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OPTIMIZATION OF REACTION TIME IN THE TRANSESTERIFICATION OF DISTILLED-WATER-WASHED USED COOKING OIL INTO FATTY ACID METHYL ESTERS

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Abstract

Fatty acid methyl ester (FAME) is an alternative to fossil diesel fuel that can be synthesized from used cooking oil (UCO). UCO requires pretreatment before it can be used for FAME synthesis because of its high acid value. Pretreatment of UCO using distilled-water washing reduced the acid value from 11.80 mg KOH/g to 3.49 mg KOH/g. The treated UCO was reacted with methanol through transesterification at reaction times of 90, 120, 150, and 180 min to produce FAME. The acid value decreased with increasing reaction time, from 1.57 mg KOH/g at 90 min to 0.97 mg KOH/g at 120 min and 0.43 mg KOH/g at 150 min. However, the acid value increased again at 180 min to 1.10 mg KOH/g, which was attributed to the reversible nature of the reaction. The FAME sample with the lowest acid value was analyzed using GC–MS, and five methyl esters were identified: methyl myristate (2.86%), methyl palmitate (65.88%), methyl oleate (19.25%), methyl stearate (10.85%), and methyl arachidate (0.10%), along with other compounds (1.06%). FAME synthesized from UCO pretreated by distilled-water washing has the potential to reduce large-scale FAME production costs because the distilled-water washing process is highly economical.

Keywords: *Used Cooking Oil, Fatty Acid Methyl Ester, Transesterification, Acid Value, GC-MS*

Introduction

Fossil fuels, such as oil, coal, and natural gas, remain the primary energy sources supporting various sectors of life, including transportation, industry, and power generation. Fossil fuels currently account for 85% of the world's energy resources and are predicted to remain dominant in the future [1]. However, non-renewable petroleum fuels are a major source of carbon dioxide (CO_2) emissions. The combustion of petroleum fuels contributes approximately 52% of total CO_2 emissions caused by human activities. The overexploitation of these fuels also negatively affects global temperatures and contributes to global warming [2]. Therefore, biodiesel derived from renewable sources is a promising alternative to petroleum fuel.

Biodiesel is also known as fatty acid methyl ester (FAME). It is produced by converting triglycerides contained in vegetable oils, such as palm oil, soybean oil, jatropha oil, sunflower seed oil, and other plant-based oils [3]. Another oil that can be used to produce FAME is used cooking oil (UCO). UCO refers to oil that has been used at either household or large-scale levels. The repeated use of vegetable oil, both for

domestic and commercial purposes, produces used cooking oil. UCO is primarily composed of triglycerides, monoglycerides, diglycerides, and free fatty acids in varying amounts, approximately 5–20% by weight, formed during the frying process [4]. In addition, the use of UCO can help reduce pollution caused by used cooking oil waste by improving its processing and utilization [5].

UCO has been widely used as a feedstock for FAME production. However, FAME production from UCO requires prior processing or regeneration because of impurities from frying residues and the high acidity of the used oil [6]. Free fatty acid content can be measured through acid value. Oils with acid values above 4 mg KOH/g may cause problems during alkaline transesterification. A high free fatty acid content tends to promote soap formation when reacting with the catalyst [7]. Therefore, regeneration or pretreatment of used cooking oil is necessary before use.

A common method used for oil regeneration is adsorption. Adsorbents such as bentonite, zeolite, sulfuric acid, and activated carbon can be used for this purpose [6], [8]. Adsorption using activated carbon from cocoa successfully reduced the free fatty acid content in used cooking oil. The acid value of the used cooking oil decreased from 14.92 mg KOH/g to 5.09 mg KOH/g after 60 min of adsorption [9]. Another reported method is oil washing using a deep eutectic solvent (DES) and short-chain alcohols. DES washing can reduce free fatty acid (FFA) content from 7.94% to 1.54% [6]. Meanwhile, short-chain alcohols, such as methanol, ethanol, and isopropanol, can remove polar contaminants through liquid–liquid extraction, thereby improving oil quality [10].

