

*Research article*

## **Combating A36 mild steel corrosion in 1 M H<sub>2</sub>SO<sub>4</sub> medium using watermelon seed oil inhibitor**

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**Abstract:** Corrosion inhibitive performance of the extracted watermelon seed oil on A36 mild steel in 1 M H<sub>2</sub>SO<sub>4</sub> medium, at 305 and 319 K corrosion reaction temperatures, was investigated. Weight loss and inhibition efficiency were determined using gravimetric method while corrosion rate and inhibition efficiency were evaluated using potentiodynamic tests. Gravimetric tests showed that watermelon seed oil inhibitor attained a better corrosion inhibition efficiency of 50% at the operating temperature of 305 K compared to an efficiency of 48% obtained at the operating temperature of 319 K. Electrochemical potentiodynamic polarization tests showed that 3 vol/vol% inhibitor concentration gave the most promising corrosion inhibiting results at the operating temperature of 305 K, while 4 vol/vol% inhibitor concentration gave the most reliable corrosion resisting one at the operating temperature of 319 K. Langmuir adsorption isotherm correctly predicted the adsorption behaviour of the watermelon seed oil on A36 mild steel surface in 1 M H<sub>2</sub>SO<sub>4</sub> medium. The negative values of  $\Delta G_{ads}$  revealed the spontaneous adsorption nature of the inhibitor on the mild steel surface and the electrochemical potentiodynamic polarization results showed that the watermelon seed oil acted as a mixed-type corrosion inhibitor. The optical image analysis revealed both the potency level of watermelon seed oil as an inhibitor as well as the optimum inhibitor concentrations of 3 vol/vol% (at 305 K) and 4 vol/vol% (at 319 K).

**Keywords:** corrosion; inhibitor; mild steel; watermelon seed oil; weight loss

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## 1. Introduction

Metals and alloys have wide areas of applications in engineering constructions and buildings, chemical and food processing industries, aviation and automobile industries, as well as petroleum companies. These metals are all prone to corrosion at rates which are primarily function of the nature of the corrosive environment, hence, concerted efforts must be progressively taken so as to reduce the corrosion risks of the metallic components and/or system involved [1,2].

The use of corrosion inhibitors is a trending and important way of combating metal corrosion process. Corrosion inhibitors are chemical substances added in little quantity to the corrosive environment in order to slow down or prevent corrosion reactions of metal from taken place [3,4]. Based on performance, corrosion inhibitors are generally classified as passivators precipitators, vapor phase, cathodic, anodic, neutralizing and absorbents [4]. But based on composition, corrosion inhibitors can be classified into two major groups: organic and inorganic inhibitors. Examples of the inorganic inhibitor include hydroxides, nitrates, molybdates, silicates and chromates of metals while examples of the organic inhibitors include the extracts from leaves, barks, nuts, seeds, fruits and roots [5,6].

Recently, the beam light of research findings on the exploration of various inhibitors has been extended to the determination of solubility, toxicity, thermal stability and cost of the inhibitors [7,8]. According to Lei et al. [7], 3,3-dithiodipropionic acid (DDA) inhibitor was found to be non-toxic, highly soluble and mixed-typed corrosion inhibitor for Q235 steel in 0.5 M  $\text{H}_2\text{SO}_4$ . It is also reported that the comparative cost analysis of inhibitors and inhibitive performance of the inhibitors are important factors to consider in the determination of suitable inhibitor to consider for a metal in a given medium [8].

Research has shown that certain inorganic inhibitors used in industries are toxic in nature thereby pose a threat to humans and their environment. And their toxicity thereby paves the way for the exploration of the environmentally friendly and biodegradable natural inhibitors. The use of natural or organic materials in corrosion control is also being supported due to the fact that they are readily available, cheaper, ecologically acceptable and processed through simple extraction procedures [9].

The efficiency of the natural inhibitors depends on the chemical structures of their organic compounds, ability to become cross-linked or compact, the number and types of bonding atoms or groups in the compounds and ability to form a solid complex with the atoms within the metal lattice. In terms of inhibitive performance, an organic corrosion inhibitor can be referred to as anodic, cathodic, or both (mixed typed) inhibitor [10,11]

Diverse research has been conducted in order to study the inhibitive properties of various plant extracts and leaves. The corrosion inhibitive behaviour of these plant extracts were found traceable to the presence of certain antioxidants which are heterocyclic in nature, these include alkaloids, flavonoids and tannins [12–16].

Few attempts have been made regarding the use of different parts of watermelon plants as corrosion inhibitors for metals subjected to different corrosive conditions. For example, extracts from the watermelon peel and watermelon leaves have been used in natural seawater to protect zinc from corrosion [17]. Watermelon seeds are waste materials that are very easy to come across and their oil can be easily extracted for corrosion inhibition purpose.

