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Identification of FAME (Fatty Acid Methyl Esters) Products from the Interesterification Reaction of Palm Oil with Methyl Ester Co-solvent

Elvianto Dwi Daryono[1]✉, Dwi Ana Anggorowati[2]

[1, 2] Study Program Chemical Engineering, Faculty of Industrial Technology, Institut Teknologi Nasional Malang, Indonesia

✉ **Corresponding Author :** Elvianto Dwi Daryono (e-mail: elviantodaryono@lecturer.itn.ac.id)

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ABSTRACT

Methyl ester co-solvent will accelerate the reaction without the need for a separation stage at the end of the reaction. The study aims to determine the effect of FAME co-solvent concentration and time of reaction on the methyl ester produced in the interesterification reaction of palm oil into biodiesel with methyl ester co-solvent. The weight of the oil used was 250 g, the mole ratio of oil: methyl acetate = 1:6, the temperature was 60°C, the concentration of the co-solvent (0 - 20%), the reaction time (30-90 min), the catalyst KOH 1% and the stirring speed was 800 rpm. The oil, methyl acetate and catalyst were placed in a three-necked flask and the reaction was carried out according to the operating conditions. The optimum conditions were obtained in the palm oil interesterification process with a FAME co-solvent concentration of 20% and a time of reaction of 30 min with a methyl ester concentration of 61,413 mg/L and an acid value of 0.28 mg KOH/g which met SNI 7182-2015. The research revealed a biodiesel production process with fewer steps, faster reaction times, higher yields, and product physical properties that meet standards. It is hoped that this process can be implemented at a pilot plant scale, bringing the selling price of biodiesel closer to that of diesel fuel.

INTRODUCTION

The process of making biodiesel by transesterification reaction is less efficient because it requires a separation stage of glycerol by-product (Chitra et al., 2005). The interesterification process is more efficient because the triacetin by-product does not need to be separated and functions as a bioadditive in fuel (Casas et al., 2010, 2011; Kusumaningtyas et al., 2016). Research on the interesterification process has been carried out with quite satisfactory results. Interesterification of used cooking oil with a molar ratio of NaOH: oil = 0.01:1 obtained a yield of 77.5% at a reaction time of 3 hours (Chuepeng & Komintarachat, 2018). Interesterification of palm oil with a NaOH catalyst obtained a yield of 87% at a reaction time of 1 hour and a stirring speed of 400 rpm and methyl esters that met the standards (Daryono et al., 2021). Interesterification of CPO with a KOH catalyst obtained a yield of 57.3% yield at 1 hour and a stirring speed of 300 rpm (Daryono et al., 2022). Interesterification of used cooking oil by electrolysis yielded a product of approximately 550 mL at an oil:methyl acetate weight ratio of 1:3 in 100 minutes (Aziz et al., 2024). Synthesis of palm oil biodiesel with 1% NaOH in a reaction time of 3 hours achieved a yield of 49% and methyl esters that met SNI standards (Sandra et al., 2021). The interesterification process of used cooking oil using aromatic compound biocatalysts also yielded satisfactory results (Daryono et al., 2024; Daryono, Wardana, et al., 2025; Daryono, Widi, et al., 2025; Daryono &

Dewi, 2022; Sativa et al., 2024). The interesterification process that has been carried out requires a relatively long time to achieve a high yield.

The biodiesel production process is more efficient if a co-solvent is added to increase the solubility of the reactants (Boocock et al., 1996). Co-solvents are soluble in alcohol and triglycerides (Daryono & Sinaga, 2017). The transesterification process of jatropha curcas oil with THF co-solvent yielded a FAME (Fatty Acid Methyl Esters) content of 95% in 10 minutes (Muyassaroh et al., 2012). Transesterification of candlenut oil with acetone co-solvent yielded a methyl ester content of 93.12% in 20 minutes (Roni et al., 2025). In the aforementioned study, the co-solvent used required separation, making the process less efficient. The co-solvent that did not require a separation step was the reaction product itself, methyl ester. Transesterification of CPO (Crude Palm Oil) with 5% FAME co-solvent for 30 minutes achieved a yield of 97.4%, a FAME concentration of 99.96%, and physical properties that met standards (Daryono & Mustiadi, 2022a). The addition of FAME co-solvent increases the solubility of the reactants, resulting in a high conversion rate (Daryono, 2020; Daryono & Mustiadi, 2017, 2022b; Daryono & Sinaga, 2016b, 2016a). The research conducted on the interesterification process using FAME as a co-solvent did not include details of the components and concentrations of the resulting methyl esters. This is crucial to determine whether the resulting methyl ester components align with the raw materials used.

