Adsorption Effect of Theobroma Cacao Extract OnSteel Surface to Protect Corrosion Rate

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Abstract. Research has been carried out to slow down the corrosion rate of steel by using biomass wasteinhibitors of cocoa pod extract (Theobroma cacao). The steel is pre-soaked in the inhibitor for24, 72, 120, and 168 hours. Then followed by immersion in corrosive HCl media with variations of time, namely 48, 96, and 144 hours. Characterization of corrosion rate by weight loss method, surface morphology by optical microscopy, Scanning Electron Microscopy(SEM), and Atomic Force Microscopy (AFM). Phase analysis that occurs with X-Ray Diffraction (XRD), and the Density Functional Theory (DFT) method to analyze the shape of the molecular structure, geometry, HOMO and LUMO contours. The weight loss method givesthe best results with the lowest corrosion rate of 0.2972 mg.cm2/hour and an inhibition efficiency of 74.7128% for steel soaked in inhibitor for 168 hours and in HCl for 48 hours. Morphological analysis using optical microscopy and SEM showed that the longer it was immersed in the inhibitor, the flatter the surface and the fewer cracks it had. The XRD results also showed that there were four sharp peaks of the crystalline Fe and C phases due to the reaction between the steel surface and the inhibitor. AFM analysis shows that the adsorption formed on the surface is directly proportional to the length of immersion in the inhibitor. Analysis of the DFT method shows that the greater the EHOMO, the easier it is for a moleculeto donate its electrons, so that the greater its ability to protect against corrosion. Optimization and calculation of tannin compounds that react with the steel surface resulted in a fairly high inhibition efficiency of the cocoa pod extract inhibitor, which was 80.2098%.

Keywords: Corrosion, Theobroma cacao, Weight loss, Adsorption

1 Introduction

Steel is a material that is widely used in various fields and uses such as building materials, household appliances, and vehicle frames. This is because steel is a material that is strong, malleable, easily oxidized, and has good electrical and thermal conductivity [1]. One of the weaknesses of steel is that it is easily corroded so that its mechanical properties decrease. Thismetal rusting occurs due to a metal reaction with the environment which can reduce the qualityof the metal itself and result in a shorter metal service life [2].

Corrosion is a problem that must be avoided because it is very detrimental and causes many

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problems. Various attempts have been made to slow down the rate of corrosion, one of which is by adding inhibitors. Giving inhibitors is one of the effective methods for controlling corrosion because it is considered more economical, effective and its application is quite easy,so it is widely applied in the industrial world [3]. Organic inhibitors are more widely used because they are cheap and easy to obtain, environmentally friendly and non-toxic compared to inorganic inhibitors [4].

Utilization of plant extracts from leaves, bark, roots, fruits, and stems of plants which contain organic compounds such as tannins, alkaloids, saponins, amino acids, and proteins caninhibit the rate of corrosion reactions in steel [5]. So organic inhibitors play an important role in corrosion control strategies. One of the plants that contains high tannins is cocoa pod skin (Theobroma cacao) [6]. Cocoa pods produce a large amount of shell waste, around 75%. Cocoapod skin has not been used optimally, and is often thrown away, thus damaging the environment. Cocoa pod shell contains tannin compounds which can form complex compoundswith Fe(III) on the metal surface which is able to block the attack of corrosive ions on the metalsurface [7].

The inhibition efficiency of cocoa shell extract against mild steel in 1.5 M HCl and NaCl media has been carried out by Yetri et al [8] using the method of weight loss and electrochemical measurements. The results showed that the inhibition efficiency increased withincreasing cocoa peel extract concentration. Optimal inhibition efficiency in 1.5 M HCl and 1.5 M NaCl is 96.26% (weight loss), 92.68% (Tafel), 95.64% (Rp), 85.78% (EIS) and 91 .93%
(weight loss), 85.90% (Tafel), 90.19% (Rp) and 75.23% (EIS) for a period of 768 hours with 2.5% v/v concentration of each inhibitor [8-10].

Yetri et al [9] have also inhibited the corrosion rate of steel using crude extracts and polar extracts of cocoa pod shells in 1.5 M HCl corrosive medium using the weight loss and potentiodynamic polarization (Tafel) method. Inhibition efficiency was measured for 192 hoursof soaking, extract concentrations of 0.5-2.5% with intervals of 0.5% and a temperature rangeof 303-323 K. The best conditions were obtained for inhibitor concentrations of 2.5% at 30 ^{o}C(303 K). The inhibition efficiency for crude extract and polar extract were 83.45% and 96.26%(weight loss), 85.23% and 92.08% (Tafel) and 72.29% and 83.95%.

