

# Sodium Methoxide Catalyzed Preparation of Nitrogen Compounds from Palm Oil Methyl Esters as Corrosion Inhibitor

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## Abstract

This research was conducted to study preparation of nitrogen compounds by reacting palm oil derived methyl esters and diethanolamine using sodium methoxide as a catalyst. The chemical composition of the product was analyzed using the gas chromatography-mass spectrometry (GC-MS) method. The product was then applied as a corrosion inhibitor for mild steel in CO<sub>2</sub> saturated 3% NaCl solution. Experimental results indicate that the reaction product contains six nitrogen compounds with a relative percentage of 63.7%, and the rest 36.7% are a mixture of unreacted diethanolamine and unreacted methyl esters. The results of the corrosion experiments show that the product obtained exhibited appreciably high performance as reflected by a percentage of protection (%P) of 96.85%. The performance of the product as a corrosion inhibitor is also supported by the SEM image of the mild steel sample, which shows that the surface of the sample is well protected. The results of this work demonstrate that nitrogen compounds obtained have promising potential as corrosion inhibitors, particularly to prevent corrosion by CO<sub>2</sub> gas.

## Keywords

Nitrogen Compounds, Palm Oil Methyl Esters, Green Corrosion Inhibitors, Mild Steel, Wheel Test

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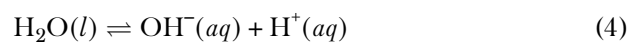
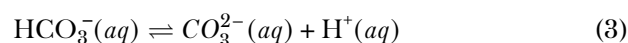
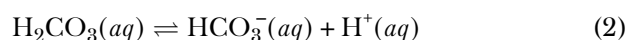
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## 1. INTRODUCTION

One of the most important infrastructure materials in petroleum industry is mild steel because this metal is relatively cheaper than other steel alloys, such as; low-alloy steel, high-strength low alloy (HSLA) steel, high-alloy steel, stainless steel, micro-alloyed steel, advanced high-strength steel (AHSS). One important use of mild steel is as a material to build a pipeline network for the transportation of crude oil from wells to processing facilities. Unfortunately, mild steel is prone to corrosion in the environment containing corrosive species, and in the context of petroleum industry, the most problematic corrosive substances present in crude oil are CO<sub>2</sub> gas and H<sub>2</sub>O. These two substances can react to form carbonic acid (H<sub>2</sub>CO<sub>3</sub>), according to the following equation:



The carbonic acid formed can undergo a series of reactions according to the following equations:



As can be seen, the last three reactions lead to the formation of H<sup>+</sup>, which means increasing the acidity of the system. As a result, the system becomes more corrosive toward metal. The most reasonable way to protect the inner surface of the pipe is by adding a corrosion inhibitor to the liquid flowing inside the pipe. The inhibitor will create a protective layer in the form of a thin film on the surface of the metal and prevent direct contact between metal surface and the corrosive species present. To overcome corrosion, different types of inhibitors that belong to inorganic and organic substances have been used. However, organic inhibitors are more desirable than inorganic inhibitors because organic inhibitors are more environmentally friendly (green inhibitors), simple to apply, relatively low cost, and renewable (Al-Amiery et al., 2023).

Commonly organic inhibitors are the compounds containing heteroatoms having lone pair electrons, primarily nitrogen

(N), oxygen (O), sulfur (S), and phosphorus (P) in conjugated systems. With the presence of heterogeneous atoms, the inhibitor can interact with the metal surface via electrostatic interaction and create a protective thin film layer on the surface. The protective layer formed can prevent direct contact between the metal and the environment (Keleş et al., 2015; Zaafarany and Abdallah, 2010; Jevremović et al., 2016).

