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Article

Methanol Extract of *Melastoma malabathricum* Leaves as Eco-Friendly Corrosion Inhibitor for Mild Steel in Sulfuric Acid

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Abstract. Methanol extract of *Melastoma malabathricum* (MEMML) contains secondary metabolites such as phenolics and flavonoids. These compounds have OH groups that can interact with Fe contained in steel and form a thin layer for corrosion inhibition on mild steel. Inhibition of mild steel corrosion using MEMML leaves in sulfuric acid solution was carried out using weight loss and potentiodynamic polarization methods. The results showed that the mild steel corrosion rate decreased and inhibition efficiency increased with the addition of MEMML. FTIR and SEM analysis showed the interaction between mild steel with MEMML forming a layer on the surface of the steel so that it inhibits corrosion. The inhibition efficiency has increased with the length of immersion time and concentration of MML methanol extract. The highest inhibition efficiency, 98.86%, was obtained at 72 hours of immersion time and 10 g / L MEMML.

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1. Introduction

Steel is a metal that is widely used in industries such as the construction of tanks, pipes, and others. Steel is very easy to corrode especially with acid solutions. Acid is very aggressive in steel material. Acid solutions are often used for acid pickling, industrial acid cleaning, and acid descaling in steel construction. Corrosion is a natural process that cannot be prevented. Inhibitors can be used to reduce the rate of corrosion. In 2017, the U.S. reported that demand for corrosion inhibitors rose to 4.1% (USD$ 2.5 billion). Using synthetic corrosion inhibitors requires expensive costs and toxic especially inorganic inhibitors such as chromate, molybdate, etc [1-3]. Therefore, research on non-toxic or slightly toxic inhibitors and low cost are very important to study.

Lately, research on corrosion inhibitors of natural products has been studied more. Natural products are environmentally friendly and contain many secondary metabolites such as flavones, alkaloids, polyphenols, steroids, etc. These secondary metabolite compounds have O, N atoms and double bonds which interact very well with iron on the surface of the steel [2][4]. Some plant extracts have studied as corrosion inhibitors are avocado seed extract [2], cassava leaves extract [5-6], *Ficus tikoua* leaves extract [4], and Ginger extract [7], etc

The Melastomaceae family has been reported to have 142 compounds consisting of polyphenolic tannins, flavonoids, steroids, phenylpropanoids, organic acids (esters), terpenoids, and others [8-10]. So that, it has the potential to be developed as corrosion inhibitors. Dissotis theifolia contains tannins and flavonoids, belonging to the Melastomataceae family. D. theifolia which is extract can inhibit the corrosion of N80 steel in 1 M HCl and the inhibition efficiency increases with increasing concentration [11-12]. Another example of the Melastomaceae family is harendong. The corrosion inhibition efficiency of harendong extract (*Melastoma Affine D. Don*) at a concentration of 5% at 300 °C is 90.345% [13-14].

Melastoma malabathricum belongs to the melastoma genus and the melastomataceae family, is a herbaceous plant and is easily available. The leaves contain flavonoids, sterols, and tannins [15-16]. In terms of family, genus and secondary metabolite compounds from *Melastoma malabathricum*, it is thought that it can be used as a corrosion inhibitor.Therefore, corrosion inhibitors from methanol extracts of *Melastoma malabathricum* are studied for their ability to inhibit corrosion on mild steel in sulfuric acid solution.

2. Experimental Section

2.1. Materials

The materials used are mild steel (Fe = 98.5%, C = 0.19%, Si = 0.22% and Mn = 0.654%, etc), *Melastoma malabathricum* leaves, H_2SO_4 p.a (Merck), impure methanol by distillation, impure acetone by distillation , aquadest, Mayer, Dragendorff and Burchard reagent, $FeCl_3$ (Merck), Mg powder (Merck), HCl p.a (Merck), ethanol p.a (Merck), and NaOH (Merck).

2.2. Instrumentation

The instrumentation used rotary evaporator Shimadzu, hot plate, Fourier Transform Infrared (FTIR) Perkin Elmer System 2000, Scanning Electron Microscopy (SEM) Hitachi S3400N, and potentiodynamic polarization eDAQ.

2.3. Preparation Methanol Extract of *Melastoma Malabathricum* Leaves (MEMML)

MEMML are cut, air dried until brown for ± 7 days. The dried leaves are mashed using a blender then macerated with methanol for 72 hours in a dark colored bottle and protected from direct light. Then filtering was carried out using filter paper to obtain MEMML. The extract obtained was separated from the solvent using a rotary evaporator at a temperature of ± 50°C until a concentrated extract was obtained. The concentrated extract was then analyzed using the FTIR instrument and phytochemical screening was performed. The concentrated extract is stored in a beaker that is covered using aluminum foil before use.

