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Effect of The Ratio of H-Zeolite Catalyst and Temperature in The Opening Ring Reaction in Bio Lube Base Oil Production from Palm Oil

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Abstract

Lubricants are materials that can reduce friction between two components. Lubricants are very important to keep the engine from being damaged quickly. Currently lubricants on the market generally comes from petroleum derivatives with limited availability. Therefore, technology needs to be developed to look for other raw materials as a substitute for petroleum based lubricating oil, namely by utilizing the potential of existing vegetable oils, one of which is palm oil. Biolubricant made from palm oil will be made using the method of transesterification, epoxidation and ring opening reaction. To increase yield and high product quality, a catalyst in the form of H-Zeolite is used. So that this study aims to determine the effect of the ratio of H-Zeolite catalyst to ring opening reactions. The quality of lube base oil could be indicated from density, viscosity index and acid number. Acid number will smaller in presence higher content in 10% weight ratio of H-Zeolite as catalyst and EPOME also higher temperature of ring-opening reaction because the catalyst H-Zeolit will work more effective in temperature 75°C. Other evidence is of density and viscosity index would show good value than smaller amount of H-Zeolit and lower temperature of reaction. Advices for the future research are the purification needs to be done not only physically but also chemically to maximize the results obtained. Preferably on the reaction of opening the epoxides rings used a long chain alcohol compounds that have a high viscosity and boiling point to obtain a better viscosities value.

Keywords: bio lubricant, palm oil, H-zeolite, ring opening

INTRODUCTION

According to Fisheries and Forestry (2003) Modern society life is supplied by energy and the use of common fossil-based products. The increase in the use of motor vehicles and the development of industrial machinery causes humans to more depend on fossil fuels including for producing lubricants. Lubricants or oils are liquids that determine the workability of the machine and motor vehicles. Lubricants are substances capable of reducing friction between two components. Erhan *et al*., (2002) states that generally, lubricants are made by combining base oils with additives to enhance the inherent characteristics of oil or to provide new performance properties to the mixture. Mobarak *et al*. (2014) Explains the current biolubricant can be made in 2 ways, namely the lubricant of hydrocarbon chemical modification and the second is the ester of vegetable oil.

Since the industrial Revolution, lubricants are used in various industrial sectors to lubricate machinery and materials. The Kline & Company Inc. Report (2006) indicates nearly 38 million metric tonnes of lubricants used around the world in 2005, with projections going up by 1.2% in subsequent decades. It is estimated that consumption for the industry reaches 32%, air and sea transportation 9.4% and lubricant for the 11.3% process. Coupled with the Statistics Indonesia (2015) that reported that the total needs of Indonesian lubricants reached 226,249 million liters in 2003. However, around 85% of lubricants used in the world are still based on fossil fuels. Birova *et al*. (2002) conveys that the negative effects of use of non-biodegradable lubricants can cause surface and

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ground water contamination, air pollution, soil contamination, and consequently, contamination of agricultural products and food. In this regard, environmental issues have increased rapidly worldwide over the last 25 years so it is necessary to do business to replace non-biodegradable lubricants into bio lubricants.

Vegetable oil is a lipid component derived from plants. Research conducted by the Agency for the assessment and application of technology (BPPT) that Indonesia has 60 types of crops that could potentially become the base material of lubricants, such as sunflower, rapeseed, soy, palm oil, and distance. In addition to the properties of its decay can equal mineral oil, vegetable oil is also easily unraveled biodegradable. Vegetable oil contains a ratio of polyunsaturated fatty acids with saturated fatty acids and high thermal stability and a low ratio of unsaturated fatty acids to polyunsaturated fatty acids (Chandu *et al.*, 2013). This is an important characteristic of lubricant. In particular, palm oil has become an attractive renewable energy resource to be processed into environmentally friendly lubricants due to its easily degraded nature, outstanding lubricating properties, high viscosity index, volatility and high flash points (Legarand and Dürr, 1998).

