Article

Environmentally Friendly Maltodextrin-Chitosan Encapsulation in Cocoa Husk Extract (*Theobroma cacao* Linn.) for Corrosion Inhibition on Steel in Corrosive Media

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Abstract

This research tested an encapsulated cocoa husk extract formulated with maltodextrin and chitosan at an 8:2 ratio for use as a steel corrosion inhibitor. The method of weight loss in 0.75 M sulfuric acid, seawater, and peat water solutions was used to test inhibitors. The analysis indicated that inhibitor efficiency increased with increasing concentration. At the same time, its efficiency decreased due to longer immersion periods, reaching a maximum of 94.07% in peat water when a 2.5 g/L concentration of inhibitors was applied for 1 day. These results indicate significant potential for encapsulated cocoa husk extract as a natural corrosion inhibitor.

Keywords: *Cocoa Husk Extract (Theobroma cacao Linn.); Corrosion Inhibitor; Corrosive Media; Encapsulation*

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Graphical Abstract

Introduction

Corrosion is a major degradation process that causes substantial damage to metallic materials, leading to significant economic losses worldwide. The detrimental effects of corrosion compel industries to allocate considerable financial resources for maintenance and repair of damaged equipment. Sectors most affected by corrosion include industrial facilities as well as reinforced concrete and steel-framed buildings [1]. Various methods have been developed to mitigate corrosion, such as surface coating, cathodic protection, and the use of corrosion inhibitors. Among these methods, corrosion inhibitors are widely applied due to their effectiveness in reducing corrosion rates while extending the service life of metallic material [2].

Recently, an increasing attention has been directed toward the use of plant-based corrosion inhibitors owing to their several advantages such environmental friendliness, biodegradability, and low toxicity. Plant extracts are rich in antioxidant compounds that can function as natural corrosion inhibitors. These extracts inhibit corrosion primarily through the adsorption of active compounds containing functional groups such as hydroxyl (–OH) and amine (–NH), as well as conjugated double bonds, which act as the main adsorption centers on the metal surface [3]. Several plant extracts have been reported as effective corrosion inhibitors, including *Andrographis paniculata* leaf extract [1], cinnamon (*Cinnamomum burmannii*) leaf extract [3], *and Macaranga gigantea bark extract* [5].

Cocoa husk extract is another promising natural corrosion inhibitor due to its high content of bioactive compounds such as tannins, polyphenols, flavonoids, alkaloids, and saponins. These compounds exhibit strong antioxidant properties and have been reported to inhibit metal corrosion effectively. In particular, tannins play a significant role in corrosion inhibition

through the formation of protective complexes on metal surfaces [4,5]. Previous studies have demonstrated that cocoa husk extract can achieve a corrosion inhibition efficiency of up to 74.7%.

Despite its potential, the application of cocoa husk extract as a corrosion inhibitor is limited by the instability of its active compounds, which are susceptible to degradation under environmental conditions such as variations in pH, exposure to oxygen, and light. One effective strategy to overcome this limitation is encapsulation. Encapsulation involves coating an active core material with a protective wall material to enhance its stability and controlled release [6]. An ideal encapsulating material should possess good emulsifying properties, film-forming ability, high solubility, and chemical inertness toward the core material. In this study, maltodextrin and chitosan were employed as encapsulating agents for cocoa husk extract. The corrosion inhibition performance was evaluated using the weight loss method. Surface morphology of the steel specimens was characterized using scanning electron microscopy (SEM), while the functional groups present in the encapsulated inhibitor were analyzed using Fourier-transform infrared (FTIR) spectroscopy.

Materials and Methods

Materials

Cocoa (*Theobroma Cacao* Linn.) husks, distilled water, 96% ethanol (By Mart), mild steel (Fe = 98.5%, C = 0.19%, Si = 0.22% and Mn = 0.654%), H_2SO_4 p.a (Merck), sea water (pH 7.8), peat water (pH 2.75) taken from Muaro Jambi Regency, tannic acid (Merck), folin denis reagent (Merck), Na_2CO_3(Merck), maltodextrin (Indo Food Chem), chitosan (Aldrich) , and acetone (By Mart).

Cocoa Husk Extraction (Theobroma Cacao Linn.).

Cocoa Husk (*Theobroma cacao* Linn.) was taken from the Pandan Makmur village of the Geragai sub-district of the Tanjung Tabung Timur Regency of the province of Jambi. After that, the filtered cocoa husk was pulverized into powder. This powder was then extracted with 96% alcohol using a 1:3 ratio for a total of three days. The solution was filtered using filter paper to obtain the filtrate. This was followed by re-soaking the sediments in alcohol for another 3 days. This re-soaking procedure was completed a total of three times. Lastly, the filtered solution was evaporated to obtain the concentrated liquid extract.

Formulation of Encapsulated Products.

The procedure for forming encapsulated products is based on the research of Musdalifa *et al*. [7] with modifications. The preparation of 50 mL of encapsulation solution was carried out by weighing maltodextrin and chitosan at a concentration of 10% (w/v) of the encapsulant solution. Maltodextrin and chitosan were weighed according to the treatment (10%: 0%; 9.5%: 0.5%; 9%: 1%; 8.5%: 1.5%; and 8%: 2% w/w) and then added with distilled water to 50 mL. The mixture was stirred using a magnetic stirrer until dissolved, then cocoa shell extract of as much as 1% of the volume of the encapsulant solution and immediately homogenized with a homogenizer for 30 minutes, then poured into a petri dish with a thickness of 3 mm and dried at a temperature of 50 ± 5^{o}C. The dried solid was ground and filtered with a 40-mesh sieve. The product results were tested for water content, and the best results were then tested for solubility in seawater, peat water, and 0.75 M sulfuric acid and tannin stability tests.

