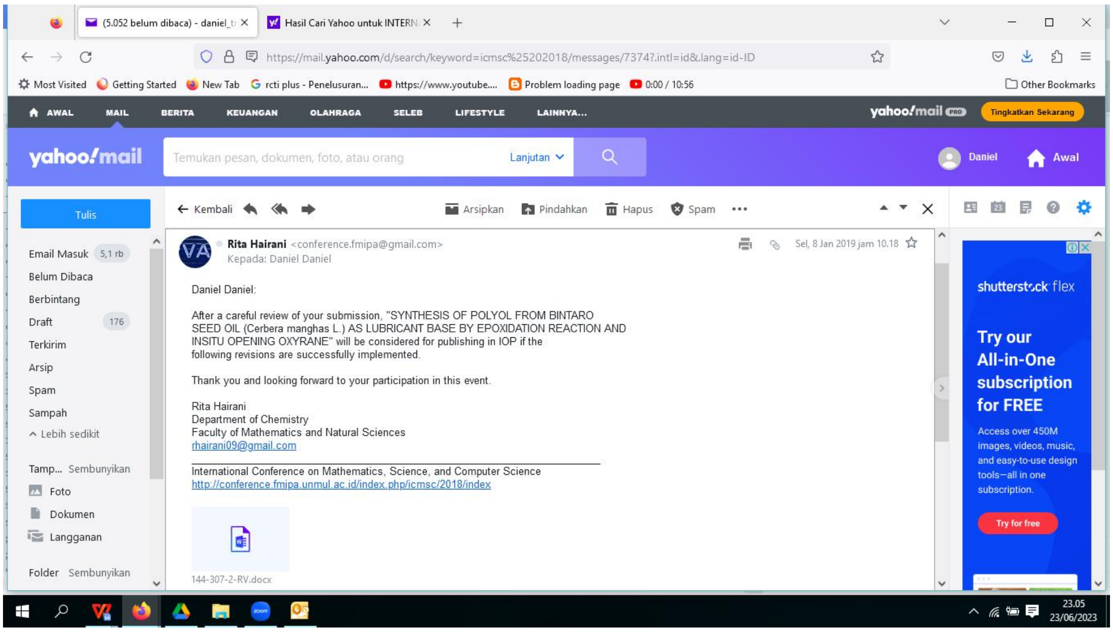

Rita Hairani <conference.fmipa@gmail.com>
Kepada: Daniel Daniel
Sel, 8 Jan 2019 jam 10.18

Daniel Daniel:

After a careful review of your submission, "SYNTHESIS OF POLYOL FROM BINTARO SEED OIL (Cerbera manghas L.) AS LUBRICANT BASE BY EPOXIDATION REACTION AND INSITU OPENING OXYRANE" will be considered for publishing in IOP if the following revisions are successfully implemented.

Thank you and looking forward to your participation in this event.

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# Synthesis of polyol from Bintaro seed oil (*Cerbera manghas* L.) as lubricant base by epoxidation reaction and insitu opening oxirane

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**Abstract**. Synthesis of polyol from Bintaro seed oil (*Cerbera manghas* L.) as base lubricant through epoxidation and opening oxirane ring directly has been carried out. The results showed that polyol can be synthesized from Bintaro seed oil and its FT-IR analysis exhibited the absorption peak typical on wave numbers of 3008.95 cm$^{-1}$ (C−H sp$^2$) and 1658.78 cm$^{-1}$ (C=C) with iodine value was 140.66 g Iod/100 g, but polyol has absorption peak typical on wave number of 3417.86 cm$^{-1}$ (-OH) and iodine value decreased to 2.11 g Iod/100 g. Besides that, another instrument analysis could be done by ascertain compounds of the product as polyol. The percentage of yield of polyol was 92.42%. The results of physical properties test showed that polyol was classified in SAE 250 on axle and manual transmission lubricant viscosity classification based on its characteristics such as kinematic viscosity at 100°C was 54.35 cSt and viscosity index was 111.36. But, its flash point and pour point were not fulfill the quality standard of SAE J306.

