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## Research Article

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## **Sesamum indicum as Green Surfactant Corrosion Inhibitor for 316L Steel in Hydrochloric Acid Solution.**

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### **Abstract**

Steel material equipment in oil and gas industries are frequently challenged with chlorides and sulfides corrosion attacks. This has been a major concerns due to regular equipment replacements or failures which consumes huge revenue and affects the economy of most oil producing nations. As a response to the above challenge, the characterization of corrosion inhibition of *Sesamum indicum* seed extract has been studied as green surfactant inhibitor for control of corrosion of austenitic 316L stainless steel in 9 M of hydrochloric acid solution using weight loss, potentiodynamic polarization, fourier-transform infrared spectroscopy analysis and optical microscopic image techniques. The inhibitor concentration, adsorption and functional group effects were inspected. Weight loss showed that the optimum inhibitor efficiency equals 51%, while the polarization measurement revealed that SI acted as mixed-type inhibitor. The adsorption behaviour of *Sesamum indicum* inhibitor on the surface of the austenitic 316L stainless steel obeyed Frumkin adsorption isotherm. The fourier-transform infrared spectrum demonstrated how the active functional groups of *Sesamum indicum* inhibitor were used up on the steel surface and confirms strong formation results. The surface morphology of stainless steel, in the absence and presence of *Sesamum indicum* inhibitor in hydrochloric acid solution was studied by optical microscopic image. Optical microscopic image reveled both the presence active chloride corrosion and the formation of protective layer of SI inhibition on the surface of the austenitic 316L stainless steel.

**Keywords:** Austenitic 316L stainless steel, Corrosion inhibition, *Sesamum indicum*, Hydrochloric acid, Economic impact, sustainable development goal.

## **Introduction**

Stainless steel materials still remain one of the most used steel materials for oil and gas equipment and structures. This frequent uses is due to its major properties such as hardness, strength, malleability, corrosion resistant and yield strength. Despite all the above excellent properties, the corrosion resistant of stainless steel equipment still fail in some high severe and corrosive chloride environments during oil and gas operations [1, 2]. For instant, the crude distillation overhead equipment is one major equipment used in the petroleum refineries, and it is mostly built with different kind of steel materials but are majorly challenged with corrosion reaction [3, 4]. This has been in existence for a long period of time, especially when high concentration chloride corrosion are present within the equipment, thereby causing severe damages to each functional part of the equipment. Also, every functional parts of the equipment are made of steels which makes the complete equipment vulnerable to corrosion attack. When corrosion causes damage to most industrial equipment, they do not only affect the industries but also the economy. This is due to the huge revenues involved in setting up an industrial equipment and its cost of proper maintenance incurred regularly.

Chlorides ions are formed during separation of crude oil constituents or process of desalting in oil and gas equipment [5-7]. When these chloride ions are directly exposed to steel equipment they causes redox reaction attacks to occur on the steel equipment [8-10], which eventually makes them to fail if not properly protected. Consequently, there is need to combat chloride corrosion affecting stainless steel equipment by seeking corrosion control method. This research seeks the use of organic inhibitor solution protection for stainless steel equipment from chlorides ion corrosion damage. The defense of steel equipment against chloride ion corrosion is of paramount importance because of its economic impact, sustainable industries and green environment.

The use of organic corrosion inhibition have been on the increasing usage for several years till date [11-16]. This is due to its excellent active properties, abundance availability, economic interest, high defense mechanism, sustainability nature and many more. They possess an hydrophobic behavior which helps to repel most aggressive or severe ions that attacks steel surfaces and causes damage to them. They form strong defense protection on steel surfaces,

via chemisorption or physisorption process. One of the interesting nature about organic inhibitors, is the great advantage of donating their free electrons (S, N, O atoms) to fill up the partial or empty d-orbitals of most transition metal steel (Cr, Fe) to make them keep their electrons rather than losing them to corrosion. This usually occurs when lone pair electrons from organic inhibitors are bonded with vacant d-orbitals of the steel surfaces. The formation of protected shield on steel surfaces are strongly guaranteed, when double or triple bond and aromatic rings of organic inhibitors helps to produce pi electron clouds. All of the above have enhanced the continuous use of organic corrosion inhibition for protection of steel structures and industrial equipment. Different forms of organic corrosion inhibitions have been examined, with excellent result and successful outcomes [17-21].