Encapsulation Moisture Content

Moisture content analysis refers to the research of Noviyanti *et al* [8]. The bottle containing the sample is dried for 1 hour in an oven at 105 ^{o}C, then weighed, and then 1 gram of sample is added (a), put into the sample bottle, dried in an oven for 4 hours at 105 C, then cooled in a desiccator for 15 minutes and reweighed. This procedure is repeated until a constant weight (b) is obtained. The moisture was calculated using the equation 1.

$$\text{Moisture Content (\%)} = \frac{(a-b)}{a} \times 100\% \quad (1)$$

Description: a is the weight of the sample before heating (g), b is the weight of the sample after heating (g)

Encapsulation Solubility

Encapsulation solubility analysis refers to the research of Noviyanti *et al* [8]. A sample of 100 mg of encapsulated product was added with 10 mL of water and then gelatinized at a temperature of 90 °C - 95 °C for 30 min with moderate stirring. Furthermore, it was cooled to room temperature and centrifuged at 2000 rpm for 30 min. The supernatant was weighed and put into an aluminum cup and dried in an oven at a temperature of 105 °C for 4 h until the weight was constant. Solubility was calculated using the equation 2 [9].

$$\text{Solubility (\%)} = \frac{M1}{M2}\ x\ 100\ \% \quad (2)$$

Description: M_1 is the weight of solids in the supernatant (g), M_2 is the weight of the supernatant sample (g)

Determination of Tannin Stability

Tannin, a secondary metabolite in cocoa pod extract, was used to benchmark the effect of storage time on encapsulated and unencapsulated extracts. Both the encapsulated and unencapsulated cocoa pod (Theobroma cacao) extracts were stored in the dark at room temperature for 0, 3, 6, 9, 12, and 15 days. The decrease in total tannin content was measured at these intervals. Tannin content was quantitatively assessed using the method described by Warnasih *et al* [10].

Weight Loss

To determine the effect of inhibitor concentration and immersion time on the efficiency of corrosion inhibition on mild steel in various corrosion media, including 0.75 M sulfuric acid solution, seawater, and peat water, the weight loss method is employed. Weight loss method was carrie out, which is based on the difference between the initial and final mass of mild steel after treatment in a corrosion medium mixed with cocoa husk (*Theobroma cacao* Linn) extract encapsulation. The corrosion rate and corrosion inhibition efficiency on mild steel are then determined using equation 3.

$$\text{Corrosion Rate} = \frac{(W0-Wf)}{A\ x\ t} \quad (3)$$

Description: r is the corrosion rate ((g))/(cm^2.day)); W_0 is the initial weight of iron (g); W_f is the final weight of iron (g); A is the surface area of the iron plate (cm^2); T is the immersion time (days)

$$\text{Inhibition Efficiency (\%E)} = \frac{r1-r2)}{r1} \times 100\ \% \quad (4)$$

Description: %E is Inhibition Efficiency (%) ; r_1 is corrosion rate without inhibitor ((g))/(cm^2.day)); r_2 is corrosion rate with inhibitor ((g))/(cm^2.day)

Result and Discussion

Phytochemical Screening

Cocoa husk extract contains secondary metabolite compounds, namely alkaloids,

flavonoids, phenolics, tannins, and saponins, while terpenoid compounds showed negative test results (Table 1). Based on the data in Table 1, these compounds show positive results for flavonoids, phenolics, tannins and saponins containing heteroatoms O, N, and/or S and have double bonds and free electron pairs that can be used as corrosion inhibitors. [4,8,9]. The results of quantum analysis indicate that molecules with electron pairs can donate electrons. Free electron pairs and double bonds can be adsorbed on the surface of mild steel to form a thin layer as a protector from corrosive environments [4].

Table 1. Results of phytochemical screening of cocoa husk extract

No	Parameters	Results
1	Alkaloids	Positive (+)
2	Flavonoids	Positive (+)
3	Phenolic	Positive (+)
4	Tannin	Positive (+)
5	Saponins	Positive (+)
6	Terpenoids	Negative (-)

Moisture Content

The lowest moisture content for various formulations was obtained by encapsulating with a maltodextrin: chitosan (8:2) coating ratio, as shown in Table 2. It shows that the moisture content becomes lower with the addition of chitosan. The decrease in moisture content is due to an increase in solids in the powder's solids content. Using chitosan as an encapsulant with maltodextrin can also prevent the particle surface from crystallizing, allowing more water inside the particle to evaporate [12]. The lowest moisture content was found in the encapsulation with an encapsulate ratio of 8:2, which was 3.51%. Lower moisture content provides stability against degradation of active ingredients, while higher water content can increase humidity and shorten shelf life due to the presence of microbes [13]. In this study, further data analysis was used on cocoa husk extract encapsulation with maltodextrin-chitosan 8:2

