Synthesis of 2-Hydroxyethyl Esters from Waste Cooking Oil and Ethylene Glycol as Potential Lubrication Bio-Additive

for Low-Sulfur Fossil Diesel

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**Abstract.** Low-sulfur diesel fuel has low lubricity and tends to cause engine wear. Lubricity-enhancing bio-additives are needed to increase the lubricity of low-sulfur diesel fuel. 2-Hydroxyethyl ester (2-HEE) is one of the modified triglyceride compounds that has the potential to be used as a lubricity-enhancing bio-additive. This compound has successfully synthesized from fatty acid methyl ester (FAME) and ethylene glycol with a molar ratio of 1:2 and 1.2% K2CO3 w/w through a transesterification reaction for 180 minutes at temperature of 130°C. FAME is obtained from waste cooking oil reacted with methanol at a molar ratio of 1:6 at a temperature of 65°C under reflux conditions. The FAME and 2-HEE component was identified using GC-MS. FAME yield reached 82.72%, and GC-MS relative abundance was 99.7% with 10 types of methyl esters formed. The conversion of synthesized 2-HEE rate was 89.08%, and a GC-MS relative abundance was 37.54% with 8 types of hydroxyethyl esters formed. This compound can be used as an alternative bio-additive to improve the lubrication of low-sulfur fossil diesel fuels.

**Keywords:** 2-Hydroxyethyl Ester, Fatty Acid Methyl Ester, Waste Cooking Oil, Ethylene Glycol, Transterification

# INTRODUCTION

The development of compounds with potential as bioadditives to improve the lubricity of diesel fuel is necessary to improve the lubricity of low sulfur diesel fuel. Acording to ASTM D5453, the maximum sulfur content of commercial diesel fuel is 10 ppm. Low sulfur diesel fuel has low lubricity because of desulfurization causes the loss of polar compounds as lubricating agents. Therefore, additives are needed to improve the lubricity of diesel fuel. In line with Sustainable Development Goal (SDG) number 7 on affordable and clean energy, the development of bioadditives is important to replace fossil fuel-based additives. The synthesis of compounds with potential as bioadditives can be carried out using vegetable oil or unused waste vegetable oil as raw materials, such as waste cooking oil (WCO) (Joshi et al., 2023).

WCO consists of varying amounts of triglycerides, diglycerides, monoglycerides and free fatty acids (5-20% by mass) produced during frying. Triglyceride is a compound consisting of three hydroxyl groups esterified with carboxyl groups of fatty acids. The triglyceride structure has high viscosity and thermal stability due to its large molecular weight (Zainal et al., 2018). Several studies have successfully synthesized compounds with potential as bioadditives using triglycerides (Joshi et al., 2023). The long polar fatty acid chains produce a high-strength lubricating film and interact strongly with metal surfaces, reducing friction and wear (Nie et al., 2020). WCO can be utilized if its chemical structure is modified first through the addition of certain compounds (Hussein et al., 2021). Chemical structure modification is generally related to acyl (C=O) and alkoxy (O-R) functional groups. The modification pathways include forming new esters through esterification/transesterification reactions, forming estolides after triglyceride hydrolysis, modifying double bonds by epoxidation to open the ring to hydroxyl groups

(McNutt and He, 2016). Among the three methods, transesterification is the most popular method for modifying the structure of triglycerides into alcohol esters, both on a laboratory and industrial scale, because it uses a more economical and environmentally friendly catalyst (Orege et al., 2022). In addition, this method is also considered practical due to its simple process and lower costs (Azeez et al., 2019).