In addition, the use of organic corrosion inhibition strongly aligns with the UN sustainable development goal (SDG), by replacing most of the toxic synthetic inhibitors, because organic inhibitors are plant based and biodegradable in nature. SDG 6 (clean water and sanitation) and SDG 12 (responsible consumption and production) are both directly in support of organic corrosion inhibitor. Organic inhibitors promotes no toxicity (less water pollution), which aligns with SDG 6's clean water and sanitation. Likewise, usage of organic inhibitors in the industries or anywhere is a responsible consumption, which is safe and their production is equally safe, as aligned with SGD 12. Most industrial consumption of organic corrosion inhibitors are safe, sustainable and clean environment for most aquatic, because they eventually dissolve in water, and also forms carbon dioxide. As a result of high consumption and demand of organic inhibitors because of their safety, it has prompted large scale agricultural production because organic inhibitors are made from agricultural waste or products, plant seed extracts, by products from food industries, etc. They reduce hazardous waste because of their biodegradable nature. Furthermore, organic corrosion inhibitors usage, also aligns with SDGs 3 (good health and well-being), 8 (decent work and economic growth), 9 (industry, innovation and infrastructure) and 13 (climate action). During usage of organic inhibitors in the industries, good health and well-being of SGD 3 is guaranteed on any individual's exposure, because they are less toxic to human being. Over many decades now, organic inhibitors have promoted economic growth of SGD 8, of most nations of the world because they are not expensive to produce. Rather, they have helped to reduce corrosion destruction that affects the economic growth of most nations due to the huge revenues used to tackle corrosion damages on most producing industries. Indirectly, organic inhibitor is a great innovation and has helped with more industries and more infrastructures as expressed by SGD 9. The creation of organic corrosion inhibitors have

improved better steel or metal materials inhibitions and lower both steel materials wastage and embodied carbon. Also, organic inhibition on steel materials have lowered carbon footprint which aligns strongly with the climate action of SDG 13.

However, in this presence research, *Sesamum indicum* is use as a green surfactant corrosion inhibitor for austenitic 316L stainless steel in high corrosive 9 M hydrochloric acid solution. *Sesamum indicum* is either a seed form: which are flat, tiny and by varieties, are white, brown, gold, black in color, or in form of oil: which possesses natural antioxidant (sesamolin, sesamol, sesamin) and contains 50 to 60% oil, high oleic and linoleic acid. It is used as cooking oil, cosmetics, lubricant, pharmaceuticals, soaps, traditional medicine (for skin, joint, hair), bakery etc. They act as decent inhibitors because they cover more surface structures due to their flat aromatic image or rings, cheap to access when compared with other synthetic ones, toxicity free, excellent electron donator, high electron density, biodegradable, possess O atoms, hydrophobic film formation (retards  $\text{Cl}^-$  or  $\text{H}^+$ ). This research deliberately apply high corrosive concentration, due to the area of application in the oil and gas industry (specially targets the crude atmospheric distillation column) and *sesamum indicum* inhibitor is used based on its numerous natural properties. The uniqueness of this research work is using electrochemical approach in high concentration of hydrochloric acid (9 M) on austenitic 316L stainless steel material and surfactant SI inhibitor protection. This research aims at maintaining the sustainable economy of affected oil and gas nations and sustainable environment.

## Experimental Analysis

### Materials

The austenitic 316L stainless-steel plate material was commercially available to use for the corrosion study and the elementary composition of the material was performed using the X-ray Fluorescence spectroscopy equipment at the University of Ibadan. The elementary composition summary result is illustrated in table 1 below:

**Table 1**

The results of Austenitic 316L stainless steel elementary composition [22].

| Element   | Cr | Ni | Mo | C    | P    | Fe    | S    | Si   | Mg | N   |
|-----------|----|----|----|------|------|-------|------|------|----|-----|
| Content % | 16 | 10 | 2  | 0.03 | 0.05 | 69.05 | 0.03 | 0.75 | 2  | 0.1 |

### Preparation of the test steel material

The square surface of  $1.0\text{ cm}^2$  were machined with thickness 0.3 cm at each ends of material. The material specimen were further prepared with abrasive papers of silicon types 80, 120, 220, 800, and 1000 grits, before being polished with  $1\text{ }\mu\text{m}$  portion diamond paste, washed with distilled water, rinsed with acetone, dried, and stored in a desiccator for the research study on weight loss analysis, while the samples for potentiodynamic polarization analysis and open circuit potential were mounted on resin with an exposed surface on one end and a connecting wire on the resin-covered end for electrochemical process.