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## 1. Introduction
Lubricant is one of the most demanded commodities in Indonesia. It can be seen through the increasing use of motor vehicles, factory machineries and others. Undeniably, it impacts on the increasing use of lubricants in Indonesia [1].

Lubricant is a substance inserted between two rubbing surfaces to reduce the friction. The composition that is widely used consists of 70-90% base lubricants mixed with additives. The most broadly applied base lubricant is mineral lubricating oil derived from petroleum [1]. However, the depletion of petroleum supplies in the world leads to the need to make alternative base lubricants such as vegetable oils that is renewable. Vegetable oil with high oleic content is considered to be the potential ingredients to replace base lubricant from conventional mineral oils and synthetic esters [2]. In general, vegetable oil has many advantages such as high flash point, high viscosity index and high lubrication. Despite the fact, some limitations of vegetables oil arise. Then the industry has not been able to overcome it, such as low resistance to oxidative degradation at high temperatures and poor low temperature properties. The reason for instability of vegetable oil is that it is structurally unsaturated in the fatty acid portion [3].

Chemical modification of vegetable oils can overcome this deficiency, by reducing or eliminating unsaturated bonds in vegetable oils. The modification significantly improves the performance of lubricant. Epoxide can be used as a high temperature lubricant and the product, obtained through the ring opening, can be used as a low temperature lubricant [4]. Bintaro seed oil has the potential to be developed into base lubricant. Bintaro seed contains 46-64% oil with triglyceride composition consisting of oleic acid (36.64%), linoleic acid (23.44%) and linolenic acid (2.37%) [5].

Based on the aforementioned explanation, it is necessary to synthesize polyol from Bintaro seed oil as base lubricants through epoxidation and direct ring opening epoxide. Then it is also required to classify the polyol as base lubricants based on the produced characteristics. This research is expected to provide information on the development of base lubricant from vegetable oils and to increase the economic value of Bintaro seeds.

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## 2. Material and Methods

### *2.1. Instruments*
The instruments used in this research were oven, heat mantles, a series of soxhletation, spatula, stirring rods, analytical balance, filter funnel, rotary evaporator, centrifuge, a series of reflux devices, magnetic stirrer, dropper funnel, volume pipette, measuring pipette, suction ball, measuring cup, measuring flask, separating funnel, beaker glass, drop pipette, erlenmeyer flask, burette, Gas Chromatography instrument and Fourier-Transform Infrared (FT-IR) spectroscopy.

### *2.2. Materials*
The materials used in this research were Bintaro seeds (*Cerbera manghas L.*), n-hexane, glacial $CH_3COOH$, $H_2O_2$ 30%, $H_2SO_{4(p)}$, distilled water, diethyl ether, anhydrous $Na_2SO_4$, ethanol 95%, phenolphthalein indicator, KOH, HCl, chloroform, Hanus iod, KI, $Na_2S_2O_3$ and amylum indicator.

### *2.3. Procedures*

*2.3.1. Extraction of Bintaro seed oil.* Bintaro seed was separated from the shell, cut into small pieces, put in the oven and then granulated. The granulated Bintaro seed was extracted using a soxhletation device. Then it obtained a mixture of oil and n-hexane solvent, afterward it was concentrated by using a rotary evaporator. The oil that has been obtained was centrifuged to separate the sap. Then, it was weighed to find out the yield of oil contained in Bintaro seeds. Later, the oil was tested for physical properties such as flash point, pour point, viscosity at 40°C and 100°C, viscosity index and chemical properties such as iodine number test, saponification number test and acid number test [6]. Moreover, the oil was analyzed by GC and FT-IR spectroscopy.