Table 2. Results of measurement of water content of cocoa husks extract encapsulation

No	Encapsulation Comparison (Maltodextrin : Chitosan)	Moisture Content (%)
1	10 : 0	6.82
2	9.5 : 0.5	5.73
3	9 : 1	5.68
4	8.5 : 1.5	4.88
5	8 : 2	3.51

Solubility

Cocoa husk extract encapsulation with maltodextrin-chitosan (8:2) is more soluble in peat water than in seawater and sulfuric acid (Figure 1). Chitosan is easily soluble in weak acid solutions. At acidic pH conditions, chitosan molecules become polycationic and cause chitosan molecules to be protonated to produce NH_3^+ ions, which affects the increase in zeta potential value. At pH > 7. Chitosan undergoes deprotonation so that chitosan molecules are precipitated due to the formation of hydrogen bonds between molecules, and the solubility of chitosan decreases. In addition, in several types of acids, such as phosphoric acid and sulfuric acid, the solubility of chitosan is very low, only around 0.5-1.1% [13], [14] [15].

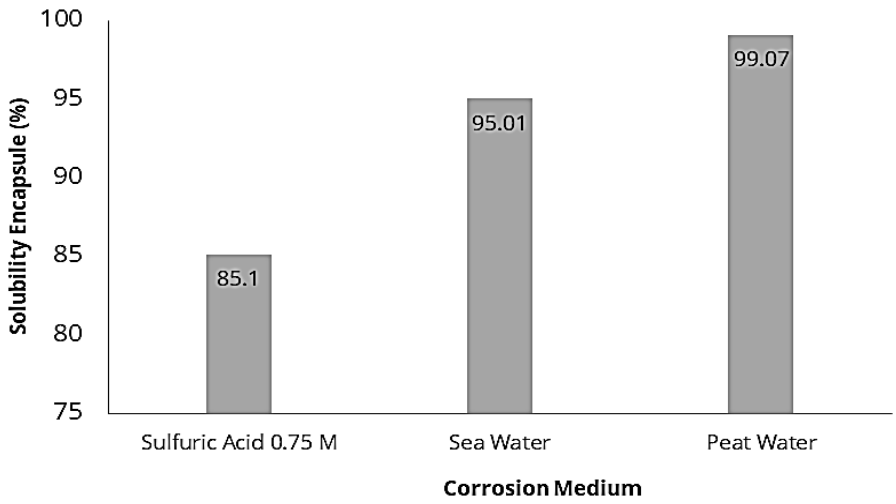

Figure 1. Comparison of the Solubility of Cocoa Husk Extract Encapsulation with Maltodextrin-Chitosan (8:2) in 0.75 M Sulfuric Acid Solution, Seawater, and Peat Water

Tannin Stability

Tannin Stability. A UV-Vis spectrophotometer was used to measure the maximum wavelength of tannic acid before testing the total tannin content of cocoa husk extract encapsulation and cocoa husk extract without encapsulation.[16]. The maximum wavelength of tannic acid obtained in this study was 750.2 nm. The presence of tannin in cocoa husk extract acts as a corrosion inhibitor. [17]. The results of the tannin stability test are shown in Table 3.

Table 3. Results of tannin stability analysis during storage time

Time Storage (Days)	Tannin Level (mg/L)		% Decrease in Tannin Levels	
	Un-encapsulated Extract	Encapsulated Extract (Encapsul)	Un-encapsulated Extract	Encapsulated Extract (Encapsul)
1	100.901	87.2745	0.00%	0.00%
2	89.137	87.2745	11.65%	0.00%
3	77.862	87.1764	22.83%	0.11%
4	71.294	86.7843	29.34%	0.56%
5	66.882	86.2941	33.71%	0.98%
6	62.764	85.5098	37.79%	2.02%

As shown in Table 3, the tannin content in the unencapsulated cocoa husk extract decreased by 37.79% after 15 days of storage, whereas the encapsulated cocoa husk extract decreased by only 2.02%. This demonstrates that encapsulation effectively protects and maintains the stability of secondary metabolite compounds, such as tannins, by preventing oxidation from environmental influences [18]

Weight Loss Analysis

Based on the variation of concentrations used, namely 0.5, 1.0, 1.5, 2.0, and 2.5 g/L, it shows a decrease in the corrosion rate along with the increasing concentration of extract from cocoa husk (Figure 2).). The results obtained in Figure 2 are in accordance with research conducted by [19] that the corrosion rate will decrease with the

increase in inhibitor concentration. The greater the inhibitor concentration, the smaller the corrosion rate that occurs in steel. Within 1 day, it shows that the decrease in corrosion rate is still relatively slow, but with increasing immersion time, the decrease in corrosion rate is getting faster. According to [20] [21]. This is due to the presence of tannin compounds in the extract. These compounds in the extract can form complex compounds with Fe(III) on the metal surface, thereby decreasing the corrosion reaction rate. This complex compound will prevent the attack of corrosive ions on the metal surface, thereby decreasing the corrosion reaction rate.