The interesterification process with the addition of FAME co-solvent is an efficient process that addresses the shortcomings of the previous process. The objective of this study was to determine the optimum conditions for the interesterification of palm oil and methyl acetate with the addition of FAME co-solvent, related to the KOH catalyst concentration and stirring speed. The results of this study will provide data on methyl ester concentration, and physical properties of the resulting product. The problem formulation is how the reaction time and FAME co-solvent concentration affect the components and concentration of methyl ester produced in the interesterification reaction of palm oil with KOH catalyst.

RESEARCH METHODS

Materials and Equipment

The materials were packaged palm oil, HCl (Sigma Aldrich, 37%), H_3PO_4 (Sigma Aldrich, 85%), ethanol (Sigma Aldrich, 96%), pp indicator, KOH pellets (Riedel-de Haen, 99%), methanol (Merck, 99.9%), methyl acetate (Sigma Aldrich, 98%), and NaOH (Riedel-de Haen, 99%). The equipment used included a three-neck flask with reflux, an Erlenmeyer flask, a pycnometer, a hot plate with a magnetic stirrer, a beaker, a graduated cylinder, a graduated pipette, a centrifuge, a complete distillation apparatus, a volumetric pipette, and a GC (Shimadzu).

Methyl Ester Co-solvent Preparation

The transesterification reaction was carried out under operating conditions: 250 g oil mass, 1% NaOH, stirring at 100 rpm, 1 hour, temperature at 60°C, and a mole ratio of 1:6. After the reaction was complete, 1 N HCl was added to neutralize the catalyst to pH 7. The reaction product was placed in a separatory funnel for approximately 12 hours. The upper layer was analyzed by GC and used as a co-solvent. The flow diagram of the FAME co-solvent manufacturing process is presented in Figure 1.