### Medium

A stock solution of 9.0 M of hydrochloric acid was diluted with distilled water, to create a simulated corrosive medium for the research analysis. The hydrochloric acid contains  $1.49\text{ g/cm}^3$  density, trademark purity is 37% and molar mass of 36.46 g/mol.

### Preparation and characterization of *Sesamum indicum* (SI)

The organic seed plant of sesame product known as *Sesamum indicum* (SI) was purchased commercially from the open market in Ota and the preparation was achieved into lipid surfactant inhibitor in line with literature approach [23]. Then saturated with n-hexane  $\text{C}_6\text{H}_{14}$  extractor solvent which was added underneath the flask for the extraction to be achieved, through heating process.

### FTIR Spectroscopy Analysis

The FTIR spectrum test samples of *Sesamum indicum* (SI), before and after the electrochemical study (liquid samples) were recorded using spectroscopy to identify the functional groups and the molecular species possessed by SI inhibitor from the range of wavenumbers of  $400\text{--}4000\text{ cm}^{-1}$ , were achieved.

### Mixture of medium and inhibitor

After, the surfactant lipid inhibitor had been achieved, the mixture of the simulated corrosive solution and lipid inhibitor were mixed. The simulated medium were prepared into 400 ml of 9.0 M HCl (37% annular grade) solution with distilled  $\text{H}_2\text{O}$ , and SI inhibitor were administrated into the medium in the proportion of 0.63%, 1.25%, 1.88%, 2.50% and 3.13% percentage concentration.

Phytochemical Analysis of *Sesamum indicum* (SI) organic compound.

The phytochemical analysis test for *Sesamum indicum* (SI) surfactant lipid inhibitor was performed at the department of Chemistry, Covenant University and the result is shown in Table 2 below:

**Table 2.**

Results of phytochemical screening analysis test for *Sesamum indicum* (SI) organic compound.

| S/N | Chemical components | SI |
|-----|---------------------|----|
| 1   | Alkaloids           | +  |
| 2   | Saponins            | +  |
| 3   | Glycosides          | +  |
| 4   | Steroids            | -  |
| 5   | Terpenoids          | +  |
| 6   | Quinones            | +  |

KEY

Present +

Absent -

### Electrochemical methods

The fundamental electrochemical approach were carried out to perform the corrosion study for this research. The weight loss method were setup experimentally by exposing the prepared austenitic 316L stainless steel material samples into the prepared simulated HCl acid solution with and without SI surfactant inhibitor, for the period of 504 hours for each 316L steel material samples. The entire experiment were exposed to the atmosphere and the samples were fully immersed at ambient temperature. Each individual samples were removed, rinsed, dried and weighed at every 24 hours for the period of 504 hours. The data were recorded and further calculations were achieved with equations 1 and 2 [24], to achieve the corrosion rate, the surface coverage, and the inhibitor efficiency, respectively.

$$C.R = \left[ \frac{87.6 \times W}{D \times A \times T} \right] \quad (1)$$

Where,  $W$  represents weight loss (g),  $D$  represents density (g/cm<sup>3</sup>),  $A$  represents area (cm<sup>2</sup>), and  $T$  represents immersion time (h). Inhibition efficiency ( $\eta$ ) was approximated from the mathematical expression;

$$\eta = \left[ \frac{\omega_1 - \omega_2}{\omega_1} \right] \times 100 \quad (2)$$

$\omega_1$  represents weight loss of the alloys without SI inhibitor in the electrolyte while  $\omega_2$  represents weight loss of the alloys at definite SI inhibitor concentrations.

Also, the potentiodynamic polarization and open circuit potential measurements were achieved by using the Gamry DY2307 work station instrument. A conventional three way electrode connection system was used with a glass like cell platinum counter electrode and a silver chloride electrode as reference electrode. The potentiostat is connected to a computer system with the appropriate software for analysis. The software is launched and the desired operation to be carried out and the parameters needed to carry out the process is input. The square shaped 316L austenitic stainless-steel was used as the working electrodes.