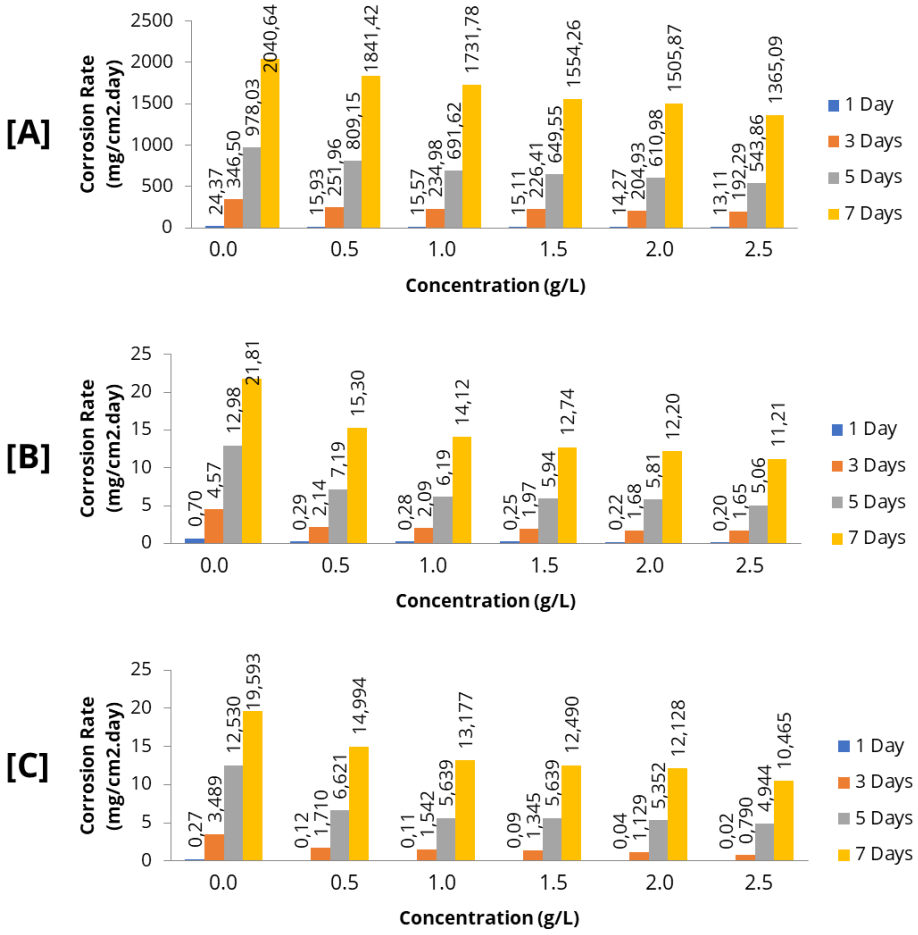

Figure 2. The effect of the concentration of encapsulated cocoa husk extract inhibitor (*Theobroma cacao* Linn.) and soaking time on the corrosion rate of steel in media (a) 0.75 M sulfuric acid (b) sea water, (c) peat water

Based on the time variations used, it shows the influence of time on the corrosion rate of steel (Figure 3). The longer the immersion time, the higher the corrosion rate on the steel. This is because the longer immersion time provides a greater opportunity for corrosion reactions to occur. The increase in corrosion rate with time is due to weakening of the interaction between secondary metabolites from cocoa husk extract (*Theobroma cacao* Linn.) on the surface of mild steel. Figure 3 shows that a relatively high corrosion rate occurs when the sample is immersed in sulfuric acid without an inhibitor. It happens because sulfuric acid is highly acidic, which allows it to attack steel. Acidity level affects the corrosion process because pH indicates the concentration of H^+ ions in water, which can accelerate ion exchange and alter the release of electrons

in metals[2]. The results of the tannin stability test are shown in Table 3.