Despite their effectiveness, adsorption and solvent-based treatments have several practical limitations. Adsorption requires additional adsorbent materials and subsequent separation or disposal steps, whereas solvent washing requires solvent recovery and management to minimize solvent loss and operating costs. Therefore, an economical and more environmentally benign pretreatment method is desirable. Water washing has been reported as a degumming process in which water hydrates and removes soluble contaminants from waste cooking oil [11]. The addition of water can form an oil-in-water emulsion stabilized by hydrophilic groups, particularly hydroxyl groups, in free fatty acids [12]. The separation of the emulsion and water phases may reduce the free fatty acid content in the oil. This method uses inexpensive, readily available, and non-toxic materials without requiring adsorbent preparation or solvent recovery. Previous studies have shown that water washing can effectively reduce contaminants in waste cooking oil. However, the detailed effect of distilled-water washing on acid value reduction and its suitability as a pretreatment step before FAME synthesis remain limited.

Therefore, this study evaluates the effectiveness of distilled-water washing for waste cooking oil purification and its subsequent application in FAME production. The synthesis process was carried out using varied reaction times to determine the optimum condition for producing FAME with the lowest acid value. The resulting FAME products were then characterized using gas chromatography–mass spectrometry (GC–MS) to identify the composition of the methyl esters formed.

Experimental section

Materials

The materials used in this study included waste cooking oil derived from palm cooking oil and obtained from local collectors, potassium hydroxide (≥99.0%; Merck), methanol (≥99.0%; Merck), 2-propanol (≥99.5%; Sigma-Aldrich), anhydrous Na_2SO_4 (Merck), *n*-hexane (Merck), diethyl ether (Sigma-Aldrich), phenolphthalein indicator (PP), distilled water, universal indicator, TLC silica gel 60 F254, and Whatman No. 42 filter paper.

Instruments

The laboratory equipment used in this study included a round-bottom flask, thermometer, burette, magnetic stirrer, Erlenmeyer flask, clamp, stand, analytical balance, stirring bar, pipette, and filter. The instruments used were an Agilent gas chromatography–mass spectrometry (GC–MS) instrument equipped with an HP-5MS column and a rotary evaporator.

Procedure

Pretreatment of Used Cooking Oil

UCO was treated using two different methods to compare their effectiveness in reducing acid value. The first method was water washing. The UCO was filtered using filter paper, washed with distilled water in a separatory funnel, and left overnight. The water layer was then removed. The oil was dried with anhydrous sodium sulfate (Na_2SO_4) and filtered. The second method used activated carbon. Activated carbon, 10 g, was added to 100 mL of UCO and stirred using a magnetic stirrer at 200 rpm for 3 h. After stirring, the oil was filtered using filter paper.

The acid value of the oil after treatment using both methods was determined using a titration method adapted from [7], [13]. First, 0.5 g of oil was mixed with 10 mL of isopropanol in an Erlenmeyer flask. Two drops of phenolphthalein were then added to the mixture. The mixture was titrated using 0.1 N potassium hydroxide (KOH). The sample was gently shaken during titration until a pink color appeared. The percentage of free fatty acids was calculated using Equation (1). The acid value was then determined based on the amount of free fatty acids obtained. The acid value was twice the free fatty acid value.

$$Acid\ Value = \frac{T \times N \times MW\ KOH}{W} \quad (1)$$

$$\%\ FFA = \frac{Acid\ Value}{2} \quad (2)$$

where:
T = volume of 0.1 N NaOH
W = weight of the sample, g
N = normality of potassium hydroxide
150
MW KOH = molecular weight of KOH

Synthesis of Fatty Acid Methyl Ester

The synthesis was carried out using a modified method reported by Firmansyah et al. (2022), using a transesterification reaction [14]. The oil was reacted with methanol at a methanol-to-oil ratio of 6:1 and with potassium hydroxide (KOH) as a catalyst at 1% w/w oil. The reaction was performed under reflux at 60°C with stirring at 500 rpm, as shown in Figure 1. The reaction time was varied at 90, 120, 150, and 180 min.