#### Tafel Polarization studies

Shining mirror like polished specimen was exposed to the simulated solution of 9.0 M HCl acid medium in the absence and presence of SI inhibitor at room temperature and permitted to demonstrate a steady state for open circuit potential study. Also, the open circuit potential was achieved at scan rate of  $1 \text{ mV s}^{-1}$ , to achieve a potentiodynamic polarization current-potential graphs. Tafel curve were produced for a potential range of -250 mV cathodically to +250 mV anodically in relation to the open circuit potential.

#### Surface Morphology Examination

The surface morphology of the austenitic 316L stainless steel specimen immersed in 9.0 M HCl solution in the absence and presence of SI inhibitor, for the exposed region were imaged by optical microscopic image equipment.

### Results and discussion

FTIR (Fourier transform infrared) spectroscopy of SI Inhibitor.

Figure. 1 indicates the spectra curves of the austenitic 316 stainless steel in 9 M HCl acid solution and SI inhibitor, showing the existing bonds formation on the steel surfaces via film adsorption protection before and after  $\text{Cl}^-$  corrosion reaction. SI organic inhibitor compound behaved as proton donating electrons by protecting the surface of the steel due to its heteroatomic nature via existing bonds such as unshared paired electron, nitrogen, oxygen, double, aliphatic, alkynes [25-27]. Observation from figure 1 shows that before corrosion the curve appears higher than after corrosion with drastic reduction in red curve, this indicates

strong adsorption of presence of functional group used up on the surface of the steel for adequate coverage. The blue curve indicates higher power potential, that is the transmittance (%) increased for SI organic inhibitor compound before corrosion occurred but the curve (red) was lowered after corrosion. This signifies that the functional groups present and molecules of SI organic inhibitor compound had been used up on the surface of 316L steel. Some of the stretching frequencies are recorded, indicating their bonds and functional groups appeared at  $1000\text{ (cm}^{-1}\text{)}$  wavenumber,  $600.17\text{ (cm}^{-1}\text{)}/\text{C-Br}$  stretch (alkyl halides),  $744.00\text{ (cm}^{-1}\text{)}/\text{C-Cl}$  stretch (alkyl halides) and  $871.00\text{ (cm}^{-1}\text{)}/\text{C-H}$  “oop” (aromatics). This suggest that SI inhibitor performed excellently with strong adsorption level.