In order to see the effectiveness of the adsorption capacity of the corrosion inhibitor of cocoa pod extract on the steel surface to inhibit the corrosion rate, in this study the steel was soaked first in the cocoa pod extract with a variation of time. The purpose of soaking in the extract is to allow adsorption of the cocoa pod extract to occur on the surface. Then immersedin 1.5 M HCl corrosive medium.

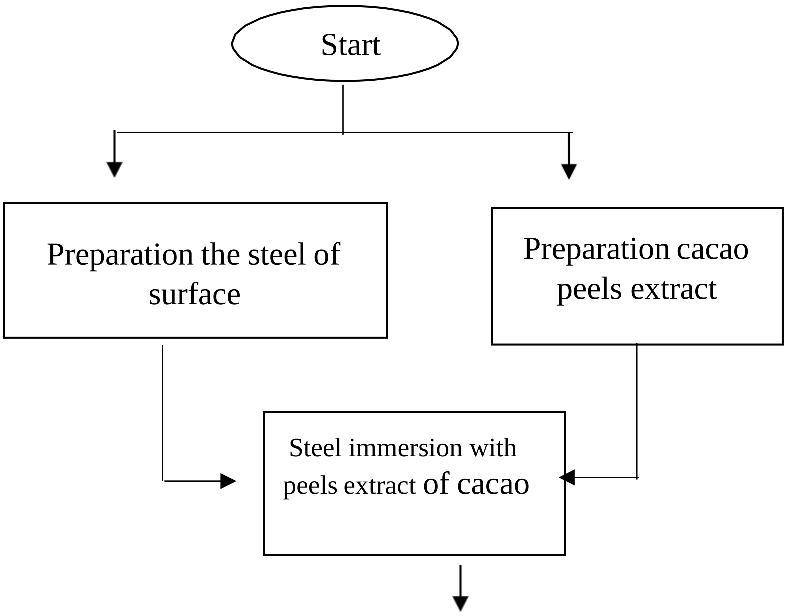
2 Research Methods

This research was conducted at the Laboratory of Materials Physics, Department of Physics, Andalas University, Padang. Corrosion rate calculation with weight loss method. XRD characterization was carried out at Padang State University. Surface morphology with optical microscopy and DFT for quantum chemistry at the Material Physics Laboratory, SEM was carried out at the Andalas University Mechanical Engineering Laboratory, AFM at the UnsyiahMechanical Engineering Laboratory.

The materials and tools needed for this research were: cocoa pod (Theobroma cacao pod), hydrochloric acid (HCl), steel plate, distilled water, ethanol, acetone, filter paper, sandpaper, aluminum foil, beaker, magnetic stirrer with hot plates, grinding machines, measuring cups, spatulas, Rotary Vacuum Evaporators, digital scales, glass containers, Erlenmeyer flasks, laptops, steel plates and tissues.

2.1 Research Procedure

The working procedure in this study, starting from the preparation of materials to analyzing theresults, can be seen in the flowchart in Figure 1.



