

PRODUCTION OF BIODIESEL WITHOUT CATALYST SEPARATION WITH PALM OIL INTERESTERIFICATION PROCESS USING ESSENTIAL OIL BIOCATALYST

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Abstract. The transesterification is a method commonly used in the production of biodiesel. Therefore, this study aimed to investigate interesterification reaction of palm oil with methyl acetate using essential oil biocatalyst, specifically focusing on eugenol and cajuput oil. The operating parameters were: 250 g of palm oil, a molar ratio of palm oil:methyl acetate of 1:6, a reaction temperature of 60 °C, a stirring speed of 300 rpm, a biocatalyst of 0.75 wt. % in relation to oil, and reaction times of 15, 30, 45, 60 and 75 min. The palm oil, methyl acetate and biocatalyst reacted in a three-necked flask as a reactor under operating conditions. Additionally, simulations were performed using ChemDraw Professional 15.0 to analyze the molecular behavior of the reacting compounds.

Keywords: interesterification, essential oil, eugenol, crude yield, molecular behavior.

1. Introduction

The transesterification method is widely used in biodiesel production. Another method of biodiesel production is the change from an alcoholic to an alcohol-free reaction, which is commonly known as interesterification. In the alcoholic route, methanol is used to supply the alkyl groups, while in the non-alcoholic route methyl acetate is the supplier of methyl groups. The reaction produces triacetin and glycerol as by-products in the non-alcoholic and alcoholic routes, respectively¹.

Several studies of biodiesel production with interesterification process have been carried out, including various catalysts and methods. These include interesterification with heterogeneous catalyst^{2–8}, homogeneous catalyst^{1,9–11}, lipase enzyme¹², and ultrasonic waves¹³. The process was also carried out under supercritical condi-

tions¹⁴, namely ultrasonic waves¹⁵, with hydrodynamic cavitation¹⁶. A catalyst was used during the investigation of the interesterification process to accelerate the reaction, except under supercritical conditions, which apply high temperatures and pressure. In the process with a homogeneous catalyst, a neutralization reaction stage and continued separation of the reaction products are essential. Meanwhile, a heterogeneous catalyst uses an activation and separation stage to prolong the process stages, resulting in higher production costs. A previous study has conducted interesterification of palm oil without catalyst at high temperature (340–420 °C) and pressure (15–25 MPa) using oil:methyl acetate molar ratio of 20:60¹⁷. This indicates that operating conditions and tools in the supercritical interesterification process require high costs and process safety standards.

A study on the biodiesel production process using an essential oil biocatalyst has not been carried out. In addition to being environmentally friendly and relatively inexpensive, the essential oil biocatalyst does not require complex and lengthy pretreatment, such as a heterogeneous catalyst. The presence of essential oils in biodiesel does not pose a significant threat, because they act as antioxidants. In addition, they can be easily separated by co-distillation with the methyl acetate left after the reaction. The use of an essential oil biocatalyst also overcomes the mass transfer limitations due to the different solubilities of the two reagents.

Essential oil was used as a biocatalyst / bio-additive in several studies. For example, the combination of rhodium (III) sulfate catalyst with clove oil was shown to elevate combustion temperature and reduce ignition delay and combustion rate¹⁸. The addition of eucalyptus oil to CPO also reduces viscosity, flash point, and ignition delay, as cineol in eucalyptus oil weakens the van der Waals bond of CPO¹⁹.

Based on the background above, this study aimed to determine the optimum conditions for the interesterification of palm oil using essential oil biocatalysts. The molecular behavior of essential oils, including eugenol and cajuput oil, was also determined in the conversion of

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palm oil into methyl esters. The major ingredient of clove oil is eugenol, which comprises 25.261 % eugenol according to GC analysis²⁰.

2. Experimental

2.1. Materials

The materials used in this study included aquadest (H₂O), acetone, potassium hydroxide (Merck, 90 %), methyl acetate (Sigma Aldrich, 99.9 %), palm oil (brand Sunco), phenolphthalein indicator (PP), commercial cajuput oil with a cineol concentration of 29.596 % and eugenol (Merck) at 56.645 %.

2.2. Methods

2.2.1. Preparation of Materials

Free Fatty Acid (FFA) and moisture content of palm oil were determined. When % FFA < 3 % and moisture content < 0.6 %, palm oil is considered suitable for immediate use in the interesterification process²¹.

