

Research Paper

Synthesis of 2-Hidroxyethyl Ester (2-HEE) and 2-Hydroxypropyl Ester (2-HPE) from Castor Oil as Bioadditives to Improve the Cold Flow Characteristic of Biodiesel

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^{doi} <https://doi.org/10.31603/ae.15860>

Published by Automotive Laboratory of Universitas Muhammadiyah Magelang

Article Info

Submitted:

07/01/2026

Revised:

11/04/2026

Accepted:

14/05/2026

Online first:

14/05/2026

Abstract

The utilization of biodiesel continues to increase along with the development of renewable energy. However, biodiesel tends to exhibit poorer cold flow properties compared to petroleum diesel. In this study, castor oil, a non-edible vegetable oil, was modified into 2-hydroxyethyl ester (2-HEE) and 2-hydroxypropyl ester (2-HPE) to improve the cold flow characteristic of biodiesel. The transesterification of 2-HEE and 2-HPE was carried out using fatty acid methyl esters (FAME) derived from castor oil with ethylene glycol (for 2-HEE) and propylene glycol (for 2-HPE), assisted by a K_2CO_3 catalyst at 150 °C, a mixing speed at 500 rpm, under a vacuum system. The optimum molar ratio of FAME to polyol was 1:10, yielding 69.63% for 2-HEE and 56.84% for 2-HPE. GC-MS analysis showed product abundances of 98.17% for 2-HEE (dominated by 2-hydroxyethyl ricinoleate at 77.4%) and 98.97% for 2-HPE (dominated by 2-hydroxypropyl ricinoleate at 77.9%). The addition of 2% v/v 2-HEE to biodiesel reduced the cloud point by 2.2 °C, the pour point by 3 °C, the flash point by 3 °C, and the density by 0.005 g/cm³, while increasing the kinematic viscosity by 0.21 cSt. Meanwhile, the addition of 2% v/v 2-HPE reduced the cloud point by 3.1 °C, the pour point by 4.3 °C, the flash point by 8 °C, and the density by 0.001 g/cm³, with an increase in kinematic viscosity of only 0.01 cSt. The 2-HPE compound showed superior performance as a bioadditive compared to 2-HEE in improving the cold flow characteristic of biodiesel without significantly altering its physical properties.

Keywords: Bioadditives; Biodiesel; Castor Oil; Cold flow

1. Introduction

Biodiesel is utilized as fuel due to concerns over the decrease of petroleum diesel fuels and the increasing carbon emissions, as it produces fewer air pollutants and greenhouse gases, is biodegradable, non-toxic, sulfur-free, and offers safer handling characteristics [1]. In general, the producing of biodiesel with transesterification reaction of vegie or animal oils with alcohols such as methanol [2]. Biodiesel quality standards are regulated by ASTM D6751 (American standard) and EN 14214 (European standard). One of the main weakness of biodiesel comparing with

petroleum diesel is its cold flow properties, particularly the cloud point and pour point parameters.

One approach to improving the cold flow characteristic of biodiesel is through the addition of bioadditives in minimal amounts [3]. Fatty acid-derived bioadditives are obtained by modifying their carboxylate groups. 5% v/v isopropyl ester of palm oil added into biodiesel can reduce the cloud point and pour point of biodiesel by 5 °C and 4 °C, respectively, while 5% v/v 2-butyl ester of palm oil can reduce the cloud point and pour point by 6 °C each [4]. In addition, more braches of alcohol compounds, can more



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reduce the cloud point of biodiesel, thereby improving its quality [5]. The addition of 10% w/w tert-butanol and 2-buthanol to biodiesel can reduce the cloud point by 5 °C and 3 °C [5].

Fatty acids in vegetable oils are generally present in triglycerides form, which are esters formed between glycerol and three fatty acids and constitute the main components of plant oils [6]. One vegetable oil that contains distinctive fatty acids is castor oil. Castor oil is a non-edible vegetable oil derived from the seeds of the castor plant, scientifically known as *Ricinus communis*. Castor oil has unique characteristics compared with other oils due to its relatively high content of ricinoleate groups compared to other fatty acids [7]. The presence of a hydroxyl group at C12 and a double bond in the ricinoleate structure enables the synthesis of various industrially valuable derivatives [8].

The modification of fatty acids from castor oil into bioadditives in the form of 2-hydroxyethyl ester (2-HEE) has been reported the lubricity enhancer of diesel fuel that has low-sulfur [9]. The synthesis of 2-hydroxypropyl ester (2-HPE) from waste cooking oil has also shown potential as a lubricity enhancer for diesel that has low-sulfur [10]. However, previous studies have not reported the application of 2-HEE and 2-HPE in improving cold flow properties. Therefore, in this study, 2-hydroxyethyl ester (2-HEE) and 2-hydroxypropyl ester (2-HPE) were synthesized as fatty acid-derived bioadditives through transesterification reaction with two step. Step one, castor oil with methanol to produce fatty acid methyl esters (FAME). Step 2, FAME was transesterified with ethylene glycol to form 2-HEE and with propylene glycol to form 2-HPE. This study also evaluated the effects of both bioadditives on the cold flow characteristic of biodiesel, represented by the cloud point and pour point parameters, as well as on its physical properties, including flash point, density, and kinematic viscosity.

2. Methods

2.1. Materials

Castor oil (commercial grade), B100 biosolar (obtained from a biodiesel factory in East Java,

Indonesia), ethylene glycol (Supelco), propylene glycol (Supelco), potassium hydroxide ($\geq 99.0\%$; Merck), potassium carbonate (Supelco), Methanol ($\geq 99.0\%$; Merck), anhydrous Na_2SO_4 (Merck), ethyl acetate ($\geq 99.0\%$; Merck), *n*-hexane ($\geq 99.0\%$; Merck), diethyl ether ($\geq 99.0\%$; Sigma-Aldrich), phenolphthalein (PP) indicator, distilled water, universal indicator, TLC silica gel 60 F_{254} plates, and Whatman No. 42 filter paper were also utilized.

2.2. The Determination of the Acid Value of Castor Oil

The determination of acid value using a method adapted from Suzehaque *et al.* [11]. Castor oil was dissolved in mixture of isopropanol and phenolphthalein (PP). The solution was titrated with 0.1 N potassium hydroxide until reach afaint pink color. The volume of KOH consumed during titration was recorded, and the acid value was calculated using Equation (1). FAME synthesis can be carried out when castor oil contains free fatty acids (FFA) of less than 2–2.5% or an acid value below 4–5 mg KOH/g [11].

2.3. Synthesis of Fatty Acid Methyl Esters (FAME)

The synthesis of FAME was carried out through a transesterification reaction adapted from the study by Zetra *et al.* [12]. Castor oil was heated, KOH (1% w/w of castor oil) was dissolved in methanol at 1:6 (oil:methanol) molar ratio. Once the castor oil reached a temperature of 60 °C, the solution of KOH and methanol was added. The reaction carried out at 60 °C and 500 rpm. The completion of the reaction was determined by thin-layer chromatography (TLC) using an *n*-hexane:diethyl ether:acetic acid (16:6:1) eluent system [13]. The product was purified following the method of Suzihaque *et al.* [11]. Themixture was left 24 hours at separatory funnel at room temperature. Two layers formed, the top layer was collected and washed with distilled water until neutral. 10 g of anhydrous sodium sulfate (Na_2SO_4) was added to absorb any remaining water. The product composition was analyzed using gas chromatography–mass spectrometry (GC–MS).

$$\text{Acid Value} = \frac{V_{\text{titrant}} \times M_{\text{titrant}} \times Mr_{\text{KOH}}}{\text{Sampel Weight}} \quad (\text{mgKOH/g}) \quad (1)$$

2.4. Synthesis of the Bioadditives 2-Hydroxyethyl Ester (2-HEE) and 2-Hydroxypropyl Ester (2-HPE)

The synthesis of the 2-hydroxyethyl ester (2-HEE) and 2-hydroxypropyl ester as bioadditive was adapted from the study by Firmansyah *et al.* with substrate modification [14], as illustrated in Figure 1 and Figure 2. FAME was heated to 150 °C, followed by the addition of polyol. The polyols are ethylene glycol (EG) to make 2-HEE and propylene glycol (PG) to make 2-HPE. and FAME with varying FAME:polyol molar ratios (1:4, 1:6, 1:8, and 1:10) and K₂CO₃ catalyst (7% w/w of FAME). The synthesis was carried out under vacuum conditions with stirring at 500 rpm. The reaction time was monitored using thin-layer chromatography (TLC) with an *n*-hexane:diethyl ether:acetic acid (16:6:1) eluent system [13]. Excess catalyst remaining in the reaction product was removed using a method adapted from Zetra *et al.* [12]. Subsequently, concentrated hydrochloric acid (HCl) was added until neutral and added ethyl acetate to extract the product. The obtained organic phase was washed with distilled water, then added anhydrous sodium sulfate (Na₂SO₄). After filtration, the ethyl acetate was evaporated with vacuum rotary evaporator at 60 °C.