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*2.3.2. Producing polyol from Bintaro seed oil.* A total of 120 mL of glacial acetic acid was put in a three-necks flask and added 60 mL of $H_2O_2$ 30% slowly while stirring. Then it was added $H_2SO_{4(p)}$ 1% (v/v) and refluxed at a temperature of 40°C - 50°C for 1 hour. Then it was added 200 mL of Bintaro seed oil slowly and refluxed at a temperature of 40°C - 50°C for 3 hours. Then the result of reaction was allowed to stand for 24 hours in a separating funnel. Then 100 mL diethyl ether was added. Diethyl ether layer was washed with saturated $NaHCO_3$ until the pH was neutral and followed by distilled water 3 times. Then the results were filtered and dried with anhydrous $Na_2SO_4$ and the filter paper. The filtration results were concentrated by using a rotary evaporator to obtain pure polyol compounds. The products obtained were tested for physical properties such as flash point, pour point, viscosity at 40°C and 100°C and viscosity index and chemical properties such as iodine number test, saponification number test and acid number test [6]. Later, the polyol was analyzed by FT-IR spectroscopy.

## 3. Results and discussion

*3.1. Extraction of Bintaro seed oil*

Bintaro seed oil is obtained by extracting it from dried Bintaro fruit seeds by using the soxhletation extraction method. The extraction process produces pure Bintaro seed oil with an oil content of 59.21%. It is in line with the results reported by Towaha et al (2011) that Bintaro seed contains 46-64% oil [5]. Then the obtained Bintaro seed oil was analyzed by gas chromatography to determine the level and type of fatty acids found in Bintaro seed oil. The results of Bintaro seed oil gas through chromatography analysis can be seen in table 1.

**Table 1**. The substance and the level of fatty acid found in Bintaro seed oil (*Cerbera manghas L.*).

| **Fatty Acid** | **Formulation** | **Level (%)** |
|---|---|---|
| Palmitic Acid | $C_{16}H_{32}O_2$ | 19.93 |
| Palmitoleic Acid | $C_{16}H_{30}O_2$ : 1 (ω-7) | 0.61 |
| Stearic Acid | $C_{18}H_{36}O_2$ | 3.63 |
| Oleic Acid | $C_{18}H_{34}O_2$ :1 (ω-9) | 36.91 |
| Linoleic Acid | $C_{18}H_{32}O_2$ : 2 (ω-6, ω-9) | 14.93 |
| Others | - | 23.99 |

Based on the result of the gas chromatography analysis, it showed that the largest composition of fatty acids of Bintaro seed oil is 52.45% unsaturated fatty acids. The result is smaller than the unsaturated fatty acid substance in Bintaro seed oil which has been reported by Towaha et al (2011) i.e. 62.45% and Endriana (2007) i.e. 69.1% [5, ]. It could be caused by the different location of the growth of Bintaro plants. Thus, the percentage of unsaturated fatty acid content is different even though it is the same type of plant. Based on the presence of these unsaturated fatty acids, it is assumed that in the production of polyol, the hydroxyl groups in the obtained polyol is formed by replacing the double bonds in the unsaturated fatty acids, namely oleic acid, linoleic acid and palmitoleic acid.

**Table 2.** The interpretation of infrared spectrum from Bintaro seed oil.

| **Wave number, (ν cm$^{-1}$)** | | **Ribbon form** | **Placement of related group** |
|---|---|---|---|
| **Spectra** | **Literature** | | |
| 3008.95 | 3100-3000 | Sharp | ν =C–H |
| 2924.09 | 3000-2850 | Sharp | ν C–H (at $CH_3$) |
| 2854.65 | 3000-2850 | Sharp | ν C–H (at $CH_2$) |
| 1743.65 | 1735-1750 | Sharp | ν C=O aliphatic |
| 1658.78 | 1680-1640 | Sharp | ν C=C non conjugation |
| 1458.18 | 1470-1450 | Sharp | γ C–H (at $CH_2$) |
| 1373.32 | 1375 | Sharp | γ C–H symmetry (at $CH_3$) |
| 1165.00 | 1000-1300 | Sharp | ν C–O–C |

