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Article

Use of Nanoemulsion-Based Merkubung (*Macaranga gigantea*) Sap Extract Inhibitor to Improve Corrosion Inhibition Efficiency on Steel

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Abstract. Corrosion frequently affects steel. Although it is an unavoidable natural process, its progression can be managed and its rate reduced. One strategy for mitigating corrosion is the use of corrosion inhibitors. Inhibitors derived from organic sources are widely employed because of their high inhibition efficiency. Sap extract from Merkubung *(Macaranga gigantea)* can function as a corrosion inhibitor due to the presence of secondary metabolites such as tannins. Tannins are promising corrosion inhibitors since their –OH groups enable them to form complexes with metal surfaces. This study evaluates the performance of a nanoemulsion containing Merkubung sap extract as a steel corrosion inhibitor. The methodology includes extraction, nanoemulsion formulation, and assessment of corrosion rate and inhibition efficiency at various concentrations and temperatures. In this work, nanoemulsion-based inhibitors were applied to improve corrosion protection of steel. The nanoemulsion demonstrated the lowest corrosion rate (0.902 mg cm^{-2} h^{-1}) and the highest inhibition efficiency (66.05 %) at 500 ppm and 30 °C. These results show that the nanoemulsion enhances inhibition performance, making it an effective corrosion inhibitor.

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1. Introduction

The use of steel for infrastructure needs in Indonesia continues to increase. Steel is usually used as the main building material in the manufacture of houses, bridges, towers, tools, and so on, because of its good mechanical properties but high susceptibility to corrosion in humid and aggressive environments [1-2]. Well-maintained steel will have a very long service life. However, the presence of environmental factors makes the quality of steel decrease, so that steel, which is expected to have a long usage time, turns out to have a shorter life than the average service life, globally, corrosion is reported to significantly reduce the durability and safety of steel-based structures and cause large economic losses [3-5]. Steel that is often exposed to water and air for a long time will experience corrosion [2-4],[6-9].

Corrosion can be detrimental, so it needs special attention due to the effects it causes, including structural failure and increased maintenance costs [3-5]. Because corrosion is a natural process, the process cannot be prevented; what can be done is to control and reduce the rate of corrosion through various strategies such as coatings, cathodic protection, material selection, and especially the use of corrosion inhibitors [1],[4-5]. One method to inhibit the corrosion rate process is by using corrosion inhibitors. A corrosion inhibitor is a chemical substance that, when added to an environment, can minimize the rate of corrosion that occurs in that environment against a metal in it [4-6],[10]. Recently, corrosion inhibitors from organic materials have been widely used. This is because the efficiency of the inhibitor is very good; it has non-toxic properties, is cheap, easy to obtain, and can be renewed [10-12].

Many organic corrosion inhibitors are derived from natural materials. Several uses of natural extracts have been proven to be effective as corrosion inhibitors [13-14]. Secondary metabolite compounds in plants, such as tannins, can function as anticorrosive substances [15-17]. Tannin compounds in plant extracts can form complex compounds with iron on the metal surface, so that the rate of corrosion reactions can decrease [18]. Natural material extract compounds that are used as inhibitors must contain N, O, P, and S atoms, and atoms that have free electron pairs that can act as ligands to form complex compounds with metals. This complex compound will block the attack of corrosive ions on the metal surface. In addition, tannins contain polyphenolic compounds that can inhibit the oxidation process so that the corrosion rate can decrease [19]. Tannin compounds can be obtained from several parts of plants, such as stems, leaves, skin, and even sap. Taking tannin compounds is done by extracting the plant part so that the substances contained in it can come out.

Merkubung (Macaranga gigantae) is a plant whose distribution is widely found in tropical rainy areas, including Indonesia. Merkubung sap extract, there are secondary metabolite compounds such as alkaloids, phenolics, flavonoids, quinones, saponins, steroids, tannins, and terpenoids, similar to many plant extracts that have shown high inhibition efficiency on mild steel in acidic media [20-21]. Tannins have great potential as corrosion inhibitors, this is because the presence of the -OH functional group in tannins can form complexes with metals so that they coat the steel surface, which has the potential to inhibit corrosion in soft steel [22-24]

The utilization of nano-sized technology can increase the efficiency of active ingredients despite their small size. Nanoemulsion can enhance the corrosion inhibiting effect with good adsorptivity. Nanoemulsion can be used to enhance the corrosion inhibiting effect. His research on nanoemulsions of plant extracts showed that large corrosion efficiency can be achieved in the presence of inhibitors even in small amounts. The purpose of this research is to determine the effect of immersion temperature on the efficiency and corrosion rate of steel coated with nanoemulsion-based merkubung sap extract inhibitor and the effectiveness of nanoemulsion-based merkubung extract inhibitor in slowing down the corrosion rate of steel [25-27].