2-hydroxyethyl (2-HEE) is an environmentally friendly base material with potential as a bio-additive that improves lubrication for diesel fuel due to its good lubricating properties, high biodegradability, low viscosity, and low volatility temperature characteristics (Zetra et al., 2023). 2-HEE is a triglyceride modifed compound in the form of a new ester with one hydroxyl group. The presence of one hydroxyl group in 2-HEE is expected to strengthen the interaction with metal surfaces, thereby forming a protective layer that can minimize direct contact between metal surfaces. Several previous studies have synthesized 2-HEE using vegetable oil such as soybean oil and peanut oil (Hariyanto et al., 2021; Zetra et al., 2021). Although the results of this research are promising, the vegetable oil sources used generally still come from foodstuffs (edible oils), thus potentially creating competition with human food needs and increasing the production costs of bioadditives. Waste cooking oil, which is usually discarded, is one of the most efficient ways to produce bioadditives without competing with food demand. Hence, the use of waste cooking oil should be given higher priority over the edible oils as bioadditive feedstock (Chhetri et al., 2008).

The use of waste cooking oil as a raw material in the synthesis of 2-HEE bioadditives has the potential to not only improve the lubricity of diesel fuel, but also reduce production costs and minimize the environmental impact of oil waste pollution. With this approach, the study is expected to contribute to the development of effective bioadditives while supporting the principles of a circular economy in resource management (Joshi et al., 2023). Therefore, this study aims to synthesize 2-HEE bioadditives from waste cooking oil and identify the types of hydroxyethyl esters formed. In addition, the potential of 2-HEE as a lubricity-enhancing bioadditive is analyzed based on a literature review.

# MATERIAL AND METHODS

## Pre-Treatment of Waste Cooking Oil (WCO)

Waste cooking oil (WCO) is filtered to separate it from frying residue, then washed using distilled water and shaken in a separating funnel, then left to settle overnight. After settling overnight, the mixture separates into two layers, where the top layer is WCO and the bottom layer is water. The water layer is separated from the oil, then 10 g of anhydrous sodium sulfate (Na₂SO₄) is added to the oil to remove the remaining water. After that, the oil containing anhydrous sodium sulfate (Na₂SO₄) is filtered again using Whatman filter paper numbers 1 and 42 to obtain clean waste cooking oil (CWCO). The CWCO acid value is measured usig a procedure adapted from the research conducted by (Suzihaque et al., 2022; Zetra et al., 2023). First, the sample (± 1.00 g) is added to 50 mL of isopropanol and 0.8 mL of 1% phenolphthalein (PP) indicator. The solution is titrated with 1N potassium hydroxide (KOH) until the indicator turns light pink. The volume of potassium hydroxide (KOH) used is used to calculate the acid value of CWCO using equations 1. The CWCO acid value is compared with the WCO acid value.

𝑉 ×𝑀 ×𝑀𝑟

𝐴𝑐𝑖𝑑 𝑉𝑎𝑙𝑢𝑒 =

(𝑚𝑔𝐾𝑂𝐻/𝑔)

𝑡𝑖𝑡𝑟𝑎𝑛𝑡 𝑡𝑖𝑡𝑟𝑎𝑛𝑡 𝐾𝑂𝐻

𝑆𝑎𝑚𝑝𝑙𝑒 𝑀𝑎𝑠𝑠

CWCO was further treated to reduce its acid number based on the research of Khuzaimah et al. (2020) using adsorption by activated carbon. A total of 200 ml of CWCO was put into an erlenmeyer. Activated carbon as much as 1 gram that has been activated is added to 200 ml of CWCO and stirred for 15 minutes (Khuzaimah and Eralita, 2020). After stirring, the activated carbon was filtered using Whatman paper number 1 and 42. The oil from activated carbon adsorption (ACWCO) was analyzed for acid number and compared with CWCO and WCO.