2.5. Component Identification and Analysis

The compositions of FAME, 2-HEE, and 2-HPE were identified using gas chromatography–mass

spectrometry (GC–MS) with an Agilent GCMSD 5975C equipped with an HP-5MS capillary column (5% phenyl methylpolysiloxane) with dimensions of 60 m × 250 μm × 0.33 μm, using helium (He) as the carrier gas. The oven temperature program was started at 70 °C and held for 5 minutes, then the temperature was increased to 300 °C at a rate of 5 °C/min and maintained for 19 minutes. The GC–MS data were analyzed using Agilent MSDA software and compound identification was performed by comparison with the Wiley 9 mass spectral library.

2.6. Evaluation of Bioadditives in Biodiesel

The synthesized 2-HEE and 2-HPE compounds were each added to biodiesel (B100) to evaluate its potential as bioadditives for improving the cold flow characteristic of biodiesel. The 2-HEE and 2-HPE were individually blended into biodiesel at concentrations of 0.5%, 1.0%, 1.5%, 2.0%, and 2.5% to investigate their effects on cold flow improvement, as indicated by the cloud point and pour point parameters. Furthermore, the concentrations of 2-HEE and 2-HPE that provided the best improvement in cold flow properties were further analyzed for physical properties, including flash point, density, and kinematic viscosity at 40 °C in accordance with ASTM D92, ASTM D4052 (pycnometer), and ASTM D445 methods.

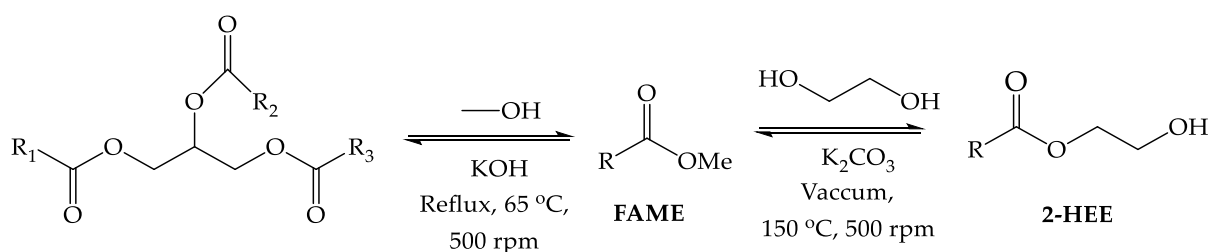


Figure 1. Synthesis pathway of 2-hydroxyethyl ester (2-HEE)

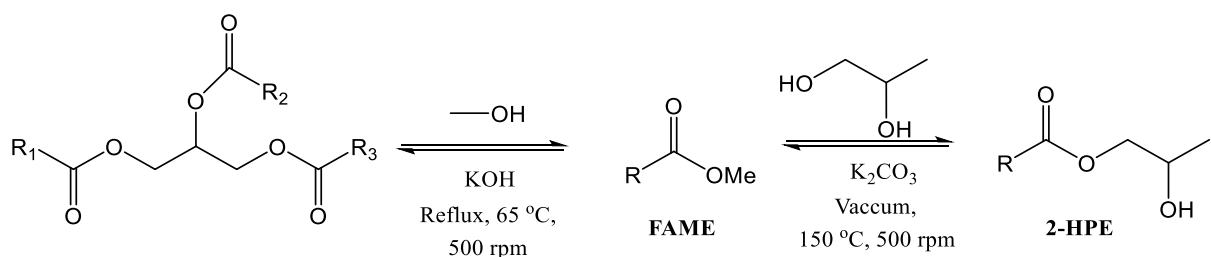


Figure 2. Synthesis pathway of 2-hydroxypropyl ester (2-HPE)

3. Results and Discussion

3.1. The Acid Value of Castor Oil

The acid value of castor oil was calculated using Eq. (1), and its comparison with results from other studies is presented in Table 1. The average acid value of the castor oil that used in this study is 1.06 mg KOH/g. The acid value content must be determined prior to transesterification because it can affect the efficiency and yield of the transesterification process [15]. A high acid value can inhibit base-catalyzed transesterification, as it tends to promote soap formation due to saponification reactions between free fatty acids and strong bases [16]. The free fatty acid content in oils or fats should not exceed 2–2.5%, as higher levels can cause difficulties at transesterification reaction [11]. The average acid value obtained is below the maximum allowable limit, indicating that the castor oil used is suitable for bioadditive synthesis.

3.2. Analysis of Fatty Acid Methyl Ester Composition (FAME)

The yield of FAME from transesterification of castor oil is 87.47%. Composition of FAME was

analyzed using TLC and GC–MS. The time of reaction done at 120 minutes, and the TLC results can be seen in Figure 3. At 120 minutes, product spots were observed, while the spots corresponding to the initial material, castor oil, were no longer visible. These results are consistent with the study by Ramezani *et al.* which reported that 120 minutes were required for the base-catalyzed transesterification of castor oil at 60 °C [17].

The total ion chromatogram (TIC) of FAME is shown in Figure 4. The chromatographic peaks were identified based on their mass spectral (MS) patterns, characterized by a base peak at m/z 74, which is typical of methyl ester fragments resulting from McLafferty rearrangement fragmentation [18].

Table 1. Acid value of castor oil

No.	Acid Value (mgKOH/g)	References
1.	1.06	This Study
2.	1.20	[16]
3.	0.57	[19]
4.	15.57	[20]
5.	1.09	[21]
6.	0.79	[22]

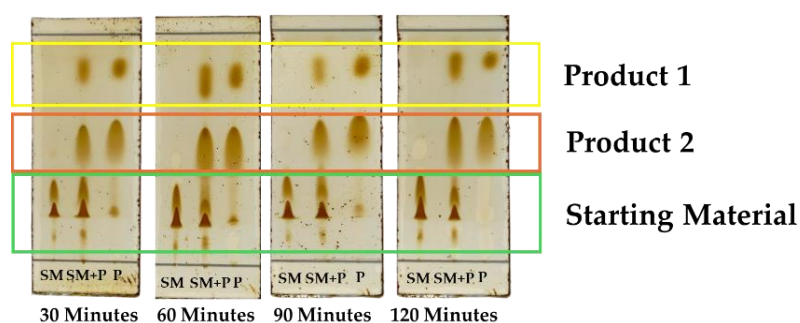


Figure 3. TLC spot (SM = Starting Materials, P = Products)

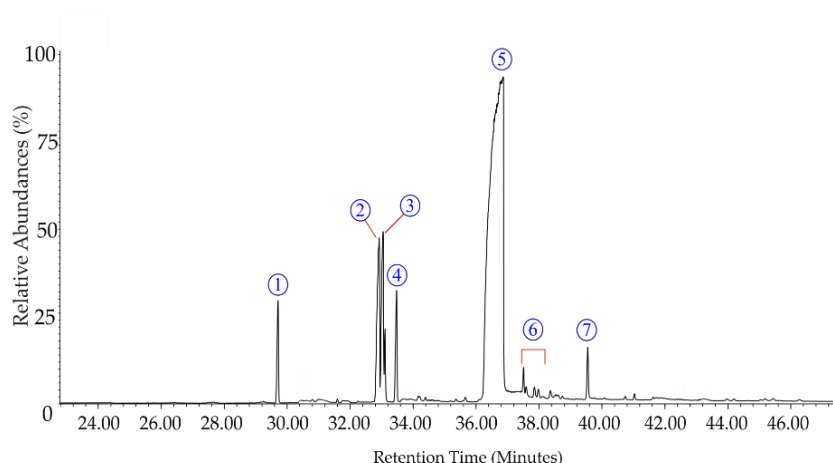


Figure 4. Chromatogram of castor oil FAME

Based on the chromatographic identification shown in Figure 5, methyl ricinoleate was found to dominate the composition with an abundance of 75.63%. In addition to undergoing the McLafferty rearrangement, methyl ricinoleate also experiences dehydration due to the presence of a hydroxyl group, resulting in the formation of an (M-18) fragment at m/z 294. Dehydration fragmentation is a common process that occurs in molecules containing hydroxyl ($-OH$) groups. This pathway produces fragment ions that are 18

mass units lower (M-18) than the parent molecular ion due to the loss of a water molecule (H_2O) [23]. The mass spectrum of methyl ricinoleate can be seen in Figure 5a, while the McLafferty rearrangement and dehydration pathways are shown in Figure 5b.

