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# Corrosion inhibition potentials of Roselle (*Hibiscus sabdariffa*) in tetraoxosulphate (VI) acid solution

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**ABSTRACT**

The harmful effects of chemical-based corrosion inhibitors on man and the ecosystem prompted the search for alternatives that are nonhazardous to the environment. Therefore, the corrosion inhibition potentials of the leaves and flowers of the green variety of Roselle (Hibiscus sabdariffa) were evaluated. This was undertaken mainly to investigate the effectiveness of the leaves and flowers of Roselle in mitigating the corrosion of mild steel in acidic medium. The gravimetric method of corrosion measurement was used to evaluate the corrosion inhibition potentials of the plant parts. The results showed that the leaves and flowers of the plant can effectively be used to mitigate the corrosion of metals in acidic medium. The maximum inhibition efficiencies of the leaves and flower extracts of the plant were 27.94 % and 22.81 % respectively. The leaves and flowers of the plant were more efficient on the first eight days of application.



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## 1. INTRODUCTION

The use of extracts from green plants to control corrosion of metals in various media is becoming popular due to their availability, low cost and eco-friendliness [1- 3]. The extracts from the leaves, seeds, roots and stems of some of these plants have been successfully utilized to control the corrosion of metals in various media.

Okafor et al [3] investigated the inhibitive effects of leaves (LV), roots (RT) and seeds (SD) extracts of Neem (*Azadirachta indica)* on mild steel corrosion in acidic medium. The results showed that the extracts acted as good inhibitors for mild steel in $H_2SO_4$ acid solution. Olawale et al [4] investigated the inhibitive effects of orange fruit seed extract on aluminum corrosion in 1M hydrochloric acid. The results showed that at inhibitor concentration of 30%, the inhibition efficiency and corrosion rate obtained in HCl solution were 38.37 % and 0.012 g/m$^2$.Oruene et al [5] investigated the inhibitive potentials of Breadfruit leaf extract on the corrosion of mild steel in hydrochloric acid solution. The results showed that maximum inhibition efficiency of 82.15 % was obtained from the gravemetric method, whereas 87.19 % was recorded from the electrochemical method. *Newbouldia leavis* leaf extract mitigated the corrosion of mild steel in sulphuric acid [6]. Inhibition efficiencies of 72 % and 90 % were obtained. Fadare et al [7], reported that ethanolic extract of *Fiscus asperifolia* and its fractions (n-hexane, ethyl acetate, butanol and aqueous) were found to be effective in mitigating the corrosion of mild steel in acidic medium.

The inhibitive action/effects of these plant materials could be attributed to the presence of the phytochemical compounds present in their extracts [8, 9]. These compounds have complicated molecular structures, large molecular weights, oxygen and nitrogen atoms contained in their structures [3]. The adsorption of the phytochemicals onto the metal surface reduces the interaction between the metal and the corrodent [3].

However, there are still many plants whose inhibition potentials have not been investigated. One of such plants is the green variety of Roselle (Hibiscus sabdariffa). The phytochemical analysis of the leaves of Roselle (*Hibiscus sabdariffa)* revealed the presence of tanins, flavonoids, saponins and steroids [10]. The phytochemical analysis of the calyses of *Hibiscus sabdariffa* revealed the presence of the following phytochemicals: tannins (17.0%), saponins (0.96%), phenols (1.1 %), glycosides (0.13%), alkaloids (2.14%) and flavonoids (20.08%) [11]. Alaga et al [12] reported the presence of saponins (1.41%), alkaloids (0.09%), tannins (0.19 %), total phenols (0.07%), flavonoids (2.41%) and glycosides (0.13%). Therefore, *Hibiscus sabdariffa* has corrosion inhibition potentials.

Therefore, the objective of this work is to evaluate the corrosion inhibition potentials of the leaves and flowers of *Hibiscus sabdariffa* in 0.5 M tetraoxosulphate (VI) acid. These plant extracts, apart from being environmentally friendly and ecologically acceptable, plant products are cheaper, readily available and renewable sources of materials.

## 2. RESEARCH METHOD

### 2.1 Processing of plant materials

Fresh leaves and flowers of Roselle plant (green variety) were obtained from a market in Jalingo, Taraba State. The leaves and flowers were thoroughly washed in water to remove dust and sand particles. The leaves and flowers were shade dried separately for fourteen (14) days, to reduce their moisture content [9]. The dried leaves and flowers were separately pulverized in an electric blender. The powdered samples were sieved with a sieve (150 µmm mesh size). The sieved samples (leaves and flowers of Roselle) were stored in different containers and labelled until needed for corrosion studies.

