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Wuluh starfruit (*Averrhoa bilimbi linn.*) leaves extract as green corrosion inhibitor in reinforced steel

Wuluh starfruit (*Averrhoa bilimbi linn.*) leaves extract as green corrosion inhibitor in reinforced steel

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Abstract. A utilization of organic inhibitors to reduce corrosion rate in metals has been widely studied compared to inorganic inhibitors because of its environmentally friendly property. In this study, the extract of Wuluh starfruit leaves (Averrhoa Bilimbi Linn.), which is contains tannins and flavonoid compounds, was used as an inhibitor to inhibit the corrosion rate in reinforced steel. The objective of this study was to analyse the effect and the efficiency of volume inhibitor variation in 3% of NaCl corrosive medium to the corrosion rate of reinforced steel. The method used is based on the losing weight, where the sample was immersed in corrosive medium mixed with an inhibitor. The study results shown that the extract of Wuluh starfruit leaves was able to inhibit the corrosion rate as 0.0004 cm/yr at the optimum inhibitor volume of 16 mL with the efficiency of 99.7%

1. Introduction

Generally, concrete is resistant to pressure but low in tensile strength. In its use as a component of building structures, concrete is reinforced with reinforcement that is able to withstand tensile forces. For reinforcement purposes, steel materials which has beneficial technical properties is used. Reinforcing steel used can be a steel rod or wire mesh in the form of steel wire rods strung together with welding techniques.

Table 1. Threaded steel weight in various nominal diameter.

Reinforcement types[a]	Weight per m (kg)
D10	0.617
D13	1.042
D16	1.578
D19	2.226
D22	2.984
D25	3.853
D29	5.185
D32	6.313
D36	7.990

[a] D10 means diameter 10 mm.

Reinforced steel is widely used in construction of factory buildings or other buildings. Reinforced steel has two types, namely, plain reinforced steel and threaded steel or deformed steel. Plain reinforcement steel is usually used for shear / stirrup / stirrup reinforcement, and has a minimum yielding voltage of 240 MPa, with a size of Ø6, Ø8, Ø10, Ø12, Ø14 and Ø16 (with Ø is a symbol of a plain diameter). Threaded steel is used for longitudinal reinforcement or elongated reinforcement, and has a minimum melting stress of 300 MPa. Nominal diameter of the threaded steel that is generally available on Market is given in table 1.

Corrosion is the process of corroding metal or converting a refined metal to a more chemically-stable form. Corrosion impact is very detrimental, especially in reinforced steel. So, further research is needed to find how to protect reinforced steel from corrosion or what types of inhibitor is needed. Reinforced steel is often easily corroded before use as shown in figure 1.

Figure 1. Corrosion in reinforced steel.

Micro crack and concrete permeability are usually the main factors that caused corrosion in reinforced steel. Reaction occurred in concrete and caused the pH changed. The reaction is

$$Ca(OH) + CO_2 \rightarrow H_2CO_3 + 2CaO \quad (1)$$

Besides, Cl-infiltration into the concrete also caused the pH of the concrete decreased, so that the passive layer was peel off [1]. Inhibitor is a substance that can slow down the corrosion rate, may come from organic or inorganic compounds. Inorganic inhibitors are inhibitors that come from minerals which does not contain carbon compounds in it, while organic inhibitors are inhibitors obtained from nature that come from plants, such as the roots, skins, leaves, fruits and stems of plants that contain certain chemical elements such as tannins and flavonoids [2]. One of organic inhibitors to inhibit the corrosion rate in reinforced steel is by using extract of Trembesi leaves [3]. This extract has been tested in NaCl corrosive medium to reinforced steel.

Many tannins and flavonoids are contained in several types of green plants. One of them is found in the leaves of Wuluh starfruit. However, type of flavonoid compounds in Wuluh starfruit leaves was not yet known, so further research is needed to determine the type of flavonoid compounds in Wuluh starfruit leaves [4].

Flavonoids are one of the largest natural phenol groups. Actually, flavonoids are contained in all green plants. In plants, flavonoid Aglycones (i.e., sugar-free flavonoids) are contained in various forms of structure. All of them contain 15 carbon atoms in their basic nucleus, which are arranged in a C6-C3-C6 configuration, where two aromatic rings connected by three carbon units. And from literature, the type of flavonoids in Wuluh starfruit leaves is not yet known [4].

Wuluh starfruit leaves contain tannins and flavonoids, that can be applied as inhibitors that can inhibit the rate of oxidation reactions in corrosion process. To obtain the content of tannins and flavonoids in the Wuluh starfruit leaves, extraction process is needed. Extraction is a process of separating components from a mixture. Extraction can be done by maceration, socletation and calcification methods. In this study, the maceration method was used to obtain tannins and flavonoids from Wuluh starfruit leaves. The extract of those compounds was then used as corrosion inhibitors in reinforced steel. Calculation method for the rate of corrosion is based on the weight loss of steel samples.

