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The Effect of Immersion Time on Corrosion Rate on St37 Carbon Steel in 3% NaCl Corrosive Medium Using Waru Leaves Extract Inhibitor

Fredison Dian Sinaga[(a)*], Ediman Ginting Suka[(b)] and Pulung Karo Karo

Department of Physics, University of Lampung, Bandar Lampung, Indonesia, 35141

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Abstract

The research of effect of immersion time on the corrosion rate of St37 carbon steel has been conducted in a 3% NaCl corrosive medium using a hibiscus leaves extract inhibitor. The samples of carbon steel St37 were immersed in a corrosive medium of NaCl 3% without being given and given the inhibitor of hibiscus leaf extract with a concentration of 15% for 3, 6, 9, and 12 days. Calculation of reduction in corrosion rate is carried out by the weight loss method. The results showed that adding the hibiscus leaves extract inhibitor effectively reduced the sample's corrosion rate with the most excellent efficiency at nine days immersion, which was equal to 78,48%. The results of XRD characterization show that the phase formed is pure Fe is a corrosion product. The results of SEM characterization showed that the surface microstructure of the sample after immersion was cracks, holes, and lumps, which indicated that the sample had been corroded. The results of EDS characterization show that in the soaked sample, there were corrosion products in the form of FeO compounds whose magnitude increased with every immersion.

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Abstrak

Telah dilakukan penelitian tentang pengaruh waktu perendaman terhadap laju korosi pada baja karbon St37 dalam medium korosif NaCl 3% menggunakan inhibitor ekstrak daun waru. Sampel baja karbon St37 direndam dalam medium korosif NaCl 3% tanpa diberi dan dengan diberi inhibitor ekstrak daun waru dengan konsentrasi 15% selama 3, 6, 9, dan 12 hari. Perhitungan penurunan laju korosi dilakukan dengan metode kehilangan berat. Hasil penelitian menunjukkan bahwa penambahan inhibitor ekstrak daun waru efektif dalam menurunkan laju korosi sampel dengan efisiensi terbesar pada perendaman selama 9 hari, yaitu sebesar 78,48%. Hasil karakterisasi XRD memperlihatkan bahwa fasa yang terbentuk adalah Fe murni yang merupakan produk korosi. Hasil karakterisasi SEM menunjukkan mikro struktur permukaan sampel setelah direndam yaitu terdapat garis retakan, lubang, dan gumpalan yang mengindikasikan bahwa sampel telah mengalami korosi. Hasil karakterisasi EDS menunjukkan bahwa pada sampel yang telah direndam terdapat produk korosi berbentuk senyawa FeO yang besarnya semakin meningkat ditiap waktu perendaman.

1. Introduction

Steel is a metal alloy with iron as the principal constituent and carbon as a reinforcing constituent. For a long time, steel-type metal products have been widely used in construction, infrastructure, and industrial areas (mater et al., 2018). Steel is divided into two types based on its application and composition: carbon steel and alloy steel. Carbon steel is one of the most essential alloys, with several industrial applications. It is widely used in the manufacturing industry because it is simple to source and manufacture. It is owing to the strength and ductility of steel (Putra, 2011). Because carbon is the major component of steel, its carbon concentration determines its qualities St37 steel is one of the most common steel used in industry. St37 steel is carbon equivalent to AISI 45, with a

* Corresponding author.
E-mail address: (a) fredisonsinaga08@gmail.com, (b)ediman.ginting@fmipa.unila.ac.id

chemical composition of carbon: 0.5 percent, manganese: 0.8 percent, silicon: 0.3 percent, and other elements, and has a hardness of 170 HB and tensile strength of 650-800. It is also classed as low carbon content, ranging from 0.468-574 percent. The letter St stands for steel. This steel is reasonably inexpensive. However, it has the disadvantage of being quickly rusted (Kirono & Amri, 2013).

