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Modification of Steel Surface with Addition of Cocoa Fruit Pod Extract Inhibitor Using Electrodeposition Method

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ABSTRACT
The steel surface modification has been carried out with the addition of an inhibitor of cocoa fruit pod extract with varying concentrations of 0.5%, 1%, 1.5%, 2.0% and 2.5%. The aim is to add inhibitors to improve the appearance, and to slow down the corrosion rate. The electrodeposition method was used to form a thin film on the surface at a voltage of 3 volts for 3 minutes. Surface characterization using optical microscopy and scanning electron microscopy (SEM). X-Ray Diffraction (XRD) is used to determine the phase that occurs. The corrosion rate was calculated using the weight loss and potentiostate methods. The results of the characterization of the steel surface with the addition of 1% fruit pod extract inhibitor at the electrodeposition showed that the surface was smoother and more even and there was no porosity. XRD analysis showed that the electrodeposition results under the same conditions contained two elemental phases, namely Cu and Fe with different intensity values. The highest intensity is located at the peak of the second position of 2-Theta 44,84860 which is the peak of the Fe crystal. The lowest intensity is located at the third peak of 259.89 at the position of 2-Theta 98.9141 which is the peak of Cu. The high intensity indicates that the particle has good crystallinity. Cocoa fruit pod inhibitor is able to slow down the corrosion rate and smooth the metal surface by electrodeposition method.

1. INTRODUCTION

The increasing use of steel in infrastructure construction, autos, shipboards, railroad, arms, and equipment, and in line with current technological developments (Afandi, Arief, & Amiadji, 2015; AL-Senani, AL-Saeedi, & AL-Mufarij, 2016). Steel is widely used because this material is easy to obtain, manufacture, and high-strength (Yetri & Jamarun, 2015). However, the weakness is that steel is easily corroded, decreasing the material's mechanical properties. Corrosion causes substantial economic losses (Wang et al., 2016). These losses reach trillions of rupiah per year or an estimated almost 3.5% of the country's Gross National Product (GNP).

There are several methods to prevent the decline in the function of steel due to corrosion, including others minimize the weak areas on the steel surface, keeping the steel clean, providing an oil layer, and can also be by insulating the steel surface (Rodríguez Torres, Valladares Cisneros, & González Rodríguez, 2016), adding inhibitors, and by coating the surface with an impenetrable layer such as paint (Victoria, Prasad, & Manivannan, 2015). The surface coating method by synthesizing a lamella on the surface of a metal is widely used because this coating method

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can isolate the surface of the metal from the surroundings, control the microenvironment on the surface of a metal, beautify the appearance or decorative (Bayuseno, 2009). The steel surface coating process can use a thin layer of Cu because Cu is relatively cheap and non-toxic, and the Cu synthesis process is relatively easy (Oktaviani, 2014).

There are several ways to synthesize thin films, such as electrodeposition, sol-gel, sputtering, and anodic oxidation methods. This electrodeposition method is more commonly used in the industrial sector (Tissos, Dahlan, & Yetri, 2018) because it has advantages compared to other methods, namely the implementation is quite simple, the substrate can be coated entirely on the top surface, and the average probability of deposition is high (Dahlan, 2009).

In addition to coating with a thin layer, the inhibitor application is still the best solution to protect metals from corrosion (Nugroho, 2015). The use of corrosion inhibitors has made significant progress in technological development since 1950 and 1960 and is expected to increase by 4.1% per year to 2.5 billion USD by 2017 (Dariva & Galio, 2014). The use of inhibitors as an effort to prevent corrosion has a weakness if the inhibitors used are inorganic inhibitors which are chemicals that are harmful to the organism, are relatively expensive, and can damage the environment (Umoren & Solomon, 2017; Yetri & Jamarun, 2015, 2016; Yetri, Jamarun, Nakai, & Niinomi, 2015). Inhibitors contain organic matter and as a solution to solve this problem, many studies have used natural ingredients as inhibitors, mainly plant extracts, be it leaves, stems, fruit, seeds, bark, or plant roots. For example, henna leaves (Chaudhari & Vashi, 2016; Zulkifli, Yusof, Isa, Yabuki, & Nik, 2017), gambier leaves (Murti, Handani, & Yetri, 2016), lime leaves (Hassan, Khadom, & Kurshed, 2016), spoon leaves (Mobin & Rizvi, 2017), elephant grass leaves (Ituen, James, Akaranta, & Sun, 2016), banana peels (Ismail & M Tajuddin, 2015), peony skins (Honarvar Nazari, Shihab, Cao, Havens, & Shi, 2017), mangosteen rinds (Turnip, Handani, & Mulyadi, 2015), peels of cocoa (Purnomo, 2015; Yetri, Emriadi, Jamarun, & Gunawarman, 2016; Yetri & Jamarun, 2015), aloe vera (Gupta & Jain, 2014), and there are still many studies using extracts of natural ingredients that can be used as inhibitors.