The novelty of this research work is in the population of the lean research databank on the use of watermelon seed oil as a corrosion inhibitor for mild steel in acidic medium. That is, few attempts have been made in the establishment of the corrosion inhibitive performance of the watermelon seed oil. Though it was reported that watermelon seed oil performed better, as inhibitor for aluminium metal in saline (sea) water compared to acetic acid or sulphuric acid with the inhibitor concentration of 0–2 wt% [18], but the inhibitive effects of watermelon seed oil on mild steel need to be considered and documented.

This research is aimed at studying the corrosion inhibitive effects of watermelon seed oil on A36 mild steel in acidic medium of  $H_2SO_4$ . The study will investigate inhibitive performance of the extracted watermelon seed oil on A36 mild steel by considering both the weight loss and potentiodynamic tests at two corrosion temperatures, the establishment of the suitable adsorption mechanism of the inhibitor, as well as the thermodynamic properties of the inhibitor at the prevailing conditions.

## 2. Materials and methods

### 2.1. Material, reagents and equipment used

A36 mild steel and watermelon seed were the materials used in the course of this research work. The analytical grade reagents used include  $H_2SO_4$  (98% purity), hexane (95%), NaOH (96%), silica gel desiccant (embedded in desiccator) and distilled water. The equipment used were digital weighing balance, soxhlet extractor and Autolab PGSTAT 302N.

### 2.2. Preparation of the inhibitor and metal samples

Oil was obtained from grinded watermelon seed by subjecting the fine powder of the grinded watermelon seed to soxhlet oil extraction process using hexane as the solvent. To every 100 g of watermelon seed powder used, 150 mL of hexane solvent was utilised during the oil extraction process operated at the temperature of 70 °C for 4 h. A36 mild steel sheet used was cut into the dimension of 2 cm × 2 cm × 0.2 cm.

Table 1 shows the elemental analysis of the A36 mild steel used. The samples were abraded with emery paper in order to remove debris on the metal surface and to attain smooth surface of the samples. The samples were then subjected to degreasing (through the use of acetone) and air drying (at 40 °C) before being stored in desiccators that were carefully packed with silica gel. The weights of the metal samples were recorded and the samples were meticulously tagged based on the experimental design.

**Table 1.** A36 mild steel constituents.

Metal	Composition%
Cu	0.22
Cr	0.07
Al	0.03
Mo	0.08
Ni	0.09
Mn	0.51
C	0.24
Fe	98.58

### 2.3. *Phytochemical analysis of the oil extracted*

The phytochemical constituents of the watermelon oil extracted were analyzed by considering the standard laboratory techniques for quantitative determinations.

#### 2.3.1. Test for tannin

3 mL of distilled water was added to 0.5 g of the oil extract in a test tube, 3 drops of dilute chloroform and 1 mL of acetic anhydride were added and shaken thoroughly. Finally, 1 mL of sulfuric acid was carefully added by the side of the test tube to the solution.

#### 2.3.2. Test for saponin

0.5 g of the oil extract was added to 25 mL of distilled water in a test tube and the mixture was vigorously shaken for about 15 min.

#### 2.3.3. Test for terpenoid

2 mL of chloroform was added to 5 mL oil extract and the mixture was heated slightly for 10 min, after the addition of 3 mL H<sub>2</sub>SO<sub>4</sub> concentrated.

#### 2.3.4. Test for flavonoids

0.5 g of the oil extract and 5 mL of distilled water were mixed together in a test tube. Few drops of NaOH solution were added to the aqueous solution.

#### 2.3.5. Test for glycosides

1 g of the oil extract and 3 mL of distilled water were added. About 5 mL of concentrated H<sub>2</sub>SO<sub>4</sub> was then added to the aqueous solution of the oil extract.

## 2.4. Corrosion tests

Different concentrations of the oil extracted (0–4 vol/vol%) and 200 mL of 1 M H<sub>2</sub>SO<sub>4</sub> acidic were used in the course of the gravimetric (weight loss) experiments. The weight loss experiments involved the suspension of metal samples in 1 M H<sub>2</sub>SO<sub>4</sub> acid solution containing varied inhibitor concentration within varied period of 72 h, for metal corrosion to occur. The weight of each of the sample was carefully determined before and after corrosion. The experiment was carried out at different temperatures of 305 and 319 K. Weight loss due to corrosion was evaluated. The inhibition efficiency (IE%) and surface coverage ( $\theta$ ) were also calculated using Eqs 1 and 2.

$$IE\% = \frac{W - W_i}{W} \times 100 \quad (1)$$

$$\theta = \frac{W - W_i}{W} \quad (2)$$

where  $W$  and  $W_i$  are the weight loss without and with inhibitor respectively.

The electrochemical experiment was made possible through the use of Autolab PGSTAT 302 N potentiostat and electrode cell containing 200 mL of 1 M HCl electrolyte (in the presence and absence of watermelon seed oil inhibitor). Platinum electrode was used as the counter electrode, Ag/AgCl as the reference electrode, and the mild steel specimens were used as working electrodes. The potentiodynamic measurements were taken between the cathodic and anodic direction ( $\pm 250$  mV) at a scan rate of 10 mV/s.