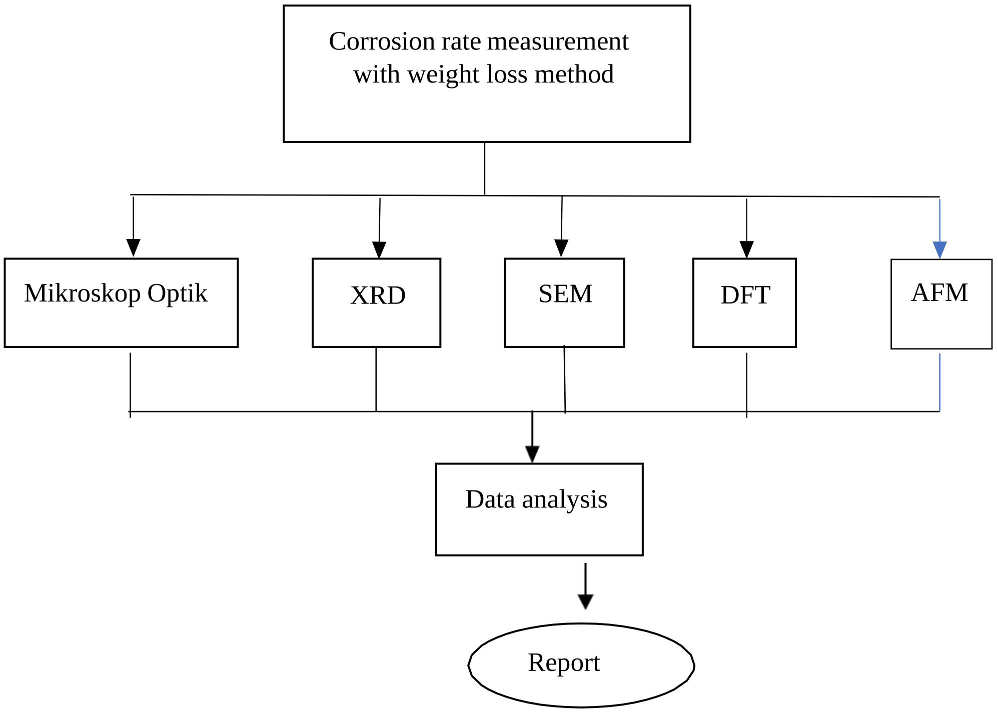


Figure 1. Research flowchart

2.2 Material preparation

The preparation of cocoa pod extract, grinding, and cleaning of the steel surface followed the procedure that had been carried out by the previous researcher [9]. After obtaining the concentrated extract of cocoa pod skin, proceed to the stage of soaking the steel into the concentrated extract of cocoa shell with variations of time for 24, 72, 120, and 168 hours. Afterthe immersion time was reached, the steel was immersed in 1.5 M HCl for 144 hours.

3 Result And Discussion

Table 1 shows the effect of immersion time in cocoa shell extract on mass loss and steel corrosion rate. This figure shows that immersion time in cocoa shell extract affects the mass change of steel produced. The increase in immersion time in the inhibitor causes the cocoa peelextract to be adsorbed more and more on the steel surface. The increase in immersion time is directly proportional to the increase in mass, where immersion of steel for 168 hours resulted in an increase in mass of 232.37 mg or 0.23 gram, while immersion for 24 hours experienced an increase in mass of 89.57 mg or 0.0896 gram.

The longer immersion time in the inhibitor causes the tannin compounds present in the cocoa peel extract to interact more with iron, thus forming a complex compound (thin layer) on the surface of the steel. The thin layer that has been formed will get thicker due to the increased immersion time, so the HCl takes longer to erode the steel surface, so that the mass loss due to surface erosion is smaller, and the corrosion rate is also getting smaller [11-12].

Table 1. Effect of Immersion Time in Inhibitor on Mass Loss, Corrosion Rate, and InhibitionEfficiency

No.	Deep Immersion Inhibitors (hours)	Weight loss (mg)	Corrosion rate	Inhibition Effisiency (IE)
1	24	232,37	11389	14,7916
2	72	147.60	0,7340	44,9568
3	120	119,60	0,5711	57,5620
4	168	89,67	0,4317	67,9056

From Table 1 it can be seen that an increase in the immersion time in the cocoa shell extract inhibitor will cause the corrosion rate to decrease. This happens because the layer of cocoa shell extract that is formed on the surface is thicker and will make the adsorption capacity higher, so it is rather difficult to be damaged by the corrosive media. Therefore, the tannin compounds present in the cocoa peel extract will form more and more complex compounds with Fe (III), so that a thicker protective layer will form on the steel surface. The length of immersion in the inhibitor also has a major effect on the efficiency of the inhibition. Because the efficiency of inhibition indicates the ability of cocoa peel extract inhibitors to inhibit the process of corrosion.

The longer it is immersed in the inhibitor, the higher the inhibition efficiency. The valueof the inhibition efficiency is inversely proportional to the value of the corrosion rate, where the smaller the corrosion rate, the higher the inhibition efficiency as shown in Table 1. The highest inhibition efficiency value reached an average of 67% for 168 hours of immersion in the inhibitor. This proves that the adsorption of cocoa shell extract on the steel surface is able to form a protective layer on the steel surface so that it can inhibit corrosion attack [9].

Characterization analysis on the steel surface was carried out using an optical microscope, SEM, and AFM. All samples were initially characterized using an optical microscope as a resultof immersion in corrosive medium HCl 1.5 M with variations in initiator immersion time of 24hours, 72 hours, 120 hours and 168 hours as shown in Figure 2.

