Coconut Fiber Extraction using Soda Pulping Method as Green Corrosion Inhibitor for ASTM A36 Steel

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Abstract. Currently, the corrosion inhibitor is a method that is widely used to control the corrosion rate of inner pipe in the chemical industry. This study aims to utilize coconut fiber as a green inhibitor, determine the effect of adding inhibitor concentration, immersing time, and determine the inhibition efficiency of coconut husk extract. The extraction method used is the soda pulping method with a solid to liquid ratio of 1:8 (w/v). The method used for testing the corrosion rate is the weight-loss method, UV-Vis Spectrophotometry and FT-IR are also used to analyze the lignin produced. The lowest corrosion rate was obtained in 1 M HCl corrosive medium with the addition of 3 g/L inhibitors at 48 hours immersion, which was 49.691 mpy. The highest inhibition efficiency was in 1 M HCl corrosive media solution with the addition of 3 g/L inhibitors at 48 hours immersion, which was 78.11%.

1. Introduction
Corrosion is damage caused by a chemical reaction between a metal, both alloy, and steel in an environment. Corrosion can be controlled or slowed down by coating the metal surface, cathodic protection, adding corrosion inhibitors, etc. To prevent proper corrosion in protecting the inside of the pipe can be done with the addition of corrosion inhibitors. Inhibitors are a flexible method of protection, capable of protecting metals in very high corrosive environments, easy to apply, and the most cost-effective. The inhibitor layer is formed very thin so that in small amounts it can provide broad protection, Komalasari et al [1]

Corrosion inhibitors can be obtained from organic or inorganic materials. Inorganic compounds are used as inhibitors such as nitrite, chromate, phosphate, and urea. These compounds are hazardous chemicals, expensive, and not environmentally friendly, Pradpta et al [2]. Meanwhile, organic compounds produced from plants can be an option because they are ecologically acceptable, inexpensive, abundantly available, environmentally friendly and, contain atoms of N, O, P, S and, other atoms that easily form complex bonds/compounds against metal and steel, Marques et al [3].

Natural ingredients contain organic compounds that have the potential as inhibitors. One of the contents of natural extracts that can be used as corrosion inhibitors is tannin and lignin, Mulyaningsih et al [4]. Lignin is a complex molecule composed of phenylpropane units bonded in a three-dimensional structure. Lignin has the ability to form complex compounds because it has elements that have lone pairs that function as electron donors to metals. almost every part of the plant contains lignin, including the coconut (Cocos nucifera L.), Pandharipande et al [5].
The purpose of this study was to extract lignin from coconut fiber as a green inhibitor, determine the effect of coconut fiber extract inhibitor on the corrosion rate of ASTM36 steel, and calculate the inhibition efficiency of coconut fiber extract.

2. Methodology

2.1. Extraction of lignin from coconut fiber
The coconut husk was cut to a size of 1 cm and then extracted using 35% Sodium Hydroxide (NaOH). The process temperature is 110 °C with a solid-to-liquid ratio of 1:8 (w/v). The extraction process of lignin for 110 minutes. The black liquor obtained was added with Sulfuric Acid (H₂SO₄) to pH 2 to form a lignin precipitate. The lignin obtained was dried in an oven at 50 °C for 3 hours.

2.2 Preparation of specimen and corrosion inhibitor medium
The materials used in this research was ASTM36 steel as specimens with a size 3 cm x 2 cm x 0.17 cm. Corrosive solution used is HCl 1 M. The added inhibitor varied from 1 g/L, 2 g/L, dan 3 g/L, and variation of immersion time 24, 36 dan 48 hours. The corrosion rate was determined from specimen weight loss of ASTM A36 steel, referring to NACE International Standard (National Association of Corrosion Engineers) [6].

2.3 Characterization of specimen and lignin from coconut fiber extraction
The morphology and type of corrosion of the ASTM A36 were characterized by Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX) to identification of its. Lignin obtained from coconut fiber extraction was analyzed using UV-Vis Spectrophotometry and FT-IR.

3. Results and Discussion

3.1. Analisis Spektroskopi Ultraviolet-Visible (Uv-Vis)
In this study, quantitative analysis was carried out to determine the absorbance value and determine phenolic compounds contained in lignin. To determine the levels of phenolic compounds in lignin, the phenol solution was used as a comparison in making standard curves because it had a hydroxyl group. The Ultraviolet-Visible (UV-Vis) spectrophotometer was operated at a maximum wavelength of 733 nm to observe the absorbance and then continued with the measurement of the absorption of coconut fiber extract. Lignin from coconut fiber extract was identified using a wavelength of 733 nm. Phenolic compounds from coconut fiber extract which have high antioxidant content can reduce Fe³⁺ ions to Fe²⁺ ions, forming a thin layer that can control the corrosion rate of metals, Suhartati [7].