2.4. Phytochemical Screening of MEMML

2.4.1. Alkaloid Test

1 mL MEMML was dissolved in 2 mL of dilute HCl then filtered and the filtrate was divided into two test tubes. The filtrate a is added 3 drops of Mayer's reagent (a solution of mercury in iodide). The occurrence of white deposits indicates the presence of alkaloid compounds. The filtrate b was added with 3 drops of Dragendorff reagent (potassium bismuth iodide solution). The occurrence of brick red deposits indicates the presence of alkaloid compounds.

2.4.2. Phenolic Test

1 mL MEMML was added with 3 drops of 1% $FeCl_3$. The formation of a blue-black color indicates the presence of phenolic compounds.

2.4.3. Flavonoid Test

1 mL MEMML was dissolved in 3 mL of distilled water and then brought to a boil and then filtered. The filtrate is added with ½ spatula of Mg powder, 1 mL concentrated HCl, and 2 mL ethanol. Shaken vigorously and left to separate. The formation of red, yellow, or orange colors in the ethanol layer indicates the presence of flavonoid compounds.

2.4.4. Quinone Test

1 mL MEMML was added with 3 drops 1 M NaOH. The formation of a red color indicates the presence of a quinone compound.

2.4.5. Saponin Test

1 mL MEMML was dissolved in 2 mL of distilled water then shaken for 1 minute and let it separate. The formation of a foam layer indicates the presence of saponin compounds

2.4.6. Steroid and Terpenoid Test

1 mL MEMML was dissolved in 2 mL chloroform and then filtered. Add 3 drops of anhydrous acetic acid to the filtrate and bring to a boil and cool again. The solution is added 3 drops of H_2SO_4. The formation of a brown ring indicates the presence of a steroid compound and a reddish brown ring indicates a terpenoid compound.

2.4.7. Tannin Test

1 mL of MEMML was added with 3 drops of 1% $FeCl_3$. The formation of a green-black color indicates the presence of phenolic compounds.

2.4.8. Preparation of MEMML Inhibitor Solution

MEMML leaves methanol extract weighed as much as 5 grams. The extract was then diluted in a 500 mL volumetric flask using 0.75 M H_2SO_4 solution to obtain an inhibitor solution with a concentration of 10 g/L. The 10 g/L inhibitor solution was diluted again so that 2, 4, 6, 8, and 10 g/L concentrations were obtained.

2.5. Immersion of Mild Steel in MEMML Inhibitor Solution

Mild steel is attached to a rope and hung on a toothpick in a 50 mL beaker glass containing an inhibitor solution with concentrations of 2, 4, 6, 8, and 10 g / L for 24, 48, and 72 hours, respectively. Steel that has been removed in accordance with the immersion time is washed using distilled water and acetone. Then the mass is measured using an analytical balance and the results are expressed as the final mass inhibitor (m_2). Then an analysis was carried out using FTIR and SEM instruments on steel with immersion treatment for 72 hours in a 10 g / L inhibitor solution.

2.6. Weight Loss Methode

The weight loss method is a method for measuring the corrosion rate and the efficiency of corrosion inhibition the corrosion rate can be calculated by the following equation [17] :

$$\text{“}C_R = \frac{W_b - W_a}{S.t}\text{”} \quad (1)$$

Where “C_R are corrosion rate, W_b dan W_a are the sample weight measured before and after soaking in a solution of corrosive. S is surface area and t is time in hour”.

The efficiency of mild steel corrosion inhibition is calculated using equation 2 [17]:

$$\text{“IE (\%)} = \frac{C_{R\,(blank)} - C_{(R\,inh)}}{C_{R(blank)}} \text{ x 100 \%”} \quad (2)$$

“$C_{R\ (blank)}$ and $C_{R(Inh)}$ indicate corrosion rate the absence and presence of MEMML in the sulfuric acid solution”

2.7. Potentiodynamic Polarization Methods

Mild steel measuring ± 3 × 1 cm is cut round lengthwise then smoothed using np iron sandpaper 120. Put in a small pipe of the same size. Then the resin is applied to one end. After that, an analysis using a potentiostat with immersion treatment of 0.75 M H_2SO_4 corrosive medium and inhibitor solution with concentrations of 2, 4, 6, 8, and 10 g/L.

