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Hydroxylation Reaction on Bio-lubricant Characteristics: A Case Study on Palm Kernel Oil

Fatmayati[1]✉, Razita Hariani[2], Nur Asma Deli[3]

[1, 2, 3] Study Program of Palm Oil Processing Engineering, Kampar Polytechnic, Indonesia

✉ **Corresponding Author :** Fatmayati Fatmayati (e-mail: fatmayati80@gmail.com)

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ABSTRACT

Historically, lubricants have relied on petroleum derivatives, but depleting resources necessitate alternatives. Vegetable oils are unstable for use due to oxidation but can be improved via chemical modification. This study examines bio-lubricant production from palm kernel oil, focusing on hydroxylation reaction time and compliance with SNI 06.7069:2012 standards. The production of bio lubricants in this study comprised three distinct stages: hydrolysis, epoxidation, and hydroxylation. Hydrolysis involves the processing of palm kernel oil to yield fatty acids and glycerol. Subsequently, the fatty acids produced during the hydrolysis reaction were epoxidized to yield the epoxy compounds. Subsequently, the epoxy compound is subjected to hydroxylation, resulting in the formation of a polyol compound that serves as the fundamental building block for the bio lubricants. Optimal bio lubricants outcomes were achieved with a hydroxylation reaction time of 240 min, resulting in a bio lubricants yield of 92.67, a kinematic viscosity value of 48.54 Cst at 313.15°K/40°C, a kinematic viscosity value of 10.89 Cst at 373.15°K/100°C, a viscosity index of 224, and a corrosion rate of 0.1496 mpy. The quality of the bio lubricants produced in this study was in accordance with the SNI 06.7069:2012 standard.

INTRODUCTION

The depletion of fossil fuel reserves has prompted the development of alternative energy sources utilising renewable raw materials (Lubad & Paramita W, 2022). Similarly, base oil is another energy source that has been affected by this depletion. In light of the current decline in petroleum availability, it is imperative to develop technology for the production of base lubricants from alternative materials. One potential source for this purpose is oil derived from plants, specifically vegetable oil, which has the potential to be developed as a bio-lubricant material (Said et al., 2017).

The processing of vegetable oil into base lubricating oil offers a number of advantages, including the ease with which it decomposes in the environment. Additionally, vegetable oil is non-toxic and does not harm the natural environment. Furthermore, it is a plentiful resource that can be replenished. One potential source of raw material for lubricating oil is palm kernel oil (Siregar, 2006). The fundamental constituents of lubricants derived from vegetable oils are unsuitable for direct utilisation due to their inherently unstable nature, particularly in the context of oxidation reactions when vegetable oils are employed as the primary lubricant component. Vegetable oils possess double bonds, which contribute to the aforementioned instability (Santi R

et al., 2016). The oxidation stability of vegetable oil lubricants can be enhanced through the modification of chemical compounds.

The process of producing bio-lubricants from vegetable oils is a sequential one, beginning with the hydrolysis of triglycerides into glycerol and fatty acids. This is followed by the conversion of unsaturated fatty acid bonds into saturated ones through the application of fatty acid epoxidation reactions, which result in the formation of fatty acid compounds containing oxirane groups. The final stage of the process of producing bio-lubricants is a hydroxylation reaction (Said et al., 2017). This involves the addition of a hydroxy group to the fatty acid oxirane group, which is also known as the oxirane ring opening reaction in the epoxide compound. The hydroxylation reaction will result in the formation of hydroxyl compounds in the form of polyol or bio-lubricants (Said et al., 2017) (Susetyo et al., 2016).

A research project was initiated based on the available information, with the objective of acquiring data regarding the impact of hydroxylation reaction time on the quality of bio-lubricant s produced in accordance with the standards set forth in SNI.06.7069: 2012. The objective of this study is to ascertain the production process of bio-lubricants derived from palm kernel oil. To determine the impact of hydroxylation reaction time on the characteristics of bio-lubricants manufactured from palm kernel oil and to evaluate whether the bio-lubricants produced from palm kernel oil comply with the requirements set forth in SNI.06.7069: 2012 (Badan Standardisasi Nasional, 2012)

RESEARCH METHODS

Materials and Equipment

The materials employed in this research experiment include palm kernel oil and palm kernel shell obtained from PTPN V KCP Tandun, distilled water (H_2O), acetic acid (CH_3COOH), phosphoric acid (H_3PO_4), sulfuric acid (H_2SO_4), 30% hydrogen peroxide (H_2O_2), 37% hydrochloric acid (HCl), sodium hydroxide (NaOH), n-hexane (C_6H_14) and Etanol (C_2H_6O). The equipment employed in this research comprises glassware, nabertherm muffle furnace, hot plate magnetic stirrer, memmert oven laboratorium, reflux condenser, rotary evaporator buchi brand, thermometer, spatula, and analytical balance.