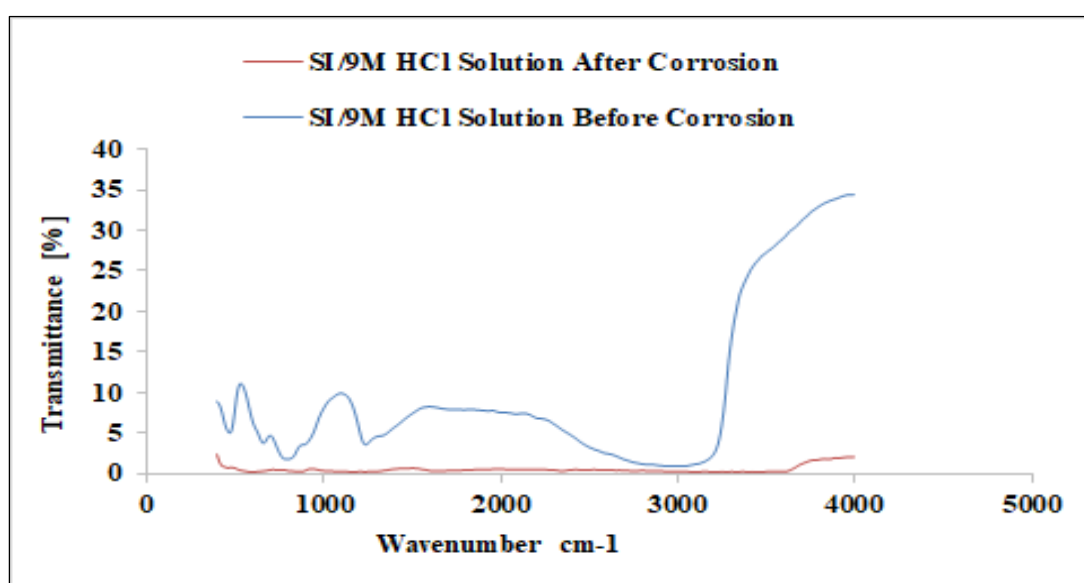


**Figure 1.** Results of Fourier transform infrared spectra of liquid residue of SI.

### Weight Loss

The influence of SI inhibitor on the corrosion reaction was determined by weight loss approach. The weight loss values observed and determined by measurement before and after corrosion and SI inhibitor are usually used to evaluate the vales for corrosion rate, surface coverage and inhibitor efficiency [28, 29]. Table 3 and Figure 1 shows the weight loss analysis parameter values and curves for austenitic 316L stainless steel in 9 M HCl acidic solution at different SI inhibitor concentrations respectively. Also, in this research, the weight loss values were obtained from weight loss experimental analysis and parameters such as corrosion rate, surface coverage, and inhibitor efficiency were all calculated using equations 1 and 2. The result from figure 2 and table 3, reveals that the control sample in 9 M HCl without SI inhibitor resulted into the highest weight loss and corrosion rate values. This also confirms the presence of strong

Cl<sup>-</sup> corrosion effect on the control sample due to the drastic decrease in weight loss from the initial measurement of the control sample. Upon increasing the addition of SI concentration, there was a drastic decrease in weight loss values at all levels of SI concentrations and increase in inhibitor efficiencies for all the 316L samples respectively. This suggests an excellent film formation shield on the surface of all 316L samples with SI concentrations that hinder further Cl<sup>-</sup> corrosion effect on those samples. This also confirms that SI inhibitor truly possess all the presence of bonds and functional groups that are responsible for strong firm protection as shown in figure 1.

**Table 3**

Results of weight loss of austenitic 316L stainless steel in 9M HCl containing different SI inhibitor concentrations.

| Sample  | WL (g) | CR (mm/y) | C (%) | SC (θ) | IE (%) |
|---------|--------|-----------|-------|--------|--------|
| Control | 0.2751 | 0.154     | 0.00  | -      | -      |
| A       | 0.1886 | 0.073     | 0.63  | 0.431  | 43.18  |
| B       | 0.1812 | 0.050     | 1.25  | 0.466  | 46.67  |
| C       | 0.1896 | 0.041     | 1.88  | 0.474  | 47.40  |
| D       | 0.1825 | 0.039     | 2.50  | 0.496  | 49.65  |
| E       | 0.1717 | 0.038     | 3.13  | 0.515  | 51.58  |

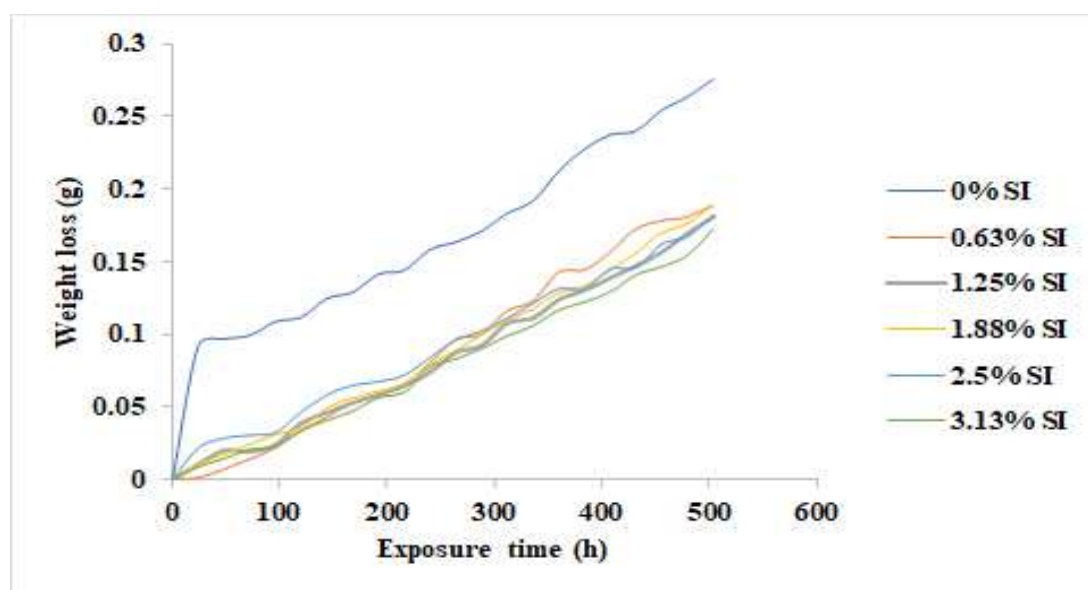


**Figure 2.** Weight loss curve for austenitic 316L stainless steel in 9M HCl containing different SI inhibitor concentrations.