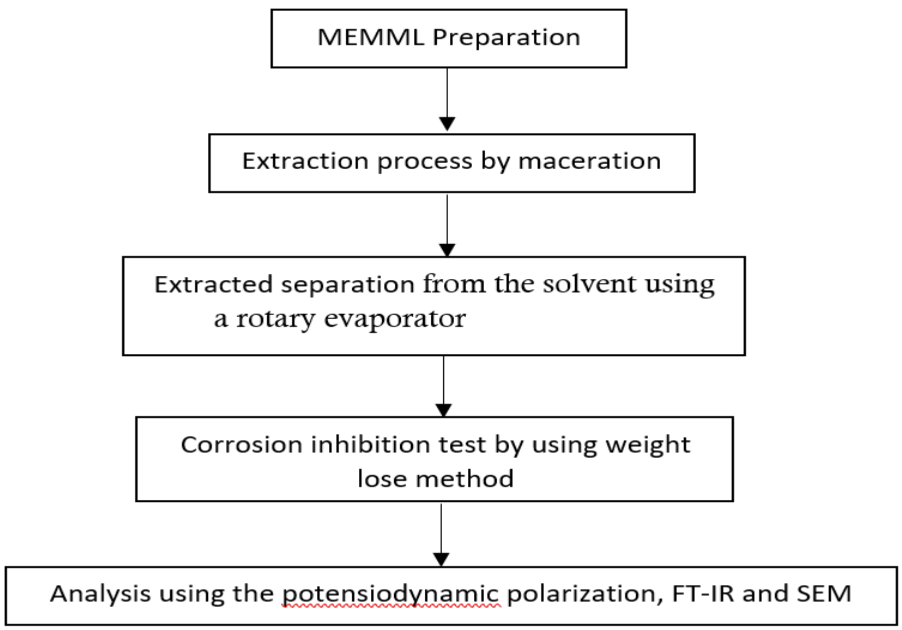

Figure 1. Research flow chart

3. Results and Discussion

3.1. Phytochemical Screening of MEMML

Phytochemical screening is carried out to identify groups of secondary metabolites found in a plant. Table 1 shows that secondary metabolite compounds contained in the MEMML leaves are phenolic, flavonoid, quinone, steroid, and tannin. These secondary metabolites have hydroxyl groups that interact with iron on the steel surface.

Table 1. Phytochemical screening results of MEMML

Secondary Metabolite	Reagent	Identified
Alkaloids	Mayer/Dragendorff	-/-
Phenolic	$FeCl_3$ 1%	+
Flavonoid	Mg, HCl, dan ethanol	+
Quinone	NaOH 1 N	+
Saponin	Aquadest	-
Steroid	Burchard	+
Tannin	$FeCl_3$ 1%	+

(+) = identified (-) = unidentified

3.2. Fourier Transform Infrared (FTIR) Spectrophotometric Analysis

Figure 2a shows the absorption of the -OH group at the wave number 3335.78 cm^{-1} and the aliphatic -CH at the wave number 2928.54 cm^{-1}. Socrates (1994) reports that the presence of aliphatic CH- absorption gives a clue to the possibility of the presence of methyl (CH_3) and methylene (CH_2) groups. This assumption is reinforced by the absorption bands of C = C and C-O at numbers 1442.58 and 1256.7 cm^{-1}. There is also the absorption of C = O groups at wave numbers 1960.01 cm^{-1} and aromatic C-H at wave numbers 650.88 cm^{-1}.