In previous studies, the use of various nitrogen compounds has been reported, among others are oligomers of 4-vinylpyridine (Ilim et al., 2021b; Ilim et al., 2016e), oligomer of 2-vinylpyridine (Ilim et al., 2021d), oligomer of 4-vinylpiperidine (Ilim et al., 2017c), and ethylenediamine and tetraethylenepentamine (Mahmmud et al., 2019). In the context of nitrogen compounds preparation, other potential raw materials are vegetable oils, considering their availability, renewability, biodegradability, and non-toxicity. One particular piece of information that was taken into consideration in conducting this present research is the work by others (Wongwanichkangwarn et al., 2021), who reported the successful synthesis of fatty acid diethanolamide by reacting fatty acid methyl ester with diethanolamine using sodium methoxide as a catalyst. This achievement implies that it is possible to produce nitrogen compounds from fatty acid methyl esters derived from various vegetable oils, such as coconut oil (Masyithah et al., 2019; Hossain et al., 2012; Ilim et al., 2022a), palm oil (Minaria and Mohadi, 2016), karanj oil (Syamsuddin et al., 2016), soybean oil (Lu et al., 2015), and rubber seed oil (Anggraini et al., 2019; Pandiangan et al., 2021).

With respect to the finding reported by Wongwanichkangwarn et al. (2021), this research was conducted to study the production of nitrogen compounds by reacting palm oil methyl esters and diethanolamine. The reaction was carried out in the presence of sodium methoxide ( $\text{NaOCH}_3$ ) synthesized from NaOH and methanol, as a catalyst. The product was then characterized using FTIR and GC-MS, and finally, the activity of the product as a corrosion inhibitor of mild steel was evaluated in a 3% NaCl medium saturated with  $\text{CO}_2$ .

## 2. EXPERIMENTAL SECTION

### 2.1 Materials

The chemicals used in this research included HCl,  $\text{Sb}_2\text{O}_3$ ,  $\text{SnCl}_2$ , NaCl, NaOH,  $\text{HNO}_3$ , methanol,  $\text{NaHCO}_3$ , diethanolamine, SAE/AISI Grade 1022 mild steel was obtained from Krakatau Steel Company, palm oil methyl esters used was laboratory stock from previous work, and silicon carbide paper (grit 60, 80, 100, 200, 400, 600, 800, and 1200). The corrosive solution was prepared by dissolving NaCl (3% w/v) and  $\text{NaHCO}_3$  ( $100 \text{ mg L}^{-1}$ ) in deionized water. Laboratory equipments used were Shimadzu GCMSQP2010 SE Chromatography–Mass Spectrometry (GC–MS), PANalytical EMPYREAN X-Ray Diffractometer (XRD), ZEISS EVO 10 Scanning Electron Microscope (SEM), Fourier Transform Infrared Spectroscopy (FTIR) NICOLET AVATAR 360 Fourier Transform Infrared (FTIR) Spectrometer, autoclave (Polytetrafluoroethylene coated stainless steel), analytical balance (METTLER AE

200), glass bottles. Micropipette, and micrometer.

