the change in mass of stainless steel in solution with inhibitor and without inhibitor, respectively.

Scanning electron microscopy (SEM) model Supra 40VP manufactured by Carl Zeiss Ltd was used to study the metal surfaces after corrosion. The corrosion inhibition mechanism of the inhibitor was studied using three different isotherms (Freundlich, El-Awady, and Temkin).

### 3.0 Results and discussion

#### 3.1 Corrosion study

Table 1 shows a summary of the data collected and results calculated from the weight loss tests. The weight loss of the stainless steel bars reduced with increasing concentration of the inhibitor, with the blank having the highest weight loss. This indicates that more corrosion took place at lower concentration and in the blank compared to the higher concentration of the \textit{Terminalia catappa} inhibitor. This behavior resulted in lower corrosion rates at higher concentrations, thus better corrosion resistance with the addition of the inhibitor. As reported by Rani and Basu [15] corrosion inhibition of metal surfaces by plants extract can be attributed to the phytochemicals in the extracts. Some studies have reported plants with the phytochemicals: tannin, saponin, phlobatin, terpene, anthraquinone, cardiac glycosides and alkaloids in their extracts as an effective corrosion inhibitor in the acidic environment [12,28,29].

The inhibition efficiency of the extract increased relative to increasing inhibitor concentration; this is not so for the temperature changes as the corrosion inhibition efficiency decreased with rising temperature. However, under certain conditions, the % IE was observed to be higher at
higher temperatures. The highest % IE (97.9 %) was achieved at 50 ºC and 0.09 g/ml concentration, while the lowest % IE was 22.7 % at a temperature of 30 ºC and inhibition concentration of 0.01 g/ml. This could be as a result of the formation of stable oxide films on the metal surface at a higher temperature. This film augments chemical inhibition and enhances the overall performance of the inhibitor against the corrosion of the metal surfaces.

Table 1: Data from weight loss tests

| Temperature (K) | Concentration (g/ml) | Δw(g) | Surface coverage (θ) | Inhibition efficiency (%IE) | Corrosion rate (mm/y) |
|-----------------|----------------------|-------|----------------------|-----------------------------|----------------------|
| 303 Blank       | 0.0436               | -     | -                    | 0.2094                      |
| 0.01            | 0.0337               | 0.2271| 22.71                | 0.1619                      |
| 0.03            | 0.0293               | 0.3280| 32.80                | 0.1407                      |
| 0.06            | 0.0022               | 0.9495| 94.95                | 0.0106                      |
| 0.07            | 0.0015               | 0.9656| 96.56                | 0.0072                      |
| 0.09            | 0.0014               | 0.9679| 96.79                | 0.0067                      |
| 313 Blank       | 0.0495               | -     | -                    | 0.2378                      |
| 0.01            | 0.0371               | 0.2505| 25.05                | 0.1782                      |
| 0.03            | 0.0351               | 0.2909| 29.09                | 0.1686                      |
| 0.06            | 0.0046               | 0.9071| 90.71                | 0.0221                      |
| 0.07            | 0.0037               | 0.9252| 92.53                | 0.0178                      |
| 0.09            | 0.0021               | 0.9434| 94.34                | 0.0101                      |
| 323 Blank       | 0.0614               | -     | -                    | 0.2949                      |
| 0.01            | 0.0466               | 0.2410| 24.10                | 0.2238                      |
| 0.03            | 0.0368               | 0.4007| 40.07                | 0.1768                      |
| 0.06            | 0.007                | 0.8860| 88.60                | 0.0336                      |
| 0.07            | 0.0051               | 0.9169| 91.69                | 0.0245                      |
| 0.09            | 0.0013               | 0.9788| 97.88                | 0.0062                      |

3.1.1 Adsorption mechanism of Terminalia catappa

Temkin, El-Awady and Freundlich isotherms were used to investigate the adsorption mechanism and behavior of the inhibitor on the surface of the metal. The data obtained from the weight loss analysis fitted in all the isotherms considered in this study (Figure 1).
Temkin isotherm relates the inhibitor concentration $C$ to the degree of surface coverage ($\theta$) as shown in Equation 5 [30].

$$\theta = \frac{-2.303\log K_{ads}}{2a} - \frac{2.303\log C}{2a}$$

(5)

A graph of surface coverage ($\theta$) versus log $C$ was obtained (Figure 1) and the $R^2$ of the graphs indicated that the weight loss data fitted in Temkin isotherm. Also, adsorption constant $K$ was obtained and applied to determine Gibb’s free energy of the process using Equation 6 [31]. The attractive parameter ($a$) from the isotherm are all negatives, which shows that there was repulsion at the interface between the metal surface and the inhibitor [32]. The Gibb’s free energy values obtained from all the three isotherms used indicated that the inhibition mechanism of *Terminalia catappa* on 304 stainless steel in 1 M HCl is by physisorption i.e it is by physical adsorption and the process was spontaneous. A similar observation was made by Eddy et al. [24] in research where *Terminalia catappa* was utilized as a corrosion inhibitor for mild steel in H$_2$SO$_4$. The summary of the parameters from the isotherms and Gibb’s free energy values obtained are shown in Table 2.

