JBAT 7(2) (2018) 156 – 163

p-ISSN 2303 0623
e-ISSN 2407 2370

Jurnal Bahan Alam Terbarukan

Terakreditasi: SK No.: 36b/E/KPT/2016
http://journal .unnes.ac.id/nju/index.php/jbat

An Analysis of Metal Surface Immersed in Based Lubricant from Mineral Oil Containing Vegetable Oil with Rice Bran Oil Based Bio-inhibitor

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DOI 10.15294/jbat.v7i2.11623

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Article Info

Article history:
Received
October 2017
Accepted
August 2018
Published
December 2018

Keywords :
Based oil;
Bioinhibitor;
Rice bran oil;
Mineral oil;
Metal surface

Abstract

The need for environmentally friendly chemical products in daily needs encourages the production of its. The green-chemistry concept is using the process and produces chemical products that are ecofriendly. Including ecofriendly chemical products are base oil and additives for lubricants, grease, and fuels. The production is expected to reduce the consumption of mineral and synthetic base oils, so it will be biodegradable and renewable. This study compares the results of analysis of metallic surfaces immersed in the mixture of mineral and vegetable base oil, with the addition of rice bran oil bioadditive, ie epoxidized methyl ester (EME) and hydroxyl alkylbenzene sulphonic acid ester (HASE). The research method consists of preparing HASE; analyzing the effect of HASE and EME bioadditives addition on the mixture of base oil to the changing of metallic weight immersed in the mixture; determining the inhibition efficiency of the EME and HSAE additions; analyzing the metal surface using SEM-EDX (Scanning Electron Microscope) / (Energy Dispersive X-ray Spectrometry) to find images of microstructure and chemical compounds contained in specimens, and testing the metal difractogram immersed in base oil mixtures with bioadditive using XRD (X-Ray Diffraction). SEM test results of carbon steel immersed in a mixture of base oil and bioadditives show corrosion in which the metal surface color immersed in EME bioadditive mixtures is brighter. EDX spectra of metal sample surfaces immersed in a mixture of base oil, EME and HASE contain carbon (C) and iron (Fe). The carbon content in carbon steel samples immersed in the mixtures and HASE is higher. XRD test results show Fe_2O_3 phases in carbon steel samples immersed in the HASE bioadditive mixture are higher than in EME. While Fe_3O_4 phases in carbon steel samples immersed in the EME bioadditive mixture are higher than Fe_2O_3 phases in samples immersed in HASE bioadditive mixture.

INTRODUCTION

The need of ecofriendly chemical products for human life encourages its production. Including ecofriendly chemical products are base oil and additives for lubricants, grease, and fuels. Preparing biolubricants using the green chemistry concept applies the process which produces environmentally friendly chemical products, both base oil and its supporting materials, additives and catalysts. The production is expected to reduce the consumption of mineral and synthetic base oils, so it will be degradable and renewable. One type of additives is a corrosion inhibitor. Substitution of mineral oil by vegetable oil is hampered by the easiness of vegetable oils oxidized, so it is necessary to add additives that can prevent the oxidation process. Schafer et al. (1994) stated that a sulphonate-containing compound can prevent corrosion of mineral oil, grease, and vegetable oils. Holser (2008) explained that epoxy methyl ester products of transesterified epoxy oil may be utilized as surfactants, additives and base oils of industrial products.

Additives are added to the cooling stage of grease preparation, so they are dispersed in the

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ISSN 2303-0623
e-ISSN 2407-2370

grease matrix. Additives affect the functional properties of grease which has oxidation stability, resistance to bearing load, anticorrosive, antiwear, and antirust. Additives may also affect the solubility of the thickener in the base oil or at the crystallization stage (Adhvaryu et al., 2005). Azlina & Ali (2010) stated that the cost is cheaper compared to chemically modifying. Patent US 5368776 A states that metal inhibitor and alkali metal salt of high molecular sulfonic acid which are the result of neutralized sulphonated oil (petroleum sulphonate, synthetic sulphonates) are widely used. High-molecular compounds are adsorbed throughout the metal surface through sulphonic acid polar groups, which can inhibit anodic or cathodic reactions. The lipophilic portion results in high solubility in mineral oil. The Use of inhibitors with high metal content is limited because it can inhibit microbial degradation, resulting in low degradability. Therefore, synthesized additive compounds resemble sulphonates can prevent corrosion when used in mineral oil, grease, and vegetable oils. Nugrahani et al. (2016) and Nugrahani et al. (2017) undertook a bioinhibitor synthesis study containing sulphonic acid based on rice bran oil and its characterization.