2. Research Methods

2.1. Materials

The tools used in this research are reflux equipment, waterbath, blender, beaker, thermometer, dropper, test tube, measuring cup, measuring flask, magnetic stirrer, ultrasonicator, centrifuge, Particle Size Analyzer (PSA), analytical balance and sandpaper. The materials used in this research are merkubung bark, distilled water, Mayer reagent, Dragendorff reagent, concentrated HCl, $FeCl_3$ 1%, NaOH 1M, H_2SO_4 0.75M, tween 80, Virgin Coconut Oil (VCO), pH 7 buffer, 96% ethanol, and steel chips.

2.2. Procedure

2.2.1 Extraction of Merkubung Sap

The bark of the merkubung tree is peeled and cleaned. The bark of the merkubung tree is peeled and cleaned from the outer skin, then blended and squeezed. The juice was collected in a beaker. A total of 100 mL of merkubung sap was put into a round flask, and distilled water was added as much as 3-4 times the weight of merkubung sap. The mixture was refluxed for 3 hours at 70-80°C. The mixture was then filtered and the filtrate was taken. Furthermore, the filtrate is evaporated using a waterbath at 70 °C until concentrated tannin is obtained.

2.2.2 Phytochemical Screening of Merkubung Sap Extract

Phytochemical screening refers to Warni et al (2022), which includes;

2.2.2.1 Alkaloid test

The alkaloid test was carried out with 0.5 grams of extract homogenized in 2 mL of dilute HCl then filtered and the filtrate was divided into two parts. Filtrate I then added 3 drops of Mayer reagent. Positive results are characterized by a white precipitate. Then, filtrate II was added 3 drops of Dragendorff reagent. Positive results are characterized by a brick red precipitate.

2.2.2.2 Flavonoid Test

The alkaloid test was carried out with 0.5 grams of extract dissolved in 3 mL of distilled water and then brought to a boil and filtered. Filtrate added ½ sudip powder Mg, 1 mL concentrated HCl and 2 mL ethanol. Shaken vigorously and allowed to separate. Positive results are characterized by red, yellow or orange color.

2.2.2.3 Phenolic Test

Alkaloid test is done with 0.5 grams of extract added 3 drops of FeCl 1%. Positive results are characterized by the formation of blue-black.

2.2.2.4 Quinone Test

Alkaloid test is done with 0.5 grams of extract added 3 drops of NaOH 1M. positive results are characterized by the formation of red color.

2.2.2.5 Saponin Test

Alkaloid test is done with 0.5 grams of extract dissolved in 2 mL of distilled water then shaken for 1 minute and allowed to separate. Positive results are characterized by the formation of a foam layer.

2.2.2.6Tannin Test

Tannin test is done with 0.5 grams of extract added FeCl 1%. Positive results are characterized by the formation of bluish green.

2.2.2.7 Terpenoid Test
The terpenoid test was carried out by reacting 0.5 grams of extract with 10 drops of H_2SO_4 through the tube wall. Positive results were indicated by the formation of green and blue colors.

2.2.3 Nanoemulsion Preparation of Merkubung Sap Extract
The merkubung sap extract was dissolved in the oil phase, namely virgin coconut oil (VCO). Tween 80 surfactant and 96% ethanol cosurfactant were added and homogenized using a homogenizer for 1 hour. The nanoemulsion was then ultrasonicated for 1 hour and mixed with pH 7 buffered aqueous phase in a ratio of 1:5.

2.2.4 Analysis of Merkubung Sap Extract Nanoemulsion
Tests were carried out on the results of the nanoemulsion of merkubung gum extract: droplet size test with Particle Size Analyzer (PSA). 1 mL of merkubung gum extract nanoemulsion was diluted with aqua pro injection as much as 250 mL. The diluted nanoemulsion sample was placed in a cuvette, then the particle size was measured. Physical stability test of nanoemulsion was conducted by centrifuging the sample at 4700 rpm for 15 minutes. A stable nanoemulsion showed no separation of the two phases. Solubility test using nanoemulsion with solvents (1:1) namely distilled water, ethanol, hexane and ethyl acetate.