In previous studies, Kuweir (2010) performed a ring opening reaction using an H-zeolite catalyst that would be substituted by monoalcoholic compounds (ethanol, butanol, octanol and Heksadanol) and glycerol. However, the resulting yield is still quite low, it can be seen from not all the glycerol group is repressed into the oxirane as desired and then continued with Åkerman (2011) which uses the solid-Amberlyst-15 catalyst but Because the price is expensive then the usage is still limited so that in the research this time will be attempted using an H-Zeolit catalyst that was received by acid later used in the opening reaction of epoxide rings. Expected reaction with this catalyst will produce the product with the desired character.

The purposes of this research are to study the manufacture of lubricating oil with palm oil as raw materials using transesterification reaction, epoxydation and ring opening, to study the influence of H-zeolite catalyst weight ratio with palm oil on viscosity, density and acid number of ring-opening reactions and assess the effect of ring opening reaction temperature on viscosity, density and acid number of the opening reaction of the ring.

METHODOLOGY

The materials used in this study are palm oil, methanol, NaOH, formic acid, H_2O_2, natural zeolite catalyst (to be activated into H-zeolite) and 10 liters Aquadest. The tools used in this study are erlenmeyer, glass beaker, pH indicator, measuring cup, drop pipette, burette, klem and statif, syringe, picnometer, volume pipette, boiling three neck, thermometer, viscosimeter, and condenser.

The research was done in Diponegoro University laboratory. Fixed variables in this research are NaOH Catalyst 1% w compared to palm oil dissolved in methanol (mol methanol: CPO = 6:1), purification of the product with Aquades (Volume POME: Aquadest 1:1) and Formic acid Catalyst (mol POME: Formic acid 1:0.3), purification of the product with Aquades (Volume EPOME: Aquadest 1:1). Meanwhile, the modified variables are H-Zeolite catalyst ratio (1% w EPOME, 5% w EPOME, 10% w EPOME) and rin opening rection's temperature (55^oC, 65^oC, and 75^oC).

The research begins with transesterification, reacting palm oil with methanol with the help of NaOH catalysts. This transesterification aims to produce POME (Palm Oil Methyl Esther). The procedure to be done is palm oil is heated to a temperature of 65^oC with constant stirring. NaOH catalysts are first dissolved in methanol. Comparison of methanol mol and moles of palm oil used is 6:1. The NaOH catalyst used is as much as 1% of the oil palm Mass. Methanol and NaOH solutions are slowly coupled with stirring. Attach the condenser to prevent methanol evaporation, the reaction is carried at 65^oC and kept constant for 1 hour. After the reaction carried out the resulting product is allowed for ± 3 hours, then the product will be separated into 2 phases namely POME and glycerin. POME and glycerin are separated by a separating funnel, then washed with aquades with a comparison of 1:1 volume and stirring evenly. After stirring is performed, the silence and POME will be separated again. Aquades are separated from POME with a separating funnel. Washing is done three times to remove the glycerin that is still mixed in POME. Heat POME in the oven until the POME is free of water (golden yellow).

Pome results in the transesterification is epoxidized with hydrogen peroxide (H_2O_2) with the help of the formic acid catalyst to produce EPOME (Epoxidised Palm Oil Methyl Esther). The procedure is to heat the POME until it reaches a temperature of 50^{0}C with stirring. Insert the formic acid catalyst into the pome which has been heated with a comparison of mole POME and the mole of the acid formic 1:0.3. Add 5 ml of H_2O_2 with continued stirring. Keep the reaction temperature at 65^{o}C. Add 5 ml of H_2O_2 with continued stirring. Keep the reaction temperature at 65 ^{o}C. Add the next 5 ml H_2O_2, keeping the temperature up to 65^{o}C back. Let stand to separate into two phases, EPOME and water with H_2O and formiat acid. Separate using a split funnel. EPOME is washed with aquades (volume ratio is 1:1) and stir evenly. After stirring is done, let stand and EPOME will be separated again. Aquades is separated from the EPOME with a separating funnel. Washing is done three times. Heat the EPOME with an oven until the EPOME is free of water.

The zeolite that will be used in the ring opening reaction comes from Gunung Kidul regency. Zeolite is purchased in powder size. Before use, zeolite is activated first chemically and in physics. Chemical activation is done with an acid solution in this case HCl 4M while heated for 70 minutes, this chemical activation aims to clean the surface of the pore and dispose of the impuritiy compounds such as CaO and MgO. While the activation of physics is done by heating and Calcination aims to vaporize the water caught in the pores of the zeolite crystals by using a furnace with a temperature of 500^{o}C for 2 hours then cooled in the desicator.

