

# The Extract of Siamih (*Ageratum conyzoides* L) Leaf as a Green Eco-Friendly Corrosion Inhibitor for the Mild Steel St. 37 in HCl Solution

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## ABSTRACT

The extract of siamih (*Ageratum conyzoides* L) leaf was tested as a corrosion inhibitor of St. 37 steel in HCl medium using weight loss method, potentiodynamic polarization, Fourier Transform Infrared (FT-IR) analysis, and Scanning Microscopy Electron (SEM) analysis. Based on the method of weight loss, it can be seen that the corrosion rate decreases with the addition of the extract of siamih leaf. The highest inhibitory efficiency was 85.33% with the addition of an extract of 8 g/L, without the addition of iodide ions. The effect of temperature causes the inhibition efficiency to decrease with increasing temperature. Potentiodynamic polarization measurements show that the extract of siamih leaf is a mixed type inhibitor. SEM analysis shows the difference in the surface of the steel immersed in HCl without and with the addition of the extract of siamih leaf. There was a change in the shift in the wavenumber shown by the FT-IR spectrum before and after the extract of siamih leaf was added. The extract of siamih leaf adsorption on the steel surface corresponds to the Langmuir adsorption isotherm.

**Keywords:** *Ageratum conyzoides* L, corrosion inhibitor, potentiodynamic polarization, Langmuir adsorption isotherm, synergetic effect.

## INTRODUCTION

Steel is one of the metals that is often used in industry because steel has many advantages such as strong, easy to use, easy manufacturing process, and low

price. However, steel has a tendency to experience corrosion, especially in acidic, alkaline, and other aggressive environments. Steel corrosion in acidic medium is a concern in academia and industry. Corrosion prevention can be done by several methods in which the base of Reviews These methods was to protect the steel surface from the attack of corrosive environments. The method used is a coating on metal surfaces, cathodic protection, anodic protection, and the addition of corrosion inhibitors. [1,2]

The use of inhibitors to date was still the best solution to protect corrosion in metals and was used as the main defense for the oil processing and extraction industry. Inhibitors are flexible protection methods, the which were Able to provide protection from less aggressive environments to environments that have very high levels of corrosivity, are Easily applied and very high levels of effectiveness, Because The layers that were formed are so thin that in small quantities it was able to provide extensive protection. [3,4] Non-toxic or less toxic inhibitors are far more strategic. Research on corrosion inhibitors that are environmentally friendly, has been Carried out with the aim of being easy to use and relatively inexpensive. [5]

Some studies have been carried out using environmentally friendly materials as corrosion inhibitors, for example, The

extract of Waru leaf as corrosion inhibitors of mild steel in HCl medium, [6] *Ocimum basilicum L* oil as aluminium corrosion inhibitors in chloride acid solutions, [7] the effect of *Artemisia herbaalba* extract as a corrosion inhibitor of steel in hydrochloric acid solution, [8] the ability of *Musa acuminata* fruit extracts against steel corrosion in HCl medium, [9] and soft steel corrosion inhibitors in HCl medium using extracts from *Musa acuminata* flowers. [9]

The extract of siamih (*Ageratum conyzoides L*) leaf is a plant that grows in Indonesia. This siamih leaf contains saponins, flavonoids, coumarin, and tannins. [10] These organic compounds were expected to be adsorbed on the steel surface and can reduce the rate of corrosion.

In this study, the extract of siamih (*Ageratum conyzoides L*) leaf as a green eco-friendly corrosion inhibitor for the mild steel St. 37 in HCl medium using weight loss methods, potentiodynamic polarization, Fourier Transform Infrared (FT-IR) analysis, and Scanning Microscopy Electron (SEM) analysis.

## EXPERIMENTAL METHOD

### Instruments and Materials

The equipment were used in this study are analytical balance, calipers (digital caliper Inoki), iron emery, watch glass, boiling flask, oven, potentiostat (eDaq), rotary evaporator (Heidolph W2000), waterbath, FT-IR (Thermo Scientific Nicolet Nicolet iS10 uses KBr pellets), hotplates, thermometers, grinders, glassware, and SEM (Hitachi S-3400N).

