

Article

Study of Combination of *Physalis angulata* Leaf and *Cassia angustifolia* Leaf Extract with a 50:50 Ratio as Green Inhibitor for API 5L in 1M HCl Solution

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Abstract: Researches for green inhibitors in corrosion protection has garnered significant attention due to environmental and safety concerns associated with synthetic inhibitors. Previous studies have explored various plant extracts, but there remains a need for more efficient and sustainable options. This research investigated the effect of combination of *Physalis angulata* leaf and *Cassia angustifolia* leaf extract as green inhibitor with a 50:50 ratio for API 5L steel. This experiment was carried in 1M HCl solution by using different concentrations as variables which is 10 ml, 30 ml, and 50 ml inhibitor extracts. Evaluation of this experiment was performed using potentiodynamic polarization and EIS. The antioxidant compound was investigated using FTIR and was found to be flavonoid. It was found that the capability of the inhibitor to inhibit the steel surface from the environment increases with increasing concentration. The combined extracts exhibited remarkable corrosion inhibition properties, achieving a maximum inhibition efficiency of 98.63% at a 50 ml concentration. This efficiency highlights the potential of these natural extracts as eco-friendly and effective alternatives to conventional inhibitors. Combination of *Physalis angulata* leaf and *Cassia angustifolia* leaf extract is identified as mixed-type inhibitor with predominant cathodic effectiveness. The adsorption of this inhibitor on the steel surface was predominantly through physisorption and obeyed Langmuir adsorption isotherm.

Keywords: Green inhibitor; EIS; Potentiodynamic polarization; FTIR; API 5L

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1. Introduction

API 5L carbon steel pipe is commonly used in oil and gas industry. The pipe is used to transport fluids between equipment in a plant or from one place to another for a successful production in the industry. API 5L is a low carbon steel pipe that more susceptible to corrosion in aggressive environments compared to other types of steel. Given its exposure to various aggressive environments, it necessitates protection against corrosion.

One of the obstacles that is found in the aggressive environment is corrosion. Corrosion is a phenomenon where the material reacts with the environment causing the material to degrade [1]. This phenomenon will caused a failure to the material, which can make the material unable to function properly. Moreover, this phenomenon can make the production process disrupted and increase the production cost. With the numerous issues related to metal corrosion, research on corrosion inhibitors must continuously be developed to find the most effective inhibitors with the highest efficiency. Additionally,

corrosion inhibitors should be affordable and environmentally friendly so they can be applied across various industrial sectors.

Inhibitor is one of the effective way to prevent the corrosion to happen. Inhibitor is known to have an effective result, which can decrease the corrosion rate up to 99% [2]. Researches into inhibitors are thus geared towards natural products, such as fruits and plants, to obtain a non-toxic and environmentally friendly inhibitors with good inhibition efficiencies [3]. Several studies about green inhibitors have been reported [3–12]. The leaf extracts from non-inoculated and inoculated *Aquilaria subintegra* were evaluated, demonstrating a high corrosion inhibition efficiency of up to 93% at 1500 ppm. This was determined through weight loss studies, electrochemical impedance spectroscopy, and potentiodynamic polarization measurements [3]. *Morinda citrifolia* (Noni) extract as a green corrosion inhibitor is effective for protecting low carbon steel in a 3.5% sodium chloride solution, achieving an optimum inhibitor efficiency of 76.92% at a concentration of 3 ppm after 288 hours of immersion, with the formation of a monolayer film indicating its mixed-type inhibitor behavior [4].

Physalis angulata leaf known to have an antioxidant activity. This leaf contains several chemical compounds including cryptoxanthin, physalin, saponin, terpenoid, flavonoid, polyphenol, and steroid [13]. *Cassia angustifolia* leaf also known to have an antioxidant activity. This leaf contains chemical compounds including anthraquinone, xanthone, and flavonoid [14, 15]. The existence of flavonoid in both leaves is believed to give an effective inhibitive effect to the material.

The experiment was done to study the corrosion inhibition of the combination of *Physalis angulata* leaf and *Cassia angustifolia* leaf with a 50:50 ratio as green inhibitor for API 5L steel. This experiment was evaluated using potentiodynamic polarization and EIS, also performed in 1M HCl solution at 25°C. Different concentrations, which are 10, 30, and 50 ml, were used as the variable for this experiment. The compounds in the leaves were identified using FTIR.