FFA Test²²

A total of 20 g of oil was weighed and heated to a temperature of 65 °C. This was followed by the addition of 50 mL of 96 % ethanol and 3 drops of PP indicator into the Erlenmeyer flask. The solution was allowed to cool at room temperature and titrated until the color turned pink with a 0.1 N KOH solution. Subsequently, the required titration volume was recorded and % FFA was calculated using equation (1):

$$FFA (\%) = \frac{ml\ KOH \times N\ KOH \times MW\ of\ fatty\ acid}{mass\ of\ sample \times 1000} \times 100 \%. \quad (1)$$

Determination of Moisture Content²³

A total of 10 g of oil was weighed, placed in an oven at 110°C for 30 minutes, and allowed to cool in a desiccator at room temperature to obtain a constant mass. Subsequently, the moisture content of palm oil was calculated using equation (2):

$$\text{Moisture content } (\%) = \frac{\text{initial mass} - \text{final mass}}{\text{initial mass}} \times 100 \%. \quad (2)$$

2.2.2. Interesterification Reaction with Essential Oil Biocatalyst

The study variables were used to weigh palm oil, methyl acetate, and biocatalyst. Palm oil was placed in an Erlenmeyer flask, while methyl acetate and biocatalyst were added to a three-neck flask, and each component was heated to 60 °C. Subsequently, palm oil was poured into a three-neck flask containing methyl acetate and biocatalyst that had been heated to 60 °C with a hot plate.

All components were introduced into a three-neck flask fitted with a reflux condenser and the reaction temperature was maintained at 60 °C. Subsequently, the mixture was stirred with a magnetic stirrer at 300 rpm, and response time was assessed based on the study factors. After the end of the reaction time, 50 g of the reaction product was taken for each variant of the reaction time.

2.2.3. Process of Separation of Interesterification Reaction Results with Essential Oil Biocatalyst

At 105 °C, the reaction mixture was distilled to separate the residual methyl acetate and water. The distillation process continued until no more droplets of distillate, which consisted of methyl acetate and water, were observed. The residues included oil / triglycerides from the reaction, methyl esters, triacetin, eugenol, or cajuput oil. After the distillation was completed, the residue from the distillation was weighed as a product of interesterification reaction and analyzed for concentration and composition using the GC analysis. The product obtained from the interesterification reaction was analyzed according to the biodiesel standard (SNI 7182-2015) and crude yield was calculated using the equation (3):

$$Crude\ yield\ (\%) = \frac{\text{Mass of product (g)}}{\text{Mass of initial oil (g)}} \times 100 \%. \quad (3)$$

2.2.3. Analysis

Density Test¹¹

The density test was performed by weighing the empty pycnometer, followed by inserting the methyl ester into the pycnometer. Subsequently, the weight of the pycnometer was determined to assess the density of the methyl ester, which was calculated using equation (4):

$$\text{Density } \left(\frac{g}{mL} \right) = \frac{\text{weight of pycno's filled} - \text{weight of pycno's empty}}{\text{pycno's volume}}. \quad (4)$$

Acid Number Test²⁴

The acid number test was carried out by making 100 mL 0.1 N KOH solution, followed by weighing 19–21 ± 0.05 grams of biodiesel sample into an Erlenmeyer flask. Subsequently, 100 mL of acetone solvent was added into a 250 mL Erlenmeyer flask, and 3 drops of PP indicator were added by pipette. The solution was titrated for 15 s until it became pink, and the acid number was determined using equation (5):

$$Acid\ number = \frac{56,1 \times \text{volume KOH} \times N\ KOH}{\text{sample weight}} \quad (5)$$

Analysis of Methyl Esters

The composition and concentration of methyl esters were determined using Shimadzu Gas Chromatography (GC).