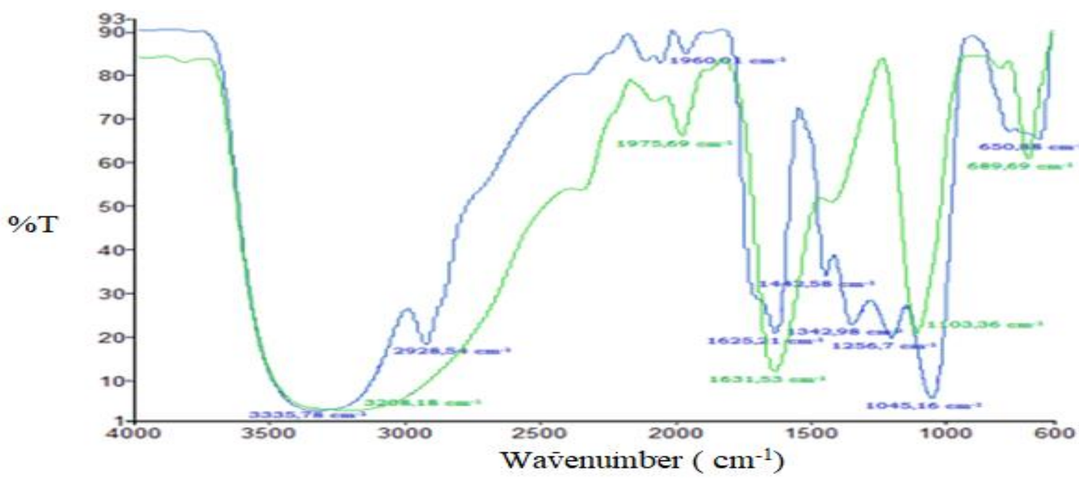

Figure 2. FTIR Spectrum (a) MEMML (blue line) and (b) Film Formed on surface mild steel after immertion with MEMML in sulfuric acid (green line)

The presence of OH, C = O, C = C, and CO absorption strengthens the presence of secondary metabolite compounds in Table 1. The blue line (2a) represents MEMML and the green line (2b) represents the layer formed on the steel surface after immersion of MEMML in sulfuric acid depicts different wavenumbers and patterns. The surface layer on mild steel after immersion for three days in H_2SO_4 solution containing MEMML showed a shift in the peak of the OH functional group from 3335.78 cm^{-1} to 3208,18 cm^{-1}, the peak of the C = O functional group from 1960.01 cm^{-1} to 1975,69 cm^{-1}, and the peak of the functional group. C-O from single OH from 1256,7 cm^{-1} to 1103.36 cm^{-1} The shift in wave numbers shows the occurrence of physical and chemical interactions between MEMML which has OH, C = O, and C-O functional groups from a single OH with Fe^{+2} on the surface of mild steel [18-19].

3.3. Scanning Electron Microscopy (SEM) Analysis

Figure 3a shows the surface of mild steel which is still clean, smooth, and there are no holes due to corrosion. The visible lines are the result of the preparation using sandpaper. Figure 3b shows a rough, uneven, soft steel surface with holes caused by corrosion by sulfuric acid. Figure 3c shows the smooth surface of mild steel compared to Figure 3a and Figure 3c. It happened because the secondary metabolite compounds contained MEMML were adsorbed on the surface of soft steel to form a thin layer and protect it from corrosion [20-21].

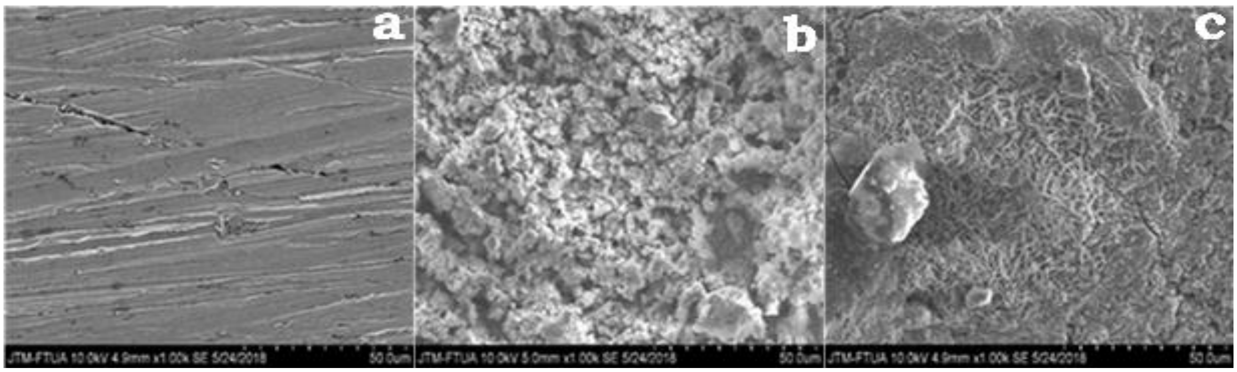

Figure 3. SEM results of soft steel: a) blank; b) immersion in 0.75 M sulfuric acid for 72 hours; c) immersion in a methanol extract inhibitor of *M. malabathricum* leaves 10 g/L for 72 hours

Effect of the concentration of MEMML on the rate of corrosion and efficiency of mild steel inhibition decreasing of corrosion rate and increasing of inhibition efficiency in figure 3 prove MEMML works well in protecting mild steel against corrosion. The secondary metabolites found in MEMML contain such as phenolics, tannins, and flavonoids (Table 1) which have many OH groups interacted with iron (Fe) on the mild steel surface and form a thin layer. The layer can inhibit the attack of H^+ ions on the steel surface, thus slowing the corrosion reaction [22-23].

