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Sarang Semut (*Myrmecodia Pendans*) EXTRACT AS A GREEN CORROSION INHIBITOR FOR MILD STEEL IN ACID SOLUTION

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ABSTRACT

In this work, *Myrmecodia pendans*, or *sarang semut*, was successfully extracted as a potential organic inhibitor. Various concentrations of *Myrmecodia pendans* extract in 1 M H_2SO_4 solution were applied in the range of 100–500 mg/L for corrosion inhibition. Gas chromatography–mass spectroscopy (GC-MS) showed that the *Myrmecodia pendans* extracts contained a high-benzenediol compound with rich oxygen atom content, which played an important role in the inhibition process. The 300 mg/L *Myrmecodia pendans* extract had the highest effect, decreasing the rate of corrosion from 177.73 mpy to 47.4 mpy. This was attributed to the chemical compounds present in the *Myrmecodia pendans* extract on the surface of mild steel, which prohibited the corrosion rate.

Keywords: Extract method; Inhibitor; Mild steel; *Sarang semut*

1. INTRODUCTION

Corrosion is a process in which material deteriorates due to its reaction with the environment (Santana et al., 2012). It can lead to a process of equipment damages that may cause disturbance in the production process. Furthermore, corrosion can represent a problem in production processes owing to the loss of the product due to corrosion of the means of transportation; this may increase the costs of the product and equipment maintenance. For these reasons, corroded surfaces need to be controlled.

Focusing on the corrosion inhibition of pipes, the corrosion process on the outside of the pipe can be inhibited by painting and cathode protection, while the corrosion process on the inside of the pipe can successfully be addressed using a corrosion inhibitor. There are two main types of corrosion inhibitors, namely inorganic inhibitors and organic inhibitors. Inorganic inhibitors have been well recognized as an efficient medium to decrease the rate of the corrosion. However, many of these substances may have a detrimental effect on the environment. Therefore, organic inhibitors are preferred as an alternative for corrosion inhibitors due to their wide availability, low cost of production, and environmental friendliness. *Sarang semut* is one of many local natural ingredients that appear to have excellent potential as an organic inhibitor adding an extract of this substance into a solution of NaCl at 3.5% showed inhibition efficiency of about 90% (Pradityana et al., 2014; Pradityana et al., 2016). *Sarang semut*, widely known as *Myrmecodia pendans,* is an epiphytic plant that grows naturally in Papua. This kind of plant is

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rich in antioxidants and flavonoids. A study carried out by Subroto showed that *Sarang semut* contains many flavonoid compounds, tannins, polyphenols, and tocopherols (Soeksmanto et al., 2010). A similar study conducted by Engida using the high-performance liquid chromatography (HPLC) method showed that this substance contains phenolic and flavonoid compounds in the orders of 330.61±2.13 mg GAE/g and 63.28 ± 1.75 mg QE/g, respectively (Engida et al., 2013).

Mild steel is a type of material that is commonly used in various applications in the industrial world, such as pipeline systems, because of its wide availability and good tensile strength. This type of steel is likely to be affected by corrosion in an acidic environment, especially in HCl and H2SO4 solution. The greatest problem occurring in pipeline systems is internal corrosion, as the flowing fluid contains many compounds, such as oxygen, acid sulfide, carbon monoxide, and sulfate-reducing bacteria (Al-Otaibi et al., 2012).

Many studies have been devoted to the subject of corrosion inhibitors that can be used for mild steel in acidic media. Santana reported that the first organic inhibitor in the pickling process using sulfuric acid was extract of *Chelidonium majus* (celandine) (Al-Otaibi et al., 2012). *Uncaria gambir* (Hussin & Kassim, 2011), *Justicia gendarussa* (Satapathy et al., 2009), green *Capsicum annuum* fruit (Ji et al., 2012), *Murraya koenigii* (Quraishi et al., 2010), *Salvia officinalis* (Soltani et al., 2012), and fennel *(Foeniculum vulgare)* (Lahhit et al., 2011) have also been reported as corrosion inhibitors (Santana et al., 2012).

