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Mechanical properties of mild steel by adding *Theobroma Cacao* Peels Extract (TCPE) inhibitor

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Abstract. The ability of corrosion inhibitor *Theobroma cacao* Peels Extract (TCPE) has been tested to improve the mechanical properties of mild steel after corrosion occurred. These properties were examined to measure its hardness, tensile and fatigue. Scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDX) and atomic force microscopy (AFM) was used to analyze the morphology of the surface. The corrosion rate was found reducing, on the contrary, the efficiency was increasing as the increasing concentration on the extract. This raising was followed by an increase in mechanical properties, namely hardness, strength and fatigue strength. Indeed, the presence of absorption on the surface of the data was reinforced by EDX, X-ray photon spectroscopy (XPS), and AFM for topography. The addition of polar extract of cacao peels in HCl 1.5M is very useful to reduce the corrosion rate on the mild steel surface, and it can retain its mechanical properties after the corrosion occurred.

1. Introduction

Corrosion of metals is a major problem that must be confronted for safety, environment, and economic reasons. It can be minimized by suitable strategies which in turn stifle, retard or completely stop the anodic or cathodic reactions or both [1]. Stell corrosion will decline the quality due to the chemical or electrochemical reaction between the steel and its environment. Hence, the electrochemical reactions take place in a neutral environment with the complex reaction:

$$\begin{array}{l} Fe + {}_{½}\,O_2 + H_2O \longrightarrow Fe^{2+} + 2OH \quad Fe^{2+} + 4OH^- \longrightarrow 2Fe(OH)_2 \longrightarrow \\ 2Fe(OH + ½O_2 \longrightarrow 2FeO\,(OH)\,H_2O \qquad 2H_2O + Fe_3O_4\ \text{(corrosion product)} \end{array} \quad (1)$$

Many methods have been done to slow the corrosion rate such as electroplating, galvanizing and inhibitor [2]. The inhibitor is one way to minimize the effect of material degradation that is often used. It slows the rate by forming a protective layer on the metal surface [3]. It is usually slightly added in the acidic environment, cooling water, steam, or other environments.

Recently, the inhibitor is the best solution to protect the metal corrosion. It is a flexible method of protection. It protectons the environment to be less aggressive on the very high corrosion level environment, and easy to apply. However, the cost of effectiveness is higher due to the very thin form of layer reason. Therefore a small amount can provide broad protection [4].

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In the present, many researchers develop various types of inhibitors, both organic and inorganic. Inorganic substances are like phosphates, chromates, dichromates, silicates, borates, tungstates, molybdates, and arsenates have been found useful as inhibitors of metal corrosion. Pyrrole and derivatives are believed to exhibit good protection against corrosion in acidic media. These inhibitors have also found a useful application in the formulation of primers and anticorrosive coatings. Otherwise a significant disadvantage is on their toxicity, and likely their use has come under severe criticism. [5]. Under these conditions, people nowadays switch their preference to use organic inhibitors which are derived from natural materials.

The new inhibitor is expected to reduce the corrosion rate of materials, especially carbon steel material. The use of corrosion inhibitors will protect against the failure of material due to the declining quality of corroded carbon steel. The use of organic inhibitors is a method to solve the corrosion problems as it is non-toxic, biodegradable and environmentally friendly [6]. The use of natural ingredients from plants such as extracts of leaves, bark, and seeds [7] has been done to get environmentally friendly inhibitors. Previous studies show that some extracts of natural materials can be used for the corrosion inhibitor purpose. For example: Oxandra asbeckii plant [8], Musa paradisica peel [9], Asteriscus graveolens [10], Spirulina pantesis [11], Molasses [12], Zizyphus spina Christi [13], Sebasnian sesban [14], Chlomolaena odorata L [15], Pongamia piñata seed [16], Mymecodia pendans [17], and Theobroma cacao peel [18]. These natural compounds have proven its ability to act as a corrosion inhibitor for some metals and alloys in several different aggressive media. Cacao peels extract an inhibitor which is capable of improving the efficiency of inhibition of mild steel up to 96% [3]. Many inhibitors inhibit corrosion by absorption to form an invisible thin layer with the thickness of several molecules. Some are caused by environmental influences in the form of visible precipitate and protects the metal from the attacks that corrodes it and produce products that form a passive layer. Meanwhile, some others eliminate aggressive constituents.