2.2.5 Preparation of Steel Samples
Steel chips were sanded, washed with distilled water and acetone and dried. Then the initial mass of steel (Wo) was weighed.

2.2.6 Steel Immersion
The acid medium was prepared by dissolving the nanoemulsion of merkubung gum extract into 0.75 M sulfuric acid solution and made concentrations of 100; 200; 300; 400; 500 ppm. Steel chips were put into the acid medium with temperature variations of 30°C, 40°C, and 50°C for 3 hours each. Afterwards, the steel chips were dried and the final mass was weighed.

2.2.7 Analisa Laju Korosi
Corrosion rate analysis is carried out after the immersion time is reached. Then the corrosion rate measurement can be calculated using the following equation:

$$CR = \frac{Wo - Wf}{A \times t}$$

2.2.8 Efisiensi Inhibitor
Inhibitor efficiency shows the percentage decrease in corrosion rate due to the addition of inhibitors. The efficiency is calculated using the equation:

$$E = \frac{(X_a - X_b)}{X_a} \times 100\%$$

Where E is the inhibitor efficiency (%), Xa is the average metal mass loss without inhibitor (gr), and Xb is the average metal mass loss with inhibitor.

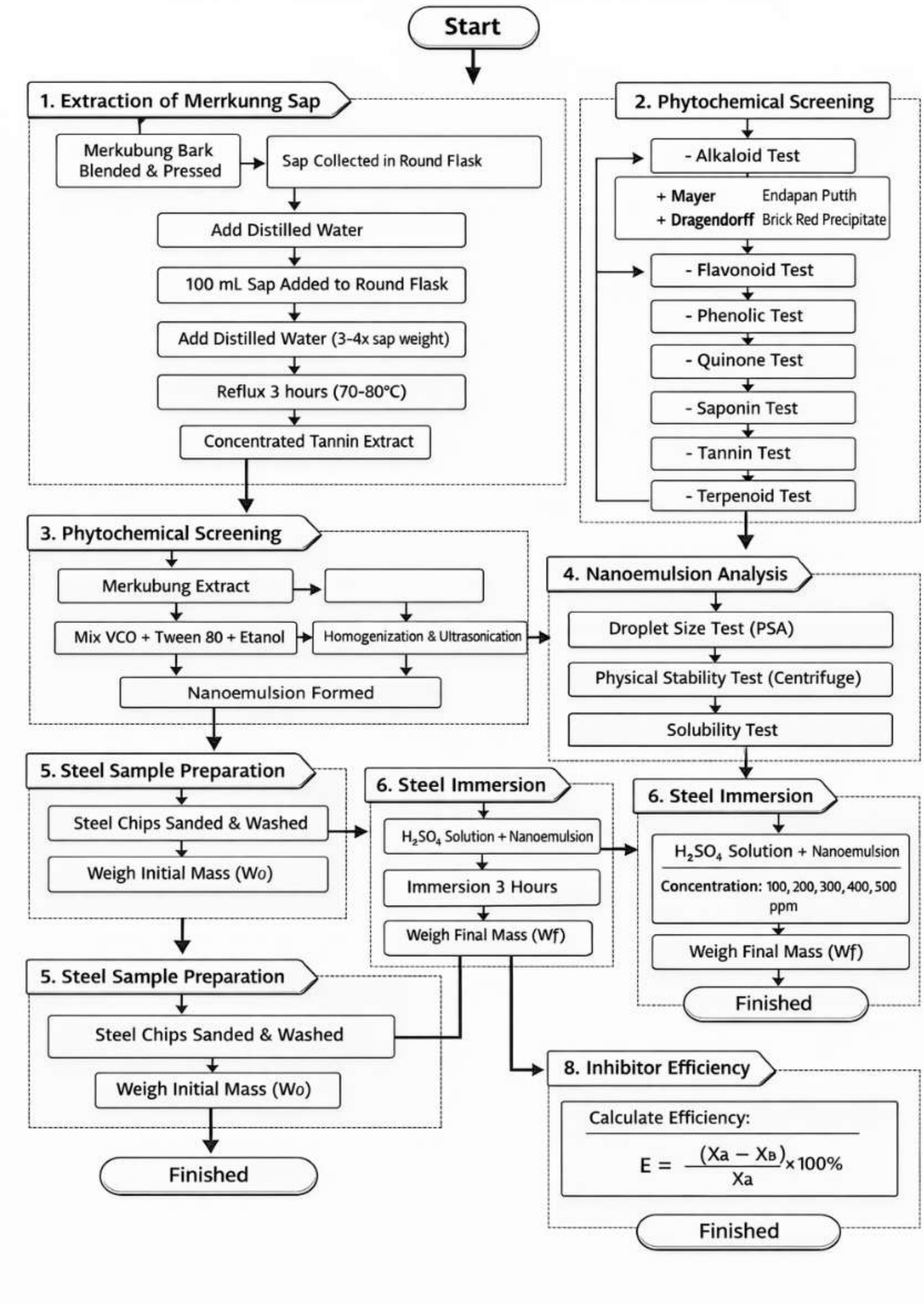


Figure 1. Flowchart Research Methods

3. Results and Discussion

3.1 Phytochemical Screening of Merkubung Sap Extract

Phytochemical screening aims to identify the class of secondary metabolite compounds contained in merkubung sap extract. The results of the phytochemical test of merkubung sap extract are shown in Table 1 below:

Table 1. Phytochemical screening results of Merkubung sap extract

Secondary Metabolites	Reagents	Result
Alkaloids	Mayer/Drgendorff	-/-
Flavonoids	Mg, HCl, ethanol	+
Phenolics	$FeCl_3$ 1%	+
Quinones	NaOH 1 M	+
Saponins	Aquades	+
Tannins	$FeCl_3$ 1%	+
Alkaloids	H_2SO_4	-

Description: (+) contains secondary metabolite compounds, (-) does not contain secondary metabolite compounds.

Table 1 shows the positive results of Merkubung sap extract, which contains secondary metabolite compounds such as flavonoids, phenolics, quinones, saponins and tannins. While the negative results are alkaloid and terpenoid compounds. Secondary metabolite compounds contained in merkubung sap extract contain O atoms and double bonds. Compounds containing O, N, or S heteroatoms that have free electron pairs and double bonds can be used effectively as corrosion inhibitors. This is because these compounds can be adsorbed to form a thin layer on the surface of soft steel as a protection from corrosive environments [28-30].

