Study on corrosion inhibition activity of nitrogen compounds derived from coconut oil methyl ester

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Abstract

This study was carried out as an attempt to convert methyl esters coconut oil into nitrogen compounds by reacting the methyl esters with diethanolamine. Three samples were prepared by carrying out experiment at 80°C for 24 h, at 100°C for 24 and 48 h. The products of the reactions were analyzed using GC-MS and then utilized as corrosion inhibitor for mild steel in CO_2 saturated brine solution using Wheel test method. The inhibition activity of the samples was evaluated in terms of percentage protection and surface morphology of the sample using SEM-EDX method. The experimental results obtained revealed that the experiments conducted at 80°C for 24 h and at 100°C for 24 h only produced nitrogen compounds as a minor component, but in the sample produced from the experiment carried out 100°C for 48 h a series of nitrogen compounds were produced, contributing 53.54% to the composition. The results of corrosion testing experiments revealed that the samples exhibit corrosion inhibition activity, with the highest protection of 97.9% was provided by the sample prepared at 100°C for 48 h. Without ignoring the need for further research, the results obtained in this study suggest that the nitrogen compounds derived from coconut oil possess promising potential as green corrosion inhibitor.

Introduction

One of serious problems faced by the gas and oil industry for years is corrosion of the inner surface of the pipe due to reaction between CO_2 gas and water to produce highly corrosive carbonic acid [1, 2]. It has been reported that carbonic acid causes more severe corrosion then that caused hydrochloric acid at the same pH [3]. The difference in corrosive properties of the two compounds is explained as a result of differences in dissociation properties where HCl undergoes complete ionization while H_2CO_3 undergoes gradual dissociation and produces several corrosive species such as HCO_3^-, CO_3^{-2}. For this reason, CO_2 corrosion has been a problem in the oil and gas industry for many years [4, 5].

Addition of corrosion inhibitor into a corrosive medium is the most effective method for controlling acid corrosion including CO_2 corrosion. In search for effective and efficient

inhibitors against corrosion, various corrosion inhibitors have been developed, and of particular interest are nitrogen compounds having both hydrophilic and hydrophobic group. The presence of lone pair electrons on the nitrogen atom provides the compound with hydrophilic character and allows the formation of a covalent bond between the inhibitor molecules and the metal atoms on the surface. As a result of this interaction, the surface of the metal will be covered by the layer of inhibitor that function to prevent direct interaction of the metal surface with the corrosive components present in the environment. Protection of the metal surface is also provided by the hydrophobic part of the inhibitor which acts as a screen to protect the surface from the water.

Driven by their good performance, various nitrogen compounds are continuously developed and tested as corrosion inhibitor for different materials and different environments. Mahmmod *et al.* [6] reported application of ethylenediamine (EDA) and tetraethylene-pentamine (TEPA) as corrosion inhibitors for Cu–Ni alloy in 6% HCl solution and reported the highest inhibition efficiency of 77% was provided by EDTA at concentration of 10 g/L and 40ºC experimental temperature. The use of imidazoline derivative demonstrated high inhibition performance in a CO_2/H_2S system with the efficiency reaches 93.94% by using 20 ppm inhibitor [7]. In our previous works, corrosions of mild steel in CO_2-saturated 3% NaCl solution were studied in the presence of three nitrogen compounds as corrosion inhibitor, include the oligomers of 4-vinylpiperidine [8], polymeric derivatives of 2-vinylpyridine [9], and 4-vinylpyridine oligomers [10].

In this study, conversion of methyl esters derived from coconut oil into nitrogen compounds was investigated. The methyl esters were reacted with diethanolamine and the product was analyzed to identify nitrogen compounds formed and then tested as corrosion inhibitor of mild steel in brine solution saturated with CO_2 gas. The performance of the compounds was evaluated by determination of the percent of protection using Wheel test method and surface characterization of the metal sample using SEM/EDX technique.