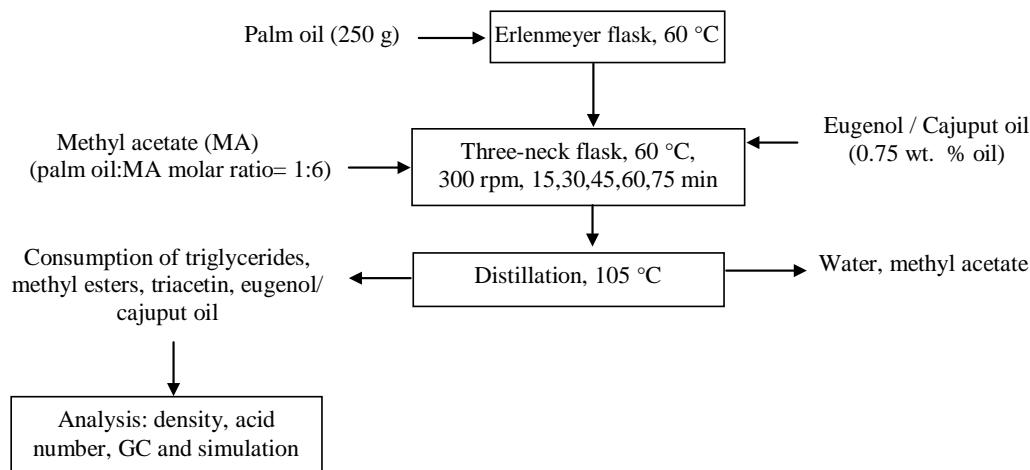


Fig. 1. Flow diagram process of palm oil interesterification with biocatalyst

2.2.4. Molecular Behavior Simulation with Software ChemDraw Professional 15.0

The molecular behavior (modeling) of the compounds participating in the interesterification reaction, namely triglycerides, methyl acetate, and biocatalyst, was studied by simulation using ChemDraw software. This software can also be used to calculate parameters related to the interactions of the compounds in the interesterification reaction. The known molecular behavior and parameters were C-C bond length before and after the addition of the biocatalyst.

3. Results and Discussion

3.1. Preparation of Materials

The analysis of palm oil raw materials showed 0.15 % FFA and 0.015 % moisture content, indicating its suitability for use in the interesterification process.

3.2. Interesterification Reaction with Essential Oil Biocatalyst

Fig. 2 shows that the optimum conditions were reached with a reaction time of 15 min using eugenol as a biocatalyst, obtaining the highest crude yield of 83.16 %. When using cajuput oil, a larger crude yield was obtained in a longer reaction time. The usage of cajuput oil did not allow us to achieve the optimal reaction time, where 75 min gave the highest crude yield of 65.88 %. The optimal results were achieved using eugenol after 15 min of the reaction. This success was attributed to the reversible nature of the interesterification process, where increasing the reaction time tended to shift the equilibrium toward the reactants^{9, 25, 26}. The limitations of a reversible

interesterification reaction became apparent when the reaction reached equilibrium. However, the use of a catalyst such as eugenol accelerated the equilibrium without changing its position. Crude yield achieved in the interesterification process with cajuput oil biocatalyst was relatively low due to the low content of 1,8-cineole compound, which was approximately 29 % in cajuput oil biocatalyst. The presence of this compound, serving as a magnetic field source to activate the reacting molecules, influences the interactions and affects product formation. Additionally, other impurities in cajuput oil affect product formation in the interesterification reaction.

In terms of economics, the interesterification reaction of palm oil with eugenol biocatalyst is the optimal condition due to its highest yield achieved within 15 min. Generally, reaction time is significantly essential in the process selection. The reason is that the shorter reaction time contributes to process efficiency and economic viability.

Table 1 shows that in the interesterification process using eugenol and cajuput oil biocatalysts, all achieved acid number values, including a maximum value of 0.5 mg KOH/g, comply with the SNI 7182:2015 standard. Moreover, smaller acid number values are desirable, showing fewer products transforming into FFA. This is attributed to the function of eugenol and cajuput oil as biocatalysts and antioxidants capable of preventing oxidation reactions to form FFA. In the interesterification process with eugenol and cajuput oil biocatalysts, all density values (from 0.85 to 0.89 g/mL) did not meet the SNI 7182:2015 standard. This is because the reaction product is a mixture of methyl ester, triacetin, residual triglycerides, and the biocatalyst. The average density for the four components in the reaction product was: triacetin = 1.16 g/mL, eugenol = 1.06 g/mL, triglycerides = 0.91 g/mL, and cajuput oil = 0.925 g/mL.

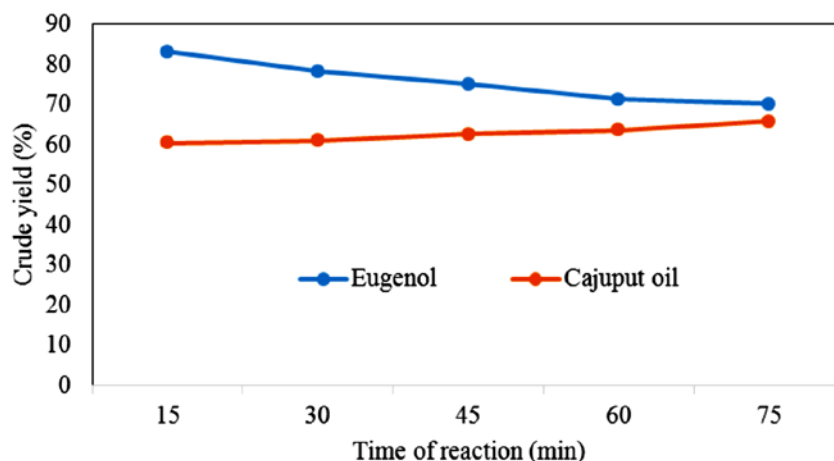


Fig. 2. The correlation between reaction time (min) to crude yield (%) with various types of biocatalyst

