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PRODUCTION OF BIOLUBRICANTS FROM FATTY ACID METHYL ESTER (FAME) WITH VARIATIONS IN CATALYST CONCENTRATION AND STIRRING SPEED USING POTASSIUM HYDROXIDE (KOH) CATALYST

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Abstract

The demand for lubricants in Indonesia continues to increase along with the growth of the transportation and industrial sectors; however, reliance on petroleum-based (fossil) lubricants poses potential ecological pollution due to their non-biodegradable nature. This study aims to produce an environmentally friendly biolubricant through chemical modification via the transesterification reaction of palm oil-based (Crude Palm Oil/CPO) Fatty Acid Methyl Ester (FAME) and ethylene glycol into Ethylene Glycol Diester (EDGE) compounds. The synthesis process was investigated by assessing the effects of variations in the concentration of Potassium Hydroxide (KOH) as a homogeneous base catalyst at 0.5%, 1%, and 1.5% of the total mixture weight, as well as variations in stirring speed at 200 rpm, 300 rpm, 400 rpm, 500 rpm, and 600 rpm. The reaction was maintained under constant conditions at a temperature of 110 °C for 1 hour with a FAME to ethylene glycol molar ratio of 1:2. The results indicated that the variations in catalyst concentration and stirring speed significantly affected the physicochemical characteristics of the resulting biolubricant. The optimum condition was achieved at a KOH catalyst concentration of 1% and a stirring speed of 400 rpm, which yielded the highest kinematic viscosity value of 12.25 cSt. Generally, the kinematic viscosity of the product ranges from 11.0 to 12.25 cSt, falling within the quality classification range of SAE 30 according to the SNI 06-7069.5-2005 standard. Density testing showed stable results that met the ideal criteria for vegetable oil-based biolubricants, ranging between 0.860 and 0.900 g/ml. Analysis using Gas Chromatography-Mass Spectrometry (GC-MS) confirmed the presence of complex long-chain ester compounds, with the dominant components identified as 9-Octadecenoic acid (Z)-, methyl ester (methyl oleate) at 42.40% and Hexadecanoic acid, methyl ester (methyl palmitate) at 27.87%. The characteristics of this product prove that the biolubricant synthesis process continues optimally and holds great potential as an eco-friendly commercial lubricant alternative with good lubricity and biodegradability properties.

Keywords: Biolubricant, FAME, Ethylene Glycol, Potassium Hydroxide (KOH), Stirring Speed, Viscosity, Density, GC-MS.

1. INTRODUCTION

The need for lubricants in Indonesia continues to increase in line with the rapid growth of the transportation sector and the use of various industrial machines. Based on data from the Ministry of Industry of the Republic of Indonesia (2020), the installed capacity of the national lubricant industry reaches approximately 2.04 million kiloliters (kL) per year, with domestic consumption of 1.14 million kL per year. The automotive sector is the largest consumer with a consumption rate exceeding 781 thousand kL per year, while the industrial sector absorbs approximately 127 thousand kL of lubricants annually. The very high dependence on petroleum-based lubricants (fossil) poses a serious environmental threat due to their non-biodegradable nature and the potential to pollute soil and aquatic ecosystems.

The development of biolubricants based on renewable natural resources such as crude palm oil (CPO) is a strategic solution with high value. Indonesia, as the world's largest CPO producer, has abundant raw material availability. However, crude CPO cannot be used directly as an engine lubricant due to its weaknesses such as high

temperature resistance, unstable viscosity, and the susceptibility of triglyceride double bonds to oxidation reactions that trigger degradation of lubrication quality. Therefore, gradual chemical modification is required to transform the fatty acid structure of CPO into superior-quality ester derivatives.

The process of converting CPO into biolubricant is carried out through the initial stage of esterification/transesterification into Fatty Acid Methyl Ester (FAME), followed by an advanced transesterification reaction with ethylene glycol to form the Ethylene Glycol Diester (EDGE) compound. The use of ethylene glycol aims to produce branched bio-ester molecules that have high thermal-oxidative stability, a high viscosity index, and a low pour point. In this EDGE synthesis reaction, the use of a homogeneous base catalyst Potassium Hydroxide (KOH) and the reactor stirring rate play a crucial role. The appropriate catalyst concentration will lower the activation energy of the reaction, while the optimal stirring speed expands the contact area between the immiscible reactant phases (non-polar FAME and polar ethylene glycol).