### Potentiodynamic polarization

Potentiodynamic polarization approach was also used to examine the effectiveness of SI on corrosion reaction of 316L steel in 9 HCl solution. Literature explains that when inhibitors are added during the plot of potentiodynamic polarization curves, shifts occurs and those shifts are used as criteria to determine the categories of inhibitors into anodic, cathodic or mixed inhibitor [30-32]. The potentiodynamic polarization parameters such as corrosion current density ( $I_{corr}$ ), corrosion potential ( $E_{corr}$ ), anodic Tafel slope ( $b_a$ ), cathodic Tafel slope ( $b_c$ ), were all obtained from the Tafel plots and recorded in table 4. Figure 3 shows the result of Tafel curves for austenitic 316L in 9M HCl acidic solution at different SI inhibitor concentrations.

From table 4 and figure 3, observation revealed that the addition of SI at all values of concentrations studied caused significant changes in the polarization curve and parameters. Firstly, as the concentrations of SI increases, there was decrease in corrosion rate (CR) and decrease in corrosion current density ( $i_{corr}$ ) values. Secondly, a slight remarkable shift in region was observed as demonstrated by the corrosion potential values ( $E_{corr}$ ), which indicates that both the cathodic and the anodic region are influenced by SI but predominantly anodic shift [33]. This also confirms that both metal dissolution and hydrogen evolution are influenced by SI concentrations [34]. Furthermore, research from literatures established that when corrosion potentials values are within the range of positive (+) or negative (-) 85 mV, the inhibition behaved both as anodic or cathodic type inhibitors, indicating mixed type of inhibitor [35-38]. However, the values of corrosion potentials indicates that SI behaved as a mixed type of inhibitor because all the values are not up to  $\pm 85$  mV, as shown in table 4.

**Table 4**

All results of Tafel polarization studies on 316L stainless steel in 9 M HCl acid solution containing different SI inhibitor concentrations.

| SI C (%) | $E_{corr}$ (V) | $I_{corr}$<br>( $\times 10^{-5} \text{ A cm}^{-2}$ ) | CR<br>(mm/y) | IE (%) | $b_a$<br>(V/dec <sup>-1</sup> ) | $-b_c$<br>(V/dec <sup>-1</sup> ) |
|----------|----------------|------------------------------------------------------|--------------|--------|---------------------------------|----------------------------------|
| 0        | -0.36          | 11.69                                                | 0.154        | -      | 26.61                           | 9.57                             |
| 0.63     | -0.37          | 2.31                                                 | 0.073        | 43.18  | 32.36                           | 2.18                             |
| 1.25     | -0.30          | 0.45                                                 | 0.050        | 46.67  | 47.11                           | 3.26                             |
| 1.88     | -0.12          | 0.39                                                 | 0.041        | 47.40  | 6.77                            | 6.24                             |
| 2.50     | -0.11          | 0.08                                                 | 0.039        | 49.65  | 18.79                           | 5.57                             |

