



# KINETICS STUDIES OF *Sida acuta* LEAVES EXTRACTS AS CORROSION INHIBITOR ON TANNERY MACHINES PARTS IN ACIDIC ENVIRONMENT



Oladunni Nathaniel\*, Yusuf Sulyman Ishola, Anweting Idongesit Bassey, Agbele Idowu Elijah and Samaila P. Oscar,  
Department of Science Laboratory Technology, Nigerian Institute of Leather and Science Technology (NILEST), Zaria, Nigeria

\*Corresponding author: [olanath2002@yahoo.com](mailto:olanath2002@yahoo.com)

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**Abstract:** Most of tannery machines are made of mild steel and these machines are subjected to harsh condition, which includes exposure to moisture, air, acid, base, salt and other environmental components that causes corrosion on machines' parts. Corrosion is the degradation of the metallic properties of a metal. The aim of this study is to generate measurable and testable data towards the control of corrosion using plant (*Sida acuta* leaves ethanolic extracts) as inhibitor. This was achieved through the determination of inhibitor's performance by weight loss techniques, determination of metal-inhibitor interaction mechanism using kinetic parameters and adsorption isotherms (Langmuir, Temkin and Freundlich), the corrosion process through the use of exposure time, phytochemicals of the plant leaves extracts, inhibitor concentration, media concentration, temperature, initial weight of metal and the weight of the metals in every 6 h for a total of 24 h. Mild steel samples were immersed in 0.1 M HCl solution in the absence and presence of varying inhibitor concentrations (blank (0.1 M HCl), 0.2, 0.4 and 0.6 g/dm<sup>3</sup>) of plant inhibitor (*Sida acuta* leaves extract). A weight loss technique was adopted and the weights of the mild steel were recorded every 6 h for 24 h. The best inhibition efficiency for the plant extract was 86.667% at 0.6 g/dm<sup>3</sup> of the extract at 313 K. The half-life (9.118 h) for the corrosion reaction occurrence for plant inhibitor was high at 0.2 g/dm<sup>3</sup> at 313 and at 343 was high at 0.4 g/dm<sup>3</sup> (9.290 h). Thus, the plant inhibitor might not be better in its performance than other inhibitors on mild steel when exposed to acidic environment but they are best in aspect of its interaction to the environment due to its ecofriendly, inexpensive and biodegradable properties.

**Keywords:** Corrosion inhibitor, Tannery machines, Langmuir, Temkin and Freundlich isotherm

## Introduction

Corrosion may be considered as a redox process in which an oxide coating is formed on the surface of the metal/alloy undergoing the process. It therefore requires oxygen and moisture to occur. It involves the transfer of electrons along the surface of the metal under the influence of a potential difference (Onen, 2000).

Inhibitors are commonly used to prevent metal dissolution as well as acid consumption and retard the corrosive attack on metallic materials.

Corrosion inhibitors are chemical compounds used in small concentrations in industry to reduce the corrosion rate of metals and alloys in contact with aggressive environment, and therefore, retard the corrosion process and keeps its rate to a minimum and thus prevents economic losses due to metallic corrosion. The mechanism action of inhibitors is of great importance and depends on their formulation as well as on their rational use in various environments. The electronic characteristic of the adsorbate molecules, the solution chemical composition, the nature of metallic surface, the temperature of the reaction, the immersion time and the electrochemical potential at the metal-solution interface determine the adsorption degree and hence the inhibition efficiency (Zarrouk *et al.*, 2010). Corrosion inhibitors synthetic chemicals are widely used to protect metals against corrosion, but, Most of them are not environment friendly due to their toxicity levels, bioaccumulation and/or biodegradability (Al Mubarak *et al.*, 2010). Hence, the research of new corrosion inhibitors non-toxic, eco-friendly, natural, at low or "zero" environmental impact are desired (Ibrahim *et al.*, 2012). Plant extract is rich source of naturally synthesized chemical compounds, readily available low-cost and eco-friendly, and can be obtained through simple extraction process with low cost as well as biodegradable (El-Etre, 2007).