Methods

The method employed utilises palm kernel oil and is comprised of three distinct stages: hydrolysis reaction, epoxidation reaction and hydroxylation reaction. The independent variable that is varied in the process of producing bio-lubricants is the variation of reaction time in the hydroxylation process. First, a flowchart illustrating the palm kernel oil hydrolysis process is presented in Figure 1.

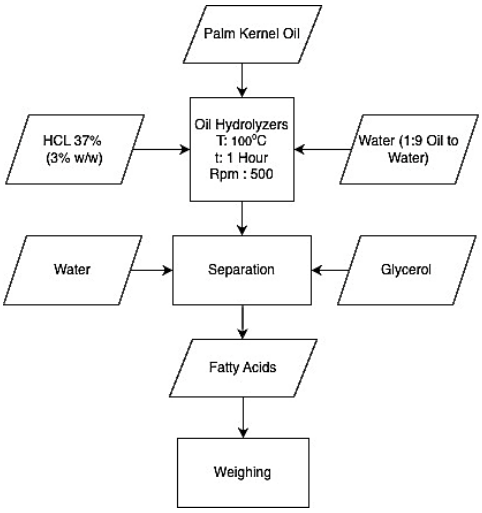

Figure 1. Flowchart of palm kernel oil hydrolysis process

The initial stage of the process of modifying palm kernel oil into bio-lubricant (polyol compounds) is the hydrolysis reaction. In this reaction, the triglyceride compounds present in palm kernel oil are broken down into glycerol and fatty acids. The reaction is conducted at 373,15°K (100°C) with the use of a hydrochloric acid catalyst for a period of one hour. The HCl solution functions as a potent acid catalyst, capable of generating the highest yield in comparison to other acid catalysts, such as H_2SO_4 and HNO_3 solutions (Dewi et al., 2018).

In the hydrolysis process, 0,2 kg of palm kernel oil is introduced into a reactor containing 1,8 kg of water, resulting in a reactant ratio of 1:9 (oil:water). In order to accelerate the reaction rate, a catalyst is added to the reaction. Once the temperature reached 373,15°K (100°C), the HCl catalyst was added in an amount equivalent to 5% of the weight of the raw material, or 0,006 kg. Upon completion of the designated reaction time, the hydrolysis product will manifest in two distinct phases. The reaction results in the formation of water and glycerol in the lower phase, and fatty acids in the upper phase. The hydrolysis results yielded a total of 0,194.2 kg, which will subsequently be transferred to the subsequent stage of bio-lubricant, namely the epoxidation reaction process, in an amount of 0,150 kg.

Secondly, a flowchart illustrating the reaction process of epoxidation compounds with palm kernel oil fatty acid compounds is provided in figure 2.