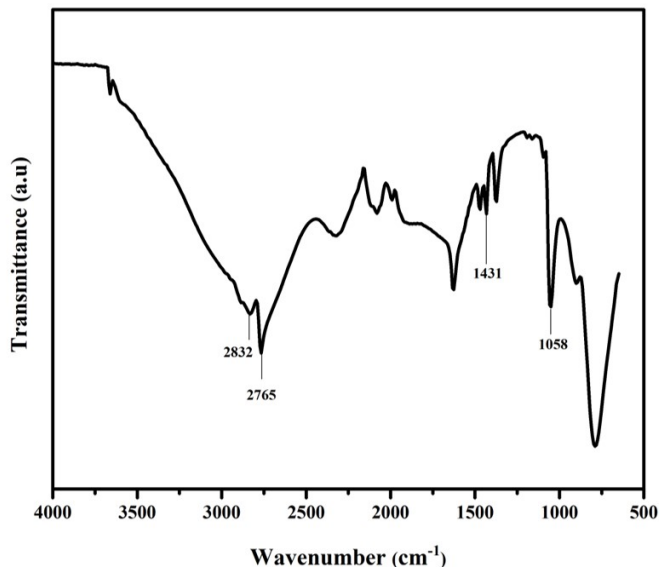


Figure 1. FTIR Spectrum of Sodium Methoxide Synthesized

### 2.2 Methods

#### 2.2.1 Preparation of Sodium Methoxide ( $\text{NaOCH}_3$ )

Sodium methoxide ( $\text{NaOCH}_3$ ) catalyst was synthesized by reacting sodium hydroxide (NaOH) and methanol analysis with a molar ratio of 1:1 adopting the ratio applied in previous research by others (Wongwanichkangwarn et al., 2021). A mass of 2.0 g of sodium hydroxide was transferred into a polypropylene container and 2.525 mL of methanol was added. The container was closed tightly and the mixture was subjected to 30 minutes of stirring, then left for 24 hours at room temperature until white crystals formed. The products obtained were then characterized using FTIR, XRD, and SEM.

#### 2.2.2 Nitrogenation of Palm Oil Methyl Esters

To carry out nitrogenation, the reaction mixture consisting of palm oil methyl esters (10 mL), diethanolamine (10 mL), and sodium methoxide catalyst (0.1g) was placed in a round bottom flask and then refluxed for 8 hours at 80 °C. After the experiment was completed, the flask was cooled to room temperature, followed by filtration to separate the catalyst from the product (filtrate). The functionality of the product was assessed using FTIR and the chemical components of the product were identified using GC-MS.

#### 2.2.3 Corrosion Inhibition Experiment

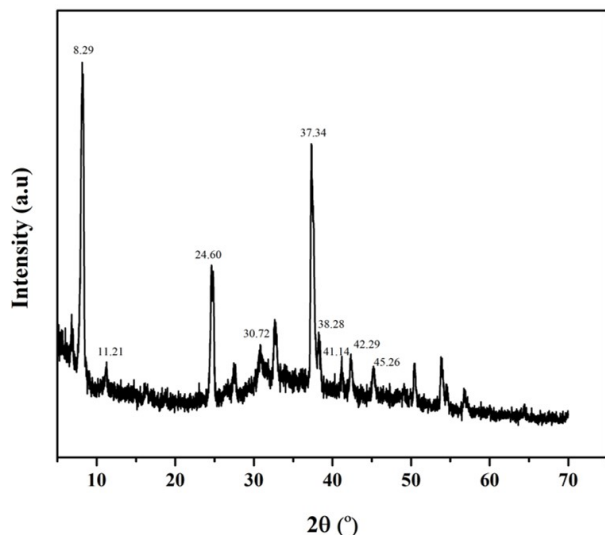
In this study, the wheel test method was applied to assess the activity of the nitrogenation product as a corrosion inhibitor. A mild steel sample in the form of coupon was prepared with dimensions of 2 cm × 1 cm and the surface of the sample was subsequently polished with silicon carbide paper with grit sizes of 60, 80, 100, 200, 400, 600, 800, and 1200. Before the

**Table 1.** Chemical Composition of the Nitrogenation Product Obtained

Retention Time (min)	Chemical Formula	Name of Compound	Relative Percentage (%)
9.25	C <sub>6</sub> H <sub>14</sub> N <sub>2</sub> O	N-Isopropyl-N-nitroso propylamine	16.09
13.61	C <sub>4</sub> H <sub>11</sub> NO <sub>2</sub>	Diethanolamine	15.43
29.46	C <sub>17</sub> H <sub>34</sub> O <sub>2</sub>	Methyl palmitate	7.01
32.71	C <sub>19</sub> H <sub>34</sub> O <sub>2</sub>	Methyl linoleate	2.31
32.84	C <sub>19</sub> H <sub>36</sub> O <sub>2</sub>	Methyl oleate	11.55
33.57	C <sub>14</sub> H <sub>29</sub> NO <sub>2</sub>	N-(2-Hydroxyethyl)-lauramide	3.38
36.71	C <sub>10</sub> H <sub>21</sub> NO <sub>2</sub>	N-(2-Hydroxyethyl)-octanamide	5.71
38.82	C <sub>9</sub> H <sub>21</sub> N	N, N-Dipropyl-1-propylamine	22.63
41.64	C <sub>6</sub> H <sub>14</sub> N <sub>2</sub>	N, N'-Dimethylpiperazine	11.98
44.18	C <sub>15</sub> H <sub>31</sub> NO <sub>3</sub>	(4S) [(Tert. Butyloxycarbonyl) amino]-2-methyl	3.91