2. Methodology

Corrosion inhibitor used in this study were from the extract of Wuluh starfruit leaves, as shown in figure 2. To extract leaves and run the phytochemicals test, similar procedure of previous study was used [5]. Initially, 3 kg of leaves weighted and dried at room temperature for 7 days to reduce the water content. Then, mashed with blender and sieved in 20 mesh of sieve tray to get the powder. The powder of Wuluh starfruit leaves then soaked in ethanol and mixed for 3 days to dissolve the active component contained in Wuluh starfruit leaves. After 3 days of soaking, filtered the mixture using a vacuum filtration to remove the leaves and get the filtrate. The filtrate from this maceration method was then evaporated in rotary evaporator at temperature of 50^{o}C, 150 rpm for 2 hr to remove the solvent. Finally, the product was tested for phytochemicals.

Figure 2. Wuluh starfruit leaves (Averrhoa bilimbi linn.).

To run the corrosion test, a corrosive medium of 3% NaCl was used (3 mg of NaCl in 97 mL of distilled water) with different volume of inhibitor added. A variation of volume of inhibitor and corrosive medium used are shown in table 2.

Table 2. Mixed volume of inhibitor and corrosive medium in several variation.

Inhibitor volume of Wuluh starfruit extract (mL)	3% of NaCl corrosive medium (mL)
0	100
4	100
8	100
12	100
16	100
20	100

Samples of reinforced steel were cut to a length of 3 cm and thickness of 10 mm. A prepared samples were pre-treated with abrasive paper to remove the coating or activate the surface, rinsed with distilled water and heated to 70^{o}C. Each sample was then weighed to get

its initial weight. After that samples of reinforced steel to test were immersed in corrosive medium with variation volume of inhibitor as given in table 2 for 14 days or 336 hr. The corrosion test of samples was shown in figure 3. After 14 days, the samples were removed from the solution and cleaned as accordance in ASTM standard. Finally, each sample was weighted to get its final weight and took a picture of its surface morphology using digital microscope (Dino-Lite).

Figure 3. Corrosion testing

Corrosion rate data were obtained from the calculation using weight loss formula in equation (2).

$$\text{Corrosion rate } \left(\frac{\text{cm}}{\text{year}}\right) = \frac{\text{W}\left(\frac{24 \text{ x } 365 \text{ hours}}{\text{year}}\right)}{\text{D. A. t}} \tag{2}$$

Where W is the lost weight (g), D is the metal density (g/cm^3), A is the corroded metal surface area (cm^2) and t is the immersion time (hr). Efficiency of inhibitor was the determination of the value of the corrosion rate effectively. Efficiency value of an inhibitor was calculated using equation (2).

$$\text{Inhibitor efficiency} = \frac{\text{Xa} - \text{Xb}}{\text{Xa}} \text{ x } 100\% \tag{3}$$

Where Xa is the corrosion rate without using an inhibitor (cm/yr) and Xb is the corrosion rate with the addition of inhibitor (cm/yr).

3. Results and Discussions

Wuluh starfruit leaves extract compounds have been identified using phytochemical test in this study, where the result shown that the extract contains tannins and flavonoids compounds. Tannin compounds in the ethanol extract of Wuluh starfruit leaves were marked with color change to blackish green after the addition of MgCl. While the flavonoid compounds change color to yellow after the addition of 0.5 g of Magnesium powder (Mg) to the test tube of HCl and Wuluh starfruit leaves extract. Effects of inhibitor volume to the corrosion rate of reinforced steel are shown in figure 4.

Reinforced steel samples were immersed in 3% of NaCl corrosive medium for 336 hours and the rate of corrosion was calculated using equation 1. From figure 4, it can be seen that Wuluh starfruit leaves extracts are able to work as inhibitor to reduce the corrosion rate in reinforced steel. In sample without inhibitor, the corrosion rate was reached to 0.14 cm/yr. Meanwhile with the addition of inhibitor from 4 to 20 mL, the corrosion rate could be

reduced until 0.0004 cm/yr. Corrosion rate of reinforced steel was reduced along with the addition of inhibitor. The optimum volume of inhibitor was 16 mL.

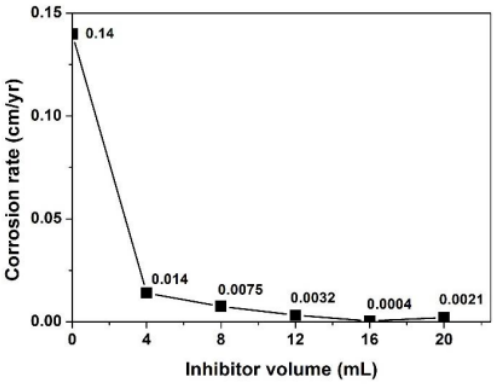

Figure 4. Effects of inhibitor volume to the corrosion rate of reinforced steel in 3% of NaCl corrosive medium.