Indonesia is the world's third largest cocoa producer, with total production reaching 1 million tonnes (Badan Pusat Statistik, 2022). Cocoa pod is an abundant natural resource. 70-75% of its production is cocoa pod waste (Yetri & Jamarun, 2015) which has yet to be processed optimally (Yetri, 2021). The choice fell on cocoa pod skin as a research topic because this pod shell contains catechin and epi catechin compounds which can form a thin layer on the steel surface.

So, it is possible in this study to combine the electrodeposition method with adding inhibitors from cocoa pod waste. Because the availability of cocoa pod skin is relatively abundant, it is easy to extract the tannin compounds, and it is not harmful to the environment. So that is why cocoa fruit pod extract is used for corrosion inhibitors. Previous studies have added nickel to form a thin layer on the surface using the electrodeposition method to enhance its appearance and have not analyzed its mechanical properties (Tissos et al., 2018). In this study, an inhibitor of cocoa pod extract will be added to the electrolyte solution in coating steel substrates with Cu using the electrodeposition method. Utilization of this waste is expected to maximize the use of cocoa pod waste which has been left to waste, thus damaging the beauty of the environment.

2. METHODS

Cocoa pods are taken from residents' plantations in Lubuk Minturun, Padang. Cu lamella was manufactured using the electrodeposition method at the Laboratory of Materials, Physics Department, Faculty of Mathematics and Natural Sciences, University of Andalas. XRD characterization at Politeknik Negeri Padang, SEM characterization at the Department of Mechanical Engineering, Faculty of Engineering, Andalas University, and corrosion rate testing with potentiodynamic polarization and weight loss methods were done at the Chemistry Department, Andalas University.