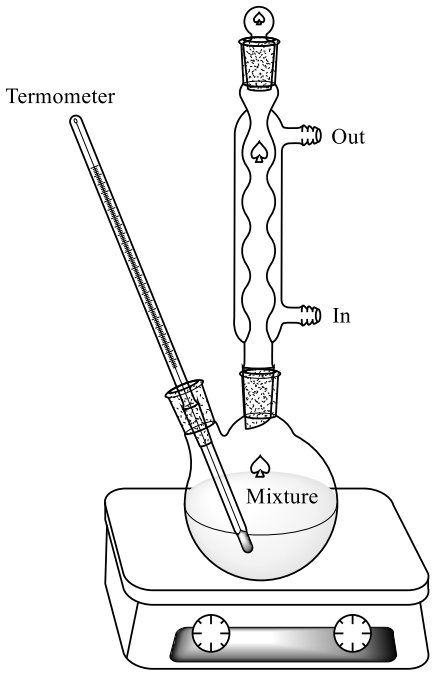

Figure 1. Reflux setup for Fatty acid methyl ester synthesis

The reaction products were left overnight. The formed FAME was present in the upper layer, whereas the lower layer consisted of glycerol, methanol, and water. After separation, the lower layer and the FAME in the upper layer were evaporated to remove the solvent and then dried using Na_2SO_4. The resulting FAME was tested for acid value using Equation (1). Product yield was calculated using Equation (2).

$$\%\ Yield = \frac{Experimental\ Mass}{Teoritical\ Mass} \times 100\% \quad (2)$$

Characterization of Fatty Acid Methyl Ester

The FAME sample with the lowest acid value was subjected to preliminary characterization using thin-layer chromatography (TLC) and further characterization using GC–MS. TLC analysis was performed using silica gel 60 F254 plates with a mobile phase consisting of *n*-hexane and diethyl ether at a ratio of 3:2 (v/v). The samples were spotted onto the TLC plate and developed in a saturated chamber until the solvent front reached an appropriate distance. The plate was then removed, dried, and visualized under UV light at 254 nm to detect the separated compounds. The retention factor (*R*f) values were calculated using Equation (3).

$$\mathrm{Rf} = \frac{\text{Compound distance}}{\textit{Eluent distance}} \qquad (3)$$

where the migration distances were measured from the origin to the center of each spot and to the solvent front, respectively.

Methyl ester composition was analyzed using an Agilent GC–MSD 5975C GC–MS instrument equipped with an HP-5MS capillary column (5% phenyl methylpolysiloxane; 60 m × 250 µm × 0.33 µm) and helium (He) as the carrier gas. The gas chromatography oven was set at an initial temperature of 70°C and held isothermally for 1 min. The temperature was then increased to 180°C at a rate of 10°C/min. Subsequently, the temperature was increased to 315°C at a rate of 4°C/min and held isothermally for 30 min. The mass spectrometer was operated in electron impact (EI) mode with an ionization energy of 70 eV.

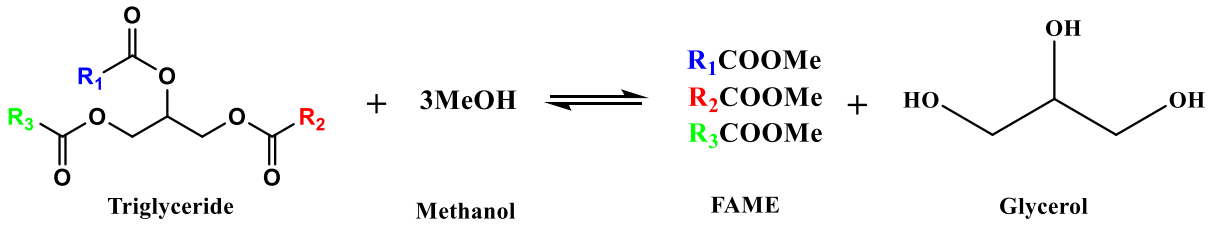

Figure 2. Fatty acid methyl ester synthesis reaction

Results and Discussion

Pretreatment of Used Cooking Oil

The acid value of the oil is shown in Table 1. Before treatment, the used cooking oil had an acid value of 11.80 mg KOH/g. This high acid value was caused by the high-temperature frying process, which changes the physical and chemical characteristics of the oil. The chemical changes include the release of free fatty acids from the partial hydrolysis of triglycerides [15].

Table 1. Acid value of used cooking oil

Oil	FFA (%)	Acid Value (mg KOH/g)
Before pretreatment	5.90	11.80
Water-washing treatment	1.74	3.49
Activated carbon treatment	1.64	3.28

After treatment, the acid value of the oil decreased significantly. In the first method, namely water washing, the acid value of the oil decreased to 3.49 mg KOH/g. The addition of water can form an oil-in-water emulsion stabilized by hydrophilic groups, particularly hydroxyl groups, in free

fatty acids. The hydroxyl groups in fatty acids interact with water, whereas the long chains in fatty acids interact with the oil phase, resulting in the formation of three phases: the oil phase, the oil-in-water emulsion, and the water phase. The emulsion and water phases are then separated. The decrease in acid value is caused by the separation of free fatty acids contained in the oil–water emulsion [12].