Reduction of corrosion rate in reinforced steel with the addition of inhibitor was due to tannins and flavonoids compounds in Wuluh starfruit leaves extracts formed a passive layer on the surface of reinforced steel. This passive layer was then protected steel from the aggressive ions of Cl in NaCl corrosive medium.

Similar effects also observed by other researchers, such as organic inhibitors from trembesi leaves extracts that can inhibit the corrosion rate of reinforced steel in 3% NaCl medium and seawater [5], and papaya leaves extracts can inhibit the corrosion rate of AISI 4140 steel in medium seawater [6]. However in our study, with 20 mL addition of inhibitor, the rate was slightly increased due to the excessive use of inhibitors can trigger the subsequent occurrence of corrosion. This phenomena was due to the decomposition reaction of the formed inhibitor layer, because it has passed the saturation in inhibitor layer.

Effects of inhibitor volume to the inhibitor efficiency in 3% of NaCl corrosive medium are shown in figure 5. The value of the inhibitor efficiency can be calculated from the corrosion rate using equation 2.

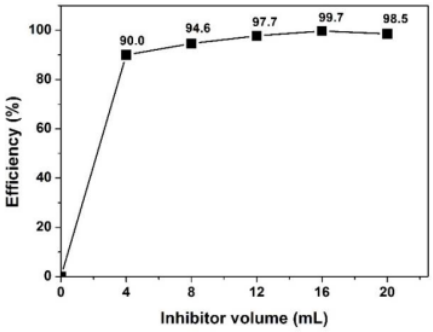

Figure 5. Effects of inhibitor volume to the inhibitor efficiency in 3% of NaCl corrosive medium.

From figure 5, it can be seen that the effect of inhibitor volume to the inhibitor efficiency in 3% of NaCl corrosive medium showed an increase in inhibitor efficiency value along with a decrease in the corrosion rate or increase in volume of inhibitor. However, the inhibitor efficiency decreased slightly with the inhibitor addition of 20 mL. With the inhibitor addition of 4 to 16 mL, the percentage value of inhibitor efficiency increased from 90% to 99.7% but decreased to 98.5% with the addition of 20 mL. This reduction in percentage is very dependent on the corrosion process occurred, where from the calculation of 20 mL inhibitor volume, the corrosion value increases slightly. This is what causes the percentage of efficiency values to decrease accordance to theory where the value of efficiency is inversely proportional to the rate of corrosion.

Surface morphology of reinforced steel after being immersed for 336 hours was observed using digital microscope (Dino-Lite), are shown in figure 6.

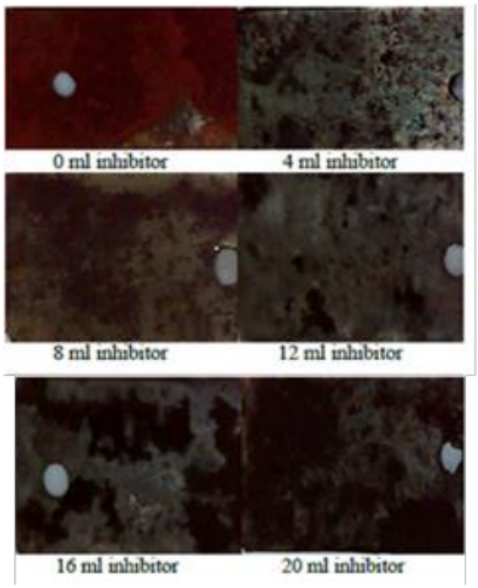

Figure 6. Surface morphology of reinforced steel after 336 hours of immersion in 3% of NaCl corrosive medium.

From figure 6, it can be seen that the colour change on the surface of the sample from several variation in volume of inhibitor used. In sample without the addition of inhibitors (control), it is clearly seen that corrosion has occurred which is characterized by the presence of brownish red colour formed on surface of metal. This corrosion occurred because of the aggressive NaCl ions easily attack the sample without protection. Whereas along with the addition of inhibitors in reinforced steel samples, corrosion formed decreases. This is because of the inhibitor which is mixed to the corrosive medium is able to protect the sample by forming a protective layer on the surface of steel samples. The optimum inhibitor volume was 16 mL with corrosion rate reduced to 0.0004 cm/yr and has 99.7% in effectivity.

4. Conclusions

Wuluh starfruit leaves extract as shown from phytochemical tests contains tannins and flavonoids compounds. From this research study, these compounds may be applied as corrosion inhibitor that inhibited reinforced steel to corrode by forming a protective layers on steel surface. Effect of wuluh starfruit leaves extract as inhibitor to decrease the corrosion

rates of reinforced steel in 3% of NaCl corrosive medium with different volume of addition from 4 to 20 mL shown good results with up to 90% in efficiency. The optimum inhibitor volume was 16 mL with corrosion rate reduced to 0.0004 cm/yr and has 99.7% in effectivity. Meanwhile, if the inhibitor addition was 20 mL, the corrosion rate was slightly increased due to the excessive use of inhibitors can trigger the subsequent occurrence of corrosion.

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