Materials and Methods

1. Materials and tools

Chemicals used in this research were purchased from Merck include NaCl, NaOH, methanol, $NaHCO_3$, HNO_3, diethanolamine, HCl, Sb_2O_3, $SnCl_2$, mild steel (SAE/AISI Grade 1022) was purchased from Krakatau Steel, rice husk was obtained from local rice milling company, food grade CO_2 gas, zeolite-A, coconut oil methyl ester, and silicon carbide paper (grade 200, 400, 600, 800, and 1200). The corrosive solution was prepared by dissolving NaCl (3% w/v) and $NaHCO_3$ (100 mg·L^{-1}) in deionized water.

Instruments used in this research were Fourier Transform Infrared (FTIR) NICOLET AVATAR 360, Gas Chromatography–Mass Spectrometry (GC–MS) Shimadzu GCMS-QP2010 SE, X-Ray Diffraction (XRD) PAN analytical type EMPYREN, Scanning Electron Microscopy-Energy Dispersive X-Ray (SEM-EDX) ZEISS EVO 10, Polytetrafluoro-

ethylene (PTFE) lined stainless steel autoclave, analytical balance METTLER AE 200, micropipette, micrometer, and glass bottles.

2. Methods

Zeolite-A and coconut oil methyl esters used in this study were the stocks prepared in our previous investigations. Detailed procedure for preparation of zeolite-A was described in Simanjuntak *et al*. [11] and detailed procedure for preparation of coconut oil methyl ester was described in Herliana *et al*. [12]. To prepare nitrogen compounds, an aliquot of 20 mL of coconut oil methyl ester was mixed with 20 mL diethanolamine and 2 grams of zeolite-A. The mixture was transferred into an autoclave and then heated in an oven. Three samples were prepared by conducting the experiments at 80°C for 24 h and at 100°C for 24 and 48 h. After the completion of the experiment, the autoclave was removed from oven and allowed to cool to room temperature, and then filtered to separate the catalyst from the filtrate. The filtrate was characterized using FTIR and analyzed using GC-MS to identify the components of the sample. The product of each of the experiments was then tested as corrosion inhibitor for mild steel in CO_2 gas saturated brine solution (NaCl 3%). For simplicity purpose, the product of the reaction at 80°C for 24 h was specified as P-1, the product of the reaction at 100°C for 24 h as P-2, and the product of the reaction at 100°C for 48 h as P-3.

To conduct corrosion experiment, mild steel coupon with the dimension of 2×1 cm was prepared and the surface of the metal was polished using silicon carbide paper with the size of 200, 400, 600, 800, and 1200 grits. Brine solution was placed in clean glass bottles with the same volume of 175 mL then purged with CO_2 gas for approximately 45 min. Corrosion inhibitor solution then added into the bottles with concentration of 150 ppm. A mild steel coupon prepared was placed into the bottle and the bottle was tightly sealed and placed in a room temperature for 24 h. After the completion of the experiment, the mild steel coupon was removed and immediately placed in Clarke's solution for 5 seconds in order to remove the corrosion product from the surface. Mild steels was dipped into water for about 5 seconds and into ethanol for another 5 seconds. The sample was dried and accurately weighed. The weight loss (W) and the corrosion rate (CR) were calculated using Equation 1 and 2.

$$W = W_i - W_t \tag{1}$$

$$CR = \frac{10 \cdot W \cdot 365}{A \cdot D \cdot t} \tag{2}$$

where W = weight loss (gram), W_i = initial weight, W_t = final weight, CR = corrosion rate (mm·y^{-1} = milimeter per year), A = area of coupon (cm^2), D = density of the metal (g·cm^{-3}) equal to 7.86 g·cm^{-3} for carbon steel, and t = exposure time (days). The efficiency of the inhibitor to reduce the corrosion rate was calculated using Equation 3.

$$\%P = \frac{CR_0 - CR_i}{CR_0} \cdot 100 \tag{3}$$

where $\%P$ = percent protection, CR_0 = corrosion rate without inhibitor and CR_i = corrosion rate with inhibitor.

To obtain visible images of the surface, untreated (original) specimens, specimens immersed in a brine solution without inhibitor, and specimens immersed in a brine solution containing inhibitor at a concentration of 150 mg·L^{-1} were characterized using SEM-EDX.

