Olive leaf as green corrosion inhibitor for C-steel in Sulfamic acid solution

H. M. Elabbasy and A. S. Fouda

ABSTRACT

The effect of Olive leaf as a corrosion inhibitor for C-steel in 10% Sulfamic acid (NH₂SO₃H) solution was tested utilizing different techniques. The effect of temperature (298–328 K) was determined and activation and adsorption thermodynamic parameters were counted and discussed. The surface morphology was analyzed utilizing a scanning electron microscope (SEM). Potentiodynamic polarization procedures evidenced that Olive leaf acts as mixed-type inhibitor for corrosion of C-steel in 10% NH₂SO₃H solution. The addition of Olive leaf produces an increase in the charge transfer resistance (R<Ct>) and reduction in capacitance of the double layer (C<dl>). The inhibition mechanism involves physisorption. The adsorption of this extract on C-steel surface follows Langmuir isotherm. All the studied techniques gave similar results.

1. Introduction

C-steel is a metal applied in several industrial objectives, such as industrialization of equipment for oil, fertilizers, and others. It is important to prevent the corrosion of C-steel by utilizing inhibitors. Organic compounds can be used for this purpose as it contains functional groups of O, N, P, S atoms, but it is considered danger and not safe environmentally (1). Plant extracts are “Green corrosion inhibitors” which are biodegradable, do not contain heavy metals or other toxic elements, renewable nature extracted source and have the perfect action of C-steel inhibition in corrosive media. This is reported by many research groups (2). Several authors tried to use plant extracts as green corrosion inhibitors (3–9).

The present research will examine the inhibitory performance of Olive leaf extract for C-steel in 10% NH₂SO₃H as a corrosive medium, that is because Olive leaf is considered as a sustainable source, biodegradable, and less expensive than other green inhibitors. Sulfamic acid is chosen as a corrosive medium for C-steel because it is widely used in the industry.

1.1. Plant extract preparation

Olive leaf was dried at room temperature away from the sun and greatly milled utilizing an electrical mill. 10 gm of the powder was dissolved by b-idistilled water and put on a hot plate, then after cooling, the solution was
filtrated. In order to determine the concentration of the dissolved part, 10 ml filtrate with 5 ml ethyl alcohol was put in a flask of evaporating purpose. After evaporating the solvent, the remainder substance weights are the same as the equivalent weight dissolved 10 ml filtrate. By this way, the desired concentration of the extract solution was obtained (10).

2. Experimental

2.1. Solutions

The aggressive solution utilized is Sulfamic acid (10% NH₂SO₃H) which was obtained from analytical reagent degree Sulfamic acid 99.8% by dilution with b-idistilled water. The needed concentrations were obtained from the stock solution by dilution.

2.2. Material samples composition

See Table 1.

2.3. Weight difference test

This test was carried out utilizing six identical C-steel pieces with dimensions of (2 × 2 × 0.2) cm, which were perfectly scraped with various degrees of emery papers. Acetone and bi-distilled water were utilized for cleaning and washing of the pieces, thereafter their weights were determined utilizing a sensitive balance. The pieces were suspended in solutions of 100 ml of 10% NH₂SO₃H in the absence and existence of various extract concentrations. Through a period of 30 min, the pieces were taken out, washed, dried, and weighted again through 3 h, at different temperatures (298–328 K) (11,12). Surface coverage (θ) and inhibition competence (IE %) can be accounted utilizing the following equation:

\[
\text{IE}\% = \theta \times 100 = \left[1 - \left(\frac{W}{W^°}\right)\right] \times 100, \tag{1}
\]

where \(W\) is the weight difference in the existence of extract and \(W^°\) is the weight difference in the absence of extract. Corrosion rates (k) were calculated utilizing the following equation:

\[
k = \frac{W}{A \times t}, \tag{2}
\]

where \(t\) is the time, min and \(A\) is the species area, cm² (13).

2.4. Electrochemical techniques

Electrochemical estimations were done utilizing the cell of (a) working electrode consists of C-steel solder with Cu-wire to conduct electricity and mounted into a glass tube of suitable diameter to make the contact area of C-steel electrode 1 cm². The working electrode is scraped as explained before. (b) Saturated calomel electrode, SCE (reference electrode). All potential values were registered vs. SCE. (c) Platinum foil (1 cm²) is the auxiliary electrode. The measurements were done utilizing Gamry instrument Potentiotstat /Galvanostat, ZRA(PCI4-G750) which consists of DC105, EIS300, and EFM140, connecting with a computer to collect and analyze the data utilizing Echem analyst V.6.03.