In addition MEMML, contains secondary metabolites such as phenolics, tannins, and flavonoids can also react with corrosive media and reduce the concentration of aggressive ions in solution, thereby slowing the occurrence of corrosion reactions in steel. Decreasing of corrosion rate in mild steel has an impact on increasing the efficiency of corrosion inhibition. The lower the corrosion rate, the higher the corrosion inhibition efficiency. In this study, the highest corrosion inhibition efficiency of 98.86% was obtained in the immersion treatment in a solution of MEMML 10 g/L

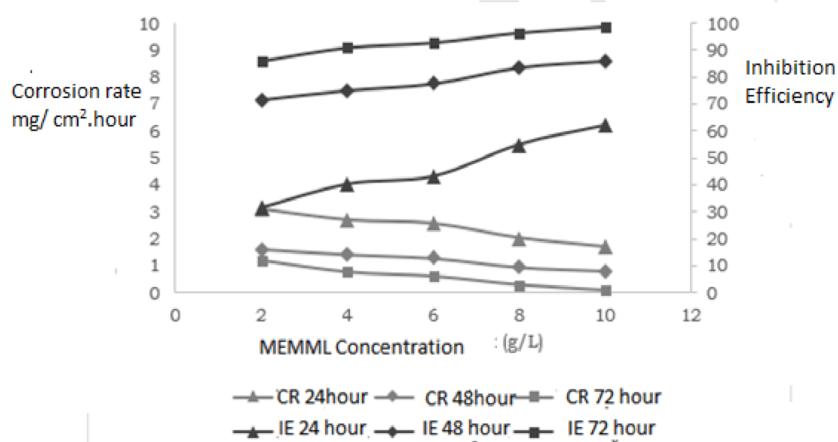

Figure 4. The effect of the concentration of MEMML on the corrosion rate and the corrosion inhibition efficiency of soft steel during 24, 48, and 72-hour immersion

3.4. Effect of Soaking Time in a Solution of MEMML Inhibitor on Corrosion Rate and Mild Steel Inhibition Efficiency

Figure 5 shows the immersion time affecting the rate of corrosion. Corrosion rate decreases with increasing immersion time [24-25]. Secondary metabolites such as phenolics, tannins, and flavonoids in MEMML has more time to interact with mild steel, so the protective layer that forms on the surface of the steel is wider and the corrosion reaction is inhibited.

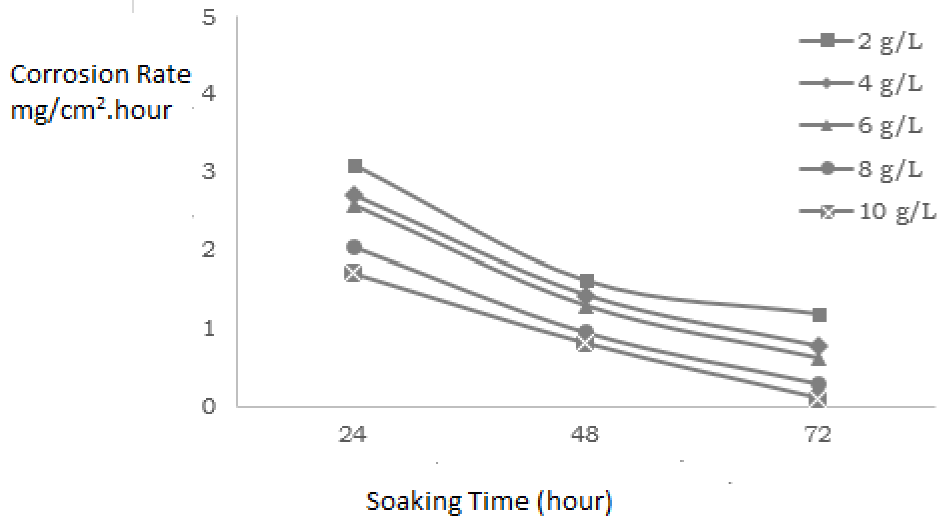

Figure 5. Effect of immersion time on the rate of corrosion of steel in the methanol extract inhibitor solution of MEMML 2, 4, 6, 8, and 10 g / L

3.5. Potentiodynamic Polarization Methods

Figure 6 shows a shift in the cathode and anode curves. The shift in the value of Ecorr towards more negatively than the blank is called a cathodic inhibitor in which the inhibitor inhibits the hydrogen evolution reaction. Whereas the Ecorr value which shifts towards more positively than blank is called anodic inhibitor wherein the inhibitor inhibits the dissolution of the anode from iron [26-27]. If the Ecorr value is shifted in the positive and negative direction of the blank, it is called a mixed inhibitor.