Results and Discussion

1. FTIR characterization

To evaluate the change in functional groups as an indication of the formation of nitrogen compounds has taken place, the FTIR spectra of the methyl esters and the three reaction products (P-1, P-2, and P-3) were recorded and shown in Figure 1.
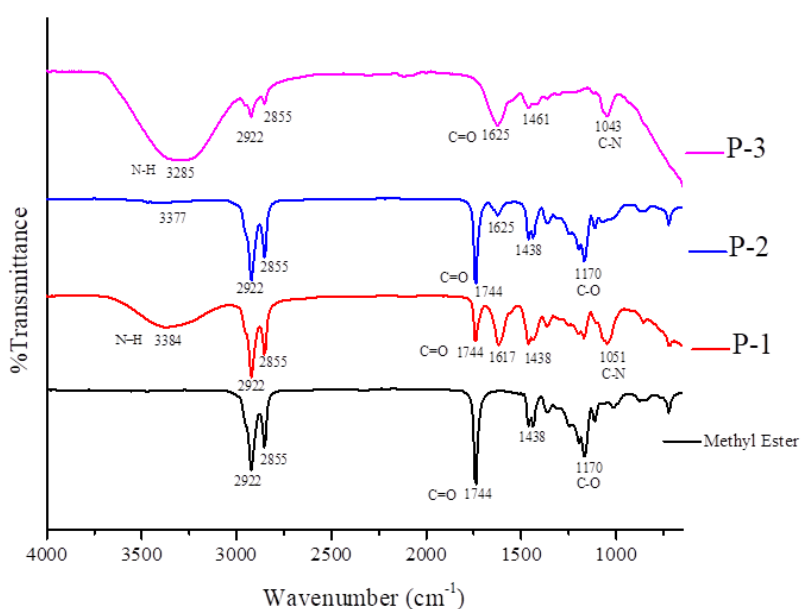

Figure 1. The FTIR spectra of methyl esters, P-1, P-2 and P-3.

As can be seen, the spectrum of methyl esters is characterized by the existence of absorption peaks that arise from vibration of distinctive functional groups of esters. The absorption bands at 2922 cm^{-1}, 2855 cm^{-1}, and 1438 cm^{-1} are attributed to the $-$C$-$H stretching and bending of the alkane group. The sharp absorption peak at a wavenumber of 1744 cm^{-1} is attributed to stretching of the carbonyl group (C=O) and the absorption band at 1170 cm^{-1} represents the C$-$O group [13, 14]. Sari *et al.* [15] reported that the amide group (C=O) was recorded at a wavenumber of 1618.34 cm^{-1} for alkanolamide from coconut oil.

Comparing the spectrum of P-1, P-2, and P-3 samples to that of methyl esters sample, the existence of some differences can be observed. The first difference is the emergence of an absorption band in the wavenumber range of 3000$-$3750 cm^{-1} in the spectra of P-1, P-2, and P-3 which represents the N$-$H group. It is also observed that the intensities of the peak are significantly higher in the spectrum of P-1 and P-3 samples compared to that in the

spectrum of P-2 sample. The existence of an N–H group is also supported by the absorption band in the wavenumber range of 1600–1630 cm^{-1} in the spectra of P-1, P-2, and P-3 samples but not detected in the spectrum of methyl esters. In overall, the FTIR spectra of the samples investigated display that the P-1 and P-3 samples are relatively richer in nitrogen compounds compared to the P-2 sample. To justify this indication, the samples were further analyzed using the GC-MS method.