Organic compounds consisting of one or more atoms of nitrogen, oxygen, sulfur, phosphor, double bonding, or an aromatic ring on their molecules can be used as corrosion inhibitors because they can be easily coated on a metal surface. The structure of the compound shows a pair of free electrons that results in adsorption of the inhibitor on the metal surface. The process leads to the formation of a layer of protection that prevents direct contact of the metal to the corrosive medium, resulting in a decreased rate of corrosion. Organic inhibitors can act as anodic and cathodic inhibitors because they can inhibit both anodic and cathodic reactions. This will result in a decreasing rate of corrosion marked by the reduced speed of the anodic, cathodic, or both reactions (Quraishi et al., 2010; Soltani et al., 2012).

In this work, the effectiveness of *sarang semut* in the corrosion inhibition of mild steel in a 1 M H_2SO_4 solution is systematically presented. The rate of corrosion is measured using the Tafel extrapolation method and electrochemical impedance spectroscopy (EIS), while gas chromatography–mass spectroscopy (GC-MS) is used to analyze the role of cluster function in *sarang semut*. This research is expected to make a beneficial contribution to the study of corrosion inhibition by organic substances; with the possible future introduction of inhibitor trading schemes, it could be used as an alternative means of corrosion inhibition in mild steel pipes, which are mostly used in the oil and natural gas industries.

2. EXPERIMENTAL PROCEDURES

2.1. Specimens and Solutions

The specimens comprised mild steel with a composition of 0.09% C, 0.49% Mn, 0.014% P, 0.007% S, 0.011% Si, and the balance Fe. For electrochemical measurement, a specimen was cut at 10×10×3 mm. One side of this specimen was connected to a copper wire covered with a plastic tube to avoid contact with the environment. The specimens were polished using emery paper of grades 500, 1000, and 1500. Then, specimens were washed with demineralized water and acetone. An aggressive solution of 1 M H_2SO_4 was prepared by the dilution of 98% H_2SO_4 from Merck using demineralized water.

2.2. Extraction

Sarang semut with a minimum diameter of 10 cm was cut and dried under sunlight for about a day, and subsequently dried in an oven for about 12 hours at 70°C. Following this, it was ground and sieved in preparation for solution in ethanol. The solution was slowly stirred and dried at 30°C for about 5 hours and filtered to obtain a homogenous solution (Soeksmanto et al., 2010) Moreover, ethanol was evaporated from the mixture using the rotary evaporation method. Finally, in this procedure, 21.3 g of extract was obtained from 100 g of *sarang semut* powder; this was stored in a glass bottle for future use.

2.3. Electrochemical Measurements

Electrochemical measurements with three electrode cells (potentiostat) were used to determine corrosion rate. Platinum was used as the counter electrode (CE), while the saturated calomel electrode (SCE) was used as the reference electrode (RE), and the mild steel specimens as the working electrode (WE). The WE surface area was prepared as described in the previous section. All electrochemical measurements were performed using the VersaStat provided by the Versa Studio software. Prior to measurement, the specimen was immersed in a corrosion medium at open circuit potential (OCP) until it reached steady state conditions. It was recorded as a function of time up to 30 min. The polarization curve was graphed with a scan rate of 0.1 mV/s. This inhibition efficiency (IE%) can defined as

$$IE\% = \frac{CR_o - CR_1}{CR_o} \; x \; 100\% \tag{1}$$

where CR_o and CR_1 are the corrosion rates in the absence and presence of the inhibitor, respectively.