Initially, C-steel electrode potential was measured against time for 1 h to obtain open circuit potential (OCP) and the steady state.

Potentiodynamic polarization tests were conducted by applying a potential range (−1200 mV to +1200 mV) to obtain corrosion current densities (\(i_{\text{corr}}\)). Values of corrosion current densities (\(i_{\text{corr}}\)) and corrosion potential (\(E_{\text{corr}}\)) were attained by extrapolating of two Tafel area. IE % and θ from potentiodynamic polarization technique were accounted utilizing the following equation:

\[
\text{IE}\% = \theta \times 100 = \left[1 - \left(\frac{i_{\text{corr}}}{i_{\text{corr}}^°}\right)\right] \times 100, \tag{3}
\]

Figure 1. \(\Delta W\) of C-steel against time in 10%NH₂SO₃H solutions in the absence and existence of various extract concentrations.
where $i_{\text{corr}}$ is the corrosion current density in the existence of extract and $i_{\text{corr}}^0$ is the corrosion current densities in the absence of extract.

Tests of electrochemical impedance spectroscopy (EIS) were done utilizing Ac signals through the frequency extent (0.1 Hz–100 kHz) and 5 mV peak to peak capacity. Analyzing and explaining of outcomes from EIS depend on the utilized equivalent circuit. ($\theta$) and (IE %) were attained by the following equation.

$$\text{IE} \, \% = \theta \times 100 \times \left[1 - \left(\frac{R_{\text{ct}}}{R_{\text{ct}}^0}\right)\right] \times 100$$

where $R_{\text{ct}}$ is the charge transfer resistance in the existence of extract and $R_{\text{ct}}^0$ is the charge transfer resistance in the absence of extract (14).

Tests of electrochemical frequency modulation (EFM) were achieved utilizing 2 and 5 Hz frequencies. The waveform was found to repeat every 1 s because the main frequency was 0.1 Hz. The spectra include current responses connected with harmonically and intermodulation current peak. Values of Tafel slopes ($\beta_c$ and $\beta_a$, $i_{\text{corr}}$), and the causality factors (CF-2 and CF-3) were acquired utilizing the large peaks (15, 16).

### 2.5. Surface check

The metal surface was analyzed, after 24 h in 10% NH$_2$SO$_3$H with and without the extract, utilizing a scanning electron microscope (SEM, JSM-T20, Japan).

### 3. Outcomes and debate

#### 3.1. Weight difference ($\Delta W$) test

Figure 1 displays $\Delta W$ against time for C-steel in 10% NH$_2$SO$_3$H solutions in the absence and existence of various extract concentrations at 298 K. It is clear that the curves with the extract are significantly below than that without the extract. This indicated that the extract molecules are adsorbed on C-steel surface forming a film which reduces the correction rate.

#### 3.2. Temperature influence

The temperature influence on the metal corrosion inhibition in acid media is very complex (17), due to the surface changes, for example, desorption or decomposition of the inhibitor molecules. Impact of temperature on IE% was examined utilizing weight loss test at (298–328 K). Figure 2 shows corrosion rate ($k$) against

| [extract] ppm | 298 K | 308 K | 318 K | 328 K | 298 K | 308 K | 318 K | 328K |
|---------------|-------|-------|-------|-------|-------|-------|-------|------|
| Blank         | 0.0287| 0.0847| 0.1979| 0.3702| –     | –     | –     | –    |
| 50            | 0.0101| 0.0227| 0.0388| 0.0710| 64.70 | 73.14 | 80.41 | 80.85 |
| 100           | 0.0067| 0.0145| 0.0274| 0.0440| 76.77 | 82.92 | 86.17 | 88.13 |
| 200           | 0.0053| 0.0105| 0.0226| 0.0334| 81.65 | 87.64 | 88.60 | 91.00 |
| 300           | 0.0041| 0.0090| 0.0176| 0.0270| 85.78 | 90.54 | 91.10 | 92.71 |
| 400           | 0.0030| 0.0061| 0.0139| 0.0174| 89.60 | 92.75 | 92.98 | 95.30 |
temperature in the absence and existence of various extract concentrations. It is evident that $k$ is inversely proportional to the extract concentration while directly proportional to temperature. Values of $k$ and IE% are shown in Table 2. Results showed that $k$ values were lower in the presence of the extract compared to the blank solution. Also, IE% increases with an increase in the extract concentration until it reaches 89.6. This result might be due to the fact that the amount of adsorption and the metal surface coverage increase with an increase in the inhibitor concentration (18). IE% decreased with an increasing temperature, indicating that at higher temperatures, dissolution of the metal predominated on extract adsorption (19,20).