3.3. Result of Density Analysis and Acid Number

Table 1. Density and acid number data

Type of biocatalyst	Time of reaction, min	Density, g/mL	Acid number, mg KOH/g
Eugenol	15	0.892	0.337
	30	0.896	0.348
	45	0.896	0.359
	60	0.9	0.359
	75	0.904	0.370
Cajuput oil	15	0.938	0.381
	30	0.912	0.393
	45	0.91	0.404
	60	0.91	0.415
	75	0.908	0.426

3.4. Results of GC Analysis

Fig. 3 shows the chromatogram of the methyl ester produced during the optimal 15-minute reaction time at the interesterification of palm oil with eugenol biocatalyst.

The FAME composition from palm oil and methyl acetate interesterification reaction with eugenol biocatalyst is shown in Table 2. The catalyst weight was 0.75 % of the oil weight, stirring speed 300 rpm, reaction temperature 60 °C, and reaction time 15 min. In this research the largest methyl ester identified is methyl hexanoate, contrasting the transesterification process with a heterogeneous catalyst. Furthermore, the transesterification process of palm oil with a CaO catalyst showed that the largest methyl ester product is octadecanoic acid or methyl stearate (39.41 %) ²². In the process with a Ca/h-clay catalyst, the highest methyl ester products are methyl palmitate and elaidic acid methyl ester, while methyl stearate and elaidic acid methyl ester are obtained in the Cu/h-clay ²⁷.

3.5. Molecular Behaviour Simulation with ChemDraw Professional Software 15.0

Fig. 4 shows the molecular dynamics of triglycerides and methyl acetate after adding cineol biocatalyst. Meanwhile, Fig. 5 shows the molecular dynamics of triglycerides and methyl acetate after the addition of eugenol biocatalyst.

Table 3 shows that the addition of a cineol biocatalyst shortens the C-C bond in TG and methyl acetate. The initial length of the C-C bond is 1.509 Å; it changed to an average of 1.5175 Å. This phenomenon occurs due to the instability of TG and methyl acetate molecules caused by the cineol biocatalyst, which weakens the C-C bond. According to the mechanism of the interesterification process, a substitution reaction occurs when the C-C bond between TG and methyl acetate are broken. In this reaction, the R group on TG will replace the R group on methyl acetate, and *vice versa*.

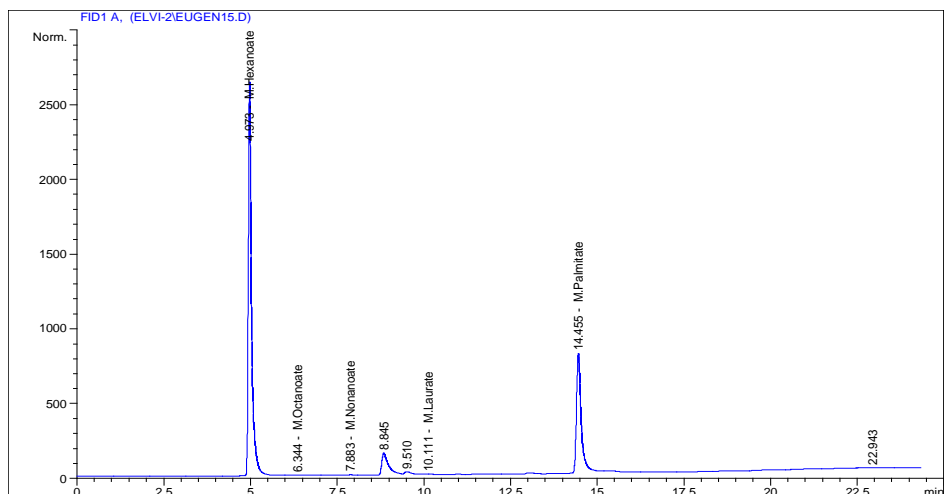


Fig. 3. Chromatogram of methyl ester with the addition of eugenol biocatalyst at a reaction time of 15 min