The values of corrosion potential ( $E_{corr}$ ), corrosion current density,  $I_{corr}$ , (A/cm<sup>2</sup>), corrosion rate and polarization resistance were obtained using the experimental measurements from the Tafel plots of potential  $E$  (V) against log current  $I$  were made. Inhibition efficiency (IE%) and surface coverage ( $\theta$ ) were also determined using Eqs 3 and 4 respectively.

$$\theta = 1 - \frac{I_{corr}}{I_{ocorr}} \quad (3)$$

$$IE\% = 1 - \frac{I_{corr}}{I_{ocorr}} \times 100 \quad (4)$$

where  $I_{corr}$  and  $I_{ocorr}$  are the current densities with and without inhibitor respectively.

## 2.5. Thermodynamics study of the inhibitor

The adsorption mechanism of the watermelon seed oil inhibitor on metal sample surfaces was determined through the consideration of the adsorption isotherms that suitably predict the manner in which the inhibitor was adsorbed. The adsorption isotherms considered for the thermodynamics study are as expressed in Eqs 5–8.

$$\text{Langmuir: } \frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (5)$$

$$\text{Langmuir-Freundlich: } \left[ \frac{\theta}{1-\theta} \right]^{\frac{1}{h}} = K_{ads} C_{inh} \quad (6)$$

$$\text{Temkin: } \exp(f\theta) = K_{ads} C_{inh} \quad (7)$$

$$\Delta G_{ads} = -2.303RT \log(55.5 K_{ads}) \quad (8)$$

where  $K_{ads}$  = adsorption equilibrium constant,  $C_{inh}$  = concentration of the inhibitor,  $f$  = factor of energetic inhomogeneity,  $h$  = heterogeneity parameter ( $0 < h < 1$ ),  $R$  = Universal gas constant ( $8.314 \text{ JK}^{-1} \text{ mol}^{-1}$ ),  $T$  = absolute temperature (K) and  $\Delta G_{ads}$  = change in Gibbs free energy.

## 2.6. Microscopic image of the metal samples

The optical image analysis of the corroded metal samples (with and without inhibitor) was carried out on the metal samples subjected to the electrochemical tests in order to evaluate the effects of the varied concentrations of the inhibitor on the metal samples.

## 3. Results and discussion

### 3.1. Phytochemical analysis of watermelon seed oil

Table 2 revealed the antioxidant compounds present in the extracted water seed oil.

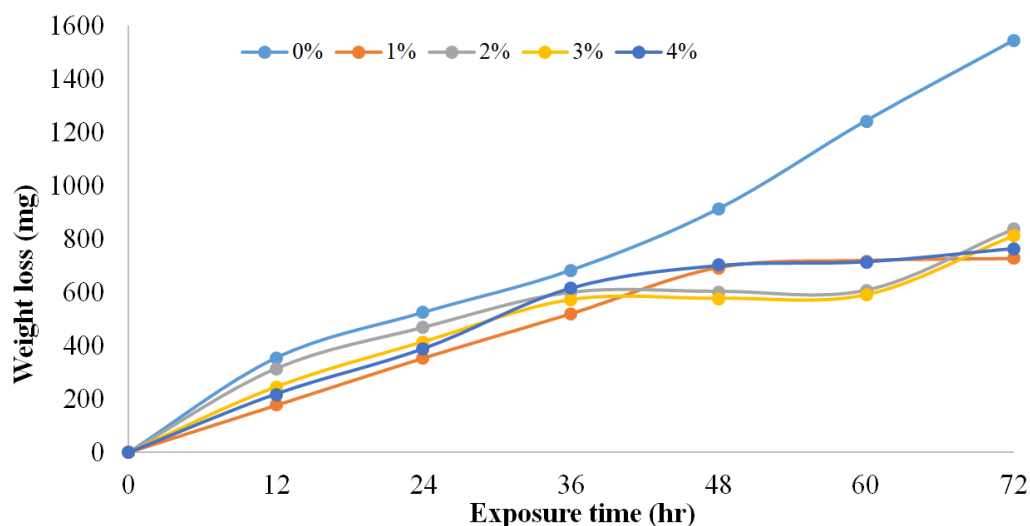
**Table 2.** Phytochemical analysis of watermelon seed oil.