### 2.2 Extraction of active ingredients in the leaves and flowers of Roselle plant

Extraction of the active ingredients in the leaves and flowers of the Roselle plant were done using the reflux method [13, 14]. Sixty (60) grammes each of the powdered leaves and flowers of Roselle plant were measured using an electronic balance into two round bottom flasks. 1000 mL of 0.5 M tetraoxosulphate (VI) acid solution was added to each of the measured samples in the two flasks. The resulting mixture was boiled for three hours [14] on a burner. The content of each flask was filtered using a filter paper. The filtrates were taken as the stock solutions.

### 2.3 Preparation of corrosion test specimens (Coupons)

Twenty-two (22) rectangular test specimens each of dimension (40 mm x 30 mm x 3 mm) were prepared from a mild steel sheet. Mild steel was selected because it is widely used in the industry for the construction of machines and its accessories.The percentage chemical composition of the mild steel sheet used for the preparation of the corrosion test specimens is: 98.82 wt% Fe, 0.143 wt% C, 0.124 wt% Si, 0.541 wt% Mn, 0.0204 wt% P, 0.307 wt% Cr, 0.00097 wt% Mo, 0.00788 wt% Ni, 0.0158 wt% Cu, 0.0224 wt% Al, 0.00027 wt% Nb, 0.00339 wt% Ti and 0.00134 wt% V. A 3mm diameter hole as shown in Figure 1 was made at the longitudinal end of each specimen. This was done to facilitate easy suspension and withdrawal of the metal samples from the corrodent.

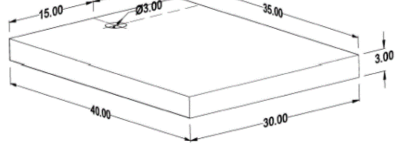


Figure 1. Corrosion test specimen (coupon)

The sharp edges formed during the cutting and drilling operations were smoothened using a hand file. The surfaces of the test specimens were further polished with abrasive paper using water as a lubricant, to produce smooth surface and to remove any trace of contaminants. The test specimens were degreased in ethanol, rinsed thoroughly in distilled water and dried in acetone. After weighing, the test specimens were kept in a desiccator until needed for corrosion studies.

### 2.4 Preparation of corrosion environment (Corrodent)

The solution of 0.5 M tetraoxosulphate (VI) acid solution was prepared by diluting 22.6 mL of concentrated tetraoxosulphate (VI) acid in 1000 mL of distilled water.

### 2.5 Gravimetric method

The gravimetric method of corrosion measurement was adopted due to its simplicity, reliability and the method has been used by several researchers to assess the corrosion of plants and equipment [15]. The experiment was conducted in two sets and at room temperature. In each set of weight loss measurements, eleven rubber containers were arranged on a table in the Hydraulics laboratory of Department of Mechanical Engineering, Taraba State University, Jalingo. 1000 mL of 0.5 M tetraoxosulphate (VI) acid solution was measured and poured into each of the labeled plastic containers in each set. The required concentrations of the extract(s) (1.0 g/L, 1.5 g/L, 2.0 g/L, 2.5 g/L and 3.0 g/L) were respectively measured from the stock solution of leaves and flowers of the Roselle plant. After measurement, the measured extracts (leaves and flowers of Roselle plant) were respectively added to each of the container containing the corrodent in each set. No plant extract was added to the eleventh container.

Each of the pre-weighed test specimens were totally immersed in each of the plastic containers containing both the corrodent and the extracts. After every four days (4), samples were retrieved from the corrodent, observed and washed to remove corrosion products. Samples were again subjected to absolute washing in absolute ethanol with a cotton wool, dried in acetone and weighed to determine the final weight. The difference in weight of the coupon was taken as the weight loss. The average weight loss value was determined by adding the weight loss value obtained from the first set of experiment to the corresponding weight loss value obtained from each coupon in the second set of experiment and the result divided by two (2). The average weight loss value was record as the weight loss. The corrosion rate was computed using Equation 1 [16]

$$CR = \frac{534W}{DAt} \quad (1)$$

where CR is the corrosion rate in mm/day, W is the weight loss in grammes, A is the sectional area in $mm^2$, D is density of the metal sample in $g/mm^3$ and t is the exposure time of the mild steel coupon in days.
The degree of surface coverage (ϴ) was computed using Equation 2 [17]

$$\Theta = 1 - \frac{W_i}{W_o} \quad (2)$$

where $W_i$ is the weight loss in the presence of inhibitor and $W_o$ is the weight loss in the absence of the inhibitor. The inhibition efficiency (I %) of the extract was computed using Equation 3 [18]

$$\text{Inhibition Efficiency (I)} = \frac{CR_{uninhibited} - CR_{inhibited}}{CR_{uninhibted}} \times 100\% \quad (3)$$

where $CR_{uninhibited}$ is the corrosion rate of the uninhibited system and $CR_{inhibited}$ is the corrosion rate of the inhibited system.

