



Article Info

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Inhibition Potential of Extract of Leaves of Sodom Apple in the Corrosion of Mild Steel in 1.0 M H₂SO₄

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Corrosion inhibition of mild steel in 1.0 M sulphuric acid by extract of leaves of Sodom apple has been studied using gravimetric and surface analysis. Varying concentrations (0.5, 1.0, 1.5 and 2.0 g/mL) of the inhibitor in 1.0 M H₂SO₄ were prepared from residue of an ethanolic extract of the sample and were used for the corrosion inhibition study. The extracts were previously confirmed by Fourier Transform Infrared Spectroscopy and screened for phytochemicals including tannins, saponins, and cardiac glycosides. The results for anticorrosion studies showed that the highest inhibition efficiency of the extract was 89.3% at 1.5 g/mL inhibitor concentration and a temperature of 323 K. Equally, the corrosion rate of the steel was observed to decrease with increase in the concentration of the extract. Scanning electron micrographs also indicated that the extract actually slows down the rate of corrosion of the steel. The study further suggests that the corrosion inhibition mechanism of the extract on steel in 1 M H₂SO₄ could be modelled using Freundlich isotherm and that the inhibition was by physisorption and formation of multilayers on the steel surface.

Keywords: Corrosion, mild steel, Sodom apple, acid, extracts.

1. Introduction

Corrosion of materials in both infrastructural and industrial settings, due to the actions of corrosive agents such as acids, bases, and salts, have been linked to several cost implications. These, include economic and cost in materials, energy, safety and even cost in human lives.¹ There is therefore the continuous need to develop materials for application in the prevention of damages to equipment and facilities caused by corrosive agents. Many materials have been used in inhibition of corrosion including various organic and inorganic materials, polymers and paints, oils and a range of hybrid materials.² These corrosion inhibitors, when added to equipment form a thin layer of protection against corrosive agents or corrosion catalysts.³

Currently, because of the huge cost and environmental impact of using some of the materials for corrosion prevention, there has been a great increase in the drive for the utilization of green inhibitors for corrosion prevention. This has the advantage of little or no effect to the environment and even human lives when they utilize equipment coated with these inhibitors. Green inhibitors are sourced majorly from various parts of plants including the roots, fruits, leaves and oils of plants¹ and sometimes

could be hybrid materials such as chitosan-CuO nanocomposite, which has been used in corrosion protection of steel in acidic medium.⁴ Ag-CuO/epoxy hybrid nanocomposite has also been applied as green coating to prevent corrosion of copper.⁵

The efficiency of plants extracts from mango and orange peels, garlic peels, castor bean, coffee powder, white tea, etc as green inhibitors of carbon steel in acidic medium was recently further demonstrated in a recent survey demonstrated.⁶

Sodom apple (*Calotropis procera*) is a plant of the family Apocynaceae that is native to tropical Africa and is widely found in Sokoto, Sokoto State, Nigeria. It has a greenish fruit and though its leaves are poisonous, they are reported to cure some illness like asthma and cough.^{7,8} Like other plants such as mango, orange, garlic, Sodom apple can be explored as a green inhibitor of corrosion. The current study thus, examine the effectiveness of leaf extract of Sodom apple as inhibitor of corrosion of mild steel in acidic medium.

2. Materials and Methods

2.1 Materials

Mild steel obtained from Ahmadu Bello Way, Sokoto was used as the metal sample for corrosion study. The steel sample had a composition (wt.%) of Fe (97.20), Mn (1.00), P (0.40), C (0.45), S (0.60) and Si (0.35). The sample was mechanically pressed cut into different coupons with dimension of 1.20 X 1.20 X 1.10 cm. Samples of Sodom apple leaves were obtained from Maberu area, Sokoto State and identified at Botany Unit of Usmanu Danfodiyo University, Sokoto. Flic Film FF8172F water bath with temperature stability of ± 0.1 °C and a Mettler Toledo™ Excellence XPR analytical balance with 0.0001 g precision were used for the varied temperature experiment, and for weighing of samples. Absolute ethanol, sulfuric acid (H₂SO₄), zinc dust, sodium hydroxide (NaOH), and acetone were purchased from Sigma Aldrich or Fisher Scientific UK and were used without further purification. Double distilled water was used throughout the study.

2.2. Pretreatment and preparation of Samples

The coupons surface treatment was done according to the ASTM G1-90 standard.⁹ The sample was degreased by washing with ethanol, rinsed with acetone and allowed to dry in air before they were preserved in a desiccator. The metal specimens were first pretreated by polishing with sandpaper and cleaning with a clean cloth prior to analysis. The leaves of Sodom apple were thoroughly washed to remove dirt, sun-dried for one week, grounded into powder and stored in glass sample bottle before analysis.