Corrosion is a redox reaction of a metal with other molecules in its surroundings that results in the formation of undesirable substances. Metals chemically react with corrosive environments to create stable compounds known as corrosion products (Raja & Sethuraman, 2008). Corrosion is caused by the migration of metal ions into solution in the active zone (anode), the transit of electrons from the metal to the acceptor in the less active region (cathode), and ionic and electronic currents in the metal (Oguzie, 2007). Corrosion causes a material's quality to deteriorate, resulting in metal becoming a less valuable material (Nasution et al., 2012). Corrosion cannot be stopped, although the pace of corrosion can be delayed by metal control and protection efforts. The inclusion of inhibitors is one of them. So far, the usage of inhibitors is extensively employed and is one of the most effective strategies to prevent corrosion. It is also ecologically benign and inexpensive (Ludiana & Handani, 2012).

An inhibitor is a method or technology that can protect the metal from substances with a high corrosion rate. Because the presence of corrosion inhibitors in the solution is critical for preserving the steel surface (El-Etre, 2006). Organic and inorganic substances are the most common sources of inhibitors. Organic inhibitors are efficient since they are easy to obtain, environmentally friendly, and inexpensive (Pradityana et al., 2007). Inorganic inhibitors, such as silicates, borates, tungstates, phosphates, chromates, and arsenates, are hazardous, expensive, and harmful to the environment (Indrayani, 2016). Environmental and health concerns influence the use of chemical compounds in diverse applications because inhibitors refer not only to efficacy but also to safety (Abdel-Gaber et al., 2011). As a result, using inhibitors that might negatively impact the environment can be replaced with natural agents that are both effective and safe for the environment (Abdel-Gaber et al., 2011). Many investigations on ecologically benign corrosion inhibitors that are produced from natural materials have been conducted at this time.

Organic inhibitors are effective at slowing the corrosion rate of metal, as evidenced by a study undertaken by (Ludiana & Handani, 2012). Antioxidant components in tea leaf extract have been shown to suppress the corrosion reaction of carbon steel in a corrosive NaCl solution. As a result, the existence of a layer generated by the inhibitor and capable of blocking the attack of aggressive ions from NaCl reduces the formation of holes or rust (Yerimadesi et al., 2013). Extracted hibiscus tiliaceus leaf is one of the natural compounds that can be utilized as an organic inhibitor. Saponins, flavonoids, polyphenols, and tannins are phytochemical substances in war leaves (Lusiana, 2013). Furthermore, hibiscus leaves are easy to get and, when appropriately utilized, are environmentally friendly. Waru has various applications, including traditional medicine for anti-inflammatory, blood cleansing, anti-swelling, and bleeding control, among others, and the leaves contain tannins and phenolics (Rustini et al., 2015).

In this investigation, St37 carbon steel was immersed in a 3 percent NaCl corrosive medium for three days, six days, nine days, and 12 days with hibiscus leaf extract utilizing the maceration method for the hibiscus leaf extraction procedure. This study aims to see if varying the immersion time of St37 carbon steel samples in a corrosive medium soaked with inhibitors of war leaf extract can inhibit corrosion rate and determine the efficiency and microstructure, as well as chemical elements and phases formed in the resulting samples. Immersion in a corrosive media with an inhibitor added. The weight loss method was used to calculate the corrosion rate. Scanning Electron Microscope (SEM), Energy-dispersive X-ray spectroscopy (EDS), and X-ray Diffraction were used to analyze the corrosion of St37 carbon steel (XRD).

2. Methodology of Research

Tissue, glass, beaker, measuring cup, measuring flask, sample bottle, spatula, dropper, funnel, aluminum foil, caliper, nylon thread, digital balance, rotary vacuum evaporator, steel cutting tool, sandpaper 320, 400, 800, 2000, filter paper, blender, SEM, EDS, and XRD were all employed in this investigation. The materials utilized in this investigation included hibiscus leaves, St37 steel, 3% NaCl, 96% ethanol, distilled water, and acetone.

2.1 Making an Inhibitor Solution from Waru Leaves

To make an inhibitor solution of warm leaf extract, dry up to 3 kg of waru leaves at room temperature for 20 days to eliminate the water content. To facilitate and maximize the extraction process, puree the dried hibiscus leaves in a blender. It is using the maceration procedure to extract hibiscus leaves that have been smoothed. For 24 hours, place the maceration results of war leaves in a container containing 96 percent ethanol. Filter the soaking results through filter paper until we have obtained the filtrate. To get a concentrated extract, evaporate the filtrate from the maceration using a rotary evaporator at 200 rpm and 50 ^{0}C.