US Patent Application 20060090393 states that epoxidized esters may reduce the corrosion of lubricants and fuels, and is good to replace parts or all of the anticorrosive used. Coupland & Smith (1981) stated that the epoxidation of the unsaturated bond of a carboxylic acid ester can be used as lubricant additives, thereby increasing the antiwear and anti-friction, forming a coating on the surface part of the moving metal. The addition of 12-hydroxy stearic acid improve antiwear properties (Farooq et al., 2011), and the addition of calcium fluoride increases the thermal stability (Azlina & Ali, 2010). Rowland & Migdal (2006) stated that the engine bearing is susceptible to corrosion, especially Cu-Pb bearing, so it is need to prevent the occurrence of wear, friction increase, loss of fuel and engine failure. Epoxidized methyl talat, epoxidized butyl talat, epoxidized 2-talat ethylhexyl, epoxidized octyl talat, epoxidized methyl oleate, epoxidized butyl oleate, epoxidized 2-ethylhexyl oleate, epoxidized octyl oleate, epoxdized methyl linoleate, epoxidized butyl linoleate, epoxidized 2-ethylhexyl linoleate, epoxidized octyl linoleate, epoxidized unsaturated oils such as epoxidized soybean oil, canola oil, cotton seed oil, palm oil, peanut oil can be used as additives. Dermawan (2011) stated the loss of some materials from the surface occurs due to abrasion, adhesion, erosion, tribochemistry, and fatigue. Engine wear can be prevented by the weight-bearing film layer, thus avoiding direct contact between the friction surfaces. This happens due to the addition of antiwear agent. Dermawan (2012) stated that molybdenum glycol, sulfonates, phenates, and salicylates - molybdenum prevent corrosion due to dissolve in the metal.

The performance test of corrosion inhibitor is done by immersing the metal in a solution for a certain time, and measuring the weight reduction, corrosion rate, Fourier Transform Infrared (FTIR), and metal surface test (Anbarasi et al., 2013). The SEM test is performed to determine the corrosion of metallic surfaces immersed into a solution containing corrosion inhibitor; XRD test is taken to determine the existence of corrosion products (Aini & Triwikantoro, 2010). The reaction occuring on corrosion testing is known from the EDX test (Bayuseno & Handoko, 2012). Ningsih (2017) analyzed the oxidative stability and corrosion resistance of the SEM and XRD test results on metal surfaces immersed in base oils of vegetable oil mixture without bioadditive on mineral lubricants oil.

This study compares the results of metallic surface analysis dipped in the mineral and vegetable base oil mixtures with the addition of Rice Bran Oil bioadditive, ie EME and HASE.

RESEARCH METHODOLOGY

Material

Epoxidized methyl ester rice bran oil (with the oxirane number : 1.7%), linear alkyl benzene sulfonic acid (LABSA), base oils of mineral oil lubricants, rice bran oil, coconut oil, aquades, and metal plate.

Equipment

Glass beaker, thermometer, magnetic stirrer, analytical scales, SEM / EDX (Scanning Electron Microscope) / (Energy Dispersive X-ray Spectrometry) Method of LUM - IK - 06-01, XRD diffractogram (X - Ray Diffraction).

Experiment

HASE Praparation

EME and LABSA were mixed with a ratio of 1: 1.3 (w / w) to the beaker glass (Nugrahani et

Table 1. The results of the metal weight change test dyed in a base oil mixture of mineral oil and vegetable oil (rice bran oil and coconut oil)

Experimental Condition	Sample 1			Sample 2
	Sample a	Sample b	Sample c	
Metal weight before dyed in base mixture oil, W_1, g	55.95	56.91	57.31	55.90
Metal weight after dyed in base mixture oil, W_2, g	56.19	56.94	57.42	55.92
Weight Changing, ΔW, g (equation 1)	0.24	0.03	0.11	0.02

al., 2016). The mixture was heated at 70°C for 4 hours while stirring with a magnetic stirrer (Nugrahani et al., 2017). The test of the product is the oxirane number.