Determination of phenolic compounds contained in lignin by quantitative analysis of UV-Vis spectroscopy. The regression equation obtained is $y = 0.0015x + 0.0227$ with a correlation coefficient (r) of 0.9952. The correlation coefficient value close to 1 proves that the regression equation is linear. The regression equation of the standard solution calibration curve was used to determine the phenol content in lignin (ppm) of the coconut fiber extract, $y$ was the absorbance value and $x$ was the concentration of phenol contained in the coconut coir lignin extract. From the results of the Ultraviolet-Visible (UV-Vis) spectrophotometry analysis, the absorbance value was 0.1444 A so that the phenol concentration value was 79.713 ppm.

Figure 1 shows calibration curve of phenol standard solution from UV-Vis spectrophotometric analysis.
**Figure 1.** Calibration curve of phenol standard solution from uv-vis spectrophotometric analysis

3.2. **Analisis Spektroskopi FTIR**
FTIR spectroscopic analysis was carried out in the wavenumber range of 450-4500 cm\(^{-1}\). The result of the FTIR analysis is a graph of wavenumber (cm\(^{-1}\)) vs percent transmittance (%).

Table 1 shows the functional groups contained in lignin, namely phenols, alkanes, alkenes, and aromatics. The phenolic groups contained in the lignin indicate that the coconut coir extract contains lignin which has potential as a green corrosion inhibitor and protects the steel from corrosion.

| Lignin produced | Lignin after protection | Wavenumber (cm\(^{-1}\)) | Functional Group Interpretation | Reference of lignin wavenumber range (cm\(^{-1}\)) (Stark et al), 2016 |
|-----------------|-------------------------|--------------------------|---------------------------------|-------------------------------------------------|
| 3615,12         | 3593,5                  | 3615,12, 12              | Phenolic group (O-H)            | 3600                                            |
| 2874,06         | 2886,6                  | 2874,06, 6              | Alkane, and Alkene group (C-H)  | 2849                                            |
| 1592,31         | 1597,13                 | 1592,31, 13             | Aromatic group (C=C)            | 1645                                            |
| 1042,57         | 1043,53                 | 1042,57, 53             | Aromatic group (C-H)            | 1050                                            |

**Figure 2** shows the presence of 2 wavelengths of lignin before the immersion process and after the immersion process. From the comparison results, it can be concluded that the HCl solution does not react with the lignin extract in the corrosion rate test process.
4. Figure 2. FTIR spectrum of lignin from coconut fiber extract

3.3. Effect of immersion time on corrosion rate

Corrosion rate can be determined by weight loss method using various inhibitor concentrations, corrosive media concentrations, and immersion time.

Figure 3, shows the effect of variations in inhibitor concentration, and immersion time on the corrosion rate of steel specimens, while Figure 4 shows the efficiency of lignin inhibition from coconut coir extract.

Based on Figure 3, it can be seen that there is a difference in the value of the corrosion rate obtained when the steel is immersed in 1 M HCl solution with the addition of inhibitor concentration and without the addition of inhibitor, where the lowest corrosion rate is 49.691 mpy at a variation of the addition of inhibitor concentration of 3 g/L with time immersion for 48 hours.

In this study, the value of the corrosion rate without the addition of inhibitors at a concentration of 1 M HCl corrosive medium and an immersion time of 48 hours was 227,069 mpy. According to Komalsari et al., [1] which states that corrosive media is one of the factors that can affect the corrosion rate. HCl media is a strong acid that is very corrosive. Steel has decreased in weight due to the erosion of Fe ions by acidic HCl. The Cl\(^{-}\) (chloride) ion acts as an aggressive ion because of its ability to destroy the surface layer of steel so that it can reduce the bond strength between metal atoms and accelerate corrosion.

Based on Figure 3, it shows that there are differences in the corrosion rate values obtained when the steel is immersed in 1 M HCl solution with the addition of inhibitor, where the lowest corrosion rate is 49,691 mpy at the variation of the addition of inhibitor concentration of 3 g/L with time immersion for 48 hours.

In this study, the value of the corrosion rate without the addition of inhibitors at concentration of 1 M HCl corrosive medium and an immersion time of 48 hours was 227,069 mpy. According to Komalasari et al., [1] which states that corrosive medium is one of the factors that can affect the corrosion rate. HCl solution is a strong acid which is very corrosive. Steel has decreased in weight due to the erosion of Fe ions by acidic HCl. The Cl\(^{-}\) (chloride) ion acts as an aggressive ion because of its ability to destroy the surface layer of steel so that it can reduce the bond strength between metal atoms and accelerate corrosion.
Figure 3. The corrosion rate of astm36 steel using coconut fiber extract with variation of immersion time and inhibitor concentration

Figure 4 shows the difference in the value of the inhibition efficiency on the immersion time of ASTM A36 steel on 1 M HCl corrosive medium. The highest inhibition efficiency was 78.11% for the addition of 3 g/L inhibitor and 48 hours immersion time. While the lowest inhibition efficiency was 36.09% for the addition 1 gr/L inhibitor and 24 hours immersion time. The inhibition efficiency increased because it was influenced by the addition of inhibitor concentration and the immersion time was increased. The greater concentration of inhibitor added and the longer immersion time of the steel plate, the inhibitor will form a layer on the metal that can protect the metal from HCl corrosive solution, Hussin [8]. The ability of the inhibitor to protect steel from corrosion will be lost or exhausted at a certain time, it’s because the longer the immersion time, more the inhibitor will be eroded by the acid solution. At a certain time, the value of the inhibition efficiency will decrease because the functional group for adsorption on the steel surface are already maximum and can no longer from a protective layer.