## 3. RESULTS AND DISCUSSION

### 3.1 Corrosion inhibition effects of the extracts

Table 1 shows the inhibitive effect of the extracts (flowers and leaves) on the corrosion of mild steel in the tetraoxosulphate (VI) acid medium. There was a significant difference ($p < 0.05$) in the corrosion rate of mild steel in tetraoxosulphate (VI) acid solution without any extracts (control) compared to the corrosion rate in the acidic medium with different concentrations of the extracts. This is an indication that the extracts

Table 1: Inhibitive effects of the extracts on the corrosion of mild steel in the acidic medium

| Concentration (g/L) | Corrosion rate (mm/day) | |
|---|---|---|
| | Flower extract | Leaves extract |
| Control | 80.99±4.83[a] | 80.99[a] |
| 1.00 | 76.17±4.14[b] | 78.34[b] |
| 1.50 | 72.65±3.73[b] | 75.99[b] |
| 2.00 | 69.84±3.17[b] | 73.08[b] |
| 2.50 | 67.26±2.86[c] | 68.78[c] |
| 3.00 | 66.01±3.05[c] | 64.68[d] |

**Values are expressed as Mean± Standard error of the mean. Values with different alphabetic superscript in the same column are significantly different ($p< 0.05$)**

mitigated the corrosion of mild steel in the acidic environment. Therefore, the flower and leaves of Roselle can effectively be used to inhibit the corrosion of mild steel in acidic medium. Potentiodynamic polarization and electrochemical impedence spectroscopy measurements also revealed that Hibiscus sabdariffa inhibit the corrosion of metals in acidic medium [19]. The low corrosion rates obtained at various concentrations of the extracts could be attributed to the adsorption of the phytochemical constituents present in the extracts on the metal surface [5, 6]. The adsorbed chemical constituents on the metal surface created a barrier between the metal and the corrodent, thereby protecting the metal surface from the corrosive action of the corrodent [5].

### 3.2 Effect of concentration of the extracts on corrosion rate

The effect of concentration of the extracts (leaves and flower) on the corrosion rate of mild steel in tetraoxosulphate (VI) acid solution are presented in Figures 2 and 3.

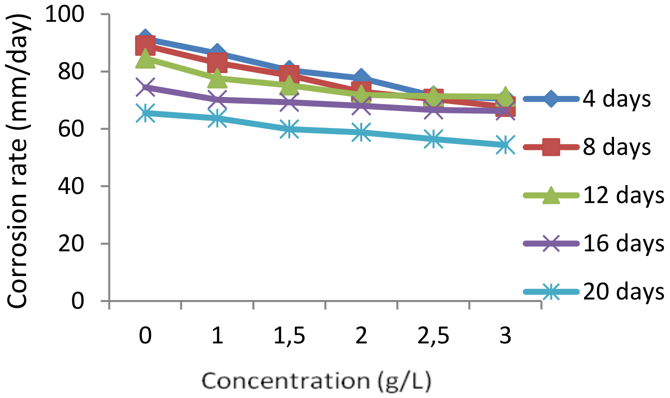


Figure 2. Effect of concentration of flower extract of Roselle on the corrosion rate of mils steel in acidic medium

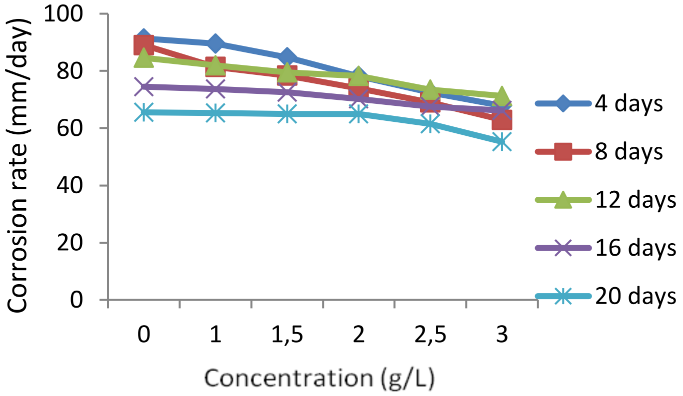


Figure 3. Effect of concentration of leaves extract of Roselle on the corrosion rate of mild steel in tetraoxosulphate (VI) acid solution