2. Materials and Experiment Methods

2.1 Extraction

The leaves were already in a powder form. A 0.375g from each leaf powder were dissolved in a 150ml 1M HCl solution. The 150ml extract was then divided into 10ml, 30ml, and 50ml inhibitor concentrations.

2.2 Sample Preparation

API 5L steel was used in this experiment. This sample has a nominal composition (wt%) of 98.7% Fe, 0.203% C, 0.524% Mn, 0.0242% P, and 0.0111% S.

The sample was cut with a band saw to 25 mm x 20 mm x 5 mm, which is cut into two samples. The samples were then drilled with a 3 mm drill bit to hook it with a string to hang the sample in the solution.

The sample was cut to 10 mm x 10 mm x 5 mm, which is cut into four samples. The four samples were then grinded and connected to a copper wire with a solder. The four samples were then mounted with resin and hardener.

2.3 FTIR

1M HCl solution was used in this experiment. The solution was prepared by diluting 37% concentrated HCl in distilled water.

2.4 Solution Preparation

Pelkin-Elmer instrument was used in this experiment with a 4000-450 cm⁻¹ wavelength. The FTIR was performed twice, to the leaf powder and to a sample soaked in a 100ml 1M HCl solution with a 50ml inhibitor concentration. This experiment was done to find out the active compound in the leaves and to find out the absorbed active compound in the steel surface. The active compound found in the steel surface known to be the active compound that gives an inhibition effect to the steel.

2.5 Extraction

Nova Autolab PGSTAT302N instrument was used in this experiment with a 0.01 V/s scanning rate and 120s open circuit potential (OCP). The result of this experiment was a polarization curve which was obtained with a Tafel extrapolation. The current density (I_{corr}) and corrosion potential (E_{corr}) were obtained from the curve. The IE % was calculated using the equation (1):

$$IE \% = \frac{I_{uninhibited} - I_{inhibited}}{I_{uninhibited}} \times 100 \quad (1)$$

2.6 Sample Preparation

Gamry Instrument was used in this experiment. A semicircle curve was obtained in this experiment. The curve was then fitted to obtain an electrical circuit where charge-transfer resistance (R_{ct}), electrolyte resistance (R_s), and double-layer capacitance (C_{dl}) were obtained from this electrical circuit. The IE % was calculated using the equation (2):

$$IE \% = \frac{R_{ct(inhibited)} - R_{ct(uninhibited)}}{R_{ct(inhibited)}} \times 100 \quad (2)$$

3. Results and Discussion

3.1. Fourier Transform Infra Red (FTIR) Spectroscopy

3.1.1. Leaf Extract

The antioxidant compound in *Physalis angulata* leaf, specifically flavonoid, were confirmed by FT-IR spectroscopy as shown in Fig. 1. Flavonoid has an O-H hydroxyl compound group as the basic structure. The peak at 3330.78cm⁻¹ is due to the existence of O-H hydroxyl group. The same result was obtained in *Cassia angustifolia* leaf as shown in Fig. 2, where flavonoid was found. The peak at 3279.66cm⁻¹ is due to the existence of O-H hydroxyl group. In both leaves, C-H, C=C, and C=O compound were also found [16].

This combination is predicted to have a synergistic effect to each other and is predicted to give a good inhibition effect to the steel.