## Fatty Acid Methyl Ester (FAME) Synthesis and Composition Analysis

FAME synthesis can be carried out directly if the acid value content in the oil is less than 4-5 mg KOH/g (Santoso et al., 2020; Suzihaque et al., 2022). With acid value content more than 4-5 mg KOH/g, the oil needs to undergo the pre-treatment process to reduce the acid value content to avoid the risk of saponification when reacted with a base catalyst (Santoso et al., 2020). This synthesis was carried out based on research by Zetra et al. (2023)

through a transesterification reaction. The synthesis begins by heating the oil (CWCO) at a temperature of 65°C in a two-neck flask (Zetra et al., 2023). The transesterification reaction was carried out by methanol with a molar ratio of methanol and CWCO of 6:1 and KOH catalyst (1% weight/volume of oil) to the heated oil (CWCO). The reaction was continued at a temperature of 60°C with a reaction time of 150 minutes through a reflux system with stirring at 500 rpm. The FAME formed in the above reaction was then purified based on the method of Suzihaque et al. (2022) by transferring the product to a separating funnel and allowing it to settle for 24 hours at room temperature. After two (2) layers formed, the upper layer was taken and washed with hot distilled water to remove the catalyst and soap residue. Next, 10 g of anhydrous sodium sulfate (Na₂SO₄) was added to absorb the remaining water (Suzihaque et al., 2022). The product formed was then calculated using equation 2.

𝑇ℎ𝑒𝑜𝑟𝑖𝑡𝑖𝑐𝑎𝑙 𝑚𝑎𝑠𝑠 𝑜𝑓 𝐹𝐴𝑀𝐸

% 𝑌𝑖𝑒𝑙𝑑 = ×100%

𝐸𝑥𝑝𝑒𝑟𝑖𝑚𝑒𝑛𝑡𝑎𝑙 𝑚𝑎𝑠𝑠 𝑜𝑓 𝐹𝐴𝑀𝐸

FAME composition was analyzed using an Agilent GCMSD5975C gas chromatography-mass spectrometer (GC-MS) instrument, capillary column type HP-5MS (5% phenylmethylpolysiloxane) film size 60 m x 250 µm x

0.33 µm using helium (He) carrier gas. The gas chromatography was set at an initial temperature of 70°C and maintained for 1 minute, then the temperature was increased to 180°C with an increase rate of 10°C/minute, then the temperature was increased to 315°C with an increase rate of 4°C/minute and maintained for 30 minutes. The mass spectrometer used was an electron impact (EI) mass spectrometer with an ionization energy of 70 eV.

## 2-Hydroxyethyl Ester (2-HEE) Syntesis and Composition Analysis

The synthesis of 2-HEE procedure referes to (Attia et al., 2020; Hussein et al., 2021) using synthesized FAME. The reaction was carried out by reacting FAME and ethylene glycol with a molar ratio of 1:2. Ethylene glycol and K2CO3 catalyst (1.2% w/w FAME) was heated in a round bottom flask at temperature of 130°C with vacuum system. After reaching 130°C, FAME was added to the flask and stirring at 500 rpm. This condition was maintained for 3 h. Next, the purification of the product is conducted by method adapted from Zetra et al. (2023) by adding hydrochloric acid (HCl) until the pH was netral (Zetra et al., 2023). The separation of the organic phase from the aqueous phase was carried out through liquid liquid extraction using 30 mL of ethyl acetate solvent (3x30 ml) in a separating funnel. The separated organic phase was washed with distilled water (3x30 ml) and dried by adding 10 g anhydrous sodium sulfate (Na2SO4) and filtered using Whatman filter paper numbers 1 and 42. The filtrate obtained was evaporated using a vacuum rotary evaporator. The reaction conversion was calculated according to equation 3.

% 𝐶𝑜𝑛𝑣𝑒𝑟𝑠𝑖𝑜𝑛 = 𝐴 − 𝐵

𝐶

A : Wt. of reaction mixture before reaction

B : Wt. of reaction mixture after reaction

C : Theoritical wt. of methanol out of reaction with respect to limiting reactant

The composition of 2-HEE was analyzed using an Agilent GCMSD5975C gas chromatography-mass spectrometer (GC-MS) instrument, capillary column type HP-5MS (5% phenylmethylpolysiloxane) film size 60 m x 250 µm x 0.33 µm using helium (He) carrier gas. The gas chromatography was set at an initial temperature of 80°C and maintained for 1 minute, then the temperature was increased to 180°C with an increase rate of 10°C/minute, then the temperature was increased to 325°C with an increase rate of 4°C/minute and maintained for 40 minutes. The mass spectrometer used was an electron impact (EI) mass spectrometer with an ionization energy of 70 eV.