It was observed fron the Figures that the corrosion rates decreased with increase in the concentration of the extracts. Similar patterns were reported by [5, 20]. The decrease in corrosion rate with extract concentration could be attributed to the increase in the adsorption of the phytoconstituents on the metal surface [21, 22]. Consequently, corrosion rate is reduced with increased concentration.

### 3.3 Effect of concentration of extracts on inhibition efficiency

Presented in Figures 4 and 5 are the results of the effect of concentration of extracts on inhibition efficiency. Inhibition efficiencies of the extracts increased with increase in the concentration of the extracts. Similar trends were obtained by [23-25]. Minimum and maximum inhibition efficiencies were obtained at extract concentrations of 1.0 g/L and 3.0 g/L respectively.

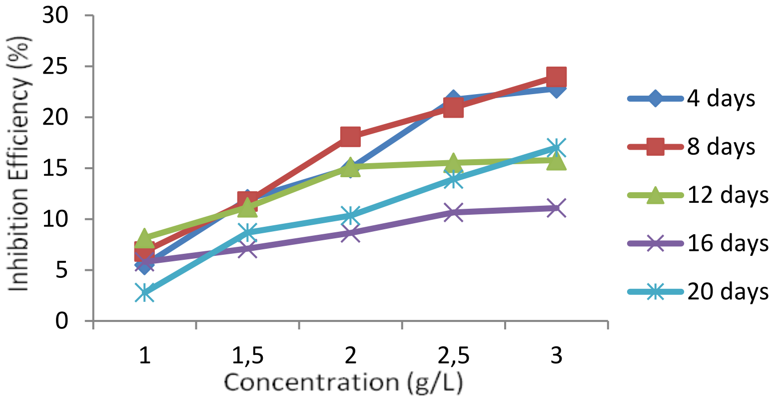


Figure 4. Effect of concentration of flower extract of Roselle on inhibition efficiency

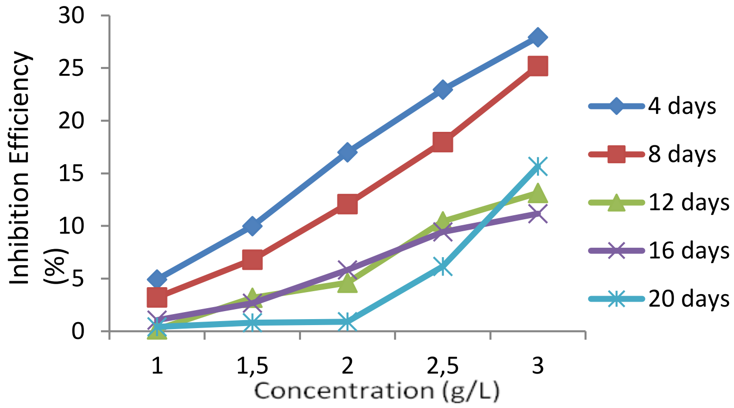


Figure 5. Effect of concentration of leaves extract of Roselle on inhibition efficiency

The increased inhibition efficiency could be due to the increase in the surface coverage area as extract concentration increases [26]. Therefore, corrosion rate is reduced, and inhibition efficiency of the extracts is increased.

**3.4 Effect of exposure time on inhibition efficiency of the extracts**

Presented in Figures 6 and 7 is the effect of exposure time on the inhibition efficiencies of the extracts.