Table 2. FAME composition from palm oil and methyl acetate interesterification reaction with eugenol biocatalyst

FAME (<i>Fatty Acid Methyl Esters</i>)	Formula	Content, %
Methyl hexanoate	$C_7H_{14}O_2$	80.8744
Methyl octanoate	$C_9H_{18}O_2$	0.7171
Methyl nonanoic	$C_{10}H_{20}O_2$	1.0922
Methyl laurate	$C_{13}H_{26}O_2$	0.7244
Methyl palmitate	$C_{17}H_{34}O_2$	16.5927

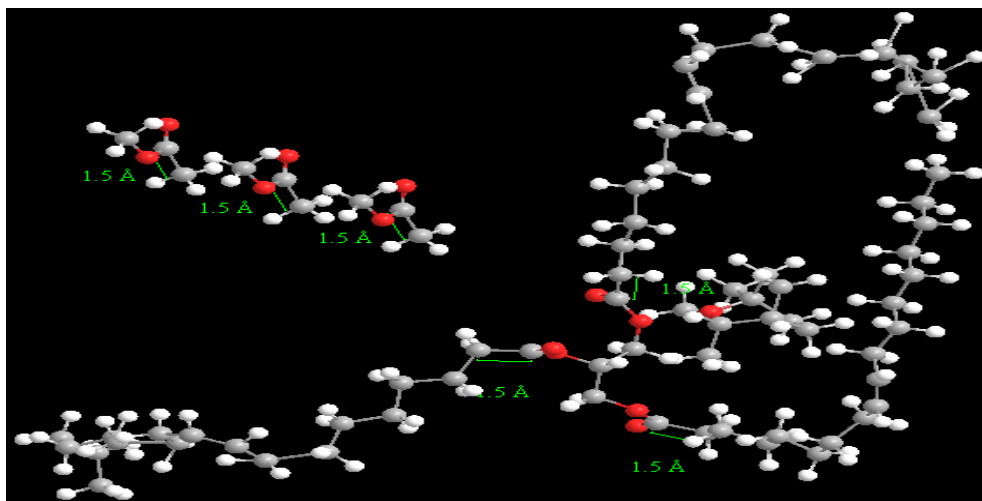


Fig. 4. Molecular dynamic of TG and methyl acetate after adding cineol (cajuput oil) biocatalyst

Table 3. C-C bond length before and after the addition of cineol (cajuput oil)

C-C bond	Actual final, Å	Optimale initial, Å
C(69) – C(70) → R of methyl acetate	1.518	1.509
C(8) – C(12) → R ₁ (palmitic acid)	1.520	1.509
C(7) – C(10) → R ₂ (oleic acid)	1.508	1.509
C(9) – C(14) → R ₃ (linoleic acid)	1.531	1.509
C(74) – C(75) → R of methyl acetate	1.510	1.509
C(79) – C(80) → R of methyl acetate	1.518	1.509
C-C bond length average		1.5175

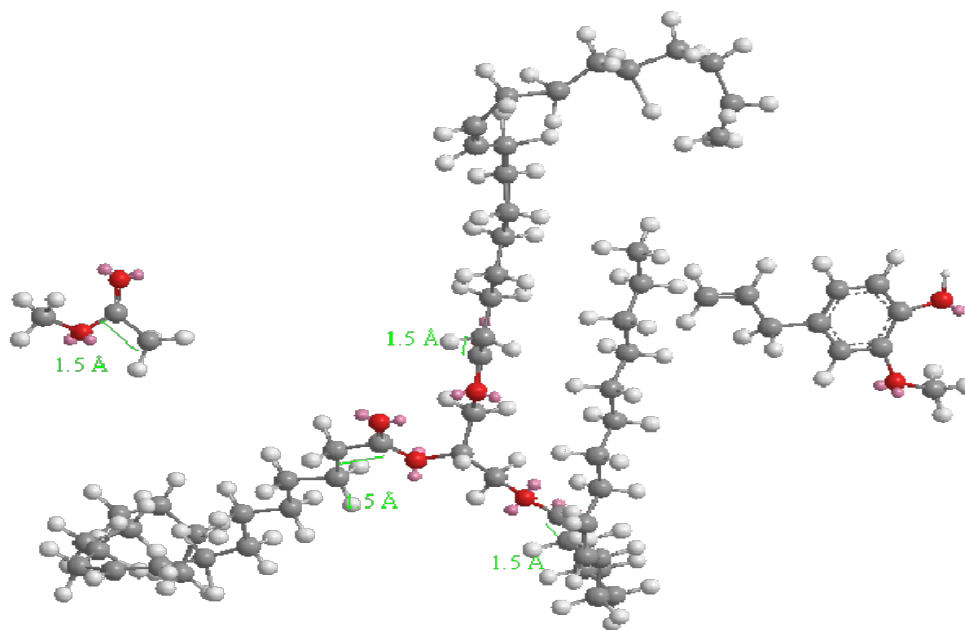


Fig. 5. Molecular dynamic of TG and methyl acetate after adding eugenol biocatalyst

