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16 February 2017

NOTIFICATION OF ACCEPTANCE

On behalf of the committee, we are pleased to announce that the following paper:

Title : UTILIZATION OF CRUDE PALM OIL TO PRODUCE BIOLUBRICANT THROUGH EPOXYDATION-HYDROXYLATION-ACETYLATION PROCESSES
Author : Muhammad Faizal
Paper ID : EST37

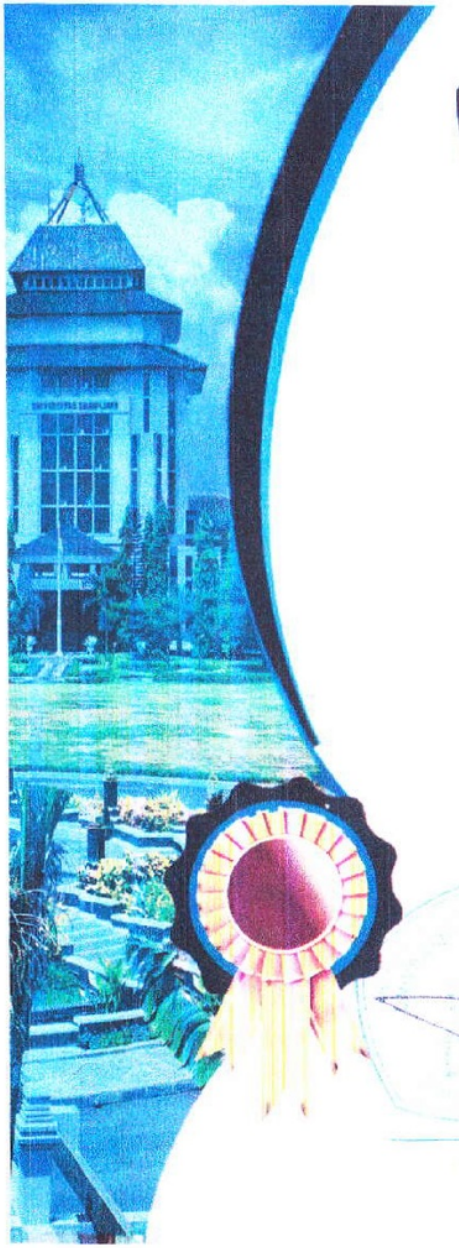
Has been accepted for **Oral Presentation** in the 7th Annual Basic Science International Conference 2017.

Please note that all presenters are required to pay the registration fee for the conference before the program is finalized.

Thank you and congratulation.

Regards,

Hari Arief Dharmawan, Ph.D.
Chairman of BaSIC 2017



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CERTIFICATE

This is to certify that

Dr. Ir. Muhammad Faizal, DEA

has been an active participant as

Presenter

with his/her paper entitled :

Utilization of Crude Palm Oil to Produce Biolubricant Through Epoxydation-Hydroxylation-Acetylation Processes

in

The 7th Annual Basic Science International Conference, held on 7 – 8 March 2017, in Malang, Indonesia.

Adi Susilo, PhD
Dean Faculty of Science,
Brawijaya University

Hari Arief Dharmawan, PhD
Conference Chairman

Utilization of Crude Palm Oil To Produce Biolubricant Through Process of Epoxydation, Hydroxylation And Acetylation

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Abstract – The aims of this research is to determine the optimum operating conditions and to study the reaction kinetics of bio-lubricant production. Biolubricant has been produced using palm oil as a raw material through epoxydation, hydroxilation and acetylation processs. The highest conversion was a reaction at a temperature of 80 °C and at 25 minutes with the conversion value of 64.14%. In this researeh, also the reaction energy activation of reaction is 4,834.4 J/mole. The reaction enthalpy at a temperature of 70 °C and 80 °C are of 1,981.5 J/mole and 1,898.3 J/mole respectively.