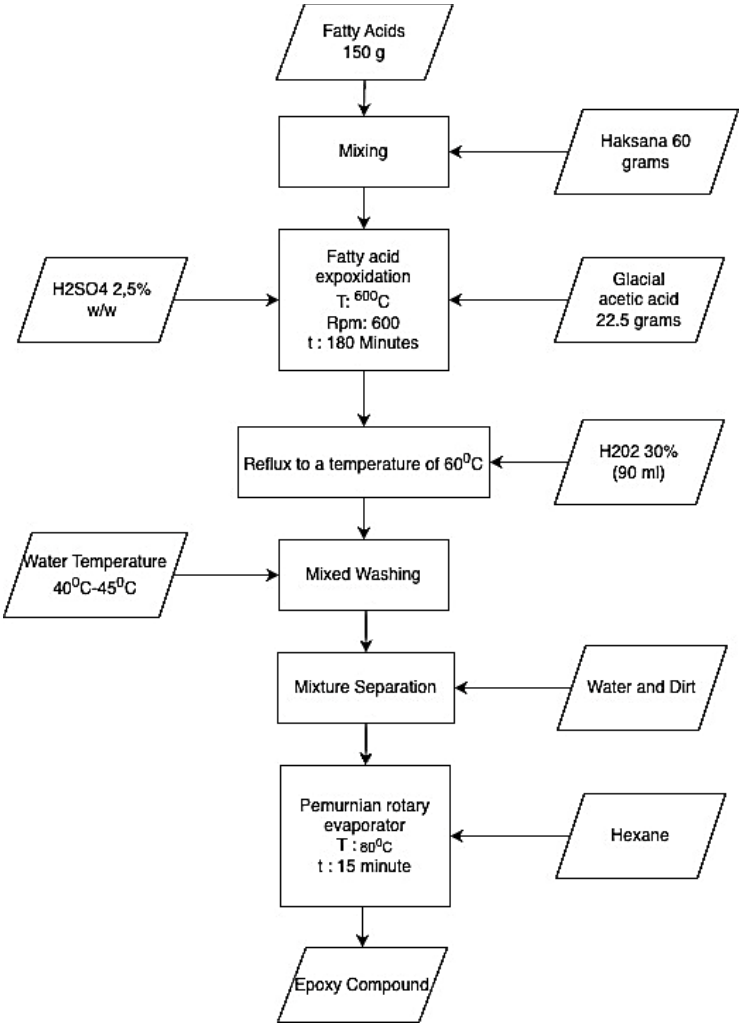

Figure 2. Flowchart of the reaction process of epoxidation compounds to palm kernel oil fatty acid compounds

The epoxidation reaction process is defined as the process of forming epoxy compounds. Epoxy compounds are defined as those containing oxirane groups. The reaction between peroxy acid and fatty acids resulting from the hydrolysis process can yield chemical compounds that

contain oxirane groups. The peroxy acid employed in the epoxidation process under investigation is peracetic acid. The rationale for selecting peroxide acid is that the compound is more readily accessible, exhibits relatively high efficiency when reacted with a relatively low-cost material, and offers a cost-effective alternative (Abdullah, 2012).

The synthesis of epoxy compounds commences with the preparation of 0,150 kg of fatty acids via the hydrolysis process. Moreover, the solvent is introduced, specifically hexane in an amount of 0,060 kg and H_2SO_4 catalyst in an amount of 2.5% (w/w), which is 0,00375 kg. Some research findings indicate that sulfuric acid is an effective catalyst for epoxidation reactions at a relatively low cost. Nevertheless, the utilisation of sulfuric acid catalysts has the potential to result in the degradation of the oxirane ring. The degradation of the oxirane ring can be mitigated by adjusting the quantity of catalyst and incorporating a solvent.

In the epoxidation process, the addition of a hexane solution is carried out with the objective of facilitating the binding of peracetic acid to the organic phase, which can ultimately result in a reduction in acid concentration in the organic phase. In order to ensure that the addition of the hexane solution has no impact on the rate of reaction in the epoxidation process (Wibowo & Rusmandana, 2013).

Thirdly, a flowchart illustrating the hydroxylation reaction process of palm kernel oil fatty acid epoxy compounds is provided in figure 3.