$$\Delta G_{ads} = -RT \log(55.5K_{ads})$$

(6)

The El-Awady isotherm is a modified Langmuir isotherm and is given as follows [33].

$$\log \frac{\theta}{1-\theta} = \log K + y \log C$$

Where, $y$ is the number of inhibitor molecules occupying an active site, $\theta$ is the surface coverage, $C$ is the inhibition concentration, $K$ is the constant associated to the adsorption constant $K_{ads}$, which is $K^{1/y}$. Values of the inverse of $y < 1$ is an indication of multilayer adsorption, while the inverse of $y > 1$ denotes that an inhibitor molecule takes up more than one active site. The reciprocal of $y$ indicates the number of water molecules that have been displaced by one inhibitor molecule [34]. The reciprocal of $y$ in this study are 0.50, 0.6, and 0.57 at 303, 313, and 323 K, respectively. The values indicate the formation of multilayers of inhibitor molecules on the stainless steel’s surface and each molecule of the extract occupied more than one active site [35].
Figure 1: (a) Temkin isotherm (b) Freundlich isotherm and (c) El-Awady isotherm

Table 2: Adsorption isotherm parameters

| Temperature (K) | El-Awady parameters | Temkin isotherm parameters | Freundlich isotherm parameters |
|----------------|----------------------|-----------------------------|--------------------------------|
|                | Y                    | ΔG_{ads} (kJ/pp m) | a | K (ppm) | ΔG_{ads} (kJ/pp m) | K | 1/n | ΔG_{ads} (kJ/pp m) | R² |
| 303            | 1.99                 | 0.069                 | -1.47 | 0.9791 | -1.61 | 3.81 | 0.9764 | 0.071 | 0.6059 | -3.46 | 0.9885 |
| 313            | 1.66                 | 0.062                 | -1.40 | 0.9685 | -1.63 | 3.67 | 0.9636 | 0.076 | 0.5798 | -3.75 | 0.9728 |
| 323            | 1.74                 | 0.069                 | -1.57 | 0.9428 | -1.70 | 3.11 | 0.9969 | 0.092 | 0.5410 | -4.38 | 0.9908 |

The values of parameter n of the Freundlich isotherm were obtained to be 1.65, 1.72 and 1.85 at 303, 313 and 323 K, respectively. The n values are greater than unity, which implies that the corrosion inhibition mechanism of the extract on stainless steel in 1 M HCl, can be modelled using Freundlich isotherm and adsorption mechanism is by physical process [36].

3.2 SEM analysis
Selected samples were analyzed for surface morphology using SEM. The micrographs show severe corrosion on the surface of the blank samples (Figures 2a, 3a, and 4a). The harshness of corrosion agents on the metal surface in blank solution (solutions without the inhibitors) increased with the rise in temperature. These images confirmed the rates at which the metal
corroded in 1 M solution at various temperatures. It could be seen from the micrographs in Figures 2b-d, 3b-d and 4b-d that the stainless steels’ surfaces were inhibited against corrosion and smoother surfaces are seen with increment in the concentration of the inhibitor.

Figure 2: SEM micrographs of stainless steels after corrosion in 1 M HCl containing *Terminalia catappa* extract at 303 K (a) without inhibitor (b) 0.06 g/ml inhibitor (c) 0.07 g/ml inhibitor (d) 0.09 g/ml inhibitor
Figure 3: SEM micrographs of stainless steels after corrosion in 1 M HCl containing *Terminalia catappa* extract at 40°C (a) without inhibitor (b) 0.06 g/ml inhibitor (c) 0.07 g/ml inhibitor (d) 0.09 g/ml inhibitor
4. Conclusion

*Terminalia catappa* was found to exhibit good corrosion inhibitory effects on type 304 austenitic stainless steel in 1 M HCl. The results obtained from the gravimetric corrosion analysis showed that *Terminalia catappa* gave an efficiency of 96.8% at concentration of 0.09 g/mL and 30 °C temperature. The corrosion inhibitor, *Terminalia catappa* was observed to inhibit the stainless steel in 1 M HCl by physisorption mechanism. The corrosion rates decreased with increase in extract’s concentrations and inhibition efficiency. The corrosion rates however, increased with increase in temperature. The corrosion analysis data fitted into all the isotherms (El-Awady, Temkin, and Freundlich) studied.
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