Testing the effects of bioadditive addition of EME and HASE on the base oil mixture.

Corrosion solution is the mixture of based oil lubricant and water. The sample was then put into the solution. The corrosion process is carried out for 5 hours (Al-Sabagh et al., (2012) had ermormed it for 48 hours), then the sample was cleaned and dried in the oven. Corrosion behavior was tested by weight loss method.

Mineral oil lubricant base oil was mixed with rice bran oil and coconut oil, and addition of bioadditive 1 (EME) and bioadditive 2 (HASE).

Sample 1 consist of Base oil mixture of mineral oil lubricant, vegetable oil (rice bran oil, coconut oil) HASE additive, and water. The metals were prepared and weighed. All ingredients were mixed until homogeneous. It was then heated at 60-70°C for 15 minutes. Each metal was placed into solution and stand for 3 days. Samples were then weighed after day 3.

Sample 2 consist of base oils mixture of mineral lubricants and vegetable oils (rice bran oil, coconut oil), EME additives, and water. Metals were prepared and weighed. All ingredients were mixed until homogeneous. It was then heated at 60-70°C for 15 minutes. The metal was placed into solution and stand for 3 days. The samples were then weghed after day 3.

Analysis of Weight Changes and Inhibition Efficiency

The metal weight loss test (Eq. 1) which has been immersed in corrosive solution can be used to find the Corrosion Inhibition (Eq. 2) (Kumpawat et al, 2012).

$$\Delta W = W_1 - W_2 \quad (1)$$

$$(\eta_w)\% = (\Delta W - \Delta Wi) / \Delta W \quad (2)$$

Where, $(\eta_w)\%$ is the percentage of inhibition efficiency, ΔW and ΔW_i are the weight loss of the metal in uninhibited and in inhibited solution respectively, W_1 and W_2 are the weight of the metal before and after immersed in solution.

Analysis of Metal Surface

On the metal surface with the largest weight changes, the SEM-EDX is tested to identify the microstructure and composition (chemical compounds) contained in the specimen, and tests the diffractogram using XRD on metals immersed in mineral oil and vegetable oil mixtures coupled with bioadditives.

RESULTS DAN DISCUSSION

Characteristics of HASE Products

HASE was synthesized from EME rice bran oil with an initial the oxirane number of 1.7% through an oxiration ring opening reaction by LABSA at a condition as in the research of Nugrahani et al. (2017) temperature of 70°C for 4 hours. The product formed was analyzed by the change of the oxirane number, which decreased to 0.72%; it shows that not all the oxirane rings are opened. Furthermore, the resulting HASE is used as a bioadditive in mineral and vegetable base oil mixtures and are compared to use EME bioadditive.

Analysis of specimen metal weight change test

The mixture of mineral oil, vegetable oil (rice bran oil and coconut oil), and the addition of bioadditive 1 (EME) and bioadditive 2 (HASE) are further tested for effects on the metal surface corrosiveness, using metal weight parameters before and after immersion in the mixture. The metal weight test results dipped in the mixture can be seen in Table 1.

Table 1 shows the changes in metal weight in Sample 1 (a,b,and c) on average of 0.126 g and in Sample 2 of 0.02g. When compared with previous research (Ningsih et al., 2017) i.e. dipping the metal into base oils mixture of mineral oil and vegetable oil without the use of additives, the change in the