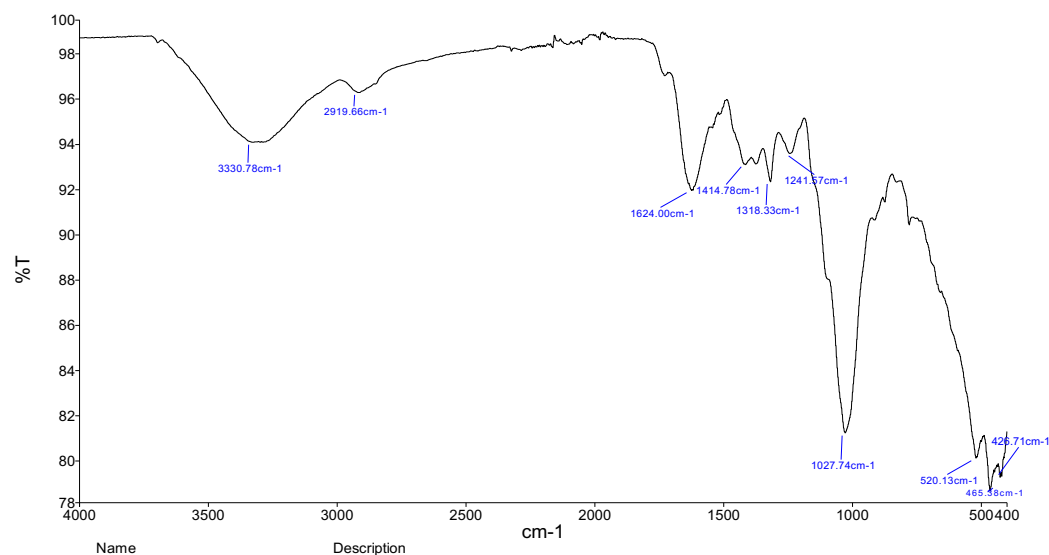


Figure 1. FTIR spectrum of *Physalis angulata* leaf

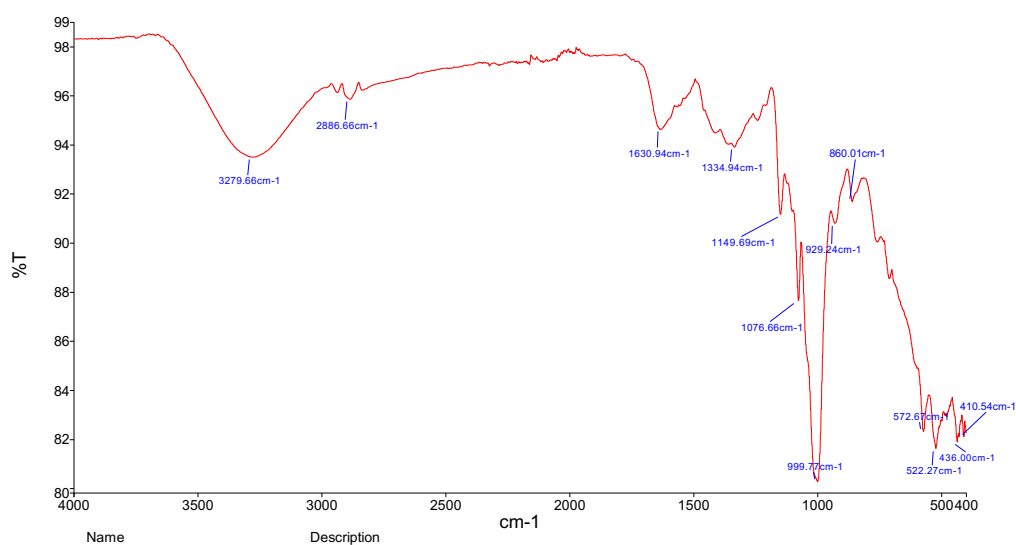


Figure 2. FTIR spectrum of *Cassia angustifolia* leaf

3.1.2. Absorbed Extract in Steel Surface

The absorbed antioxidant compound in steel surface were also investigated using FT-IR spectroscopy. The result is shown in Fig. 3. The peak at 3350.78cm⁻¹ is due to the existence of O-H hydroxyl compound group. C=C and C=O compound were also found in the abdsorbed extract [16]. This indicates that the extract was successfully absorbed in the steel surface. Flavonoid is confirmed to be the active compound in the extract with the existene of O-H hydroxyl compound group in the extract and in the absorbed extract in the steel surface.