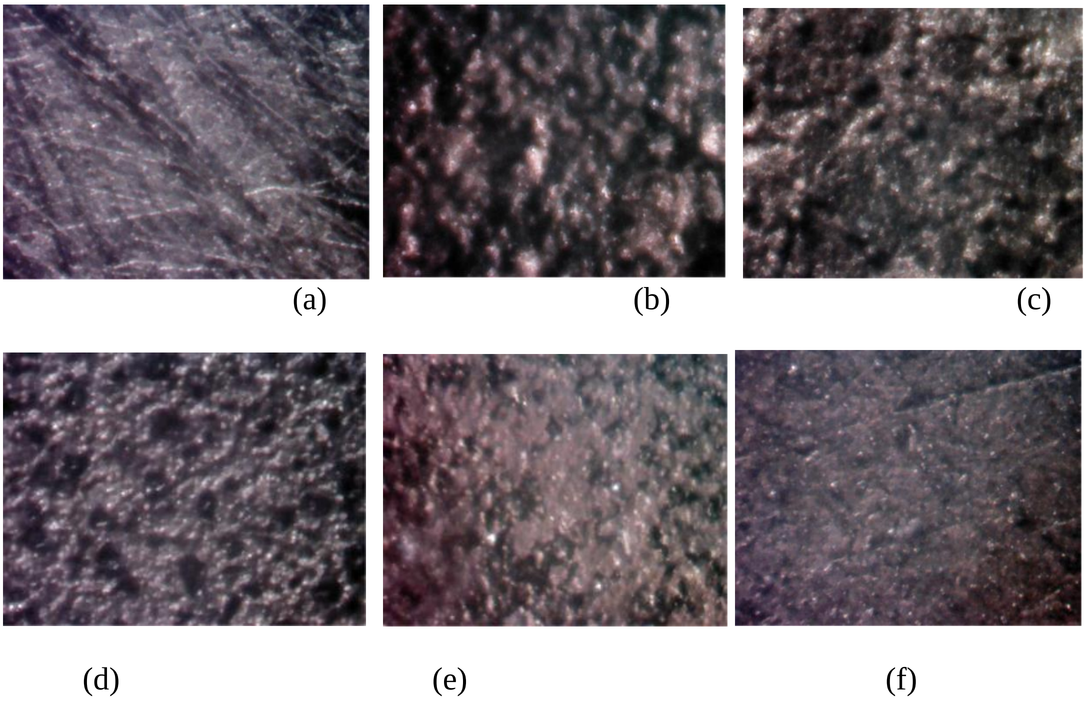
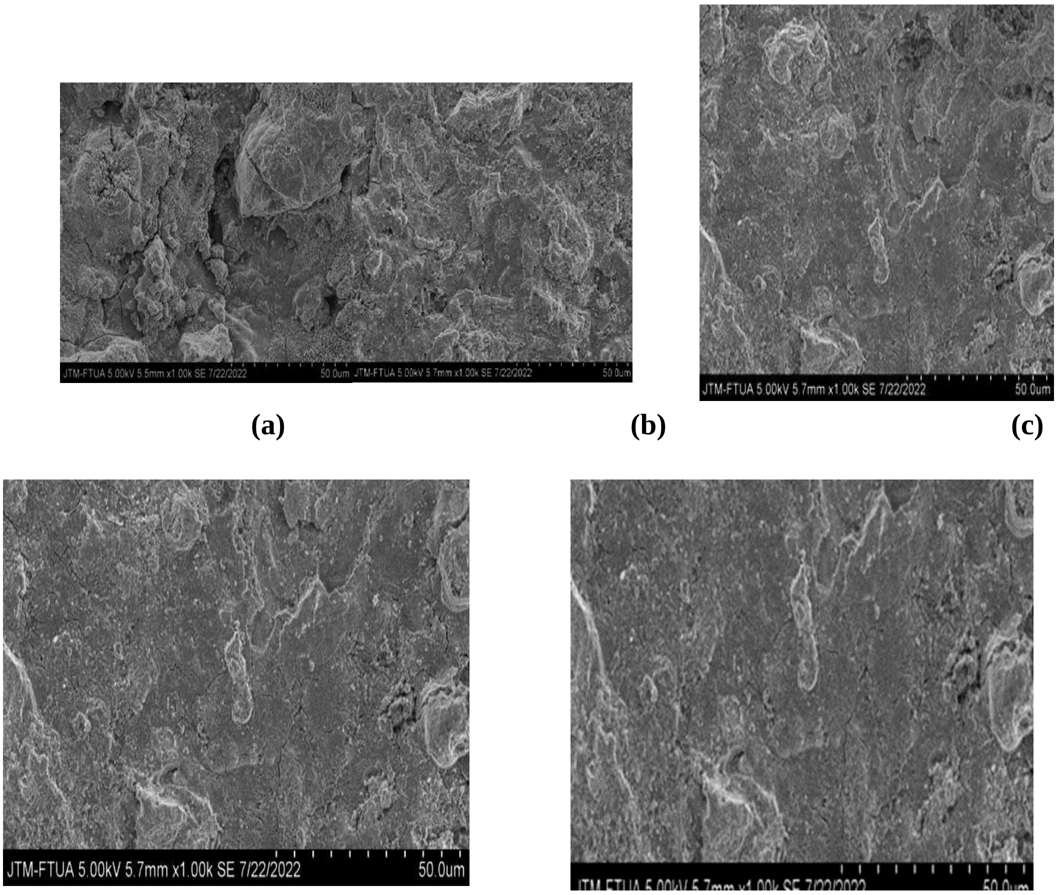