2.3. Leaf Extraction

The extraction was done as described in Madu *et al.* (2019).¹⁰ 300 g of the prepared leaf powder was soaked in a solution of ethanol (1 L) for 48 h. The mixture was filtered using a muslim cloth and then Whatman No. 1 filter paper. The filtrate was then concentrated using a rotary evaporator to obtain a semi-solid extract which was further dried to a solid residue at 45°C.

2.4. Phytochemical screening

The solid residue was subjected to phytochemical analysis using standard procedures as described in literature¹¹⁻¹³ in order to identify phytochemicals including tannins, flavonoids, alkaloids, cardiac glycosides, and saponins present in the extracts.

2.5. Fourier Transform Infrared (FTIR) analysis

FTIR spectrum of the solid residue of the extract was recorded on a Bruker Alpha spectrometer fitted with a Platinum ATR module (4000 – 600 cm⁻¹).

2.6. Preparation of inhibitor for anticorrosion studies

The solid residue of the extract sample was used in preparing different concentrations (0.5, 1.0, 1.5 and 2.0 g/mL) of the extract solutions for anticorrosion studies by dissolving appropriate masses of the extract sample in 1 L of 1.0 M H₂SO₄ respectively. These standard solutions were used in the corrosion inhibition studies.

2.7. Corrosion inhibition studies

Corrosion inhibition studies was done using two methods: (i) gravimetric method and (ii) surface analysis.

2.7.1 Gravimetric analysis

Gravimetric analysis was used in the corrosion inhibition studies. In the gravimetric experiment, a previously weighed metal steel coupon was completely immersed in 250 mL of the test solution in an open beaker. The beaker was inserted into a water bath maintained at various temperature (303, 313, 323 and 333 K). After every 24 h, each sample was withdrawn from the test solution, washed in a solution containing 50 per cent NaOH and 100 mg of zinc dust. The washed coupon was rinsed in acetone and dried in air before re-weighing. The difference in weight for a total period of 21 days was taken as total weight loss. The analysis was conducted in triplicates and the average measurement was taken. From the weight loss results, the inhibition efficiency (% IE) of the inhibitor was calculated using the formula:

$$\% IE = \frac{\text{Weight loss in control} - \text{Weight loss in sample}}{\text{Weight loss in control}} \times 100$$

Also, the corrosion rate (CR) was determined using the formula:

$$CR (mm/yr) = \frac{K \Delta W}{\rho A T}$$

where W, ρ , A and T are the mean weight loss (mg), density (gcm⁻³), surface area (cm²) and exposure duration (h) respectively.

2.7.2 Surface analysis

Surface analysis of the steel sample was examined before and after the corrosion studies using Scanning Electron Microscopy (Phenom Prox, Malaysia), operated at an accelerated voltage of 10 kV.

2.8. Mechanism of Inhibition

The mechanism of inhibition was investigated by fitting the data obtained from the corrosion studies into two isotherm models – the El-awady and Freundlich.

3. Results and Discussion

3.1. Phytochemical screening of ethanol extract of sodom apple leaves

The results for phytochemical screening for the leaf extract are shown in Table 1. The essence of identifying the phytochemicals was to develop an idea of the phytochemicals responsible for corrosion inhibition activity. The screening showed the presence of flavonoids, tannins, saponins, and cardiac glycosides.

Table 1: Phytochemicals in the extract of leaves of Sodom apple.

Phytochemicals	Extract of Sodom apple leaves
Tannins	+
Saponins	+
Condensed tanins	+
Alkaloids	-
Cardiac glycosides	++

Key: + = present; ++ = very present; - = absent

3.2. FTIR Spectrum

The FTIR spectrum of the extract, which is presented in Figure 2 shows peaks that could be assigned to functional groups expected in the extract of Sodom apple leaf. The result compares well with FTIR absorptions observed in the spectrum of tannins extracted from Sodom apple.¹⁴ The band at 3268 cm⁻¹ was assigned to phenolic O-H stretching whereas the peak at 2933 cm⁻¹ was assigned to the C-H functional group in alkanes. Other assigned peaks were aromatic (C=C) stretching at 1630 cm⁻¹, nitro (N-O) group stretch observed at 1513 cm⁻¹, and bands observed at 1402 to 1036 cm⁻¹ indicating the presence of asymmetric stretch of ester (C-O).

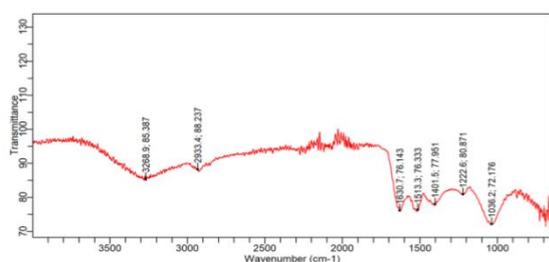


Figure 2: FTIR spectrum of Sodom apple leaf extract.