Other methyl ester components are listed in Table 2. The presence of a cyclic ketone was also identified, with a molecular ion peak at m/z 280 and an abundance of 2.08%. The formation of cyclic ketones during the transesterification of

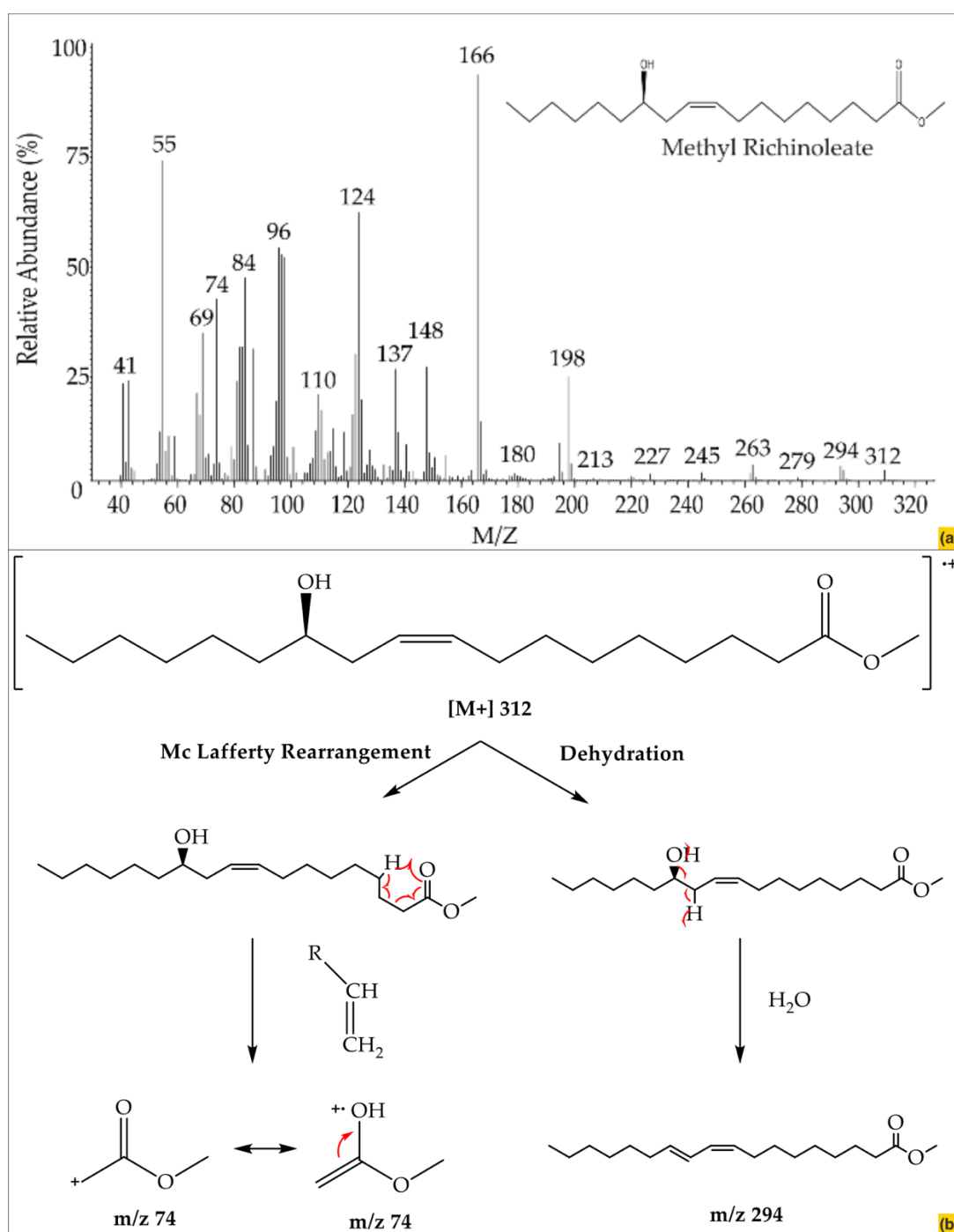


Figure 5. (a) Mass spectrum; (b) McLafferty rearrangement and dehydration of methyl ricinoleate

Table 2. Identification of methyl ester composition in FAME from castor oil

Peak	Retention Time (minutes)	Compound	Molecular Formula	Molecular Weight	Relative Abundance (%)
1	29.718	Methyl Palmitate	C ₁₇ H ₃₄ O ₂	270	3.12
2	32.933	Methyl Linoleate	C ₁₉ H ₃₄ O ₂	294	7.34
3	33.057	Methyl Oleate	C ₁₉ H ₃₆ O ₂	296	7.58
4	33.489	Methyl Stearate	C ₁₉ H ₃₈ O ₂	298	2.94
5	36.875	Methyl Ricinoleate	C ₁₉ H ₃₆ O ₃	312	75.63
6	37.531	Cyclic Ketone	N/A	280	2.08
7	39.571	Methyl Arachidate	C ₂₁ H ₄₂ O ₂	326	1.26

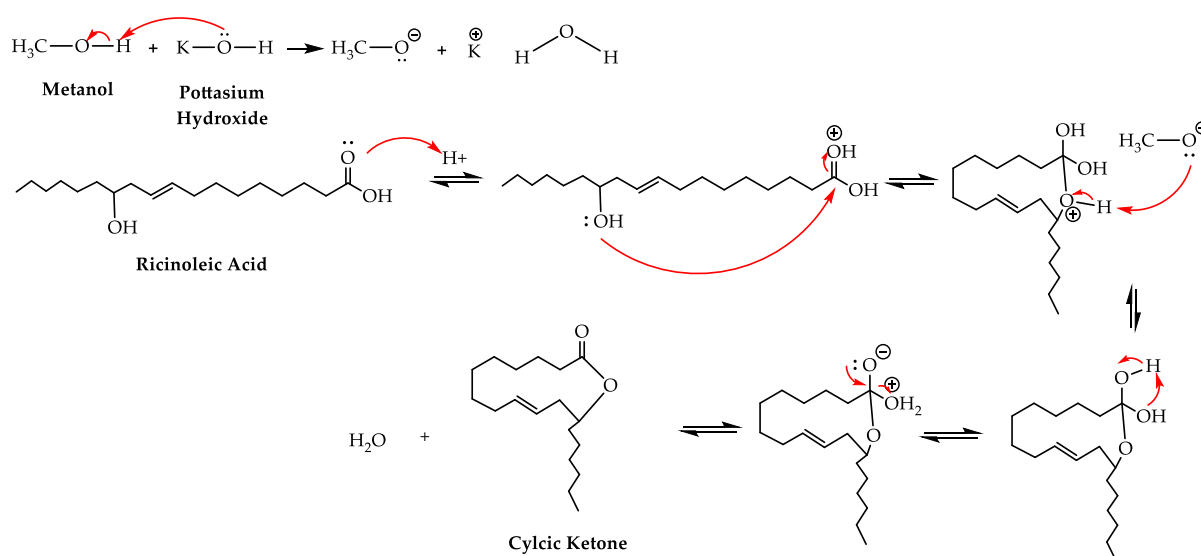
castor oil occurs due to intramolecular esterification of ricinoleic acid through nucleophilic addition to the carbonyl group [14]. KOH, as a strong base, deprotonates methanol to form potassium methoxide by abstracting a proton from methanol. Ricinoleic acid in castor oil captures this proton, causing the carbonyl carbon of ricinoleic acid to become electrophilic. Consequently, the hydroxyl group of ricinoleate attacks the carbonyl group, leading to cyclization and the formation of a cyclic ketone. The proposed mechanism for cyclic ketone formation was adapted from the study by Firmansyah *et al.* [14], as illustrated in Figure 6.

3.3. Composition Analysis of 2-HEE and 2-HPE

Synthesis was carried out using an excess of reactant with FAME-to-reactant molar ratios of 1:4, 1:6, 1:8, and 1:10. K₂CO₃ was first dissolved in each reactant prior to mixing with FAME to allow potassium carbonate to deprotonate the reactant in advance. The resulting deprotonated species acts as a nucleophile that attacks the α -carbon of FAME. The proposed reaction mechanisms for the

formation of 2-HEE and 2-HPE are shown in Figure 7.