In the second method, namely activated carbon adsorption, the acid value of the oil decreased to 3.28 mg KOH/g. The decrease in acid value in the activated carbon adsorption method was caused by contact between the free fatty acids and the activated carbon, allowing the free fatty acids to be adsorbed onto the activated carbon [8].

Although activated carbon adsorption resulted in a slightly lower acid value, the difference between the two methods was relatively small. This finding indicates that water washing can effectively reduce free fatty acids in used cooking oil to a level suitable for subsequent transesterification. In addition, water washing offers several practical advantages, including the use of inexpensive and readily available materials, shorter preparation requirements, and the absence of adsorbent regeneration or replacement steps. Therefore, despite the slightly better performance of activated carbon adsorption, water washing was selected for subsequent FAME synthesis as a simple and cost-effective pretreatment method.

Synthesis of Fatty Acid Methyl Ester

FAME was synthesized from UCO through a transesterification reaction between triglycerides (TG) and methanol using potassium hydroxide (KOH) as a base catalyst. The ratio of methanol and catalyst to triglycerides in UCO is a key parameter in the conversion of triglycerides into methyl esters. According to the stoichiometry of the TG transesterification reaction, one mole of TG requires three moles of methanol to produce three moles of FAME and one mole of glycerol. The reaction equation is shown in Figure 2.

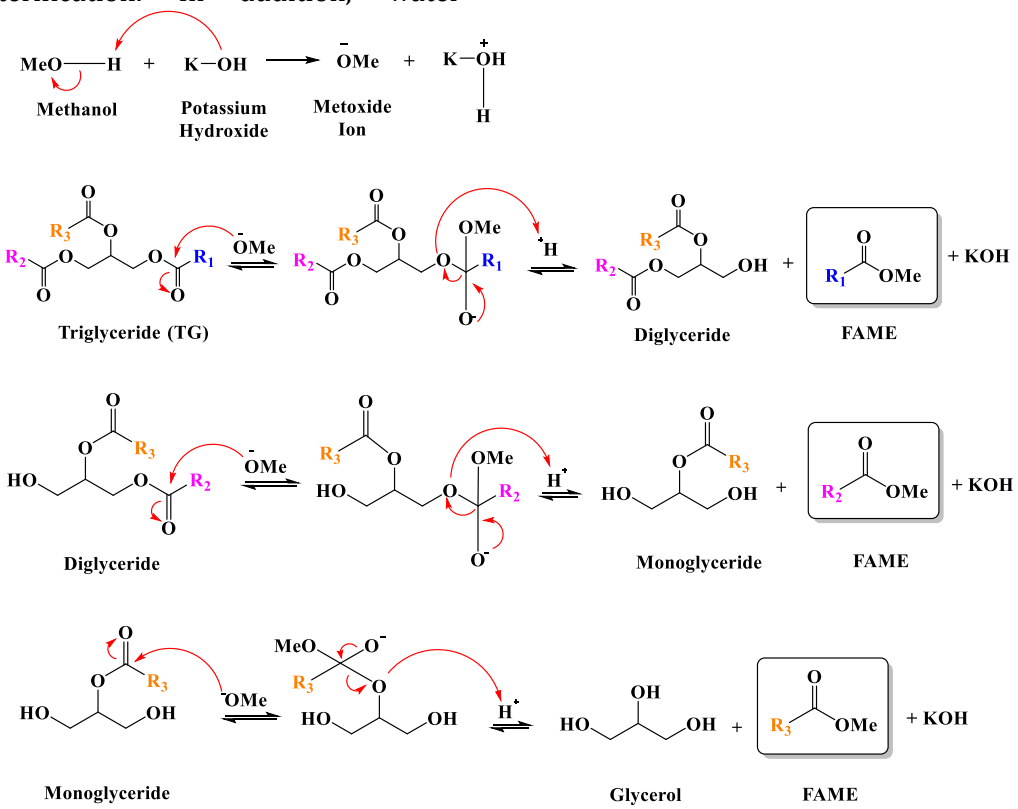

Figure 3. Fatty acid methyl ester synthesis reaction mechanism

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The transesterification reaction between used cooking oil and methanol can be explained through the reaction mechanism shown in Figure 3. The reaction begins with the deprotonation of methanol initiated by the base catalyst potassium hydroxide (KOH), resulting in the formation of methoxide, which acts as a nucleophile. The nucleophile attacks the carbonyl carbon of one of the triglyceride chains, producing one mole of FAME and a diglyceride. This step proceeds through a bimolecular nucleophilic substitution (S_N2) reaction. The reaction continues until three moles of FAME are formed, with glycerol as the by-product.