By the increasing number of cacao production in Indonesia to one million ton/ year, resulting in the production of cacao peels increases as well. This raise holds tremendous promise for cacao peels extract to be an environmental friendly inhibitor, which can retain and restore the mechanical properties of steel after the corrosion.

2. Material and method

2.1. Preparation Materials

Equipment and materials of this study which included the preparation of the extracts of cacao, mild steel, and media corrosive hydrochloric acid (HCl), has been described in the previous article [3]. As a note, the cacao peels extract as an inhibitor was obtained from maceration. The concentration of inhibitor used was varied, they are 0.0; 0.5; 1.0; 1.5; 2.0 and 2.5%. Commercial plain carbon steel plate which was used for general construction is taken as the sample. Bars form of mild steel was cut 12 mm in diameter and 2-3 mm in thickness for the specimen of corrosion rate determination. Subsequently, the preparation specimens for mechanical properties test was based on ASTM standards. All of the specimens were sanded until they were smooth and finally washed them with acetone. Media corrosive hydrochloric acid took Merk as the brand with 1.5 M in concentration. The rate of Corrosion Testing Methods applied the three methods which have been comprehensively described in previous articles [3], [18].

2.2. The Corrosion Rate of Testing Methods

Weight loss, potentiodynamic and impedance were the method taken to determine the rate of corrosion. The working procedure and the formula used by these three methods have been clearly described in the previous articles [3], [17].

2.3. The Surface Morphology of Steel

The previous article has described corrosion surface morphology analysis that took *Stereo Carton Trinoculer Photo Optic* and S-3400N *Scanning Electron microscope*. Morphology analysis of the surface aims to see mild steel surface with and without cacao peels extract in the corrosive medium of HCl 1.5 M. *Electron Dispersion X-Ray* (EDX) *EMAX software* was utilized to determine the composition of the elements contained in the steel surface with and without the inhibitor. Besides, XPS and XRD were chosen to determine the type of absorption elements on the surface, whereas AFM to view its topography.

2.4. Absorption Surface

Their absorption on the surface was one of the parameters to determine the effectiveness of the inhibitor extract from the natural product that is used. XPS analysis is taken to determine the absorbed elements on the surface. The work of AES-XPS tool ESCA 2000 which was based on the existence of high-resolution separation of the binding energy on electrons in the core level emitted by the photoelectric effect would come from the X-ray irradiation. Simply, XPS worked based on photon sources which were derived from the X-ray irradiation, which passed on a sample. Electrons were located near the core or inner bark which emitted out, and then captured by the analyzer and detected in the form of binding energy of the electrons in the core rate. The bond energy of electrons near the core of the interface/ software is displayed in the form of bonds to the intensity of the energy spectrum. The bond energy eventually interpreted as the presence of certain molecules or atoms.

Atomic Force Microscopy (AFM), the nanosurs type, is a tool for viewing and manipulating atoms nano dimension (size <100 nm). AFM is widely used to investigate the structure, function, and absorption on the surface. This device is capable of displaying images that are smaller than 20 ms. This microscope might also display images of soft crystals and the polymer surface [19].

2.5. Testing Mechanical Properties

The mechanical properties will test tensile, hardness and fatigue of mild steel, with and without extract of cacao peels in the corrosive medium of HCl 1.5 M. Tensile test used a *Universal Testing Machine type RAT-30p CAP 30tf*. The test specimen was prepared in accordance with ASTM E-8 standard. The Rockwell Hardness Tester TH 550 with the load 980,7N is utilized to determine surface hardness of mild steel. The tested preparation includes cutting and sanding, where the load and time pressure were set in accordance with the desired result [20]. Meanwhile, the fatigue specimen test applied the ASTM E466-2002 procedure.