The relationship of $k$ with $T$ can be shown in the following equation (21):

$$\log k = \log A - \frac{E_a}{2.303 RT}, \tag{5}$$

where $E_a$ is the activation energy, $T$ is the absolute temperature (K), $R$ is the universal gas constant (joule/k.mole), and $A$ is the frequency factor. Plots of log $k$ of C-steel in 10% NH$_2$SO$_3$H against 1/$T$ without and with various extract concentrations are shown in Figure 3.

![Figure 3](image)

**Figure 3.** Plots of log $k/T$ against 1000/$T$ for C-steel in 10% NH$_2$SO$_3$H in the absence and existence of various extract concentrations.

Table 3. Activation parameters for C-steel in 10% NH$_2$SO$_3$H in the absence and existence of various extract concentrations.

| [extract] ppm | $E_a$ (kJ mol$^{-1}$) | $\Delta H^*$ (kJ mol$^{-1}$) | $-\Delta S^*$ (J mol$^{-1}$ K$^{-1}$) |
|---------------|-----------------------|-----------------------------|--------------------------------------|
| Blank         | 23.4                  | 20.7                        | 49.38                                |
| 50            | 32.4                  | 29.7                        | 117.33                               |
| 100           | 34.7                  | 32.0                        | 122.72                               |
| 200           | 35.5                  | 32.8                        | 124.49                               |
| 300           | 36.4                  | 33.7                        | 122.81                               |
| 400           | 39.8                  | 37.1                        | 134.04                               |

It is obvious from $E_a$ values (in Table 3) that as the extract concentration increases, the value of $E_a$ increases, which points that at larger extract concentration, the corrosion reaction will be more pushed to the surface sites with higher $E_a$ values (22). Meaning that a barrier layer is formed through the extract adsorption on the surface of C-steel, this layer impedes the metal corrosion reactions (23), explaining that the increase in $E_a$ is due to the reduction in the adsorption of the extract as the temperature increases. Lower adsorption means more desorption, thus large surface area of C-steel connects with the acid medium, prompting the rise of corrosion rates. But this trouble not present at high extract concentration due to the reduction in surface coverage that occurs when close to saturation (24). Higher $E_a$ values in the existence of the extract show physical adsorption (electrostatic interaction) (25), actually extract molecule with heteroatom forms cation in acid solution, thus the electrostatic adsorption of the cation gives perfect protective characteristics of the extract.

The enthalpy of activation change ($\Delta H^*$) and entropy of activation change ($\Delta S^*$) were calculated by plotting log $k/T$ versus 1/$T$ (Figure 4) according to the following equation (26):

$$k = \left(\frac{RT}{NH}\right) \exp \left(\frac{\Delta S^*}{R}\right) \exp \left(-\frac{\Delta H^*}{RT}\right), \tag{6}$$

where $h$ is Planck’s constant and $N$ is Avogadro’s number. This relationship results in straight lines, where $\Delta H^*$ is obtained from slopes and $\Delta S^*$ from the intercepts. $\Delta H^*$ and $\Delta S^*$ values are displayed in Table 3. $\Delta H^*$ has a positive sign, which points that C-steel dissolution is endothermic and slow (27). $\Delta S^*$ has a negative sign,
which reveals that the activated complex performs an association instead of dissociation, signals that from reactants to the activated complex the decrease in disorder occurs (28).

