VOL. 18, NO. 13, JULY 2023 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

THE EFFECTS OF REACTION TIME ON KINEMATIC VISCOSITY AND CONVERSION IN THE MANUFACTURING OF BIO-LUBRICANT ALTERNATIVES FROM OLEIC ACID WITH PALM KERNEL SHELL CATALYST AS AN APPROACH TO RENEWABLE ENERGY

Mersi S. Sinaga, Rondang Tambun, Maulida and Fina D. P. Ardhiyani
Department of Chemical Engineering, Universitas Sumatera Utara, Medan, Indonesia
E-Mail: mersisinaga68@yahoo.com

ABSTRACT

The majority of lubricants commonly used in everyday life are based on petroleum which is a non-renewable resource, is toxic to the environment, and has poor biodegradability which can become a big problem for the ecosystem. Bio- based lubricants (biolubricant) can be an alternative to environmental and energy issues such as biodegradability, toxicity, health, emissions, and fuel economy. Biolubricant can be defined as a lubricant developed from basic ingredients in the form of animal fats, plant oils, or synthetic esters. Lubricants based on plant oils are also renewable. More than 98% of biolubricant decompose in the soil, unlike some synthetic lubricants and mineral lubricants which only decompose 20% to 40%. In addition, vegetable or animal lubricants used in engines reduce almost all forms of air pollution compared to the use of petroleum. This research is focused on making biolubricant through an epoxidation process from palm oleic acid raw material and ring opening with the heterogeneous catalyst of palm kernel shells (PKS). The purpose of this study was to determine the effects of the catalyst and the length of reaction time on the characteristics of the biolubricant produced and to determine the potential of palm kernel shell as a raw material for the manufacture of biolubricant. For the research variables, the catalyst concentrations were 2.4 and 6% and the reaction time variables were 60, 90, 120, 150, and 180 minutes. The analysis carried out is the analysis of density, viscosity, kinematic 2 viscosity, and Fourier Transform Infrared Spectroscopy (FT-IR). This study obtained the results of kinematic viscosity and conversion in the range of 17.8592-21.2048 cSt and 77.92-91.80%, respectively. The biolubricant produced in this study meets the ISO VG 22 standard which is equivalent to SAE 5W with a kinematic viscosity of 21.2048 cSt from a catalyst variation of 6% at a reaction time of 180 minutes. So it can be concluded that oleic acid from palm oil has potential as a biolubricant raw material.

Keywords: oleic acid, biolubricant, epoxidation, palm oil shell catalyst, ring-opening reaction.

Manuscript Received 17 October 2022; Revised 23 August 2023; Published 13 September 2023

INTRODUCTION

Oil palm (*Elaeis guineensis*) is an excellent source of vegetable oil raw materials for the production of vegetable lubricants. Lubricants made from palm oil are chosen because they contain a unique fat chain and do not contain much protein. However, palm oil contains many polyunsaturated fatty acids, therefore palm oil has the potential to be a raw material for making biolubricants [19]. In addition, Indonesia is known for being the world's largest palm oil producer since 2006, beating Malaysia. The abundant availability of palm oil raw materials in Indonesia is the basis for choosing palm oil as a raw material for making biolubricants [22].

Oleic Acid (C18) is the most common group of fatty acids used in the manufacture of biolubricants. Oleic acid is a chain of polyunsaturated fatty acids contained in palm oil. Biolubricants produced from oleic acid have good lubrication and stability characteristics [1].

The epoxidation reaction is the most suitable reaction to saturated vegetable oils and can cause an increase in oxidation resistance. The process of epoxidation reaction takes place gradually; first, the vegetable oil will undergo the opening of the double bond by being initiated by peroxide to form an oxidant (ether). At this stage, there is an increase in oxidation to increase the pour point in vegetable oils [27]. A commonly used method to synthesize epoxide is the reaction of alkenes with peroxide acid and the process is called epoxidation. Peroxide is an electrophilic source of oxygen and reacts with nucleophilic bonds from alkenes. Epoxide compounds contain an oxyrane group formed through an epoxidation reaction between peroxy acid (permeate) and olefinates or unsaturated compounds [31]. The majority of lubricants or lubricating oils commonly used in everyday life are petroleum-based which is a non-renewable resource, toxic to the environment, and has poor biodegradability which can be a big problem for ecosystems [8]. With the increasing sense of wanting to be safe and secure, the demands for the use of environmentally friendly and renewable materials are also getting more attention [22]. The increasing concern for environmental and energy issues has resulted in stricter lubricant specifications related to environmental and energy issues such as biodegradability, toxicity, health and safety, emissions, and fuel economy. Bio-based lubricants can play an important role as the base material for lubricants that are in their class because of the combination of their up-to-date properties and good

lubrication performance [5]. Research on renewable sources as raw materials for the production of environmentally friendly products such as biolubricants can be used for industrial and ecological purposes. In this regard, synthetic lubricants made from vegetable materials are an alternative to the use of petroleum [1].