The materials were used in this study are St.37 Steelas a working electrode (WE), Pt electrodes as a auxiliary electrode (AE), and Ag/AgCl electrodes (Metrohm, Swidzeland) as a reference electrode (RE), distilled water, acetone (Merck, Germany), HCl (Merck, Germany), and methanol (Merck, Germany), The Siamih leaf was taken from Andalas University Limau Manih, Padang, Indonesia.

## Research Procedure

### Preparation of Steel

Shaped cut steel bars with a diameter of  $\pm 2.5$  cm, and a thickness of  $\pm 0.1$  cm. Then smooth the surface using sandpaper iron and rinse with distilled water. Then rinse with acetone to remove fat that might stick to the specimen. Furthermore, the steel was dried in an oven at 60°C. After drying, the steel was weighed, and the weighing results were expressed as an initial weight ( $m_1$ ).

### Preparation of The Extract of Siamih (*Ageratum conyzoides L*) Leaf

Fresh leaves were dried and then mashed. Then weighed 400 g then extracted with 2000 ml of methanol for 3 days (3 times). The extract was filtered and evaporated obtained by the solvent using a rotary evaporator. The concentrated extract obtained about 5 g, then put into a 100 mL glass beaker to make an inhibition solution with different concentrations of 1 g/L, 2 g/L, 4 g/L, 6 g/L, and 8 g/L.

### Phytochemical Test of Siamih Leaf

The method of checking the content of flavonoids, triterpenoids, steroids, and phenolic compounds items, namely: 1 g of fresh sample was inserted into a test tube, then macerated with methanol, which has been heated (on a water bath) for 15 minutes. Then filter the heat into another test tube, and let all the methanol evaporate to dry. Then added chloroform and water with a ratio of 1:1 each as much as 5 mL, shake well, then transfer into a test tube, allow a moment to form two layers of chloroform-water. Chloroform layer at the bottom was used for examination of triterpenoid compounds, and steroids. While the water layer was used for examination of phenolic compounds and flavonoids.

### Determination of Corrosion Rate by Weight Loss Method

The steel was immersed in 50 mL of 5N HCl corrosive solution at various concentrations, with the addition of the extract of siamih leaf and without the

addition of the extract of siamih leaf with temperature variations of 30°C, 40°C, 50°C, and 60°C for 7 hours using a water bath. Then the steel was cleaned, rinsed, and dried in an oven at 60°C. After drying, the steel was weighed and the weighing results were stated as the final weight ( $m_2$ ).

### Potentiodynamic polarization

Potentiodynamic polarization was used St. 37 steel as a WE with a diameter of 0.02 cm, and surface area of 0.055 cm<sup>2</sup>, Pt electrode as a AE, and Ag/AgCl as a RE. Potentiodynamic polarization measurements were carried out in 1N HCl medium, and HCl medium in inhibitor 1 g/L; 2 g/L; 4 g/L; 6 g/L, and 8 g/L, respectively. The three electrodes were immersed in vessels containing corrosive HCl medium without and with the addition of various inhibitors concentration. Then it was connected to the potentiostat and regulated to the potential so that the relationship curve between the potential (E) vs. current (I) was obtained. In potentiodynamic polarization measurements, the relationship curve between potential (mV) and ln current (mA/cm<sup>2</sup>) was obtained. The potential were used is from -1000 mV to -500 mV to obtain the potentiodynamic polarization curve.

### Analysis of Fourier Transform Infrared (FT-IR)

Analysis of FT-IR was carried out by taking corrosion products attached to steel, corrosion products were then dried and Analyzed by FT-IR using KBr pellets plates, FT-IR measurements were Also Carried out for the extract of siamih leaf.

### Analysis of Scanning Electron Microscopy (SEM)

The steel was immersed in 1N HCl 1N without and with the addition of the extract of siamih leaf for 7 hours. Then dried and analyzed with SEM.

## RESULTS AND DISCUSSION

### Phytochemical Test ofThe Extract of Siamih Leaf

Phytochemicals testing aims to identify the content of secondary metabolites contained in the extract of siamih leaf. Table 1 shows that in the extract of siamih leaf contains compounds Several secondary metabolites such as alkaloids, flavonoids, phenolics, and triterpenoids roommates were indicated resources by positive signs (+). While steroids are not contained in the extract of siamih leaf which was indicated resources by the sign (-).