3.2 Nanoemulsion Analysis of Merkubung Sap Extract

3.2.1 Droplet size test with Particle Size Analyzer (PSA)

The nanoemulsion of merkubung sap extract is a type of oil-in-water emulsion. In general, oil-in-water emulsions have an average droplet size of 1-100 nm. The type of nanoemulsion depends on the composition of the ingredients used. Oil-in-water nanoemulsions are oil droplets dispersed in an oil phase. In this research, the dispersed phase is the oil phase (virgin coconut oil (VCO)) and the dispersing phase is the water phase (buffer pH 7). The dispersed phase greatly affects the droplet size and stability of the nanoemulsion formed. VCO, as the oil phase contains Medium Chain Triglyceride (MCT) so that it can produce a stable and clear nanoemulsion. The ability of the oil phase to form droplets in the dispersion medium is aided by surfactants and cosurfactants [26],[31].

The use of 96% ethanol acts as a cosurfactant that functions to reduce surface tension so that a more stable nanoemulsion is obtained. While tween 80 acts as a nonionic surfactant. The ability of surfactants and cosurfactants to reduce the emulsion surface tension between the oil phase and the water phase affects the phase separation in nanoemulsion preparations. As the ability of surfactants and cosurfactants to reduce the interfacial tension increases, a more stable nanoemulsion will be formed. Table 3 shows that the physical stability of the nanoemulsion of merkubukung gum extract is stable. A good emulsion can maintain its particle globules when given the gravity of centrifugation, but no separation between the two phases occurs [32-33].

Table 2. Stabilization and Particle Size of Nanoemulsion

Sample	Physical Stability	Particle Size	IP
Merkubung sap extract	Stable	16.8 nm	0.387

Table 2 shows the results that the nanoemulsion of merkubung extract has met the nanoemulsion droplet size requirement of ≤ 100 nm. In this study, the particle size of nanoemulsion of merkubung gum extract was 16.8 nm with a polydispersity index value of 0.387. A polydispersity index value of <0.5 indicates a uniform particle size distribution. The PI value that is closer to zero, the more the particle distribution shows the homogeneity of the nanoemulsion preparation produced [34].