Biolubricants are lubricants that are rapidly degradable (biodegradable) and non-toxic for humans and the environment. Biolubricants are developed from basic ingredients in the form of animal fats, growth oils, or synthetic esters. Lubricants based on plant oils are biodegradable and non-toxic, as well as renewable. Biolubriants decompose more than 98% in the soil, while synthetic and mineral lubricants decompose only 20%-40%. In addition, vegetable and animal lubricants reduce almost all forms of air pollution compared to using petroleum. Biolubricants can be made from various types of crops such as oil palm, soybeans, sunflowers, jatropha, and so on [22].

Therefore, this study was conducted to see the influence of catalysts and the length of reaction time on the manufacture of biolubricants so that they can produce the characteristics of the biolubricants produced by standards so that they can be used domestically or abroad, biodegradable, safe for living things and the environment, and also in the form of products that have economic value.

EPOXIDATION

Vegetable oils have been used as environmentally friendly lubricants due to their easily decomposing properties, good lubrication, higher viscosity index, and non-volatile properties. Fatty acids are an alternative source that can be used to make bio-lubricants [8].

Palm oil itself is a good source of vegetable oil raw materials for producing vegetable lubricants because it has a low chain of free fatty acids (FFA) and good lubrication potential properties. In addition, it is also edible and biodegradable, thus it can be used for bio-foodgrade lubricants (in the food and pharmaceutical industries) that are safe for human health and environmental sustainability [15].

Oleic Acid (C18) is the most common group of fatty acids used in the manufacture of biolubricants. Esters synthesized from these fatty acids have one or more ester bonds from polyols with alkyl chains. These resulting esters have a lower pouring point than natural oils, can maintain biodegradability, have good lubrication characteristics, and can also improve chemical and thermal stability [1].

An alternative method of making biolubricants utilizes fatty acids to be synthesized into methyl oleic epoxy. Epoxy of methyl oleic is the result of the reaction of epoxy of oleic acid with peroxide with an acid catalyst. The resulting epoxy is then reacted with alcohol with a ring opening reaction assisted by a solid acid catalyst. Then the reaction results show an increase in pour point, rheology, and lubrication [8].

The reaction of making biolubricants can be seen as follows:

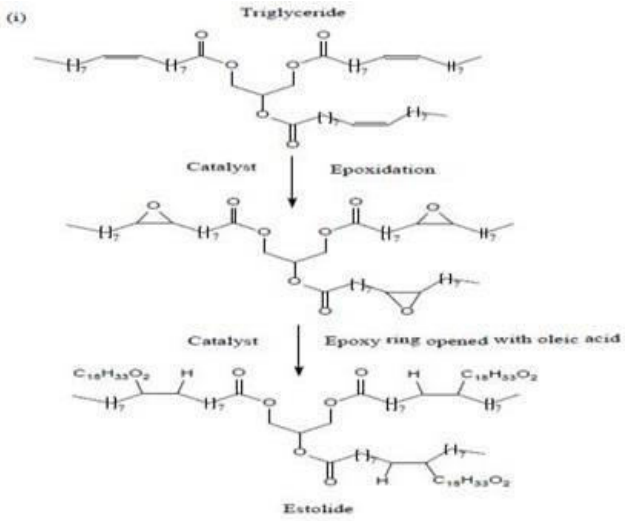

Figure-1. Biolubricant manufacturing reaction [29].