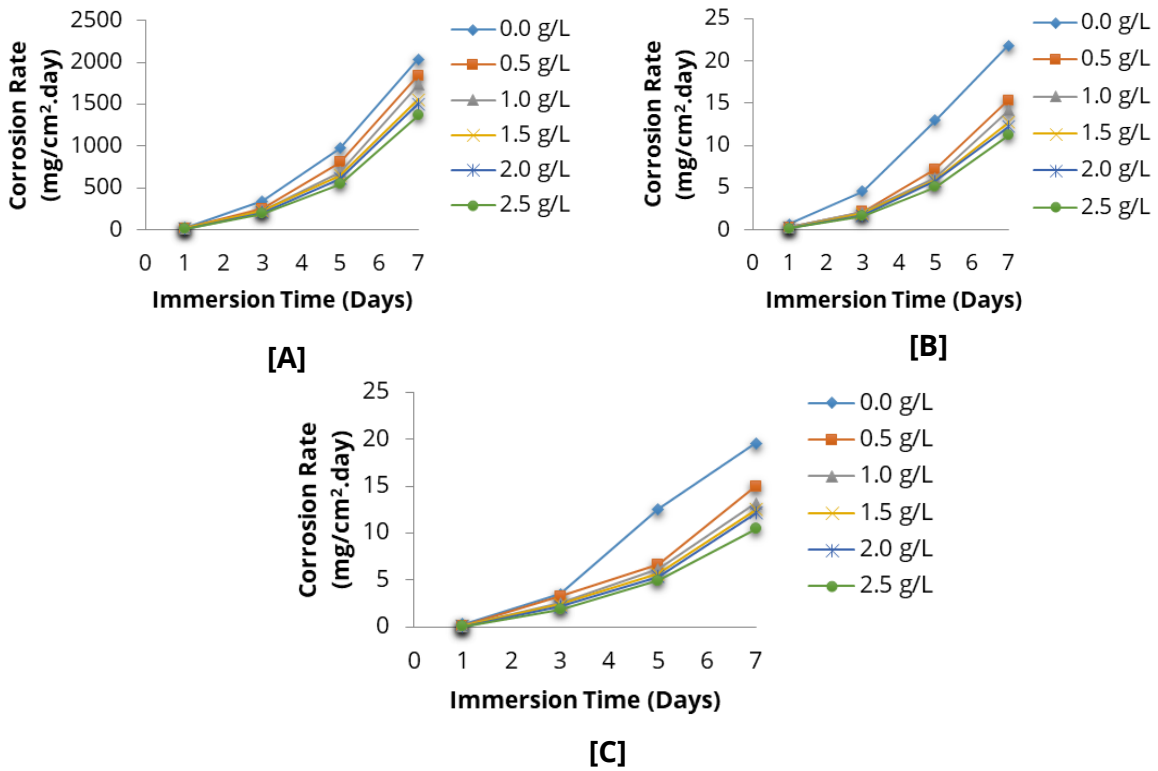

Figure 3. The effect of soaking time and concentration of encapsulated cocoa husk extract inhibitor (*Theobroma cacao* Linn.) on the corrosion rate of steel in media (a) 0.75 M sulfuric acid (b) sea water, (c) peat water

Effect of Cocoa Husk Extract (Theobroma cacao Linn.) Encapsulated Inhibitor Concentration and Soaking Time on Inhibition Efficiency

The inhibition efficiency of encapsulated cocoa husk extract (Theobroma cacao Linn.) was strongly influenced by both inhibitor concentration and soaking time, as shown in Figure 5. Increasing the inhibitor concentration from 0.5 to 2.5 mg/mL resulted in a consistent and significant improvement in inhibition efficiency at all immersion times. This trend indicates that higher concentrations provide a greater availability of active compounds, particularly tannins and polyphenols, which adsorb onto the steel surface and form a protective barrier that limits the access of corrosive species. As the concentration increases, surface coverage by inhibitor molecules becomes more extensive and compact, thereby enhancing corrosion resistance

These results are consistent with established corrosion inhibition theory, which states that inhibition efficiency increases with inhibitor concentration due to enhanced adsorption and improved film continuity on the metal surface. The encapsulation of cocoa husk extract using maltodextrin and chitosan further contributes to this behavior by stabilizing the active compounds and promoting controlled release, allowing more effective and sustained interaction between the inhibitor and the steel surface.

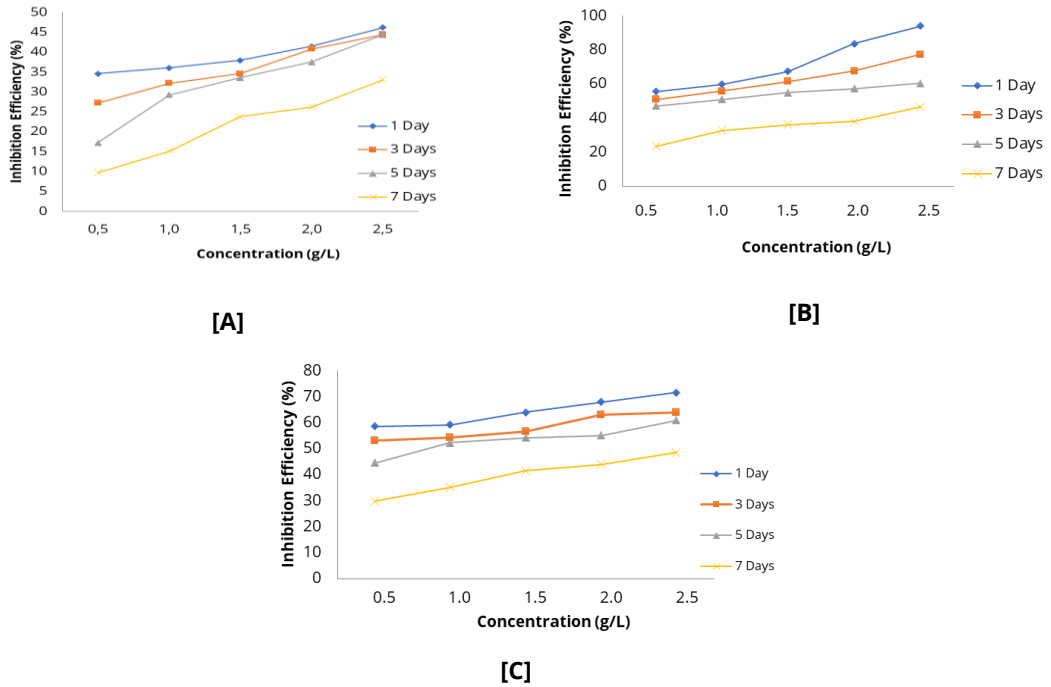

Figure 4. The effect of the concentration of encapsulated cocoa husk extract inhibitor (*Theobroma cacao* Linn.) and soaking time on the efficiency of steel inhibition in media (a) 0.75 M sulfuric acid (b) sea water, (c) peat water