# RESULT AND DISSCUSSION

## WCO Acid Value Analysis

Acid number analysis was carried out by simple titration method on waste cooking oil before pre-treatment (WCO), after pre-treatment using water (CWCO), and after activated carbon adsorption (ACWCO). Acid number is a parameter that determines the amount of free fatty acids contained in the oil. The acid number value is expressed

in mgKOH/g or equivalent to the amount of KOH in mg to neutralize 1 gram of oil. The acid number data from each stage is presented in Table 1.

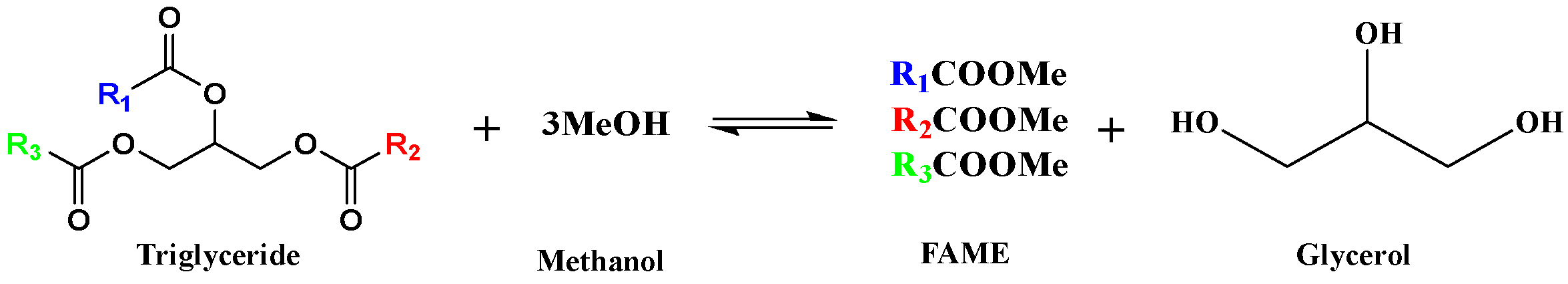
**TABLE 1.** Acid Value

|  |  |  |
| --- | --- | --- |
| **Sample** | **Step** | **Acid Value (mgKOH/g)** |
| WCO | Before pre treatment | 11.8 |
| CWCO | Water pre treatment (first treatment) | 3.49 |
| ACWCO | Active carbon adsorption (second treatment) | 3.28 |

The WCO has an acid number of 11.8 mgKOH/g and decreased after pre treatment as shown in Table 1. The high acid number of WCO caused by the presence of water and impurities in the oil. The presence of water in oil results in hydrolysis. Other than that, food that contains more water also facilitates the growth of fungi. These fungi excrete certain enzymes that degrade triglycerides into free fatty acid and glycerol. After the first pre treatment process, the acid value significantly decreases. The decrease in acid value is caused by a reduction in water content during extraction and drying with Na₂SO₄. The second treatment also lowers the acid value because the stirring pallows free fatty acids to come into greater contact with the activated carbon. These collisions facilitate greater absorption into the walls and pores of the activated carbon (Khuzaimah and Eralita, 2020). Based on these results, CWCO was chosen as a substrate in FAME synthesis because it already has acid value less then 4 mg KOH/g without further treatment. The acid value content shloud less than 4-5 mg KOH/g to avoid the risk of saponification when reacted with a base catalyst (Santoso et al., 2020).