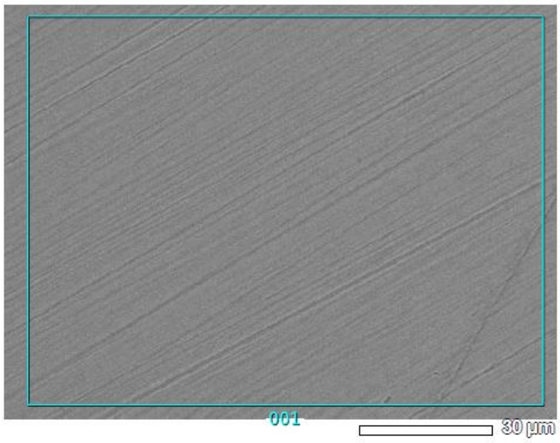

Figure 1. Results of SEM carbon steel test in Sample 1 (Base Oil Mixture and Bioadditive HASE)

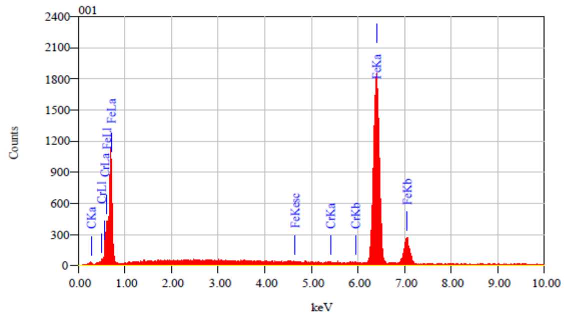

Figure 2. Results of the EDX carbon steel Test in Sample 1 (Base Oil Mixture and Bioadditive HASE).

metal weight is even 0.36g; this indicates that the addition of bioadditive affects minimizing the change in metal weight.

HASE and EME can each serve as a corrosion inhibitor that may be caused by the addition of vegetable oils. HASE contains sulfonic groups, in accordance with US Pat. 5368776 A that inhibitors of metal and alkali metal salts of high molecular sulfonic acid which are the result of neutralization of sulfonated oil (petroleum sulfonate, synthetic sulfonate) are widely used. High-molecular compounds are adsorbed throughout the metal surface through sulphonic acid polar groups, which can inhibit anodic or cathodic reactions. The lipophilic portion results in high solubility in mineral oil. Use of inhibitors with high metal content is limited because it can inhibit microbial degradation, resulting in low degradability. While EME is the epoxidation product of unsaturated carboxylic acid bonds; an ester can be used as a lubricant additive enhancing antiwear and antifriction, forming coatings on the surface of moving metal parts (US Pat. 4244829 and Holser, 2008).

The results show the effect of HASE addition to changes in metallic weight is greater than EME, this is because HASE contains acid derived from LABSA, so it is likely to result in greater corrosion or scale compared to the addition of EME .

The inhibition efficiency can be calculated using the weight change (equation 1) of Table 1 and the efficiency is calculated using equation 2. The inhibition efficiency with HASE addition is 65% and with EME addition is 94%; these values indicate that EME is more effective in preventing the occurrence of corrosion due to the base oil mixing of vegetable oils and mineral oil. In a study conducted by Kumpawat et al. (2012) the weight change in the tin metal immersed in 3.0 M HNO_3 solution without inhibitors was 285 mg and after the addition of bioinhibitor from basil leaf extract (Tulsi) showed an inhibitory efficiency of 92.38% (w/w).

Analysis of metal surface test **results immersed in a base oil mixture**

SEM / EDX Test Results

The metal surface test is performed on metals dyed on Sample 1 (with HASE bioadditive) compared to Sample 2 (with EME bioadditive) using the SEM/EDX test. The test is to determine the microstructure image of the specimen and the composition (chemical compound contained) on the specimen. The results of the test with the addition of HASE bioadditive can be seen in Figures 1 and 2.

Figure 1 is the test result of SEM carbon steel which is immersed in a mixture of mineral oil with rice bran oil dan coconut oil, with the addition of a HASE bioadditive at concentration of 3.8% (v/v), thereby reducing the lowest metal weight in Sample 1a Table 1 . This indicates that in the metal micro-structure there is corrosion, showing dark grey color, white and black lines. The semi-quantitative calculations of EDX with the HASE bioadditive can be seen in Figure 2 and Table 2.

Figures 2 and Table 2 show carbon content in carbon steel samples with HASE bioadditive, it can be concluded that the sample is included in the classification of high carbon steels and the sample is exposed to corrosion.