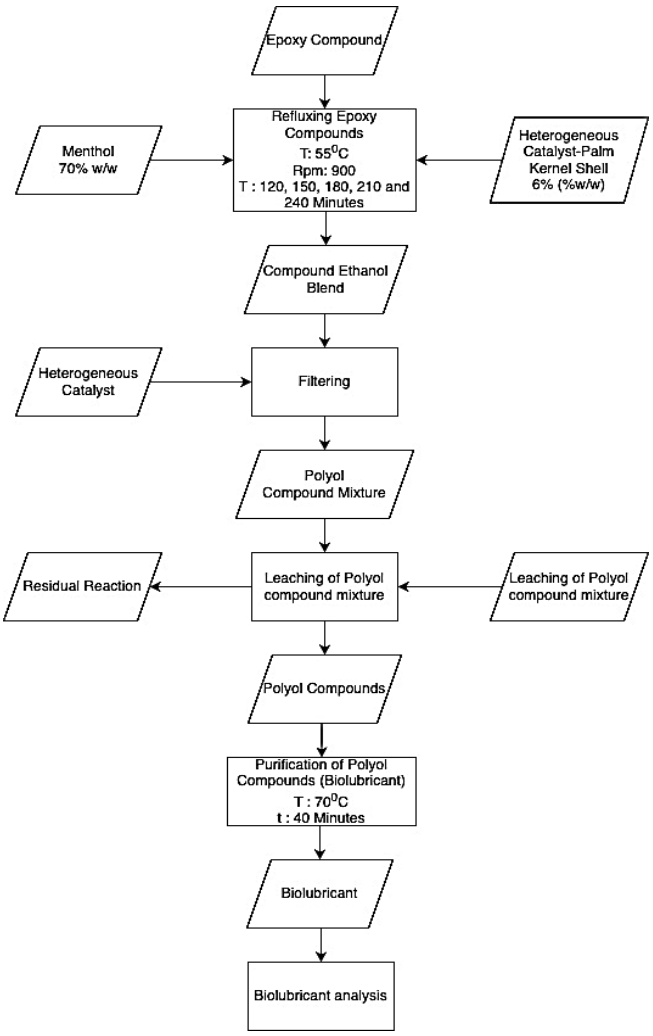

Figure 3. Flowchart of the hydroxylation reaction process of palm kernel oil fatty acid epoxy compounds.

Once the epoxy compound has been produced, the hydroxylation process is initiated. In this process, the epoxidised oil is reacted with methanol, and a palm shell heterogeneous catalyst is added in order to facilitate the formation of hydroxyl groups from the opening of the epoxide ring. The initial step involved the weighing of the resulting epoxy compound and the addition of methanol at a ratio of 70% of the mass of the epoxy compound. In this study, methanol was selected as the reactant due to its status as the smallest molecular weight alcohol compound. Methanol (MeOH) has a boiling point of 337,85°K, but during the course of the reaction, it will evaporate before reaching this temperature. MeOH is an alcohol compound that rapidly reacts with triglycerides due to its shortest carbon chain.

Additionally, it is relatively inexpensive (Dr. Ir. LA IFA, ST, MT, IPM et al., 2018). Once the temperature reached 55°C, the palm kernel shell heterogeneous catalyst was introduced into a two-neck flask in an amount corresponding to 6% of the weight of the raw material. The reaction was then conducted over a range of time periods, namely 120, 150, 180, 210 and 240 minutes. The utilisation of catalysts in the hydroxylation reaction is intended to facilitate the acceleration of the reaction time. Once the reaction had reached completion, the catalyst was separated by filtration and the reaction mixture was transferred to a separatory funnel containing warm water for further processing. Moreover, the reaction products were subjected to a rotary evaporation process at 343,15°K (70°C) for 40 minutes, with the objective of eliminating any residual unreacted reagents and water from the previous washing stage.

Testing Bio-lubricants from Palm Kernel Oil

The bio-lubricant product produced in this study was subsequently evaluated for its characteristics with the objective of determining whether the observed characteristic value was in alignment with the established standard for bio-lubricant quality parameters as outlined in SNI.06.7069.9:2012 (Badan Standardisasi Nasional, 2012).

The testing procedures for bio lubricant characteristics were in accordance with the Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (ASTM D445, 2018). Viscosity index is a quality parameter for bio-lubricants. The viscosity index value was derived from the kinematic viscosity value at 313.15°K (40°C) and 373.15°K (100°C). The aforementioned value was then tested in accordance with the procedures set forth in ASTM D2270 (ASTM-D2270, 2016).

This study also sought to ascertain the corrosion rate of a material that was applied to the bio-lubricants produced in this study. The objective of the corrosion rate test is to ascertain the efficacy of bio-lubricants in preventing corrosion in a material. The methodology employed for the corrosion rate testing procedure was based on the findings of a previous study that analyzed the corrosion rate of carbon steel plates (Kurniawan Afandi et al., 2015).

RESULTS AND DISCUSSION

The process of synthesising polyol or biolubricant compounds from palm kernel oil encompasses a series of stages. The initial stage involves the preparation of the raw materials; the hydrolysis reactions employed to obtain the fatty acids present in the palm kernel oil. Following this, the second stage entails the epoxidation reaction process, while the final stage comprises the hydroxylation reaction process

The experiments conducted in this study employed the in situ epoxidation process, which entails the simultaneous production of peracetic acid (a reaction between hydrogen peroxide and acetic acid) within the reactor. Glacial acetic acid was weighed in quantities of up to 0,0225 kg at a temperature of 333,15°K (60°C). Thereafter, 30% hydrogen peroxide was added in quantities of up to 150ml, with the temperature maintained at a constant level. In order to separate the epoxy compound from any residual impurities, such as residual hydrogen peroxide and unreacted catalysts, it is necessary to mix the compound with hexane and then separate it using a rotary vacuum at 353,15°K (80°C) for 15 minutes. The data pertaining to the epoxidation process are presented in Table 1.