### 3.3. Adsorption isotherms

Adsorption is considered the first step in inhibition process. The adsorption relies on inhibitor structure, absolute temperature and electrochemical potential at the interface of metal/solution. So the adsorption procedure can be considered as the aquasi-exchange between organic compound in solution [Org(Sol.)] and adsorbed water particle [H$_2$O(ads.)] (29).

$$\text{Org}_{\text{Sol.}} + x\text{H}_2\text{O}_{\text{ads.}} \leftrightarrow \text{Org}_{\text{ads.}} + x\text{H}_2\text{O}_{\text{Sol.}},$$ (7)

where $x$ is the number of replaced water molecule. The interaction between extract and the metal surface can be demonstrated utilizing adsorption isotherms. Among different isotherms such as Langmuir, Temkin, Frumkin and Flory-Huggins, the ideal fit was Langmuir. This isotherm was utilized for other inhibitors (30,31).

Langmuir isotherm condition is as per the following:

$$\frac{C}{\theta} = \frac{1}{K_{\text{ads.}}} + C,$$ (8)

Table 4. Langmuir adsorption isotherm results for C-steel in 10% NH$_2$SO$_3$H with the extract at various temperatures.

| Temp. (K) | Slope | $K_{\text{ads.}}$ (M$^{-1}$) | $-\Delta G^\circ_{\text{ads.}}$ (kJ mol$^{-1}$) | $\Delta H^\circ_{\text{ads.}}$ (kJ mol$^{-1}$) | $\Delta S^\circ_{\text{ads.}}$ (J mol$^{-1}$K$^{-1}$) |
|-----------|-------|-------------------------------|---------------------------------------------|---------------------------------------------|---------------------------------------------|
| 298       | 1.037 | 37.70                         | 19.0                                        | 60.07                                       | 265.2                                       |
| 308       | 1.040 | 48.78                         | 20.2                                        | 60.8                                        | 260.8                                       |
| 318       | 1.048 | 56.92                         | 21.3                                        | 255.9                                       |                                              |
| 328       | 1.063 | 458.72                        | 27.7                                        |                                              | 267.5                                       |

where $K_{\text{ads}}$ is the equilibrium constant. Figure 5 displays plots of $C/\theta$ versus $C$ at various temperatures, from which linear relationships are acquired with slopes almost unity, which indicate that Langmuir isotherm is good obeyed in our study.

The values of Gibbs free energy ($\Delta G^\circ$) were accounted utilizing the following equation:

$$\Delta G^\circ_{\text{ads.}} = -RT\ln(55.5K_{\text{ads}})$$ (9)

where 55.5 is the water molarity in the solution. Straight line is acquired from plotting $\log K_{\text{ads}}$ versus $1/T$ (Figure 6), as the slope equals enthalpy of adsorption ($\Delta H^\circ_{\text{ads.}}$) according to the following equation:

$$\log K_{\text{ads}} = \left(-\frac{\Delta H^\circ_{\text{ads.}}}{2.303RT}\right) + \text{constant}$$ (10)

Adsorption parameters for C-steel in 10% NH$_2$SO$_3$H with the extract are grouped in Table 4. It is spotted that estimations of $K_{\text{ads}}$ are high, which indicates stronger extract adsorption on the metal surface because of the existence of heteroatom and $\pi$-electrons in the various molecules present in the extract. The inhibition process occurs by these various molecules at different contents, i.e. mixture of inhibitors from major to minor components, higher inhibition efficiency (32) is due to the intermolecular synergistic effect of these various molecules in the extract.

$\Delta G^\circ_{\text{ads.}}$ have negative values that signalize spontaneous adsorption process. In general, the estimations of $\Delta G^\circ_{\text{ads.}}$ until $-20$ kJ mol$^{-1}$, demonstrate physisorption process, while those about $-40$ kJ mol$^{-1}$ or more, show chemisorption process as an outcome of sharing or transmit of electrons from molecule on metal surface (33). As evident here, $\Delta G^\circ_{\text{ads.}}$ estimations were in extent of $-19$ and $-27$ kJmol$^{-1}$, suggesting that the adsorption
mechanism collected physisorption and chemisorption, together (34), anyway, the primary contributor to the adsorption mechanism was physisorption whereas chemisorption just a little contributed. The positive sign of $\Delta H_{\text{ads}}^\circ$ indicates endothermic adsorption of extract molecule on C-steel.