2.2 Preparation of Steel Samples

Cut eight pieces of St37 steel with 20 x 20 x 5 mm^3 dimensions for sample preparation. To remove dirt and scratches caused by cutting, use 320, 400, 800, and 2000 grid sandpaper to clean and smooth the steel surface. Dip the steel in acetone to remove any grime that has clung to it.

2.3 Sample Code

Table 1 shows a sample code used to help with data presentation and analysis.

Table 1. Sample Code.

No	Code Samples	Adverb
1	St37 3.0	Immersed time 3 days inhibitor 0 %
2	St37 6.0	Immersed time 6 days inhibitor 0 %
3	St37 9.0	Immersed time 9 days inhibitor 0 %
4	St37 12.0	Immersed time 12 days inhibitor 0 %
5	St37 3.15	Immersed time 3 days inhibitor 15 %
6	St37 6.15	Immersed time 6 days inhibitor 15 %
7	St37 9.15	Immersed time 9 days inhibitor 15 %
8	St37 12.15	Immersed time 12 days inhibitor 15 %

2.4 Make a 3 percent NaCl Corrosive Medium Solution

Distilled water dilutes NaCl to make a 3 percent NaCl solution. The dilution was done by pouring 3 ml of NaCl solution into a beaker and diluting it with 97 ml of pure water.

2.5 Inhibitor Solution Preparation

To make a 15% concentration inhibitor solution, put 15 ml of warm leaf extract inhibitor solution into a beaker and dilute it with 85 ml of distilled water.

2.6 Evaluation

During this immersion step, eight samples were used, four of which had inhibitors added and four did not. The samples were then immersed in a 3 percent NaCl corrosive medium for three, six, nine, and twelve days.

2.7 Corrosion Rate Calculation

The weight loss approach provided in equations 1 and 2 can be used to calculate corrosion rate and inhibitor efficiency.

$$CR = \frac{k \times w}{D \times A \times T} \quad (1)$$

with CR = Corrossion Rate (mm/y), K = Corrosion rate constant ($8{,}76 \times 10^4$), W = Mass difference (g), A = Surface area (mm^2).

$$EI\ (\%) = \frac{CRo - CR}{CRo} \times 100\% \quad (2)$$

with, *EI* = Efficiency inhibitor (%), *CRo* = Corrosion rate of inhibitor (mm/y), *CR* = Corrosion rate on inhibitor (mm/y) (Santoso, 2019).

2.8 Characterization

The corroded steel sample is next analyzed using SEM-EDS to detect the surface structure of the sample and to see the chemical elements contained in the sample, as well as XRD to determine the phase developed in the sample.

3. Discussion and Results

The effect of the immersion period on the corrosion rate of St37 carbon steel in 3 percent NaCl corrosive media employing an inhibitor of war leaf extract with soaking times of three days, six days, nine days, and twelve days is investigated in this study.

3.1. Corrosion Rate Calculation

The weight loss method calculated the corrosion rate (Kayadoe & Turalely, 2016). Weigh the initial mass of the sample before it corrodes, the soaking sample is cleaned and dried, and the final mass of the sample is weighed. The link between immersion time and corrosion rate is depicted in **Figure 1** below.