Almost all organic molecules containing heteroatoms such as nitrogen, sulphur, phosphorous, and oxygen show significant inhibition efficiency. Despite these promising findings about possible corrosion inhibitors, most of these substances are not

only expensive but also toxic non-biodegradable thus causing pollution problems. Hence, these deficiencies have prompted the search for their replacement. Plants are sources of naturally occurring compounds, some with complex molecular structures and having different chemical, biological, and physical properties. The naturally occurring compounds are mostly used because they are environmentally acceptable, cost effective, and have abundant availability. These advantages are the reason for use of extracts of plants and their products as corrosion inhibitors for metals and alloys under different environment. Different plant extracts can be used as corrosion inhibitors commonly known as green corrosion inhibitors. Tannins and their derivatives can be used to protect steel, iron, and other tools from corrosion. To protect mild steel in 2 M HCl solutions from corrosion, extracts from leaves can be used. Extracts of tobacco from twigs, stems, and leaves can protect steel and aluminium in saline solutions and strong pickling acids (Davis *et al.*, 2001; Buchweishaija, 2013). These processes however, require the use of corrosion inhibitors in order to reduce acid damage on metallic materials. In the chemical, oil, gas, automobile and transportation industries metallic degradation is one of the main factors influencing the dependability of the systems (Koch *et al.*, 2002).

## Materials and Methods

### Experimental

#### Materials

Hydrochloric acid, ethanol, acetone, distilled water, Digital Analytical balance, Volumetric flasks (1000 cm<sup>3</sup>), Water bat, Thermometer, Oven, Measuring cylinder (100 cm<sup>3</sup>) Different sets of beakers (Pyrex), Press knife, Desiccator, mild steel (2 × 4 cm<sup>2</sup> coupons) and *Sida acuta* leaves.

#### Methods

##### Analysis of composition of mild steel

The composition of the sample was analyse to determine the percentage of the following elements in the sample (Iron, Carbon, Manganese, Silicon, Sulphur, phosphorus, Nickel, Molydenum and chromium.

**Materials preparation**

The mild steel sheet was mechanically press cut into 2.0 x 4.0 cm area coupons and was polished with emery papers. The mild steel was degreased in detergent, dried in acetone and stored in moisture free desiccators before corrosion studies commenced.

**Extraction of plants extracts**

The leaves of the *Sida acuta* was cut into pieces which were then oven dried at 105°C for two hours and cooled. The leaves was grounded into powder and then sieved through 4 μm pore size sieve and the filtrate was soaked in container containing ethanol for seven days in order to extract the juice by leaching. The juice extract was distilled at 79°C to remove the ethanol from the juice extracts and concentrate the inhibiting chemical(s). The juice extracts was stored in a clean glass bottle and covered properly.

**Phytochemicals screening**

**Test for alkaloids**

One (1) cm<sup>3</sup> of the extract was measured and it was stirred with 2 cm<sup>3</sup> of 1% HCl on a steam bath. Few drops of distilled water were added followed by few drops of Wagner's reagent. A reddish brown precipitate indicate present of alkaloid.

**Test for tannins**

Two 2.0 cm<sup>3</sup> of the extract was measured and mixed with 10% ferric chloride (FeCl<sub>3</sub>) solution. The bluish colour indicates the presence of tannins.

**Test for anthraquinones**

One (1) g of the extract was weighed and mixed with 2 cm<sup>3</sup> of benzene. 12% of ammonia solution was added and was shook vigorously the formation of the pink, red of violent colour on the ammoniacal phase indicating presence of anthraquinones.

**Test for flavonoids**

Two (2) cm<sup>3</sup> of dilute NaOH was taken and added to 2 cm<sup>3</sup> of the extract the appearance of a yellow colour indicate presence of flavonoids.

**Test for saponins**

One (1) cm<sup>3</sup> of distilled water was added to 1 cm<sup>3</sup> of the extract and shaken vigorously. Stable persistence froth indicates presence of saponins.

**Test for phenol**

Equal volume of extract and FeCl<sub>3</sub> was mixed together; a deep bluish green solution indicate presence of phenol.

**Test for cardiac glycosides**

Two (2) cm<sup>3</sup> of the extract was dissolve in 2 cm<sup>3</sup> of glacial acetic acid containing a drop of FeCl<sub>3</sub> followed by 2 cm<sup>3</sup> of conc. H<sub>2</sub>SO<sub>4</sub>. A brown ring formation indicates presence of deoxy sugar.