After epoxidation rection, the next is ring opening reaction. The purpose of the ring opening reaction is to create the the Epoxides group formed in the epoxydation process becomes unreactive. This reaction procedure takes place with 2 variations of the temperature variation and the ratio of the catalyst/raw material. Heat up the mixture of ethanol and EPOME up to 50^{o}C with stirring, heat again the mixture up to variables changes of temperature (55^{o}C, 65^{o}C and 75^{o}C) without stirring. Add variable changes of H-zeolite ((1% W, 5% W and 10% W EPOME) for each variable change of temperature. The product is formed, then test product characteristics consisting of density, viscosity, and acid number.

RESULTS AND DISCUSSION

The increase in the quality of basic lubricating oils can be done by 3 stages of the process of transesterification carried out to obtain Palm Oil Methyl Ester compounds by reacting triglycerides and methanol with the help of NaOH catalysts, unstable double bonds, enhanced by performing epoxydation by peroxide acid and resulting Epoxidised Palm Oil Methyl Ester, after stabilized, ring opening reactions performed to re-enhanced unreactive product with ethanol and H-zeolite catalysts and base on research resulting data (Table 1)

From Table 1 we sould know that increasing value of density and viscosity index followed by decreasing of acid number in every variable. Quality of lube base oil could be indicated from density, viscosity index and acid number. Acid number will smaller in presence higher content in 10% weight ratio of H-Zeolite as catalyst and EPOME also higher temperature of ring-opening reaction in value 1.847 NaOH/100g Oil because the catalyst H-Zeolit will work more effective in temperature 75^{o}C. Other evidence is increment of density and viscosity index would show better product than smaller amount of H-Zeolit and lower temperature of reaction show the best value in density 0.936 gr/ml and VI 278.7.

The Effect Ratio of H-zeolite Catalyst and Epoxidized Palm Oil Methyl Ester to Viscosity, Index Viscosity, Density and Acid Number of Ring-opening Reactions

The existing EFAME of the glycerol group will experience increased density compared to the previous one. The larger the increase in density illustrates the increasing number of glycerol clusters. It is described in the following reactions.

The catalyst will open the ring on the EFAME so that there is an excess proton (CH +), then the glycerol group will attack the area. The more the glycerol groups that stick and the molecular weight will be increased, therefore the density will increase (Kuweir, 2010). The product density of the ring opening reaction can be seen in the following bar chart.

From the image above shows that the greater the number of catalysts added then the density of the resulting product is greater. This is because more and more catalysts on variables, the greater the decline in activation energy and the more numerous the epoxides rings that the efame opened by the catalyst along with the increase in the number of catalysts added so that it will The greater the number of glycerol that can be radiated (Sanjaya, 2008).

Acid number is a weak and strong organic acid size in the lubricant. The smaller the number of acids from a lubricant, the better the quality of the lubricant. The high acid number will lead to the formation of a viscous layer consisting of resin/varnish, increasing the viscosity of the lubricant to reduce the flow/pump efficiency, the risk of corrosion of the machine especially when there are water pollutants, etc. (Susilowati, 2016).

Table 1. Result Epoxidised Palm Oil Methyl Ester

Variables		Product Analysys				
		Density (g/ml)	Kinematic Viscosity		Viscosity Index	Acid Number
T	%W H-Zeolit		at 40°C (cSt)	at 100°C (cSt)		
CPO		0.9024				0.701
POME		0.866				0.606
EPOME		0.88				6.216
55°C	EPOME + 1%w H-zeolit	0.92	23.551	4.696	118.432	5.780
	EPOME + 5%w H-zeolit	0.924	23.777	5.052	145.402	5.533
	EPOME + 10%w H-zeolit	0.928	23.444	5.105	153.701	2.723
65°C	EPOME + 1%w H-zeolit	0.927	23.7	5.573	187.498	4.598
	EPOME + 5%w H-zeolit	0.928	23.745	5.727	198.356	4.264
	EPOME + 10%w H-zeolit	0.93	24.019	6.06	218.103	2.185
75°C	EPOME + 1%w H-zeolit	0.932	24.36	6.5	242.963	4.342
	EPOME + 5%w H-zeolit	0.935	24.623	6.901	266.382	3.473
	EPOME + 10%w H-zeolit	0.936	24.787	7.141	278.676	1.847