Figure 2. Morphological results of steel surfaces using an optical microscope in 1.5 M HCl medium for 144 hours with variations in immersion time in inhibitors (a) without treatment, (b) without inhibitors, (c) 24 hours, (d) 72 hours, (e) 120 Hours, (f) 168 Hours

The results of observing the surface morphology of the steel without and with added inhibitors in 1.5 M HCl corrosion media using an optical microscope can be seen in Figure 2. Figure 2a shows that the surface of the steel without treatment looks smooth because it has notbeen contaminated with oxygen. Whereas Figure 2b is immersed in a corrosive medium without an inhibitor, it can be seen that the surface is very rough and has been damaged due tocorrosion. Figures 2c to 2f immersion in 1.5M HCl with various inhibitors 24, 72, 120, and 168hours), it can be observed that the surface morphology of the steel has corrosion, and it becomesthinner with increasing immersion time in the inhibitor. This proves that the more adsorption of cocoa pod extract on the steel surface the corrosion rate decreases.



(a) **(b)** **(c)**

(d) (e)

Figure 3. Morphological results of steel surfaces using a SEM in 1.5 M HCl medium for 144hours with variations in immersion time in inhibitors (a) without inhibitors, (b) 24 hours, (c)72 hours, (d) 120 hours, (e) 168 Hours

The surface morphology characterization of the steel was also carried out using the SEM with a magnification of 1000x. Figure 3e is a sample with the lowest corrosion rate when immersed in inhibitor for 168 hours in HCl for 144 hours. Figure 3 shows that the longer it is immersed in the inhibitor the more adsorption of cocoa pod extract occurs on the steel surface.So that the adsorption in the form of a layer is able to protect the metal and its parts so that thecorrosion rate can be prevented. In the figure it can be seen that the longer the immersion timein the inhibitor the smoother the resulting surface, because the corrosion rate decreases, and the damage caused by corrosion decreases. The layer formed is more even with lots of adsorption formed on the surface, although there are still cracks and bubbles due to immersionin HCl. This proves that an increase in immersion time affects the morphology of the steel surface. Due to the long soaking time, there will be many interactions between the tannin polyphenolic compounds and iron. This will make the tannin compound more absorbed into the steel surface, thus forming a complex compound with Fe (III) which will protect the steel surface from corrosion attack (13-14)

3.1 X-Ray Diffraction Analysis

Characterization with X-Ray Diffraction (XRD) was carried out on samples with the highest and lowest corrosion rates in 1.5 M HCl for 144 hours with variations in the time of immersion in the inhibitor 24, 72, 120 and 168 hours. The results of the XRD characterization are in the form of a diffractogram between the angle of 2θ and the intensity as shown in Figure 4. The results of the X-Ray Diffraction characterization are in the form of a peak-to-peak graph wherethe y axis represents the peak intensity and the x axis represents the measured diffraction angle.To find out the crystalline phases and compounds contained in the immersing steel samples, aqualitative analysis was carried out on the XRD results, where the intensity data and diffractionpeak positions obtained were then matched with ICDD (International Center for Diffraction Data) reference data using the search match analysis method or data matching method.