Compound	Remark
Tannin	Present
Saponin	Present
Terpenoid	Absent
Flavonoid	Present
Glycoside	Present

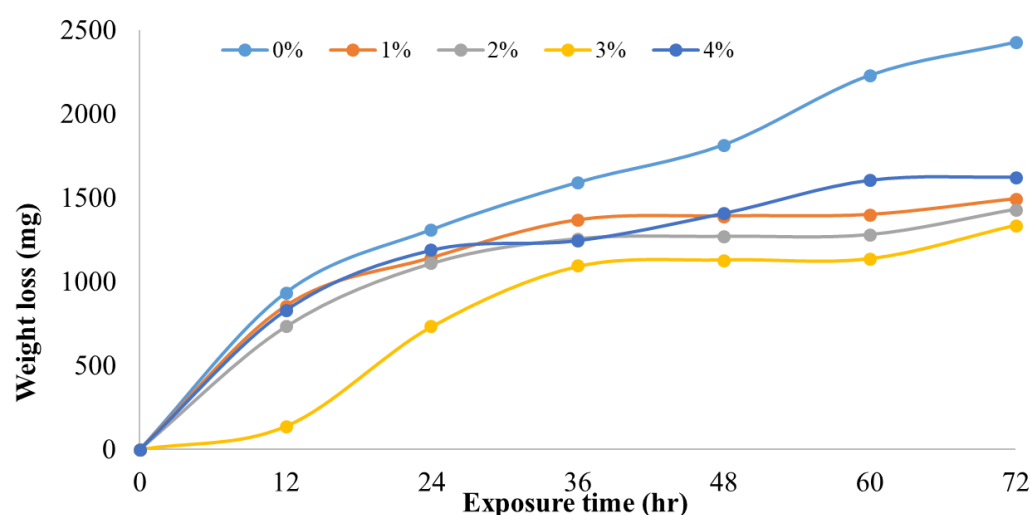
The presence of tannin was confirmed by a greenish coloration observed from the test carried out, saponin presence in the inhibitor was established through the formation of a foam layer at the top of the mixture in the test tube. From the flavonoids confirmation test, an intense yellow color was observed and the presence of glycoside was established by the formation of a reddish brown color solution. That is, the analysis indicated the presence of inhibitive (antioxidant) compounds such as tannins, saponins, flavonoids and glycosides that aided in the inhibitive action of the oil by serving as chemical barriers on the surface of the mild steel through adsorption thereby hindered (inhibit) the corrosion reactions [13].

### 3.2. Results of the weight loss tests

Gravimetric test results obtained are as shown in Figures 1 and 2 at operating temperatures of 305 and 319 K respectively. In general, it could be observed that the weight loss increased as the metal exposure time to corrosion increased. Also, the weight loss observed without the use of the watermelon seed oil inhibitor was more compared to the result obtained from the use of inhibitor, during corrosion process. This is an indication that watermelon seed oil was very effective as corrosion inhibitor.