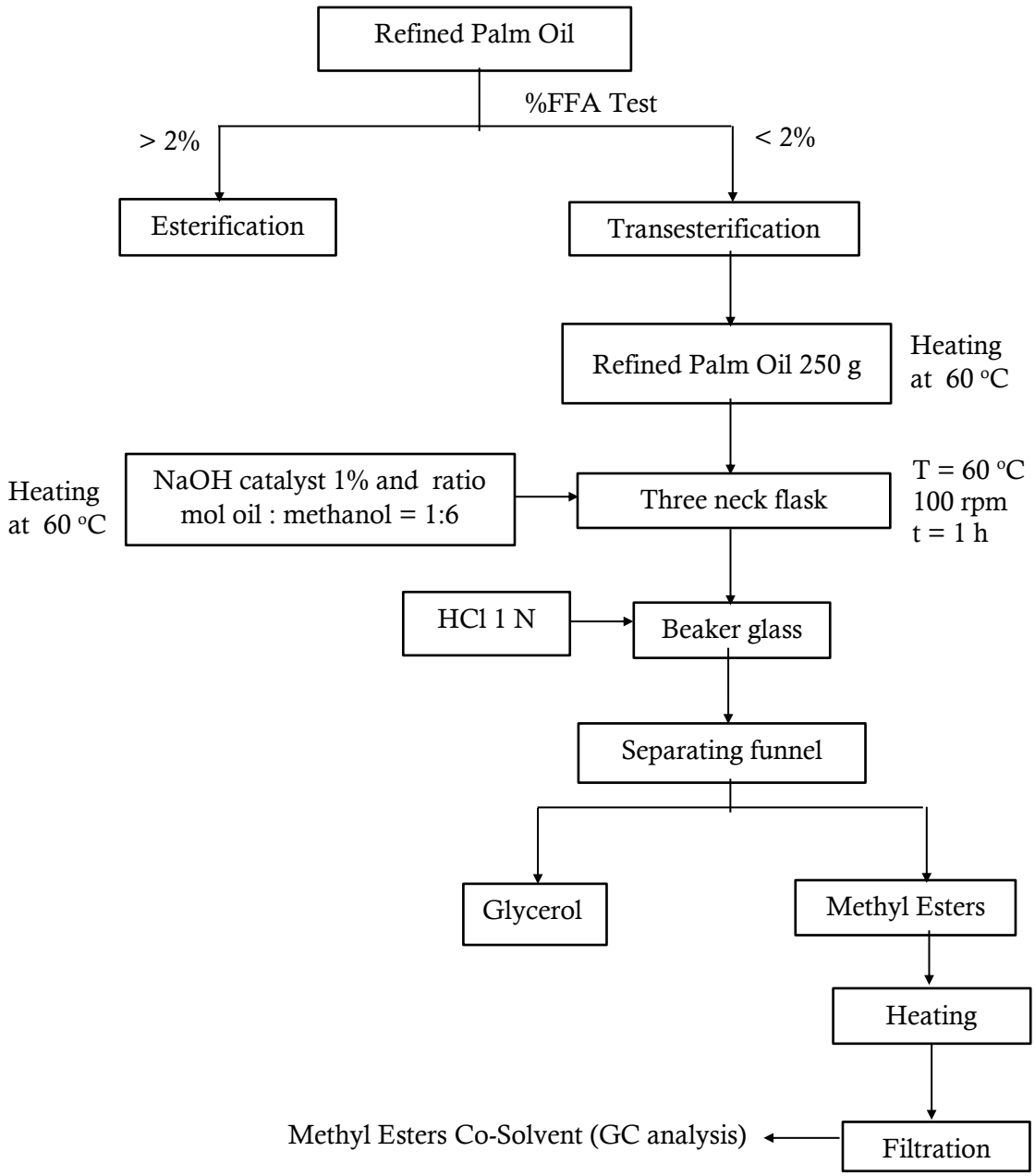

Figure 1. Flowchart of Co-Solvent Production

Interesterification Reaction of Palm Oil with FAME Co-solvent

A 250 g oil, methyl acetate at a mole ratio of 1:6 and KOH catalyst 1%wt. oil were heated to 60°C. After reaching the desired temperature, FAME co-solvent variable concentrations (0, 5, 10, 15, 20% wt. oil) was added to a three-necked flask equipped with a condenser. The mixture was heated at a constant temperature of 60°C using a hot plate equipped with a magnetic stirrer at stirring speed 800 rpm for various time of reaction (30, 60, 90 minutes). For each reaction time variable, a sample of 50 g was taken and H_3PO_4 was added to the reaction mixture until the pH was neutral to neutralize the KOH catalyst and stop the reaction. The flow diagram of the interesterification reaction of palm oil with FAME co-solvent is presented in Figure 2.