**Test for steroid**

Two (2) cm<sup>3</sup> of the extract was mixed with 1ml of acetic anhydride followed by 2 cm<sup>3</sup> of H<sub>2</sub>SO<sub>4</sub>. The colour change from violet to blue or green in the sample indicated the presence of steroid.

**Preparation of inhibitor concentrations**

The preparation of the plant inhibitor was performed by weighing the 4 g of *Sida acuta* leaves ethanolic extracts (SALEE) on a weighing balance. It was then dissolved in 1000 cm<sup>3</sup> standard flask using distilled water making it 4 g/dm<sup>3</sup> (which was used as the stock solution for further dilution). The inhibitor was further diluted in water to concentrations of 0.2, 0.4 and 0.6 g/dm<sup>3</sup> by diluting volume of 5, 10 and 15 cm<sup>3</sup> into 100 cm<sup>3</sup> measuring cylinder. 100 cm<sup>3</sup> of 0.1 M HCl acid medium and 100 cm<sup>3</sup> were poured into the corrosion cell for further corrosion studies.

**Weight loss experiment**

Weighed test specimens were fully immersed in each of the test media contained in a 250 cm<sup>3</sup> beaker for 24 h. Experiments were performed with hydrochloric acid test medium in which some had the solution extracts added. The specimens was taken out of the test media every 6 h, washed

with distilled water, rinsed with ethanol, dried in acetone, and re-weighed. Plots of weight loss versus the exposure time and of calculated corrosion rate versus time of exposure will be made. All the experiments were performed at 313 K and 343 K temperature(s) (Omotosho, 2016).

**Determination of corrosion rate**

The corrosion rate expressed as millimeter per year was calculated using the formula;

$$\text{Corrosion rate (mpy)} \text{ CR} = \frac{87.63 \text{ W} \times 1000}{\text{DAT}} \quad (1)$$

**Where:** W = Weight loss = Initial weight (W<sub>1</sub>) – Final weight (W<sub>2</sub>) in milligrams; A = Total surface area of specimen in cm<sup>2</sup>; T = Total time sample was immersed in hours; D = Density of the specimen in g/cm<sup>3</sup>; CR = Corrosion rate in millimeters per year.

**Surface coverage**

The degree of surface coverage (Θ) was calculated from equation

$$\Theta = \frac{\text{CR}_0 - \text{CR}_1}{\text{CR}_0} \quad (2)$$

**Determination of percentage of inhibition efficiency**

The percentage inhibitor efficiency, P, was calculated from the equation:

$$\text{IE (\%)} = \frac{\text{CR}_0 - \text{CR}_1}{\text{CR}_0} \times 100 \quad (3)$$

Where CR<sub>0</sub> and CR<sub>1</sub> are the corrosion rates in the absence and the presence respectively, of a predetermined concentration of inhibitor. The percentage inhibitor efficiency was calculated for all the inhibitors throughout the exposure period.

The area and density of the specimen used are 8 cm<sup>2</sup> and 7.86 g/cm

**Adsorption isotherms**

The dependence of surface coverage on concentration was studied using the following isotherms.

Langmuir isotherm: log (θ/1) vs log C (4)

Temkim: θ vs log C (5)

Freundlichisotherm: log θ vs log C (6)

The correlation co-efficient (R<sub>2</sub>) values were used to determine and to select the best fit isotherm.

**Results and Discussion**

This study investigated the problems associated with the effect of corrosion on industrial chemical environments usually found in the leather industries.

**Phytochemical screening of plant material**

The data from this study revealed that this plant (*Sida acuta*) leaves extract has a rich content of phytochemicals namely, alkaloids, flavonoids, and cardiac glycosides with functional group containing (nitrogen, Oxygen and carbon), aromatic rings present in (OH) constituent in the SALEE (*Sida acuta* Leaves ethanolic extract) chemical structure as depicted by the phytochemical test in Table 1 also enhance the process of corrosion inhibitor adsorption on the mild steel. This also corroborates the work of (Harbone, 1978; Sofowora, 1993). These active constituents are had been reported to be to promote the corrosion inhibition of mild steel in aggressive acid media and they are responsible for the inhibition efficiency of the extract, especially that of the alkaloid due to the presence of heterocyclic atoms of nitrogen (Okafor and Ebenson, 2007). The presence of the carbonyl double bond carbon group in the SALEE also suggests it to be a good corrosion inhibitor on mild steel in an acidic medium.