3. Result and discussion

3.1. The corrosion rate analysis

To be noted that further discussions would be the continuance of the previous article [18]. Referred to the results of corrosion test by implementing the three methods, it showed that there might be the addition of inhibitors which minimizes the weight loss. Besides that, it slowed down the rate of corrosion of the initial rate before inhibitor was given. Conversely, the increase of inhibitor concentration raises the efficiency of corrosion inhibition on mild steel surface as well, as shown in Fig.1. It was due to the larger the steel surface that had contact with the solution, the more steel surface coated with the polar extract of cacao peels. The phenomenon correspondently matched the protection mechanisms that occurred. It confirmed that cacao peels extract contained polyphenol compounds that had a lone pair of electrons [3]. This atom served as electron donors so that it can produce a complex compound with iron. It resulted in the stable complex compounds that were uneasily oxidized and covered the steel metal surface. Thus, it inhibited the corrosion rate [21].

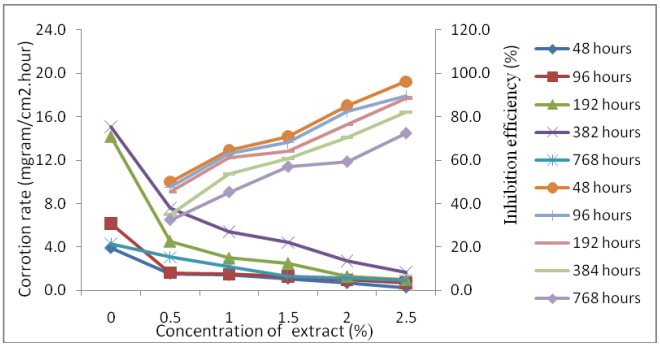


Figure 1. Corrosion rate and Inhibition efficiency against Concentration of extract.

3.2. Analysis of Mechanical Properties

The testing result of hardness, tensile, and fatigue for mild steel is shown in Table 1. Tensile, hardness, and fatigue presented an increase with the concentration on the extract, supported by the data of mechanical properties. The increasing concentration of the extract at the same time increases the resistance of steel against corrosive ions in the corrosive media so that the rate of corrosion can be inhibited. It means that the higher the extract concentration, the higher the amount of extract absorbed on the surface, made the surface area of mild steel covered by peels extract of cacao [20]. The extract chemically absorbed on the surface and formed a thin film coating which was difficult to be damaged [22- [23].

Table 1. The mechanical properties of mild steel in HCl 1.5M with various concentrations of cacao peels extracts.

Consentration of extract (%)	Hardness (HRB)	Tensile strength (MPa)	Mean fatigue strength (MPa)
Mild steel (as received)	90,04	691.1	700
0,0	52,84	495.2	550
0,5	56,27	540	600
1,0	62,29	581.8	650
1,5	64,47	601.9	700
2,0	67,97	658.4	700
2,5	72,24	675	700

There were 26 pieces samples for fatigue test which was treated similarly by using various concentrations on the extract of HCl 1.5M like in the previous research. This test was performed based on ISO 12107 standard, with a statistical estimate where the fatigue strength was given in fatigue life.

The test conditions are as follow: f = 10 Hz, R = 0.1 (tensile-tensile), Fatigue limit (given fatigue life): 1 x 105 cycles, UTS S35C measured by a tensile strength after being given 875 MPa inhibitor cacao peels extract. The strength of S35C fatigue, after experiencing immersion in a solution of HCl was dropped from 675 MPa to 550 MPa. Hence, its condition was back to the original force after receiving additional inhibitor as seen in Table 1. Undoubtedly, the fatigue strength increased by the raising of concentration because of the additional inhibitor. It was due to the absorption of the cacao peels extracts on steel surfaces. Indeed, the series of mechanical properties that have been carried out on the steel remarked that there was improvement happened on mechanical properties after more inhibitor added.