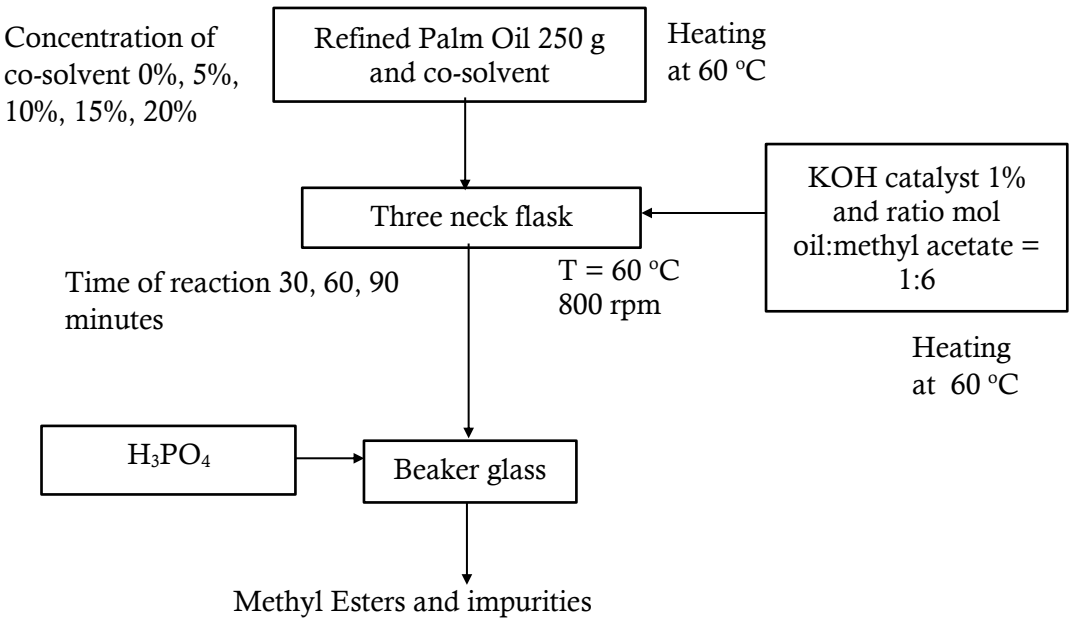

Figure 2. Flowchart of Interesterification Process with FAME Co-Solvent

Separation of Reaction Products from Remaining Methyl Acetate

The reaction products were separated by centrifugation at 1500 rpm for 1 minute to separate the reaction products and potassium phosphate precipitate. The products and residues were separated by distillation at 110°C. The distillate consisted of methyl acetate and residual water, while the residue consisted of methyl ester and triacetin. The residue was then filtered and stored in a refrigerator at 17°C before analysis. The residue was analyzed using GC (Gas Chromatography). The highest concentration of methyl ester resulting from GC analysis will be analyzed for physical properties, namely density and acid value. The flow diagram of the separation of reaction products interesterification reaction of palm oil with FAME co-solvent is presented in Figure 3.

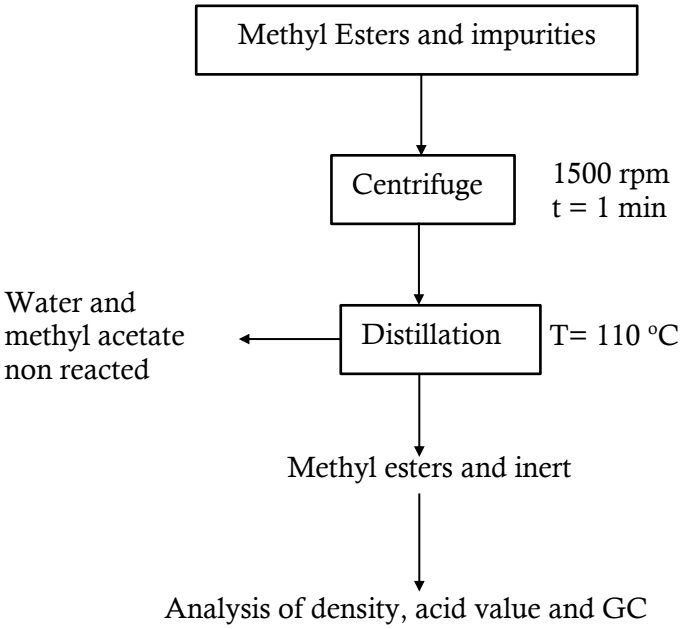

Figure 3. Flowchart of Interesterification Process with FAME Co-Solvent

Physical Property Analysis

Physical property analysis, namely density and acid value, was only performed on samples under optimum research conditions.

Density Test

An empty pycnometer was weighed. The pycnometer was filled with the methyl ester sample until it was full, heated to 40°C, and then reweighed. The density of the methyl ester was calculated using equation 1.

$$Density\ (\frac{g}{mL}) = \frac{Weight\ of\ picnometer\ filled - weight\ of\ picnometer\ empty}{Volume\ of\ picnometer} \quad (1)$$

Acid Value Test

A sample weighing 19-21 ± 0.05 g was weighed, 100 mL of acetone and 3 drops of PP indicator were added. The sample was titrated with 0.1 N KOH until a pink color persisted for at least 15 seconds. The acid value was calculated using equation 2.

$$Acid\ value\ (mg\frac{KOH}{g}) = \frac{56{,}1\ x\ volume\ of\ KOH\ x\ N\ KOH}{Weight\ of\ sample} \quad (2)$$

Optimization Criteria Used

Optimized Research Variables:

Concentration of FAME co-solvent : 0%, 5%, 10%, 15% and 20%

Time of Reaction : 30, 60 and 90 minutes

Optimized Result Response:

The FAME concentrations in mg/L from the GC analysis for each research variable are shown in Table 1 below.

Table 1. Research Result Data Design

Time of Reaction (minutes)	Concentration of Co-solvent (%)	Konsentrasi FAME (mg/L)
30	0	
	5	
	10	
	15	
	20	
60	0	
	5	
	10	
	15	
	20	
90	0	
	5	
	10	
	15	
	20	

The research data is presented in the form of a figure showing the relationship between reaction time and co-solvent concentration and the concentration of methyl ester product.