Table1.Phytochemicals Test Results

Chemical Compounds	Test results
Alkaloids	+
Flavonoids	+
Phenolics	+
Triterpenoids	+
Steroidz	-

Description: + declare the content of these chemical compounds in the extract of siamih leaf

### Analysis of Weight Loss Method

#### Effect of Concentration on Corrosion Rate and Efficiency of Corrosion Inhibition of Steel with Temperature Variations in 1N HCl Medium

Figure 1 shows the effect of the corrosion rate with variations in concentrations of 0 g/L, 1 g/L, 2 g/L, 4 g/L, 6 g/L, and 8 g/L the extract of siamih leaf at temperatures of 30, 40, 50, and 60°C. Figure 1 shows that the corrosion rate decreases with an increase in the concentration of the extract of siamih leaf. The corrosion rate is only reached 0.11 mg/cm<sup>2</sup>.hours in 1N HCl corrosive medium in the presence of the extract of siamih leaf at 8 g/L. In addition, the corrosion rate will be even greater with an increase in temperature. This was due to the increase in temperature, the kinetic energy between the particles are also greater so that it can increase the rate of corrosion of steel. Also Figure 1 indicates that the extract of siamih effectively leaf was used as a corrosion inhibitor. This was the caused by groups of secondary metabolite compounds contained in the extract of siamih leaf bonded to metals contained in

steel to form more stable complex compounds, so as to slow down the corrosion reaction in steel.

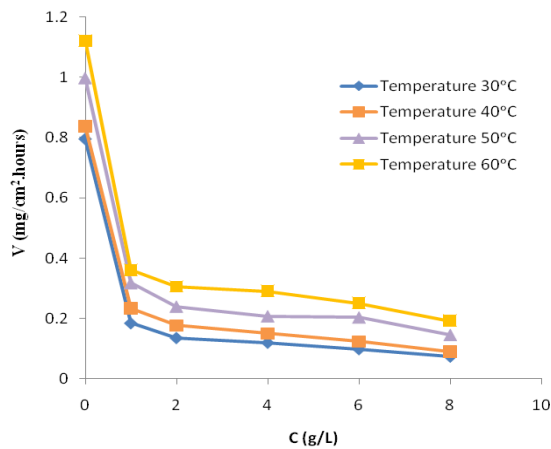


Figure 1. Effect of concentration on the corrosion rate (mg/cm<sup>2</sup>. hours) of steel in 1N HCl solution (7 hours).

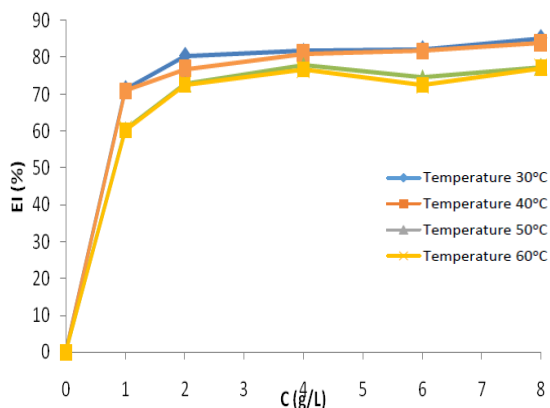


Figure 2. Effect of the concentration of the extract of siamih leaf on the efficiency of inhibition (%) corrosion of steel in 1N HCl solution at a temperature variation for 7 hours.

Figure 2 shows the percent efficiency of inhibition by varying the concentration of siamih leaf extract. Figure 2 shows that the efficiency of inhibition increases with an increase in the concentration of siamih leaf. Corrosion inhibition efficiency reaches 85.33% at a concentration of 8 g/L in 1 N HCl corrosion medium.