The reaction times for the synthesis of 2-HEE and 2-HPE are presented in the graph shown in Figure 8. The stoichiometric ratio of 1:10 (FAME:reactant) resulted in the fastest synthesis times, requiring 4 hours for 2-HEE and 8 hours for 2-HPE. The reaction time depends on the stoichiometric ratio between FAME and the reactant, as well as the type of reactant used. The time required for the synthesis of both 2-HEE and 2-HPE decreased as the FAME-to-reactant ratio increased. Reaction rates increase with increasing reactant concentration [24]. Meanwhile, at the same concentration, the synthesis of 2-HEE proceeded faster than that of 2-HPE. Unlike ethylene glycol, propylene glycol contains a methyl group that introduces steric hindrance around one of its hydroxyl groups. Steric interference occurs when the spatial arrangement of atoms or groups surrounding or near the reaction site obstructs or slows down the reaction [25]. In organic molecules that can act as nucleophiles at multiple positions (e.g., polyols),

**Figure 6.** Proposed mechanism formation of cyclic ketone

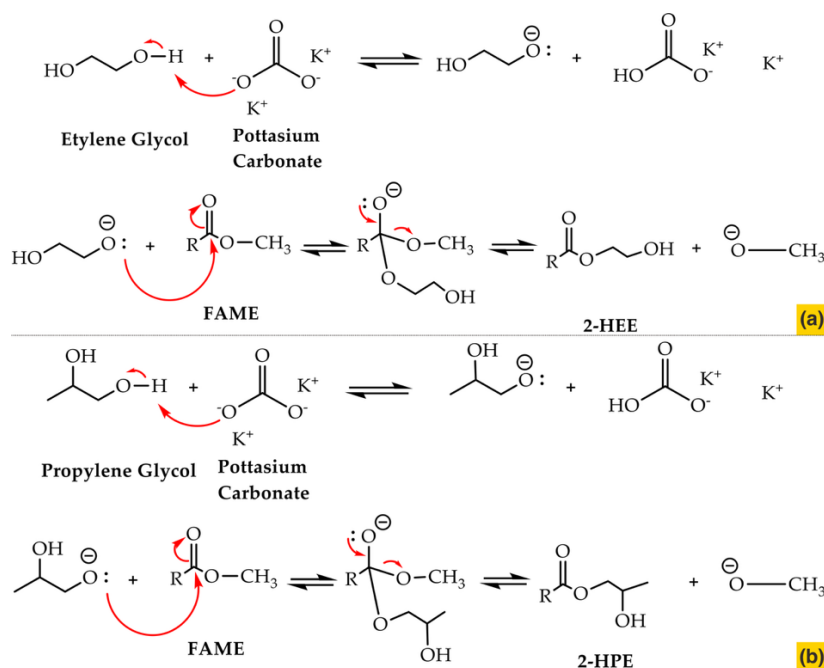


Figure 7. Mechanism reaction of (a) 2-HEE and (b) 2-HPE

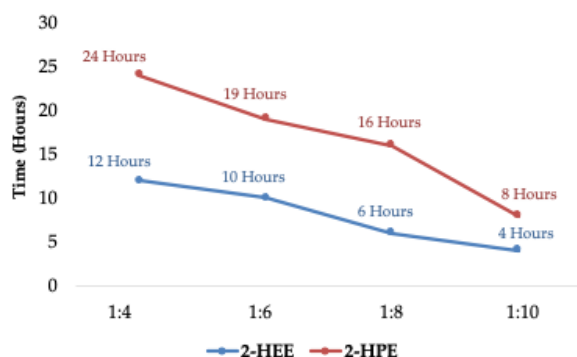


Figure 8. Graph of the effect of the FAME : reactant stoichiometric ratio on the reaction time for 2-HEE and 2-HPE

the presence of more nucleophilic sites increases the probability of effective collisions between reactants, thereby enhancing the reaction rate under identical conditions [26].

The yields of 2-HEE and 2-HPE at each stoichiometric ratio are shown in the graph in Figure 9. The highest yields were obtained at a 1:10 (FAME:reactant) ratio, with yields of 69.63% for 2-HEE and 56.84% for 2-HPE. Increasing the amount of reactant led to higher yields. For both products, 2-HEE and 2-HPE, the highest yields were achieved at the 1:10 (FAME:reactant) ratio. According to principle of Le Chatelier, increasing the concentration of a reactant moves the equilibrium toward the product, resulting in a greater amount of product formation [27]. In the case of 2-HEE, the yield increases significantly as

the stoichiometric ratio is increased. This phenomenon occurs because a higher amount of residual ethylene glycol increases the polarity of the aqueous phase (formed during the neutralization of potassium carbonate with hydrochloric acid), thereby reducing emulsion formation and accelerating phase separation.

3.4. Analysis Composition of 2-HEE and 2-HPE

The synthesis reactions of 2-HEE and 2-HPE were monitored using the TLC method. The fastest reaction times and the best yields were obtained at a stoichiometric ratio of 1:10, as shown in Figure 10.

The 2-HEE and 2-HPE products were analyzed using GC-MS, and the resulting total ion chromatograms (TICs) are presented in Figure 11.

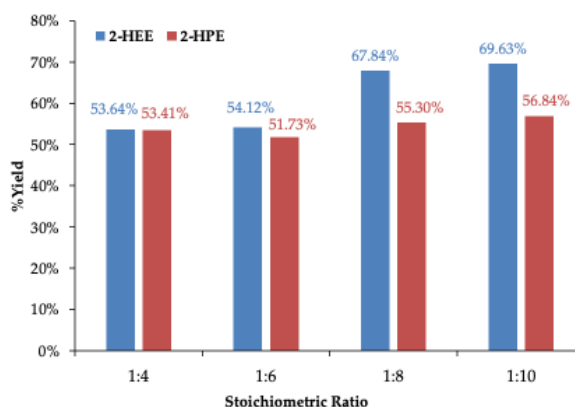


Figure 9. Graph of the effect of the FAME : reactant stoichiometric ratio on the yield of 2-HEE and 2-HPE