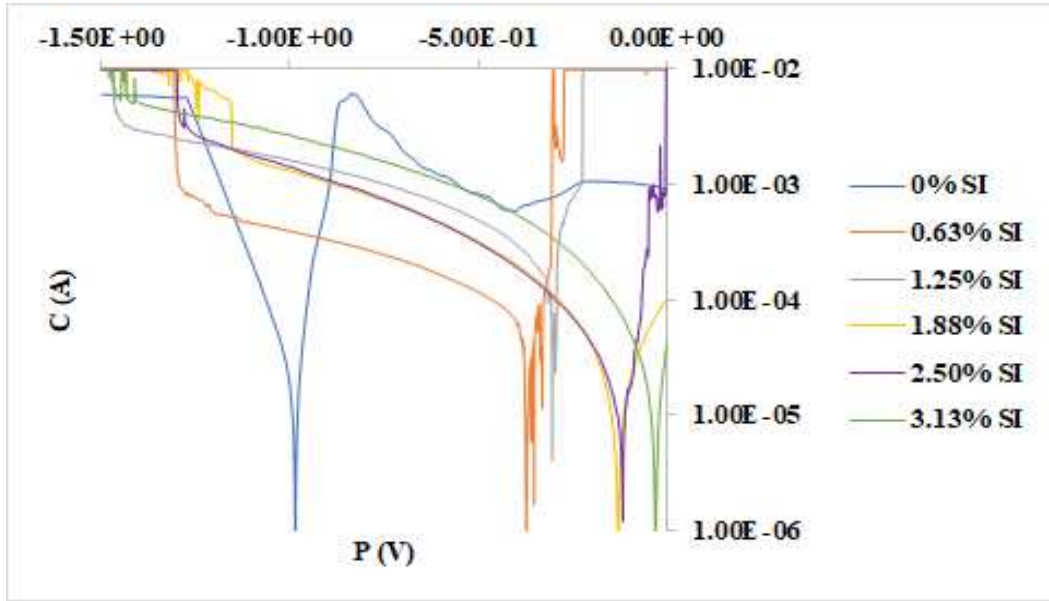


**Figure 3.** The Tafel polarization curves for austenitic 316L stainless steel in 9 M HCl acid solution at different SI inhibitor concentrations.

#### Adsorption Isotherm Behaviour

The interaction that creates the allowance of inhibition formation to occur between the steel (metal) surface and the inhibitor molecules is known as the adsorption isotherm behaviour. The surface coverage ( $\theta$ ) for each different SI concentrations was obtained from the weight loss studies. The surface coverage data obtained was further applied to various adsorption isotherms, such as, Temkin, Langmuir, Frumkin and Freundlich in order to realise the best fit for the research. After different test with the surface coverage ( $\theta$ ) data on the various adsorption isotherms behaviours, Frumkin was found to be the best fits with regression value of 0.9972 and was represented with the relationship in equation 3 [39]:

$$\beta C = \frac{\theta}{1-\theta} \exp (-2g\theta) \quad (3)$$

The experimental data plot is  $\ln \left[ \frac{\theta}{1-\theta} \right]$  vs  $\theta$  and the slope –  $g$ , and the intercept is written as  $\ln (\beta C)$ , where  $\beta$  = adsorption equilibrium constant,  $C$  = concentration of adsorbate in bulk solution,  $g$  = Frumkin interaction parameter and  $\theta$  = surface coverage, which is computed with the relation in equation 4 [40]:

$$\theta = \left[ \frac{\omega_1 - \omega_2}{\omega_1} \right] \quad (4)$$

where  $\omega_1$  represents weight loss of the alloys without SI inhibitor in the electrolyte while  $\omega_2$  represents weight loss of the alloys at definite SI inhibitor concentrations. Also, the plot of  $\text{Log} \frac{\theta}{1-\theta}$  vs  $\theta$  resulted in a straight line with an intercept of  $\beta$ . Therefore, the Frumkin adsorption isotherm for the adsorption of SI inhibitor on austenitic 316L stainless steel surface in 9 M HCl acidic solution are displayed in figure 4.