Table 1. Yield of Epoxidation Reaction

Sample	*Yield* (kg)
V1	0,16367
V2	0,15824
V3	0,15996
V4	0,16239
V5	0,15714

Once the epoxy compound has been obtained, the next stage in the production of this bio-lubricant is the hydroxylation reaction. Subsequently, the epoxidation reaction must be immediately followed by the hydroxylation reaction in order to prevent the formation of undesired by-products. The formation of by-products may impact the yield of the subsequent hydroxylation reaction. The data pertaining to the hydroxylation process are presented in Table 2.

Table 2. Hydroxylation reaction yields

Sample	*Yield (kg)*
V1 = 120 minutes	0,1368
V2 = 150 minutes	0,1384
V3 = 180 minutes	0,1381
V4 = 210 minutes	0,1388
V5 = 240 minutes	0,1392

Table 2 presents the findings of the hydroxylation reaction. The results of the hydroxylation process are organic compounds in the form of hydroxyl compounds, also known as polyol compounds (bio-lubricant). Subsequently, the bio-lubricant was subjected to a series of tests to ascertain its characteristics and compare them with those of the bio-lubricant produced from palm kernel oil as a result of the research.

Characteristics of Bio-lubricants from Palm Kernel Oil

The quality of a bio-lubricant is contingent upon the characteristics of the lubricant in question, which must align with the standards set forth in the Indonesian National Standard SNI 06.7069: 2012 (Badan Standardisasi Nasional, 2012).

1. Yield of bio-lubricants

The data pertaining to the calculation of the yield of the bio-lubricant produced in this study can be found in Figure 4.

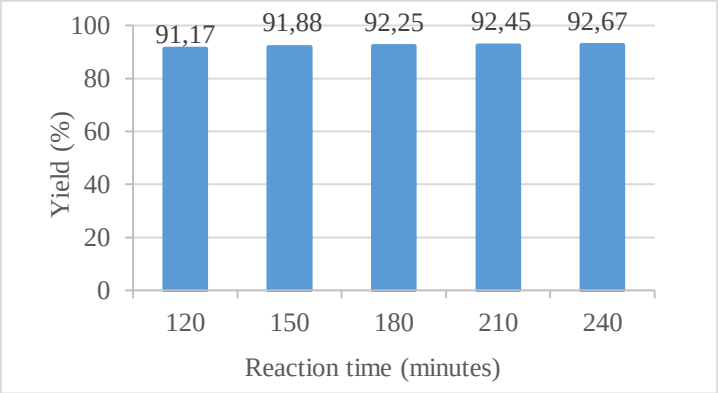

Figure 4: Graph of the effect of hydroxylation reaction time on bio-lubricant *yield*.

Figure 4 illustrates the yield of the bio-lubricant produced in this study. The highest yield of bio-lubricant was obtained at 240 minutes of hydroxylation reaction time, representing a yield of 92.67%. Conversely, the lowest yield was observed at 120 minutes of reaction time, with a yield of 91.17%. The yield of bio-lubricant in this study is higher than that of oleic acid-based

bio-lubricant (Mardhiyani, 2021), which at a reaction time of 180 minutes produces a yield of 91.80%. The yield of palm oil-based bio-lubricant is also still lower than the results of this study where theyield at a reaction time of 120 minutes was 51.985% (Prianto et al., 2022).

The observed increase in bio-lubricant yield is consistent with the observed increase in hydroxylation reaction time. This phenomenon occurs due to the fact that as the reaction time is extended, the reactant compounds that undergo reaction will have an increased opportunity to collide with one another, thereby resulting in an elevated production of polyol compounds (bio-lubricant) (Habibie Yursal et al., 2019).

2. Kinematic Viscosity Value of Bio-lubricant

Viscosity is a significant factor in the evaluation of bio-lubricant quality. Kinematic viscosity measurements were conducted at temperatures of 313,15°K (40°C) and 373,15°K (100°C). The objective of measuring viscosity is to ascertain whether an oil can be transported at a specific temperature. The viscosity of a substance is inversely proportional to its density; thus, the lighter the oil fraction, the smaller the viscosity. The data obtained from the kinematic viscosity tests on the bio-lubricants are presented in Figures 5 and 6.