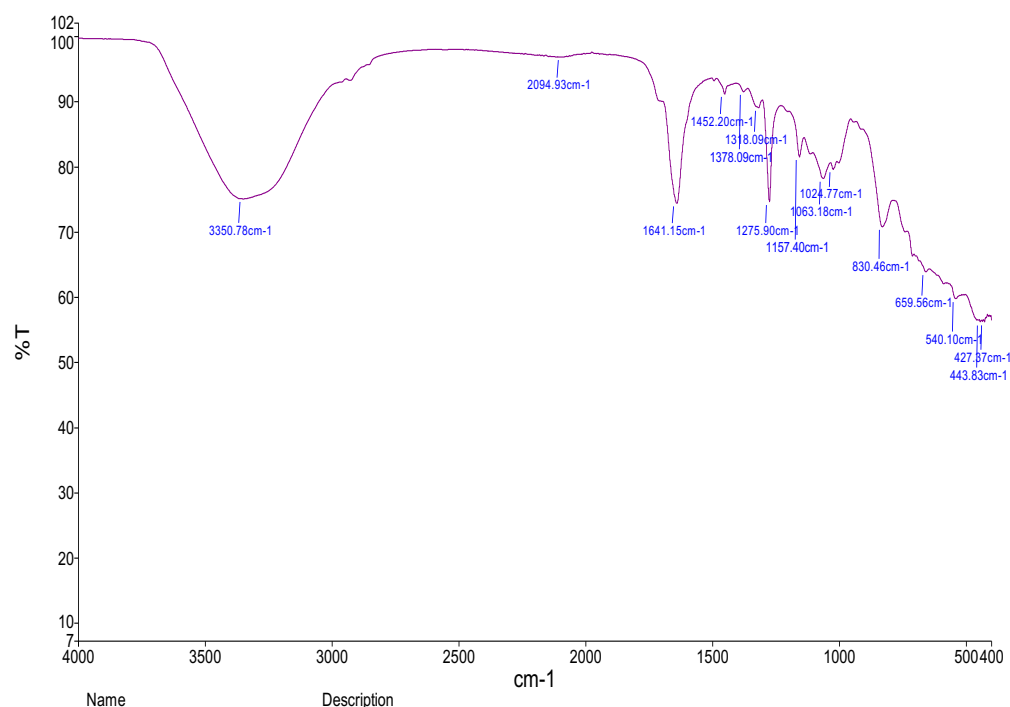


Figure 3. FTIR spectrum of the absorbed extract in the steel surface

3.2. Potentiodynamic Polarization

The polarization study for the API 5L carbon steel in various concentrations of the combination of the leaves extract is shown in Figs. 4-5. The result of the parameters calculated from the study are shown in Table 1. The corrosion rates and the corrosion current densities were measured from extrapolation of cathodic and anodic Tafel lines with respect to the corrosion potential (E_{corr}). The corrosion rate decreases as the inhibitor concentration increases. Without the addition of the inhibitor, the corrosion rate was found to be 58.53 mm/year and decreases to 10.078, 1.6828, and 0.93657 mm/year for each 10, 30, and 50ml inhibitor concentration. The decrease in corrosion potential is caused by the adsorption of the extract in the steel surface and causing the passivity of the surface in the HCl solution increased [3,17]. The decrease in corrosion rate is proportional to the decrease in corrosion current density and the increase in corrosion potential. Without the addition of the inhibitor, the current density was found to be 503700 μA and decreases to 867.330, 144.820, and 80.6000 μA for each 10, 30, and 50ml inhibitor concentration. At the same time, the corrosion potential increases from -443.26 to -445.54, -469.69, and -488.43 mV for each 0, 10, 30, and 50ml inhibitor concentration. The decrease in the corrosion current density shows that the corrosion reaction was successfully suppressed by the extract [17].

An inhibitor can be classified as a cathodic or anodic inhibitor if the E_{corr} of the inhibitor displaces more than 85 mV with respect to the blank. From the Table 1, the E_{corr} of all the inhibitor concentrations did not displace more than 85 mV with respect to the blank. This indicates that the extract could not be classified as cathodic or anodic inhibitors, instead this extract is classified as a mixed-type inhibitor [18,19].

Fig. 6 shows the the polarization curve of API 5L steel without and with the inhibitor. The curve shifted to a more negative values with the increasing of inhibitor concentration. This shows that this inhibitor is a mixed-type inhibitor but work predominantly by inhibiting the cathodic reaction. This is also confirmed by the value of the cathodic current densities (b_c) of the extract had a lower b_c compared to blank. This suggests that the addition of the inhibitor does not alter the hydrogen evolution mechanism, instead the hydrogen evolution was controlled [20].

From Table 1, the efficiency of the inhibitor is known to be increased. From Figs. 4-6 and Table 1, it is known that the concentration that gives the best result was 50ml inhibitor concentration.