The solution to improve the oxidation stability of vegetable lubricants is modification. One of the modified vegetable lubricants synthesis processes is epoxidation, hydroxylation, and acetylation. Epoxidation reaction is the reaction of converting the unsaturated bond of a compound into a saturated bond in the form of an oxidizing group by oxidizing the compound [24]. The epoxidation reaction can be seen in the following figure:

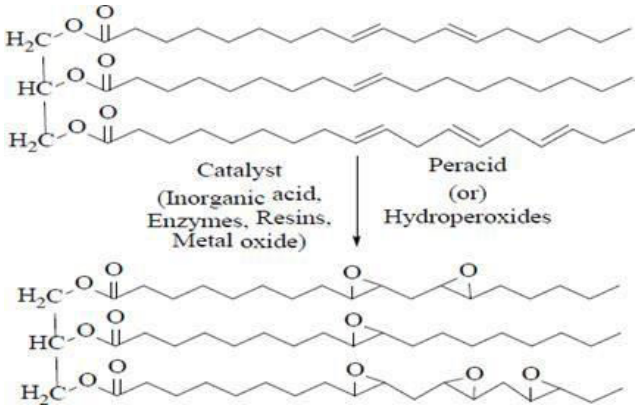

Figure-2. Epoksidation reaction [29].

The ring opening reaction or esterification is the reaction of opening the oxyrane group into a hydroxyl group carried out on epoxy compounds. Hydroxylation is a reaction that adds a hydroxy group to an organic compound. In this reaction, it can also be called the reaction of opening the oxidant ring in epoxide compounds. The hydroxylation process of epoxy oil is carried out by reacting epoxy compounds and methanol with the help of heterogeneous catalysts. The result of the hydroxylation process is organic compounds in the form of hydroxyl compounds also known as polyol compounds [24].

Catalyst design is important to achieve a good reaction and get a high yield. Some wastes such as dolomitic stone, gypsum, fishery waste, zeolite, and palm oil industrial waste have been used as catalysts. These

wastes are active in assisting reactions with yields above 90% [15]. One of the resolutions that emerged was the manufacture of renewable catalysts derived from biomass. Carbon-based heterogeneous acid catalysts are considered ideal catalysts for many reactions due to their chemical properties, and good mechanical and thermal stability. Theoretically, solid catalysts are derived from biomass containing a large number of lignin and carbohydrate species. The structure of the carbon backbone depends on the reaction temperature and time during the carbonization process. H_2SO_4 is commonly used as a sulfonation agent for carbon-based solid catalysts because it will provide a high acid density to the catalyst. The selection of palm kernel shells as raw materials for heterogeneous acid catalysts was due to abundant and easily available resources, besides that the high carbon content reaches 20-22% [6].

Lubricants are usually used to reduce wear between contact surfaces, improve the efficiency of the system, prevent corrosion, and also as a heat-resistant liquid. It is prepared from mineral oils and vegetable oils as raw materials. Most of the lubricants available on the market are Mineral oil derivative lubricants and synthetic esters. Synthetic esters are specifically formulated to meet government regulations and are harmless and biodegradable. According to [29].

The availability of petroleum from exploration in the country is projected to be exhausted in about 20 years. If only petroleum is relied on as a supply of lubricant raw materials, Indonesia will become an importer in meeting national lubricant needs. In addition to the availability of raw materials, the problem of using lubricants from petroleum is that it is not environmentally friendly because it is difficult to degrade naturally and the presence of toxic substances triggers natural damage when lubricants are thrown into the environment. Therefore, alternative solutions to this problem are needed, one of which is a lubricant based on vegetable materials. In terms of availability and environmentally friendly properties, vegetable lubricants are indeed suitable as an alternative solution to replace lubricants [24].

Biolubricants decompose more than 98% in the soi*l,* unlike some synthetic lubricants and mineral lubricants that decompose only 20% to 40%. In addition, vegetable oils used in machines reduce almost all forms of air pollution compared to the use of petroleum. Biolubricants can be produced from various types of plant oils and animal oils [7].

Lubricants based on vegetable oils are known to be biodegradable and non-toxic to life. In addition, biolubricants also show a higher viscosity index, good lubrication, and flash point and also lower volatility compared to petrochemical-based lubricants [8]. Being environmentally friendly has good characteristics and abundant availability of raw materials are the reasons bio lubricants are used as an alternative to petroleum lubricants. Manufacture of polyol compounds are intermediate compounds for the production of vegetable lubricants formed from the hydroxylation reaction of epoxy compounds [24].

The main function of the lubricant is to reduce friction and wear between two planes or surfaces that are in contact, as a heat carrier/coolant medium, prevent rust, and as a force continuer (hydraulic medium). The basic principle of lubrication itself is to prevent friction between two moving metal surfaces so that the movement of each metal can be smooth without much energy being wasted [13].