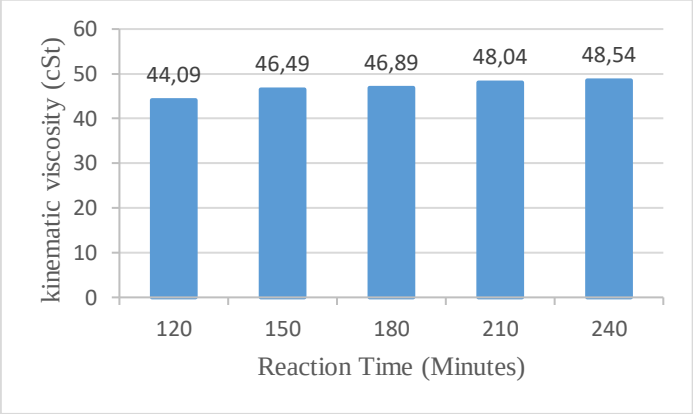

Figure 5: Graph of the effect of hydroxylation reaction time on the kinematic viscosity of bio-lubricant at 313,15°K (40°C).

Figure 5 illustrates the viscosity value of the bio-lubricant at 313,15°K (40°C). The highest viscosity value of bio-lubricant was obtained at 240 minutes of hydroxylation reaction time, with a value of 48.54 cSt. Conversely, the lowest viscosity value was observed at 120 minutes of reaction time, with a value of 44.09 cSt. The kinematic viscosity value of the bio-lubricant produced from palm kernel oil is higher than that produced from oleic acid. The kinematic viscosity value of the oleic acid-based bio-lubricant was 21.2048 cSt at a reaction time of 180 minutes (Mardhiyani, 2021). This value is lower than that of the palm kernel oil-based bio-lubricant from this study, which had a kinematic viscosity value of 46.89 cSt at a reaction time of 180 minutes.

Figure 5 also illustrates that an increase in reaction time is accompanied by an increase in kinematic viscosity. This phenomenon can be attributed to the rising number of bio-lubricant compounds formed (Fenny Lasma Hilde S, Irdoni, 2017). An increase in the quantity of bioprotein compounds results in an elevated viscosity value for the bioprotein (Rochmat et al., 2019). Thus, the observed increase in viscosity value as the reaction time progresses can be attributed to the conversion of greater quantities of epoxy into bio-lubricant. This is directly proportional to the reaction time and the conversion of the yield of the resulting bio-lubricant, whereby an increase in yield results in a corresponding increase in viscosity. In accordance with the specifications set forth in SNI Standard 06.7069.9.2012 (Badan Standardisasi Nasional, 2012), the ISO VG 46 viscosity standard encompasses a range of 41.4 to 50.6 Cst for biop lubricant viscosity at 313,15°K (40°C). Therefore, the findings of this study demonstrate that the bio-lubricant produced has attained the requisite viscosity characteristics of ISO VG 46 lubricants at 313,15°K (40°C).

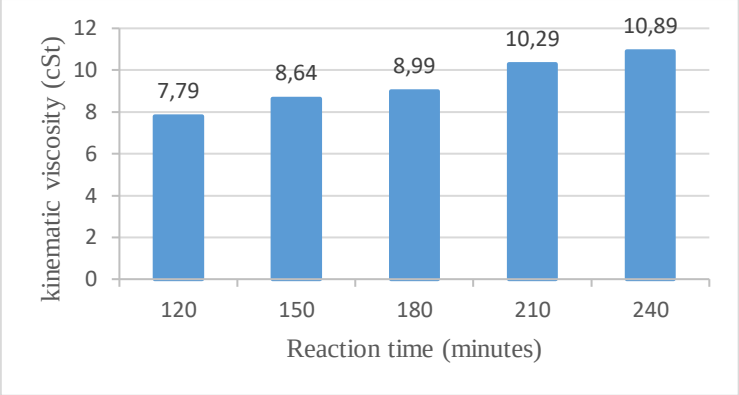

Figure 6 : Graph of the effect of hydroxylation reaction time on the kinematic viscosity of bio-lubricants at 373,15 °K (100°C).