Variations in the transesterification reaction time of oil with methanol produced FAME with different acid values, as shown in Figure 4. The 90 min reaction produced FAME with the highest acid value, 1.57 mg KOH/g. The acid value decreased with increasing reaction time. However, the acid value increased again at a reaction time of 180 min. This was because the transesterification reaction in this reflux system is reversible [16]. The reaction time that produced FAME with the lowest acid value was 150 min, with an acid value of 0.43 mg KOH/g.

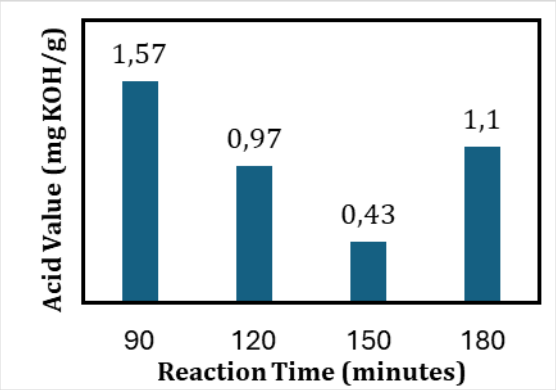

Figure 4. Acid value of fatty acid methyl ester

FAME yield was calculated using Equation (2). The yield obtained is shown in Figure 5. Similar FAME yields were obtained across all reaction-time variations, indicating that reaction time had a limited effect on the overall FAME yield. The difference in yield was strongly influenced by each stage of the synthesis and FAME purification processes. The highest yield was obtained at a reaction time of 150 min.

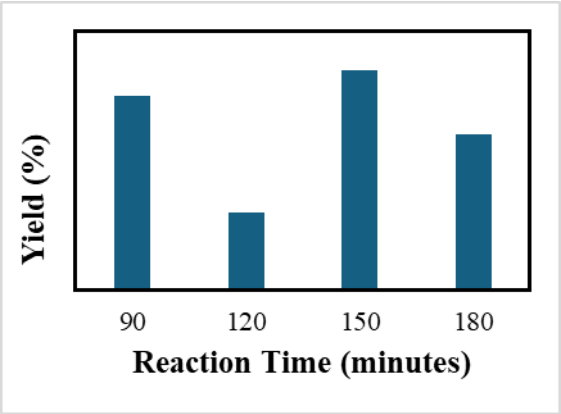

Figure 5. Yield (%) of fatty acid methyl ester

Characterization of Fatty Acid Methyl Ester

The FAME sample with the lowest acid value was preliminarily characterized using TLC, as shown in Figure 6. The TLC results for used cooking oil showed the presence of FFA and TG spots, with approximate *R*f values of 0.53 and 0.11, respectively. After 150 min of reaction, a new spot identified as FAME appeared, with an approximate *R*f value of 0.83. The TG spot disappeared, and the FFA spot decreased. This indicates that FFA decreased, TG reacted completely, and FAME was formed.

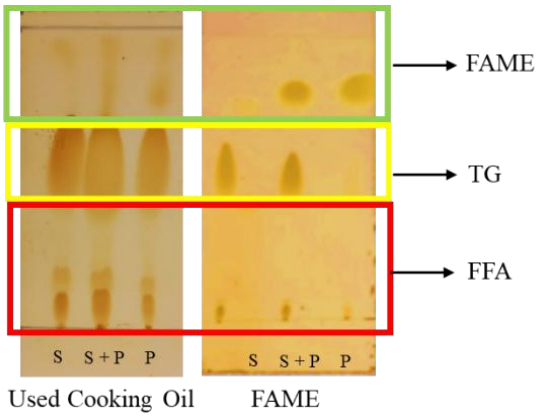

Figure 6. TLC analysis of fatty acid methyl ester

The methyl ester content in FAME was analyzed using GC–MS, as shown in Figure 7 and Table 2.