The EIS method was used to determine the resistance of the charge transfer and the electric bilayer in the interface specimen with the solution. When the measurement was conducted using the EIS method, the WE was mounted in the electrochemistry cell in front of the platinum auxiliary electrode at a distance of about 2.5 cm, while the RE was in free position. The SCE was then entered to 1 M H_2SO_4 with various concentrations of *sarang semut* inhibitor from 100 to 500 mg/L in multiples of 100 mg/L. EIS was performed at OCP in the frequency range of 10,000 to 0.1 Hz using a signal amplitude of 1 mA. In addition, the impedance data were analyzed using ZView software to determine the parameters of the proposed equivalent electrical circuit models. The content transfer of the resistance value (R_{ct}) was obtained from the diameter of a half-circle Nyquist plot. The inhibition efficiency was calculated using the following equation:

$$IE\% = \frac{R_{ct} - R_{ct,o}}{R_{ct}} \; x \; 100\% \tag{2}$$

2.4. Gas Chromatography–mass Spectroscopy Measurement

This GC-MS inspection was carried out to investigate the specific compound inside the *sarang semut* that was thought to decrease the corrosion rate. GC is known as a spectroscopy technique that is carried out by distinguishing components through the difference in migration speed of each component. It is often used to identify a compound in a mixture of gas and to determine the concentration of compound in the gas phase. MS is known as a method used to determine molecular weight. In practice, the use of GC can be combined with MS. This combination will lead to more accurate data in identifying the compound, as well as its molecule structure.

GC-MS was carried out via injection of the extract powder dissolved in 96% ethanol. The solution was injected into GC-MS instrument column of an Agilent 6980N Network GC System with an autosampler using the injection port, which was heated at 500°C for 1 minute.

This solution was brought into the tubes containing carrier gas. The thickness of the film that was determined by the detector in the certain period resulted in the production of graphic that showed the compound having the greatest area together with compound names inside the extract.

3. RESULTS AND DISCUSSION

3.1. Potentiodynamic Polarization Study

The Tafel curves of different concentrations of extract inhibitor for mild steel in 1 M H_2SO_4 solution were studied using the potentiodynamic polarization technique. The Tafel curves are shown in Figure 1. The Tafel polarization technique was used to evaluate the corrosion inhibition efficiency of *sarang semut* extract. In addition, some electrochemical parameters, such as corrosion current density (I_{corr}), corrosion potential (E_{corr}), anodic Tafel slope (β_a), and cathodic Tafel slope (β_c), could be obtained, as shown in Table 1.