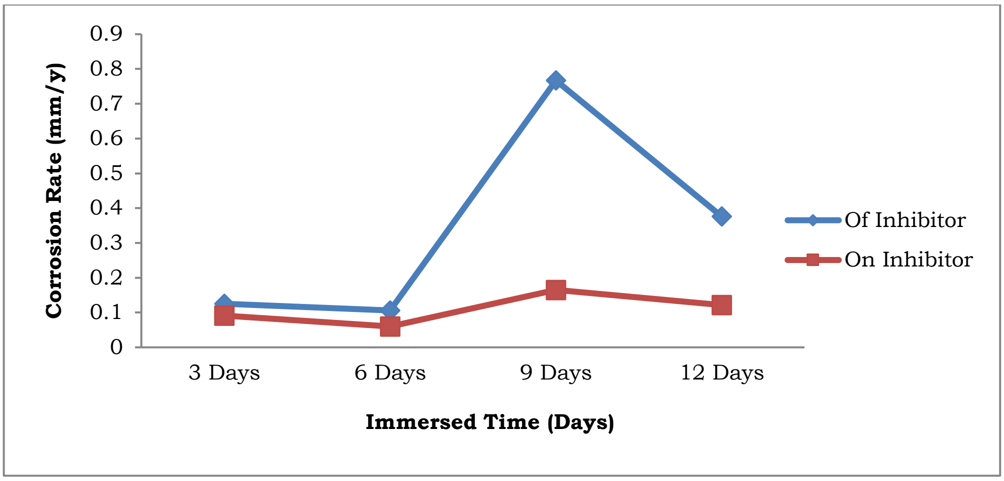


Figure 1. Corrosion rate versus immersion time chart.

Figure 1 depicts the results of the corrosion rate versus immersion time calculation, which can be seen that the steel immersed without an inhibitor has a higher corrosion rate value because the compounds contained in the corrosive medium NaCl are very aggressive in attacking the steel surface. Hence, the corrosion rate of the steel continues to increase. Compared to the corrosion rate with inhibitors, the results are lower because the tannin components included in the hibiscus leaf extract inhibitor can protect steel against NaCl attack. Because the tannins have been adsorbed, which separates the steel surface from the corrosive media, these tannin compounds can create Fe-tannate on the steel surface (Ali et al., 2014). Furthermore, calculations were performed to assess the efficacy of the hibiscus leaf extract inhibitor, as shown in **Figure 2**.

Figure 1 depicts the results of the war leaf extract efficiency inhibitor calculation. The link between immersion time and efficiency inhibitor is depicted in Figure 2 below.

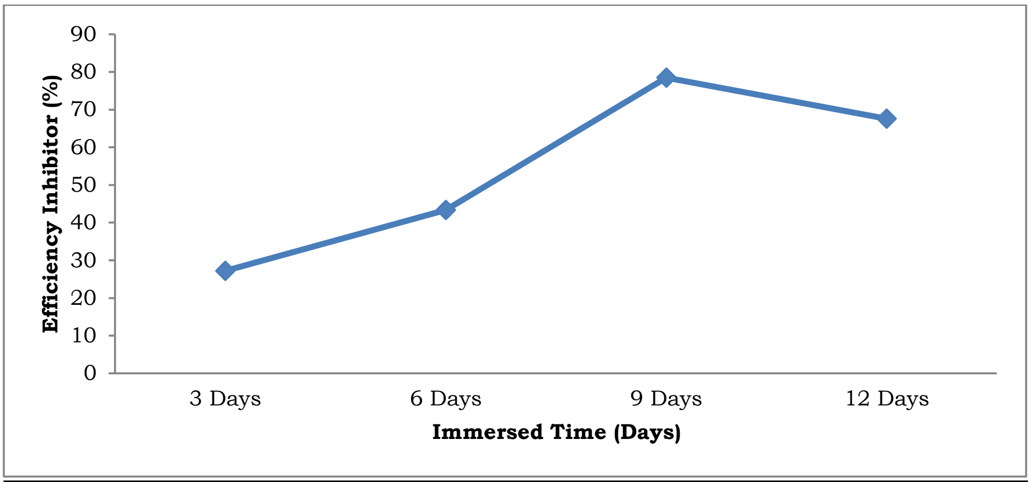


Figure 2. Efficiency inhibitor on the immersed time chart.

3.2 Analysis of X-*Ray Diffraction* (XRD)

The phase generated from St37 steel is determined using an XRD test. The XRD test is performed at a wavelength of 1.54060. The XRD analysis diffractogram is displayed in Figure 3 as the analysis result.