The characteristics of a good lubricant include several aspects such as low volatility, ideal cleanliness, high biodegradibility, good lubrication solvency, stable oxidative, low temperature, hydrolytic stability, and high viscosity tendency [16].

RESEARCH METHODOLOGY

Materials and Equipment

The main ingredients used in this study were palm oil oleic acid obtained from PT BRATACO in Medan, aquadest (H_2O), acetic acid (CH_3COOH), sulfuric acid (H_2SO_4), 30% hydrogen peroxide (H_2O_2), palm kernel shell, n-hexane (C_6H_{14}), n-ethanol (C_2H_6O). The tools used in this study were beaker glass, separator funnel, erlenmeyer, furnace, hot plate, triple neck flask, magnetic stirrer, oven, pipit drops, reflux condenser, stative and clamp, thermometer, picnometer, and Oswald viscometer.

PROCEDURES

Palm Kernel Shell Catalyst Preparation

The procedure for preparing the catalyst for empty bunches of oil palm can be seen as follows: The palm kernel shell is cleaned. Palm kernel shells are pureed using a crusher. Then sifted using a sieve of 60 mesh and 100 mesh. Palm kernel shell powder that passed the 60 mesh sieve but did not pass the 100 mesh sieve was used in this study. Palm kernel shell powder is calcined at a temperature of 500 ^oC in the furnace. The calcined palm kernel shell powder was then activated with H_2SO_4 10 N with a ratio of 1:9 and heated at a temperature of 60 ^oC for 6 hours. Then, the pre-activated palm kernel shell powder is washed with aquadest and dried. Palm kernel shells that have been chemically activated are then physically activated again using a furnace with a temperature of 500 ^oC for 3 hours.

Manufacture of Epoxy Compounds

This research began with measuring 100 grams of raw materials, then placed into a three-neck flask that had been assembled with a reflux condenser, hot plate, thermometer, and magnetic stitter. Then add solvents, namely 40 grams of hexane, then 15 grams of glacial astetaic acid, and 2.5 grams of H_2SO_4 catalysts. After the temperature reaches 60 ^oC, 65 grams of 30% hydrogen peroxide is slowly added and the temperature must be kept constant. After the addition of hydrogen peroxide is

complete, the reaction temperature is increased to 60 °C with a reaction time of 150 minutes. After the reaction stops, the mixture is washed with hot water at a temperature of 40-45 °C to separate from impurities, after the epoxidation is completed; the next stage is the oxyrant ring opening reaction.

Ring Opening Reaction

The opening of the oxidant ring in the epoxy of oleic acid is carried out using ethanol, for nucleophilic substitution in the epoxy group. With the ratio of solvent: epoxy is 9:1 M was inserted into the three-neck flask. Then this mixture is heated to a temperature of 65 °C and stirred with a stirrer with a stirring speed of 900 rpm. After reaching the desired temperature, the empty palm bunch acid catalyst is inserted into the three-neck flask with a variation of 2, 4, 6 (% w/w), and the reagent is carried out with a time variation of 60; 90; 120; 150; 180 (minutes) was adapted and performed according to Salih et al. (2017). After the reaction is completed, the catalyst is separated by filtration, and then rinsed using hot water to remove excess unreacted reagents.

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RESULTS AND DISCUSSIONS

Epoxy Characteristics of Oleic Acid as Raw Material for Making Biolubricants

Fourier Transform Infra-Red (FT-IR) analysis of oleic acid epoxy raw materials was performed to identify functional groups contained in the raw materials. The results of the analysis of functional groups on raw materials can be seen in Figure 3 contain a sulfonate group as an active site to assist esterification reactions in the manufacture of biolubricants from oleic acid. This is shown from the peak absorption spectrum at wavelengths 1707.1 and 1617.7 which are strong S=O groups, and also on the peak absorption spectrum at wavelengths 782.5 which are strong S-O groups [20]. Heterogeneous catalysts offer ease of separation, conversion efficiency, and also their use that can be done repeatedly.