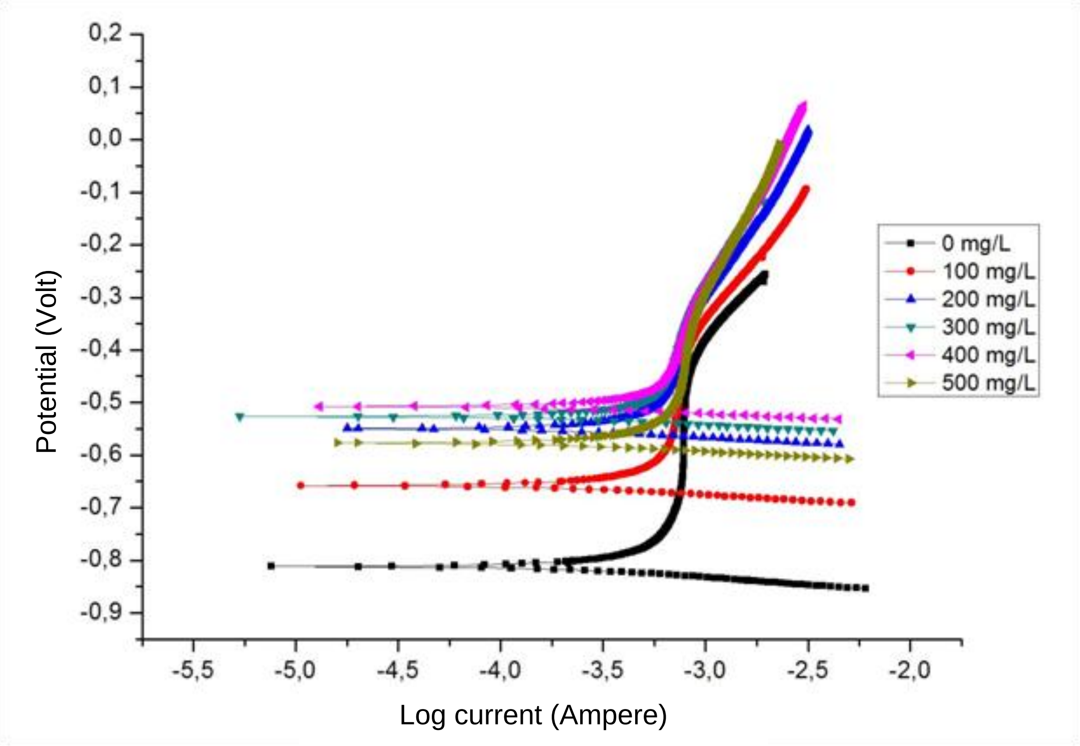


Figure 1 The representative Tafel polarization curve of the various concentrations of *sarang semut*

It can be observed from the Tafel curves that the different concentrations of extract inhibitor shifted the cathodic and anodic curves. An inhibitor can be called a cathodic or anodic inhibitor (depending on the direction) if the E_{corr} shift is more than 85 mV (Hussin & Kassim, 2011; Satapathy et al., 2009). In the presence of *sarang semut* extract inhibitor, the E_{corr} shifted to more positive values; there was no considerable variation with different concentrations of inhibitor. The max corrosion potential (E_{corr}) of mild steel shifted 303.63 mV to the right in comparison to that of the blank (anodic displacement). Therefore, the *sarang semut* extracts acted as an anodic inhibitor in these experiments. The anodic constant (β_a) value is attributed to metal dissolution and adsorption of the inhibitor at anodic potentials, whereas the cathodic constant (β_c) is related to hydrogen evolution at cathodic potentials (Ji et al., 2012). As shown in Table 1, the values of β_a and β_c decreased gradually in 100–300 mg/L concentrations of *sarang semut* extracts, but the β_a value behaved regularly, β_c increased at 500 mg/L. These patterns indicated that the presence of the inhibitor affected both the anodic and cathodic reactions (Quraishi et al., 1010; Li et al., 2009a). Table 1 also shows that adding inhibitor could decrease the value of I_{corr} in 1 M H_2SO_4 with a *sarang semut* concentration of 300 mg/L. The lowest value of I_{corr} was 103.43 µA/cm^2. The inhibition efficiency was compared from the value of I_{corr} in the absence or presence of *sarang semut* extracts. The increasing efficiency varied until 73.33% with the additional concentration at 100–500 mg/L.

Table 1 The corrosion parameter produced by the Tafel curves

Inhibitor concentration (mg/L)	E_{corr} (mv/SCE)	I_{corr} (µA/cm^2)	β_a (mV/dec)	β_c (mV/dec)	Corrosion rate (mmpy)	IE%
0	−811.257	387.818	38.612	215.827	177.73	-
100	−657.574	250.592	30.827	167.798	114.84	35.38
200	−542.337	220.958	28.597	142.676	101.26	43.03
300	−526.185	103.430	24.320	88.154	47.40	73.33
400	−507.627	165.267	20.872	97.176	75.74	57.39
500	−576.348	194.755	26.332	98.343	89.25	49.78

3.2. Electrochemical Impedance Spectroscopy (EIS) Measurement

Electrochemical parameters were obtained from EIS plots for mild steel in 1 M H_2SO_4 with and without *sarang semut* extract at concentrations of 100, 200, 300, 400, and 500 mg/L. These are shown in Table 2. In addition, Table 2 presents the calculated data obtained by the ZView fitting program. R_s (solution resistance), R_{ct} (charge transfer resistance) and CPE is the constant phase element for the double-layer capacitance. The R_{ct} values were calculated from the difference in impedance at lower and higher frequencies, as suggested by Elkadi et al. (2000). To obtain the double-layer capacitance (C_{dl}), the frequency at the maximum ($-Z_{i\max}$) imaginary component of impedance was determined and C_{dl} values were calculated from the following equation (Elkadi et al., 2000):