3.3. Topography analysis

SEM micrograph of some sample surfaces can be seen in Fig. 3, whereas a relatively smooth surface of the polished sample befortoe immersion seen in Fig. 3a. Notably, the surface of the specimens after immersion in the TCPE inhibitor only showed a thin surface layer covering the entire surface of mild steel (Fig. 3b). Fig. 3c and 3d highlights the steel surface morphology after experiencing 32 days immersion in a solution of corrosive hydrochloric acid 1.5 M with and without the addition of the cocoa peels extract.

Meanwhile, heavy damage was found on the surface of mild steel due to heavy corrosion attack of the acid (Fig. 3c). Seen that there was a surface crack on the rust layer, and it became initial crack and stress concentration sites. This condition was not only strongly related to the loss of elongation (embrittlement) but also responsible for the decrease of the strength and hardness of the steel. The presence of TCPE inhibitor was able to diminish corrosion attack so that the rust disappeared from the surface (Fig. 2d). Obviously, 2.5% addition of TCPE inhibitor within the acid could resist corrosion rate by forming a full barrier on the surface of mild steel.

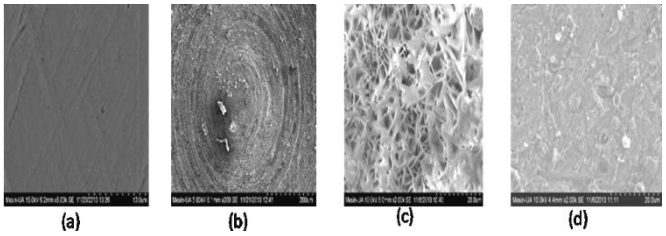


Figure 2. SEM images of mild steel in 1.5M HCl after 32 days immersion at room temperature (a) initial surface (polished) (b) immersion in inhibitor only, (c) immersion into HCl without inhibitor, (d) immersion in HCl with 2.5 % inhibitor.

The amount of extract absorbed on the surface illuminated by the EDX test was shown in Table 2. To some extent, the increase on carbon content on the surface of mild steel increased both mechanicals properties of mild steel and covered surface [24]. The rise on the hardness surface would decrease the rate of corrosion [20] details, third relations mechanical properties can be seen in Table 1.

Table 2. Content of the elements identified in EDX testing

Treatment	Contain element (% weight)		
	C	Fe	O
Mild steel	0.32	98.79	-
Mild steel + Extract	6.19	92.66	4,33
Mild steel + HCl 1.5M	1.5	29.39	63.54
Mild steel + HCl 1.5M + Extract	16.9	37.43	44.89

3.4. XPS analysis

The XPS test was aimed to see the coating film formed by the absorption on the steel surface as shown in Fig. 4. The spectrum appeared in Fig. 4a showed that the coating film on the surface contained elements of Fe, O, and C. Meanwhile, Fig. 4b explained that bond of carbon (C, C-C, C=C) that emerged at the top of 284.5 eV was detected. It was the formation of carbon-oxygen single bonds (-CO) on 285,7ev also a carbon-oxygen double bond (-C=O) on 288.4ev, and a combination of single and double bonds (OC= O) on 289,3ev [27]. The data mentioned above was supported by the analysis of FT-IR spectrum at a wavelength of 1019 cm-1, 1162 cm-1, 1435-1459 cm-1 and 1617-1654 cm-1

[3]. The combination was assumed as a complex compound peels extract of cacao with oxygen in the form of $TCPE_2O_3$, TCPE $(OH)_3$ and TCPE-O-C product.

However, the O1s spectrum occurred, representing the signal of 529,8ev oxide and oxygen of the hydroxyl group at 531,4ev and 533,0ev as seen in Fig. 4c [27]. The manifested hydroxyl groups in XPS analysis was matched with the results of FT-IR analysis at a wavelength of 3376-3422 cm^{-1}. Fig. 4d pointed out there was a peak appeared which was consisted of a mixture of iron corrosion products like Fe3O4, Fe2O3, FeO, $FeCO_3$, and FeOOH, at 710 and 724ev [28], [29]. The oxides analysis results from XPS then was strengthened by FT-IR for mild steel oxide at 668 cm^{-1} on wavelength.