2. GC-MS Analysis

To evaluate whether the formation of nitrogen compounds was achieved as expected, the three samples were characterized using GC-MS. The GC chromatogram of P-1 sample is shown in Figure 2 and the components of the sample is shown in Table 1.
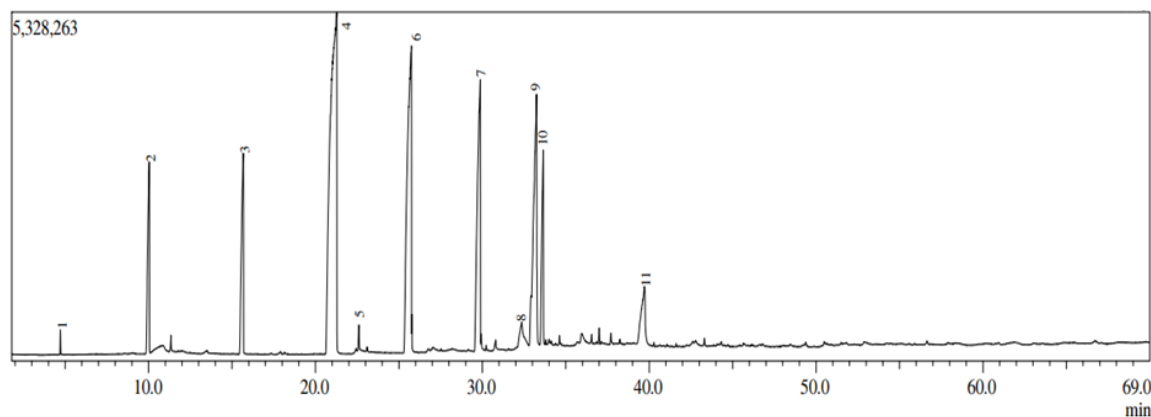

Figure 2. GC chromatogram of P-1 sample.

Table 1. Chemical composition of P-1 sample.

No.	Compound Name	Chemical Formula	Relative Percentage (%)
1	Methyl caproate	$C_7H_{14}O_2$	0.17
2	Methyl caprylate	$C_9H_{18}O_2$	5.01
3	Methyl caprate	$C_{11}H_{22}O_2$	6.04
4	Methyl laurate	$C_{13}H_{26}O_2$	35.01
5	Lauric acid	$C_{12}H_{24}O_2$	0.32
6	Methyl myristate	$C_{15}H_{30}O_2$	20.06
7	Methyl palmitate	$C_{17}H_{34}O_2$	13.04
8	Decanamide, N-(2-hydroxyethyl)-	$C_{12}H_{25}NO_2$	2.02
9	Methyl oleate	$C_{19}H_{36}O_2$	13.13
10	Methyl stearate	$C_{19}H_{38}O_2$	5.06
11	Stearic acid	$C_{18}H_{36}O_2$	0.14

As can be seen in Table 1, the sample is still dominated by the methyl esters, with only one nitrogen compound was identified. In addition, this nitrogen compound only contributes 2.02% to the composition of the sample, implying that the reaction condition applied to produce P-1 was not suitable for formation of nitrogen compounds.

The GC chromatogram of the P-2 sample is shown in Figure 3 and the compounds composing the sample are listed in Table 2.
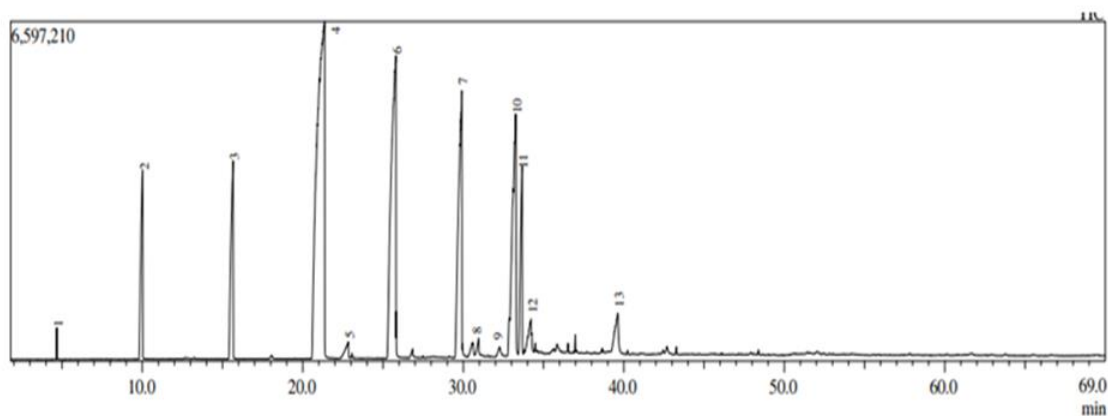

Figure 3. GC chromatogram of P-2 sample.