**Table 2.** The Results of Wheel Test Experiments

Inhibitor	A (cm <sup>2</sup> )	W (g)	CR (mm/y)	%P
-	8.0658	0.0057	0.3282	-
Synthesized product	7.5018	0.0002	0.0103	96.85

**Figure 2.** XRD Diffractogram of Sodium Methoxide Synthesized

corrosion experiment was conducted, the mass of the sample was weighed. The corrosive medium with a volume of 175 mL was transferred into a clean glass bottle, and the medium was saturated with CO<sub>2</sub> by flowing the gas into the bottle for around 45 minutes. The inhibitor solution with a concentration of 150 ppm was then added to the bottle. The mild steel coupon was then immersed in the solution and the bottle was tightly closed. The experiment was run at room temperature for 24 hours. After the completion of the experiment, the coupon was taken out from the bottle and directly immersed in Clarke's

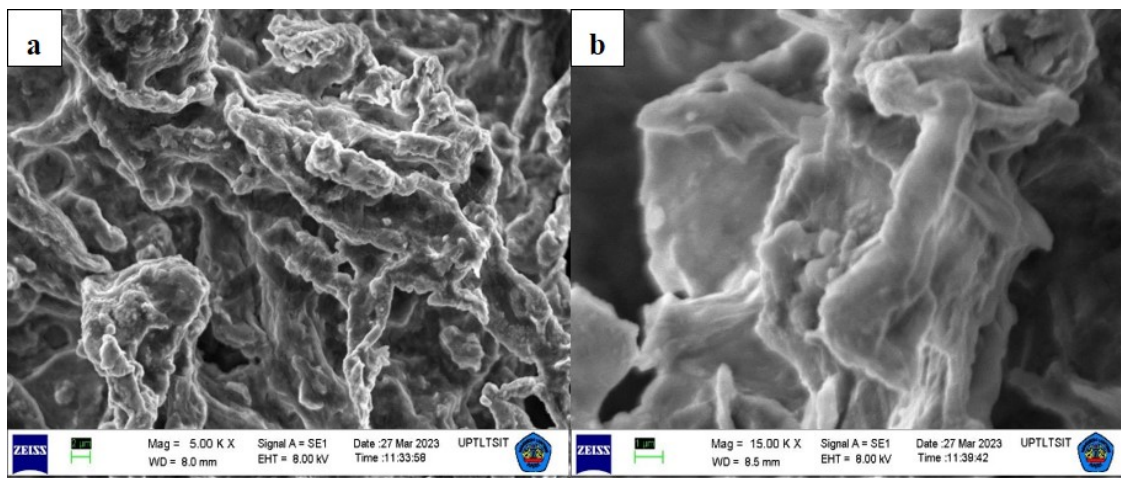
solution for 5 seconds to dissolve corrosion products formed on the surface. The coupon was subsequently soaked in distilled water for approximately 5 seconds and in ethanol for 5 seconds. The coupon was dried and finally weighed. The corrosion rate and protection percentage were calculated as reported in a previous investigation (Ilim et al., 2022a). To obtain the surface morphology, an untreated sample, the sample subjected to experiment medium without inhibitor, and the sample treated with inhibitor were characterized using SEM.