**Figure 1.** Weight loss of A36 mild steel in 1 M H<sub>2</sub>SO<sub>4</sub> at 305 K at intervals of 12 h.

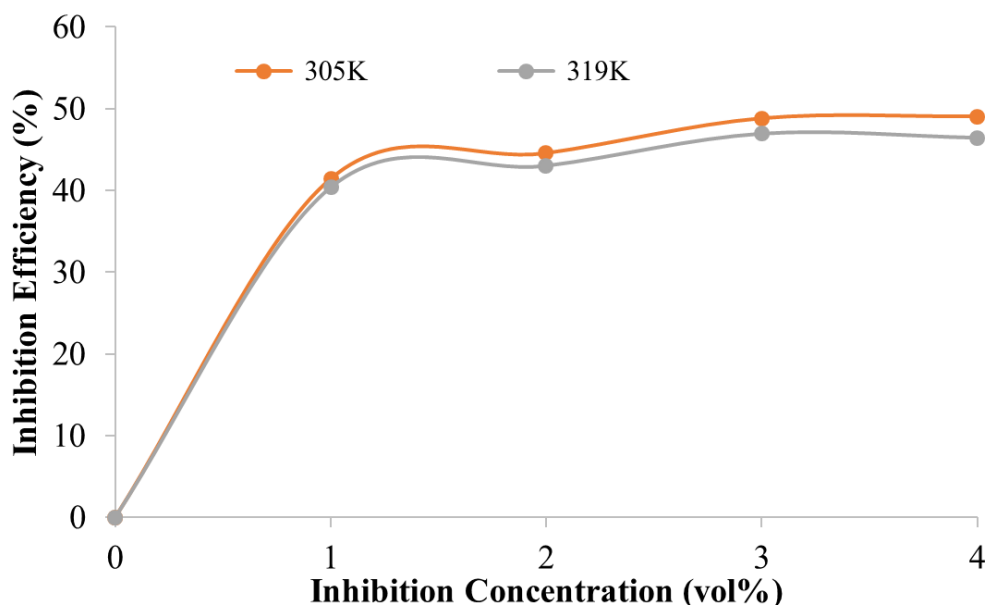


**Figure 2.** Weight loss of A36 mild steel in 1 M H<sub>2</sub>SO<sub>4</sub> at 319 K at intervals of 12 h.

The weight loss recorded at corrosion reaction of 319 K when a particular inhibitor concentration was used was higher than the corresponding weight loss obtained at corrosion reaction of 305 K for the same inhibitor concentration. That is, increase in corrosion reaction temperature amounted to increase in corrosion reaction rate (as measured in terms of weight loss). These results support the Arrhenius principle, as commonly reported in literatures [19]. Considering the inhibitor concentrations at a particular temperature, it could be seen that the weight loss obtained reduced as the inhibitor concentration increased, though the least weight loss value was noted at the concentration of 3 vol/vol% (both at temperature of 305 and 319 K). It is logical to conclude that the increase in inhibitor concentration retards the corrosion reaction thereby promotes the formation of the inhibitive layer at the metal surface through adsorption process [14].

### 3.3. Inhibitor efficiency of the gravimetric tests

Figure 3 shows the inhibition efficiency of watermelon seed oil inhibitor at corrosion reaction temperature of 305 and 319 K. The results showed that an increase in the inhibitor concentration brought about increase in the efficiency of the inhibitor. Also, it could be seen that the higher efficiency was noticed at corrosion temperature of 305 K compare to the values obtained at temperature of 319 K, under same inhibition concentration. The inhibition efficiency results obtained commensurate and confirmed the authenticity of the results obtained from the weight loss.



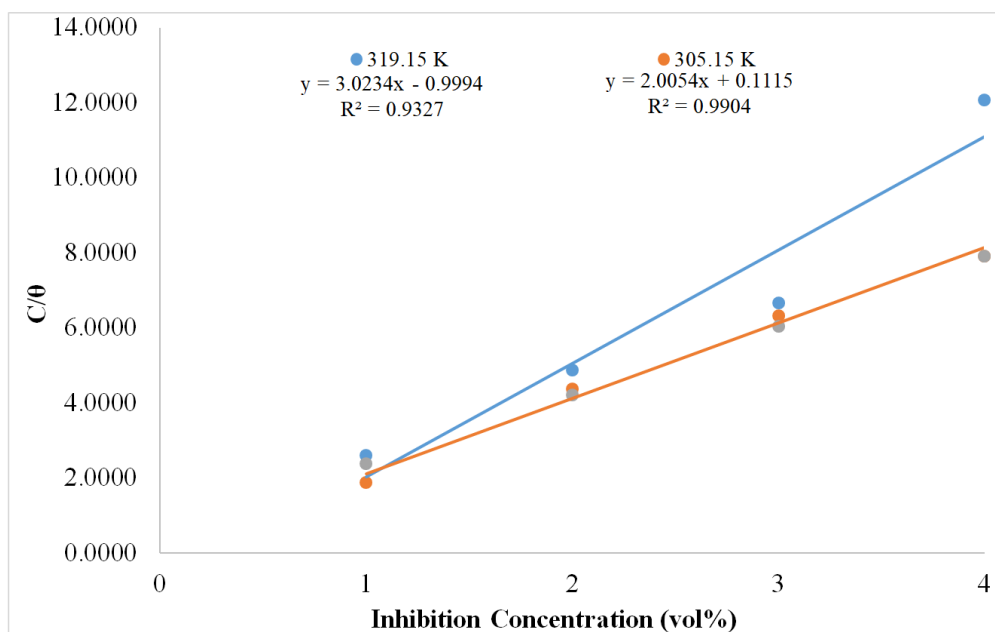
**Figure 3.** Inhibition efficiency of watermelon seed oil inhibitor at corrosion reaction temperature of 305 and 319 K.

### 3.4. Adsorption results of the inhibitive performance of watermelon seed oil

The adsorption characteristics is used to deduce the nature and inhibitive effect of watermelon seed oil. The data obtained from the experiments were considered in the establishment of the adsorption isotherm that describe the adsorption behaviour of the watermelon seed oil inhibitor. Figure 4 shows Langmuir adsorption plot for the varied concentrations of the inhibitor adsorbed on A36 mild steel. Langmuir adsorption isotherm was found suitable for the adsorption behaviour prediction. This is because, a gradient of approximately one ( $R^2$  of 0.9327 at 305 K and  $R^2$  of 0.9904 at 319 K) was obtained for each of the two temperatures considered. That is, there was a formation of inhibitive layer (passive film) on the mild steel surface and this layer prevent active interaction of the metal and its corrosive acidic environment [16].

This was in agreement with the findings obtained the weight loss experiments. That is, increase in the watermelon inhibitor concentration increased the adsorption performance of the adsorbed inhibitor by covering more reaction sites on metal surfaces [15]. This adsorption behaviour of the inhibitor could be traced to the presence of the antioxidants (tannins, saponins, flavonoids and glycosides) that formed a thin layer which retard the corrosion reaction [16].