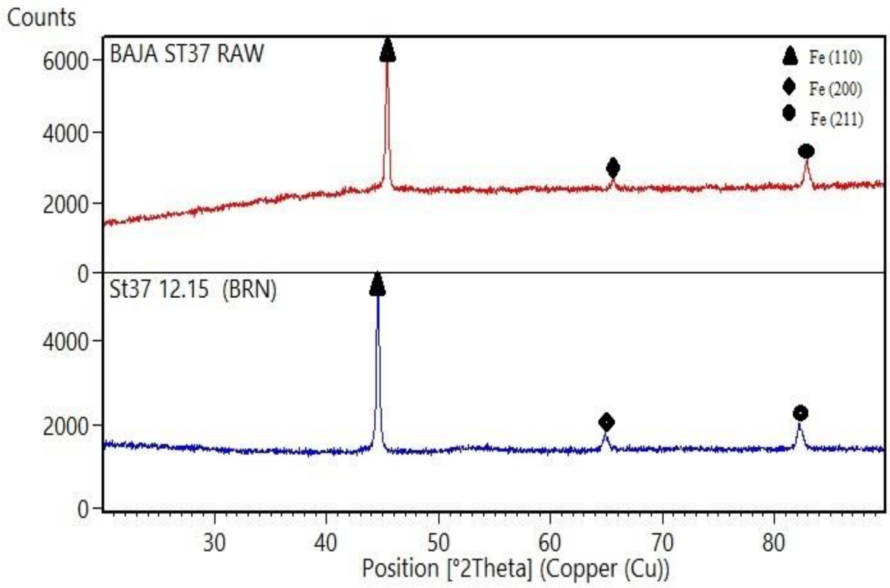


Figure 3. XRD diffractogram analysis: St37 raw, and St37 12.15

The samples tested are the raw St37 sample and the 12.15 St37 sample shown in Figure 3. The data show firm peaks, indicating the presence of a crystalline component. A qualitative examination of the XRD results was performed using the search match analysis approach to determine the phase created.

Figure 3 shows that the three most significant peaks observed in the raw St37 sample were crystalline phases in the form of Fe with lattice planes (110), (200), and (300). (211). While sample St37 12.15 detected three of the highest peaks, they are the Fe phase with lattice planes (110), (200), and (300). (211). It implies that the diffraction peaks were lowered during immersion using inhibitors due to the addition of inhibitors, implying that the inhibitors were effective.

3.3 Analysis of Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)

The microstructure and elements produced from corrosion products were determined using SEM equipped with EDS.

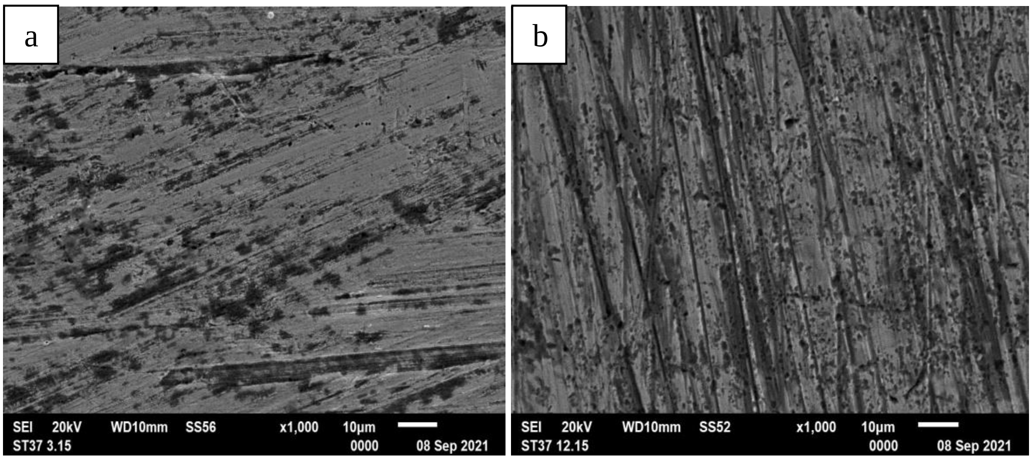
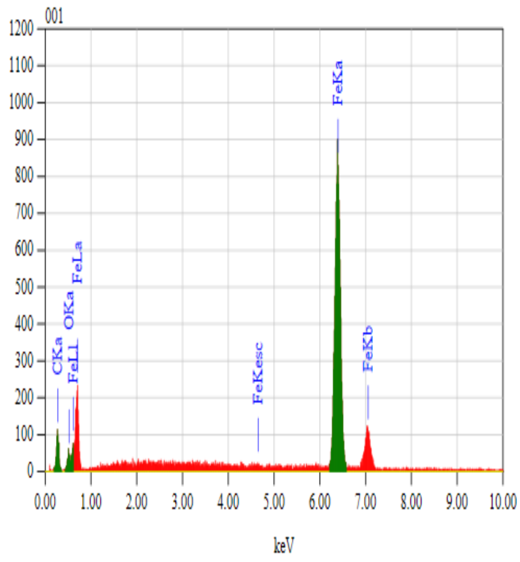
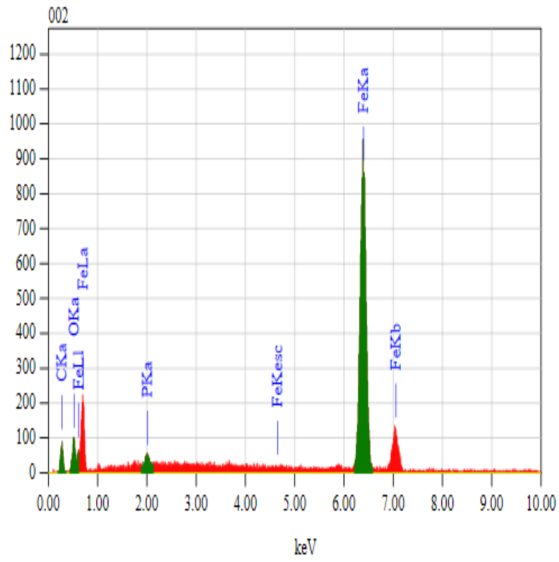