**Table 1: Results of the phytochemical of *Sida acuta* leaves used**

S/N	Tests	Results
1.	Alkaloids	++
2.	Tannins	-
3.	Anthraquinones	-
4.	Flavonoids	+
5.	Saponins	-
6.	Phenols	-
7.	Cardiac glycoside	+
8.	Steroids	-

++ Abundant; + Trace; - Absent

**Table 2: Variation of masses of coupons with time for inhibitor in 0.1 M HCl at 313 K**

Conc. (g/dm <sup>3</sup> )	Initial mass (g)	Time (hrs)			
		6	12	18	24
Blank (0.1 M HCl)	4.036	4.004	3.968	3.937	3.901
0.200	3.613	3.607	3.601	3.595	3.589
0.400	3.580	3.575	3.570	3.564	3.559
0.600	3.604	3.600	3.595	3.591	3.586

**Table 3: Variation of masses of coupons with time for inhibitor in 0.1 M HCl at 343 K**

Conc. (g/dm <sup>3</sup> )	Initial mass (g)	Time (hrs)			
		6	12	18	24
Blank (0.1 M HCl)	4.105	3.842	3.576	3.282	3.042
0.200	4.254	4.232	4.208	4.188	4.165
0.400	4.062	4.049	4.033	4.022	4.010
0.600	3.996	3.987	3.978	3.968	3.956

**Mass loss studies**

From the mass values, the inhibition efficiency (IE %) and surface coverage ( $\theta$ ) and corrosion rate were calculated using the equations in 3, 4 and 5. The results obtained from mass loss measurements are presented in Tables 2, 3, 4, and 5 which show the variation of the mass of the mild steel coupons and time in 0.1 M hydrochloric acid for an immersion period of 6 to 24 h and Table 6 shows the inhibition efficiency (IE %) and surface coverage ( $\theta$ ) and corrosion rate.

**Table 4: Weight loss for mild steel corrosion in 0.1 M HCl of plant inhibitor (*Sida acuta*) at 313 K**

Conc (g/dm <sup>3</sup> )	Time (hrs)	W <sub>0</sub> (g)	W <sub>1</sub> (g)	$\Delta W$ (g)	Log(W <sub>1</sub> -W <sub>0</sub> ) (g)
0.2	6	3.613	3.607	0.006	-2.222
	12	3.613	3.601	0.012	-1.921
	18	3.613	3.595	0.018	-1.745
	24	3.613	3.589	0.024	-1.620
0.4	6	3.580	3.575	0.005	-2.301
	12	3.580	3.570	0.010	-2.000
	18	3.580	3.564	0.016	-1.796
0.6	24	3.580	3.559	0.021	-1.678
	6	3.604	3.600	0.004	-2.398
	12	3.604	3.595	0.009	-2.046
0.6	18	3.604	3.591	0.013	-1.886
	24	3.604	3.586	0.018	-1.745

**Table 5: Weight loss for mild steel corrosion in 0.1M HCl for different concentration of *Sida acuta* inhibitor at 343 K**

Conc. (g/dm <sup>3</sup> )	Time (hrs)	W <sub>0</sub> (g)	W <sub>1</sub> (g)	$\Delta W$ (g)	Log (W <sub>1</sub> -W <sub>0</sub> ) (g)
0.2	6	4.254	4.232	0.022	-1.658
	12	4.254	4.208	0.046	-1.337
	18	4.254	4.188	0.066	-1.180
	24	4.254	4.165	0.089	-1.051
0.4	6	4.062	4.049	0.013	-1.886
	12	4.062	4.033	0.029	-1.538
	18	4.062	4.022	0.040	-1.398
0.6	24	4.062	4.010	0.052	-1.284
	6	3.996	3.987	0.009	-2.046
	12	3.996	3.978	0.018	-1.745
0.6	18	3.996	3.968	0.028	-1.553
	24	3.996	3.956	0.040	-1.398