Figure 6 illustrates the viscosity value of the bio-lubricant at 373,15°K (100°C). The highest viscosity value of the bio-lubricant was obtained at 240 minutes of hydroxylation reaction time (10.89 Cst), while the lowest viscosity value was observed at 120 minutes of reaction time (7.79 cSt). Figure 7 also demonstrates that the kinematic viscosity of the bio-lubricant at 373,15°K (100°C) increases with the prolongation of the reaction time. This is directly proportional to the kinematic viscosity at 40°C. In accordance with the requirements set forth in SNI Standard 06.7069.5:2012 (Badan Standardisasi Nasional, 2012), the viscosity range of SAE 20-30 at 100°C is 5.6-12.5 cSt. Consequently, the findings of this investigation demonstrate that the bio-lubricant in question has attained the requisite viscosity characteristics for classification as an SAE 20-30 lubricant at 373,15°K (100°C).

3. Viscosity Index Value of Biolubricants

Viscosity index (VI) is a numerical scale that indicates the extent to which lubricant viscosity is altered in response to changes in temperature. The viscosity index value is derived from the viscosity measurement of polyol compounds at 313,15°K (40°C) and 373,15°K (100°C). This viscosity index serves as a foundation for determining the viscosity characteristics of lubricants in response to temperature fluctuations. A lower change in viscosity with temperature corresponds to a higher viscosity index. The viscosity index test data for bio-lubricant is presented in Figure 7.

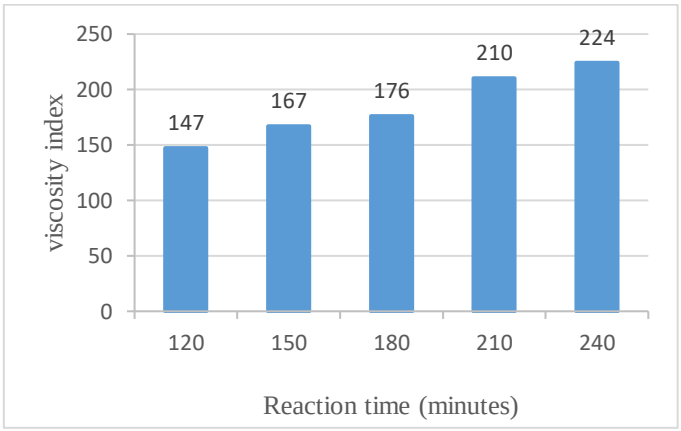

Figure 7: Graph of the effect of hydroxylation reaction time on the viscosity index of bio-lubricants

Figure 7 illustrates the viscosity index value of the bioprocessed lubricant. The highest viscosity index value of the bio-lubricant was obtained at a reaction time of 240 minutes, equating to a value of 224. Conversely, the lowest value was obtained at a reaction time of 120 minutes, resulting in a viscosity index value of 147. The viscosity index value of the bio-lubricant produced in this study is higher than that of the bio-lubricant produced from castor oil at a

reaction time of 2 hours (240 minutes), which has a viscosity index value of 146.189 (Debbie A, 2016).

The findings of the viscosity index testing of bio-lubricants in this study demonstrate the production of a notably high viscosity index. The results demonstrate that the palm kernel oil-based bio-lubricant exhibits characteristics of a lubricant with a high viscosity index (HVI), as indicated by a viscosity index value exceeding 90. The viscosity index value is a key parameter in the evaluation of lubricant products. A higher viscosity index value for a lubricating oil indicates a reduced likelihood of viscosity changes occurring at elevated temperatures during the utilisation of the bio-lubricant (Pratama, 2019). A lubricating oil with a high viscosity index value enables optimal lubrication work processes to be conducted over a wider range of temperature differences.

Due to their relatively stable physical resistance to temperature changes, bio-lubricants can function as a medium to reduce or eliminate wear between parts in an engine that rub against each other. In accordance with the requirements set forth in SNI 06.7069.9: 2012 (Badan Standardisasi Nasional, 2012), the minimum viscosity index standard for ISO VG ≥ 32 is 90. The findings of this study demonstrate that the bio-lubricant in question has fulfilled the requisite standard for viscosity index characteristics associated with lubricants.