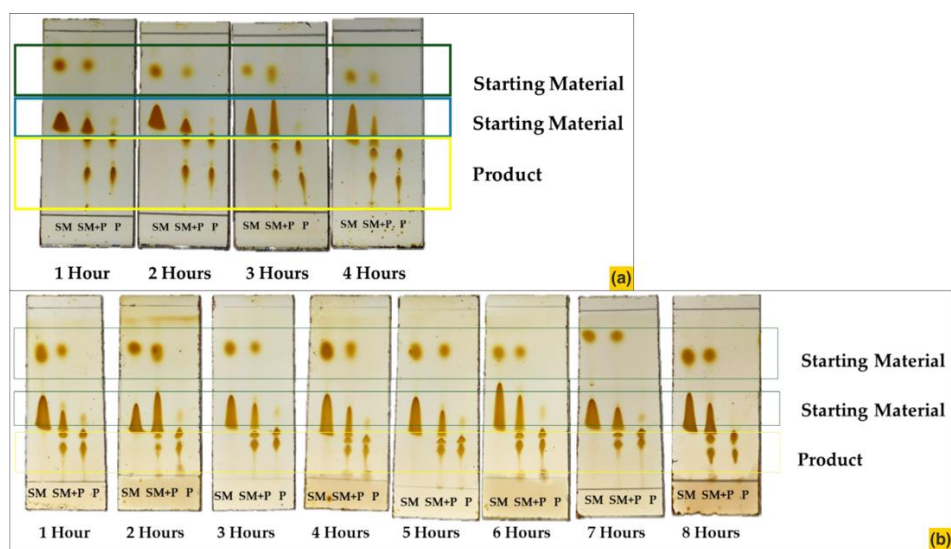


Figure 10. TLC results of (a) 2-HEE and (b) 2-HPE at 1:10 stoichiometric ratio

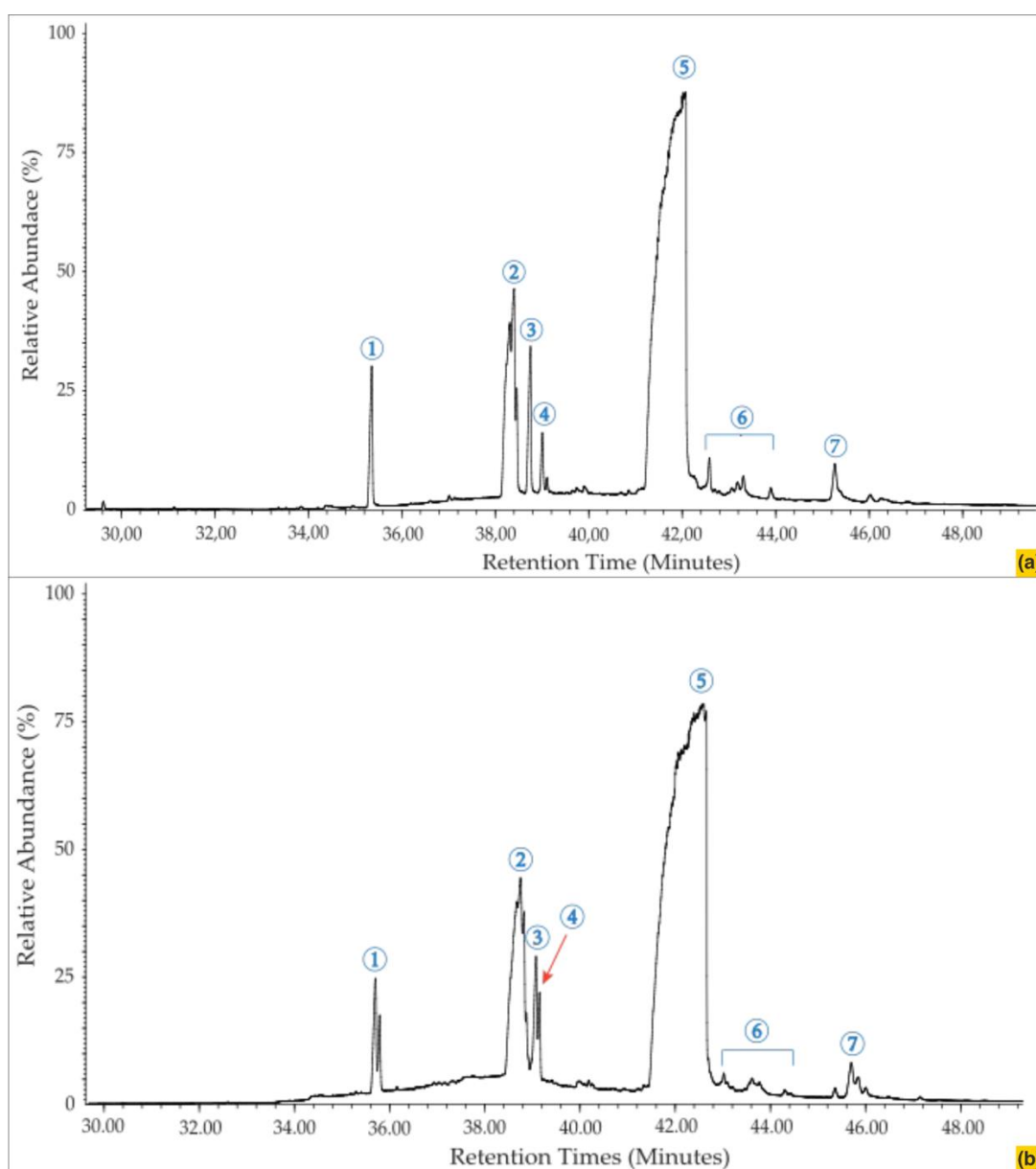


Figure 11. Chromatogram result of (a) 2-HEE and (b) 2-HPE

The GC–MS results indicate that 2-hydroxyethyl ricinoleate was the dominant component with a total abundance of 77.4% (Figure 11a), while Figure 11b shows that 2-hydroxypropyl ricinoleate was the dominant component with an abundance of 77.9%.

The identification of 2-hydroxyethyl ester compounds in the 2-HEE bioadditive was based on the existence of a characteristic molecular ion peak at m/z 104 [14]. The m/z 104 fragment in 2-HEE is attributed to McLafferty rearrangement [12]. Similar to methyl ricinoleate, 2-hydroxyethyl

ricinoleate, which has the highest abundance of 77.4%, contains a hydroxyl group that leads to the formation of a fragment at m/z 324 due to dehydration. The mass spectrum of 2-hydroxyethyl ricinoleate can be seen in Figure 12a, while the McLafferty rearrangement and dehydration pathways are shown in Figure 12b.

The identification of 2-hydroxypropyl ester compounds in the 2-HPE bioadditive was based on the presence of a characteristic molecular ion peak at m/z 118 [28]. 2-Hydroxypropyl ricinoleate, with the highest abundance of 77.9%, undergoes

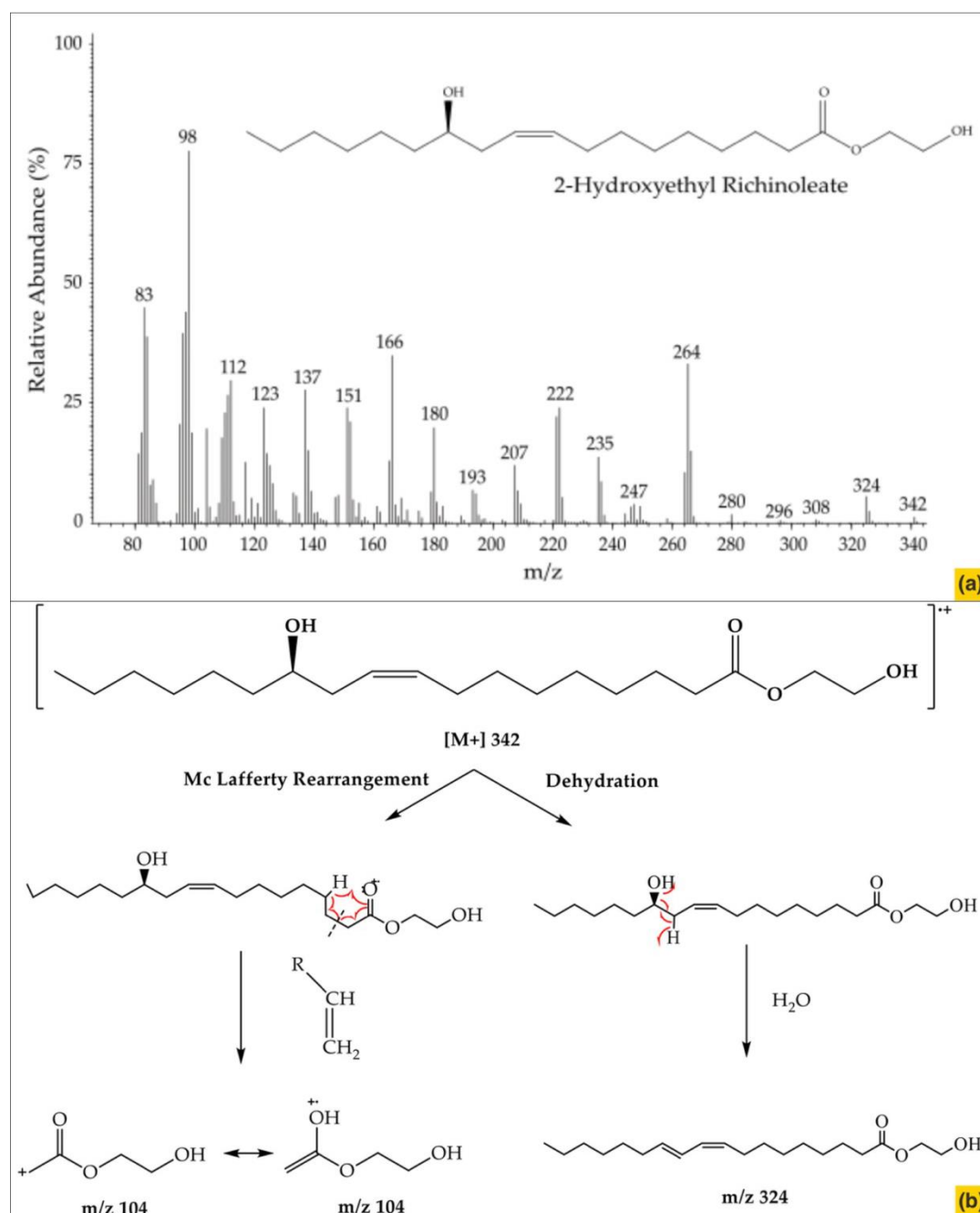


Figure 12. (a) Mass spectrum; (b) McLafferty rearrangement and dehydration of 2-hydroxyethyl ricinoleate

McLafferty rearrangement and dehydration, similar to methyl ricinoleate and 2-hydroxyethyl ricinoleate. Dehydration in 2-hydroxypropyl ricinoleate is initiated by the loss of a methyl branch, forming the $[M-15]^+$ peak at m/z 341. This fragment then undergoes dehydration, resulting in a peak at m/z 323. The mass spectrum of 2-hydroxypropyl ricinoleate can be seen in Figure 13a, while the McLafferty rearrangement and dehydration pathways are shown in Figure 13b.