## Fatty Acid Methyl Ester (FAME) Synthesis and Composition Analysis

Fatty acid methyl esters (FAME) have been synthesized from clean waste cooking oil (CWCO) through a transesterification reaction between triglycerides in CWCO with methanol. The ratio of methanol is a key parameter for the conversion of triglycerides to their methyl ester form. In the stoichiometry of the triglyceride transesterification reaction, one mole of triglycerides requires three moles of methanol to produce three moles of FAME and one mole of glycerol. The reaction is shown in Figure 1.



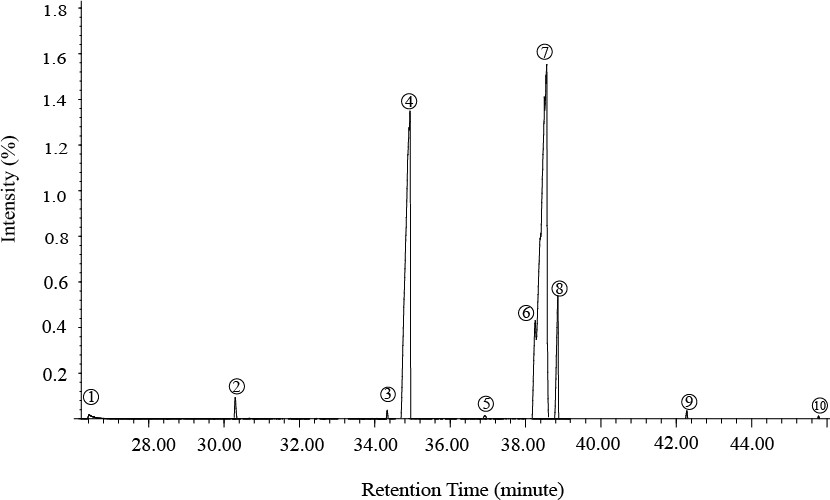
**Figure 1.** Triglyceride transesterification reaction

Increasing the mole ratio of triglyceride to methanol will also increase the yield of FAME. Increasing the amount of methanol will shift the reaction to the right or towards the product (FAME), thus increasing the yield of FAME (Oko et al., 2021). However, synthesized FAME produced a yield of 86.55%. The composition of the FAME formed was identified using GC-MS. The chromatogram in Figure 2 shows ten peaks with different intensities. The peaks obtained were identified the type of methyl ester based on its mass spectrum. Based on the analysis, the type of methyl ester in FAME can be identified with a total abundance of 99.78%. The results of this analysis are presented in Table 2, where 42.63% of the components are saturated methyl esters and 57.15% are unsaturated methyl esters.

**TABLE 2.** FAME Composition

|  |  |  |  |
| --- | --- | --- | --- |
| **No.** | **FAME Composition** | | **Abundance (%)** |
| 1 | ME Dodecanoate | C12:0 | 0.17 |
| 2 | ME Tetradecanoate | C14:0 | 1.19 |
| 3 | ME Hexadecenoate | C16:1 | 0.53 |
| 4 | ME Hexadecanoate | C16:0 | 34.68 |
| 5 | ME Heptadecanoate | C17:0 | 0.13 |
| 6 | ME Octadecadienoate | C18:2 | 5.79 |
| 7 | ME Octadecenoate | C18:1 | 51.36 |