Figure 4. Study analysis SEM on magnification 1000x: (a) sample St37 3.15, and (b) sample St37 12.15.

Figure 4a illustrates the presence of lumps, which are corrosion products on the surface of the sample after immersion in a 3 percent NaCl solution, as well as holes on the surface of the sample, which are the main reasons driving corrosion. While in **Figure 4b**, a crack line can be seen, indicating the development of stress corrosion cracking. There are substantial variations between the two samples that have been evaluated, with the sample in Figure 4b having more holes and being practically spread across the whole surface of the sample (Nurhayati et al., 2020). The SEM data in **Figure 4b** support the corrosion rate calculation results, which show that the sample St37 12.15 has a higher corrosion rate value of 0.122 mm/y. Figure 5 and Table 2 show the findings of the EDS study.



(a)



(b)

Figure 5. EDS study analysis: (a) sample 6-0, (b) sample 6-4

Table 2. Percentages study analysis EDS sample St37 3.15 dan St37 12.15

Sample	Percentages Components (%)			Corrosion Products (%)
	C	O	Fe	
St37 3.15	15.69	3.38	80.92	84.3
St37 12.15	11.83	6.15	81.05	87.2

Table 2 shows the percentages of components in the sample that contain carbon (C), oxygen (O), and iron (Fe), all of which are corrosion products. Sample St37 3.15 has fewer elements of O and Fe than sample St37 12.15, which has more components of O and Fe (Simanjuntak et al., 2019). The corroded samples were dominated by O and Fe content, with the St37 12.15 sample containing a more significant element when the inhibitor's capacity to shield the sample from corrosion began to decline, or the inhibitor was already in a saturated state (Nurhayati et al., 2020).

4. Conclusions

According to the research, immersion duration's influence on carbon steel's corrosion rate is that the longer the immersion time, the larger the corrosion rate produced on carbon steel without inhibitors. With a 9-day immersion time of 78.48 percent, abundant St37 had the highest inhibitor efficiency. According to the XRD analysis results, three peaks are generated, indicating that the phase created is Fe. The carbon steel sample has been corroded. According to SEM and EDS examination, it can be shown that the EDS analysis on the St37 sample with a 12-day immersion duration in an inhibitor yields an 87.2 percent corrosion product. The corrosion rate recorded after nine days of immersion resulted in the most significant corrosion rate of 0.767 mm/y without inhibitor.

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