**Table 6: Calculated values of corrosion rate, inhibition efficiency and surface coverage for plant inhibitor (*Sida acuta*)**

Temp. K	Conc. (g/dm <sup>3</sup> )	CR (mpy)	IE%	Log I%	Surface coverage
313	Blank	7.836	-	-	-
	0.2	1.393	82.222	1.915	0.822
	0.4	1.219	84.444	1.927	0.844
	0.6	1.045	86.667	1.938	0.867
343	Blank	61.704	-	-	-
	0.2	5.166	34.074	1.532	0.341
	0.4	3.018	61.481	1.789	0.615
	0.6	2.322	70.370	1.847	0.704

**Temperature effect on corrosion behaviour of mild steel in 0.1 M HCl acid with inhibitor**

The effect of temperature on corrosion inhibition of mild steel in 0.1 M hydrochloric acid with 0.2, 0.4, 0.6 g/dm<sup>3</sup> of inhibitor between the temperature of 313 K and 343 K had been studied. The data were calculated and shown in the Tables 4, 5 and 6; there was much mass loss at higher temperature (343 K) for all of the inhibitor used. From the Table 5, it can be observed that the rates of corrosion of mild steel in 0.1 M hydrochloric acid with inhibitor are found to be decreased with increase in concentration of inhibitor at all the temperature, while the percentage inhibition efficiency increases when compared to blank.

This signifies that the dissolution of the metals increased at higher temperatures. This observation is attributed to the general rule guiding the rate of chemical reaction, which says that chemical reaction increases with increasing temperatures. Also an increased temperature favors the formation of activated molecules, which may be doubled in number, with 10°C rises in temperature, thereby increasing the reaction rate. This is because the reactant molecules gain more energy and are able to overcome the energy barrier more rapidly (Ita *et al.*, 1997). An increase in temperature may also increase the solubility of the protective films on the metals, thus increasing the susceptibility of the metal to corrosion (Okafor *et al.*, 2004). The solubility of oxygen gas decreases with increase in temperature.

Thus, oxygen concentration is expected to be more at higher temperature which in this case is higher at 343 K than at 313

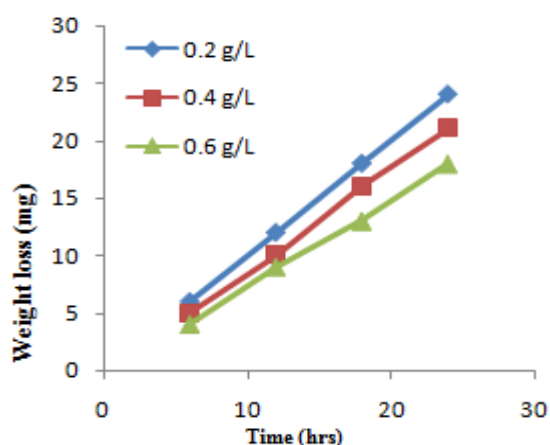
K. The presence of high concentration of oxygen thereby causes the metal to corrode faster. Also for solids, solubility generally increases with increasing temperature. This explains why the protective film which is solid becomes more soluble as the temperature is increased.

The highest inhibition efficiency (96.096%) was attained at 343 K for 0.6 g/dm<sup>3</sup> concentration of the inorganic inhibitor, further increase in the temperature, inhibition efficiency was decreased.

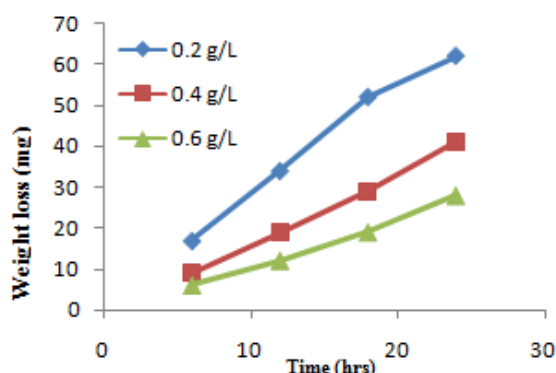
The least inhibition efficiency (34.074%) was attained at 343 K for 0.2 g/dm<sup>3</sup> green inhibitor. This is may be due to the desorption of metal surface (Zhang *et al.*, 2002).