### 3. RESULTS AND DISCUSSION

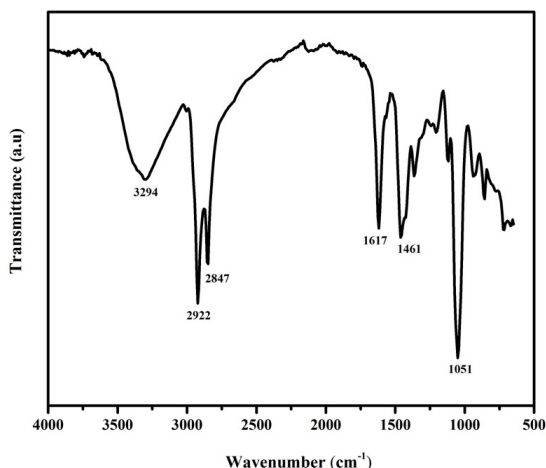
#### 3.1 Characterization of Sodium Methoxide (NaOCH<sub>3</sub>)

To evaluate the characteristics of sodium methoxide synthesized, the sample was subsequently characterized using FTIR, XRD, and SEM techniques. Figure 1 is the FTIR spectrum obtained by scanning the sample over the wavenumber range of 4000 to 500 cm<sup>-1</sup>. Based on the previous study (Chandran et al., 2006), the absorption bands at wavenumbers of 2832, 2765, and 1431 cm<sup>-1</sup> are assigned to C-H functional group. The absorption band at 1058 cm<sup>-1</sup> is a characteristic band representing C-O functional group. The presence of this band indicates that the hydrogen atom from the -OH group in the methanol has been replaced by sodium atom resulting in the formation of an O-Na bond.

The next technique applied to characterize the sample was XRD, producing a diffractogram presented in Figure 2. The diffractogram in Figure 2 shows that the sample is a mixture of crystalline and amorphous materials. The diffractogram of the sample is in agreement with the diffractogram of sodium methoxide available in the JCDPS 19-1876 file. As displayed by Figure 2, the diffractogram is marked by the presence of diffraction peaks at 2θ of 11.21; 24.60; 30.72; 37.34; 38.28;



**Figure 3.** SEM Micrograph of Sodium Methoxide Synthesized in this Study with Magnification of 5.000× (a) and 15.000× (b)



**Figure 4.** FTIR Spectrum of the Nitrogenation Product

41.14; 42.29; 45.26; and 49.14°. As a comparison, the diffraction peaks of sodium methoxide according to the JCDPS 19-1876 file are located at  $2\theta$  of 11.8; 23.7; 31.4; 36.2; 38; 41.6; 43.3; 47.2; and 48.4°. In the sample prepared, there is a peak at 8.29° which is not listed in the JCDPS 19-1876 file, suggesting the possibility of the existence of crystal with different spacing between atoms, creating an interference pattern that appear as a peak on the diffractogram.

Characterization of the sample using SEM produced micrograph shown in Figure 3. The micrograph in Figure 3 indicates that the sample is dominated by amorphous particles, agglomerating to produce a continuous structure. According to the JCPDS 19-1876 file, sodium methoxide is indexed as a tetragonal system, however, the presence of tetragonal particles in Figure 3 cannot be observed. The most likely reason for this phenomenon is the domination of the amorphous phase in the

sample, as has been indicated by the diffractogram in Figure 2.

### 3.2 Characterization of Nitrogenation Product

Characterization of nitrogenation products was conducted using FTIR and GC-MS techniques. Characterization using FTIR was aimed to gain information regarding the functional groups that can be related to the reaction between palm oil methyl esters and diethanolamine. The FTIR spectrum obtained is displayed in Figure 4.

The FTIR spectrum in Figure 4 is characterized by the presence of absorption bands that might be related to nitrogen compounds. The absorption band at a wavenumber of 3294  $\text{cm}^{-1}$  is the band that resulted from vibration of the N–H group. Other absorption bands that strongly indicate the existence of other functional groups are a band at 2922  $\text{cm}^{-1}$  (assigned to C–H group), the band at 1617  $\text{cm}^{-1}$  (assigned to the C=O group), the band at 1461  $\text{cm}^{-1}$  (representing bending vibration of  $\text{CH}_2$ ), and a band at 1051  $\text{cm}^{-1}$  (representing stretching vibration of CN functional group) (Adewuyi, 2019; Surya et al., 2013; Sari et al., 2018). To identify nitrogen compounds formed, the sample was analyzed using the GC-MS technique. The GC-chromatogram is presented in Figure 5 and the components of the sample identified with the aid of MS library database (Wiley7.Lib) are listed in Table 1. As can be seen in Table 1, besides dimethyl amine there are 6 nitrogen compounds identified, with a total relative percentage of 63.7%. These results demonstrated that the performance of the preparation method utilized was appreciably effective in producing nitrogen compounds from palm oil methyl esters.