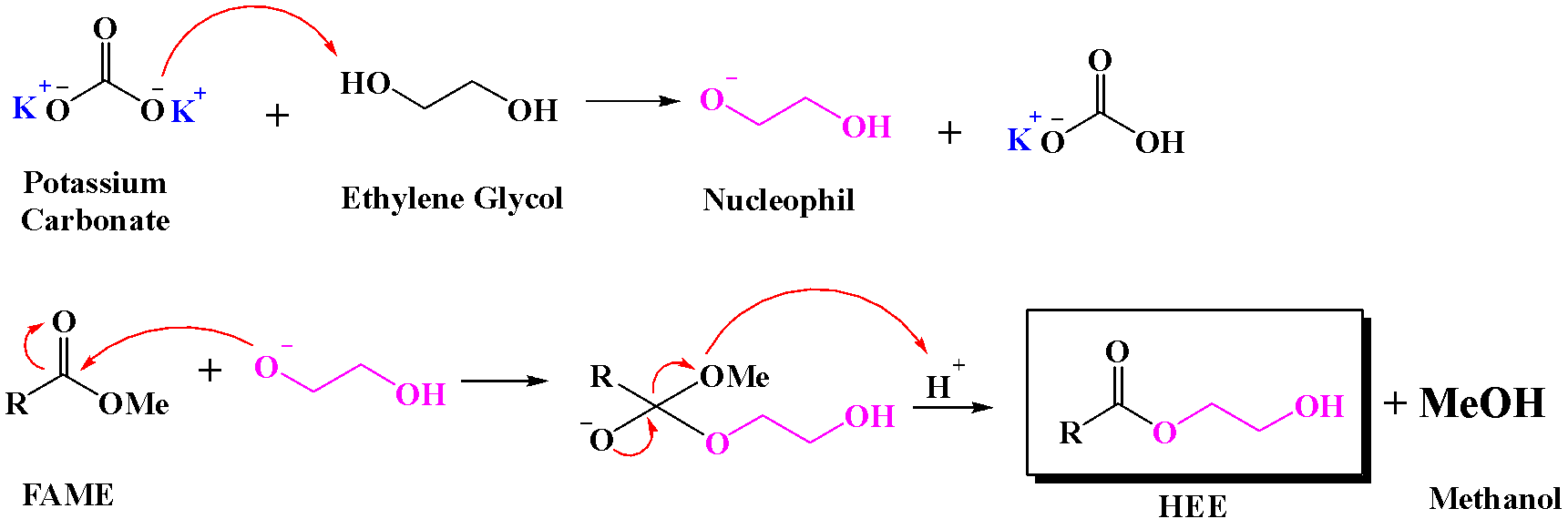
|  |  |  |  |
| --- | --- | --- | --- |
| **No.** | **FAME Compo** | **sition** | **Abundance (%)** |
| 8 | ME Octadecanoate | C18:0 | 5.43 |
| 9 | ME Icosanoate | C20:0 | 0.50 |
| 10 | ME Lignokerat | C24:0 | 0.11 |
| TOTAL | | | 99.78 |
| ∑Unsaturated | | | 57.15 |
| ∑Saturated | | | 42.63 |



**Figure 2.** FAME GC-MS chromatogram

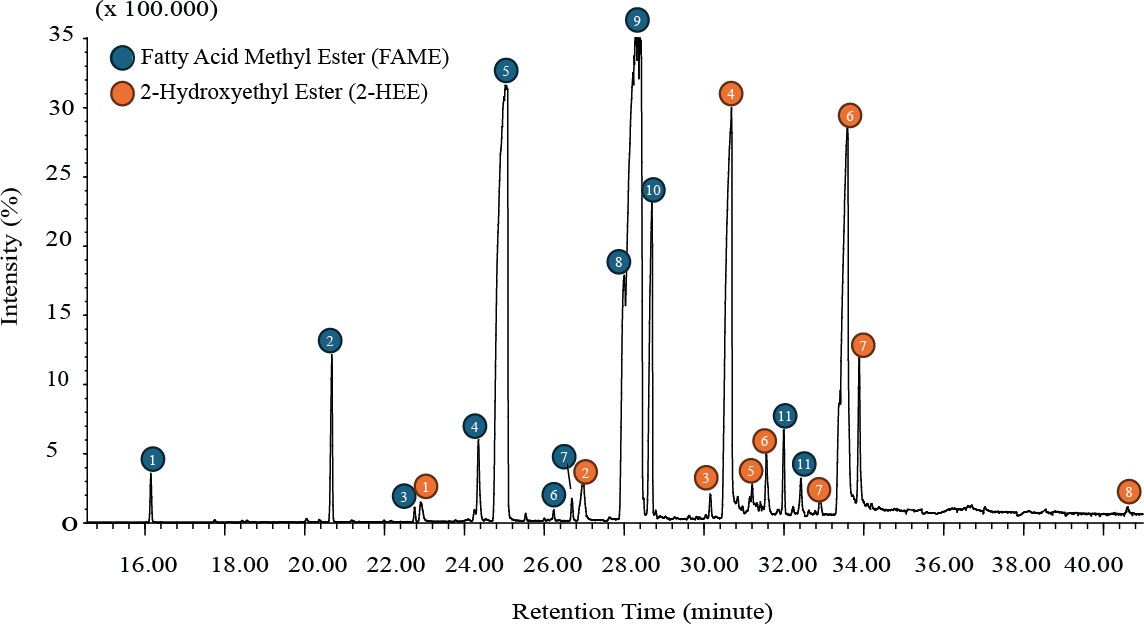
## 2-Hydroxyethyl Ester (2-HEE) Synthesis and Composition Analysis