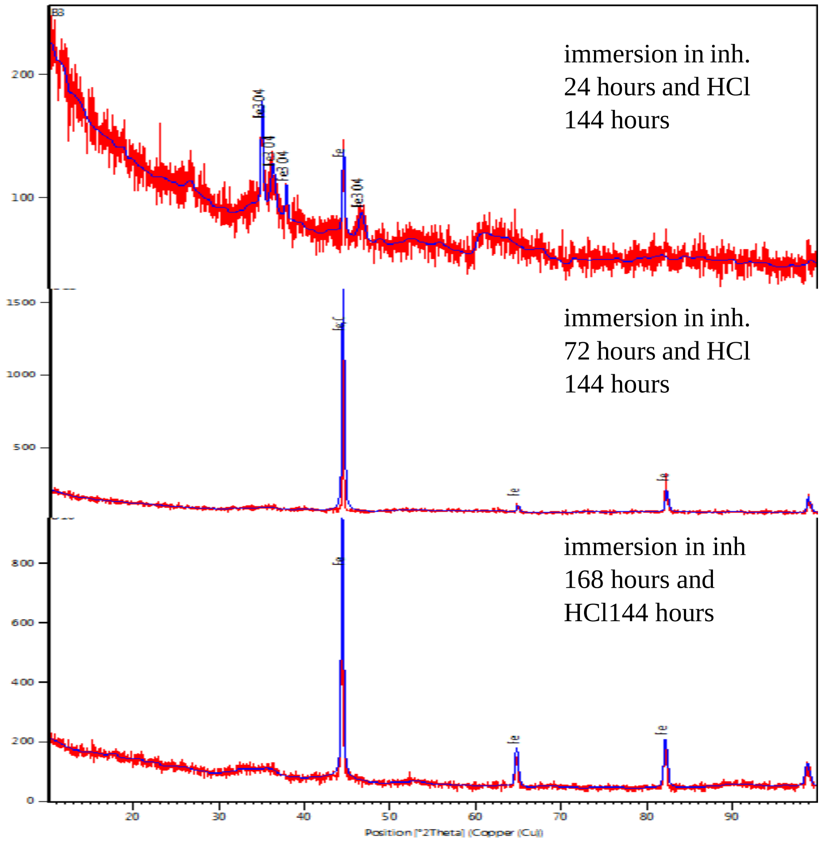


Figure 4. XRD characterization results and for samples (a) 24 hours, (b) 72 hours, and (c) 168hours

Table 5. 2θ Value and Highest Peak Intensity XRD Curves of Steel Samples in 1.5M HCl for144 Hours and Immersion in Inhibitors for 24 Hours

Peak	2θ	Intensitas	FWHM	d [Å]	Fasa
1	35.0251	100.00	0.3070	2.56198	Fe_3O_4
2	36.2815	44.67	0.6140	2.47609	Fe
3	37.8466	32.74	0.3070	2.37722	Fe_3O_4
4	44.5142	83.68	0.3070	2.03541	Fe_3O_4
5	46.6377	25.79	0.8187	1.94756	Fe_3O_4

The results of the first part of the XRD characterization showed that the sample with the highest corrosion rate contained magnetite (Fe_3O_4) as an excessive corrosion product. The magnetite phase is formed due to the interaction between Fe and OH^-. The highest intensity islocated at the first peak at position 2θ, namely 35.0251° with an intensity of 100% which is thepeak of Fe_3O_4. The lowest intensity is located at the fifth peak with position 2θ which is 46.6377° and an intensity of 25.79% which is also the peak of Fe_3O_4. It can be observed in thefirst graph Figure 11 and Table 5 states that the first, third, fourth and last peaks in this samplehave a magnetite (Fe_3O_4) phase, while the second peak has an iron (Fe) phase, because the steelsurface has not been coated with inhibitors and is experiencing a degree of high corrosion.

Table 6. 2θ Value and Highest Peak Intensity XRD Curves of Steel Samples in 1.5M HCl for144 Hours and Immersion in Inhibitors for 72 Hours

Peak	2θ	Intensity	FWHM	d [Å]	Phasa
1	44.3699	100.00	0.3070	2.04169	Fe, C
2	64.7198	14.85	0.4093	1.44037	Fe
3	82.1028	20.30	0.4093	1.17389	Fe
4	98.6537	9.70	0.5117	1.01648	Fe

Table 7. 2θ Value and Highest Peak Intensity XRD Curves of Steel Samples in 1.5M HCl for144 Hours and Immersion in Inhibitors for 168 Hours

Peak	2θ	Intensity	FWHM	d [Å]	Phasa
1	44.2716	100.00	0.3070	2.04600	Fe
2	64.6859	3.35	0.3070	1.44104	Fe
3	82.0503	11.89	0.3070	1.17451	Fe
4	98.6194	8.82	0.3070	1.01674	Fe

From Figure 4, the second and third sections show that there is no significant difference in the results of the XRD testing for the two samples from the peaks of the XRD graphs. Each produces four sharp peaks indicating the formation of a crystalline phase. Table 6 proves that the resulting intensity is very high for the Fe and C phases, namely 100% with the 2θ positionof 44.3699o, while for the other three peaks it produces the Fe phase. This stated that the samplecontained a lot of Fe and C elements because the longest immersion in the inhibitor was 168 hours, as a result the reaction that occurred was a reaction between Fe and tannin compounds from cocoa shell extract.