The decrease in corrosion rate and increase in inhibition efficiency with the addition of siamih leaf concentration was caused by the adsorption of the extract of siamih leaf on the steel surface so that a thin layer was formed which protects the steel surface. [11,12] The compound contained in the extract of siamih leaf acts as a ligand because it has a lone pair of electrons donated to the center of Fe<sup>3+</sup>. This complex compound which protects the steel from attack by its corrosive HCl medium. Increased inhibition efficiency with the extract of siamih leaf was adopted on the steel surface. This steel surface will block aggressive ions to the steel surface so that it will reduce the corrosion rate of the steel in the corrosive HCl medium. [13]

### Determination of Activation Energy

Figure 3 is the Arrhenius channel showing the relationship between 1/T, and ln v, where the slope value of the straight line equation can be used to determine the activation energy value. [14]

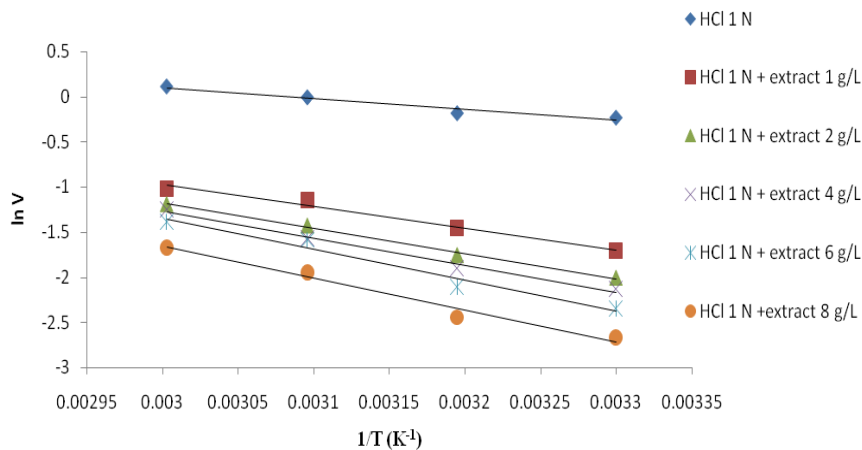


Figure 3. Arrhenius 1/T (K<sup>-1</sup>) vs ln v channel for steel corrosion in 1N HCl medium with 7 hour immersion time.

Table2.Values of the activation energy (Ea), entalpi ( $\Delta H^\circ$ ) dan entropi  $\Delta S^\circ$

Medium	Activation energy (kJ/mol)	$\Delta H^\circ$ (kJ/mol)	$\Delta S^\circ$ (J/mol)
HCl 1 N	7.07	4.43	34.35
HCl 1 N + 1 g/L extract	17.88	15.24	70.48
HCl 1 N + 2 g/L extract	16.84	14.20	17.29
HCl 1 N + 4 g/L extract	14.47	11.83	26.09
HCl 1 N + 6 g/L extract	20.71	18.07	5.94
HCl 1 N + 8 g/L extract	20.76	18.12	7.04

Table 2 shows the activation energy with the addition of the extract was greater than the activation energy without the addition of extract (blank). The high activation energy in the addition of this extract shows that the reaction was difficult to occur because of the inhibitor of the extract of siamih leaf which was adsorbed on the steel surface that forms a layer. This layer can inhibit the attack of the HCl medium or can inhibit corrosion. [11]

Values of  $\Delta H^\circ$  positive during immersion indicates that the steel undergoes an endothermic reaction. The value of  $\Delta H^\circ$  with the addition of extract greater than the blank indicates that it takes a lot of energy to be able to process the corrosion of steel. In addition, the average value of  $\Delta H^\circ > 40$  kJ/mol indicates the bond between the compound contained in the extract, and the metals on the steel surface are the chemical bond that forms the monolayer layer. The value of  $\Delta S^\circ$  decreases with the addition of extracts indicating that the addition of the extract of siamih leaf as an inhibitor can slow the rate of corrosion. [13]

### Adsorption Isotherms

Figure 4 is an adsorption isotherm based on the measurement of weight loss method that meets the langmuir adsorption isotherm because the flow between C and C/θ gives a straight line with a correlation coefficient ( $R^2$ ) of 0.99, where C is the concentration of the extract of siamih leaf, and C/θ concentration of the extract of siamih leaf per degree closure. [15]

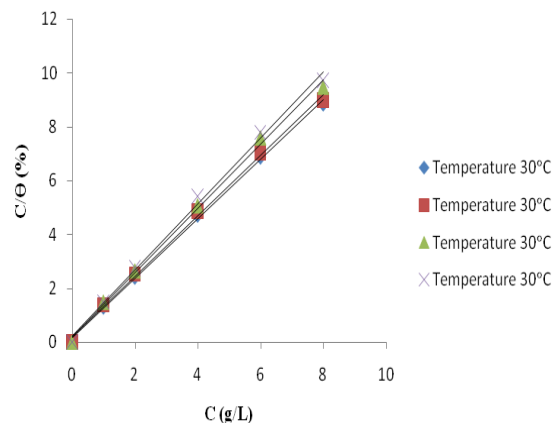


Figure 4. The Langmuir adsorption isotherm for corrosion of steel in 1N HCl medium with 7 hour immersion time based on the measurement of weight loss method.