3.3. Corrosion inhibition studies

The results for anticorrosion studies on the metal coupons using various concentrations of the extract of Sodom apple and at the temperature

range 303 to 333 K are presented in Figures 3 to 4. It is observed that the inhibition efficiency increases with increasing concentration up to 1.5 g/mL before it started decreasing. Also, on the effect of temperature, it was observed that the inhibition efficiency increases with increase in temperature up to 323 K before starting to decrease. Thus, the highest inhibition efficiency was 89.3% at 1.5 g/mL inhibitor concentration and temperature of 323 K. Generally, no inhibition was observed with the blank.

The corrosion rate on the other hand decreases with increase in the concentration of the extract. The rate was however observed to increase with increase in temperature and peaked at 323 K. The highest corrosion rate was observed for the blank at 323 K. The observed increase in corrosion inhibition efficiency with increasing temperature (303 to 323 K) has been explained to be due to an increase in the medium's corrosive activity as a result of an increase in the thermal agitation of its molecules and an increase in its conductivity.⁴ It was also explained that the decrease in the inhibition efficiency at 333 K might be due to the desorption of the adsorbed inhibitor on the metal's surface due to increased thermal agitation. Also, the increasing inhibition efficiency with increasing concentration could be linked to the increasing surface coverage of the metal with increasing concentration.¹⁰

The corrosion inhibition effect observed for the extracts can be ascribed to the presence of the phytochemicals such as tannins, saponins, etc identified in the extracts as reported in earlier studies.^{15, 16}

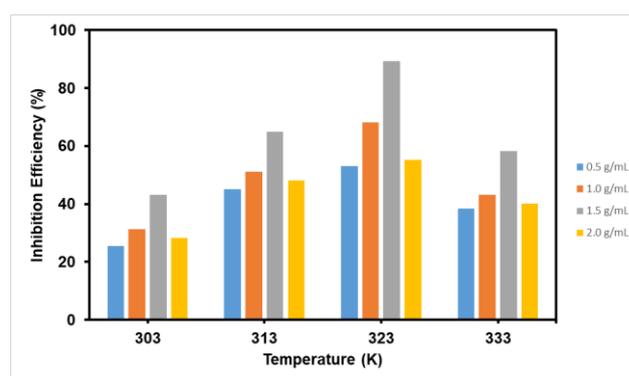


Figure 3: Inhibition Efficiency (%) of Sodom apple leaf extract (0.5, 1.0, 1.5 and 2.0 g/mL) on the corrosion of mild steel in 1.0 M H₂SO₄ at temperatures of 303, 313, 323 and 333K.

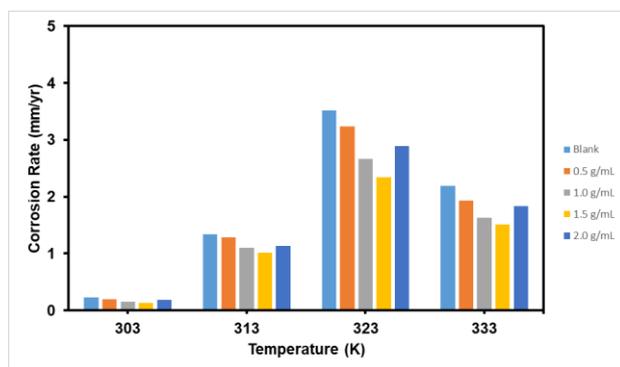


Figure 4: Corrosion Rate (mm/yr) of mild steel in 1.0 M H₂SO₄ at temperatures of 303, 313, 323 and 333 K using Sodom apple leaf extracts of 0.5, 1.0, 1.5 and 2.0 g/mL concentration.

3.4. Surface Analysis by Scanning Electron Microscopy (SEM)

In a bid to better understand the anticorrosion effect of the extract, the coupon was examined before and after the corrosion studies using SEM analysis. The SEM micrographs for the coupons are presented in Figure 5. The micrographs show rough surfaces for both samples immersed in the 1.0 M H₂SO₄ solution, however, the sample without the inhibitor was rougher than the sample with the inhibitor indicating that the inhibitor actually slows down the rate of degradation.