The analysis results showed five peaks in the chromatogram, indicating the presence of five types of methyl esters in the FAME produced from used cooking oil: methyl myristate (2.86%), methyl palmitate (65.88%), methyl oleate (19.25%), methyl stearate (10.85%), and methyl arachidate (0.10%), along with other impurities (1.06%). The main components of the FAME produced from this used cooking oil were methyl palmitate, methyl oleate, and methyl stearate. The main FAME composition in this study is consistent with the main FAME composition of used cooking oil reported by Hussein et al. (2021) and Zamanhuri et al. (2020) [13], [17].

The mass spectrum of methyl palmitate in Figure 8 shows a peak at *m/z* 74 as the base peak, obtained through the McLafferty rearrangement fragmentation shown in Figure 9 [18].

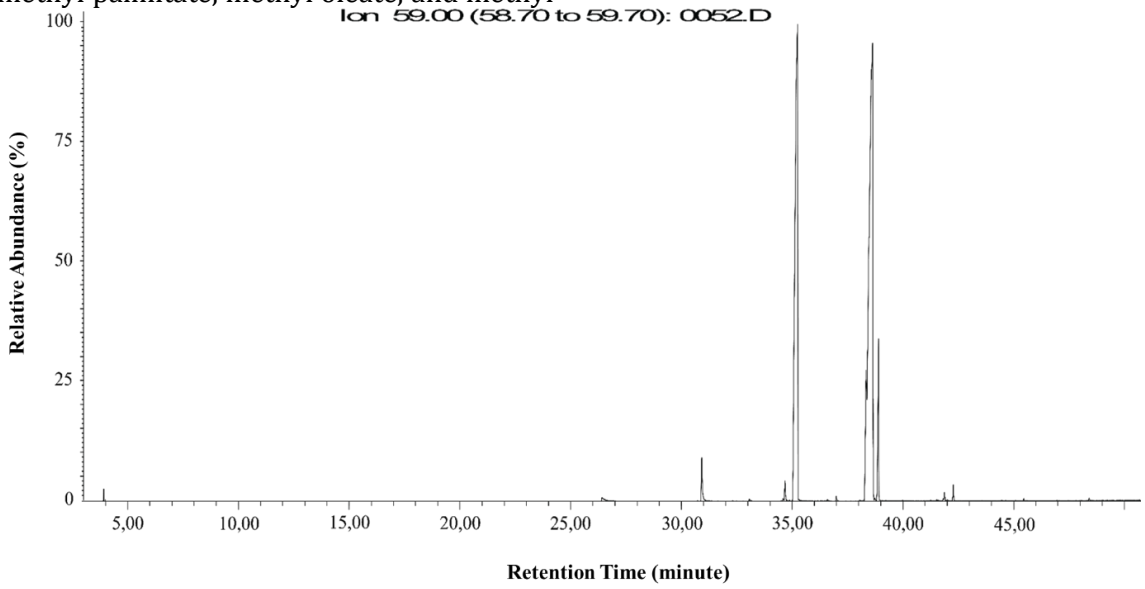

Figure 7. Fatty acid methyl ester chromatogram

Table 2. Fatty acid methyl ester composition

No.	FAME Composition		Molecular Formula	Molecular Mass	Abundance (%)
1	Methyl myristate	C14:0	$C_{15}H_{30}O_2$	242	2.86
2	Methyl palmitate	C16:0	$C_{17}H_{34}O_2$	270	65.88
3	Methyl oleate	C18:1	$C_{19}H_{36}O_2$	296	19.25
4	Methyl stearate	C18:0	$C_{19}H_{38}O_2$	298	10.85
5	Methyl arachidate	C20:0	$C_{21}H_{42}O_2$	326	0.10
6	Others	-	-	-	1.06