Figure 4. Inhibition efficiency of coconut fiber extract inhibitor with immersion time variation

3.4. Analysis: Scanning Electron Microscope-Energy Dispersive X-Ray Spectroscopy (SEM-EDX)
Figure 5. Surface morphology of ASTM A36 steel using inhibitor 3 g/L, dan immersion time 48 hours (a) magnification 500x (b) magnification 1000x (c) magnification 3000x (d) magnification 5000x

Table 2. Identification of element in ASTM36 steel from EDX analysis

| Treatment                      | Fe  | C   | O   | Cl  |
|--------------------------------|-----|-----|-----|-----|
| Steel before treatment         | 81.84 | 18.16 | - | - |
| Steel without protection      | 47.20 | 13.04 | 39.66 | 0.10 |
| Steel with protection          | 63.76 | 9.72 | 26.50 | 0.02 |

Based on the results of the EDX analysis in Figure 5 and Table 2, it can be seen that the percentage of Fe is the highest, because the main component of ASTM A36 steel is Fe. Specimens that have been in contact with HCl solution, detected the presence of elements of oxygen and chloride, this indicates that the specimens have undergone corrosion. The EDX results show that the carbon steel that is corroded is dominated by the content of Fe and O atoms which indicates that the corrosion process leads to the modification of the surface layer containing oxides and hydroxides. ASTM A36 steel which is corroded in the HCl corrosion medium to inhibitor has been added has a higher Fe atomic composition than ASTM A36 steel without the addition of an inhibitor, because steel without the addition of inhibitor, more Fe atoms are oxidized so that iron will lose more electrons and lose weight, Ramlah [9].

4. Conclusion
In this study, lignin from coconut fiber extract can be used as an inhibitor that protects metals from corrosion. The inhibition efficiency obtained was 78.11% at the inhibitor concentration of 3 g/L and the immersion time for 48 hours was the highest. As the inhibitor concentration increases and the steel
immersion time increases, the corrosion rate will decrease where the lowest corrosion rate was obtained at the inhibitor concentration of 49.691 g/L with an immersion time of 48 hours and an inhibitor concentration of 3 g/L.

References

[1] Komalasari, Utami, S P, Fermi M I, Aziz Y, and Irianty R S 2018 Corrosion Control of Carbon Steel Using Inhibitor Banana Peel Extract in Acid Diluted Solutions in IOP Conference Series: Material Science and Engineering 345(1) IOP Publishing

[2] Pradipta I, Kong D, and Tan J B L 2019 Natural Organic Antioxidants from Green Tea Inhibitor Corrosion of Steel Reinforcing Bars Embedded in Mortar Journal of Construction and Building Material 227(1) 1-14

[3] Marques F P, Silva L M A, Lomonaco D, Rosa M F, and Leitao R C 2020 Steam Explosion Pretreatment to Obtain Eco-Friendly Building Blocks from Oil Palm Mesocarp Fiber Journal of Industrial Crops and Product 143(1) 1-11

[4] Mulyaningsih N C, Pramono, and Prasetyo R T 2018 Pengaruh Penambahan Inhibitor Organik Ekstrak Eceng Gondok Terhadap Laju Korosi Journal of Mechanical Engineering 2(2) 39-45

[5] Pandharipande S L, Gujrati N, Mulukutkar, and Pandey S 2018 Comparative Study of Extraction & Characterization of Lignin from Wet and Dry Coconut Husk International Journal of Engineering Sciences & Research Technology 7(4) 659-666

[6] NACE International 2005 Standard recommended practice: preparation, installation, analysis, and interpretation of corrosion coupons in oilfield operations NACE International United States RP0775

[7] Suhartati T 2017 Dasar-dasar Spektrofotometri UV-Vis dan Spektrofotometri Massa Untuk Penentuan Struktur Senyawa Organik CV Anugrah Utama Raharja : Lampung

[8] Hussin M H, Rahim A A, Mohamad I M N and Brosse N 2016 The capability of ultrafiltrated alkaline and organosolv oil palm (Elaeis guineensis) fronds lignin as green corrosion inhibitor for mild steel in 0.5 M HCl solution Journal of the International Measurement Confederation 78(1) : 90-103

[9] Ramlah, Wijaya M and Pratiwi D E 2020 Efektivitas Ekstrak Daun Beluntas sebagai Inhibitor Korosi pada Material Baja Karbon dalam Media NaCl 3,5% Jurnal Chemica 21(1) : 86-99