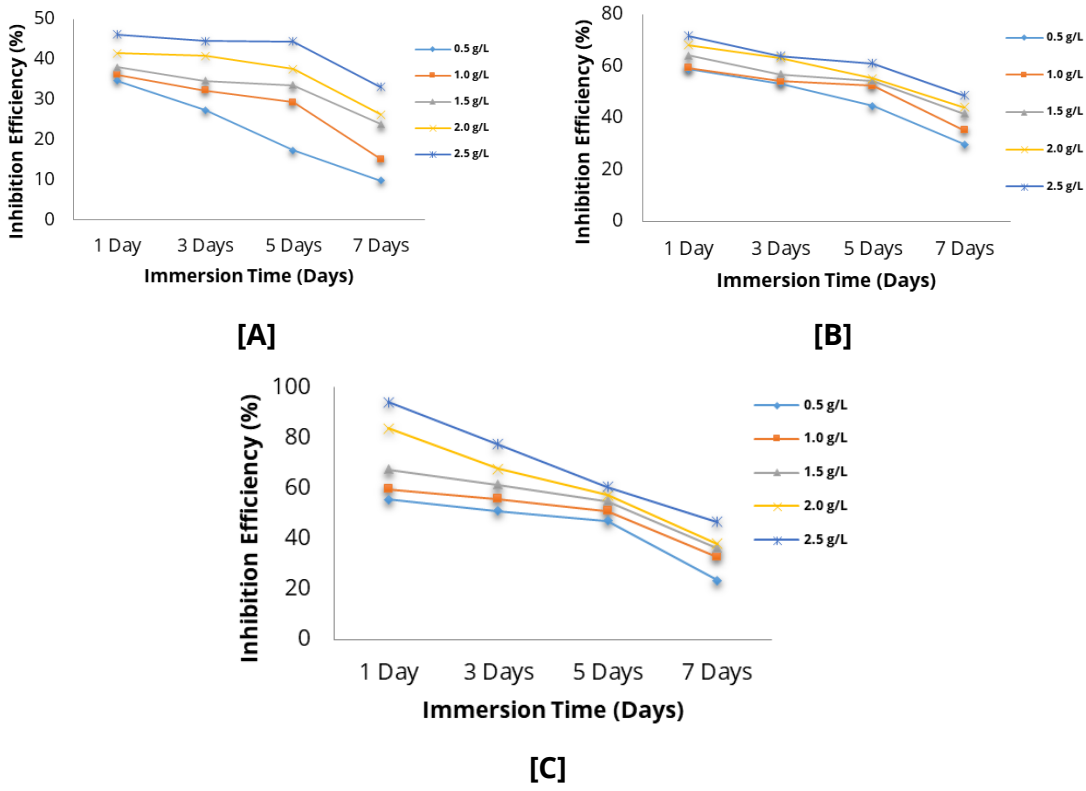

Figure 5. The effect of soaking time and concentration of encapsulated cocoa husk extract inhibitor (*Theobroma cacao* Linn.) on the efficiency of steel inhibition in media (a) 0.75 M sulfuric acid (b) sea water, (c) peat water

In contrast, inhibition efficiency decreased with increasing soaking time, even at constant inhibitor concentrations. This decline can be attributed to the gradual

degradation or oxidation of active phenolic compounds during prolonged exposure to the corrosive environment, which reduces their adsorption capability and protective effectiveness. Additionally, extended immersion may lead to partial desorption or deterioration of the inhibitor film, exposing portions of the steel surface to the corrosive medium. Although encapsulation improves the stability of the extract, the results indicate that its protective performance diminishes over extended soaking periods.

Overall, the findings demonstrate that the corrosion inhibition performance of encapsulated cocoa husk extract is maximized at higher inhibitor concentrations and shorter soaking times. These results confirm the potential of encapsulated cocoa husk extract as an effective and environmentally friendly corrosion inhibitor, while emphasizing the importance of optimizing both concentration and exposure duration for practical applications.

Surface Morphology Analysis using Scanning Electron Microscopy (SEM).

Based on Figure 6, the 0.75 M sulfuric acid corrosion media create the roughest, most uneven, and most pitted steel surface appearance, followed by seawater corrosion media and then peat water. This may indicate that the corrosiveness of 0.75 M sulfuric acid is greater than that of seawater, followed by peat water without the addition of inhibitors.