Table 1. Potentiodynamic polarization result for API 5L steel in 1M HCl solution

Inhibitor Concentration (ml)	b_a (mV/dec)	b_c (mV/dec)	E_{corr} (mV)	I_{corr} (μA)	Corrosion Rate (mm/yr)	IE (%)
0	810,350	866,260	-443,26	503700	58,53	-
10	100,670	85,962	-445,54	867,330	10,078	85,25
30	103,530	73,9690	-469,69	144,820	1,6828	97,54
50	85,6200	97,1110	-488,43	80,6000	0,93657	98,63

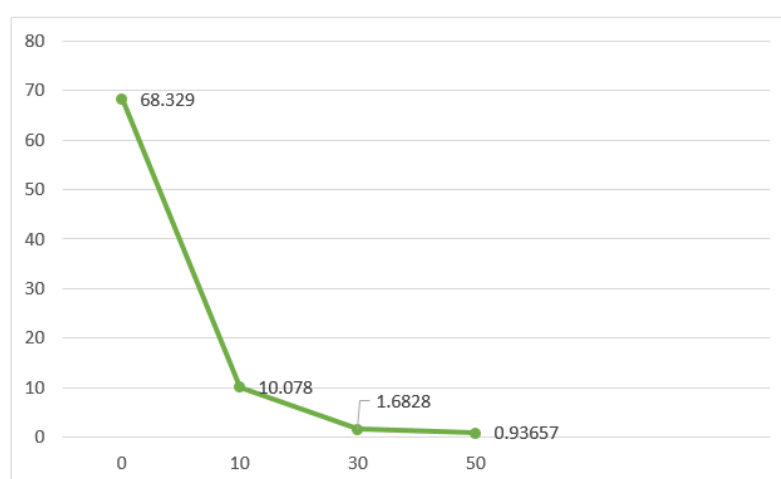


Figure 4. Plot of corrosion rate versus inhibitor concentration of API 5L steel in 1M HCl solution

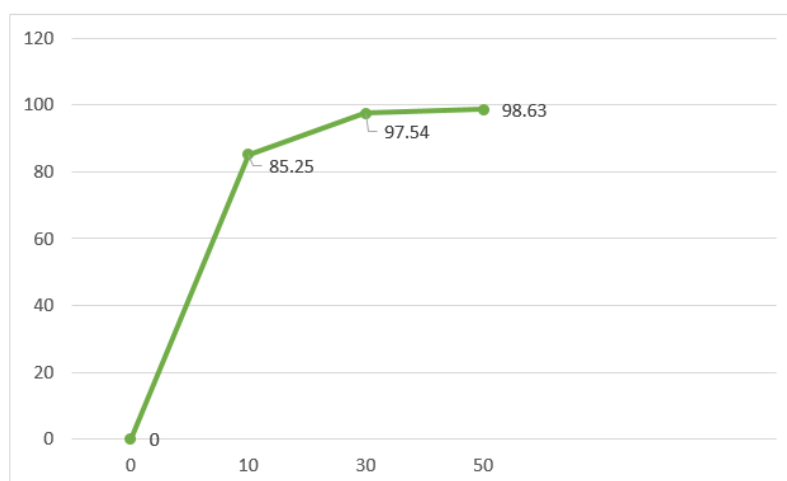


Figure 5. Plot of inhibitor efficiency versus inhibitor concentration of API 5L steel in 1M HCl solution

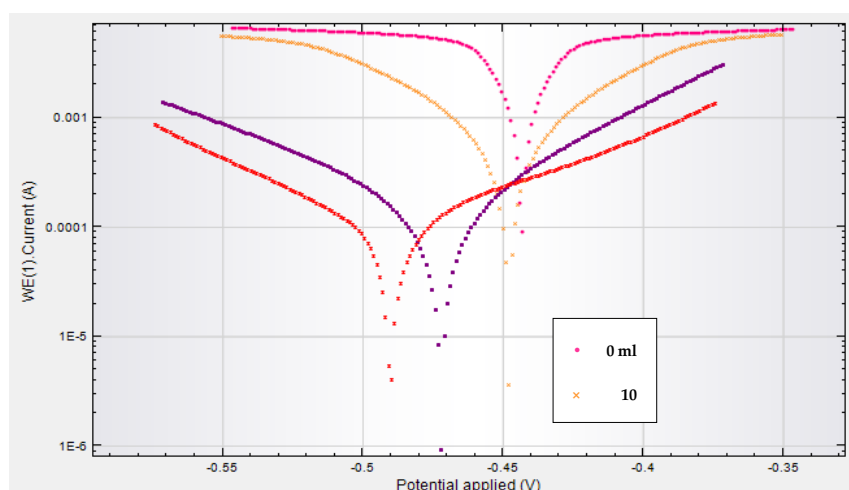


Figure 6. The polarization curve of API 5L steel without and with inhibitor in 1M HCl solution