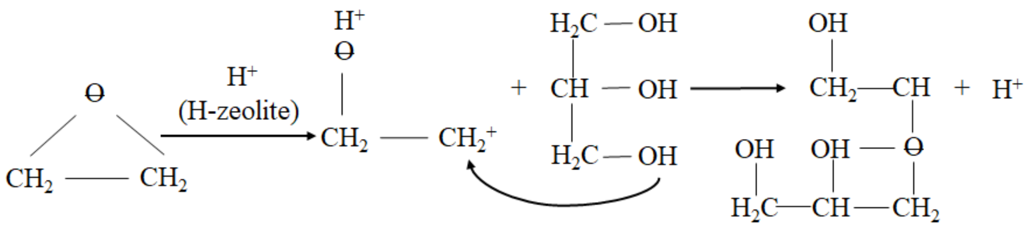

Figure 1. Increase of glycol cluster mechanism

A good lubricant should have a very small acid figure. If we compare with mineral lubricant HVI 160 and Yubase which has successive acid number 0.98 and 0.58, then all products of this research have a much higher acid number. The high number of acids can be caused by the presence of the catalyst's residual acid impurities that are not completely lost at the time of washing. To handle this, it can be done by the neutrality of the residual acid using Na_2CO_3 on the washing process (Arizona, 2010). Na_2CO_3 will react with acids and the following neutrality reaction occurs.

$$HCOOH + Na_2CO_3 \leftrightarrow HCOONa + CO_2 + H_2O$$

The viscosity of a lubricant is a measure of the large resistance given by the lubricant to flow or in other words is a viscosity measure of the lubricant. The greater the viscosity (the more viscous) means the greater the durability to flow. The viscosity index is a measure of the viscosity change to temperature (Godfrey *et al*, 1995). The viscosity of the lubricant will decrease if the temperature rises, otherwise the viscosity will rise if the temperature drops. This change will not be the same for all lubricants. A good lubricant is expected to have a sufficiently low viscosity at low temperatures, so that it can flow easily when the machine starts up. Conversely, at high temperatures, the viscosity should be high enough to keep it flowing and coat the surface of the machine well.

The viscosity value of the product is influenced by its molecular structure. The more the Hydroxylol group is the radiated then the viscosity value will be increased, this is due to the increasing group of glycerol containing the polar Group – OH the radiation, the tensile style of intermolecular attraction will be greater which results The greater the obstacles he had to flow (Utami, 2011). The following is the viscosity of the products produced at various ratios of H-zeolite catalysts.

Figure 4 Indicates that the value of viscosity tends to increase with increasing number of added H-zeolite catalysts. The larger the number of H-zeolite catalysts added, the more glycerol groups containing polar guus – OH the radiation. So, the tensile style of pulling between molecules will be greater which will result in the greater resistance that is possessed by the resulting product to flow (Kuweir, 2010). This leads to a higher catalyst ratio, so the product viscosity increases.

The Effect of Ring-opening Reaction's Temperature on Viscosity, Index Viscosity, Density and Acid Number

Ring-opening reaction do after EPOME which have oxirane ring structure. From the graph we could see that in temperature reaction at 55°C have highest value of density and followed by 75°C and the lowest value density is 65°C, this condition occure in all weight catalyst ratio variables