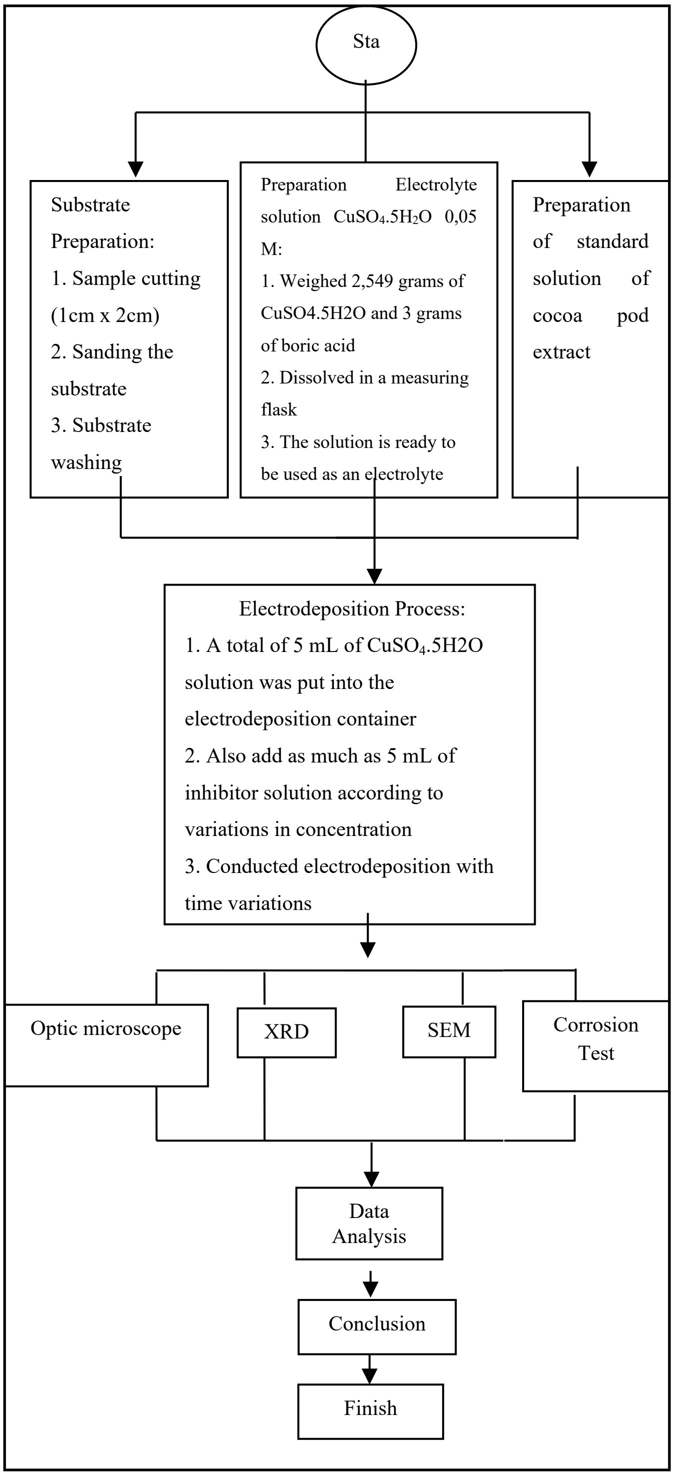

Figure 1. The Research Flowchart

The equipment needed to support the research are OHAUS GALAXYTM 160 digital balance, Power Supply, Ultrasonic bath, Basicmeter, Desiccator, Oven, chemical glassware, tweezers, Disposable syringe, spatula, gloves, mask, Vacuum Rotary Evaporator, distillation equipment, Potentiostat e-Corder 416, optical microscope, X-ray Diffraction (XRD) PanAlytical, Scanning Electron Microscope (SEM) Hitachi S-3400N, Hot Plate Magnetic Stirrer C-MAG HS 7. The materials used in this study included: commercial steel specimens, Merck's $CuSO_4.5H_2O$ as an electrolyte solution, Merck's HCl p.a, Boric Acid (H_3BO_3), Cocoa pods, Methanol 70%, Alcohol p.a 96%, sterile Aquades, sandpaper with various kinds of roughness sizes, and filter paper.

2.1. Preparation of Solution

First, an electrolyte solution is made for the electrodeposition process. CuSO4.5H2O weighed 2.495 grams, and HBO3 weighed as much as 3 grams. Then, the two chemical compounds were dissolved in a 200 mL volumetric flask to obtain an electrolyte solution with a concentration of 0.05 M (Jamaluddin, 2012). Meanwhile, the manufacture of cocoa pod extract, corrosive media, and corrosive media solutions with the addition of an extract of cocoa pod inhibitor was carried out by Yetri Y et al. 2015.

2.2. Electrodeposition Process

The process of electrodeposition and determination of the rate of corrosion using the method of weight loss follows what has been done by Tissos 2018, Silfa 2018, and Jamaluddin, 2012. For more details on the work steps in this study, see the flow chart in Figure 1.

3. RESULT AND DISCUSSION

3.1. Surface Analysis with Optical Microscope

In this study, the interaction between Cu and cocoa pod extract will form a thin layer on the surface to block the attack of corrosive ions so that it will inhibit the rate of corrosion. The Cu layer was synthesized using the electrodeposition method using an electrolyte solution of CuSO4.5H2O with variations in the cocoa pod extract solution concentration as an inhibitor solution of 0.5; 1.0; 1.5; 2.9; and 2.5%. The variation of electrodeposition time is 1; 2; 3; 4, and 5 minutes. The results of sample characterization using an optical microscope can be seen in Figure 2.