In addition, Bintaro seed oil was analyzed by FT-IR spectroscopy. The result of FT-IR spectroscopic analysis of Bintaro seed oil can be seen in table 2. The FT-IR analysis aims to determine the functional groups contained in Bintaro seed oil. Then it is compared to the functional groups in polyols so that polyols can be formed or not. The result of FT-IR spectroscopy analysis of Bintaro seed oil polyol is a spectrum with absorption peaks in wave numbers of 2924.04 cm$^{-1}$ and 2854.65 cm-

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$^{1}$. It shows a typical absorption of C–H sp$^3$ stretching vibration that supported by vibration peaks in the wave numbers of 1458.18 cm$^{-1}$ and 1373.32 cm$^{-1}$. It indicates the typical absorption of C – H sp$^3$ bending vibration. Wave number of 1743.65 cm$^{-1}$ shows the typical absorption of carbonyl group (C=O). Later, wave number of 1165.00 cm$^{-1}$ shows typical absorption of C – O – C group which indicates the presence of ester. Wave number of 3055.24 cm$^{-1}$ shows the typical absorption of C–H sp$^2$ vibration stretching and wave number of 1658.78 cm$^{-1}$ shows the typical absorption of double bonds (C=C) which indicates the presence of unsaturated bonds in Bintaro seed oil.

### *3.2. Producing polyol from Bintaro seed oil*

The production of polyol is done through epoxidation and direct ring opening epoxide (hydroxylation) on Bintaro seed oil. In the production, epoxidation and ring opening of the epoxide (hydrolysis) of Bintaro seed oil are carried out without the separation the epoxide compound at first. Thus, the production of polyol occurs directly or one step only, as indicated by the one-stage reaction scheme in figure 1.

The obtained polyol was in the form of white liquid with a fairly large percentage of the yield i.e. 92.42%. It shows that polyols from Bintaro seed oil can be formed well through epoxidation and direct ring opening epoxide (one-stage). It is quite effective compared to polyol produced indirectly, because it is done only one stage so the percentage of errors in the production is smaller.

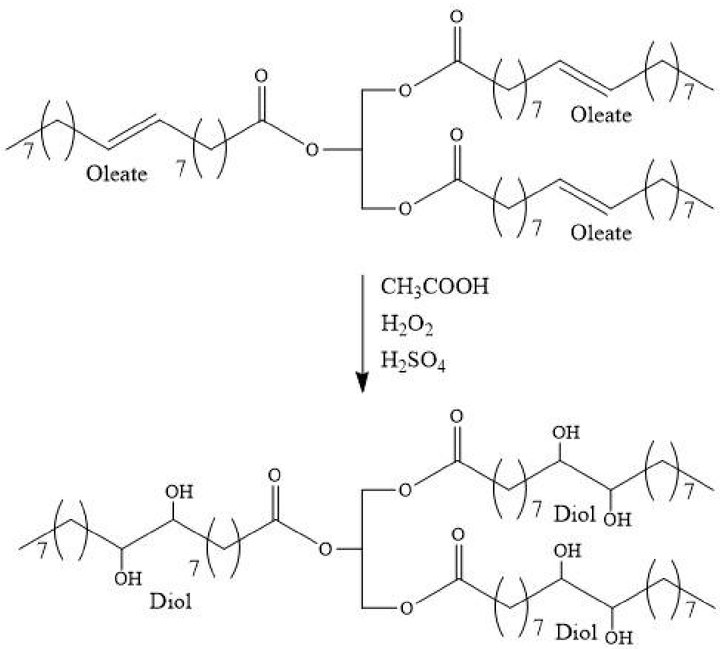


**Figure 1.** Scheme of one-stage polyol reaction reaction.

After that, the polyol was analyzed by FT-IR spectroscopy to find out the functional groups contained in the polyol so that it could be compared with the functional groups contained in Bintaro seed oil. Figure of spectrum comparison of FT-IR spectroscopic analysis of Bintaro seed oil and polyol can be seen in figure 2.