**Figure 4.** Langmuir adsorption isotherm plot for watermelon seed oil inhibitor at temperatures of 305 and 319 K.

Table 3 shows the adsorption equilibrium constant ( $K_{ads}$ ) and change in Gibbs free energy ( $\Delta G_{ads}$ ) obtained from the inhibitor adsorption process. The  $K_{ads}$  values 1.0006 and 8.9686 obtained (values above 1) indicated that the adsorption process is spontaneous. And the negative sign of  $\Delta G_{ads}$ , indicated the amount of work energy loss to the environment during adsorption process, while the large value obtained connotes that the process was both spontaneous and physisorption (physical adsorption) in nature [9]. As noticed in weight loss and inhibition efficiency plots, values of the thermodynamic parameters ( $K_{ads}$  and  $\Delta G_{ads}$ ) obtained also support better performance of the inhibitor at operating temperature of 305 K.

**Table 3.** Adsorption equilibrium constant and change in Gibbs free energy of adsorption.

T (K)	$R^2$	$K_{ads}$	$\Delta G_{ads}$ (kJ/mol)
319	0.9327	1.0006	-10658.7
305	0.9904	8.9686	-15755.2

### 3.5. Results of the electrochemical tests

Tables 4 and 5 revealed the results of the electrochemical analysis of the mild steel metal samples in 1 M  $H_2SO_4$  (with and without watermelon seed oil inhibitor). From Tables 4 and 5, it could be seen that better results were obtained when inhibitor was used compared to the results obtained when watermelon inhibitor was not considered. Also, it could be seen that as the inhibitor concentration applied increased, the current density ( $i_{corr}$ ) decreased, corrosion potential decreased ( $E_{corr}$ ), polarization resistance ( $R_p$ ) increased, corrosion rate decreased and the inhibitor efficiency of the watermelon seed oil increased. All of these pointed to the fact that increase in

inhibitor concentration introduced promoted the retardation of the corrosion process through the formation of anticorrosion inhibitor layer adsorbed on the metal surface.

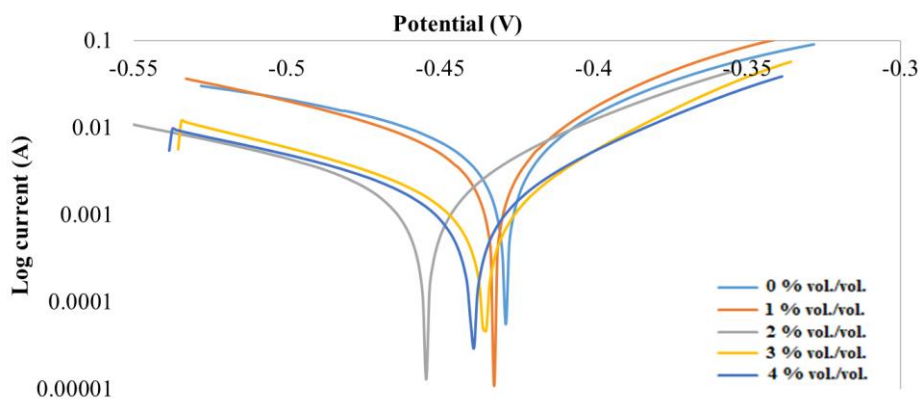
**Table 4.** Electrochemical parameters from potentiodynamic polarization of mild steel at 305 K.

Inhibitor concentration (vol/vol%)	Current density, $i_{\text{corr}}$ ( $\mu\text{A}/\text{cm}^2$ )	Corrosion potential, $E_{\text{corr}}$ (mV)	Polarization resistance $R_p$ ( $\Omega$ )	Anodic Tafel constant, $b_a$ (mV/decade)	Cathodic Tafel constant, $b_c$ (mV/decade)	Corrosion rate (mm/year)	Inhibition efficiency (%)
0	192.20	-428.77	3.1690	1.1841	3.2794	1.3853	0.000
1	108.97	-432.40	2.3344	3.4590	0.7052	1.2662	8.597
2	35.97	-454.68	9.5214	1.0400	3.2606	0.4179	69.832
3	24.53	-439.36	18.269	1.7534	2.5061	0.2849	79.428
4	27.46	-435.54	17.042	2.0669	2.2511	0.3190	76.966