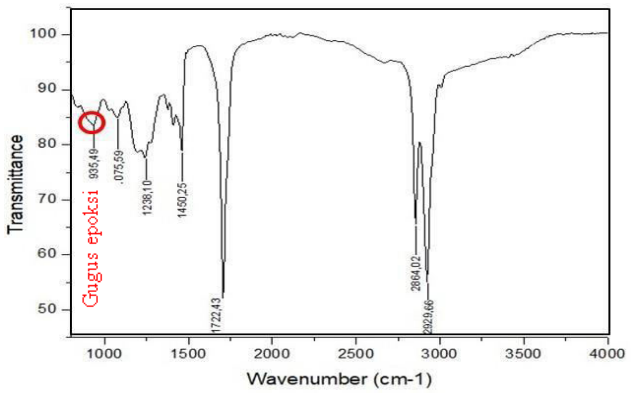

Figure-3. Results of FT-IR Analysis of Epoxy Compounds with stirring speed of 600 rpm, catalyst concentration of 2.5 %, and 60 ml H2O2.

In Figure-3 FT-IR analysis of epoxy compounds from oil palm oleic acid shows peaks of uptake that may indicate a group as a special characteristic of a compound. The peak of absorption at a wave number of 935.49 cm-1 in epoxy compounds indicates the presence of an oxygen group. The presence of this oxyrane group is caused by the oxidation reaction of the double bond of oil by active oxygen that forms epoxide compounds [26].

Characterization of Palm Kernel Shell Catalysts

The catalyst used in making biolubricants in this study was a heterogeneous catalyst for palm kernel shells (PKS) with a size of 60 mesh. The selection of palm kernel shells as raw materials for heterogeneous acid catalysts was due to abundant and easily available resources, in addition to that the high carbon content reaches 20-22% (Farabi, et al., 2019). The catalyst was synthesized using the H_2SO_4 acid activation method with calcined PKS. H_2SO_4 is commonly used as a sulfonation agent for carbon-based solid catalysts because it will provide a high acid density to the catalyst. Acid activation is carried out to improve the performance of catalysts in the reaction of biolubricant formation. To find out whether the catalyst preparation has been successfully carried out and produced the expected catalyst product, FT-IR was carried out to see the function groups that show the PKS catalyst has an active site. FT-IR testing was performed on a PKS catalyst with an H_2SO_4 concentration of 10 N, a reaction time of 6 hours, and a reaction temperature of 60 °C. The results of the Fourier Transform Infrared Spectroscopy (FT-IR) analysis for heterogeneous catalysts that have been activated with H2SO4 can be seen in Figure-4.

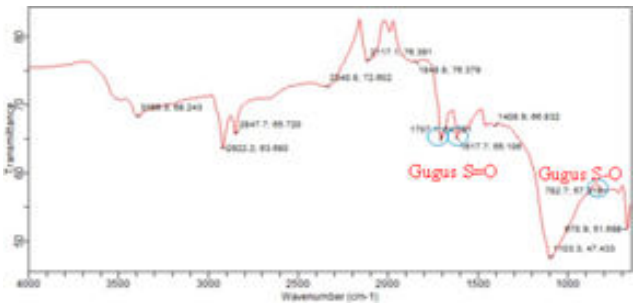

Figure-4.

From Figure-4, it can be seen that there is a vibration peak at the wavelength of 1250 cm-1 to 1750 cm-1 which indicates the presence of a SO_3H sulfonate group (Purwanto, *et al.,* 2013). Thus, FT-IR readings show evidence those heterogeneous catalysts of palm kernel shells

Biolubricant Product Characteristics

In this experiment, oleic acid epoxy oil was used as a raw material in the manufacture of biolubricants. The results of the Fourier Transform Infrared spectroscopy (FT-IR) analysis of the best biolubricant with a speed of

900 rpm, a temperature of 65 °C, and a catalyst of 6% at a time of 180 minutes can be seen in Figure-5 below:

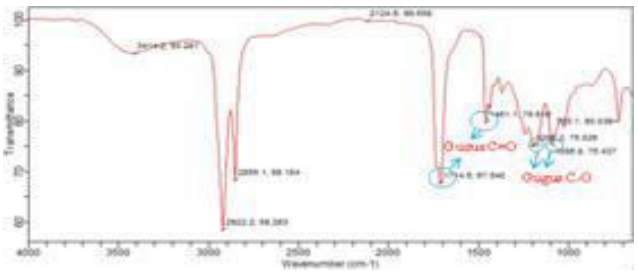

Figure-5. Results of FT- IR Biolubricant analysis with stirring speed of 900 rpm, catalyst concentration of 6% at 180 minutes.