The results of FT-IR spectroscopic analysis of Bintaro seed oil polyol provide a spectrum with absorption peaks at wave numbers of 2924.04 cm$^{-1}$ and 2854.65 cm$^{-1}$. It indicates a typical absorption of C-H sp3 stretching vibration. Then it is supported by vibration peak at wave numbers of 1458.18 cm$^{-1}$ and 1373.32 cm$^{-1}$ that shows the typical absorption of C-H sp3 bending vibration. Wave number of 1743.65 cm$^{-1}$ shows the typical absorption of carbonyl group (C=O). Wave number of 1165.00 cm$^{-1}$ shows the typical absorption of C-O-C group which indicates the presence of ester.

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**Table 2**. The interpretation of infrared spectrum from Bintaro seed oil.

**Wave number, (v cm$^{-1}$)**

**Ribbon form**

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Figure 2 shows the differences of functional groups found in Bintaro seed oil and polyol. In Bintaro seed oil, the absorption peak is found at wave number of 3008.95 $cm^{-1}$ (C-H sp2) and at wave number of 1658.78 $cm^{-1}$ (C=C). Then, in polyol of Bintaro seed oil gives a spectrum with absorption peak at wave number of 3417.86 $cm^{-1}$ which is a typical absorption of hydroxyl group (–OH) and at wave number of 1743.65 $cm^{-1}$ which is a typical absorption of carbonyl group (C=O). It shows that an oxidation has occurred in each π bond of alkene group, not on the carbonyl group on Bintaro seed oil to form diol.