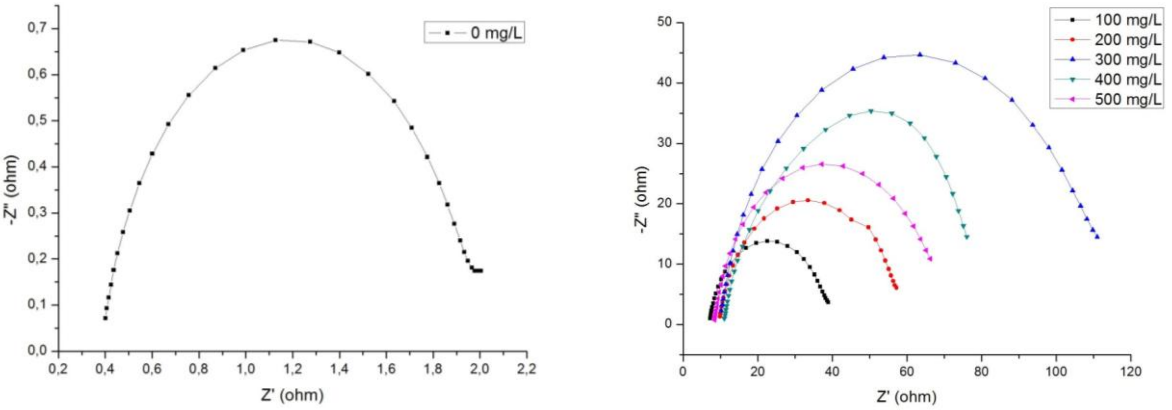
$$f\,(-Z_{imax}) = \frac{1}{2\pi C_{dl} R_{ct}} \tag{3}$$

Table 2 Parameters obtained from *Electrochemical Impedance Spectroscopy* measurements

Inhibitor concentration (mg/L)	R_s (Ω cm^2)	R_{ct} (Ω cm^2)	C_{dl} (µF/cm^2)	IE%
0	0.4	1.605	0.147023	-
100	7.256	31.679	0.000364	18.74
200	9.909	47.909	0.000162	28.86
300	10.251	105.394	0.000034	64.68
400	11.102	69.603	0.000065	42.38
500	8.452	62.122	0.000097	37.71

As can be seen in Table 2, the presence of inhibitors in acid media caused increased R_{ct} and decreased C_{dl}. The decrease of C_{dl} was associated with the decrease of dielectric local constants and/or the increase in the thickness of the double layers (Santana et al., 2012). This shows that *sarang semut* extract acted through adsorption on metal- solution interface. The presence of inhibitor adsorption on the surface of the electrode could reduce the electric capacity where the inhibitor molecules replaced water molecules and other ions were initially adsorbed on the surface of the steel (Rajalakshmi et al., 2008). Meanwhile the increase in the value of R_{ct} shows that the number of inhibitor molecules adsorbed on the surface of the steel increased and was absorbed to form a protective layer on the electrode surface; consequently. This becomes a barrier to impede the mass and charge transfer and increase the inhibition efficiency. In addition, the impedance spectrum obtained consisted of a capacitive loop indicating the presence of adsorption which occurred between the inhibitor and the steel's surface and *sarang semut* extract. This acted as the primary interface inhibitor (Li et al., 2009a).

The effect of varying concentrations of *sarang semut* extracts in inhibitor solution was also studied using EIS. The semicircles of the Nyquist plots (Figure 2) indicated the characteristic of solid electrodes. Which was attributed to the roughness and inhomogeneity of the solid surface (Hussin et al., 2011; Satapathy et al., 2009; Quraishi et al., 2010; Soltani et al., 2012). The value of R_{ct} increased gradually with various concentrations of *sarang semut* extracts (100–300 mg/L) in the inhibitor. This value is related to the formation of a protective layer on the mild steel surface. A higher value indicates stronger formation and an evenly distributed protective layer at the metal–solution interface (Awad et al., 2006). Unfortunately. the value of R_{ct} decreased with increasing concentrations of *sarang semut* extracts (400–500 mg/L). This was probably because the protective layer dissolved soon after adding the concentrations of *sarang semut* extracts in 1 M H_2SO_4.