Table3. Values of correlation coefficient ( $R^2$ ) on someadsorption isotherm.

No.	Adsorption isotherm	Correlation coefficient ( $R^2$ )
1.	Langmuir	0.99
2.	Freundlich	0.99
3.	Temkin	0.98

### Potentiodynamic

#### Polarization Measurement

Figure 5 shows that the HCl curve is located on the right and left of the curve with the addition of the extract of siamih leaf. This indicates that the extract of siamih leaf is a mixed inhibitor. Potentiodynamic polarization curves were extrapolated using the Tafel method. In addition, the potentiodynamic polarization curve shows the interaction between the extract of siamih leaf with the St-37 steel electrode.

Table 4 shows reactions occurring at the anodic and cathodic. The extract of siamih leaf was able to inhibit corrosion in the anodic by means of the adsorption of the extract of siamih leaf so that it inhibits the oxidation reaction or solubility of the Fe metal to be reduced, and inhibits the occurrence of cathodic corrosion by the reaction between  $H^+$  ions from HCl with the extract of siamih leaf which was added so that the process of reducing the  $H^+$  ions to  $H_2$  decreases, therefore the leaf extract acts as a mixture type of inhibitor. This can be seen from the value of the corrosion potential ( $E_{corr}$ ) which was shifting towards the anodic and cathodic. Corrosion potential values up and down indicates the stability of the extraction of siamih leaf adsorption on

the steel surface. Corrosion currents were caused by the movement of oxidized steel electrons in the HCl medium. [16]

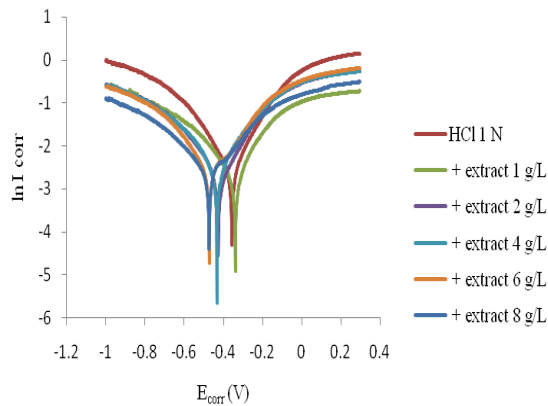


Figure 5. Potentiodynamic polarization curves without and in the presence of the extract of siamih leaf in 1N HCl medium.

Table 4. Corrosion potential values, corrosion currents, and steel corrosion inhibition efficiency without and with the addition of the extract of siamih leaf.

Extract Concentration (g/L)	$E_{corr}$ (V)	$I_{corr} \times 10^{-3}$ ( $\mu A$ )	EI (%)
0	-0.38	57.40	-
1	-0.36	54.90	4.50
2	-0.42	17.30	69.80
4	-0.43	10.90	80.94
6	-0.46	8.70	84.86
8	-0.46	6.90	87.97

Table 4 can be seen the value of the corrosion current decreases with increasing

concentration of the extract of siamih leaf added. The decrease in the current value was due to the addition of the extract of siamih leaf the movement of oxidized steel electrons in the 1N HCl medium decreases. The value of inhibition efficiency increases with increasing concentration of the extract of siamih leaf. The highest inhibitory efficiency value of the Tafel extrapolation channel value on the addition of the extract of siamih leaf with a concentration of 8 g/L, that was equal to 87.97%. [17]

### Analysis of FT-IR

Analysis of FT-IR each compound has a specific functional group of each. Compounds that can be used as corrosion inhibitors are compounds that have a hydroxyl (OH), carboxyl (-COOH), carbonyl (=CO), =CO-, C-H, =CH<sub>2</sub>, -C=C<sub>2</sub>, -C≡C-, -C-Cl, amine (-C≡N), and others which have electron pairs that can bond directly to form stable complexes on the metal surface. The infrared spectrum was used to determine the functional groups that act as corrosion inhibitors of secondary metabolite compounds contained in the extract of siamih leaf. [18]