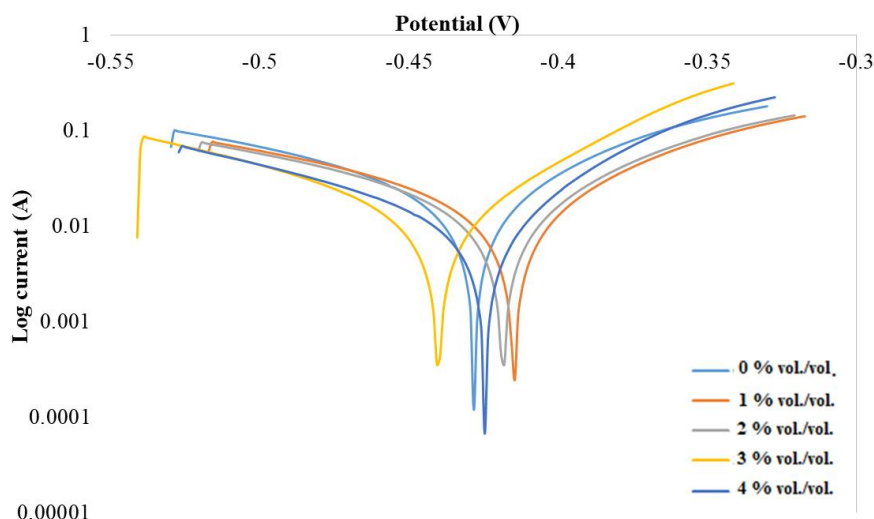
**Table 5.** Electrochemical parameters from potentiodynamic polarization of mild steel at 319 K.

Inhibitor concentration (vol/vol%)	Current density, $i_{\text{corr}}$ ( $\mu\text{A}/\text{cm}^2$ )	Corrosion potential, $E_{\text{corr}}$ (mV)	Polarization resistance $R_p$ ( $\Omega$ )	Anodic Tafel constant, $b_a$ (mV/decade)	Cathodic Tafel constant, $b_c$ (mV/decade)	Corrosion rate (mm/year)	Inhibition efficiency (%)
0	320.75	-428.20	1.0788	3.2002	1.0609	3.7271	0.000
1	238.18	-414.90	1.8731	1.6347	2.7644	2.7676	25.744
2	232.62	-415.60	2.0638	2.0338	2.4217	2.7030	27.477
3	226.50	-440.20	2.1032	2.3066	2.0272	2.6920	27.772
4	215.63	-424.55	1.5708	3.2831	1.0229	2.5056	32.773

It can also be inferred from the Tables 4 and 5 that a more reliable results (in terms of combating corrosion) were obtained at the corrosion temperature of 305 K, compared to the results obtained at the corrosion temperature of 319 K. At the operating temperature of 305 K, 3 vol/vol% inhibitor concentration gave the most promising results while 4 vol/vol% inhibitor concentration gave the most reliable one at the operating temperature of 319 K. These results were further justified by the results obtained from the mixed-typed tafel plots (Figures 5 and 6).



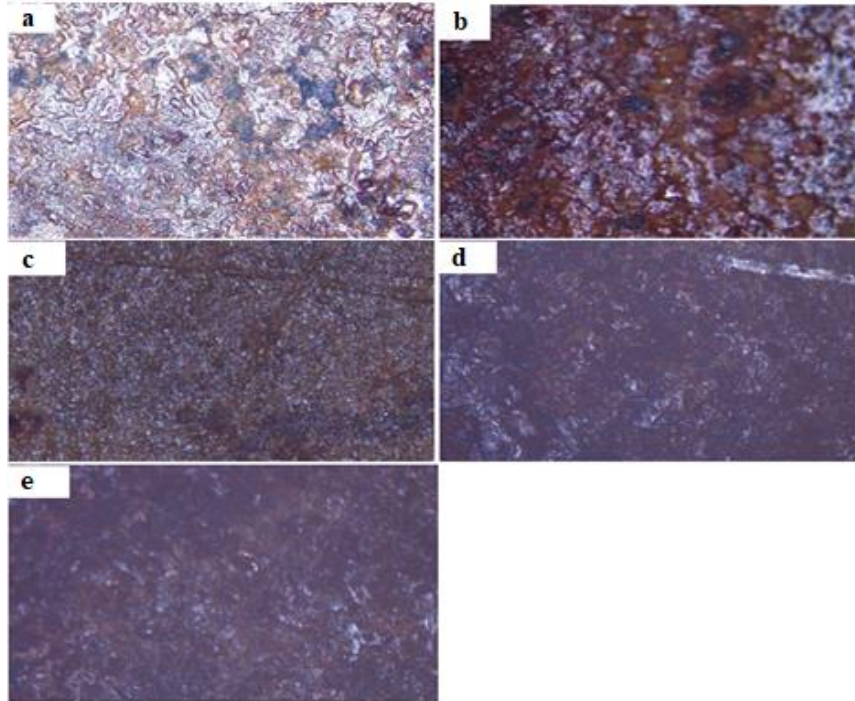
**Figure 5.** Tafel plot of current against potential at 305 K.