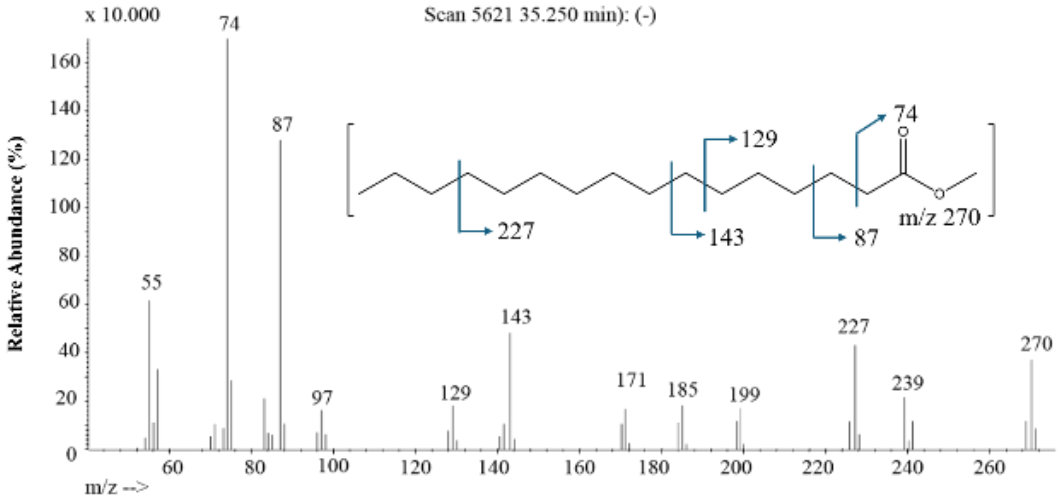

Figure 8. Methyl palmitate mass spectrum

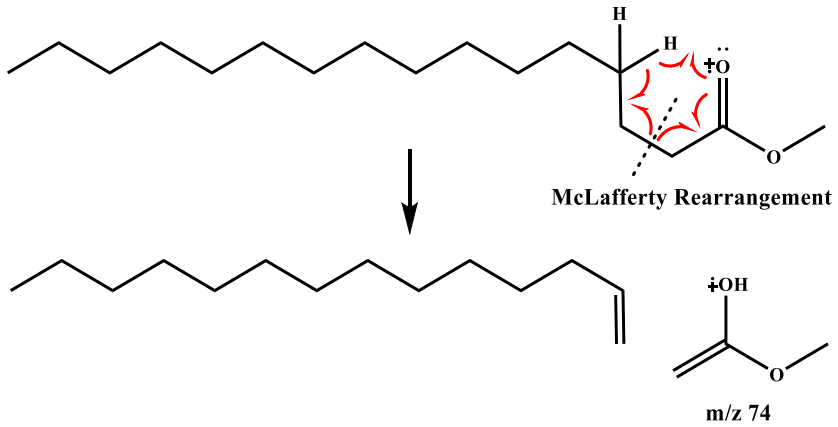

Figure 9. McLafferty rearrangement

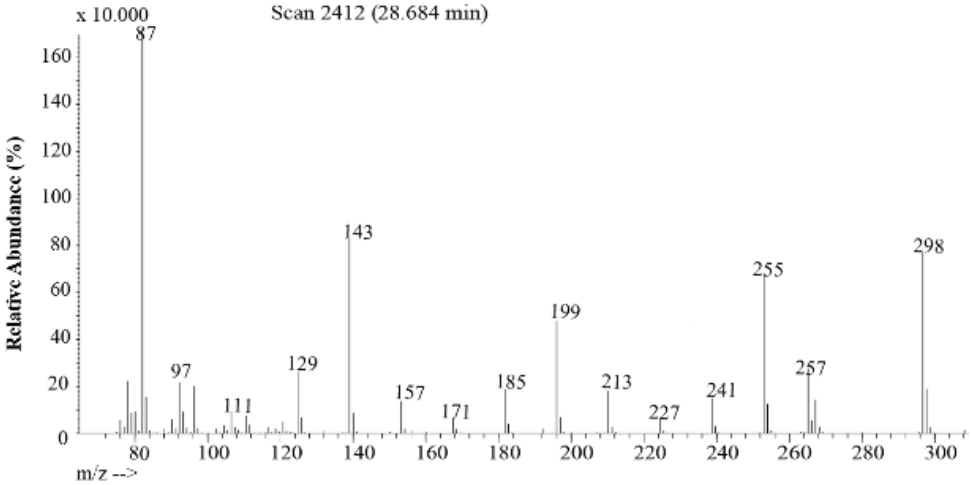

Figure 10. Methyl stearate mass spectrum

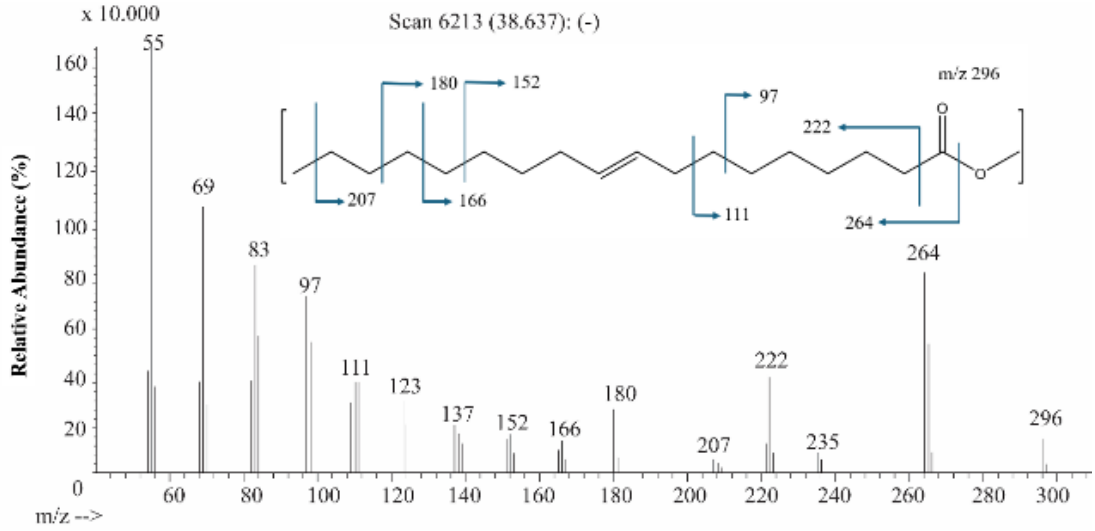

Figure 11. Methyl oleate mass spectrum

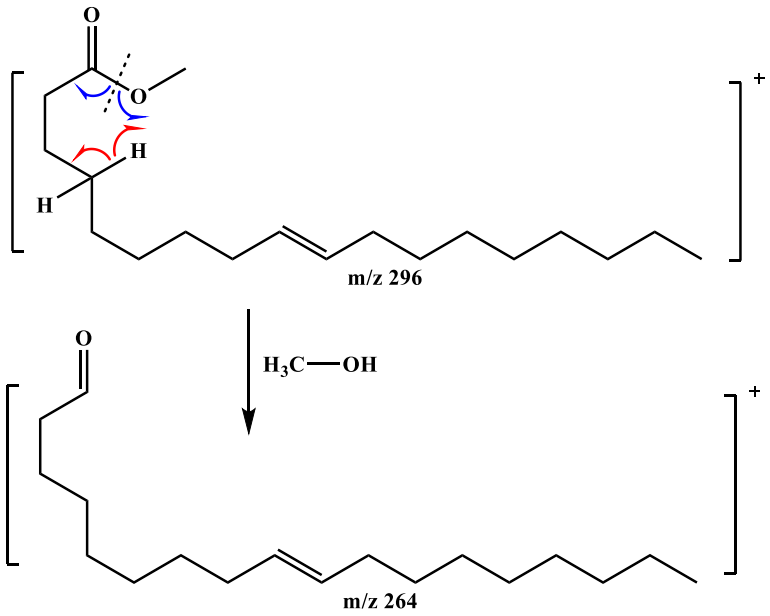

Figure 12. Fragmentation at *m/z* 264

The mass spectrum of methyl stearate in Figure 10 also shows a base peak at *m/z* 74, which originates from the McLafferty rearrangement. Unlike the previous two methyl esters, methyl oleate (see Figure 11) does not show a peak at *m/z* 74 but instead shows a base peak at *m/z* 264. The molecular ion peak at *m/z* 296 $[C_{18}H_{36}O_2]^+$ fragments into ions at *m/z* 264 $[C_{17}H_{33}O]^+$, as shown in Figure 12 [19].

Conclusion

Pretreatment of UCO by distilled-water washing successfully reduced the acid value of UCO from 11.80 mg KOH/g to 3.49 mg KOH/g. FAME was successfully synthesized from UCO and methanol. Variations in FAME synthesis time produced FAME with different acid values. The acid value decreased with increasing reaction time but increased again at 180 min because of the reversible nature of the reaction. FAME composition was identified using GC–MS and showed the presence of five types of

methyl esters: methyl myristate (2.86%), methyl palmitate (65.88%), methyl oleate (19.25%), methyl stearate (10.85%), and methyl arachidate (0.10%). The UCO pretreatment method using distilled-water washing successfully produced FAME with a methyl ester content of up to 98.04%. This treatment method can reduce the cost of large-scale FAME production because the distilled-water washing process is highly economical.

Acknowledgement

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