Table 4. C-C bond length before and after the addition of eugenol

C-C bond	Actual final, Å	Optimale initial, Å
C(70) – C(71) → R of methyl acetate	1.783	1.509
C(8) – C(12) → R ₂ (oleic acid)	1.521	1.509
C(7) – C(10) → R ₁ (palmitic acid)	1.548	1.509
C(9) – C(14) → R ₃ (linoleic acid)	1.529	1.509
C(87) – C(88) → R of methyl acetate	1.830	1.509
C(92) – C(93) → R of methyl acetate	1.771	1.509
C-C bond length average		1.664

Table 4 shows that the C-C bond length in TG and methyl acetate are changed when the eugenol biocatalyst is added, increasing from 1.509 Å to an average of 1.664 Å. The eugenol biocatalyst causes the TG and methyl acetate molecules to become reactive, weakening the C-C bond. Furthermore, the average C-C bond length produced by the eugenol biocatalyst is larger than the cineol (cajuput oil). This shows that the eugenol biocatalyst caused the molecule to move more actively, making it easier to break than the cineol (cajuput oil).

Cajuput and clove oil contain aromatic compounds, namely 1,8-cineole and eugenol, which have aromatic rings. The localization of electrons in these rings generates a magnetic field^{18, 28}, which is attracted by triglycerides and methyl acetate, which have paramagnetic C=O bonds. As the C=O bond is pulled through the magnetic field, the distance between all the atomic bonds in the triglyceride and methyl acetate becomes longer. During this process, the C-C bond with the lowest binding energy is broken first, after which a group exchange reaction occurs in triglycerides and methyl acetate to form a reaction product.

4. Conclusions

1. This study showed that the eugenol biocatalyst was more effective in the palm oil interesterification process compared to cajuput oil.

2. A crude yield of 83.16 % was obtained under optimum conditions: the eugenol biocatalyst 0.75 wt. %, a palm oil: methyl acetate molar ratio 1:6, reaction temperature 60 °C, reaction time 15 min.

3. Under optimum conditions a density of 0.892 g/mL and acid number of 0.3 mg KOH/g were achieved, which meet the SNI 7182:2015 biodiesel standard.

4. Based on the simulation performed using the ChemDraw program, the use of eugenol biocatalyst caused the movement of the most reactive triglyceride and methyl acetate molecules. Furthermore, the average C-C bond length of 1.664 Å was greater than the cineol (cajuput oil).

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ВИРОБНИЦТВО БІОДИЗЕЛЯ БЕЗ КАТАЛІТИЧНОЇ СЕПАРАЦІЇ ЗА ДОПОМОГОЮ ПРОЦЕСУ ІНТЕРЕСТЕРИФІКАЦІЇ ПАЛЬМОВОЇ ОЛІЇ З ВИКОРИСТАННЯМ БІОКАТАЛІЗАТОРА З ЕФІРНОЇ ОЛІЇ

Анотація. Трансестерифікація – це метод, який широко використовують у виробництві біодизеля. Мета цієї роботи – дослідити реакцію інтерестерифікації пальмової олії метилацетатом із використанням біокатализатора з ефірної олії, зокрема, евгенолу та кадзупутувої олії. Робочі параметри були такими: 250 г пальмової олії, мольне співвідношення пальмова олія : метилацетат 1:6, температура реакції 60 °C, швидкість перемішування 300 об./хв, біокатализатор 0,75 % мас. щодо олії, час реакції 15, 30, 45, 60 і 75 хв. Пальмова олія, метилацетат і біокатализатор реагували в тригорлій колбі як реакторі в робочих умовах. Додатково виконано моделювання з використанням ChemDraw Professional 15.0 для аналізу молекулярної поведінки сполук, що реагують.

Ключові слова: інтерестерифікація, ефірна олія, евгенол, вихід сирової речовини, довжина зв'язку.