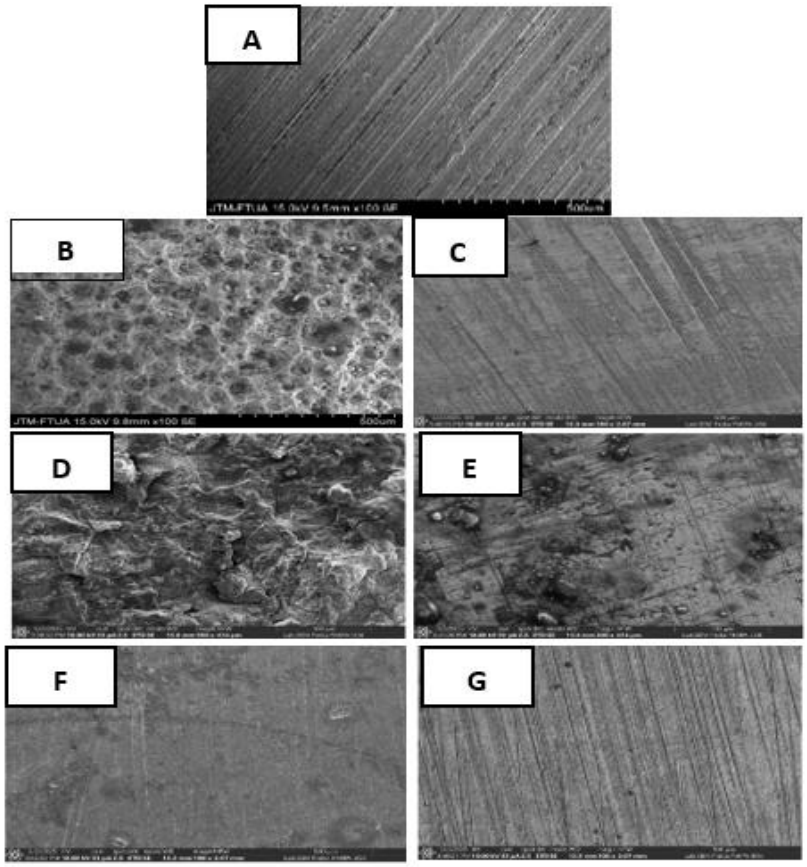

Figure 6. SEM results of steel: a) before treatment, b) after immersion in 0.75 M H_2SO_4 without inhibitor, c) after immersion in 0.75 M H_2SO_4 with the addition of encapsulated inhibitor, d) after immersion in seawater without inhibitor, e) after immersion in seawater with the addition of encapsulated inhibitor, f) after immersion in peat water without inhibitor, g) after immersion in peat water with the addition of encapsulated inhibitor.

Figure 6 also shows that the presence of cocoa husk extract encapsulation as a corrosion inhibitor can protect steel, so that the steel surface is only slightly pitted and looks covered by a protective layer, with the best surface appearance being with cocoa husk extract encapsulation inhibitor in peat water corrosion media, then followed by sea water, and then 0.75 M sulfuric acid.

Functional Group Analysis

Figure 7 shows the results of wavenumber absorption in FT-IR spectroscopy for maltodextrin (7A), chitosan (7B), cocoa husk extract (7C), encapsulation of cocoa husk extract (7D), adsorption layer on steel in sulfuric acid with the presence of encapsulation of cocoa husk extract (7E), adsorption layer on steel in seawater with the presence of encapsulation of cocoa husk extract (7F), and the adsorption layer on steel in peat water with the presence of encapsulation of cocoa husk extract (7G). Figure 7 is the result of the FTIR spectra. Absorption wave numbers for each compound. It was found that the cocoa husk extract encapsulation (Figure 7D) had a wave number absorption pattern similar to that of maltodextrin (Figure 7A), chitosan (Figure 7B), and cocoa husk extract (Figure 7C). Adsorption layer on steel in sulfuric acid with the presence of encapsulation of cocoa husk extract (Figure 7E), adsorption layer on steel in seawater with the presence of encapsulation of cocoa husk extract (7F), and the adsorption layer on steel in peat water with the presence of encapsulation of cocoa husk extract (7G). The FTIR spectrum of the research results, as compared to the literature, is presented in Table 4.