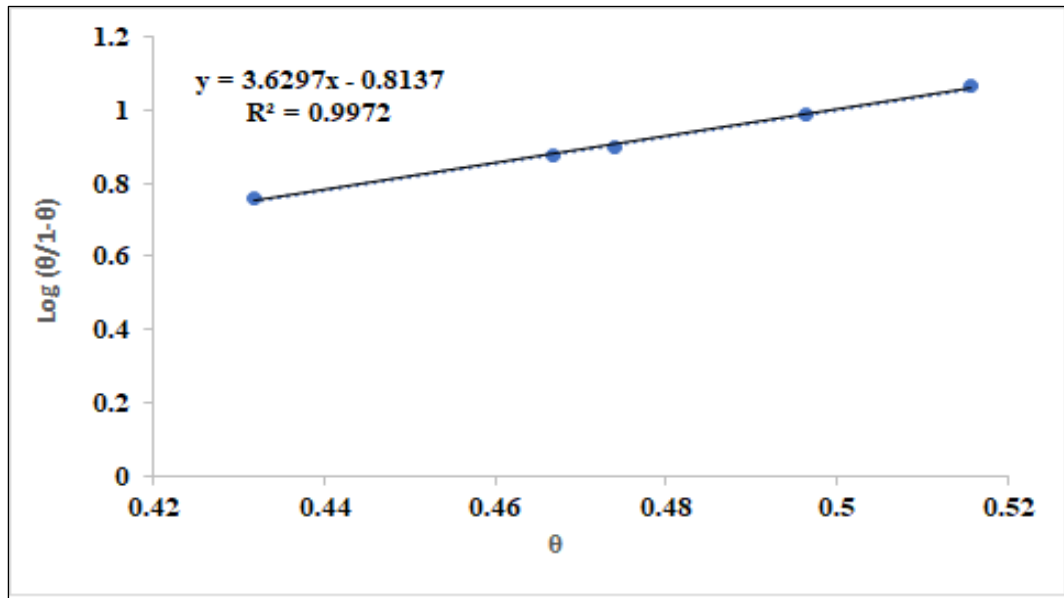


**Figure 4.** Frumkin adsorption isotherm for the adsorption of SI on austenitic 316L stainless steel surface in 9 M HCl acidic solution.

### Surface Morphology

The surface morphology test of austenitic 316L stainless steel in chloride corrosion and SI treatment is performed by using optical microscopic image (OMI). The OM image of a shining surface of austenitic 316L stainless steel before immersion is given in figure 5 without chloride corrosion attack. A close view of the OM image in figure 6 shows slight spot of uniform and pitting corrosion which confirms the presence of  $\text{Cl}^-$  ion corrosion attacks on the surface of the 316L stainless steel. Few forms of pits were observed on the surface of the steel and uniform corrosion across the surface of the austenitic 316L stainless steel. Figure 7 represents the austenitic 316L stainless steel surface after treatment with SI inhibitor. It confirms the reduction of corrosion attack and indicates the formation of protective layers of SI inhibitor on the surface of austenitic 316L stainless steel. OM image has proved excellent confirmation for the morphology characterization of steel materials surfaces and literatures have showed results of the test [41, 42].

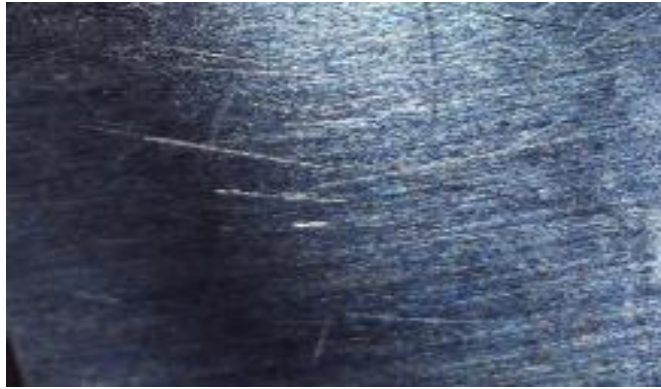


**Figure 5.** Optical Microscopic image of austenitic 316L stainless steel before immersion. (mag x 10).



**Figure 6.** Optical Microscopic image of austenitic 316L stainless steel after immersion in 9 M HCl solution. (mag x 10).



**Figure 7.** Optical Microscopic image of austenitic 316L stainless steel after immersion in 9 M HCl solution and SI. (mag x 10).

## **Conclusion**

According to the results of all the examination performed in this research, the following conclusions are drawn:

1. SI performed as a good green surfactant inhibitor for corrosion mitigation of austenitic 316L stainless steel in 9 M HCl acidic solution.
2. SI aligns with about six (6) United Nations sustainable development goal (SDG).
3. SI contains excellent phytochemical constituents such as alkaloids, saponins, glycosides, terpenoids, and quinones which all aided strong inhibition mechanisms.
4. The inhibitor efficiency of SI increases as SI concentrations increases.
5. FTIR spectrum confirms that SI was used up on the surface of austenitic 316L stainless steel and revealed numerous functional groups and bonds possessed by SI.
6. SI acted as mixed type inhibitor by inhibiting both anodic and cathodic reaction but predominantly anodic reaction.
7. The adsorption isotherm behaviour of SI on the surface of austenitic 316L stainless steel follows Frumkin adsorption isotherm.

### **Declaration of Fund**

The authors declare that no financial commitment or interest that could influence the study report of this research article.

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