1. INTRODUCTION

Production of lubricants made from vegetable oil (bio-lubricant) is as part of efforts to reduce the use of lubricant made from petroleum. This bio-lubricant can be used motor vehicles and heavy equipment. The raw materials of bio-lubricants can be derived from vegetable oils such as palm oil. Vegetable oil is available in large quantities and can be renewed, and sustainable. Bio-lubricants has good characteristics: stable to heat and evaporation, low emissions. The term bio-lubricants applies to all lubricants, which are both rapidly biodegradable and non-toxic to humans and other living organisms, especially in aquatic environments [1]. Waste cooking oil (WCO), which otherwise finds no immediate potential utilization can be successfully used to synthesize bio-lubricant [2]. Plant seed oils are renewable alternatives to petrochemicals, but they cannot be used in their raw form except they are suitably modified [3]. The production of biolubricants from palm oil and Jatropha oil through two stages of transesterification has been studied [4]. Utilization of vegetable oil to be converted to polyurethanes has been carried out [5]. The exploiting fatty acids in the preparation of biobased polyols and polyurethanes has been carried out [6]. The aims of this research are to determine the optimum operating conditions and to study the reaction kinetics of bio-lubricant production.

2. METHOD

2.1 Chemicals

The chemicals used in this work were refined palm oil, 30% H_2O_2, methanol, anhydride acetic acid, glacial acetic acid, sulfuric acid 2%, bentonite, sodium hydrocarbonate, violet crystal indicator, HBr 0.1 N, indicators phenolpthaleine, ethanol, 0.1 N NaOH, and distilled water.

2.2 Procedures

A volume of 200 ml of palm oil and 40 ml of acetic acid (purity 99%) were put in a reactor. This mixture then was heated at 70 °C. The hydrogen peroxide (H_2O_2) and sulfuric acid of were added drop by drop to palm oil. The reaction was carried out for 120 minutes. After completed reaction, the mixture was then cooled. Epoxy compound then was purified by addition of water of 200 ml and then saturated by addition of $NaHCO_3$ solution in water of 100-200 ml. A 150 ml of epoxy and 100 ml of methanol was reacted to produce polyol at 40 °C for 120 minutes. Benthonit was used as a catalyst. Finally, 60 ml of polyol compound was converted to bio-lubricant by the addition of 6 ml of anhydrate acetic acid of 97% at a temperature of 70 °C and 80 °C. Bentonite was used as a catalyst. The polyoester sampling was analyzed for each 5, 10, 15, 20, 25 minutes. Figure 1 represents the scheme of experimental procedure of biolubricant production.

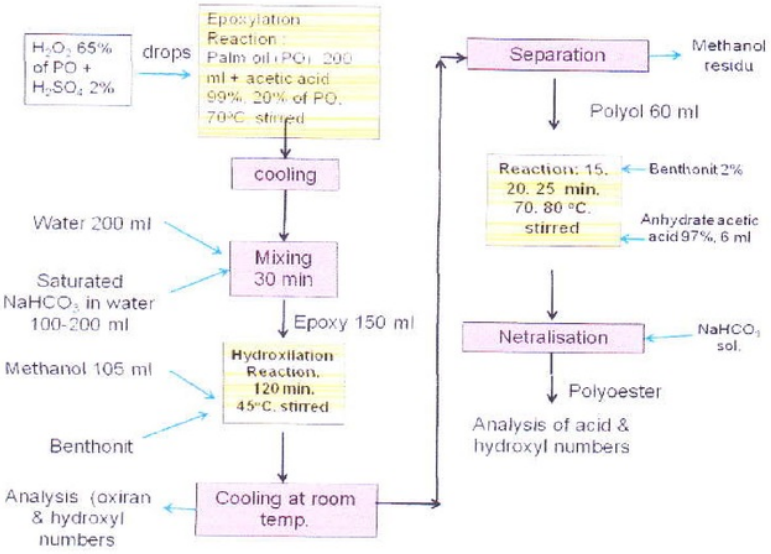


Figure 1 Scheme of Experimental Procedure of Biolubricant

3. RESULT AND DISCUSSION

Table 1 represents the physical and chemical characteristics of the standard of refined palm oil, and palm oil as raw material respectively. Based on Table 1, it can be seen that the quality of palm oil used in this study meet the standard value so that palm oil can be used as a raw material for making polyolester through epoxydation and hydroxylation process