3.2.2 Solubility test

The solubility of the nanoemulsion of merkubung sap extract is shown in Table 3. The solubility test aims to compare the solubility of the nanoemulsion and the extract. The solubility of a substance is based on the principle of like dissolves like or polarity between solvent and solute. Polar substances will dissolve in polar solvents, while nonpolar substances will dissolve in solvents that are nonpolar as well. In Table 2, the solubility of nanoemulsion and Merkubung gum extract showed the same results, namely soluble in polar solvents (aquades and ethanol) and insoluble in non-polar solvents (hexane and ethyl acetate). This shows that there is no change in the properties of the merkubung sap extract even though it was converted into a nanoemulsion. The solubility of nanoemulsion is also influenced by its small particle size. The size of nanoemulsion that is less than 100 nm has a large surface area, so the level of solubility is better [32],[34-35].

Table 3. Solubility test results of nanoemulsion of Merkubung gum extract.

Solvent	Solubility of Extract	Solubility of Nanoemulsi
Aquades	+	+
Ethanol	+	+
Hexane	-	-
Ethyl Acetate	-	-

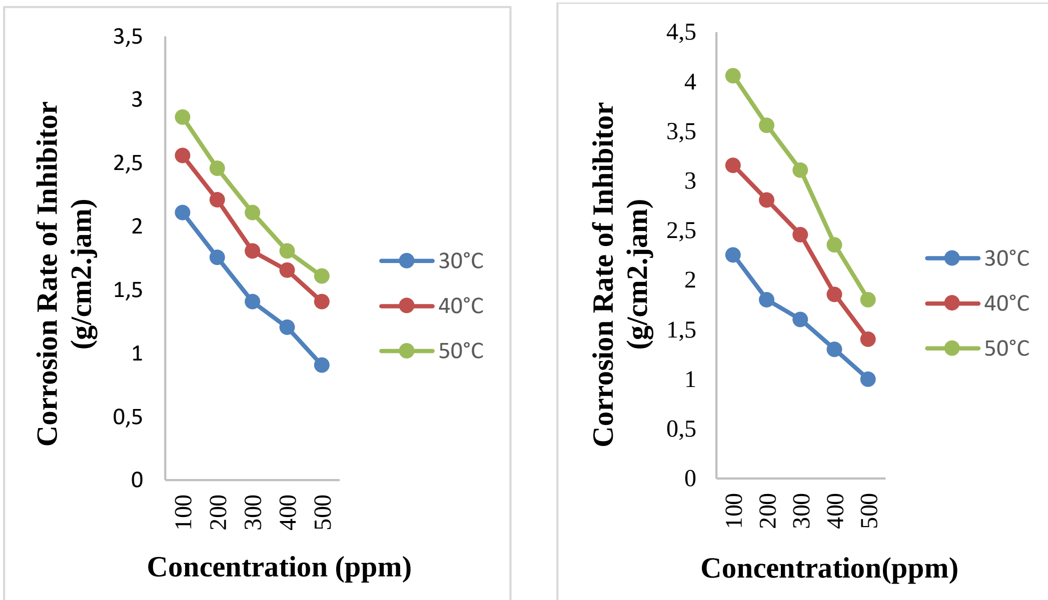
Description: (+) Soluble, (-) Insoluble

3.3 Analysis of Corrosion Rate and Inhibition Efficiency of Steel

3.3.1 Relationship between Temperature and Concentration Variations on Corrosion Rate

Figure 2 shows that the concentration is inversely proportional to the corrosion rate. The greater the concentration of corrosion inhibitor, the smaller the corrosion rate. This is because the more inhibitor is added, the more merkubung gum extract is adsorbed on the surface of the steel plate. The formed layer will be able to inhibit the corrosion rate on the steel surface. However, the corrosion rate value is directly proportional to the temperature. The higher the temperature, the higher the corrosion rate. The increasing temperature causes the particles that react at kinetic energy to increase so that the magnitude of the activation energy value is exceeded, resulting in the corrosion rate also getting faster [36-38].

Figure 1(a) shows the corrosion rate of steel with merkubung sap extract nanoemulsion inhibitor. The smallest corrosion rate value is 0.902 mg/cm^{2}h at a concentration of 500 ppm with an immersion temperature of 30°C. While Figure 1(b) shows the corrosion rate of steel with merkubung sap extract inhibitor, with the smallest corrosion rate value of 1.002 mg/cm^{2}h at a concentration of 500 ppm, with an immersion temperature of 30°C. The corrosion rate of steel using nanoemulsion inhibitor has a smaller value than that of mercurial gum extract. This is because nanoemulsions have high kinetic stability and a smaller droplet size. The smaller droplet size causes a larger surface area so that the nanoemulsion adsorption process on steel is faster and greater.