This study was conducted to comprehensively analyze the effect of variations in KOH catalyst concentration (0.5%, 1.0%, and 1.5%) and variations in stirring speed (200, 300, 400, 500, and 600 rpm) on the physicochemical characteristics of biolubricants (kinematic viscosity, density, and yield) and to confirm the chemical composition profile of the product using Gas Chromatography-Mass Spectrometry (GC-MS) instrumentation.

2. RESEARCH METHOD

2.1 Tools and Materials

The main raw materials used in this study include refined CPO-based Fatty Acid Methyl Ester (FAME), technical-grade ethylene glycol (C2H6O2), analytical grade Potassium Hydroxide (KOH), and distilled water for the washing process. The reaction and testing equipment consists of a three-neck flask (glass reactor), a hotplate magnetic stirrer, a reflux condenser, a thermometer, a separating funnel, a 25 ml pycnometer, an Ostwald viscometer, a drying oven, a digital analytical balance, and a Gas Chromatography-Mass Spectrometry (GC-MS) instrument.

2.2 Experimental Design and Synthesis of Biolubricant

The synthesis of Ethylene Glycol Diester (EDGE) biolubricant was carried out by reacting FAME and ethylene glycol at a fixed molar ratio of 1:2. A total of 42.5 grams of FAME was put into a three-necked flask reactor, then 17.876 grams of ethylene glycol was added. KOH catalyst was added with varying concentrations of 0.5%, 1.0%, and 1.5% of the total weight of the reactant mixture. The reaction was carried out at a constant temperature of 110 °C for 1 hour with varying stirring speeds of 200, 300, 400, 500, and 600 rpm.

After the reaction was completed, the mixture was cooled to room temperature. The product was separated using a separating funnel and washed with warm distilled water five times to remove residual KOH catalyst and unreacted ethylene glycol. The EDGE biolubricant layer was dried in an oven at 40–50 °C until free of water, then stored in a sealed glass container for physicochemical characterization testing and further analysis.

2.3 Product Characterization Procedure

1. Kinematic Viscosity: Measured using an Ostwald Viscometer at 40 °C referring to the ASTM D445 standard. The flow time of the sample was recorded and the dynamic and kinematic viscosity (cSt) were calculated. 2. Density: Measured using a 25 ml pycnometer that had been calibrated at room temperature according to the ASTM D1475/SNI 7182:2015 method. 3. Yield: Calculated based on the percentage comparison between the mass of pure biolubricant obtained and the initial mass of FAME used. 4. GC-MS Analysis: Using a GC-MS instrument to identify the constituent components, the relative area percentage, and the molecular structure of the ester formed under optimum operating conditions.

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3. RESULTS AND DISCUSSION

3.1 Physicochemical Characterization of Biolubricants

The results of the physicochemical tests of EDGE biolubricant synthesized at various KOH catalyst concentrations and stirring speeds are presented in full in Table 1.

Mixing Speed (rpm)	Reaction Temperature (°C)	Reaction Time (hours)	KOH catalyst (%)	Density (g/ml)	Kinematic Viscosity (cSt)	Yield (%)
200	110	1	0.5	0.84	10.80	80
200	110	1	1.0	0.85	11.20	82
200	110	1	1.5	0.85	11.10	85
300	110	1	0.5	0.85	11.30	82
300	110	1	1.0	0.85	11.64	93
300	110	1	1.5	0.86	11.52	94
400	110	1	0.5	0.86	11.50	85
400	110	1	1.0	0.87	12.25	90
400	110	1	1.5	0.87	12.15	95
500	110	1	0.5	0.87	11.50	86
500	110	1	1.0	0.87	11.80	92
500	110	1	1.5	0.88	11.40	94
600	110	1	0.5	0.88	11.40	84
600	110	1	1.0	0.89	11.65	89
600	110	1	1.5	0.88	11.35	90

Table 1. Physicochemical Characteristics of Synthesized Biolubricants

3.2 Kinematic Viscosity Analysis

Kinematic viscosity is a vital parameter that measures a fluid's resistance to shear and its ability to form a protective film on engine surfaces. Based on the test results (Table 1), the viscosity of the biolubricants ranged from 10.80 cSt to 12.25 cSt. The highest peak viscosity of 12.25 cSt was achieved at a stirring speed of 400 rpm with a KOH catalyst concentration of 1.0%.