### 3.3 Corrosion Inhibition Tests

The results of wheel test experiments are presented in Table 2, synthesized inhibitors possess the ability to inhibit the corrosion of mild steel samples in the corrosive environment used. The results obtained show that the nitrogenation product exhibits a fairly high ability to protect metal samples as indicated by a %P of 96.85%. Considering the contribution of the ni-

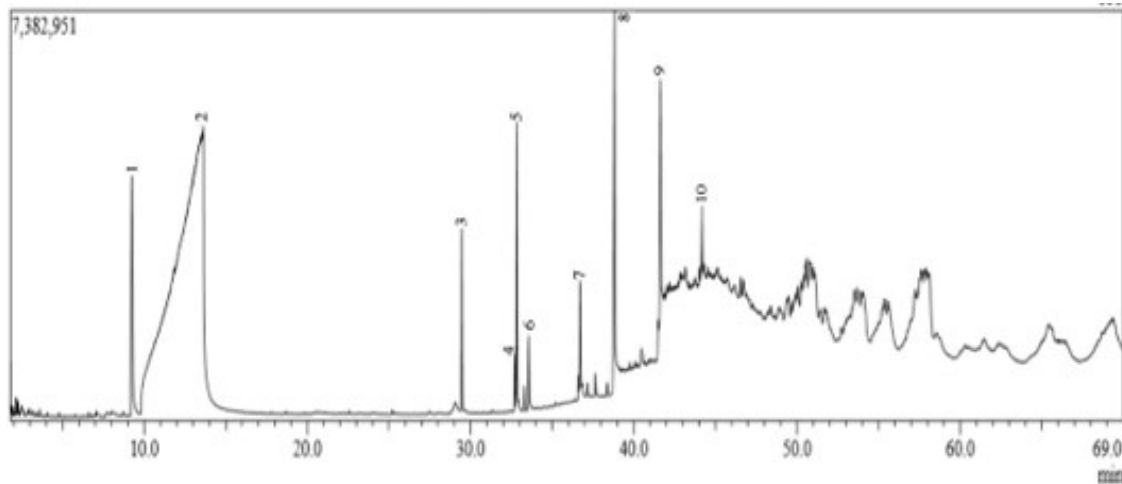


Figure 5. GC-chromatogram of the Nitrogenation Product Obtained

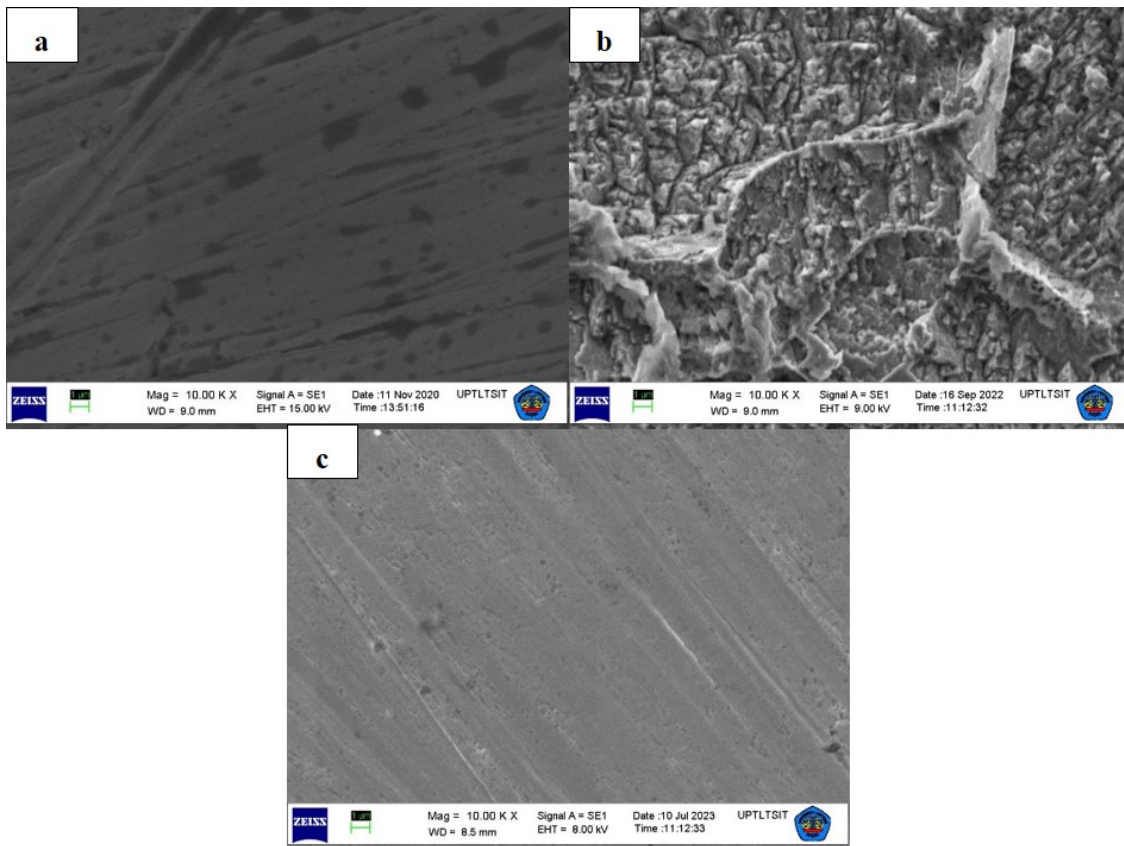


Figure 6. Surface Morphology of the Original Mild Steel (a), Mild Steel Treated without Inhibitor (b), and Mild Steel Treated with Inhibitor Product (c), The Images were Recorded with a Magnification of 10,000×

trogen compounds (63.7% to the relative composition of the sample), it is reasonable to consider that this %P value is related to the content of nitrogen compounds in the product. The performance of nitrogenation product as a corrosion inhibitor observed in this study is in agreement with the findings reported in previous studies (Shahin et al., 2002; Avdeev et al.,

2021) where the activity of corrosion inhibitors increases with increasing amounts of nitrogen compounds composing the inhibitor. The relationship between the activity of the inhibitor and the content of nitrogen compounds derived from palm oil methyl esters found in this present study is supported by the finding that diethanolamine does not have corrosion inhibitory

activity, as reported in our previous study (Ilim et al., 2022a).

The performance of inhibitors in protecting mild steel samples is also reflected by the SEM micrographs shown in Figure 6. The micrographs of the metal samples presented in Figure 6 display the ability of the inhibitor to protect the sample, as also demonstrated by the %P data obtained from wheel test experiments shown in Table 2. The untreated mild steel sample (Figure 6a) is characterized by a smooth surface. The scratches on the surface of this original sample are due to the polishing process. The surface of the sample immersed in a corrosive medium without inhibitor (Figure 6b) is seriously damaged, which suggests that corrosion has attacked the entire surface of the sample. A very different image is displayed by the sample treated with the inhibitor (Figure 6c), where the sample surface remains smooth with only minor scratches, confirming the appreciably high ability of the inhibitor to protect the sample from corrosion attack.

#### 4. CONCLUSION

The experimental data obtained demonstrated that the production of nitrogen compounds by nitrogenation of palm oil methyl esters was achieved, although it should be acknowledged that complete conversion of the methyl esters into nitrogen compounds has not been achieved. Regardless of this incomplete conversion, the nitrogenation product was found to offer promising potential as corrosion inhibitor, as reflected by the percentage of protection (96.85%) and well protected surface of the metal sample as displayed by the SEM image.

#### 5. ACKNOWLEDGMENT

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