5. Corrosion Rate of Bio-lubricant

The corrosion rate can be defined as the speed at which material quality deteriorates over time. The WGL (weight gain loss) method is a means of calculating corrosion rates, expressed in mpy (milli inch per year). In order to ascertain the corrosion rate, it is necessary to calculate the mass of the metal that has been cleaned of oxides. This mass must be expressed as the initial mass and then subjected to a corrosive environment for a specified period of time. The data obtained from the corrosion rate test on the bio-lubricant produced in this study can be found in Figure 8.

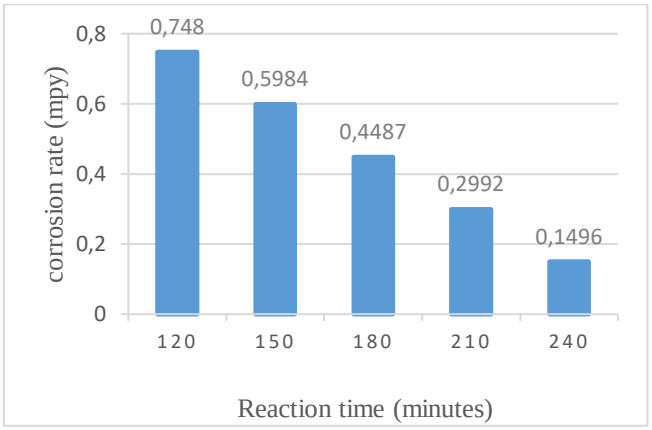

Figure 8. Graph of the effect of hydroxylation reaction time on corrosion rate.

Figure 8 illustrates the viscosity index value of the bio-lubricant research results. The highest corrosion rate value of the bio-lubricant is obtained at a reaction time of 120 minutes, with a value of 0.7303 mpy. In contrast, the lowest value is observed at a reaction time of 240 minutes, with a value of 0.1496 mpy. The findings indicate a direct proportionality between the corrosion rate and the reaction time of the resulting bioprocessed product. This is due to the fact that an extended reaction time results in the formation of a highly viscous bio-lubricant, which becomes more firmly attached to the lubricated object due to its increased viscosity. This leads to a reduction in corrosion rate. The resistance of a material to corrosion is typically quantified using a corrosion rate value between 1 and 200 mpy. The classification of material resistance based on corrosion rate can be observed in Table 3.

Table 3. Corrosion resistance level based on corrosion rate

Relative Corrosion resistance	Approximate Metric Equivalent				
	mpy	mm/year	µm/yr	nm/yr	pm/sec
Outstanding	< 1	< 0.02	< 25	< 2	< 1
Excellent	1 - 5	0.02 - 0.1	25 - 100	2 - 10	1 - 5
Good	5 - 20	0.1 - 0.5	100 - 500	10 - 50	5 - 20
Fair	20 - 50	0.5 - 1	500 - 1000	50 - 100	20 - 50
Poor	50 - 200	42125	1000 - 5000	150 - 500	50 -200
Unacceptable	200+	5+	5000+	500+	200+

Source : (Kurniawan Afandi et al., 2015)

Table 3 indicates that the corrosion rate obtained in this study is within the outstanding category, as the range is less than 1 (mpy). Therefore, the lubricant produced has demonstrated an excellent level of corrosion resistance.

CONCLUSION

The research conducted on the production of bio-lubricants from palm kernel oil has led to the conclusion that the manufacturing process involves three main stages: hydrolysis, epoxidation and hydroxylation. This study demonstrates that the reaction time has a significant impact on the quality of the resulting bio-lubricant. Specifically, an increase in reaction time leads to an improvement in the quality of the bio-lubricant. The optimal bio-lubricant outcomes were observed with a 240-minute hydroxylation reaction time, resulting in a bio-lubricant yield of 92.67%, a kinematic viscosity (40°C) of 48.54 Cst, a kinematic viscosity (100°C) of 10.89 Cst, a viscosity index of 224, and a corrosion rate of 0.1496 mpy. The quality of the bio-lubricants produced in this study using palm kernel oil as a raw material is in accordance with the quality parameters set out in SNI.06.7069:2012 for physical properties of lubricants.

It is recommended that future research employ solvents other than methanol, such as butanol or octanol, in order to facilitate a comparison of the effects of solvent usage on bio-lubricants. Furthermore, it is advised that a more comprehensive characterisation of the bioprocessed lubricants be conducted, utilising techniques such as FT-IR, GC-MS, flash point and pour point analysis. This will enhance the reliability of the product analysis results.

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