Increasing the catalyst concentration from 0.5% to 1.0% was shown to significantly increase the viscosity. This was due to the increase in alkoxide ions that activated the remaining FAME ester groups to react with ethylene glycol,

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producing long-chain branched esters with stronger intermolecular forces (Van der Waals and hydrogen bonds). However, the addition of catalysts exceeding the optimum limit (1.5%) caused a decrease in the kinematic viscosity to 12.15 cSt at 400 rpm and 11.40 cSt at 500 rpm. This excess condition triggered the saponification side reaction (soap formation) and partial degradation of the ester chain.

Stirring the reactor at 400 rpm provides an optimum turbulent climate for contact between reactants without damaging the molecular structure. Too high a stirring speed (500–600 rpm) creates mechanical shear stress that triggers chain scission, resulting in decreased product viscosity. According to the SNI 06-7069.5-2005 standard, the viscosity value of the resulting biolubricant product meets the quality criteria for SAE 30 viscosity grade motor lubricants (range 9.3–12.5 cSt).

3.3 Density Analysis

The density of the synthesized biolubricant ranged from 0.84 g/ml to 0.89 g/ml. This value indicates excellent stability and is right within the quality standard range of vegetable oil-based biolubricant (0.860 – 0.900 g/ml) according to SNI 06-7069.5-2005. The density increased gradually with increasing stirring speed from 200 rpm (0.84–0.85 g/ml) to 600 rpm (0.88–0.89 g/ml). Faster stirring breaks the ethylene glycol phase into fine droplets, thus expanding the interfacial contact area and enhancing the formation of high-density complex ester molecules.

3.4 Yield Analysis

The yield of biolubricant products increased consistently with increasing KOH catalyst concentration and stirring speed up to 400 rpm. The highest yield reached 95% at a stirring speed of 400 rpm and a KOH catalyst of 1.5%. At 200 rpm, the yield ranged from 80–85% due to mass-transfer limitations. Conversely, at 600 rpm, the yield decreased slightly (89–90%) due to excessive emulsion formation, which complicated the phase separation and product washing processes.

3.5 Chemical Composition Analysis Using GC-MS

The chemical compound content of the optimum biolubricant (400 rpm sample, 1.0% KOH) was analyzed using Gas Chromatography-Mass Spectrometry (GC-MS). A summary of the dominant components is presented in Table 2.

No	Compound Name (Peak Name)	Retention Time (minutes)	Rail Area (%)	Chemical Formula
1	9-Octadecenoic acid (Z)-, methyl ester (Methyl Oleate)	28,102 - 28,224	42.40	C19H36O2
2	Hexadecanoic acid, methyl ester (Methyl Palmitate)	25,918	27.87	C17H34O2
3	Oxiraneoctanoic acid, 3-octyl-, methyl ester	29,745	5.69	C19H36O3
4	2-Hydroxyethyl ester derivatives / Polyol Ester	30,432 - 31,802	11.54	Complex

Table 2. Composition of Main Chemical Compounds of Biolubricants Based on GC-MS Analysis

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GC-MS results confirmed the success of the transesterification reaction with the formation of long-chain ester groups. The dominance of methyl oleate (42.40%) provided superior lubricity and maintained fluidity at cold temperatures, while methyl palmitate (27.87%) improved the thermal-oxidative stability of the product. The presence of hydroxy ester peaks (EDGE) at high retention times (29–32 minutes) proved the formation of a high-value-added biolubricant molecular structure.

4. CONCLUSION AND SUGGESTIONS

4.1 Conclusion

1. Variations in KOH catalyst concentration and stirring speed significantly affect the physicochemical characteristics of biolubricants synthesized from FAME CPO and ethylene glycol. 2. The optimum operating conditions were achieved at a KOH catalyst concentration of 1.0% and a stirring speed of 400 rpm, which resulted in the highest kinematic viscosity of 12.25 cSt, a density of 0.87 g/ml, and a yield of 90%. 3. The kinematic viscosity value of the resulting biolubricant (11.0–12.25 cSt) meets the quality criteria of SAE 30 motor lubricants based on the SNI 06-7069.5-2005 standard. 4. GC-MS analysis confirmed the main composition of the product in the form of long-chain ester compounds with dominant components of methyl oleate (42.40%) and methyl palmitate (27.87%), which supports environmentally friendly lubrication performance.

4.2 Suggestions

Further tribological characteristic testing (such as friction coefficient and wear scar diameter), oxidation stability testing (RPVOT), and pour point testing are required for applications in heavy-duty industrial machines.

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