RESULTS AND DISCUSSION

Preparation of Methyl Ester Co-solvent

The concentration and components of the methyl ester co-solvent were analyzed using GC. Figure 4 shows the GC chromatogram of the methyl ester co-solvent. The analysis results showed a methyl ester concentration in the co-solvent of 1,23933.10^5 mg/L.

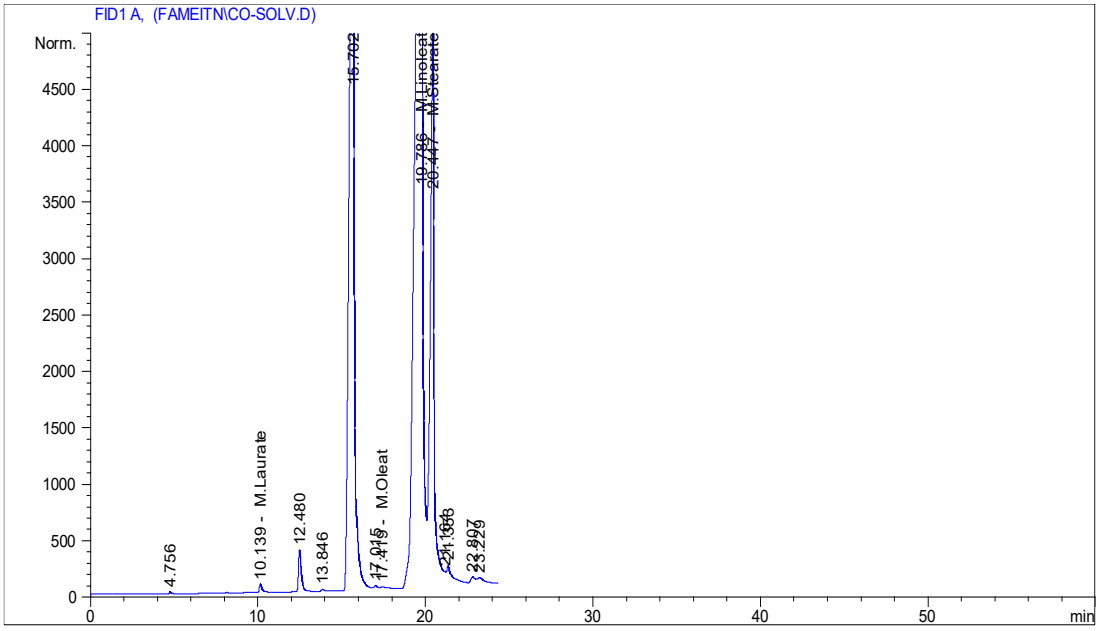

Figure 4. GC chromatogram of FAME co-solvent

Interesterification Reaction of CPO with FAME Co-solvent

The levels of methyl ester formed in the interesterification reaction of CPO with methyl ester co-solvent are presented in Figure 5.

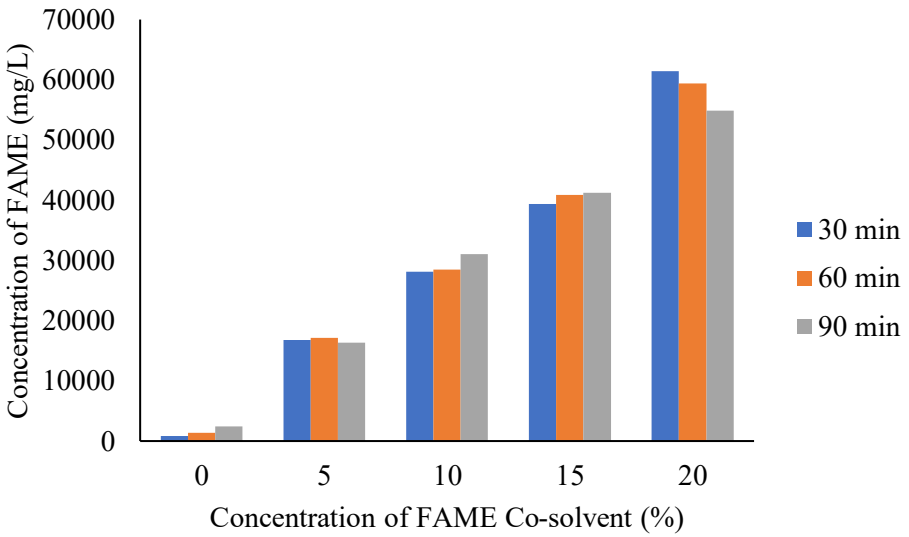

Figure 5. Relationship between reaction time and FAME co-solvent concentration and methyl ester concentration