3.5. Effect of 2-HEE and 2-HPE Bioadditives on Biodiesel (B100)

3.5.1. The characteristic of cold flow

The characteristic of cold flow used in this study were the cloud point (CP) and the pour point (PP). CP refers to the condition at which biodiesel start forming wax crystals. The wax crystals can make fuels become cloudy and indicating the onset of solidification. If the temperature keeps dropping, the PP is reached,

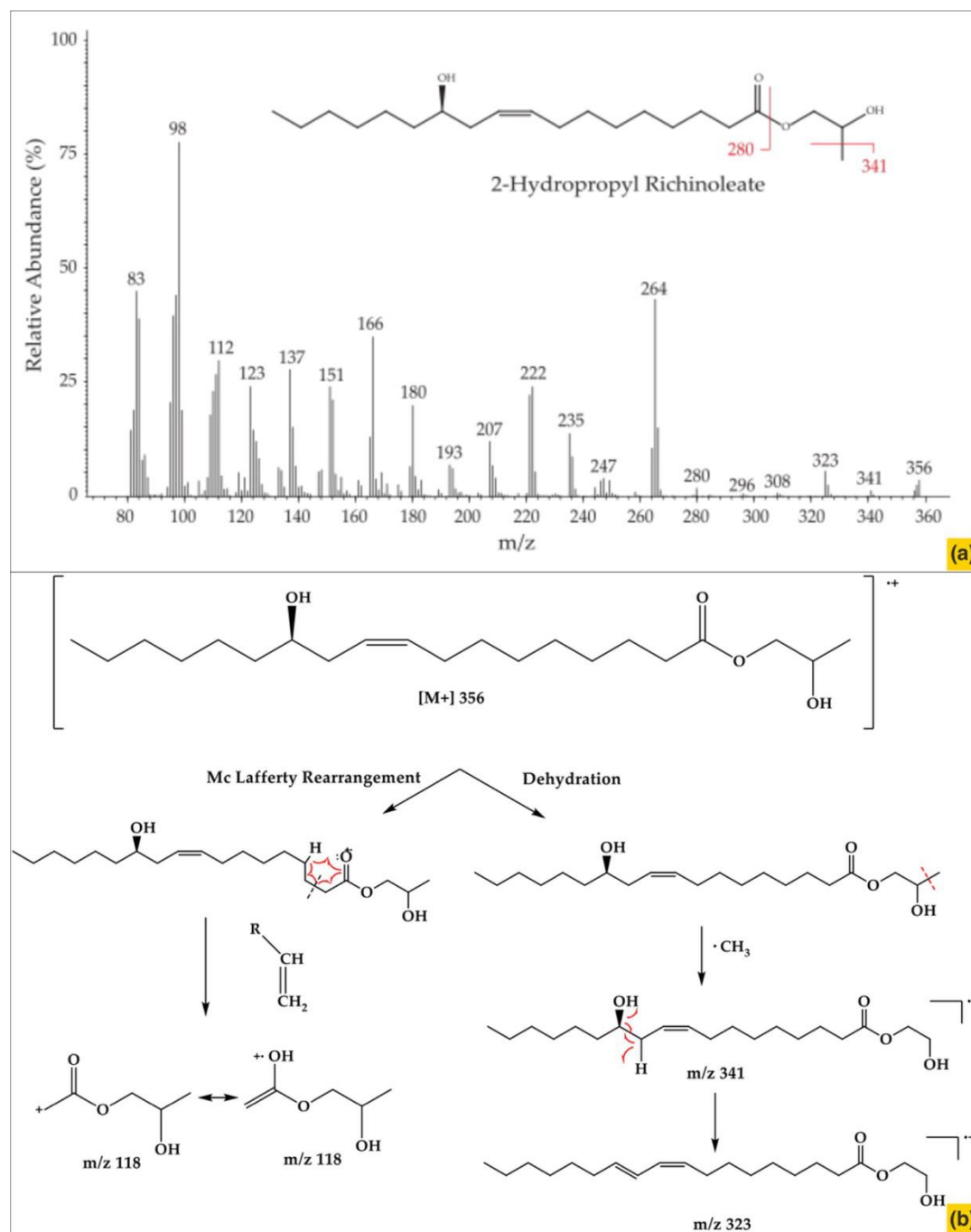


Figure 13. (a) Mass spectrum, (b) McLafferty rearrangement and dehydration of 2-hydroxypropyl ricinoleate

Table 3. Identification of 2-hydroxyethyl ester from **Figure 11a**

Peak	Retention Time (Minutes)	Compound	Molecular Formula	Molecular Weight	Relative Abundance (%)
1	35.358	2-Hidroxyethyl Palmitate	C ₁₈ H ₃₄ O ₃	300	2.91
2	38.314	2-Hidroxyethyl Linoleate	C ₂₀ H ₃₆ O ₃	324	7.27
3	38.403	2-Hidroxyethyl Oleate	C ₂₀ H ₃₈ O ₃	326	6.29
4	38.754	2-Hidroxyethyl Stearate	C ₂₀ H ₄₀ O ₃	328	3.00
5	41.530	2-Hidroxyethyl Ricinoleate	C ₂₀ H ₃₈ O ₄	342	77.4
6	42.582	Cyclic Ketone	N/A	280	1.75
7	45.264	2-Hidroxyethyl Arachidate	C ₂₂ H ₄₄ O ₃	356	1.30

Table 4. Identification of 2-hydroxypropyl ester from **Figure 11b**

Peak	Retention Time (Minutes)	Compound	Molecular Formula	Molecular Weight	Relative Abundance (%)
1	35.705	2-Hidroxypropyl Palmitate	C ₁₉ H ₃₈ O ₃	314	2.92
2	38.676	2-Hidroxypropyl Linoleate	C ₂₁ H ₃₈ O ₃	338	12.8
3	39.078	2-Hidroxypropyl Oleate	C ₂₁ H ₄₀ O ₃	340	2.14
4	39.151	2-Hidroxypropyl Stearate	C ₂₁ H ₄₂ O ₃	342	2.06
5	42.033	2-Hidroxypropyl Ricinoleate	C ₂₁ H ₄₀ O ₄	356	77.90
6	43.02	Cyclic Ketone	N/A	280	1.00
7	45.698	2-Hidroxypropyl Arachidate	C ₂₃ H ₄₆ O ₃	370	1.15

which is the lowest temperature at which biodiesel can be poured. Under this temperature, the fuel becomes too thick to be poured or pumped [29].

Biodiesel tends to have poor flow due to its chemical structure as fatty acid methyl esters (FAME). Pure biodiesel used in this study were obtained from Indonesian biodiesel producers whose product has been certified to meet the SNI 7182:2015 standard. The chemical composition of biodiesel from palm oil is saturated FAME dominated by methyl palmitate. In detail, the FAME composition is as follows: methyl palmitate 41.72–56.26%; methyl oleate 29.93–42.62%; methyl linoleate 6.38–9.93%; methyl stearate 4.71–5.92%; methyl myristate 0.71–1.11%; methyl palmitoleate 0.11–0.21%; methyl laurate 0.10–0.16%; methyl caprate 0.00–0.10% [30]. Higher saturated fatty acid in FAME, higher potential of wax crystal formation [31]. The formation of wax crystal occurs due to interactions between saturated molecules in fame with van der Waals interactions [32]. Molecules of saturated FAME have straight and no branch chains, it makes them to pack neatly and closely together. This regular arrangement of FAME molecules enhances van der Waals interactions between molecules [33]. As a result, molecular attractions become stronger and wax crystals form more readily at low temperatures. The effects of adding 2-HEE and 2-HPE to biodiesel are shown in the graph in **Figure 14**.

The major compounds detected in 2-HEE and 2-HPE were ethyl ricinoleate and propyl ricinoleate, respectively. Ricinoleate has polar hydrocarbon chain because of the existence of a hydroxyl group and a double bond can disorder the wax crystal formation with participating in wax crystal formation on FAME molecules. Then, the polar structures of 2-HEE and 2-HPE, which contain an acyl group and a hydroxyl group, limit the extent of co-crystallization by restricting crystal-to-crystal attachment. At the same concentration, the addition of 2-HPE to biodiesel tended to result in a greater reduction in cold flow properties compared to 2-HEE. This is attributed to the presence of an additional methyl branch in the polar head structure of 2-HPE that can create geometric disruption and more prevent crystal-to-crystal attachment. The addition of additives containing polar groups within their hydrocarbon chains allows them to participate in wax crystal formation (co-crystallization) and disrupt van der Waals interactions [32]. Acyl group and a hydroxyl group, limit the extent of co-crystallization by restricting crystal-to-crystal attachment [34]. Branching creates geometric disruption, preventing molecules from packing neatly to form crystals [29]. The proposed interactions between 2-HEE and biodiesel, as well as between 2-HPE and biodiesel, are illustrated in **Figure 15**, adapted from the mechanism of turpentine oil as a fluidity improver for B40 biodiesel [35].