**Table 7: The values of rate constant (K) and half-life (t<sub>1/2</sub>) for mild steel in 0.1 M HCl in different concentrations of the plant leaves extracts**

Conc. (g/dm <sup>3</sup> )	Rate constant (K)		Half-life (t <sub>1/2</sub> )	
	313	343	313	343
0.2	0.0760	0.0760	9.118	9.118
0.4	0.0797	0.0746	8.695	9.290
0.6	0.0813	0.0820	8.524	8.451



**Fig. 1: Variation of weight loss with time (hrs) for mild steel in 0.1 M HCl for plant inhibitor at 313 K**



**Fig. 2: Variation of weight loss with time (hrs) for mild steel in 0.1 M HCl for plant inhibitor at 343 K**

**Temperature effect on corrosion behaviour of mild steel in 0.1 M HCl acid with inhibitor**

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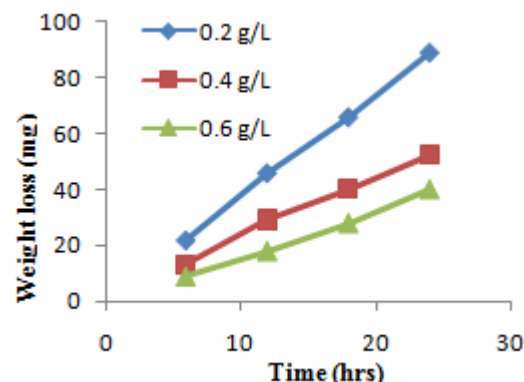
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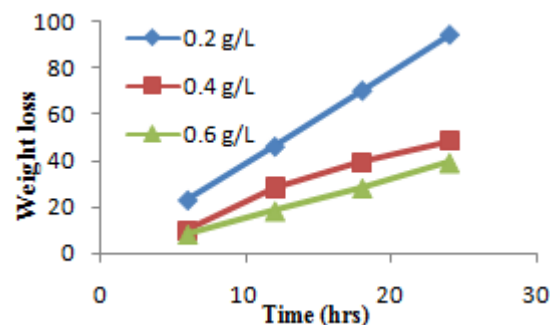
Thus, oxygen concentration is expected to be more at higher temperature which in this case is higher at 343 K than at 313 K. The presence of high concentration of oxygen thereby causes the metal to corrode faster. Also for solids, solubility generally increases with increasing temperature. This explains why the protective film which is solid becomes more soluble as the temperature is increased.

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**Fig. 3: Variation of weight loss with time (hrs) for mild steel in 0.1 M HCl for inorganic inhibitor (K<sub>2</sub>CrO<sub>4</sub>) at 313 K**



**Fig. 4: Variation of weight loss with time (hrs) for mild steel in 0.1 M HCl for inorganic inhibitor (K<sub>2</sub>CrO<sub>4</sub>) at 343 K**

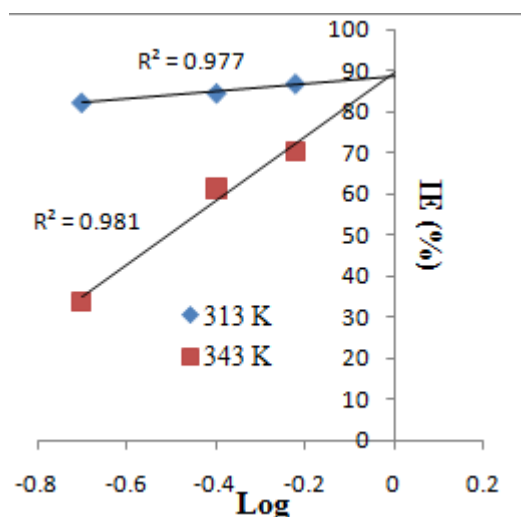


Fig. 5: Temkin adsorption isotherm plotted as IE against log C for plant inhibitor (*Sida acuta*) at 313 K and 343 K