3.3. Electrochemical Impedance Spectroscopy (EIS)

The corrosion behaviour of API 5L steel in 1M HCl without and with the inhibitor was investigated by the EIS method. The result was a semicircle curve named Nyquist diagrams which is shown in Fig. 7. The impedance parameters deduced from the analysis of Nyquist diagrams and value of IE are given in Table 2. The impedance parameters that are obtained were charge-transfer resistance values (R_{ct}) and double layer capacitance values (C_{dl}). The value of the R_{ct} increases with the increase of the addition of the inhibitor concentration, whereas the value of the C_{dl} decreases. Without the addition of the inhibitor, the R_{ct} was found to be 24.74Ω and increases to 74.83Ω , 168Ω , and 190.4Ω for each 10, 30, and 50ml inhibitor concentration. While for the C_{dl} , without the addition of the inhibitor, the value was found to be 178.8×10^{-6} F and decreases to 62.98×10^{-6} F, 41.21×10^{-6} , and 36.72×10^{-6} F. The increase of the R_{ct} values indicates that the protective

layer, called double layer, was formed in the interface between the specimen and solution [21]. The increase of the R_{ct} values also indicates that the corrosion reaction rate decreased because of the decrease of the active corrosion zone [22]. Meanwhile, the decrease of the C_{dl} values indicates that the thickness of the double layer increases. Double layer was formed because of the adsorption of inhibitor molecule on the interface between the specimen and solution. The changes of the R_{ct} and C_{dl} values are also because of the exchange of the water molecules with the inhibitor molecules [21].

Table 2. EIS result for API 5L steel in 1M HCl solution

Inhibitor Concentration (ml)	R_s (Ω)	R_{ct} (Ω)	C_{dl} / CPE (10^{-6} F)	IE (%)
0	$718,4 \times 10^{-3}$	24,74	178,8	-
10	$613,9 \times 10^{-3}$	74,83	62,98	66,94
30	1,029	168	41,21	85,27
50	$735,1 \times 10^{-3}$	190,4	36,72	87

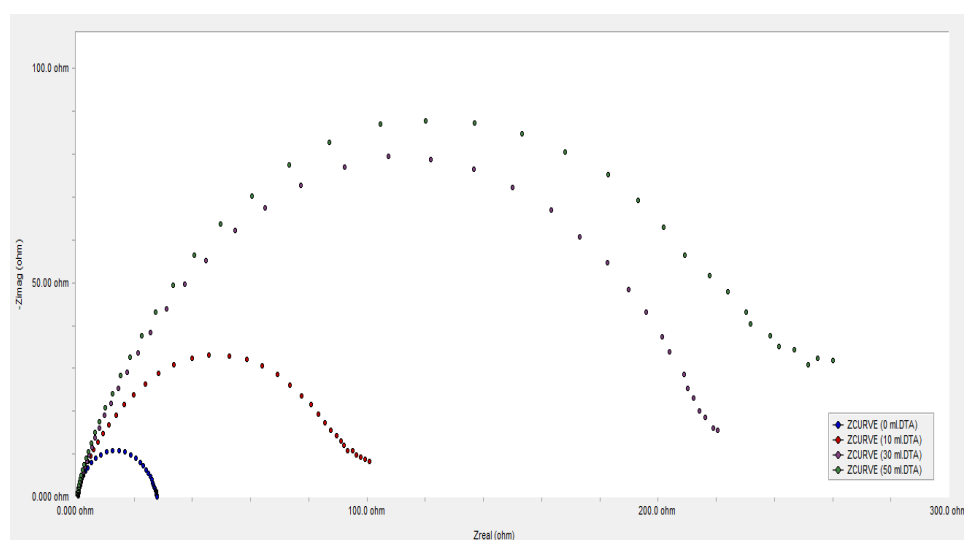


Figure 7. Nyquist diagrams of API 5L steel without and with inhibitor in 1M HCl solution

3.4. Adsorption Isotherm Model

The mode of adsorption of the inhibitor on the API 5L steel was tested with several adsorption inhibitors. Langmuir, Frumkin, Temkin, Flory-Huggins, and thermodynamic/kinetic model of El-Awade isotherm were the most frequently used isotherm. The interaction between molecule and metal surface must be higher than that of the water molecule and the metal surface to make sure that the inhibitor molecules were

absorbed on the metal surface [23]. The experimental data were tested using various isotherms and among various adsorption isotherms tested, Langmuir adsorption was found to be the best fit. The Langmuir adsorption isotherm can be expressed by Equation (3).