Table 2. Chemical composition of P-2 sample

No.	Compound Name	Chemical Formula	Relative Percentage (%)
1	Methyl caproate	$C_7H_{14}O_2$	0.19
2	Methyl caprylate	$C_9H_{18}O_2$	5.01
3	Methyl caprate	$C_{11}H_{22}O_2$	5.86
4	Methyl laurate	$C_{13}H_{26}O_2$	34.23
5	Lauric acid	$C_{12}H_{24}O_2$	0.71
6	Methyl myristate	$C_{15}H_{30}O_2$	19.47
7	Methyl palmitate	$C_{17}H_{34}O_2$	12.31
8	Palmitic acid	$C_{16}H_{32}O_2$	0.66
9	Decanamide, N-(2-hydroxyethyl)-	$C_{12}H_{25}NO_2$	0.45
10	Methyl elaidate	$C_{19}H_{36}O_2$	12.20
11	Methyl stearate	$C_{19}H_{38}O_2$	4.68
12	Elaidic acid	$C_{18}H_{34}O_2$	1.99
13	Lauric diethanolamide	$C_{16}H_{33}NO_3$	2.23

As demonstrated by the data in Table 2, the composition of the P-2 sample is also dominated by the methyl esters as the case with the P-1 sample. Despite the similarly of the

two samples, it can be noted that in the P-2 sample, there are two nitrogen compounds contributing 2.68% to the composition, while in the P-1 sample there is only one nitrogen compound with slightly smaller contribution (2.02%) to the composition.

Analysis of the P-3 sample produced the chromatogram as shown in Figure 4 and the identified chemical components of the sample are listed in Table 3. As can be seen, the chromatogram of the P-3 sample is significantly different from the chromatograms of the previous two samples, suggesting that this last sample is different in composition.
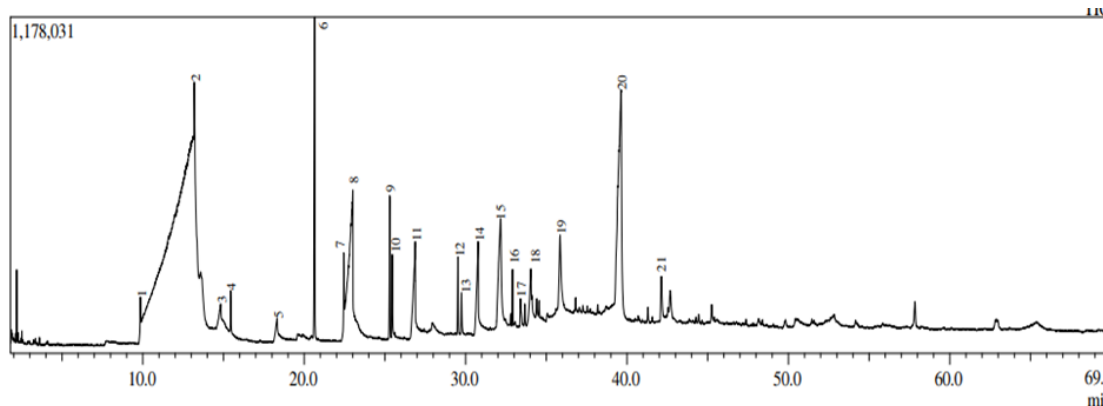

Figure 4. GC chromatogram of P-3 sample.

Table 3. Chemical composition of P-3 sample.