(a) absence inhibitor (b) presence inhibitor

Figure 2 Impedance diagram obtained from the potential corrosion of the various concentrations of *sarang semut*

The Nyquist plots of mild steel at the various concentrations of *sarang semut* extracts in the inhibitor exhibited a similar pattern. This indicates that the inhibition mechanism (a charge–transfer process between the inhibitor and the metal surface) was also similar (Al-Otaibi et al., 2012; Lahhit et al., 2011). From the Nyquist plot, it is clear that the size of the capacitive loop increased with the increasing concentration of *sarang semut* extract. That is the inhibition efficiency increased and the rate of steel corrosion in the acidic solution decreased. Maximum efficiency occurs with the addition of a 300 mg/L concentration. With the addition of 400 and 500 mg/L the inhibition efficiency decreased. Nyquist plot semicircles are usually obtained for the electrode/interface solution which is normally associated with the roughness surface of the electrode (Bouyanzer et al., 2006). The efficiency of protection confirms that *sarang semut* extract acts as an efficient inhibitor for mild steel to a solution of 1 M H_2SO_4. At concentrations 400 and 500 mg/L. inhibition efficiency decreased. Likely due to increased aggressive ions attacking the corrosive solution. Thus a thin layer of protection suffered from abrasion and this reduced the thickness of the protective layer.

The impedance parameters were obtained after performing a simulation on an equivalent circuit as shown in Figure 3. This was used for fitting the impedance data. Analysis of the impedance spectra was performed by means of a Randles circuit which is the most common equivalent circuit.

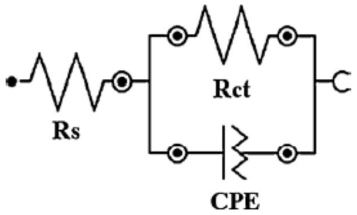


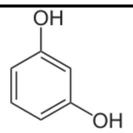
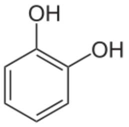
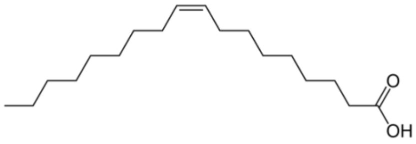
Figure 3 Equivalent electrical circuit model

Based on the EIS measurement, the highest efficiency of the use of *sarang semut* extract was revealed at a concentration of 300 mg/L in 1 M H_2SO_4, amounting to 64.68%. This result is similar to that of potentiodynamic polarization measurement. This shows that *sarang semut* carries out passive layer formation, thereby reducing the corrosion rate. If there was a difference in the values of the measurement results, it was due to different measurement times, which caused changes in the tool calibration such that the value was sometimes different.

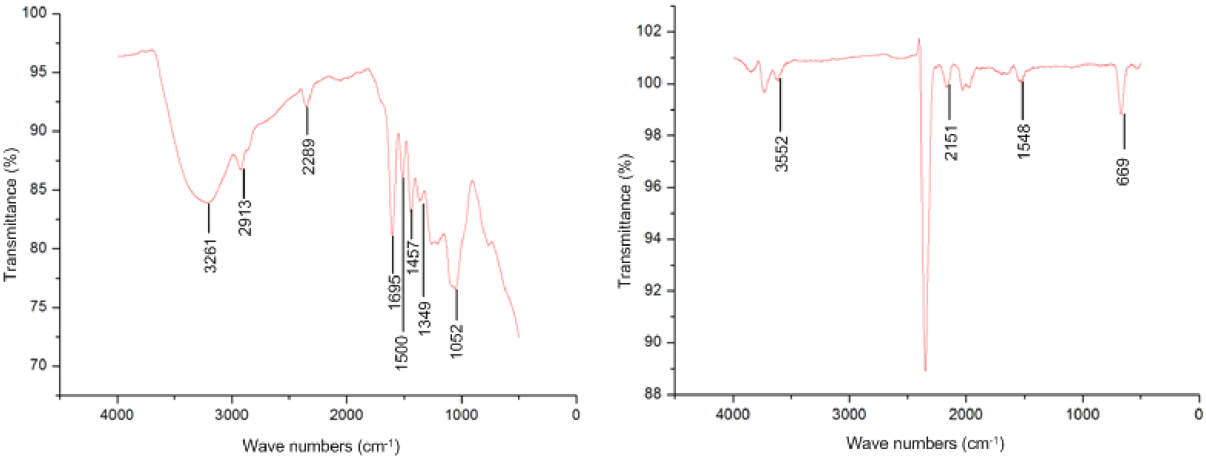
3.3. Characterization of *Sarang semut* Extracts