2-HEE has been synthesized through a transesterification reaction with ethylene glycol and potassium carbonate (K₂CO₃) base catalyst. The main parameters in the synthesis of 2-HEE are the molar ratio of FAME : ethylene glycol, also the catalyst. Theoretically, to obtain one mole of 2-HEE, one mole of FAME is required. In this study, the ethylene and FAME ratio used was 2:1 v/v, referring to Attia et al. (2020), with a reaction time of 3 hours at 130˚C under vacuum. Excess ethylene glycol can shift the reaction to the right or to the product to obtain more optimal results. The catalyst concentration used was 1.2% w/b FAME, referring to Attia et al. (2020) and Hussein et al. (2021), which is the optimum catalyst concentration (Attia et al., 2020; Hussein et al., 2021). The K2CO3 catalyst was chosen because at the same concentration with a shorter reaction time, K2CO3 can produce a higher conversion percentage compared to KOH (Nie et al., 2020). The product formed was 2-HEE with methanol as a by-product. The reaction in a vacuum was carried out with the aim of separating methanol from the mixture. One indicator that the reaction was complete was that methanol stopped dripping in the vacuum tube. Reaction conversion is expressed as the ability of reactants to convert substrates into products or the amount of reactants that can react to form products. The conversion value was calculated using equation three (3) and obtained a value of 89.08%. This indicates that 89.08% of EG as the reactant has reacted with FAME as the substrate to form the 2-HEE product. The conversion obtained was only 89.08%, which may have been caused by the limited amount of effective reactants or suboptimal reaction conditions (Hussein et al., 2021).



**Figure 3.** 2-HEE synthesis reaction mechanism

2-HEE was then identified using GC-MS and a chromatogram was obtained which is shown in Figure 4. The peaks in the chromatogram obtained were then identified based on their mass spectra. Based on the results of GC-MS analysis, 2-HEE was obtained with a GC-MS relative abundance of 37.54%. The hydroxyethyl ester composition with the highest abundance is octadecanoate hydroxyethyl ester (C18:1) at 16.85% which is an unsaturated hydroxyethyl ester, and hexadecanoate hydroxyethyl ester (C16:0) at 14.35% which is an unsaturated hydroxyethyl ester. The composition obtained in the synthesis results is in accordance with the composition of FAME.