No.	Compound Name	Chemical Formula	Relative Percentage (%)
1	Methyl caprylate	$C_9H_{18}O_2$	0.77
2	Glycerine	$C_3H_8O_3$	2.35
3	Caprylic acid	$C_8H_{16}O_2$	1.36
4	Methyl caprate	$C_{11}H_{22}O_2$	1.06
5	Capric acid	$C_{10}H_{20}O_2$	1.77
6	Methyl laurate	$C_{13}H_{26}O_2$	9.78
7	Tripropylamine	$C_9H_{21}N$	1.85
8	Lauric acid	$C_{12}H_{24}O_2$	1.75
9	Methyl myristate	$C_{15}H_{30}O_2$	3.96
10	Hexanamide, N-(2-hydroxyethyl)-	$C_8H_{17}NO_2$	2.39
11	Myristic acid	$C_{14}H_{28}O_2$	5.60
12	Methyl palmitate	$C_{17}H_{34}O_2$	2.02
13	Caprylic acid monoethanol amide	$C_{10}H_{21}NO_2$	1.22
14	Palmitic acid	$C_{16}H_{32}O_2$	5.03
15	Decanamide, N-(2-hydroxyethyl)-	$C_{12}H_{25}NO_2$	12.38

No.	Compound Name	Chemical Formula	Relative Percentage (%)
16	Methyl oleate	$C_{19}H_{36}O_2$	1.53
17	Methyl stearate	$C_{19}H_{38}O_2$	1.50
18	Oleic acid	$C_{18}H_{34}O_2$	2.44
19	Stearic acid	$C_{18}H_{36}O_2$	5.53
20	Lauric diethanolamide	$C_{16}H_{33}NO_3$	33.23
21	1-Piperazineethanol	$C_6H_{14}N_2O$	2.47

By comparing the results obtained for the three samples, it can be seen that the P-3 sample significantly different from P-1 and P-2 samples. As can be seen in Table 3, as many as six nitrogen compounds were found in the P-3 sample, with a total contribution of 53.54% to the composition of the sample, making the nitrogen compounds the dominant components instead of methyl esters as found in the P-1 and P-2 samples. With respect to the results obtained for the three samples, it can be concluded that the experimental condition applied to produce P-3 sample is the best condition.

3. Corrosion rate measurement using the wheel test method

In this study, corrosion experiments were carried out using the P-1, P-2, and P-3 samples. As a comparison, an experiment without inhibitor was also conducted. The corrosion rates (CR) and percentage of protection (%P) are presented in Table 4.

Table 4. Percentage of protection achieved using different inhibitors.

No.	Inhibitor	C (ppm)	CR (mm/y)	%P
1	–	–	0.3362	0
2	P-1	150	0.0329	90.2
3	P-2	150	0.1222	63.6
4	P-3	150	0.0070	97.9

The experimental data in Table 4 indicate that the three inhibitors possess the ability to protect the sample from corrosion. Although no trend relating the performance of the inhibitor to the nitrogen content, it can be seen that the highest protection (97.9%) was provided by the P-3 sample, which is the sample containing the nitrogen compounds with the highest relative amount (53.54%), compared to less than 3% in the other two samples.

Although the need for further investigation should be acknowledged, the significantly different results obtained with the P-1 and P-2 samples is most likely associated with the presence of N-(2-hydroxyethyl)-decanamide. Despite the need for further investigation, the results obtained in this study demonstrated that the methyl esters of coconut oil could be

converted into nitrogen compounds and the nitrogen compounds produced exhibit promising potential as green corrosion inhibitor.

As a comparison with previous investigations, percent protections achieved in this study are relatively higher than that achieved using N and S co-doped carbon dots (N, S-CDs) as corrosion inhibitor for mild steel, in which 93% protection was reported [16]. In another study [17], the inhibition effect of tall oil diethylenetriamine imidazoline (TOFA/DETA imidazoline) on corrosion of mild steel in CO_2-saturated 3 wt% NaCl solution was investigated and found relatively lower protection (92%). On the other hand, relatively better result (99% protection) was reported with the use of benzimidazole derivative (BPMB) as corrosion inhibitor for carbon steel in CO_2-saturated brine solution under high turbulent flow condition [18]. The significant role of nitrogen compounds as corrosion inhibitor observed in this investigation is in agreement with the findings reported in previous studies [19–20] in which the activity of corrosion inhibitor increases with increased amount of nitrogen compounds composing the inhibitor.