In Figure 5, it can be seen that the reaction time and FAME co-solvent concentration are directly proportional to the methyl ester concentration obtained. At a FAME co-solvent concentration of 0%, the best condition was obtained at a reaction time of 90 minutes with a methyl ester concentration of 2,455 mg/L. At a FAME co-solvent concentration of 5%, the optimum

condition was obtained at a reaction time of 60 minutes with a methyl ester concentration of 17,173 mg/L. At a FAME co-solvent concentration of 10%, the best condition was obtained at a reaction time of 90 minutes with a methyl ester concentration of 31,036 mg/L. At a FAME co-solvent concentration of 15%, the best condition was obtained at a reaction time of 90 minutes with a methyl ester concentration of 41,220 mg/L. At a FAME co-solvent concentration of 20%, the best condition was obtained at a reaction time of 30 minutes with a methyl ester concentration of 61,413 mg/L. The concentration of methyl ester increases with the increase in the FAME co-solvent concentration and reaction time until the optimum conditions are achieved. If the optimum conditions have been achieved but the FAME co-solvent concentration and reaction time continue to be increased, the reaction equilibrium will shift towards the reactants because the interesterification reaction is reversible (Daryono et al., 2022). By shifting the reaction equilibrium to the left, the formed product will be further broken down, thereby reducing the methyl ester formed. Overall, the optimum conditions for the study were obtained at a FAME co-solvent concentration of 20% with a reaction time of 30 minutes. In the interesterification reaction of palm oil with 15% FAME co-solvent, 0.5% KOH catalyst, and a stirring speed of 600 rpm for 1 hour, a methyl ester concentration of 4.8% was obtained (Daryono, 2020). In this study, a reaction time of 1 hour was used as a fixed variable, so the reaction may have reached equilibrium before 1 hour, resulting in a decrease in conversion. This decrease in conversion also caused the FAME concentration to decrease because the reaction had shifted toward the reactants after 1 hour. The results of this study were better because a concentration of 6.82% was obtained in a reaction time of 30 minutes.

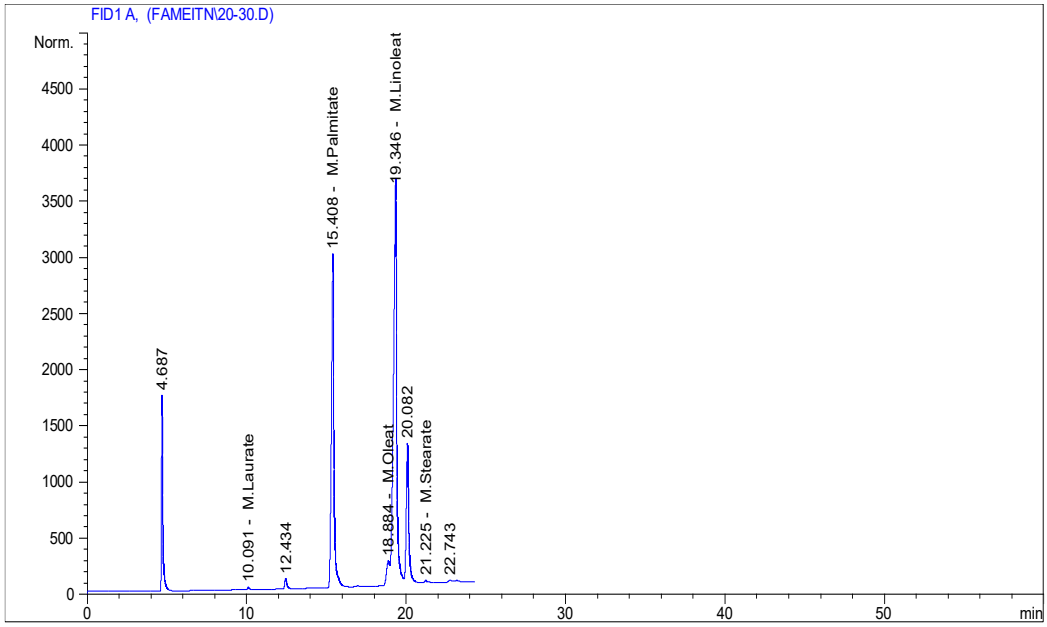

Figure 6. GC chromatogram of methyl ester under optimum process conditions

Table 2. Methyl ester concentration (%) from GC analysis at a FAME co-solvent concentration of 20% with a reaction time of 30 minutes.