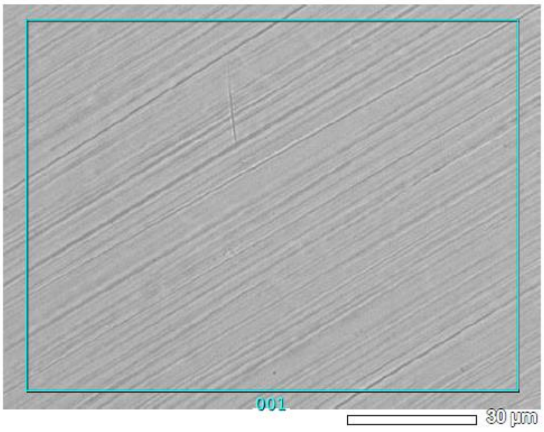

Figure 3. Results of SEM carbon steel test in Sample 2 (Base Oil and EME Bioadditive mixture)

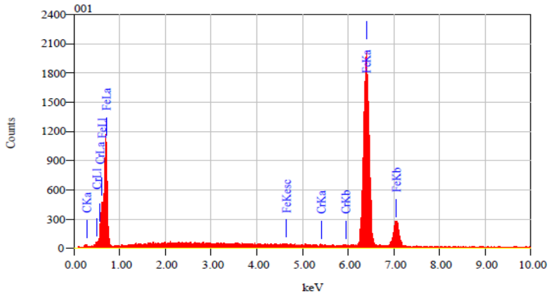

Figure 4. Results of the EDX carbon steel Test in Sample 2 (Base Oil and EME Bioadditive Mixture).

Table 2. Semi-quantitative calculation of the SEM-EDX spectrum with HASE bioadditive.

No	Elements	Content (% mass)
1	Carbon (C)	1.91
2	Chrome (Cr)	0.07
3	Iron (Fe)	98.02

Spectrum EDX (energy dispersive spectroscopy) on the surface of metal samples immersed in a mixture of mineral oil, vegetable oil, and HASE is shown in Figure 2. Qualitative results of EDX show the sample material is carbon steel with carbon content of 1.91%. Analysis result of using EDX obtained that most element contained in this carbon steel sample is iron (Fe). There are some other elements contained in this carbon steel in a relatively small amount as shown in Table 2.

Further, the test results of metallic surfaces immersed in a mixture of mineral oil and vegetable oils, with the addition of bioadditive EME can be seen in Figures 3 and 4.

Figure 3 is the test result of a SEM carbon steel immersed in a mixture of mineral oil and vegetable oil with the addition of an EME bioadditive at a concentration of 4% (v/v), thereby reducing the metal weight (Sample 2 in Table 1). This indicates that on the metal microstructure there is corrosion, showing brighter gray color (as compared to Figure 1), white and black lines. Semi-Quantitative calculations of EDX with EME bioadditive can be seen in Figures 4 and Table 3.

Figure 4 and Table 3 show carbon content in carbon steel samples with EME bioadditive, it can be concluded that the sample belongs to high carbon steels classification and the sample is exposed to corrosion.

Spectrum EDX (energy dispersive spectroscopy) on the surface of metallic samples immersed in a mixture of mineral oil, vegetable oil, and EME is shown in Figure 4. The qualitative EDX results show that the sample material is carbon steel with a carbon content of 1.64%. The analysis result by using EDX obtained that the most element contained in this carbon steel samples is iron (Fe). There are other elements contained in this carbon steel in a relatively small amount as shown in Table 3.

Table 3. Semi-quantitative calculations of EDX with EME bioadditive

No	Elements	Content (% mass)
1	Carbon (C)	1.64
2	Chrome (Cr)	0.17
3	Iron (Fe)	98.19

The carbon content of carbon steel samples immersed in a mixture of mineral oil, vegetable oils and bioadditives HASE is higher than the carbon content of carbon steel samples immersed in a mixture of mineral oils, vegetable oils and EME bioadditive. It can be concluded that the samples include to the high carbon steels classification and the sample is exposed to corrosion.