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (3)$$

where C_{inh} is the corrosion inhibitor concentration in the solution and K_{ads} is the equilibrium constant of the adsorption process [24]. Meanwhile, the free energy of adsorption, ΔG_{ads}° , can be calculated by Equation (4) [25].

$$K_{ads} = \frac{1}{55.5} \exp\left(-\frac{\Delta G_{ads}^{\circ}}{RT}\right) \quad (4)$$

The plot of the Langmuir adsorption, C_{inh}/θ vs. C_{inh} , displayed a straight line with an intercept of $1/K_{ads}$ for the tested inhibitor. The plot is shown in Fig. 8. The Langmuir adsorption isotherm for the studied inhibitor derived the thermodynamic parameters.

The interaction of the adsorption is described into two types of interaction which are called physisorption and chemisorption [26]. The type of the interaction can be known through the value of the ΔG_{ads}° or Gibbs free energy. The interaction type would be physisorption if the value of the Gibbs free energy is around -20 kJ mol⁻¹ or less negative, while the interaction type would be chemisorption if the value of the Gibbs free energy is around -40 kJ mol⁻¹ or more negative [24]. The Gibbs free energy was calculated and the result was -16.3398693 kJ mol⁻¹ for API 5L steel in 1M HCl solution. These values indicate that the type of interaction of the adsorption is physisorption. Meanwhile, the negative sign on the values of the Gibbs free energy indicates that the adsorption reaction is spontaneous [27].

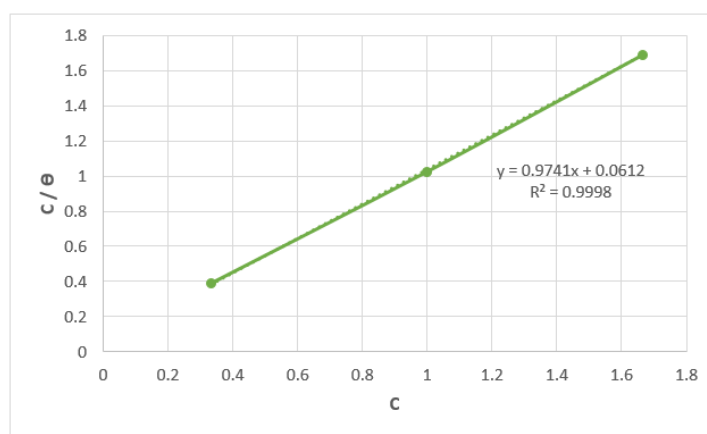


Figure 8. Langmuir's adsorption isotherm plots for the adsorption of the inhibitor on API 5L steel in 1M HCl solution

4. Conclusions

In this study, combination of *Physalis angulata* leaf and *Cassia angustifolia* leaf extract as green inhibitor with a 50:50 ratio experiments has been conducted to solve the corrosion problem for API 5L low carbon steel. The analysis evaluate using potentiodynamic polarization, EIS and FTIR led to the following conclusions:

1. The strong active compound that gives the inhibition effect of the combination of *Physalis angulata* leaf and *Cassia angustifolia* leaf extract to the surface of API 5L steel was found to be flavonoid.
2. The combination of *Physalis angulata* leaf and *Cassia angustifolia* leaf extract with a 50:50 ratio as green inhibitor was found to be a mixed-type inhibitor.
3. The combination of *Physalis angulata* leaf and *Cassia angustifolia* leaf extract with a 50:50 ratio as green inhibitor gave a synergetic effect to each other, as the efficiency of the inhibitor increases with the increase of inhibitor concentration from 10, 30, and 50 ml.
4. The best inhibitor concentration between 10, 30, and 50 ml was found to be 50 ml.
5. The adsorption inhibitor model was found to be Langmuir adsorption isotherm and the type of the interaction of the inhibitor with the specimen was found to be physisorption.

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