Table 1 The result of palm oil quality used in this research

No	Parameter	Unit	Standard of Refined palm oil	Result of this research
1	Density	kg/m^3	923	922
2	Viscosity	centipoise	49.4	49.5
3	Oxiran Number	% O	0.04	0.041
4	Acid Number	mg NaOH/gr of oil	0.704-0.728	0.720

Table 2 represents the physical and chemical characteristics of epoxy and polyol compounds. The physical and chemical properties of intermediate products (epoxy and polyol) were changed. After hydroxilation reaction, there were the changes of physical and chemical characteristics between epoxy and polyol. Based on Table 2, the density decreased from 939.1 kg/m^3 (epoxy) to 924.0 kg/m^3 (polyol), But both densities of epoxy and polyol are greater than the density of the raw material (922 kg/m^3). For the value of kinematic viscosity, at temperatures of 40 ° C, the kinematic viscosity of epoxy compound (56.1 eSt) is higher than the kinematic viscosity of polyol (40.5 eSt). This different kinematic viscosity of both compounds is caused by differences in the structure of molecular between the epoxy compound and polyol compound. At a higher temperature (100 °C), there was a decreasing of kinematic viscosity for each type of epoxy compound and polyol compound. This phenomena is due to the increasing of temperature that the distance between the molecules was longer. In addition, the movement of each molecule will more rapid due to an increasing in the kinetic energy of each molecule. The viscosity index value between the epoxy compound and polyol produced quite different. The viscosity index is a number that indicates changes in viscosity due to temperature change. As we know, this viscosity index can describe the stability of a chemical substance or physics to changes in temperature. If the viscosity index greater the viscosity, the stability of the compound is better. Stable properties of these compounds indicate that the bonds between the compounds present in these substances are also more stable. Stable properties of a compound are very important in a lubrication system of an engine. Therefore, with stable properties, the lubrication properties of the machine can work well, either at low temperatures or at high temperatures. The viscosity index of the epoxy compound (162.1) obtained was greater than the viscosity index of polyol (143.5). This different viscosity index can be said that in terms of stability against temperature, the epoxy compound is better than polyol compound. However, for the lubricating oil to be produced later is a polyoester compound. The changes

in the chemical properties of the epoxy compound into a polyol compound comes from the change of oxyrane numbers. The difference is very clear in physical properties between epoxy and polyol in the term of oxyrane number. The oxyrane numbers of epoxy compound of 4.3 %O are greater than the oxyrane number of polyol of 36% O. This condition due to the epoxy compound had oxyrane-oxygen groups, while for polyol, the oxyrane group (COC) change into $COCH_2COH$ group.

Table 2 Physical and Chemical Characteristics of Epoxy and Polyol Compounds

No	Physical and Chemical Characteristics	Unit	Value	
			Epoxy	Polyol
1	Density	kg/m^3	939.1	924.0
2	Kinematic Viscosity (at 40 °C)	cSt	56.1	40.5
	(at 100 °C)	cSt	14.0	7.7
3	Viscosity Index	-	162,1	143.5
4	Oxyran Number	% O	4.3	0.36
5	Acid Number	mg NaOH/mg sample	0.91	1.22
6	Hydroxil Number	mg NaOH/mg Polyol	169.1	351.0