Retention Time (min)	Component	Concentration (mg/L)
10.091	Methyl Laurate	409.3767
15.408	Methyl Palmitate	41,005
18.884	Methyl Oleate	3731.4586
19.346	Methyl Linoleate	16,166
21.225	Methyl Stearate	101.4279
	Total	61,413

Table 2 shows that the two largest methyl ester components are methyl palmitate and methyl linoleate. The two largest methyl ester components correspond to the fatty acid content of palm oil: palmitic acid and oleic acid (Marlina et al., 2020). Figure 6 shows the GC chromatogram of the methyl ester components under optimum process conditions.

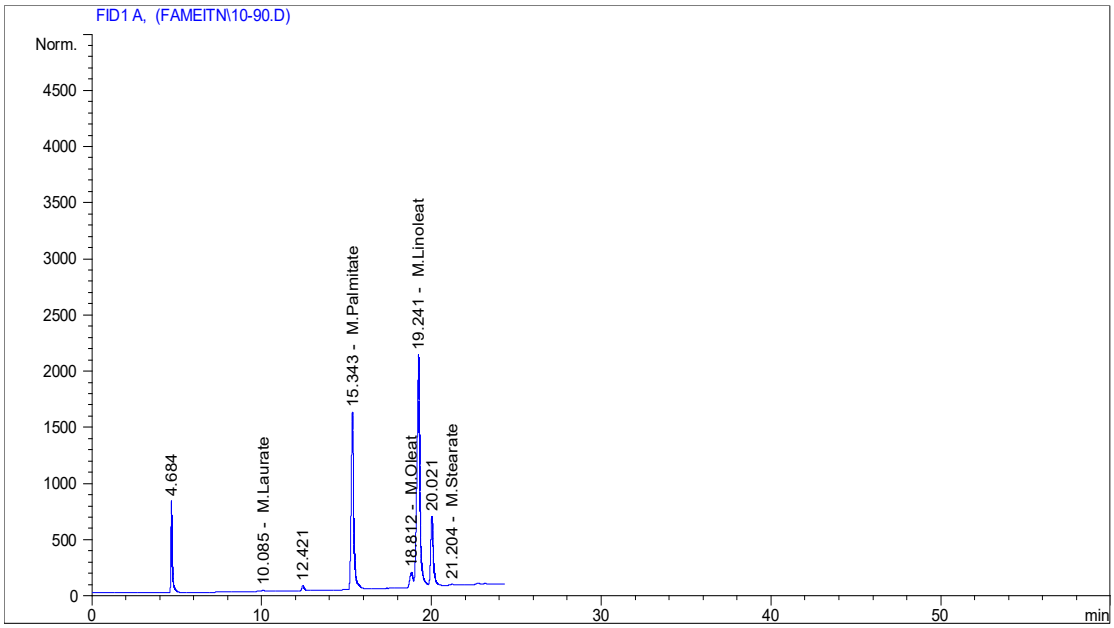

Figure 7. GC chromatogram of methyl ester at 10% co-solvent and 90 minutes reaction time

Table 3. Methyl ester concentration (%) from GC analysis at a FAME co-solvent concentration of 10% with a reaction time of 90 minutes.

Retention Time (min)	Component	Concentration (mg/L)
10.085	Methyl Laurate	302.9071
15.343	Methyl Palmitate	20,556
18.812	Methyl Oleate	2135.5356
19.2416	Methyl Linoleate	8023.5509
21.204	Methyl Stearate	18.3958
	Total	31,036

Table 3 shows the residence time, concentration and components of the methyl ester formed that interesterification reaction of palm oil with a co-solvent concentration of 10% and a reaction time of 90 minutes. The two largest methyl ester components are methyl palmitate and methyl linoleate. Figure 7 shows the GC chromatogram of methyl ester at 10% co-solvent and 90 minutes reaction time.