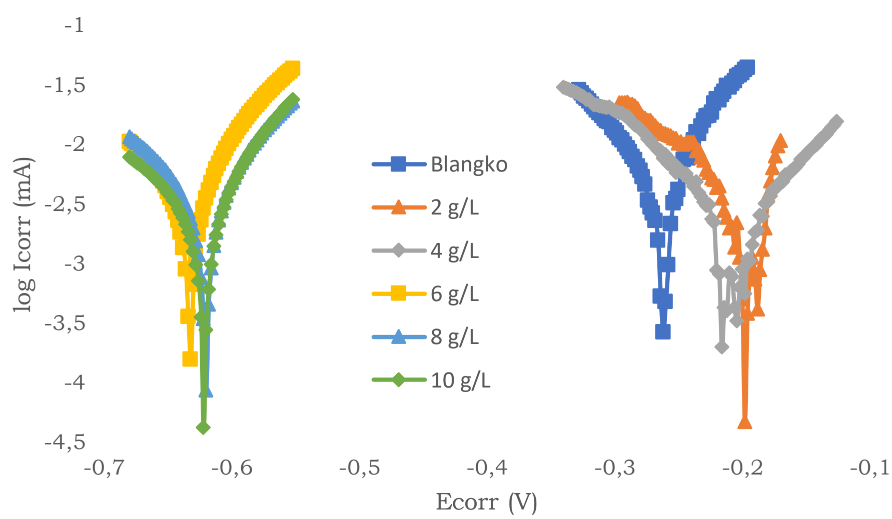

Figure 6. Mild steel potentiodynamic polarization curves in MEMML inhibitor solution

The treatment of mild steel using a solution of MEMML inhibitors with concentrations of 2 and 4 g / L causes the Ecorr value to shift in a positive direction from the blank, whereas for the use of an inhibitor solution with concentrations of 6, 8, and 10 g/L, the Ecorr value shift in the negative direction of the blank. This proves that the MEMML is a mixed inhibitor, but tends to be cathodic because 3 out of 5 treatments show the value of Ecorr is shifting towards the negative.

Anodic Tafel Constants (βa) and Cathodic Tafel Constants (βc) in Table 2 obtained using Equation 4 and Equation 5 show changes after the addition of MEMML [28-29]. It indicates that the MEMML reduces iron dissolution at the anode and inhibits the hydrogen evolution reaction at the cathode [30]. Small changes of βa and βc (<1) indicate that the corrosion mechanism does not change with the addition of MEMML [30-31]. The efficiency of inhibition using a 10 g / L inhibitor solution in the potentiodynamic polarization method is 80.047%, while the weight-loss method is 98.864%. The difference in the value of inhibition efficiency can occur because the two methods use different immersion times [32].

Table 2. Potentiodynamic polarization data of mild steel in solutionof MEMML inhibitor

Concentration (g/L)	I_{corr} (mA)	β_c (V/mA)	β_a (V/mA)	IE (%)
0	0.005	0.08	0.038	-
2	0.004	0.10	0.667	20.567
4	0.002	0.115	0.073	55.332
6	0.002	0.057	0.05	60.189
8	0.002	0.052	0.052	68-337
10	0.001	0.045	0.056	80.047

4. Conclusion

The methanol extract of *M. malabathricum* leaves (MEMML) can inhibit the rate of corrosion of mild steel in sulfuric acid medium at 98.86% at a concentration of 10 g / L and soaking for 72 hours. SEM analysis on mild steel without treatment shows mild steel surfaces that are still clean, smooth, and there are no holes due to corrosion. Soft steel after immersion in H_2SO_4 corrosive medium shows a rough, uneven surface, and there are holes due to corrosion. Whereas soft steel after immersion in an M. malabathricum corrosion inhibitor solution shows a smoother surface compared to mild steel immersed in a corrosive medium solution. The MEMML which is used as mild steel corrosion inhibitor in H_2SO_4 corrosive medium is a mixed type inhibitor because it can inhibit iron dissolution at the anode and hydrogen evolution reaction at the cathode.

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