Comparing with previous studies without bioadditives, the results of the SEM/EDX spectrum indicate an element O (Ningsih et al., 2017), this indicates that the wet metal surface results in the least oxygen contained in the electrolyte. This

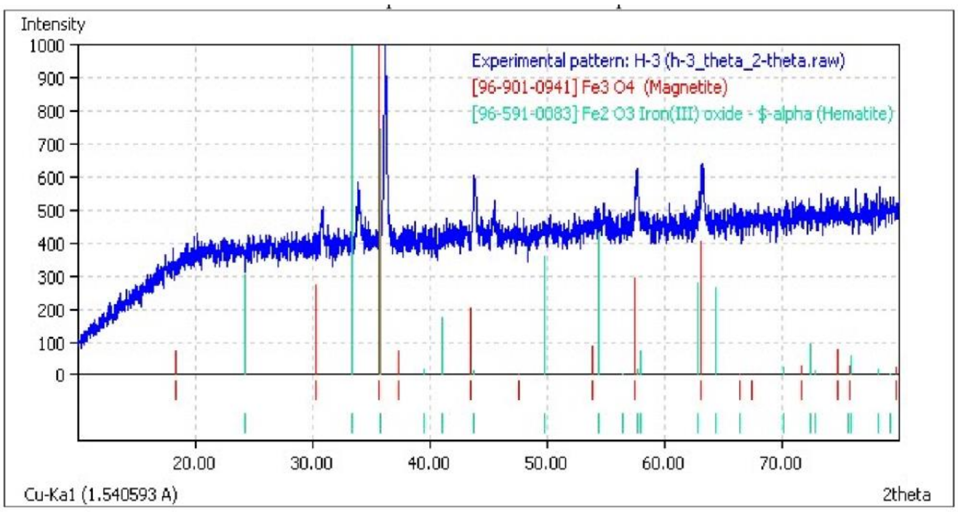

Figure 5. XRD carbon steel test results in metal sample 1a, in base oil and HASE bioadditive mixture.

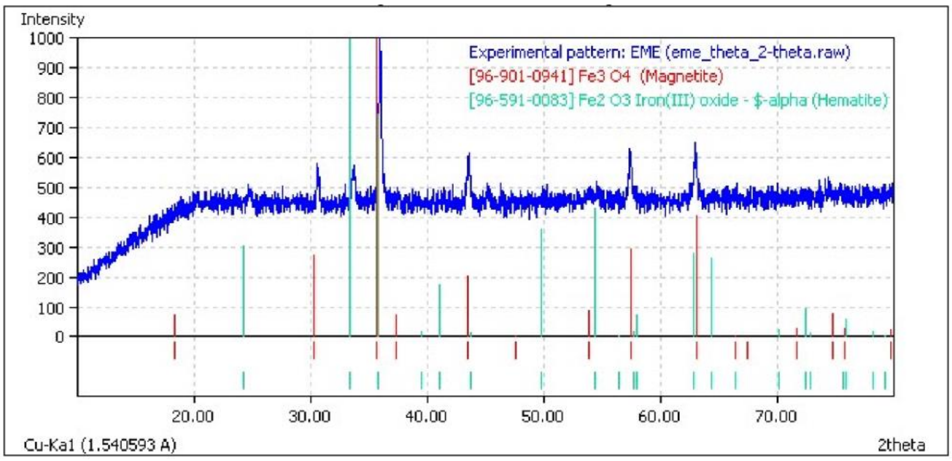

Figure 6. XRD carbon steel test results in metal sample 2, in base oil and EME bioadditive mixture

results in a path through which oxygen passes toward the metal acting as an anode. The occuring metal dissolution causes the precipitation of corrosion products around the pitting and rust formed (Aini & Triwikantoro, 2012).

Samples of carbon steel immersed in mineral oil and vegetable oil mixtures with the addition of EME belong to high carbon steels; while some elements contained in this carbon steel may not stand on their own but bond to one another forming certain minerals according to the largest content of steel carbon. Thus a structural analysis is required to identify the phases presenting in carbon steel soaked in a mixture of mineral oil and vegetable oils by addition of EME.