**Figure 4.** 2-HEE GC-MS chromatoram

**TABLE 3.** 2-HEE composition

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **No.** | **Compound** |  | **Cemical Formula** | **Mr** | **Abundance (%)** | **Total Abundance (%)** |
| **Fatty Acid Methyl Ester (FAME)** | | | | | | |
|  | 1. ME dodecanoate | C12:0 | C13H34O2 | 214 | 0,47 | 61,62 |
|  | 2. ME Tetradecanoate | C14:0 | C15H34O2 | 242 | 2.13 |
|  | 3. ME Pentadecanoate | C15:0 | C16H34O2 | 256 | 0.14 |  |
|  | 4. ME Hexadecenoate | C16:1 | C17H32O2 | 268 | 1.23 |  |
| 1 | 5. ME Hexadecanoate | C16:0 | C17H34O2 | 270 | 21.64 |  |
|  | 6. ME Heptadecanoate | C17:0 | C18H36O2 | 284 | 0.25 |  |
|  | 7. ME Octadecadienoate | C18:2 | C18H34O2 | 294 | 5.62 |  |
|  | 8. ME Octadecenoate | C18:1 | C18H36O2 | 296 | 23.49 |  |
|  | 9. ME Octadecanoate | C18:0 | C18H38O2 | 298 | 5.95 |  |
|  | 10. ME Icosanoate | C20:0 | C21H42O2 | 326 | 0.70 |  |
|  | **Hidroxyethyl Ester (HEE)** | | |  |  |  |
| 2 | 1. HE Dodecanoate | C12:0 | C14H28O3 | 244 | 0.42 | 37.54 |
|  | 2. HE Tetradecanoate | C14:0 | C16H32O3 | 272 | 1.18 |  |

**No. Compound**

**Cemical Mr Formula**

**Abundance (%)**

**Total Abundance (%)**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| 3. HE Hexadecenoate | C16:1 | C18H34O3 | 298 | 0.31 |
| 4. HE Hexadecanoate | C16:0 | C18H36O3 | 300 | 15.36 |
| 5. HE Octadecadienoate | C18:2 | C20H36O3 | 324 | 1.04 |
| 6. HE Octadecenoate | C18:1 | C20H38O3 | 326 | 16.65 |
| 7. HE Octadecanoate | C18:0 | C20H40O3 | 328 | 2.46 |
| 8. HE Lignokerat | C24:0 | C26H52O3 | 410 | 0.12 |
|  | ∑Unsaturated | |  | 48.46 |
|  | ∑Saturated | |  | 50.70 |

## 2-HEE Potential as Bioadditive for Low-Sulphur Diesel Fuel Lubricity Improver

The hydroxyl group in 2-HEE is responsible for its lubricating properties. Therefore, hydroxyethyl ester can be used as a lubricity-enhancing bioadditive in low sulfur diesel fuel (LSD) (Firmansyah et al., 2022). The higher the polarity of the compound used as a bioadditive, the higher the potential of the bioadditive to reduce wear in LSD analyzed using a High-Frequency Reciprocating Rig (HFRR). The presence of hydroxyl (OH) groups in bioadditive compounds can increase their polarity. Compounds that have polar groups in their structure provide tribological capabilities in reducing wear. Hydroxyl (-OH) groups bound to ester groups (RCOOR) are absorbed at the ball interface, producing a lubricating film with higher polarity that results in greater shear strength and better protection against wear. This behavior is caused by strong intermolecular hydrogen bonds (Hsiao et al., 2021). 2-HEE from castor oil can reduce wear marks on low-sulfur fossil diesel from 279 μm to 198.5 μm (Firmansyah et al., 2022).

# CONCLUSION

The 2-hydroxyethyl ester (2-HEE) was successfully synthesized from waste cooking oil through transesterification with a conversion of 89.08% and a relative abundance of 37.54% based on GC–MS analysis. The main components were hydroxyethyl ester octadecenoate (C18:1, 16.65%) and hydroxyethyl ester hexadecanoate (C16:0, 15.36%). These results demonstrate that waste cooking oil can be utilized as a sustainable feedstock for producing 2-HEE. Due to the presence of hydroxyl groups, 2-HEE has the potential to act as a lubricity enhancer when applied as a bio-additive for low-sulfur diesel fuel.

# ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial support provided by Institut Teknologi Sepuluh Nopember (ITS) through the Penelitian Keilmuan – Pendanaan Penelitian Keilmuan scheme under Contract No. 1179/PKS/ITS/2024.

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