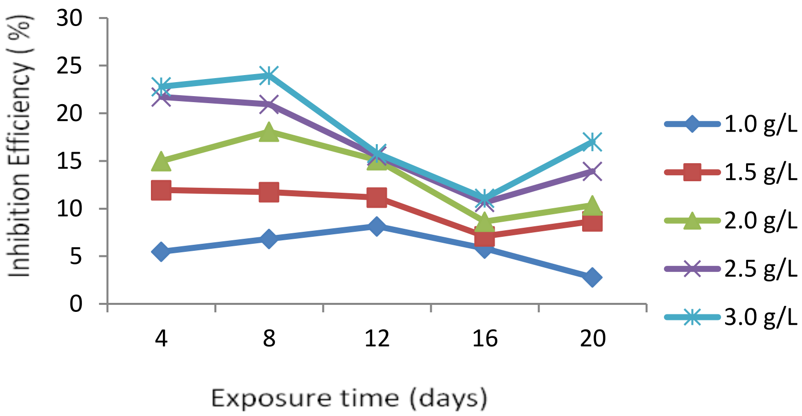


Figure 6. Effect of exposure time on the inhibition efficiency of the flower extract of Roselle

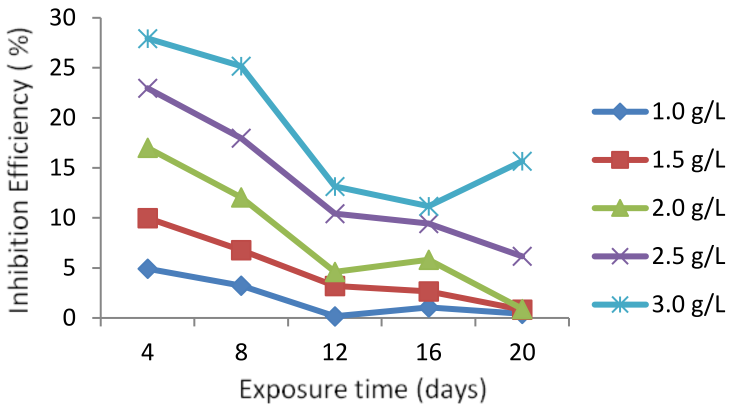


Figure 7. Effect of exposure time on the inhibition efficiency of the leaves extract of Roselle

Figure 6 shows the effect of exposure time on the inhibition efficiency of the flower extract of Roselle. Inhibition efficiency was observed to increase from day four to day twelve after which it began to decline till the end of the exposure time at 1.0 g/L of the extract. The increase in inhibition efficiency from day four to day twelve could be due to the formation of a protectictive film on the metal surface [6], thereby reducing the rate of corrosion attack. Therefore, the corrosion rate is reduced, and inhibition efficiency of the extract is increased. The decrease in the inhibition efficiency of the extract could be due to the fact that the extract was completely used used up and the metal surface is exposed to the acidic environment [6]. Consequently, the corrosion rate is increased, and the inhibition efficiency of the extract is reduced. At 1.5 g/L and 2.5 g/L of the flower extract, inhibition efficiency of the extract decreased as the exposure time increased from day four to day sixteen after which it began to increase with time. At 2.0 g/L and 3.0 g/L of the extract, inhibitor efficiency increased as exposure time increased from day four to day eight after which it began to decline as exposure time increased to day sixteen and subsequently it increased with time.

Presented in Figure 7 is the effect of exposure time on the inhibition efficiency of the leaves extract of Roselle. The inhibitor efficiency was observed to decrease as exposure time increased from day four to day twelve after which it increased as exposure time increased to day sixteen and subsequently it began to decrease with time at concentrations of 1.0 g/L and 2.0 g/L. At 1.5 g/L and 2.5 g/L, inhibition efficiency decreased with increased exposure time. Inhibition efficiency was observed to decrease as exposure time increased from day four to day sixteen after which it began to increase with time at concentration of 3.0 g/L.

### 3.5 Adsorption Isotherm

The possible mode of adsorption of the extracts can be investigated by testing the experimental data generated with several adsorption isotherm models [27]. The surface coverage (Ɵ) values for each extract were fitted into the Langmuir and Freundlich isotherm models.

#### 3.5.1 Langmuir adsorption isotherm

The Langmuir adsorption isotherm model is based on the assumption that adsorption takes place at certain sites within the adsorbent [27]. The Langmuir adsorption isotherm is represented by Equation (4) [28, 29].

$$\frac{Cinh}{\theta} = \frac{1}{Kads} + C_{inh} \quad (4)$$

Where $K_{ads}$ ($Lg^{-1}$) is the adsorption equilibrium constant, $C_{inh}$ is the concentration of the inhibitor in g/L and Ɵ is the degree of surface coverage. The plots of $\frac{C_{inh}}{\theta}$ against concentration ($C_{inh}$) for the adsorption of the flower extract on the metal surface is presented in Figure 8. Straight lines were obtained from the plots. The Langmuir adsorption isotherm parameters obtained from the plots are presented in Table 2. Table 2 is the Langmuir adsorption isotherm parameters for the adsorption of the flower extracts onto the metal surface in tetraoxosulphate (Vl) acid solution. The coefficient of correlation ($R^2$) obtained from the plots are not close