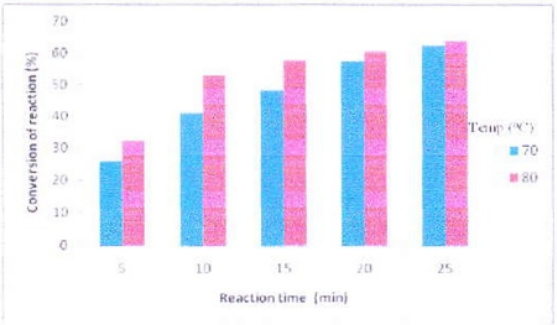
Table 3 shows density, kinematic viscosity, viscosity index and an acid number of produced polyester. According to the Table 3, the minimum density of polyoeser compound was of 931.5 kg/m^3 at temperature 70 ° C and at a reaction time of 10 minutes and a maximum value of 937.0 kg/m^3 at a temperature of 80 °C and at reaction time for 20 minutes. The minimum kinematic viscosity of polyolester at 40 ° C measurement was of 53,97 cSt at temperature reaction of 70 °C, and for a reaction time of 10 minutes. The maximum kinematic viscosity of polyolester at 40 ° C measurement was of 58.50 cSt at temperature reaction of 80 ° C and at a reaction time of 25 minutes. At a temperature of 100 °C, a minimum kinematic viscosity of polyolester was the value of 12.69 cSt at a temperature of reaction of 70 °C and for a reaction time of 20 minutes and a maximum kinematic viscosity of 15.88 cSt at a temperature of a fraction of 80 ° C, and at a reaction time of 25 minutes. The kinematic viscosities arehigher than the standard value of SAE 5W30. The minimum viscosity index of polyolester was of 160.0 at a temperature of 70 °C and for a reaction time of 20 minutes and a maximum viscosity index of polyolester of 164.7 was found at a temperature of 80 °C and for a reaction time of 5 minutes.

Table 3 Density, Kinematic Viscosity, Viscosity Index and Acid Number of polyester

No	Acetylation Variable		Density (kg/m^3)	Kinematic Viscosity (cSt)		Viscosity Index	Acid Number (mg NaOH/mg sample)
				40°C	100°C		
	Temp °C	Time (minute)		± 62.3	± 10.7	± 163	
1		5	931.9	54.29	13.62	162.7	0.54
2		10	931.5	53.97	14.04	164.6	0.36
3	70	15	933.4	54.54	14.12	163.6	0.41
4		20	933.6	54.79	12.69	160.0	0.64
5		25	933.5	54.34	14.50	164.6	0.67
6		5	931.7	54.36	14.80	164.7	0.58
7		10	932.4	54.31	15.07	164.6	0.52
8	80	15	933.6	54.15	14.60	164.5	0.77
9		20	937.0	55.40	15.49	164.6	0.68
10		25	936.9	58.50	15.88	163.0	0.33

The value lubricating viscosity SAE 5W-30 is of ± 163. If the viscosity index value of a lubricant does not meet the standards, then the polyolester compound can be added an additives to increase viscosity stability. The minimal of acid number (0.33 mg NaOH/mg) was found at 80 °C and reaction time of 25 minutes. The maximum acid number (0.77 mg NaOH/mg) was obtained at 80 °C and reaction time of 15 minutes. If acid number of biolubricant is small, so the quality of biolubricant is better. High acid number of lubricant used can cause corrosion in the machine. To minimize the acid number close to zero, then the polyolester compound can be added an additives such as pH-neutralizing compound.

Figure 2 represents the reaction conversion of polyol to polyester at temperature 70 °C and 80 °C for 5, 10, 15, 20 and 25 minutes of reaction time.



According to the Figure 2, increasing of reaction temperature, the conversion of polyol to polyoester (bio-lubricant) was increased. Also, increasing of reaction time, the conversion of polyol to polyoester was increased. The highest conversion was a reaction at a temperature of 80 ° C and at 25 minutes with the conversion value of 64.14%. In this research, also the reaction energy activation of reaction is 4,834.4 J/mole. The reaction enthalpy at a temperature of 70 °C and 80 °C are of 1,981.5 J/mole and 1,898.3 J/mole respectively.

4. CONCLUSIONS

The conclusion of this work are:

a. The conversion value of polyol to polyester through acetylation process was increased if reaction temperature and reaction time increased.

b. The highest conversion of polyol to polyester (64.14%) was obtained at 80 °C and reaction time of 25 minutes.

c. The reaction activation energy E was found of 4,834.4 J/mol, enthalpy of acetylation reaction at 70 °C and at 80 °C was obtained of 1,981.5 J/mol and of 1,898.3 J/mol respectively

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