Zengshe *et al*. (2010) doing experiment in ring-opening polymerization reaction was conducted at different temperature ranging from 0 to 50°C. The glass transition temperature (Tg) of insoluble Epoxidised Vegetable Oil Ester polymers after extraction was measured by DSC. It is well known that the crosslinking density influences Tg and as the crosslinking density decrease, the free volume of a material will increase, thereby, decreasing Tg correspondingly. Ring-opening polymerization of 3-membered rings is thermodynamically favored (ΔG, free-energy change is negative) (Chanda, 2000). Considering the thermodynamic relation

$$\Delta G = \Delta H - T\, \Delta S$$

where ΔH is the enthalpy change, ΔS is entropy change, T is temperature (K). ΔH is the major factor for determining ΔG for ring opening in 3-membered rings, while ΔS is very important for 5- and 6-membered rings. ΔH was reported negative (-113.0 kJ/mol) for the 3-membered ring-opening (Allcock, 1970). Reaction temperature 55 °C have lowest value of density because of its temperature glass, so the volume increase and decreasing density. But, by raising temperature, the required conditions were optimized, so it creates increment of density in temperature 75°C (Karadeniz, 2015).

Acid number is important to know because lube base oil will use as lubricant of engine or the other lubricant. The occurrence is vaue of acid number is decreasing with higher temperature on ring-opening reaction.

The total acid number (TAN) of the starting material Epoxidised Oil Methyl Ester, and reaction product were determined to check if there was any formation of free fatty acids during the reaction. Palm oil has a TAN value of 1.12 g of NaOH/100g, EPOME has 0.12 g of NaOH/100g, and all four reaction products have more than 0.5 g of NaOH/100g. This further confirms that there is hydrolysis of a triacylglycerol backbone to form free fatty acids in these reaction conditions, and also decreasing of acid number means with increasing the temperature, catalyst H-Zeolit work more optimal (Josia, 2016).

The kinematic viscosity is the most important physical property of lubricating base oil (Sharma *et al.*, 2006). It is an index for analyzing of internal resistance in the motion of the lubricating base oil. The kinematic viscosity of lubricating base oil possible to change with temperatures; it increases while the temperature decreases. The effect of molecular weight and molecular structure of alcohols on the kinematic viscosity at 40°C and 100°C of alkyl esters are expressed. The viscosity

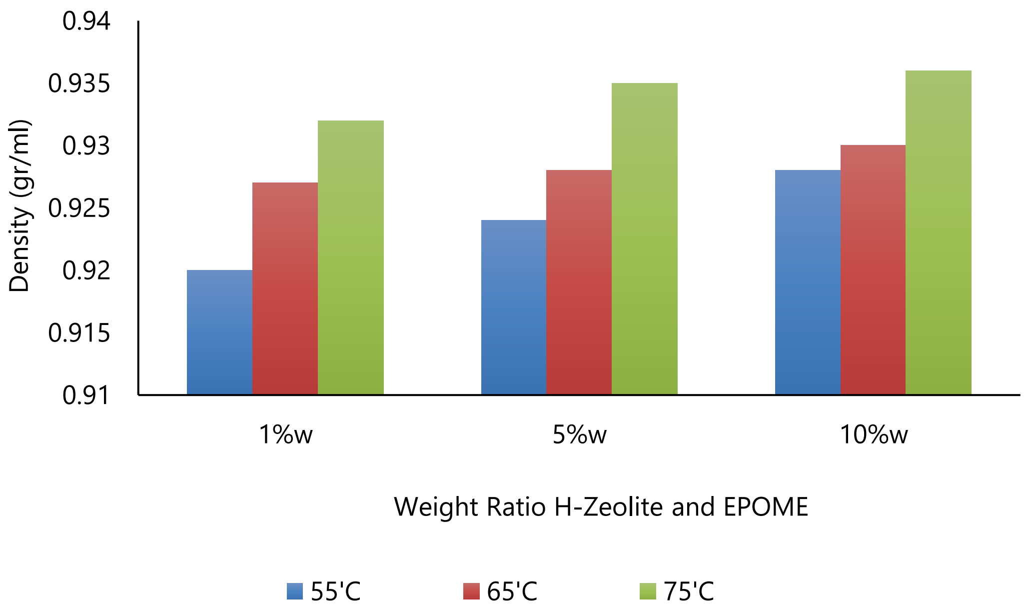

Figure 2. Effect ratio of H-zeolite catalyst and EPOME to density of bio-lube base oil