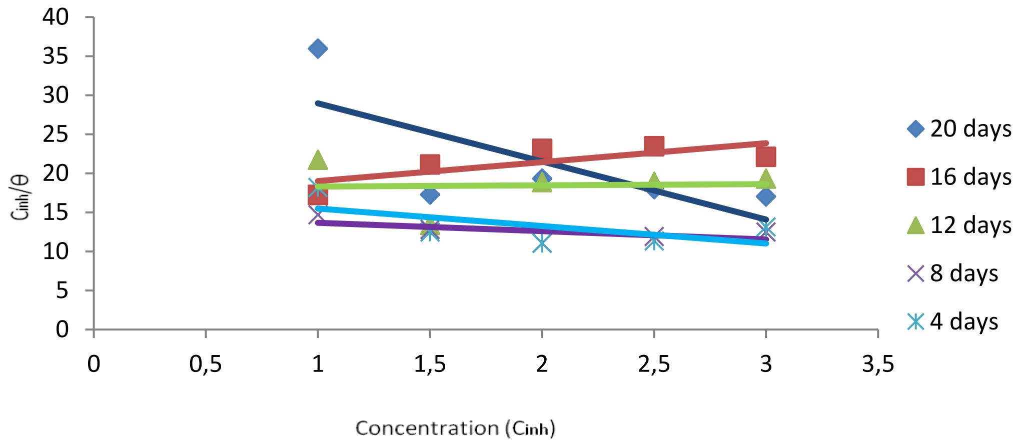


Figure 8. Langmuir adsorption isotherm plots for the adsorption of the flower extract on the metal surface in the acidic medium

Table 2: Langmuir adsorption isotherm parameters for the adsorption of the flower extract on the metal surface in the acidic solution.

| | 4 days | 8 days | 12 days | 16 days | 20 days |
|---|---|---|---|---|---|
| Slope | -2.237 | -1.066 | 0.156 | 2.427 | -7.438 |
| Intercept | 17.724 | 14.727 | 18.148 | 16.574 | 36.396 |
| $R^2$ | 0.376 | 0.385 | 0.002 | 0.581 | 0.524 |
| $K_{ads}$ | 0.060 | 0.070 | 0.060 | 0.060 | 0.030 |

to unity, therefore the adsorption behaviour of the flower extract does not obey Langmuir adsorption isotherm model. Isotherm models with regression coefficients less than 0.8 are considered as unfit [30]. The adsorption equilibrium constant ($K_{ads}$) obtained from the plot on day eight was higher compared to the values obtained on other days. This is an indication that a better and stronger interaction between the inhibitor and metal occurred on day eight compared to other days [31]. Therefore, the flower extract was more efficient as an inhibitor on day eight compared to other days.

Langmuir adsorption isotherm plot for the adsorption of the leaves extract on the metal surface in the acidic solution is presented in Figure 9. Straight lines were obtained on all days. The Langmuir adsorption isotherm parameters obtained from the plots for the adsorption of the leaves extract on mild steel in the acidic medium are presented in Table 3. It was observed from the table that the coefficient of correlation ($R^2$)