Table 3 shows the retention time (t_R) and identified compounds obtained from *sarang semut* extract. The analysis of GC-MS results for *sarang semut* extract shows some hydroxyl (-OH) compounds, which are usually found in natural materials.

Table 3 Retention time (t_R) of the main chromatogram peak in GC-MS measurement

No.	t_R (minutes)	Sum (%)	Compounds	Molecular structures
1.	26.894	67.176	1.3 Benzenediol	
2.	22.507	13.76	1.2 Benzenediol	
3.	48.750	5.733	9 Octadecenoic acid	
4.	45.502	5.039	Hexadecanoic acid	

These organic compounds have been used as organic corrosion inhibitors for metals. Benzenediol is one of the major compounds that was identified in *sarang semut* extract. In contrast, the adsorption of organic molecules may have been due to the presence of an oxygen atom (a heteroatom) π electron of aromatic rings or electron donating groups. In other words, heteroatoms such as oxygen are the major adsorption center in organic compounds due to their interaction with the metal surface. Adsorption can also occur via electrostatic interaction between a negatively charged surface which is provided by a specifically adsorbed anion (SO_4^{2-}) on iron and the positive charge of the inhibitor (Tebbji et al., 2005). Furthermore, according to the literature (Hu et al., 2015) an organic acid component can itself form a passive film on substrate surface. From the presented investigation, it can be deduced that the inhibitor molecules adsorbed on the metal surface by blocking the active corrosion sites (Rocca et al., 2004). This conclusion is supported by the polarization and impedance measurements which showed that the extract could inhibit the corrosion process by blocking it through the formation of a thin layer on the metal surface. Thus, the presence of *sarang semut* extract causes a potential shift toward more positive values indicating that the anodic reaction is inhibited. We can conclude that the decreasing oxidation rate of the substrate in corrosive media is due to the formation of a protective layer.



(a) FTIR spectra of Myrmecodia Pendans

(b) FTIR spectra of Myrmecodia Pendans + Metal + H_2SO_4 solution

Figure 4 Fourier transform infrared (FTIR) spectra

This research has been carried out through Fourier transform infrared (FTIR) testing to determine whether there is new bonding in the solution. We carried out FTIR testing to assess the possibility that bonding that occurs between metal surfaces and the inhibitor. The FTIR results showed that a new bonding occurs wherein Fe-O bonds were present in solution with the inhibitor added. This supports research done by Xianghong Li et al. (Li et al., 2009b) who found that the presence of Fe-O, Fe-N, and Fe-N-H bonds showed the formation of complex compounds of 6-benzylaminopurine extract with Fe^{2+} in the surface of the material as a thin layer of protection.

4. CONCLUSION

Myrmecodia pendans or *sarang semut* was successfully applied as a potential organic inhibitor in 1 M H_2SO_4 solution for mild steel specimens. In the presence of *sarang semut* extract, the E_{corr} values shifted to the positive voltage and acted as an anodic inhibitor. GC-MS showed that the *Myrmecodia pendans* extracts contained a high benzenediol compound with rich oxygen atom content playing an important role in the inhibition process. The 300 mg/L concentration of *Myrmecodia pendans* extract had the highest effect decreasing the rate of corrosion from 177.73 mpy to 47.4 mpy. This was attributed to the chemical compounds present in the *Myrmecodia pendans* extract on the surface of mild steel which prohibited a high corrosion rate.

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