Based on Figure-5 above, the result of Fourier Transform Infrared Spectroscopy (FT-IR) analysis of the best produced at a speed of 900 rpm, a temperature of 65oC, and a catalyst of 6% at 180 minutes can be seen. From the results above, we will see whether in this study biolubricants have been successfully created by looking at the groups shown from the absorption spectrum.

The reaction of the formation of biolubricants from oxidized oil palm oleic acid can be described by the following reaction:

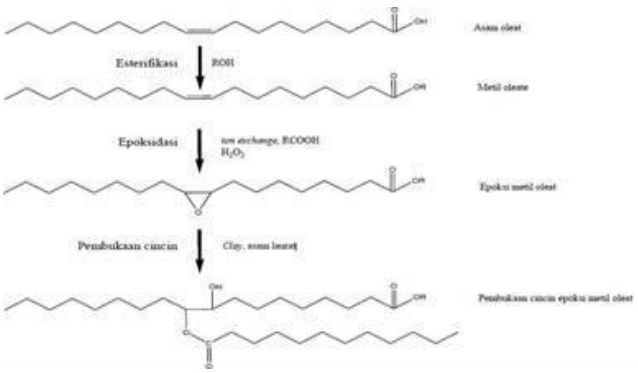

Figure-6. Biolubricant formation reaction [33].

At the peak of absorption from Figure-6, it can be seen that there is a vibration signal from the ester functional group, indicated by the absorption spectrum at the wave numbers 1714.6 cm-1 and 1461.1 cm-1 which indicates the C=O ester group supported by C-O at wave numbers 1200.2 cm-1 and 1095.8 cm-1. The results of the analysis above are the results of FT-IR analysis of the best biolubricants obtained in this study, with a speed of 900 pm, a temperature of 65 °C, and a catalyst of 6% at 180 minutes. Wavelength 1050-1300 cm-1 indicate the presence of the C-O bond which is the identity of the ester functional group, and wavelengths 1690-1760 cm-1 indicate the presence of the C=O bond which is also the identity of the ester functional group [25].

The results of the FT-IR analysis show that the raw material of oleic acid in the form of carboxylic acid has been converted into a biolubricant (in the form of esters). So it can be concluded that this shows that the process of esterification of oleic acid into biolubricants with heterogeneous catalysts of palm kernel shells has been successfully carried out.

Effect of Reaction Time on the Kinematic Viscosity of Biolubricants

Viscosity is one of the important parameters in the determination of the quality of a bioluricant. Kinematic viscosity measurements are carried out at a temperature of 40 °C. The viscosity value of the bioluricant will indicate how much resistance it has to flow. The greater the viscosity value of the bioluricant means that the thicker it is the greater the resistance to flow. Ideally, viscosity should produce a strong layer to separate two surfaces rubbing against each other. Based on the reference (ASTM D 445), kinematic viscosity is a measure of the resistance (viscosity) of a liquid at the force of gravity. The function of viscosity is to provide information for the determination of the operation of equipment.

The relationship of reaction time to kinematic viscosity values can be seen in the following graph:

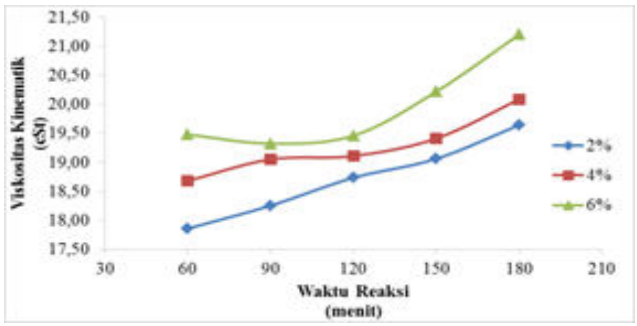

Figure-7. Graph of the effect of reaction time on the kinematic viscosity of biolubricants.

From Figure-7 it can be seen that the kinematic viscosity of biolubricants increases more and more as the reaction time increases. This is by the reference according to (Hilde, 2017), a relatively long reaction time affects the viscosity of the resulting biolubricant. The higher the viscosity of the biolubricant due to the increasing content of esters formed, the more esters formed causing the viscosity of the biolubricant to increase [21].