Entropy of adsorption ($\Delta S_{\text{ads}}^\circ$) was deduced from the following equation (35):

$$D_{\text{G}}^{\circ\text{ads}} = D_{\text{H}}^{\circ\text{ads}} - T \Delta S_{\text{ads}}^\circ \quad (11)$$

Estimations of $\Delta S_{\text{ads}}^\circ$ (in Table 4) were positive, indicating that the entropy increases through the adsorption process, due to the desorbed solvent molecules from the C-steel surface. So the disorder of the system increases.

### 3.4. Open circuit potential (OCP)

Figure 7 shows impact of various Olive leaf extract concentrations on OCP of C-steel in aerated non-stirred 10% NH$_2$SO$_3$H solution at 298 K. OCP was discovered to be high negative than it at $t = 0$, indicating that the oxide film from air that formed before immersion must dissolve before the steady state is attained (36). This potential ($E_{\text{corr}}$), attained through nearly 10 min, indicates the free bare metal corrosion (37). Obviously ($E_{\text{corr}}$) becomes more negatively without variation of the public feature of $E/t$ curve.

Furthermore, increasing of extract concentration shifts the OCP, initially to low negative values (because of corrosion inhibition) reaching a maximum, then at definite time, depends on the extract concentration, the potential decreases until a steady value (because of metal dissolution). This will move the OCP towards more negative values. These results revealed that Olive leaves inhibit C-steel corrosion in 10% NH$_2$SO$_3$H solutions efficiently.

### 3.5. Potentiodynamic polarization tests

Figure 8 includes plots from potentiodynamic polarization tests of C-steel in 10% NH$_2$SO$_3$H in the absence and existence of various extract concentrations. IE% values from polarization tests were counted utilizing Equation (3). Electrochemical parameters related with polarization tests ($i_{\text{corr}}, E_{\text{corr}}, Tafel slopes (\beta_a, \beta_c)$ and IE %) are shown in Table 5.

It is known that iron dissolution in acid occurs spontaneously according to the following equation:

$$\text{Fe} = \text{Fe}^{2+} + 2e^- \quad (12)$$

Joined by the following cathodic equation:

$$2\text{H}^+ + 2e^- = \text{H}_2 \quad (13)$$

Anodic and cathodic Tafel sections indicate a bend over the entire utilized potential range. The anode bend may be because of a non-passive film produced from the products from corrosion or the deposited impurities on C-steel (for

![Figure 8. Curves from potentiodynamic polarization tests for C-steel in 10% NH$_2$SO$_3$H solutions in the absence and existence of various extract concentrations.](image)

![Figure 9. Impedance curve together with the equivalent circuit utilizing for stimulation the registered EIS data for C-steel in 10% NH$_2$SO$_3$H solution at 298 K.](image)

| [extract] ppm | $i_{\text{corr}}$ $\mu$A cm$^{-2}$ | $E_{\text{corr}}$ mV vs. SCE | $\beta_a$ mV dec$^{-1}$ | $\beta_c$ mV dec$^{-1}$ | IE % |
|---------------|-----------------|-----------------|-----------------|-----------------|-------|
| Blank         | 199.0           | 470             | 76              | 235             | –     |
| 50            | 94.6            | 438             | 40              | 199             | 52.5  |
| 100           | 79.0            | 433             | 38              | 195             | 60.3  |
| 200           | 64.2            | 431             | 33              | 190             | 67.7  |
| 300           | 59.7            | 433             | 32              | 189             | 70.0  |

Table 5. Electrochemical parameters that related with polarization tests ($i_{\text{corr}}, E_{\text{corr}}, \beta_a, \beta_c$ and IE %) for C-steel corrosion in 10% NH$_2$SO$_3$H solutions in the absence and existence of various extract concentrations.
example, Fe₃C). For cathodic section, stationary solution makes the diffusion of H⁺ slow, and concentration polarization works to diminish the area of cathodic linear.

Since acidic solution is aerated 10% NH₂SO₃H solution, the solution will contain two cathodic reactions (evolution of hydrogen and reduction of dissolved oxygen) with the anodic reaction (metal dissolution). The complicated cathodic part may show no linearity or show short Tafel area as shown here (38). So, we depend on the software to obtain the Tafel slopes and i_corr values although we do not trust it much. Like polarization, EIS cannot be used to determine i_corr values. Unlike polarization, EFM is a perfect method to obtain the values of Tafel slopes and i_corr.