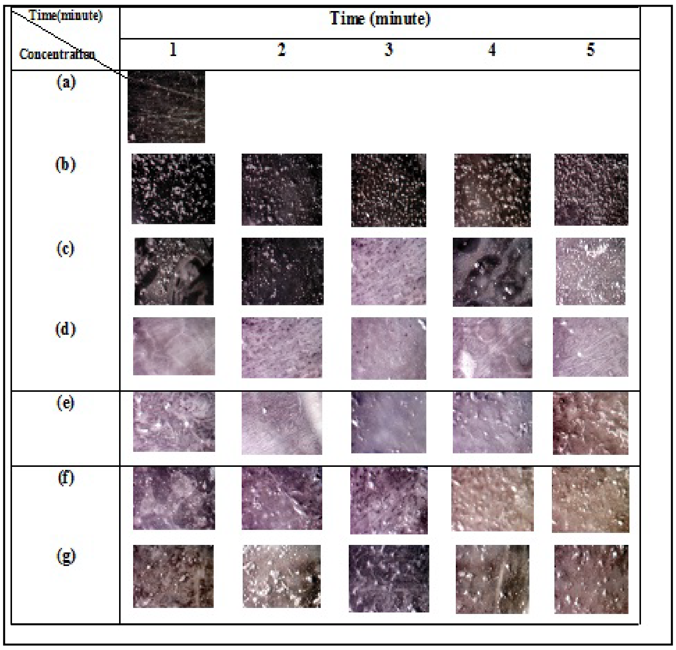

Figure 2. Characterization Results with Optical Microscopy (a) Samples of Steel Before Electrodeposition, and After Electrodeposition (b) Added 0% Inhibitor (c) 0.5% Inhibitor (d) 1% Inhibitor (e) 1.5% Inhibitor (f) 2% Inhibitor (g) 2.5% Inhibitor

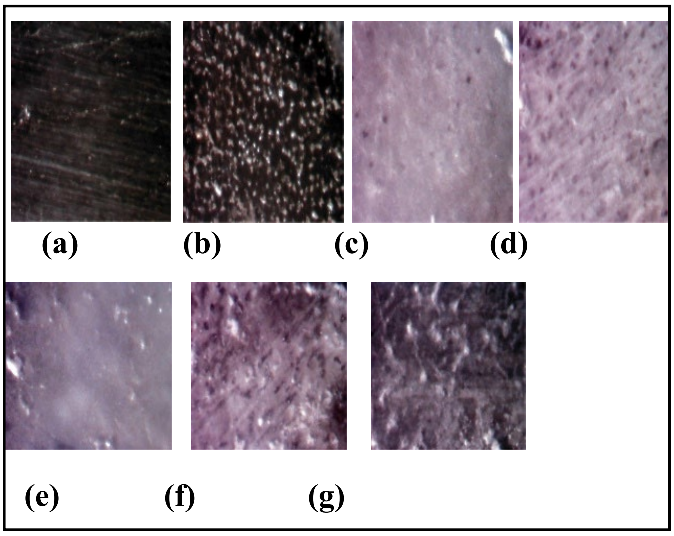

Figure 3. Characterization Results with Optical Microscopy (a) Sample of Steel Before Electrodeposition, and After Electrodeposition (b) Plus 0% inhibitor (c) 0.5% Inhibitor, (d) 1.0% Inhibitor, (e) 1.5% Inhibitor, (f) 3.0 Inhibitor, and (g) 2.5 Inhibitor

Figure 3(a) is a photo of the original steel after grinding and sanding without electrodeposition, which shows only fine lines of the influence of sanding on the surface of the steel. Figure 3b shows the electrodeposition results without adding an inhibitor which was electrodepositioned with an electrodeposition time of 3 minutes. The electrodeposition result is uneven, the particles accumulate, and the grain thickness is coarse. The granules are Cu particles that are starting to form. During the addition of 1% and 1.5% inhibitors (Figures 3c and 3d) with the same electrodeposition time, the deposition results were much smoother and more evenly distributed, there was no accumulation of material in some parts of the deposition results, and the formation of another layer covering the Cu particles.

The results of deposition are reasonable when compared to steel without the addition of inhibitors in Figure 3a. While in Figures 3e, 3f, and 3g, the deposition results are still smooth and even, but other layers are starting to rise to accumulate in some parts. Based on Figure 3, it can be analyzed in general that the increase of concentrations of inhibitor solution 1% and 1.5% was able to improve the deposition results to be more even and smoother, reduce the buildup of particles formed, and produce smaller particles, and produce a layer that protects Cu particles. This layer is produced from the inhibitor of cocoa pod extract added in the electrodeposition process.