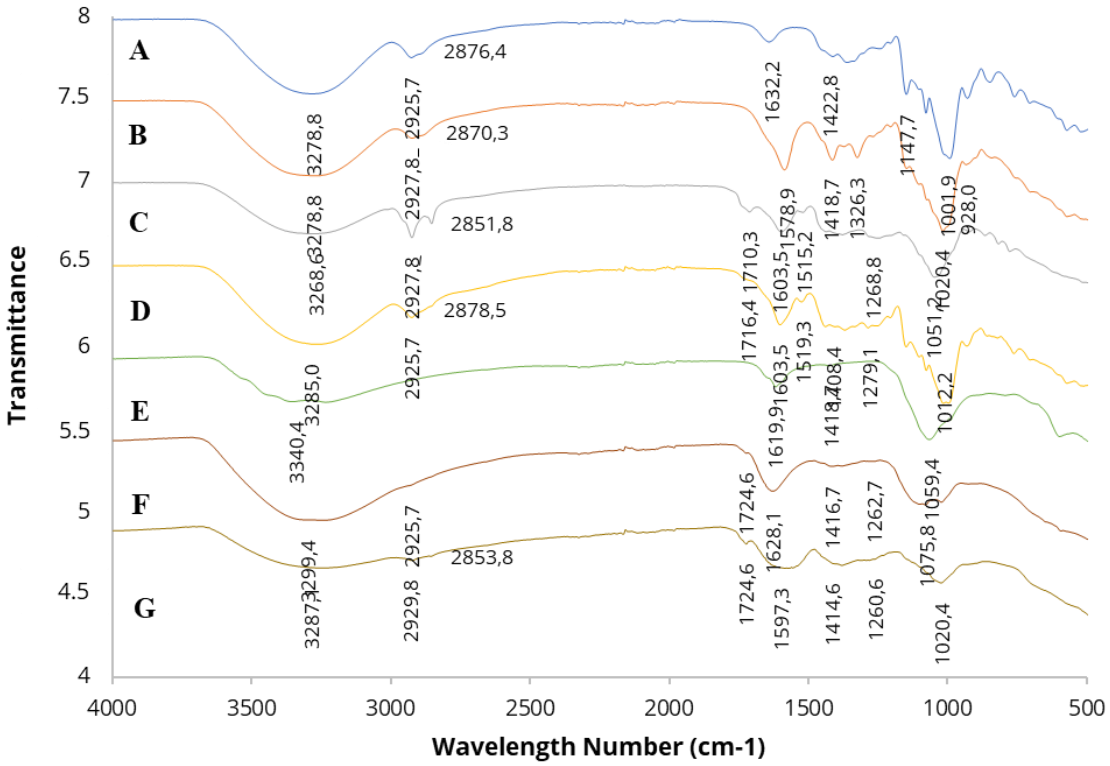

Figure 7. FTIR spectra of maltodextrin (7A), chitosan (7B), cocoa husk extract (7C), encapsulation of cocoa husk extract (7D), adsorption layer on steel in sulfuric acid with the presence of encapsulation of cocoa husk extract (7E), adsorption layer on steel in seawater with the presence of encapsulation of cocoa husk extract (7F), and the adsorption layer on steel in peat water with the presence of encapsulation of cocoa husk extract (7G)

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Table 1. Comparison of FTIR Spectrum Obtained with Literature

A	B	C	D	E	F	G	References [22] [23]	Functional Group
3278.8	3278.8	3268.6	3285.0	3340.4	3299.4	3287.1	3600-3200	O-H
-	3278.8	3268.6	3285.0	3340.4	3299.4	3287.1	3500-3100	N-H
2925.7	2927.8	2927.8	2925.7	-	2925.7	2929.8	3000-2850	C-H
-	-	1710.3	1716.4	-	1724.6	1724.6	1760-1690	C=O
1422.8	1578.9 & 1418.7	1515.2	1519.3 & 1408.4	1418.7	1416.7	1519.3 & 1414.6	1620-1400	C=C
1001.9	1020.4	1051.2	1012.2	1059.4	1075.8	1020.4	1300-1000	C-O
-	1326.3	1268.8	1279.1	1059.4	1262.7	1260.6	1350-1000	C-N
-	-	1603.5	1603.5	1619.9	1628.1	1597.3	1690-1590	C=N

Figures 7E, F, and G show similarity patterns with Figures 7A, B, C, and D. When compared with Figure 6d, the shift in wave numbers in Figure 7F and G is seen in the OH group, namely, in Figure 7d there is absorption in the 3285.0 cm^{-1} region shifting to 3340.4 cm^{-1}, 3299.4 cm^{-1}, and 3287.1 cm^{-1} in Figure 7E, F, G, respectively. Then there is a shift in the wave number for the C=O functional group, which was originally at a wave number of 1716.4 $cm^{-1,}$ shifting to 1724.6 cm^{-1} in Figures 7F and G. Furthermore, there is a shift in the wave number for the CN functional group, namely at wave number 1279.1 cm^{-1} shifting to 1262.7 cm^{-1} and 1260.6 cm^{-1} in Figure 7 F and G and there is also a shift in the wave number for the CO functional group, namely at wave number 1012.2 cm^{-1} shifting to 1059.4 cm^{-1}, 1075.8 cm^{-1}, and 1020.4 cm^{-1} in Figure 7E, F, G respectively. Thus, there is a shift in the wave number for the OH, C=O, CN, and CO functional groups due to the interaction between these functional groups and the steel surface, where these functional groups interact with Fe^{2+} on the steel surface through coordination bonds which cause the formation of a protective layer on the steel surface that can protect the steel from corrosion attacks. Functional groups with heteroatoms (oxygen and nitrogen) and double bonds (C=O, C=C, and C=N) are observed in the product spectrum.k (coating) mild steel corrosion containing cocoa shell extract encapsulated inhibitor. The presence of organic compounds containing nitrogen, oxygen, sulfur and double or triple bonds facilitates absorption on the metal surface, forming a protective barrier that minimizes the corrosion process [22]

Conclusions

The best formulation was obtained with maltodextrin:chitosan with a ratio of 8:2,

which was then used as a steel corrosion inhibitor. The weight loss method was used in 0.75 M sulfuric acid solution, seawater, and peat water to test the inhibitor. The analysis showed that the inhibitor efficiency increased with increasing concentration. At the same time, its efficiency decreased due to longer immersion periods. The high inhibition efficiency reached a maximum of 94.07% in peat water when an inhibitor concentration of 2.5 g/L was applied for 1 day. These results indicate the significant potential of encapsulated cocoa shell extract as a natural corrosion inhibitor.

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Author Contributions

The contribution of each author to this article is : "Conceptualization F.T and D.R.G; Methodology, F.T., D.R.G., A.N and F.F ; Software, F.A., T.R., and D.R.G..; Validation, D.R.G., A.N., and N.A.T.,.; Formal Analysis, F.T., D.R.G., and F.F.; Resources, D.R.G., A.N; Writing – Original Draft Preparation, .F.A and D.R.G; Writing – Review & Editing, D.R.G., A.N., N.A.T; Supervision, D.R.G.; Project Administration, D.R.G.; Funding Acquisition, F.A., D.R.G.

Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

Ethical Standards

This article does not contain any studies involving human or animal subjects.

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