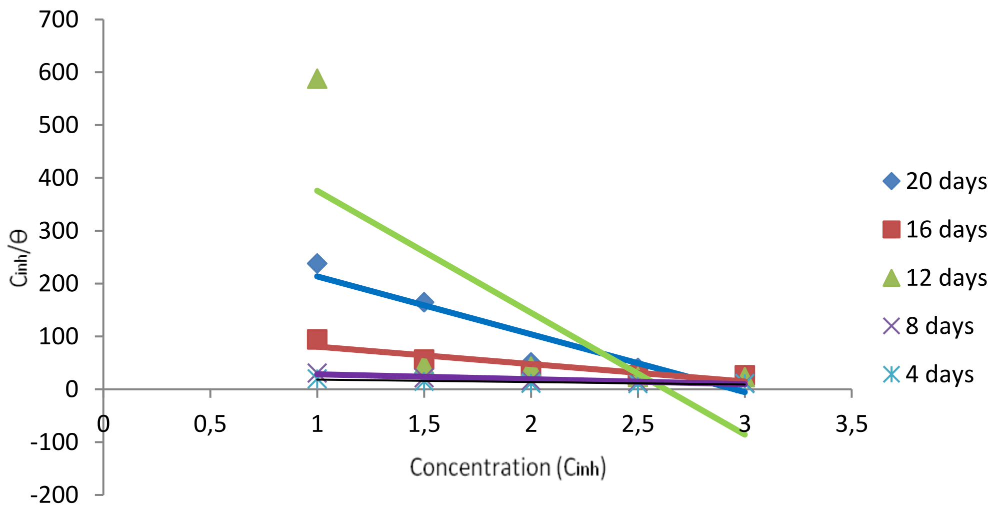

Figure 9. Langmuir adsorption isotherm plots for the adsorption of the leaves extract on the metal surface in the acidic medium

Table 3: Langmuir adsorption isotherm parameters for the adsorption of the leaves extract on the metal surface in the acidic solution

| | 4 days | 8 days | 12 days | 16 days | 20 days |
|---|---|---|---|---|---|
| Slope | -4.660 | -9.304 | -230.010 | 33.010 | -109.49 |
| Intercept | 23.061 | 37.72 | 606.620 | 113.770 | 323.22 |
| $R^2$ | 0.822 | 0.912 | 0.541 | 0.822 | 0.869 |
| $K_{ads}$ | 0.040 | 0.030 | 0.002 | 0.009 | 0.003 |

obtained on day four, eight, sixteen and twenty are close to unity, therefore the adsorption of the extract obeyed the Langmuir adsorption isotherm model [23, 30, 32]. The coefficient of correlation ($R^2$) on day twelve is not close to unity; therefore the adsorption of the extract does not obey Langmuir adsorption model.The $R^2$ value obtained on day eight is higher compared to the values obtained on other days. This is an indication that the adsorption of the leaves extract on the metal surface on day eight fiited more into the Langmuir adsorption isotherm model compared to other days [32]. The adsorption equilibrium constant ($K_{ads}$) obtained on day four is higher compared to the values obtained on other days. This means that the leaves extract was more efficient on day four compared to other days.

### 3.5.1 Freundlich adsorption isotherm

Freundlich isotherm is mostly employed to describe the adsorption characteristics of heterogeneous systems [33]. The linearized form of Freundlich isotherm is represented by the expression in Equation 5 [34]

$$\text{Log } \Theta = \text{Log } K_{ads} + n\log C \quad (5)$$

Where $K_{ads}$ is the adsorption equilibrium constant, C is the inhibitor concentration, ϴ is the surface coverage and n is the adsorption intensity.

Figure 10 shows the Freundlich adsorption isotherm plots for the adsorption of the flower extract on mild steel in the acidic medium. Straight lines were obtained. Straight lines were also obtained by [7, 34]. The values of n and $K_{ads}$ were evaluated from the slopes and intercepts of the plots [31]. The values of the Freundlich adsorption isotherm parameters obtained from the plots are presented in Table 4. The coefficient of correlation ($R^2$) obtained on day four, eight and sixteen were close to unity. This means that the adsorption of the extract on the metal surface conform to the Freundlich adsorption isotherm model. The $R^2$ values obtained on day twelve and twenty were not close to unity, therefore the adsorption of the extract does not fit into Freundlich adsorption isotherm model on those days.

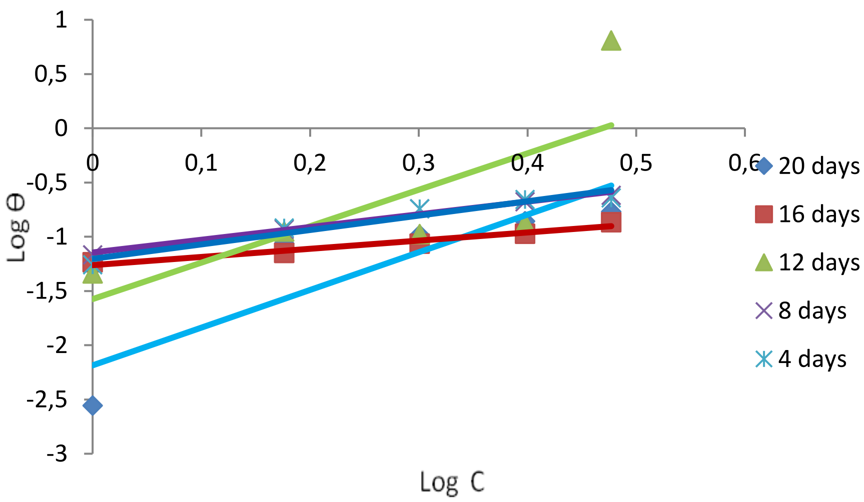

Figure 10. Freundlich adsorption isotherm plots for the adsorption of the flower extract on the metal surface in the acidic medium