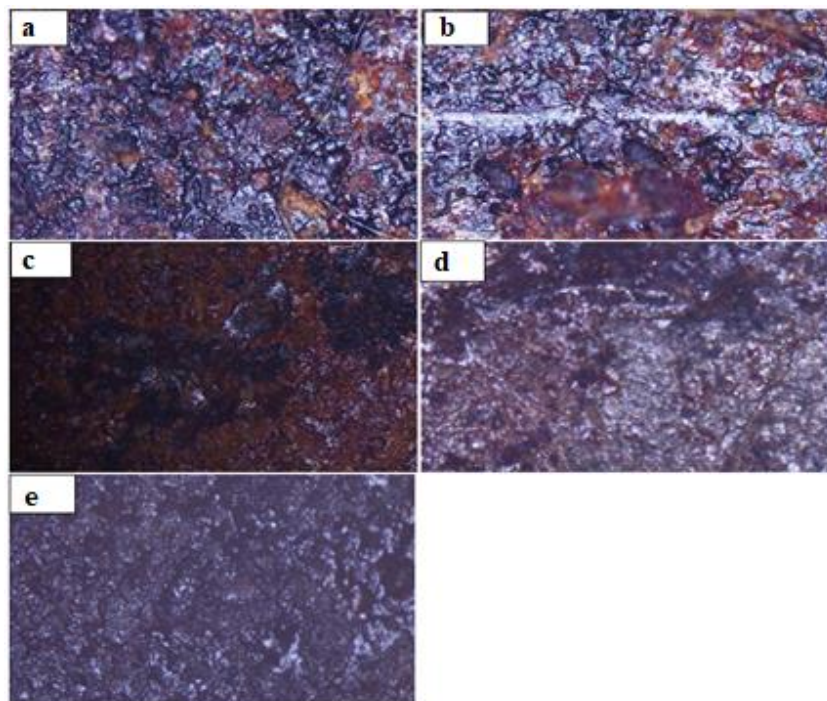
**Figure 6.** Tafel plot of current against potential at 319 K.

### 3.6. Analysis of the magnified metal surfaces

Figures 7 and 8 revealed the optical image analysis results of A36 mild steel samples (with and without inhibitor) at different inhibitor concentrations (0–4 vol/vol%) and different reaction temperature 305 and 319 K) from the electrochemical tests. The metal surface magnification ( $\times 10$ ) confirmed the fact that the metal samples in the acidic medium with no inhibitor suffered corrosion attack the most, at the two operating temperatures. And this implied that watermelon seed oil performed a good role of resisting (or inhibiting) corrosion process. Also, it could be seen that the degree of the corrodibility of the A36 mild steel samples (in terms of surface roughness, burnt and pits formation) decreased as the concentrations of the watermelon seed oil inhibitor introduced increased, considering the two different operating temperatures. The level of damage due to corrosion was mild at the inhibitor concentration of 3 and 4 vol/vol%, and suggest that corrosion attack was best addressed at these two concentrations. It is important to mention that the sample structural analysis also justified the fact that corrosion resistance was better achieved at the operating temperature of 305 K, compared to the analysis obtained at the operating temperature of 319 K.



**Figure 7.** Optical image results of A36 mild steel samples ( $\times 10$ ) at 305 K with inhibitor concentration of (a) 0 vol/vol%, (b) 1 vol/vol%, (c) 2 vol/vol%, (d) 3 vol/vol% and (e) 4 vol/vol%.



**Figure 8.** Optical image results of A36 mild steel samples ( $\times 10$ ) at 319 K with inhibitor concentration of (a) 0 vol/vol%, (b) 1 vol/vol%, (c) 2 vol/vol%, (d) 3 vol/vol% and (e) 4 vol/vol%.

#### 4. Conclusions

The following conclusion can be made from this research work:

- a. Watermelon seed oil proved to be an effective corrosion inhibitor of A36 mild steel in an acidic medium of 1 M H<sub>2</sub>SO<sub>4</sub>, due to the presence of some antioxidants such as tannin, saponin, flavonoid and glycoside.
- b. Gravimetric tests on A36 mild steel showed that watermelon seed oil inhibitor attained a better corrosion inhibition efficiency of 50% at the operating temperature of 305 K compared to an efficiency of 48% obtained at the operating temperature of 319 K.
- c. Electrochemical potentiodynamic polarization tests showed that 3 vol/vol% inhibitor concentration gave the most promising corrosion inhibiting results at the operating temperature of 305 K, while 4 vol/vol% inhibitor concentration gave the most reliable corrosion resisting one at the operating temperature of 319 K.
- d. Langmuir adsorption isotherm correctly predicted the adsorption behaviour of the watermelon seed oil on A36 mild steel surface in 1 M H<sub>2</sub>SO<sub>4</sub> medium. And the negative values of  $\Delta G_{\text{ads}}$  revealed the spontaneous adsorption nature of the inhibitor on the mild steel surface.
- e. The electrochemical potentiodynamic polarization results showed that the watermelon seed oil acted as a mixed-type inhibitor.
- f. The optical image analysis did not only reveal the potency level of watermelon seed oil as an inhibitor, it also showed optimum inhibitor concentrations of 3 vol/vol% (at 305 K) and 4 vol/vol% (at 319 K).

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#### Conflict of interest

All authors declare no conflicts of interest in this paper.

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