XRD Test Result

The metal surface test to know the difractogram is carried out on metals soaked in a mixture of mineral oil and vegetable oil with the addition of HASE (sample 1a) compared to Sample 2 using XRD test can be seen in Figures 5 and 6.

XRD carbon steel test result in Sample 1a (base oil and HASE bioadditive mixture)

Figure 5 shows that the material surface is both rusted and oxidized; it is made clear by the change of the chemical composition of the material surface by the bioadditive, where there is an oxygen element contained in the material. When oxygen reacts with the metal surface, there will be a reaction between oxygen and metal. The reaction is the oxidation reaction, which is the combination of oxygen with metal to form rust. Figure 5 shows the analysis results of XRD (X-Ray Diffraction) identifying the presence of passiva layers on the steel surface which is in the form of Fe_3O_4 (Magnetite) and Fe_2O_3 iron (III) oxide with a ratio of 67.75% to 32.35% (Finger & Hazen, 1980; Gatta et.al., 2007).

XRD carbon steel test results in sample 2 (base oil and EME bioadditive mixture)

Figure 6 shows the material surface was rusted and oxidized; this is made clear by the change in chemical composition of the surface material by the bioadditive, where there is an oxygen element contained in the material. In the case of carbon steel samples, when oxygen reacts with the metal surface there will be a reaction between oxygen and metal; this is the oxidation reaction, which is the combination of oxygen with metal to form rust. Figure 6 shows the analysis results of XRD (X-Ray Diffraction) identifying the presence of pasiva layers on the steel surface in the form of Fe_3O_4 (Magnetite) and Fe_2O_3 iron (III) oxide with a ratio of 68.43% to 31.57% (Finger & Hazen, 1980; Gatta et.al., 2007)

Fe_2O_3 phases in carbon steel samples immersed in the mixture with HASE bioadditive are higher than Fe_2O_3 phases in samples immersed in mixtures with EME bioadditive. While Fe_3O_4 phases in carbon steel samples immersed in mixtures with EME bioadditive are higher than Fe_2O_3 phases in samples immersed in mixtures with HASE bioadditive.

Being compared with previous studies without bioadditives, the results of the SEM / EDX spectrum indicate the presence of element O; this indicates the metal dissolution that causes the precipitation of the corrosion product. The EDX spectrum peak indicates the FeO corrosion product, as well as the corroded area (Ningsih et al, 2017).

CONCLUSIONS

SEM test result of carbon steel immersed in the mixture of mineral oil, vegetable oil and the addition of both HASE and EME bioadditives at concentration of 3.8% - 4% (v / v) decreased weight; this shows on the metal microstructure there is corrosion. The color of metallic surfaces immersed in the mixture of bioadditive EME is a brighter compared to HASE. The EDX spectrum on the metal sample surface immersed in a mixture of mineral oil and vegetable oil with EME and HASE contains carbon (C) and mostly is iron (Fe); in addition there are several other elements in relatively small amounts. The carbon content of carbon steel samples immersed in the mixture of mineral oil and vegetable oils with HASE bio-additive is higher than the carbon content of carbon steel samples immersed in a mixture of mineral oils and vegetable oils with EME bioadditive. It can be concluded that the samples include into the high carbon steels classification and is exposed to corrosion. XRD test results show Fe_2O_3 phases on carbon steel samples immersed in mixtures of HASE bioadditive is higher than Fe_2O_3 phases on samples immersed in mixtures with EME bioadditive. While Fe_3O_4 phases in carbon steel samples immersed in mixtures of EME bioadditive is higher than Fe_2O_3 phases in samples immersed in mixtures with HASE bioadditive.

ACKNOWLEDGEMENTS

Gratitude is highly sent to the Director of Research and Community Service, Directorate General Research Strengthening and Development of the Ministry of Research, Technology and Higher Education. In accordance with the Letter of Agreement on the Implementation of Research Grant No. 0404 / K3 / KM / 2017, May 24, 2017. Abundant thank is also sent to the Department of Chemical Engineering, Universitas Muhammadiyah Jakarta, for all facilities provided during this research.

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