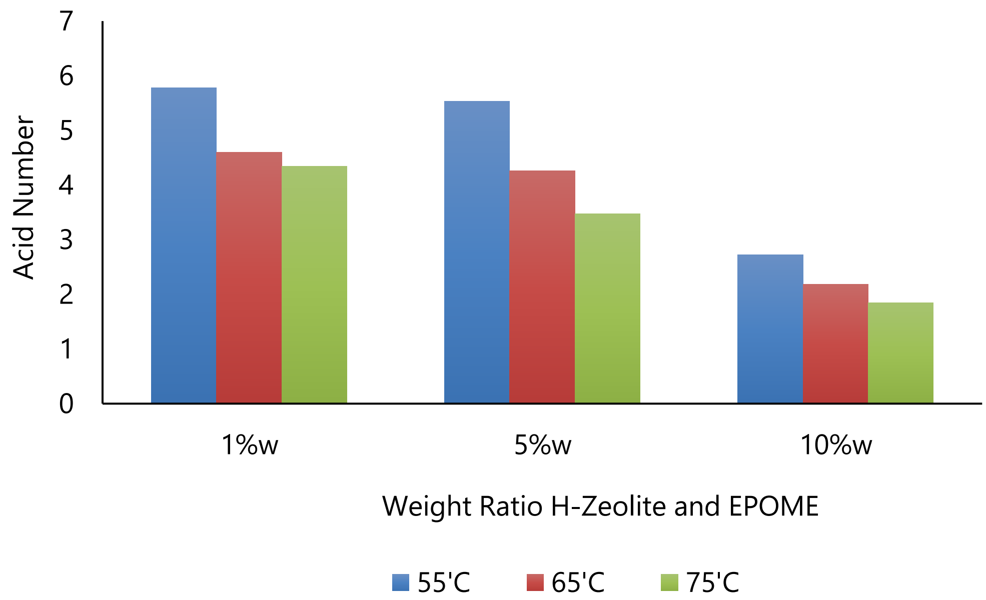

Figure 3. Effect ratio of H-zeolite catalyst and EPOME to acid number of bio-lube base oil

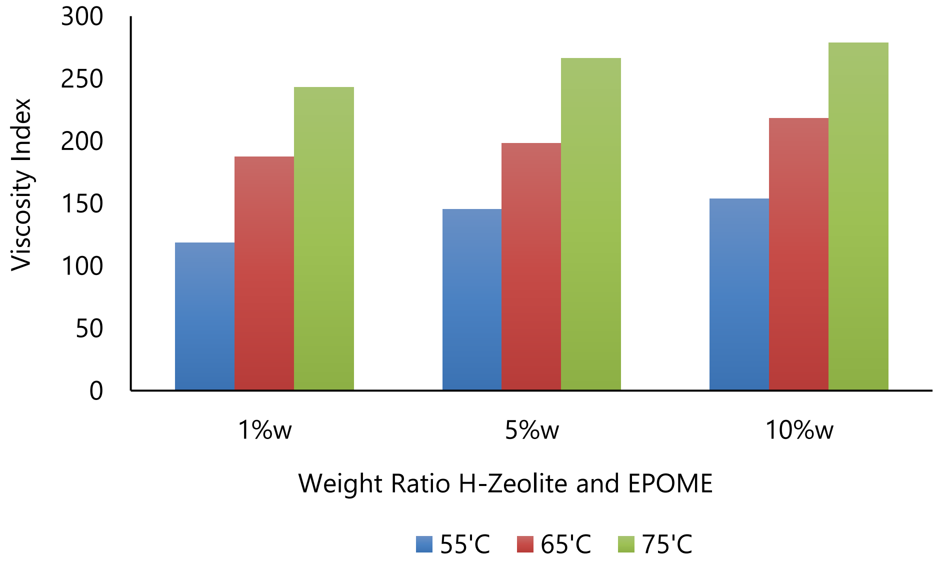

Figure 4. Effect ratio of H-zeolite catalyst and EPOME to viscosity index of bio-lube base oil