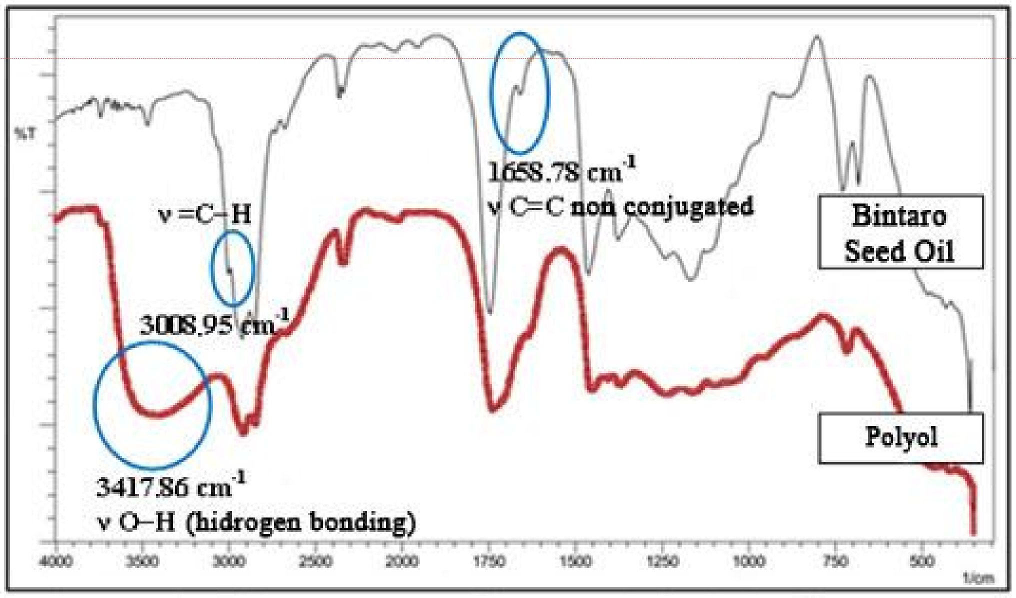


**Figure 2.** The comparison of FT-IR spectrum of Bintaro seed oil and polyol

*3.3. Physical test of Bintaro seed oil and polyol*

*3.3.1. Kinematic viscosity test.* Viscosity is the most important property of lubricant. If the viscosity of a lubricant is higher than needed, the engine load becomes heavier. Otherwise, if the viscosity of a lubricant is too low, the lubricant would not able to meet the ability to withstand the required load by the equipment [7]. The results of the kinematic viscosity test of Bintaro seed oil and polyol, at temperature 40ºC, are 32.70 cSt and 818.83 cSt, respectively. Furthermore, the kinematic viscosity test of it, at temperature of 100ºC, are 6.65 cSt and 54.35 cSt, respectively. If it is compared with the viscosity classification for industrial lubricants along with ISO, the viscosity of Bintaro seed oil at 40ºC is 32.70 cSt at ISO VG 32. Moreover, the viscosity of polyol at 40ºC is 818.83 cSt set between ISO VG 680 and ISO VG 1000. If it is compared to the viscosity classification for engine lubricant according to SAE J300, the viscosity of Bintaro seed oil is at the level of SAE 15W or 20W. While the polyol viscosity is not in that classification. In addition, if it is compared to the viscosity classification for the lubricating gear/manual transmission and axle in line with SAE J306, the viscosity of Bintaro seed oil is at SAE 75W, then the polyol viscosity is at SAE 250. Based on the comparison of the existing lubricant standard, the polyol of Bintaro seed oil is most appropriate with the SAE 250 on viscosity classification for the lubricating gear/manual transmission and axle in line with SAE J306. However, it does not rule out the possibility for the use at the classification of other lubricant standards by mixing existing standard lubricants or additives to improve the lubricating properties of Bintaro seed oil polyols.

*3.3.2. Index of viscosity.* The viscosity index is the measuring of kinematic viscosity at temperature of 40ºC and 100ºC. Thus, the higher viscosity index, the viscosity is more stable or the possibility of

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viscosity change tends to smaller. It has been previously known that polyol of Bintaro seed oil fits to SAE 250 on viscosity classification for lubricating gears/transmissions manual and axle in line with SAE J306. The quality of minimum lubricant performance for lubricating gears/manual transmissions and axle along with SAE J306 is API GL-4. It has the official results of laboratory test. Based on the performance quality specifications of API GL-4, then the polyol viscosity index has met the minimum quality standard i.e. 90.

*3.3.3. Flash point test.* The flash point test is used to find out the minute when the lubricant is burned or when a flame is on the engine. It will harm the engine and result in waste of lubricant. The flash point can also indicate the possibility of highly volatile and flammable material in a material that is relatively non-volatile or not burnt [8]. The flash point test result of Bintaro seed oil is 29.44$^{o}$C and the flash point of Bintaro seed polyol oil is 35$^{o}$C. Based on the API GL-4 of the performance quality, the flash point of polyol included in SAE 250 (monograde), on the viscosity classification for the lubricating gear/manual transmission and axle, does not meet the quality standard which has a minimum flash point temperature at 200$^{o}$C.

*3.3.4. Pour point test.* The pour point of lubricant is the indicator of the lubricant to freeze at a certain temperature. If the lubricant quickly freezes, it will cause the engine hardly to be turned on. It is because the lubricant cannot be pumped and lubrication does not occur. In addition, it also indicates the type of used base oil. Therefore, the characteristic of pour point needs to be limited to its maximum value [9]. The result of the pour point test of Bintaro seed oil is -3.89$^{o}$C and the pour point of Bintaro seed oil polyol is 23.89$^{o}$C. Based on the API GL-4 of the performance quality, the polyol pour point included in the viscosity classification for lubricating gear/manual transmission and axle does not meet the quality standard which has a maximum pour point temperature of -20$^{o}$C. The increase of the pour point of Bintaro seed oil to be polyol is caused by the reduction of the unsaturation of Bintaro seed oil to be polyol [10].