The surface absorption supported by the analysis results of surface topography using Atomic Force Microscopy (AFM) ParkNX10 was clearly shown that there had been an absorption on the steel surface (see Fig. 4). Fig. 4a pointed out the mild steel surface after being polished with sandpaper. However the surface smoothness was uneven. Whereas, Fig. 4b pointed out the mild steel surface after immersed in cacao peels extracts. It was a visible absorption occurred on the steel surface which was coated by its extracts in the form of a thin smooth and flat layer. Fig. 4c pointed out the mild steel surface after immersed into HCl 1.5M solution.

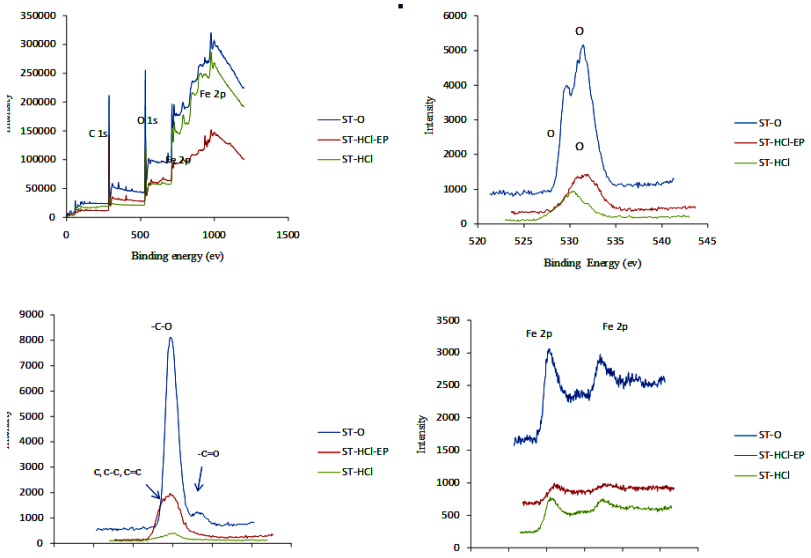
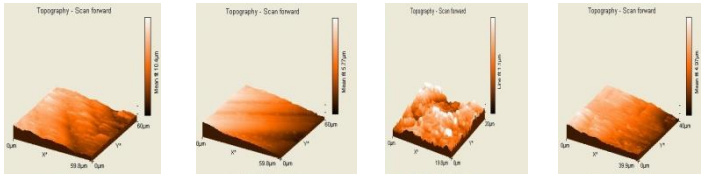


Figure 3. XPS spectrum of (a). Samples were analyzed, (b). Inhibits oxygen and are not inhibits, (c). Carbon absorbed, and (d). Fe which appears on the surface of mild steel



a. b. c. d.

Figure 4. a. Only mild steel, b. Mild steel + Extract, c. Mild steel + HCl, d. Mild steel + HCl + Extract

The picture above showed an uneven surface because it was damaged by a corrosive solution and was visible. Then in Fig. 5c could be seen that this damage was minimized by the addition of cacao peels extracts into HCl 1.5M solution. It's result mentioned in Fig. 5d. The holes of the former damage which then covered by the extracts were absorbed on the surface of the mild steel.
As a result of this absorption, the corrosion rate and the damage could be reduced.

3.5. Corrosion Inhibition Mechanism

The inhibitor molecule on the mild steel surface, occurred because of the absorption. Where this absorption was due to the adhesion force between the inhibitor with the mild steel surface. Absorption inhibitor molecules on the surface of mild steel produced a thin layer (film) which inhibited the corrosion rate. In this case, the extract inhibitor of cacao peels formed a thin layer on the surface that served as a control of the corrosion rate by separating between the steel and corrosion media [25]. The absorption process of this peels extract was in functional groups [22].