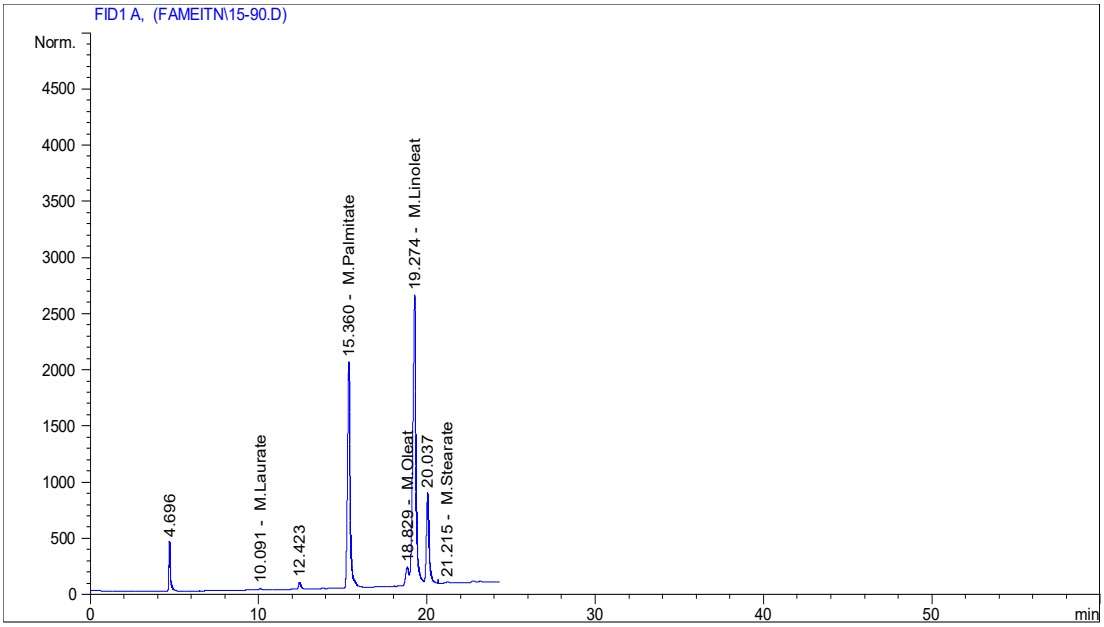

Figure 8. GC chromatogram of methyl ester at 15% co-solvent and 90 minutes reaction time

Table 4. Methyl ester concentration (%) from GC analysis at a FAME co-solvent concentration of 15% with a reaction time of 90 minutes.

Retention Time (min)	Component	Concentration (mg/L)
10.091	Methyl Laurate	333.8351
15.360	Methyl Palmitate	27,611
18.829	Methyl Oleate	2761.0652
19.274	Methyl Linoleate	10,802
21.215	Methyl Stearate	46.1903
	Total	41,220

Table 4 shows the residence time, concentration and components of the methyl ester formed that interesterification reaction of palm oil with a co-solvent concentration of 15% and a reaction time of 90 minutes. The two largest methyl ester components are methyl palmitate and methyl linoleate. Figure 8 shows the GC chromatogram of methyl ester at 15% co-solvent and 90 minutes reaction time.

Physical Property Analysis

The results of the density and acid value analysis of the samples under optimum process conditions, namely the CPO interesterification reaction with a FAME co-solvent concentration of 20% with a reaction time of 30 minutes, are presented in Table 5.

Table 5. Density and Acid Value Data Under Optimum Process Conditions

Time of reaction (min)	Concentration of FAME co-solvent (%)	Acid value (mg KOH/g sample)	Density (g/mL)
30	20	0,29	0,90

Table 4 shows that the density does not comply with SNI 7182:2015 (0.85 - 0.89 g/mL). This is because there is still a lot of unreacted oil, so the density value of the reaction product is still close to the initial oil density of 0.93 g/mL. The acid value has met SNI 7182:2015 (max. 0.5 mg-KOH/g).

CONCLUSION

The greater the concentration of FAME co-solvent and the higher the time of reaction, the greater methyl ester concentration obtained until the optimum conditions are reached. The optimum conditions were obtained in the interesterification reaction of CPO and methyl acetate with a KOH catalyst 1%, stirring at 800 rpm, a time of 30 min, a temperature of 60°C, a co-solvent weight of 20% and a mole ratio of 1:6. The methyl ester concentration of 61,413 mg/L and an acid value of 0.28 mg KOH/g which met SNI 7182-2015. The research results obtained a faster and more efficient biodiesel production process so that it can be applied on a pilot plant scale.

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