Increasing viscosity values over time the reaction indicates more and more epoxys are converting into biolubricants. From the results of the study, the highest kinematic viscosity value was obtained at a reaction time of 180 minutes with the use of a 6% catalyst, which was 21.2048 cSt. The lowest viscosity produced in this study was obtained at a reaction time of 60 minutes with the use of a 2% catalyst, which was 17.8592 cSt.

The increasing viscosity of biolubricants is caused by more and more epoxy compounds converting into ester compounds (biolubricants) as the concentration of catalysts used increases. Thus, the more ester compounds resulting from such reactions can increase the viscosity value and lower the corrosion value of the resulting biolubricant. So it can be concluded that the concentration of catalysts to the kinematic viscosity of

biolubricants is directly proportional. Thus it can be said that the resulting biolubricant can meet the characteristics of the ISO VG 22 viscosity standard.

Effects of Reaction Time on Biolubricant Conversion

The relationship between the reaction time to biolubricant conversion at reaction times of 60, 90, 120, 150, 180 minutes with the use of CKS catalysts of 2%, 4%, and 6% can be seen in Figure-8 below:

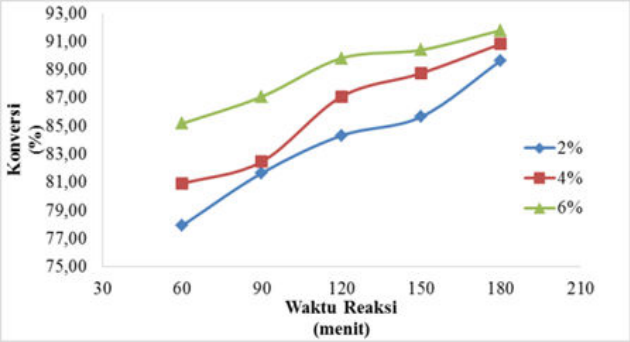

Figure-8. Graph of Biolubricant conversion reaction time effect.

From Figure-8 it can be seen that the conversion of biolubricants tends to increase as the reaction time increases. From the results of the study, the highest conversion was obtained at a reaction time of 180 minutes with the use of a catalyst of 6% which was 91.80%, while the lowest conversion was found at a reaction time of 60 minutes with the use of a catalyst of 2% which was 77.92%. This is to the reference (Yusral, 2019), where the longer the reaction time, the more product conversion will increase. The conversion gain of biolubricants is directly proportional to the reaction time; this is because the number of esters formed during the reaction increases as time goes by.

Comparison of Biolubricant Characteristics with Commercial Lubricants

The biolubricants produced from this research will have their characteristics identified to see if the biolubricants produced have characteristics that are by existing standards. The biolubricants to be compared are biolubricants produced from a reaction time of 180 minutes and 6% catalyst with commercial lubricants with ISO VG 22 standards.

The physical characteristics of commercial lubricants according to ISO VG 22 standards can be seen in the following table:

Table-1. Classification of lubricants (with ISO VG grade standards).

ISO VG Grade	22	32	46	68	100
Part Number	ALT	ATH	AFS	ALS	AM
Density @ 15ºC	0.86	0.87	0.87	0.88	0.88
Viscosity @ 40ºC (cSt)	20.9	33.8	47.4	67.7	94.8
Viscosity @ 100ºC (cSt)	4.19	5.62	6.91	9.05	10.8
Viscosity Index	102	100	100	100	98
Closed Flash Point (ºC)	200	210	218	210	230
Pour Point (ºC)	-30	-24	-18	-18	-18

CONCLUSIONS

This study concludes that the kinematic viscosity and conversion of the resulting biolubricants increase as the catalyst concentration and reaction time increase. The elevated conversion at a reaction time of 180 minutes with a catalyst concentration of 6% which is 91.80% and the highest kinematic viscosity is obtained from a reaction time of 180 minutes with a catalyst concentration of 6% which is 21.2048 cSt. In the results of the FT-IR analysis of the resulting biolubricant, there is a vibration signal from the ester function group, indicated by the absorption spectrum at wave numbers 1714.6 cm-1 and 1461.1 cm-1 which indicates the C=O ester group supported by C-O at wave numbers 1200.2 cm-1 and 1095.8 cm-1. The results of FT-IR analysis shows that oleic acid raw materials have been converted into biolubricants (in the form of esters).

ACKNOWLEDGEMENTS

The source of funding in this study comes from the University of North Sumatra Talenta 2022.

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