4. Surface analysis

Another method to evaluate the ability of corrosion inhibitor to protect a sample is surface analysis using scanning electron microscopy (SEM). This method gives information about surface morphology of the sample, therefore, the morphological change due to corrosion can be visualized. SEM micrographs of the samples investigated in this study are shown in Figure 5.
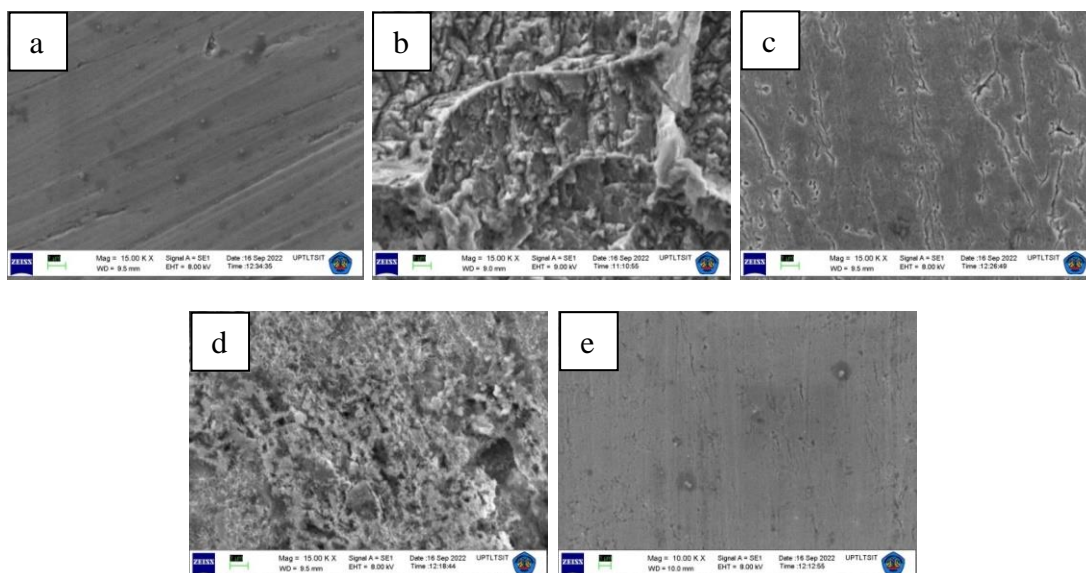

Figure 5. Micrographs of the mild steel samples investigated: (a) original sample, (b) sample treated without inhibitor, (c) sample treated with P-1 as inhibitor, (d) sample treated with P-2 as inhibitor, and (e) sample treated with P-3 as inhibitor.

Micrograph in Figure 5 showed clearly the effect of addition corrosion inhibitor to mild steel surface. Mild steel without treatment (Figure 5a) has a smooth surface appearance, with

fine scratches from the preparation process. This is very different from the surface morphology of mild steel treated without the use of inhibitor (Figure 5b). The surface morphologies of the samples treated with inhibitors are in agreement with the results of corrosion rate measurement experiments as previously described. Of particular interest is the result obtained with the use of P-3 inhibitor, in which the SEM image shows that the sample is well protected from the corrosion.