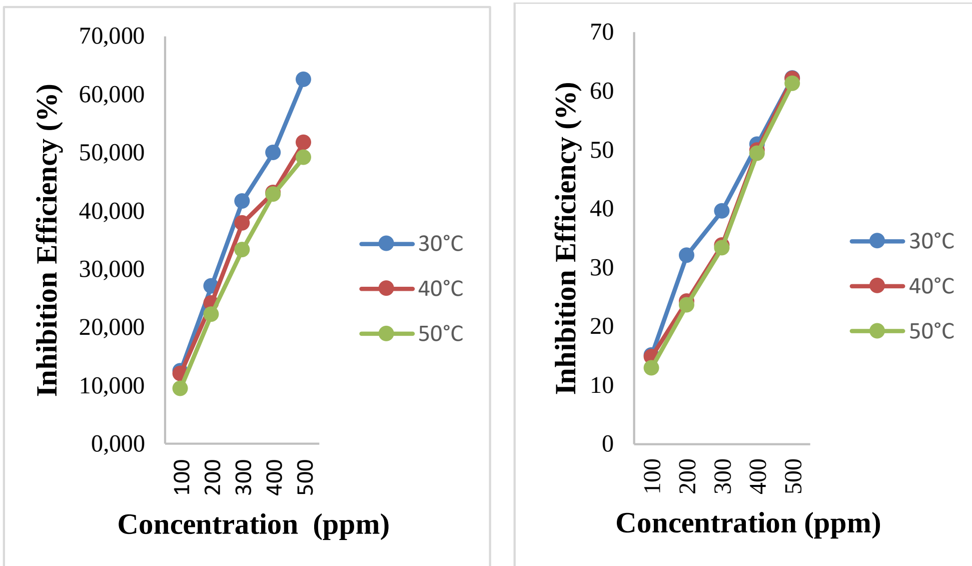
(a) (b)

Figure 2. Graph of the effect of temperature and inhibitor concentration on corrosion rate using: (a). merkubung extract nanoemulsion inhibitor; (b). merkubung sap extract inhibitor

3.3.2 Temperature and Concentration Relationship to Inhibition Efficiency

Figure 3 shows that the corrosion inhibition efficiency value increases with increasing inhibitor concentration, but decreases with increasing temperature. The increase in corrosion inhibition efficiency is due to the presence of an inhibitor layer attached to the steel surface [37,38]. The more inhibitor adsorbed on the steel surface results in inhibition of mass and charge transfer thus preventing further corrosion [11],[39-40].

In addition, the presence of tannin compounds in the extract can act as ligands in forming complex compounds with Fe(III), so that the corrosion rate will decrease. The value of the corrosion rate with the inhibition efficiency value is inversely proportional, the more the corrosion rate decreases, the more the inhibition efficiency value increases. Figure 3(a) shows the inhibition efficiency value using nanoemulsion inhibitor of merkubung sap extract has the largest efficiency value of 66.05% at a concentration of 500 ppm with a temperature of 30°C. While Figure 3(b) shows the efficiency value with the largest merkubung sap extract inhibitor, namely 62.26% at a concentration of 500 ppm with a temperature of 30°C.



(a) (b)

Figure 3. Graph of the effect of temperature and inhibitor concentration on inhibition efficiency in immersion using: (a). merkubung extract nanoemulsion inhibitor; (b). merkubung sap extract inhibitor

4. Conclusion

The results of this study demonstrate that nanoemulsion-based merkubung (Macaranga gigantea) sap extract is effective in reducing the corrosion rate of steel in acidic media. The lowest corrosion rate of 0.902 mg/cm²·h and the highest inhibition efficiency of 66.05% were achieved at a concentration of 500 ppm and a temperature of 30°C. In general, increasing inhibitor concentration leads to a significant decrease in corrosion rate and an increase in inhibition efficiency, whereas increasing temperature accelerates corrosion and reduces inhibitor performance.

These findings indicate that the nanoemulsion system enhances the adsorption of active compounds on the steel surface due to its small particle size and high kinetic stability, resulting in improved corrosion protection compared to the non-nano extract. Therefore, the use of nanoemulsion-based natural inhibitors offers a promising and environmentally friendly approach for enhancing corrosion resistance of steel materials.

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