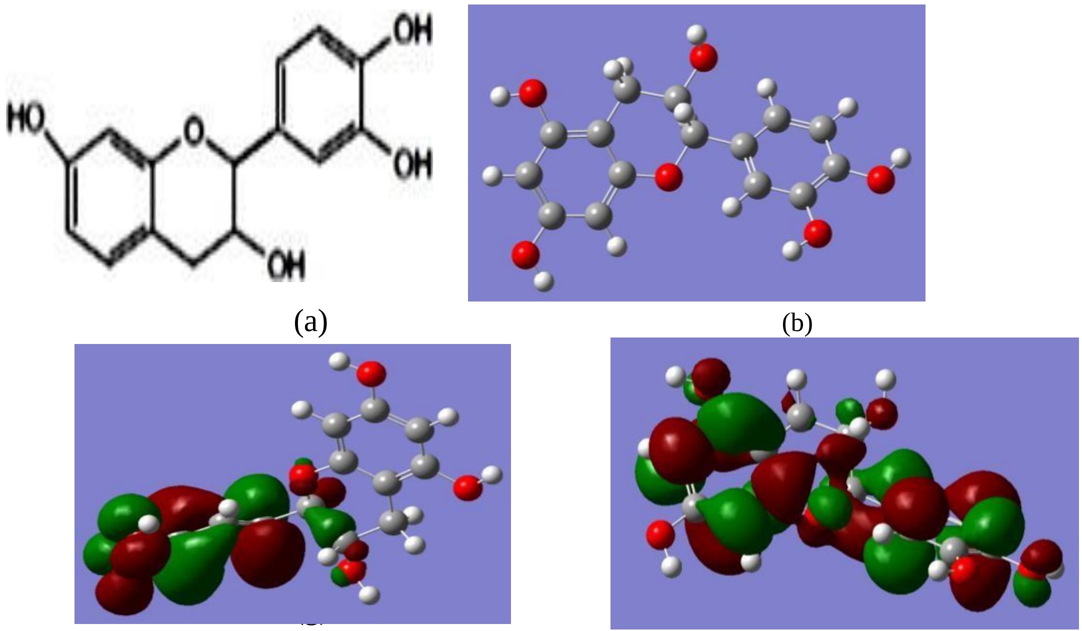
The samples started in Table 7 give XRD characterization results that are not much different, this is evidenced in Figure 4 which consists of four sharp peaks, where the highest intensity on the first peak is at position 2θ 44.2716°, while the lowest intensity is 8.82%. at position 2θ 98.6194°. Samples with the longest variation of immersion time in inhibitor and HCl had four peaks with the same phase, namely Fe. This indicates that the sample still containsa lot of Fe and this is in accordance with the research conducted by Bundjali et al [29] that lowcarbon steel produces the Fe phase.

3.2 Analysis of AFM

The AFM analysis results show that the adsorption formed on the steel surface is formed gradually according to the increase in the immersion time of the inhibitor used. The longer it is immersed in the inhibitor, the more adsorption is formed on the steel surface. The adsorptionforms a thin layer on the surface which can form a complex with tannins from the extract. Thecomplex that is formed is able to withstand attacks from corrosive ions that attack the steel surface, so that it is able to withstand the corrosion rate [9, 14, 17].

3.3 Structure of Tannin and Tannin-Fe Compounds

Corrosion inhibitor is a compound that can inhibit corrosion on a metal, one of the corrosion inhibitor compounds is tannin. Tannin compounds can be used as inhibitors because they have lone pairs of electrons and π electrons in their molecular structure. The tannin compound was optimized by applying Gaussian 16W using the DFT method and B3LYP/6-31G basis set.



(a) (b)

(c) (d)