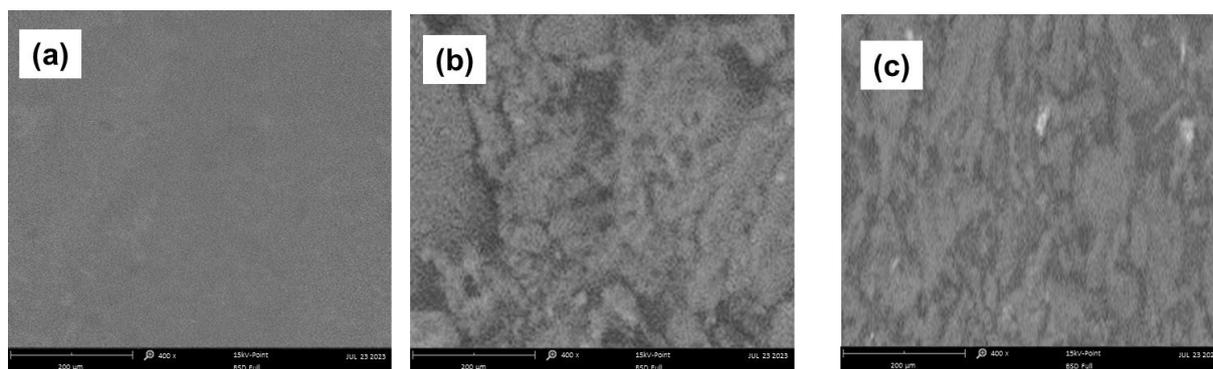


Figure 5: SEM micrographs of metal steel coupon (a) original sample (b) after corrosion in 1.0 M H₂SO₄ without inhibitor and (c) after corrosion in 1.0 M H₂SO₄ with 1.5 g/mL inhibitor concentration at 323 K.

3.5. Mechanism of Corrosion Inhibition

To understand the mechanism of inhibition by the extract, the data were fitted into El-Awady and Freundlich isotherms and the results are presented in Figure 6 and Table 2. The R² values reveal that the data fitted better with the Freundlich isotherm than the modified Langmuir (El-Awady) isotherm. However, the reciprocal of θ in this study, 0.55, 0.59, 0.39, and 0.61 at 303, 313, 323 and 333 K, respectively are less than unity indicating the formation of multilayers of

inhibitor molecules on the steel's surface. The n values of the Freundlich isotherm were also determined and were found to be greater than unity. That is, 1.69, 1.82, 1.95 and 1.96 at 303, 313, 323 and 333 K respectively. This suggest that the corrosion inhibition mechanism of Sodom apple extract on steel in 1 M H₂SO₄ can be modelled using Freundlich isotherm and that the inhibition mechanism is physisorption.^{10, 17}

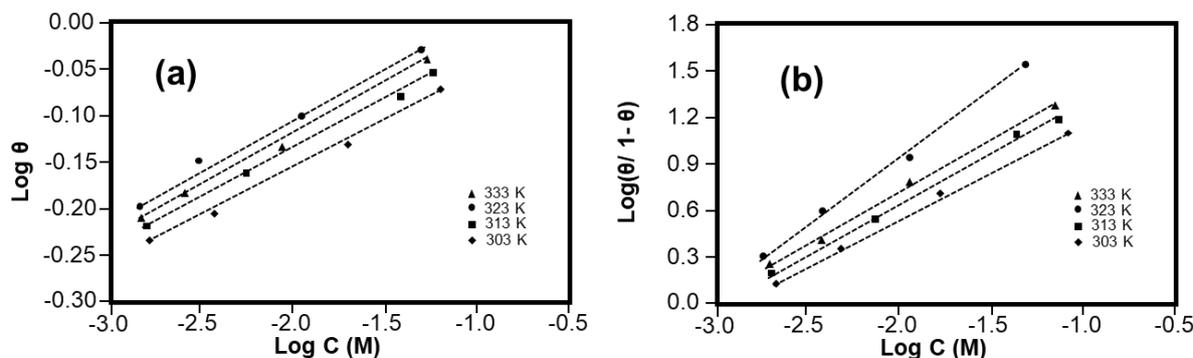


Figure 6: (a) El-Awady isotherm and (b). Freundlich isotherm.

Table 2: Adsorption isotherm parameters

Temperature (K)	El-Awady Isotherm Parameters			Freundlich Isotherm parameters		
	Y	K _{ads}	R ²	K _F	1/n	R ²
303	1.83	0.065	0.9712	0.069	0.5934	0.9953
313	1.71	0.063	0.9748	0.073	0.5493	0.9914
323	1.52	0.059	0.9432	0.091	0.5134	0.9948
333	1.64	0.063	0.9492	0.097	0.5099	0.9688

4. Conclusion

This study has shown that extracts of leaves of Sodom apple can be applied as green inhibitors for prevention of corrosion of steel in acidic medium. The inhibition efficiency of the extract was observed to have an optimum value at a concentration of 1.5 g/mL and temperature of 323 K. The extract was also observed to show multilayer adsorption on the metal and to follow a physisorption mechanism. The findings of this study highlight the potential of extracts of Sodom apple leaves as a low cost green alternative to corrosion prevention of steels in aqueous environments.

Conflict of interest

The authors declare no conflict of interest.

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