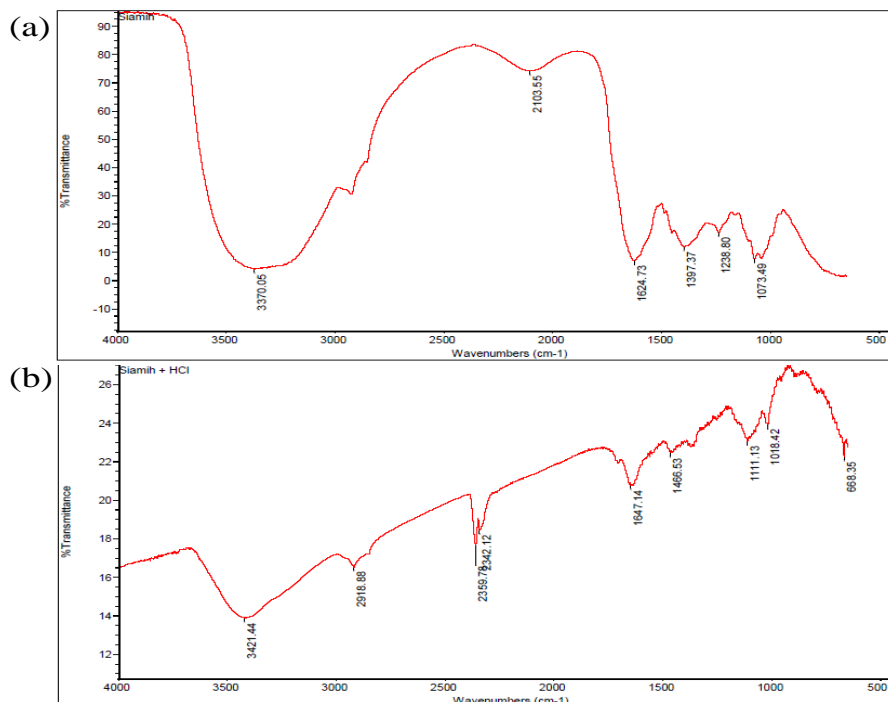


Figure 6. FT-IR spectrum of the extract of siamih leaf(a); corrosion products on steel immersion for 7 hours in 1N HCl with the addition of 8 g/L of the extract of siamih leaf(b).

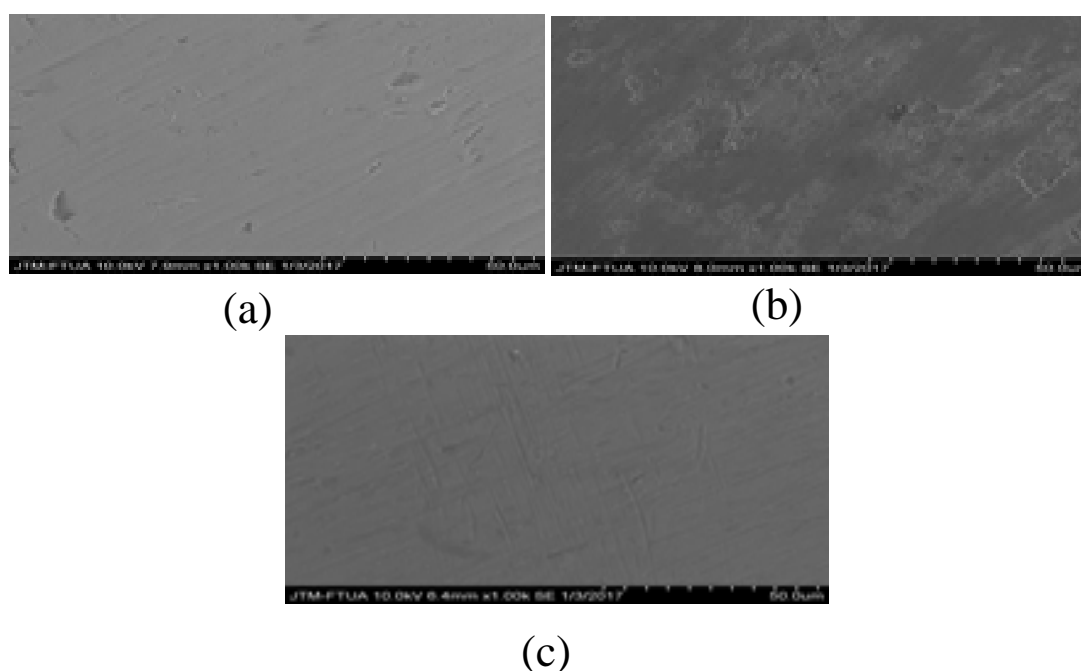
Figure 6(a) is a spectrum of the extract of siamih leaf. The spectrum shows the hydroxy group (-OH) ( $3500-3000\text{ cm}^{-1}$ ), the stretching CH group ( $3000-2500\text{ cm}^{-1}$ ), and the C=C stretching group ( $1640-1610\text{ cm}^{-1}$ ), CO stretching ( $1042-1448\text{ cm}^{-1}$ ), and low-intensity  $\text{CO}_2$  vibrations at the wave number  $2360.37\text{ cm}^{-1}$ . Figure 6(b) shifts the hydroxy peak (-OH) from wave number  $3393.75\text{ cm}^{-1}$  to  $3432.19\text{ cm}^{-1}$ , and also shifting group C=C stretching from wave number  $1616.78\text{ cm}^{-1}$ - $1637.66\text{ cm}^{-1}$ , besides shifting in the group. The shift also occurs in other functional groups. This happens because of the interaction between the extract of siamih leaf with the steel surface, and forms a thin protective layer on the steel surface [18].