3.2. Scanning Electron Microscopy Analysis

Analysis Based on the best results in Figure 3, the surface morphology characterization with SEM was carried out, as shown in Figure 4. Figure 4a shows the surface of the Cu layer on the steel before being electrodepositioned, the deposition results are uneven, and a buildup of particles of non-uniform size is formed. This condition indicates that the Cu coating is imperfect during the electrodeposition process, although macroscopically, the layer looks uniform. In Figure 4b, adding 1% inhibitor shows a smoother, more homogeneous deposition despite a thin gap from the resulting layer. However, the layers are deposited evenly, and particles are not accumulated in some parts. The effects of sanding are not visible on the steel surface.

Furthermore, using a 1.5% inhibitor solution, the characterization results in Figure 4c shows that the deposition results are still evenly distributed, and there is a passive layer that can slow down the corrosion rate on the steel surface. This layer is a barrier against the attack of corrosive ions on the surface of the steel sample (Yetri & Jamarun, 2015). The barrier formed can protect the metal surface (Arista, Dahlan, & Syukri, 2016). Meanwhile, when heating at 600°C in electrodeposition using 1% inhibitor, aggregate buildup occurs in several parts, and the protective layer (barrier) becomes invisible (Tissos et al., 2018). It is shown that heating can damage the layer formed on the steel surface (Rahmawatf, Wahyuningsih, & Handayani, 2008; Sujatno, Salam, Bandriyana, & Dimyati, 2017).

3.3. Characterization by X-Ray Diffraction

XRD analysis was carried out on three samples that were electrodepositioned for 3 minutes, namely the original steel before being electrodepositioned, the steel that was electrodeposited without the addition of an inhibitor, and the steel that was electrodepositioned with the addition of a 1% cocoa pod extract inhibitor are showed in Figure 5. The results showed that the surface was more even, the grain level was finer than the raw cocoa pod extract, and the thickness of the deposition was relatively thin.

The result of XRD characterization is a diffractogram between 2-Theta (2θ) angle and intensity. The 2-Theta angle used is in the range of 20o to 100o. From the data on the intensity and position of the diffraction peaks produced by the X-ray diffractometer and then compared with the Joint Committee on Powder Diffraction Standards (JCPDS) standard data, it is known that the phase of the crystalline element layer on the original steel before electrodeposition.

The XRD characterization data of steel before being electrodepositioned produced three X-ray diffraction peaks, namely peaks at an angle of 2-Theta for the intensity, with their respective values shown in Table 1. By referring to JCPDS, each 2-Theta angle can be identified in phase. At the same time, the XRD data of the Cu layer on the steel that was electrodeposited with the addition of a 1% cocoa pod extract inhibitor showed five X-ray diffraction peaks, namely peaks at an angle of 2-Theta to the intensity with each value shown in Table 2. Each peak has a value of different value intensity towards different 2θ positions. Referring to the data on JCPDS, each 2-Theta angle can be identified in phase (Ismail & M Tajuddin, 2015; Jannah, 2007).

The characterization results show that the electrodepositioned steel with adding a 1% cocoa pod extract inhibitor contains two elemental phases, namely Cu and Fe, with different intensity values, as shown in Table 2. The highest intensity is at the peak of the two positions 2-Theta 44,84860, which is the peak of the Fe crystal. The lowest intensity is located at the third peak of 259.89 at position 2-Theta 98.9141, which is the peak of Cu. The high intensity indicates that the particle has good crystallinity. The data on XRD results of electrodeposition steel with the addition of 1.5% cocoa pod extract inhibitor are shown in Table 3 below.

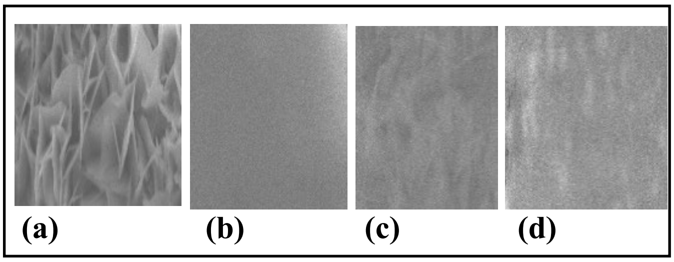

Figure 4. Surface Coating using SEM with 10000x Magnification (a) Sample Before and After Electrodeposition (b) with 1% Inhibitor, (c) 1.5% Inhibitor, and (d) 1% Inhibitor and Heated at 600°C