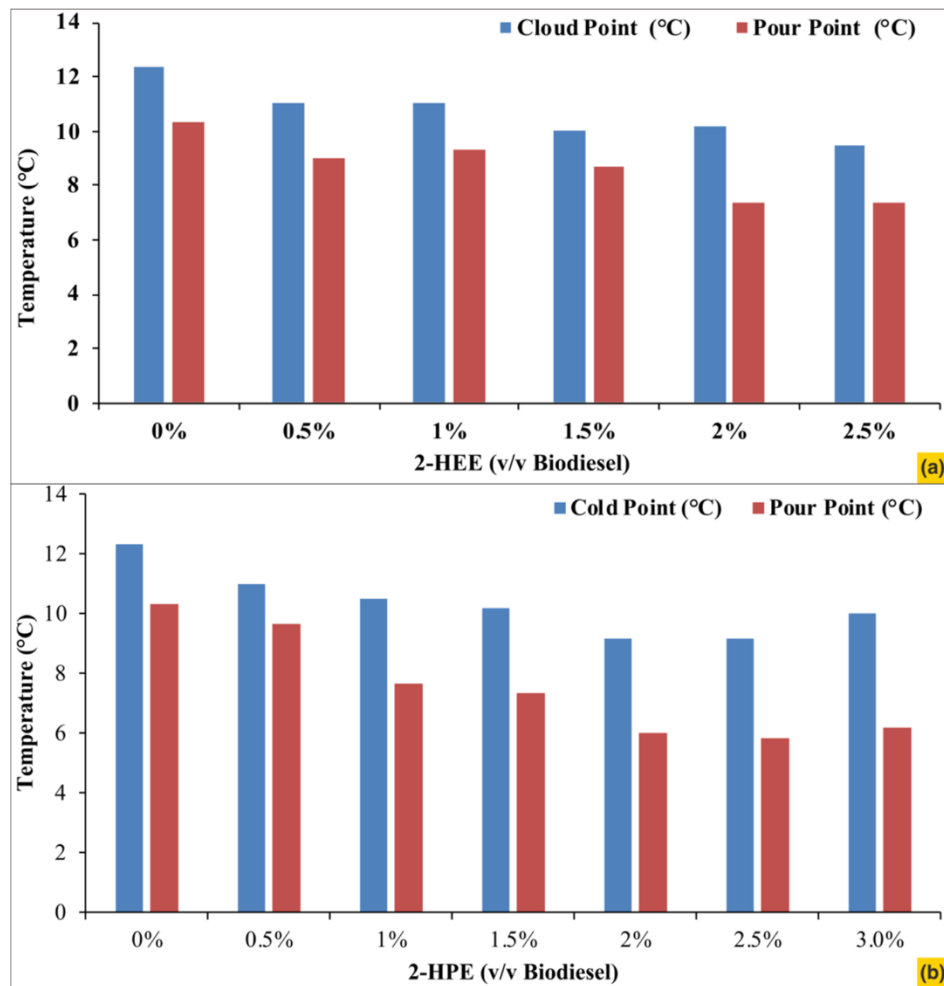


Figure 14. Cold flow properties of the addition of (a) 2-HEE and (b) 2-HPE to biodiesel (B100) at various concentrations

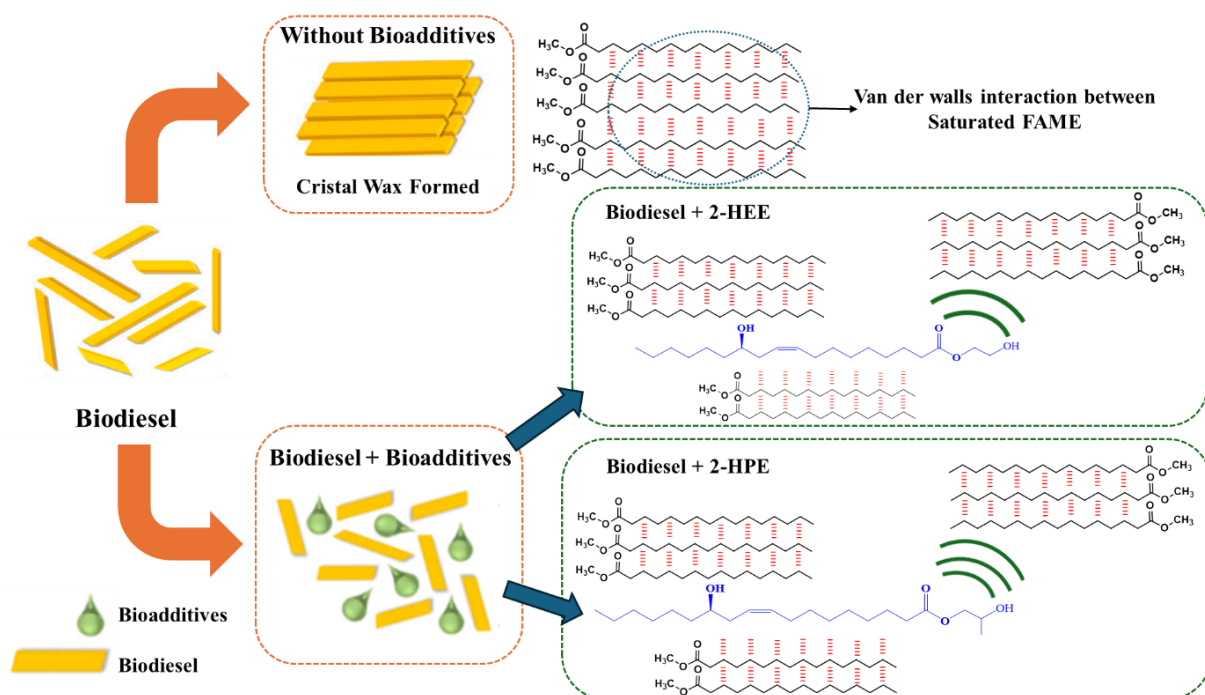


Figure 15. Proposed interactions between bioadditives (2-HEE and 2-HPE) and biodiesel (B100)

The addition of the 2-HEE additive to biodiesel was allowed to reduce the CP and PP 10.1 °C and 7.3 °C, respectively, at a concentration of 2% v/v. Increasing the 2-HEE concentration to 2.5% v/v resulted in a further decrease in the CP but did not produce a significant change in the PP. Therefore, the optimum concentration of 2-HEE addition to biodiesel was determined to be 2% v/v. Meanwhile, the addition of the 2-HPE additive to biodiesel reduced the CP and PP to 9.2 °C and 5.8 °C, respectively, at a concentration of 2.5% v/v. However, when the 2-HPE concentration was increased to 3% v/v, both parameters showed an increase. Thus, the optimum concentration of 2-HPE was established at 2.5% v/v. The increase in biodiesel cold flow properties upon the addition of bioadditives above the optimum concentration occurs because the bioadditives associate with the biodiesel through hydrogen bonding, thereby participating in crystal formation [36]. For further physical property testing, a concentration of 2% v/v of both 2-HEE and 2-HPE in biodiesel was used to allow for direct comparison. The results were compared other additives and are presented in Table 5.

3.5.2. The characteristic of flash point

The flash point is the lowest temperature at which the vapor from a liquid or solid substance can burn instantly (causing a flame) when it comes into contact with a heat source, spark, or flame [37]. In the context of fuels, an excessively high flash point makes the fuel difficult to ignite, resulting in inefficient combustion [38]. A slightly lower flash point can reduce the energy required

for combustion initiation [39]. The biodiesel standard requires the flash point to be not less than 130 °C. This standard needs to be considered because the lower the flash point, the more easily the substance ignites and the greater the risk of fire and explosion [37]. The effect of adding 2% 2-HEE and 2% 2-HPE to biodiesel, which resulted in a decrease in the biodiesel flash point, is shown in Figure 16.