To justify that the protection of the sample from corrosion attack is attributable to the presence of nitrogen compounds, the sample treated with P-3 inhibitor was also analyzed using EDX together with the sample treated without inhibitor, and the results are presented in Figure 6.
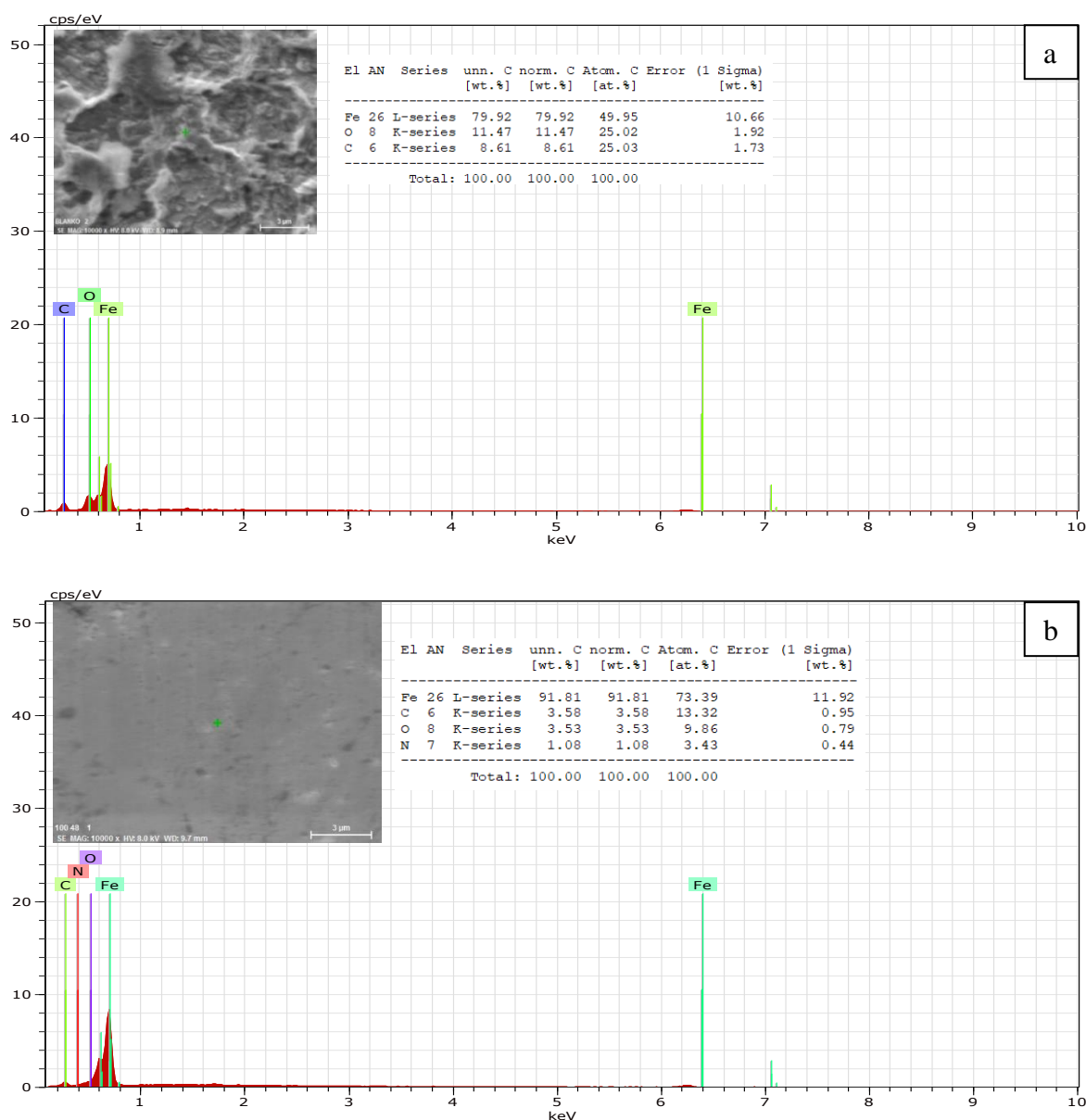

Figure 6. The elemental composition of the sample detected using EDX: (a) the sample treated without inhibitor, and (b) the sample treated with P-3 as inhibitor.

As can be seen in Figure 6, no nitrogen atom was detected in the sample treated without inhibitor (Figure 6a), while in the sample treated with P-3 inhibitor, the presence of nitrogen atom is clearly detected, thus justifies that the nitrogen compounds were attached onto the surface of the sample and function as protective layer.

Conclusion

The result obtained in this research demonstrated that methyl esters of coconut oil could be converted into nitrogen compounds. The results of corrosion testing experiments revealed that the samples exhibit corrosion inhibition activity, with the highest protection of 97.9% was provided by the sample prepared at 100°C for 48 h. The protection of the sample was also supported by the SEM image of the samples and the presence of nitrogen atom on the surface as shown by the EDX results. Without ignoring the need for further research, the results obtained in this study suggest that the nitrogen compounds derived from coconut oil possess promising potential as green corrosion inhibitor.

Acknowledgements

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