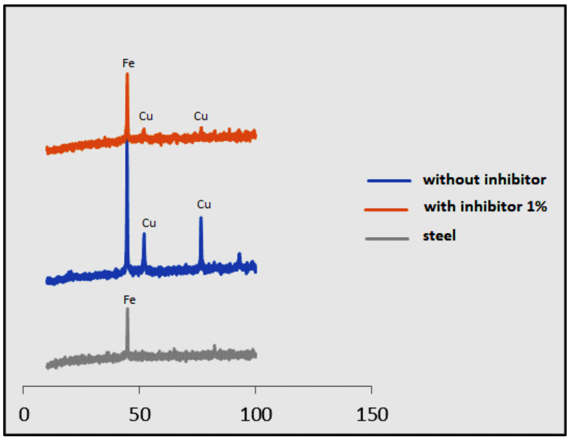

Figure 5. Three X-ray Diffraction Peaks

Table 1. Value of 2θ and Highest Peak Intensity of Steel XRD Curve Before Electrodeposition

Peak	2θ	Intensity	FWHM	D (A°)
1	44.7434	540.95	0.2362	2.02551
2	64.9524	51.52	0.3542	1.43577
3	82.3163	77.82	0.4723	1.17139

Table 2. Value of 2θ and Highest Peak Intensity of Steel XRD Curve After Electrodeposition with the Addition of 1% Cocoa Pod Extract Inhibitor

Peak	2θ	Intensity	FWHM	D (A°)
1	43.4831	530.63	0.3149	2.08125
2	44.8486	3891.77	0.3739	2.02100
3	65.0336	402.43	0.3542	1.43418
4	82.3599	630.20	0.3149	1.17088
5	98.9141	259.89	0.6298	1.01450
6	43.4831	530.63	0.3149	2.08125

Table 3. Value of 2θ and Highest Peak Intensity of Steel XRD Curve After Electrodeposition with Addition of 1.5% Cocoa Pod Extract Inhibitor

Peak	2θ	Intensity	FWHM	D (A°)
1	43,6442	283,89	0,6298	2,07394
2	44,9627	5018,75	0,3739	2,01614
3	65,1290	458,60	0,3936	1,43231
4	82,4614	703,40	0,3936	1,16970
5	98,9576	301,63	0,3936	1,01417

Table 4. Comparison of 2θ Angle of X-Ray Diffraction Peaks of Steel Before and After Electrodeposition with Inhibitor

Peak	2θ		
	Before Electrodeposition	After Electrodeposition	
		Steel + Inhibitor 1%	Steel + Inhibitor 1.5%
1	44.7434	44.8486	44.9627
2	64.9524	65.0336	65.1290
3	82.3163	82.3599	82.4614

Table 3 shows that the XRD results of electrodeposition steel with the addition of 1.5% cocoa pod extract inhibitor had the five highest X-ray diffraction peaks with different intensity values. The identification results show that the electrodeposition steel with adding 1.5% cocoa pod extract inhibitor contains two phases of crystalline elements, namely Cu and Fe, with different intensity values. The highest intensity is located at the fifth peak, 2-Theta 98.95760, which is 301.63, the peak of the Fe crystal. The lowest intensity is located at the first peak, 2-Theta 43,64420, 283.89, the peak of Cu.

The results of XRD characterization are diffraction patterns which are characteristic peaks of the crystal structure formed in the Cu layer, and the patterns of diffraction of the layer are identified at 2θ angle. The result of XRD characterization for the three samples produced three X-ray diffraction peaks. Apart from that, the diffractogram shows that the phase produced in the steel before electrodeposition is still amorphous, characterized by not many peaks formed and no Cu peaks formed. The comparison of the three peaks in each sample can be seen in Table 4.

From its table, the 2-Theta angle shift is at the first peak, and all peaks experience a shift. The shift reveals a reduction in the interplanar distance of the crystal lattice upon the substitution of Cu atoms (Ong et al., 2014). The first peak is observed, and all steel peaks have diffraction peaks at identical 2-Theta angles. It is shown that adding a cocoa pod inhibitor solution extract in the electrodeposition process of steel samples does not change the crystal structure (Jamaluddin, 2012).

3.4. Corrosion Test Analysis

The polarization curve in the corrosive medium HCl 1 N, which has been extrapolated into Tafel solution, has been published previously. The Tafel curve shows a shift in the value of the corrosion potential to a more positive and negative direction, indicating that the added extract is anodic and cathodic (Honarvar Nazari et al., 2017).