It is clear that, as the extract concentration increases, (E_corr) becomes more positive and (i_corr) decreases. The IE % increases with the increasing extract concentrations, until reaching 77.2. This shows that the extract is a perfect corrosion inhibitor. The inhibiting influence of the extract may be concerning on adsorption on the steel surface to form a barrier layer. Based on the results, it is found that the extract limits the disintegration of C-steel, also delays hydrogen evolution reaction. This referred that the extract molecules absorb upon both anodic and cathodic sites of the metal surface, thus the extract could be classified as a mixed type inhibitor (39).

Values of Tafel slopes are slightly changed at the addition of extract, this result reveals that Olive leaf is efficacy mixed type green corrosion inhibitor. The process of inhibition does not change the mechanism of corrosion, only deactivates a fraction of the surface in the destructive solution (40). IE% values were found to increase by increasing the extract concentration.

### 3.6. EIS tests

Figure 9 represents (CPE) an equivalent circuit was suggested for corrosion of C-steel electrode in 10% NH₂SO₃H solutions. Where R_s is the resistance of solution, R_ct is the resistance of charge transfer, and CPE is the constant phase element, utilized to give further exact fit and determine (C_dl) the capacitance double layer (41). The semicircle appears due to the stationary time of charge-transfer and double layer capacitance (42). This semicircle produces an angle about 68.5° with an actual axis and affords 4.8 Ω cm² through its intersection for the resistance of the solution (Rs) found between reference and working electrodes.

It is obvious from Nyquist plots (Figure 10) with and without Olive leaf extract, the shapes of the plots do not change indicating that, as obtained from polarization tests, the existence of extract does not change the mechanism of the corrosion reaction. The same results were obtained in past studies (43). (Rs + R_ct) values were obtained from the intersection point between semicircle and actual axis. The greatest frequency (f_max) of the maximal imaginary ingredient of the impedance was applied in the following equation to obtain C_dl.

\[
C_{dl} = \frac{1}{2\pi f_{max}R_{ct}}
\]

IE% values from EIS tests were attained by R_ct values utilizing Equation (4). Through the C-steel corrosion in 10% NH₂SO₃H solutions, the surface roughness increases. The obtained EIS parameters for C-steel corrosion in 10% NH₂SO₃H solutions in the absence and existence of various extract concentrations at 298 K are given in Table 6.

| [extract] (ppm) | R_s (Ω cm²) | R_ct (Ω cm²) | C_dl (μF cm⁻²) | IE% |
|----------------|-------------|--------------|----------------|-----|
| Blank          | 4.632       | 73.18        | 182.0          | —   |
| 50             | 3.87        | 168.3        | 84.06          | 56.5|
| 100            | 4.019       | 213.2        | 77.86          | 65.7|
| 200            | 4.162       | 226.4        | 73.02          | 67.7|
| 300            | 4.42        | 233.6        | 70.06          | 68.7|
| 400            | 4.8         | 256.2        | 58.84          | 71.5|

![Figure 10](image-url)
with Helmholtz model, given by the following equation:

\[ C_{dl} = \varepsilon \varepsilon_0 A / d \]  

(15)

where \( d \) is the double layer thickness, \( \varepsilon \) is the dielectric constant, \( \varepsilon_0 \) is the vacuum permittivity and \( A \) is the effective surface area.

Results in Table 6 illustrate that the inhibition efficiency increases as the extract concentration increases. This outcome assures the results obtained from the previous measurements and confirms that Olive leaf extract is efficacy green corrosion inhibitor.

### Table 7

| [extract] (ppm) | \( i_{corr} \) (µA) | \( \beta_a \) (mV dec\(^{-1}\)) | \( \beta_c \) (mV dec\(^{-1}\)) | CF-2 | CF-3 | IE% |
|-----------------|---------------------|---------------------|---------------------|------|------|-----|
| Blank           | 271.8               | 57.07               | 235.3               | 1.9  | 2.9  | –   |
| 50              | 121.5               | 49.6                | 156.5               | 1.9  | 2.9  | 55.3 |
| 100             | 95.34               | 48.01               | 149.1               | 1.9  | 2.6  | 64.9 |
| 200             | 88.78               | 45.93               | 146.1               | 1.9  | 2.7  | 67.3 |
| 300             | 74.49               | 46.62               | 140.5               | 1.9  | 2.8  | 72.6 |
| 400             | 69.47               | 46.5                | 138.7               | 1.8  | 2.8  | 74.4 |

**Figure 11.** Current response with the frequency for C-steel in 10% NH\(_2\)SO\(_3\)H in the absence and existence of various extract concentrations.