Figure 6 shows that the infrared spectrum of the extract of siamih leaf, and the infrared spectrum of corrosion products on steel surfaces have relatively similar chemical changes. But there was a slight difference in

the strength or weakness of the absorption peak produced, and there were some absorption peaks that are lost and there were also new absorption peaks that appear. In addition, from the shift in the infrared spectrum wave number shift, this shows the interaction between the compounds contained in the extract of siamih leaf with steel through extraction adsorption on the steel surface. So the extract of siamih leaf has a protective effect and can inhibit the rate of corrosion of steel.

### Analysis of SEM

Analysis of SEM is one way to prove effect of adding the extract of siamih leaf to changes in the corrosion rate of steel. SEM provides information about the morphological shape of the steel before treatment Figure 7(a), after immersion in 1N HCl Figure 7(b), and after immersion in 1N HCl with the addition of the extract of siamih leaf Figure 7(c).



**Figure 7.** Results of SEM (a) steel before it soaked, (b) steel after being soaked with a solution of HCl, (c) steel after being soaked with 1N HCl, and extract with a concentration of 8 g/L ( $30^\circ\text{C}$  with a massive 1000x).

Figure 7 (a) is a photograph of the surface of St. 37 steel without treatment at 1000x magnification, the surface of the steel still looks good, non-porous, and does not crack because there was no interaction with

the HCl medium. Figure 7(b) is a photograph of steel surfaces that have been immersed in 1N HCl media for 7 hours. The surface of the steel undergoes corrosion which was characterized by damage to the

surface of the steel with visible brown and rust holes. This happens because of the interaction between the steel surface with  $H^+$  and  $Cl^-$  ions.

Figure 7(c) is a photograph of steel surfaces that has been immersed in 1N HCl media with the addition of 8 g/L the extract of siamih leaf. The surface of the steel was less damaged compared to the surface of the steel which was immersed with 1N HCl medium without siamih leaf extract. This was because the protective layer of siamih leaf extract was formed on the steel surface.

## CONCLUSION

Research that has been done can be concluded that the extract of siamih leaf can be used as a corrosion inhibitor. The inhibitory efficiency extract value at the optimal concentration of 8 g/L in 1N HCl is 85.33%. This proves that the extract of siamih leaf can be used as a corrosion inhibitor of steel. Potentiodynamic analysis shows that the corrosion current decreases with increasing concentration of siamih leaf extract. The activation energy value increases with increasing extract concentration which shows that the extract of siamih leaf, corrosion will be difficult. The SEM results also showed a significant change between without and in the presence of the extract of siamih leaf. There was a change in the shift in the number of waves shown by the FT-IR spectrum before, and after the extract of siamih leaf was added.

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