Table 5. Corrosion Parameters of Steel Samples in 1 N HCl Corrosive Media

Concentration of Inhibitor (%V/V)	I corr (mA/cm^2)	-E corr (V)	IE (%)
0	0.0072	0.51	-
1	0.0122	0.59	70.5
1.5	0.0131	0.58	80.1

Table 6. Corrosion Rate of Steel in Corrosive Medium HCl 1N

Steel + Inhibitor	Δm (gram)	A (cm^2)	T (hour)	v (g/cm^2.hour)
Steel + 0%	0.0003	2.00	6	$2.50x10^{-5}$
	0.0067	2.10	12	$2.66\ x10^{-4}$
	0.0113	2.10	18	$2.99\ x10^{-4}$
	0.0182	2.10	24	$3.61\ x10^{-4}$
Steel + 0.5%	0.0003	2.42	6	$2.01\ x10^{-5}$
	0.0052	2.42	12	$1.79\ x10^{-4}$
	0.0083	2.10	18	$2.19\ x10^{-4}$
	0.0154	2.10	24	$3.06\ x10^{-4}$
Steel + 1.0%	0.0002	2.10	6	$1.59x10^{-5}$
	0.0045	2.42	12	$1.55\ x10^{-4}$
	0.0071	2,42	18	$1.88\ x10^{-4}$
	0.0102	2,42	24	$2.02\ x10^{-4}$
Steel + 1.5%	0.0002	2.42	6	$1.58\ x10^{-5}$
	0.0035	2.42	12	$1.20\ x10^{-4}$
	0.0067	2.42	18	$1.53\ x10^{-4}$
	0.0095	2.42	24	$1.64\ x10^{-4}$
Steel + 2.0%	0.0001	2.10	6	$7.94\ x10^{-6}$
	0.0026	2.10	12	$1.04\ x10^{-4}$
	0,0059	2,42	18	$1,35\ x10^{-4}$
	0,0075	2,1	24	$1,49\ x10^{-4}$
Steel + 2.5%	0,0001	2,42	6	$6,89\ x10^{-6}$
	0,0015	2,1	12	$5,95\ x10^{-5}$
	0,0039	2,1	18	$1,03\ x10^{-4}$
	0,0069	2,42	24	$1,19\ x10^{-4}$

Table 6 shows an increase in mass loss and corrosion rate directly proportional to the time the steel is immersed in a corrosive medium. It means that the longer the time of steel immersion in corrosive media, the greater the corrosion rate (Guo et al., 2017). However, with the same immersion time, the corrosion rate will decrease as the inhibitor concentration of cocoa pod extract is added. Table 6 indicates a decrease in mass loss as the concentration of cocoa pod extract inhibitor increases. In the table, it is clear that the corrosion rate decreases with the increase in the concentration of the inhibitor of the cocoa pod extract. It is because the more cocoa pod extract inhibitors are added, the more cocoa pod extract is adsorbed on the steel surface so that the layer formed on the surface can inhibit the attack of the corrosive rate on the surface of steel so that the rate of corrosion of steel can be inhibited (Wang et al., 2016; Yetri & Jamarun, 2015). This shows that the compounds contained in cocoa pod extract can be used as inhibitors to decrease steel corrosion rate. The inhibition efficiency of steel increases with increasing inhibitor concentration while the corrosion rate decreases (AL-Senani et al., 2016).

4. CONCLUSION

Based on the data and analysis carried out on the study's results, it can be concluded that with a voltage of 3 Volts, the optimum results of morphological electrodeposition were obtained at 3 minutes, and the concentration of inhibitor of the cocoa pod extract was 1%. Then the results of characterizing the electrodepositioned steel surface showed a smoother and more even surface. There was no porosity in the electrodepositioned steel sample with the addition of a 1% concentration of cocoa pod extract inhibitor. Meanwhile, the rate of corrosion test using the weight loss method and potentiodynamic polarization showed that the corrosion rate decreased and the efficiency of inhibition increased along with the rise in the concentration of inhibitor of cocoa pod extract added. Cocoa pod inhibitors can decrease the corrosion rate and smooth the metal surface by electrodeposition.

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