Table 4: Freundlich adsorption isotherm parameters for the adsorption of the flower extract on the metal surface in the acidic solution

| | 4 days | 8 days | 12 days | 16 days | 20 days |
|---|---|---|---|---|---|
| n | 1.326 | 1.172 | 3.359 | 0.753 | 3.474 |
| $K_{ads}$ | 0.063 | 0.072 | 0.027 | 0.055 | 0.007 |
| $R^2$ | 0.944 | 0.979 | 0.562 | 0.962 | 0.780 |

The adsorption equilibrium constant ($K_{ads}$) obtained on day eight is higher compared to the values on day four, twelve, sixteen and twenty. This implies more adsorption and better inhibition efficiency of the flower extract on day eight compared to other days. The values of n ranged between 0.753 and 3.474. The value of n greater than unity is an indication that the adsorption of the extract on the metal surface was based on heterogeneous interaction [34].

The Freundlich adsorption isotherm plot for the adsorption of the leaves extract on the metal surface is presented in Figure 11. Straight lines were obtained. The Freunlich adsorption isotherm parameters for the adsorption of the leaves extract on the metal surface is presented in Table 4. The coefficient of correlation

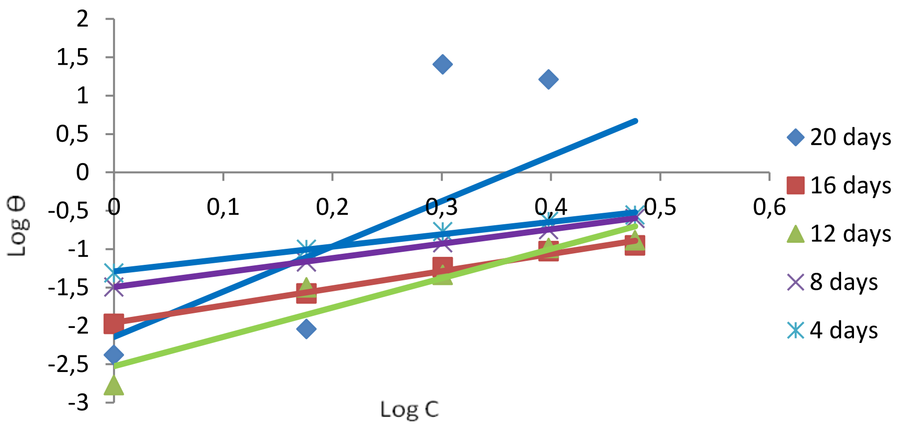


Figure 11. Freundlich adsorption isotherm plots for the adsorption of the leaves extract on the metal surface in the acidic medium

Table 5: Freundlich adsorption isotherm parameters for the adsorption of the leaves extract on the metal surface in the acidic solution

| | 4 days | 8 days | 12 days | 16 days | 20 days |
|---|---|---|---|---|---|
| n | 1.611 | 1.880 | 3.818 | 2.237 | 5.898 |
| $K_{ads}$ | 0.051 | 0.032 | 0.003 | 0.011 | 0.007 |
| $R^2$ | 0.992 | 1.000 | 0.904 | 0.988 | 0.390 |

($R^2$) obtained form the plots were close to unity on day four, twelve, sixteen and unity on day eight. This implies that the adsorption of the extracts conform to the Freundlich adsorption isotherm model. The $R^2$ value obtained on day twenty is less than unity and that implies poor fit of the experimental data to the isotherm model. The adsorption equilibrium constant obtained on day four is greater than the values obtained on day eight, twelve, sixteen and twenty. This is an indication that the extract was more effective on day four compared to other days. The values of n were greater than unity, which is indicative of a heterogeneous interaction.

## 4. CONCLUSION

The corrosion rates obtained in the presence of the extracts were lower compared to the values obtained in the blank solution. The corrosion rates decreased with increase in the concentration of the extracts. The inhibition efficiencies of the extracts increased with concentration. Therefore, leaves and flowers of the green variety of Hibiscus sabdariffa have been identified as corrosion inhibitors for mild steel in acidic medium. The leaves and flower extracts of the plant are more efficient as inhibitors in the first eight days of application.

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