EFM is a small signal ac method that utilizes two sine waves (at various frequencies) with one another to cell. Without period information of Tafel constant, EFM immediately gives estimations of the corrosion current.

In EFM tests, the current response to potential is non-linear. The current response includes two enter frequencies, and its ingredients (total, difference, and multiples). The two enter frequencies must be small, integer multiples of an main frequency which is responsible for the

3.7. EFM tests

EFM is a small signal ac method that utilizes two sine waves (at various frequencies) with one another to cell. Without period information of Tafel constant, EFM immediately gives estimations of the corrosion current.

In EFM tests, the current response to potential is non-linear. The current response includes two enter frequencies, and its ingredients (total, difference, and multiples). The two enter frequencies must be small, integer multiples of an main frequency which is responsible for the
experiment length. Figure 11 shows the current response as a function of frequency, as acquired from EFM tests. IE % was calculated from Equation (3). The parameters gained from the EFM method (IE %, icorr, βa, βc and causality factors (CF-2 and CF-3)) at various concentrations of the extract, are seen in Table 7.

The large quality of EFM comes from causality factor that utilizes as an inward test on the rightness of the EFM tests (45). As illustrated in Table 7, causality factors are compatible with theoretical values, that is 2.0 and 3.0 for CF-2 and CF-3, respectively, which confirms the rightness of Tafel slopes and icorr. It is apparent that the outcomes acquired from all utilized tests were in large agreement with each other.

3.8. SEM analysis

Figure 12(a–c) shows SEM micrographs for surface of C-steel prior and after its putting in aerated 10% NH₂SO₃H solution without and with the addition of 400 ppm of Olive leaf. As expected, Figure 12(a) indicates metallic surface is evident, whereas in Figure 12(b), without extract, the metallic surface is deteriorated by aerated 10% NH₂SO₃H solution, while in Figure 12(c), with extract, the metallic surface is not affected by acid corrosion.

Figure 13 shows a comparison of the inhibition efficiencies derived from different investigated corrosion estimations methods. As shown in the figure, the different methods gave approximately similar results. This confirms the valid obtained results.

3.9. Inhibition mechanism

From experiment results, we notice that there is a logical inhibition mechanism for corrosion operation of C-steel in 10%NH₂SO₃H solutions by Olive leaf as a green corrosion inhibitor, may be delineated on the adsorption foundation.
4. Conclusions

Weight loss, OCP, Tafel, EIS, EFM, and SEM, were utilized to check the inhibitory action of Olive leaf extract on corrosion of C-steel in 10% NH2SO3H solutions. The principal conclusions were

- IE% values increase as the Olive leaf extract concentration increases.
- OCP becomes more positive as the Olive leaf extract concentration increases.
- Increasing in Olive leaf concentration leads to decrease in $i_{corr}$.
- $R_{ct}$ values increase with increasing of Olive leaf concentration, while $C_{dl}$ decreases.
- Physisorption is suggested as a corrosion mechanism.
- IE% acquired from chemical and all electrochemical tests were concurrent with each other.

Disclosure statement

No potential conflict of interest was reported by the authors.

Notes on contributors

H.M. Elabbasy currently works as lecturer of chemistry in essential sciences department at Misr higher Institute of Engineering and Technology, Mansoura, Egypt. Main research interests are in corrosion inhibition of metals and alloys using some synthesis organic compounds and green corrosion inhibitors.

A. S. Fouda currently works at Mansoura University, Mansoura, Egypt. He is professor of physical chemistry at chemistry department (College of Science). Their current project is ‘synthesis and characterization of some organic compounds as corrosion inhibitors’.

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ORCID

A. S. Fouda @ http://orcid.org/0000-0002-3239-4417