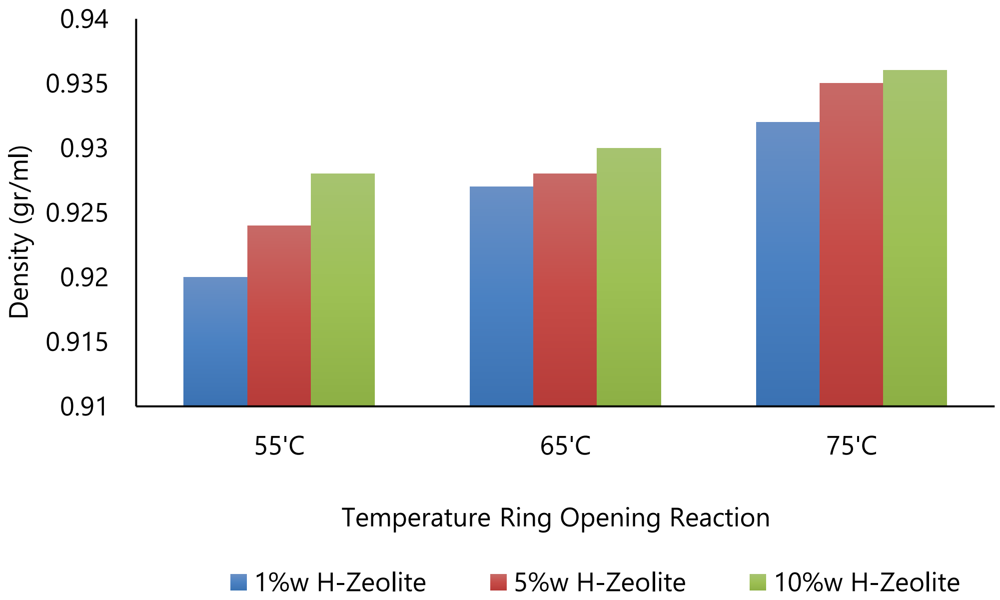

Figure 5. Effect temperature of ring-opening reaction to density of bio-lube base oil

index is an arbitrary number indicating the effect of changing temperature on the kinematic viscosity of alkyl esters. A high viscosity index signifies the relatively small change of kinematic viscosity with temperature. The study results demonstrated that viscosity performance was suitable for biolubricant production (Inkerd, 2015).

Results of Syaima, (2015) clearly demonstrate that when the temperature increased, the reaction rate increased. This has been anticipated since an increase in temperature would absolutely accelerate the process. High estrate of reaction was should at 75°C. With the increasing temperature reaction, catalysts H-Zeolite will more reactive and could do ring-opening reaction better. But, common problem with performing this reaction with strong acid is that epoxides of the major fatty acid component in the vegetable oil were found a significant amount of structure and this structure depend of the variety of the strong acid itself via intra-molecular etherification or inter-molecular polymerizing. This self-etherification/ polymerization leads to undesired product with increased molecular weight and viscosity (Benecke, 2017). This viscosity increase may be due to a higher molecular weight resulting from the ring opening polymerization of polyols and may also be due to the free hydroxyl groups. EPOME in 75°C has a much higher viscosity than EPOME in 65°C due to its higher molecular weight resulting from the high degree of chain coupling (Karadeniz, 2015).