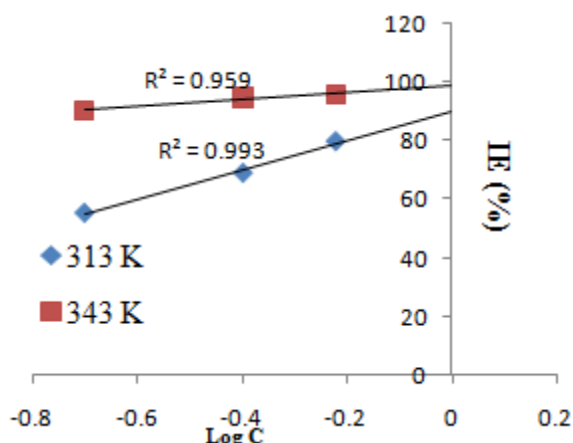


Fig. 6: Temkin adsorption isotherm plotted as IE against log C for inorganic inhibitor ( $K_2CrO_4$ ) at 313 K and 343 K

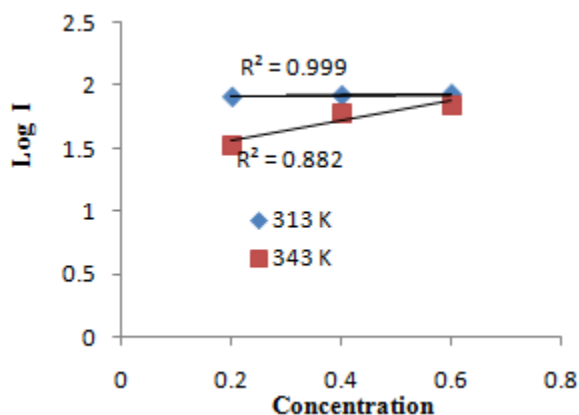


Fig. 7: Langmuir adsorption isotherm plotted as log I against Concentration for Plant (*Sida acuta*) inhibitor at 313 and 343 K

**Adsorption mechanism for the plant inhibitor**

The inhibition of metal corrosion by organic compounds is attributed to either the adsorption of inhibitor molecule or the formation of a layer of insoluble complex of the metal on the

surface which acts as a barrier between the metal surface and the corrosive medium (Okafor *et al.*, 2008).

Since no insoluble material was observed on the metal surface, the inhibitive action of the extract may be due to its adsorption on the metal surface. Investigations of the leaves of *Sida acuta* revealed the presence of the essential chemical compounds: saponins, flavonoids, alkaloids, glycosides. This makes it difficult to assign the observed inhibiting effect to a particular constituent. The net adsorption of these organic matters on the corroding mild steel surface creates a barrier that isolates the metal from the corrodent.

The relationship between inhibition efficiency and the bulk concentration of the inhibitor at constant temperature, which is known as isotherm (Tsuru *et al.*, 1978), gives an insight into the adsorption process. Several adsorption isotherms were attempted to fit surface coverage values to classical isotherms of Langmuir, Temkin, and Freundlich (Christov and Popova, 2004). The value of the correlation ( $R_2$ ) was used to determine the best fit isotherm which was obtained for Langmuir, and Freundlich isotherms.

**Conclusion**

*Sidaacuta*leaves ethanolic extracts (SALEE) was shown to be an effective green inhibitor for corrosion of mild steel in 0.1 M hydrochloric acid. The inhibition efficiency increased with increase in extract concentration, but decreased with increase in temperature. The inhibitive action is suggested to be realized through adsorption of extract phytochemical compounds onto mild steel surface as demonstrated by fitting to Langmuir adsorption isotherm. Thermodynamic data obtained support physisorption and spontaneous inhibition process. The presence of the extract increases the activation energy of the corrosion reaction. Activation energy, enthalpy and entropy were relatively small and thus independent of extract concentration.

The various indices that characterized the inhibitor behaviour in curtailing metallic deterioration and essentially prolonging the lifespan of the metal are presented in the research conclusion as follows:

- The plant inhibitor (SALEE) produced its best IE value of 86.667% at a concentration of 0.6 g/dm<sup>3</sup> at 313 K in 0.1 M HCl media, respectively for mild steel.
- The metal-inhibitor surface interaction mechanisms of all the green inhibitor systems were characterized by the Langmuir adsorption isotherm.
- The green inhibition systems showed mixed type inhibition mechanism. The inhibitors impacted the anodic metallic dissolution and the cathodic hydrogen evolution reactions (Omotosho, 2016).

**Conflict of Interest**

Authors declare that there is no conflict of interest related to this study.

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