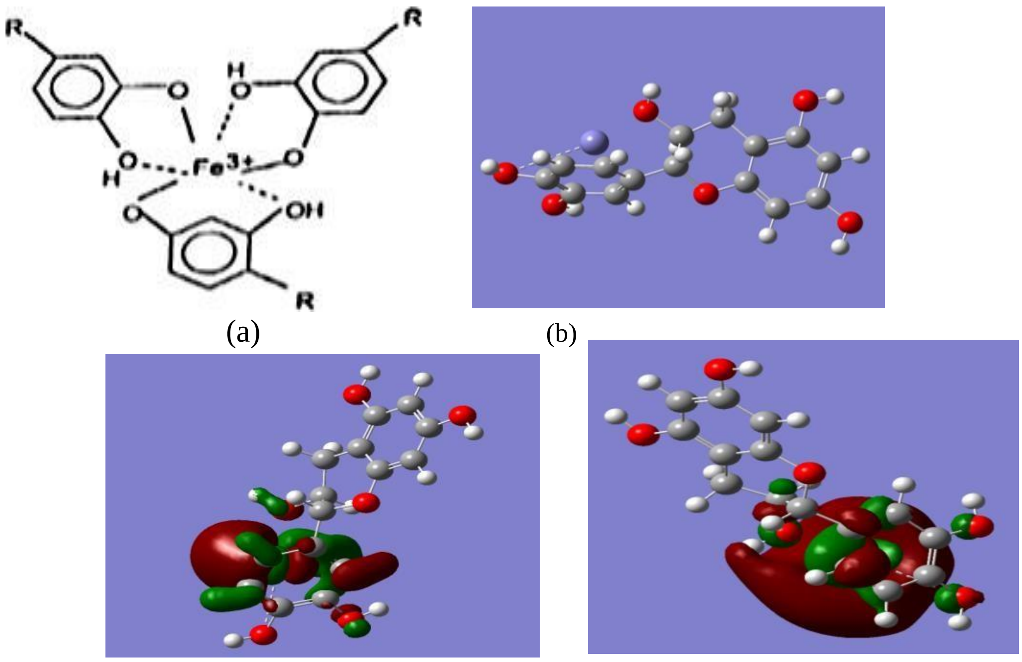
Figure 5. Optimization Process for Tannin Compounds, (a) Molecular Structure, (b) Geometry Structure, (c) HOMO Contour, and (d) LUMO Contour

Figures 5 and 6 show the molecular structure, optimal geometric structure, HOMO and LUMOcontours for pure tannin and tannin-Fe compounds. The HOMO and LUMO contours can predict absorption centers and inhibitor molecule interactions. The HOMO contour shows areas that are electron donors while the LUMO contour shows areas that are electron acceptors. It can be seen in Figure 5 that the HOMO and LUMO contours are centered on tannin compounds,this indicates that tannin compounds have good properties as electron donors and acceptors, while in Figure 6 it can be seen that the HOMO and LUMO contours are centered on tannin compounds containing Fe atoms. This explains that when tannins are reacted with Fe, there will be a good donor and electron transfer between tannin compounds and Fe [20, 24].

The HOMO energy indicates the ability of a molecule to donate electrons. The greater the HOMO energy, the easier it is for a molecule to donate electrons, while the LUMO energyis the ability of a molecule to accept electrons. The smaller the LUMO energy, the easier it is for the molecule to accept electrons. The inhibitor molecule functions to donate its lone pair ofelectrons to ferrous metal, so that the more reactive inhibitor is the one with the greater

HOMOenergy and large LUMO energy [19, 25].

Corrosion inhibition efficiency (EI %) of tannins was calculated using the DFT- B3LYP/6-31G method. Table 9 shows that through quantum chemical calculations, the inhibition efficiency of tannin compounds is high, namely 80.2098%, while the maximum inhibition efficiency obtained in this study is 74.7128%. This proves that between experimentsand tests with the Gaussian software the results are not much different, which means that tannin compounds have a good ability to reduce or inhibit the corrosion rate of steel by forming complex compounds with iron, so that the steel surface will be covered and protected. from corrosion attack [20, 25].



(a) (b)

(b) (d)

Figure 6. Optimization Process for Tannin-Fe Compounds, (a) Molecular Structure, (b) Geometry Structure, (c) HOMO Contour, and (d) LUMO Contour

Table 9. Efficiency of Corrosion Inhibition of Tannin Compounds

Iadd (%)	EIadd	EIteori (%)
7,3575	0,05497	80,2098

4 Conclusion

Based on the results of research that has been done, it can be concluded as follows:

1. The corrosion rate obtained in a corrosive medium with the same time and concentration is inversely proportional to the immersion time in the inhibitor. The increase in immersion time in the inhibitor causes the corrosion rate to decrease. The lowest rate was obtained for samples with immersion time in the inhibitor for 168 hours.
2. The morphology of optical microscopy and SEM for samples immersed in 1.5 M HCl corrosive media for 144 hours with variations in the immersion time in the inhibitor shows that the longer the sample is immersed in the inhibitor, the more adsorption is formed on the surface of the sample.
3. XRD analysis produced a crystalline phase for samples with the highest corrosion rate,namely the presence of a magnetite phase, while for samples that were soaked in inhibitors for longer periods, they produced Fe and C phases.
4. Quantum chemical characterization results in molecular structure, geometry, EHOMO and ELUMO, as well as other parameters which support that tannin compounds are ableto interact with Fe and produce good inhibition efficiency in inhibiting corrosion, whichis 80.2098%.

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