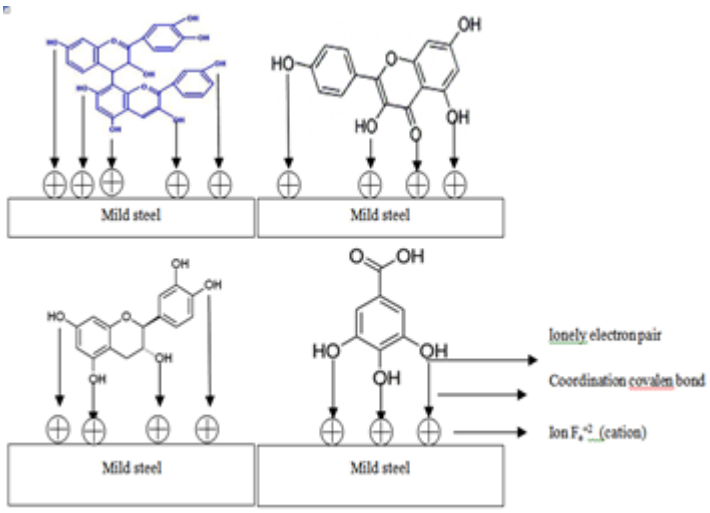


Figure 5. The absorption process cacao peels extracts compound on the surface of mild steel.

The absorption process could explain the inhibition mechanism on the surface of the steel and the components structure which was contained in its extract (see Fig. 6). This inhibition was due to the absorption of phytochemical components contained in the extract through oxygen and/or nitrogen atoms on a metal surface [22]. This complex structure might be absorbed onto the surface of the mild steel by van der Waals force to form a protective layer, to prevent the mild steel from corrosion [26]. Some of the major components of this cacao peels extract were catechin, campferol, gallic acid, and prosianidin (see Fig. 5) [18]. All of these compounds have heteroatoms groups that could donate an electron to form a complex compound on the surface of the mild steel. On the other hand, the effective inhibitor performance also depended on the size of phytochemical constituents of this extract to interact with the metal surface to slow the rate of corrosion [22], [27].

The absorption mechanism in Figure 6 explains that the absorption has formed a thin layer on the steel surface [8], [18]. These layers are wider with the increase of extract concentration which was added to the corrosive solution. This was supported by the data of the closure surfaces degree which increased after the extract concentration was added (see Fig. 7). The result of EDX analysis in Table 2 also stated the increasing composition of the elements contained on the surface after the addition of the cacao peels extracts.

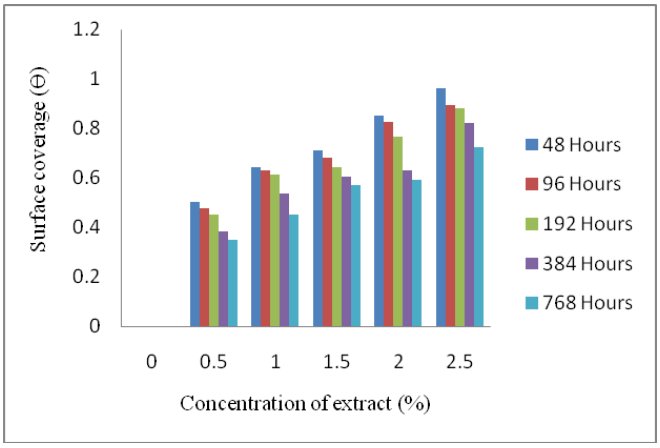


Figure 6. Surface coverage against Concentration of extract in HCl 1.5M

4. Conclusion

The increase of the cacao peels extracts concentration makes the surface coverage on the surface of mild steel increasing as well. The extent of the surface coverage is strongly influenced by surface absorption on the surface. Certainly, the increasing concentration of cacao peels extract will increase surface absorption on the surface. Absorption is formed due to the interaction between the donor atoms of the extract with a mild steel surface. The mechanical properties of mild steel can influence this absorption. As a result when the level of carbon absorption on the surface increase, the mechanical properties of hardness, tensile and fatigue strength also increase.

Overall, it can be said that polar extract of cacao peels can improve the mechanical properties of the mild steel which has been attacked by corrosions.

Acknowledgement

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