Biodiesel has a flash point of 166 °C. The addition of 2% v/v 2-HEE to biodiesel reduced the flash point by 3 °C to 163 °C. Meanwhile, the addition of 2% v/v 2-HPE reduced the flash point by 8 °C to 158 °C. The lower the flash point of a fuel, the higher its volatility [40]. The addition of 2% v/v 2-HEE and 2% v/v 2-HPE to biodiesel caused a decrease in the flash point. Similar to their effects on the cold flow characteristic of biodiesel, 2-HEE and 2-HPE disrupt van der Waals interactions among biodiesel molecules, increasing intermolecular spacing and weakening

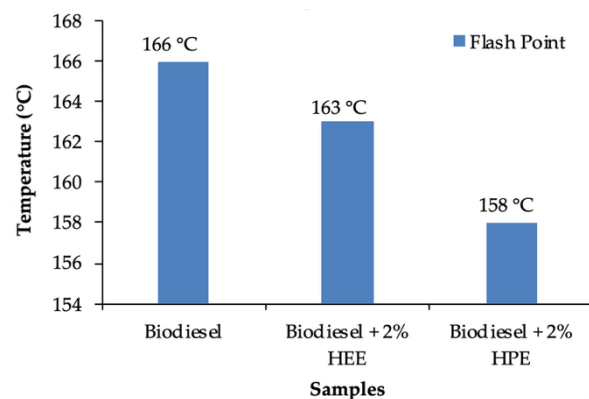


Figure 16. Graph of the effect of 2-HEE and 2-HPE addition biodiesel (B100) flash point

Table 5. Characteristics of cold flow

Samples	CP (°C)	PP (°C)	Changes (°C)		References
			CP	PP	
Standard	<18	<15	-	-	SNI
Biodiesel	12.3	10.3	-	-	This Study
Biodiesel + 2-HEE 2%	10.1	7.3	2.2	3	This Study
Biodiesel + 2-HPE 2%	9.2	5.8	3.1	4.5	This Study
Biodiesel + SoLE 0.5%	18	17	3	2.7	[41]
Biodiesel + DTMPTE	-	-6	-	-	[42]
Biodiesel + 2-butanol 10%	-	-	4	-	[5]
Biodiesel + tert-butyl alcohol 10%	-	-	5	-	[5]
Biodiesel + IEMS 5%	11	10	5	4	[4]
Biodiesel + 2-BEMS 5%	10	8	6	6	[4]
Biodiesel + DHFA 1%	-5	-2	3.5	1	[43]

Descriptions : CP (Cloud Point), PP (Pour Point), 2-HEE (2-Hydroxyethyl Ester), 2-HPE (2-Hydroxypropyl Ester), SoLE (Solketal Levulinate Ester), DTMPTE (Di-trimethylolpropane Tetra Ester), IEMS (Isopropyl Ester of Palm Oil), 2-BEMS (2-Butyl Ester of Palm Oil), DHFA (Dihydroxy Fatty Acid).

intermolecular forces [44]. The presence of branched esters makes biodiesel more prone to evaporation at the liquid surface, thereby shifting the flash point to lower temperatures [45]. The addition of 2-HPE, which contains a branch, renders its molecular structure more “bulky” than that of 2-HEE, further reducing intermolecular contact area and weakening van der Waals forces to a greater extent [46].

3.5.3. The characteristic of density

The Density is generally defined as mass per unit volume [47]. Density influences atomization, spray formation in injectors, and combustion efficiency. A high fuel density increases power output but reduces thermal efficiency [48]. The range standard of biodiesel's density is from 0.850 to 0.890 g/cm³. This specification is aligned with diesel engine requirements so that biodiesel can behave similarly to petroleum diesel in terms of flow, injection, and combustion processes [49]. The effect of adding 2% v/v 2-HEE and 2% v/v 2-HPE on biodiesel density is shown in the graph in Figure 17.

The density of biodiesel with the addition of 2% v/v 2-HEE and 2% v/v 2-HPE decreased slightly, by 0.005 g/cm³ and 0.001 g/cm³, respectively. This reduction in density is not desirable, as the biodiesel used as a baseline already had a density lower than the standard, so the decrease does not provide a physical advantage. The density reduction occurs because van der Waals interactions among biodiesel molecules are disrupted by the presence of 2-HEE and 2-HPE, leading to a less coordinated, looser molecular arrangement and a lower density [50]. More polar additives with lower densities can further reduce the packing of FAME molecules, increasing the mixture's specific volume and causing a greater decrease in density [51]. 2-HEE tends to be more polar than 2-HPE because branched alcohols have lower polarity than straight-chain alcohols; for example, 1-propanol is more polar than 2-propanol [52].

3.5.4. Viscosity cinematic at 40 °C

Viscosity is a measure of a fluid's thickness and describes how resistant it is to flow. It directly relates to temperature, pressure, and the formation of fluid layers. Viscosity significantly affects atomization in injector nozzles; if too high,

fuel droplets become larger, spray angles narrow, and mixing with air is less effective, resulting in incomplete combustion [53]. The standard kinematic viscosity at 40 °C ranges from 2.3 to 6.0 cSt. The effect of adding 2% v/v 2-HEE and 2% v/v 2-HPE on biodiesel viscosity is shown in the graph in Figure 18.

The viscosity of biodiesel increased by 0.21 cSt with the addition of 2% v/v 2-HEE and by 0.01 cSt with 2% v/v 2-HPE. Hydroxyl groups are polar and can form hydrogen bonds between molecules, both among additive molecules and between additives and the methyl esters in biodiesel [54]. The hydroxyl groups in 2-HEE and 2-HPE facilitate hydrogen bonding between the solute (2-HEE or 2-HPE) and the solvent (biodiesel) [36]. The higher viscosity observed with 2-HEE compared to 2-HPE is attributed to stronger hydrogen bonding interactions in straight-chain alcohols compared to branched alcohols [55]. Nevertheless, the changes in biodiesel viscosity are not significant, and both remain within the safe range according to standard viscosity values.

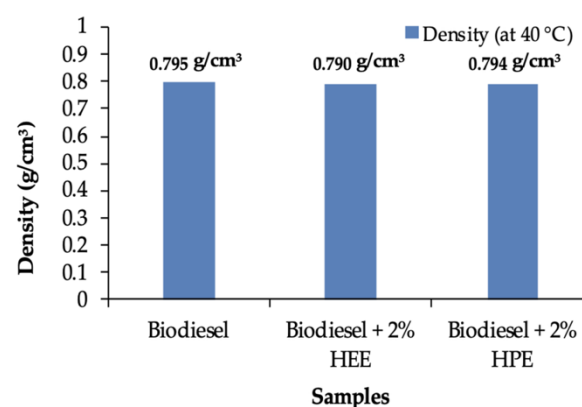


Figure 17. Graph of the effect of 2-HEE and 2-HPE addition on biodiesel (B100) density

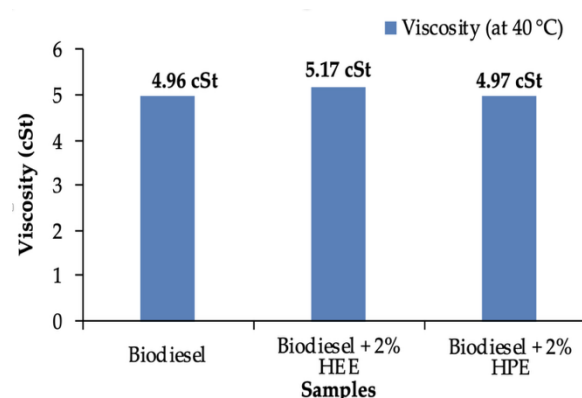


Figure 18. Graph of the effect of 2-HEE and 2-HPE addition on biodiesel (B100) viscosity at 40 °C

4. Conclusion

The synthesis of 2-HEE and 2-HPE from castor oil FAME using K_2CO_3 as a catalyst showed that a stoichiometric ratio of FAME to polyol of 1:10 produced the highest yields: 69.63% for 2-HEE and 56.84% for 2-HPE. The total abundance of 2-HEE was 98.17%, dominated by 2-hydroxyethyl ricinoleate at 77.4%, while 2-HPE reached 98.97%, dominated by 2-hydroxypropyl ricinoleate at 77.9%. Evaluation of the cold flow characteristic of biodiesel with added 2-HEE and 2-HPE indicated that a 2% v/v dose was optimal for 2-HEE, resulting in a reduction of the cloud point by 2.2 °C and the pour point by 3 °C. For 2-HPE, a 2% v/v addition reduced the cloud point by 3.1 °C and the pour point by 4.3 °C, with the optimal condition for 2-HPE being 2.5% v/v (cloud point decrease of 3.1 °C and pour point decrease of 4.5 °C). Physical property analysis at 2% v/v showed that the addition of 2-HEE and 2-HPE decreased the flash point by 3 °C and 8 °C, respectively; reduced the density by 0.005 g/cm³ and 0.001 g/cm³, respectively; and increased the kinematic viscosity at 40 °C by 0.21 cSt and 0.01 cSt, respectively. Overall, the addition of 2-HPE was more effective than 2-HEE in improving the cold flow characteristic of biodiesel without significantly altering other physical properties. However, further investigation is needed regarding other physical properties, particularly water content, as the synthesized bioadditive is susceptible to hydrolysis reaction.

Acknowledgements

The authors thank the Direktorat Riset Pengabdian Kepada Masyarakat (DRPM) which has provided local funding through the Scientific Research program Institut Teknologi Sepuluh Nopember in 2025.

Author's Declaration

Authors' contributions and responsibilities

The authors made substantial contributions to the conception and design of the study. The authors were responsible for data analysis, interpretation, and discussion of the results. The authors read and approved the final manuscript.

Funding

Scientific Research program Institut Teknologi Sepuluh Nopember in 2025.

Availability of data and materials

All data are available from the authors.

Competing interests

The authors declare no competing interest.

Additional information

No additional information from the authors.

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