*3.4. Chemical properties test of Bintaro seed oil and polyol*

*3.4.1. Iod-number test.* The Iodine number shows the unsaturation of fatty acids which make up oil and fat. The unsaturated fatty acids are able to bind iodine and form saturated compounds. The amount of tied iodine shows the number of double bonds that oil has. The iodine number is the number of grams of iodine which is bound by 100 grams of oil or fat [11]. The obtained iodine number of Bintaro seed oil and polyols respectively are 140.66 g Iod/100 g and 2.11 g Iod/100 g. Based on the comparison of the iodine number of the Bintaro seed oil and polyol compounds, it has decreased. It indicates that there has been an oxidation on the double bonds in unsaturated fatty acids into saturated compounds. It is supposed to occur due to the epoxidation and the opening of the oxirane ring, where at first the double bonds form the cyclic bonds oxirane oxygen in the epoxidation. Then it forms the -OH group in the oxirane ring opening reaction.

*3.4.2. Saponification number test.* In this test, the sample was refluxed in an alcoholic KOH solution until it was saponified, then it was titrated by using a standardized HCl solution. The use of alcoholic KOH solution aimed to saponify the oil gram, then the remaining alkaline solution is determined by the titration by using acid (HCl solution). Then the amount of alkali that reacts is identified by doing blank test. The blank test shows the first used KOH in the saponification. The test is accomplished by triplicate. It results that the saponification numbers of Bintaro seed oil and polyol respectively were 266.23 mg KOH/g and 270.60 mg KOH/g. The saponification number of Bintaro seed indicates that Bintaro seed oil cannot be used as a base lubricant since it does not fit to the range of values as reported by Sudradjat (2007) that the properties of vegetable oils that can be used as a base lubricant is 186-198 mg KOH/g [1]. It is because the saponification number indicates the amount of oil that can be

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saponified, the high molecular weight of oil will have a lower saponification number than the lower molecular weight of oil.

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**Table 3.** Physical and chemical properties test of Bintaro seed oil and polyol.

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| Parameter | Bintaro Seed Oil | Polyol |
|---|---|---|
| Kinematic Viscosity 40 °C (cSt) | 32.70 | 818.83 |
| Kinematic Viscosity 100 °C (cSt) | 6.65 | 54.35 |
| Index of Viscosity | 148.40 | 111.36 |
| Flash Point (°C) | 29.44 | 35.00 |
| Pour Point (°C) | -3.89 | 23.89 |
| Iod Number (gr I/100 gr sample) | 140.66 | 2.11 |
| Saponification Number (mg KOH/g) | 266.23 | 270.60 |
| Acid Number (mg KOH/g) | 2.58 | 37.01 |
| Free fatty acids (%) | 1.30 | 18.61 |

*3.4.3. Test of acid number and free fatty acid level.* Acid number is the number of milligrams of KOH needed to neutralize free fatty acids in one gram of oil or fat. The higher acid number impacts on the lower quality of it [11]. The obtained acid numbers of Bintaro seed oil and polyol respectively are 2.58 mg KOH g and 37.01 mg KOH/g. The obtained free fatty acid levels respectively are 1.30% and 18.61. %. Based on the comparison of acid number values of Bintaro seed oil and polyol compounds which have increased, it is assumed due to the presence of acidic residues from the reaction. In addition, the increase of acid numbers occurs due to the process of hydrolysis or oxidation of the oil, especially in the double bond [12].

## 4. Conclusions

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Polyol can be synthesized from Bintaro seed oil (*Cerbera Manghas L.*) through the epoxidation and direct ring opening epoxide. It is proved by the results of FT-IR analysis and iodine number tests that Bintaro seed oil has a typical absorption peak at wave numbers of 3008.95 $cm^{-1}$ (C – H $sp^2$) and 1658.78 $cm^{-1}$ (C=C) and the iodine number is 140.66 g Iod/100 g. Meanwhile, the polyol has a typical absorption peak at wave number of 3417.86 $cm^{-1}$ (-OH) and the iodine number decreases into 2.11 g Iod/100 g. The obtained result is in the form of white liquid with the percentage of yield is 92.42%.

The result of physical properties test shows that polyol is classified into SAE 250 on the classification of lubricating viscosity of manual transmission and axle. It is in line with the result of kinematic viscosity test at temperature of 100°C that is 54.35 cSt and the viscosity index is 111.36. However, the flash point and the pour point do not meet the SAE J306 quality standard.


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