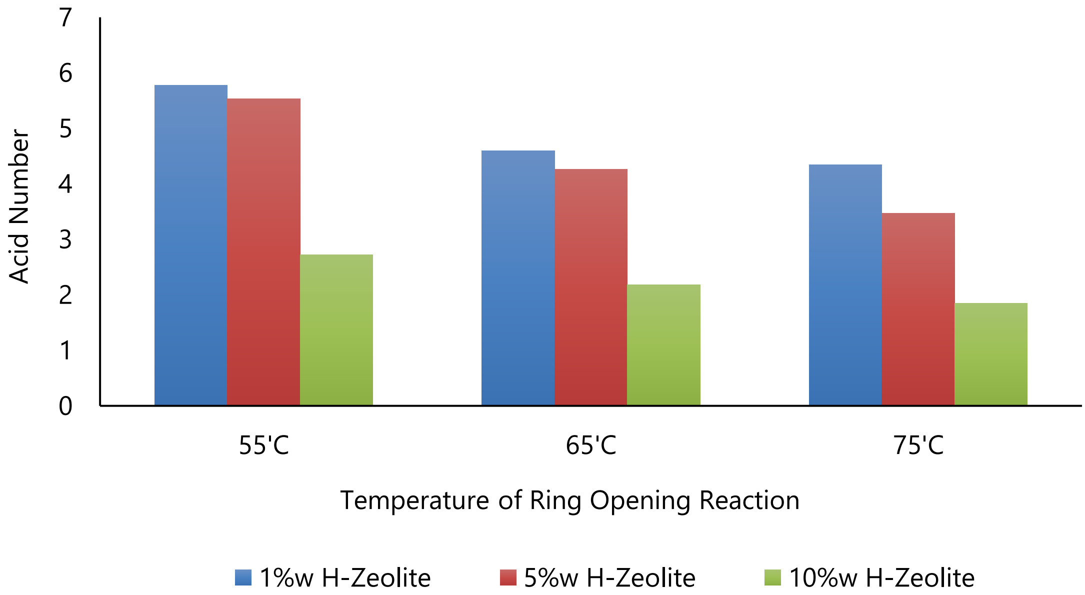

Figure 6. Effect temperature of ring-opening reaction to acid number of bio-lube base oil

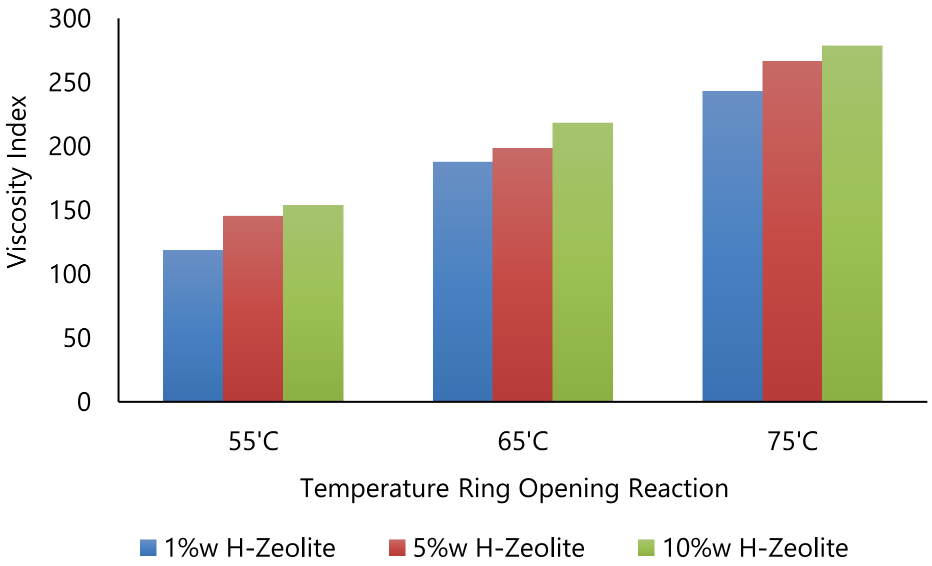

Figure 7. Effect temperature of ring-opening reaction to viscosity index of bio-lube base oil

But, in temperature 75ºC the viscosity is higher than 65ºC. This fact suggests that EPOME undergoes complete ring opening under the reaction condition to form thio-ether derivatives of hydroxy vegetable oils. The consequent generation of free hydroxyl groups resulted. The presence of these intermolecular hydrogen bonds also causes an increase in viscosity of the reaction product (Sharma, 2006).

CONCLUSION

Lube base oil was successfully made from palm oil which was processed by transesterification, epoxidation, and ring-opening reaction. 75ºC and 10%weight ratio of H-Zeolite as catalyst and EPOME provide the optimum result. Effect of weight ratio H-zeolite catalyst and EPOME to density, viscosity and acid number is higher ratio will decrease the activation energy of reaction and increase capability of reactant to add glycerol groups and improve the properties of bio lube base oil such density, viscosity and also decreasing acid number. Effect of ring opening reaction temperature to density, viscosity and acid number is, higher temperature will makes reaction reach optimum condition and improve the properties of bio lube base oil such density, viscosity and also decreasing acid number, but several problem like self-etherification/ polymerization leads to undesired product makes several product doesn't meet the acid number standard.

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