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Research Article

Synthesis of FAME for Sustainable Aviation Fuel Based on Microwave Irradiation

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Abstract:
The need to reduce greenhouse gas emissions from the aviation industry has increased the search for alternative sustainable fuels. This study suggests a new method of using microwave irradiation to produce fatty acid methyl esters (FAME) based on esterification from waste cooking oil (WCO). FAME can be further converted into Sustainable Aviation Fuel (SAF) through hydrodeoxygenation and isomerization processes. In this study, we aimed to achieve a 95% synthesis ratio within 10 minutes by using microwave-assisted FAME synthesis from waste cooking oil. The research included pre-treatment and neutralization, followed by FAME synthesis under various conditions. FAME yield ratio was measured by GC method. Experimenta1 results showed that microwave irradiation could significantly reduce reaction time and energy use compared to traditional heating methods. The results confirmed that a 95% yield ratio was achieved within 10 minutes. This study highlights microwave-assisted FAME synthesis as a viable way to produce eco-friendly aviation fuels, helping to meet the aviation sector's sustainability goals.

Keywords: *FAME, Sustainable aviation fuel, Microwave irradiation, Synthesis, Waste cooking oil*

1. Introduction

Global energy consumption is increasing year by year, as this consumption grows, the demand for fossil fuels like petroleum, coal, and natural gas also rises. However, this leads to serious problems such as the depletion of fossil fuels and global warming caused by the CO_2 emissions from burning these fuels. In this context, sustainable aviation fuel (SAF) has gained attention as an alternative to traditional fossil fuels. SAF is promising because it can reduce CO_2 emissions, and it's applicable to most current aircraft and engines. SAF is made from vegetable oil, animal fats, waste oils or greases and so on, these are all environmentally friendly raw materials. It is expected that its production will increase worldwide, and the market will expand. One of the reasons for this is the statement made by Japan government on October 26, 2020, declaring that Japan aims to achieve carbon neutrality by 2050[1]. To support this goal, SAF is considered a realistic and effective measure. However, mainstream methods like Fischer-Tropsch process, Hydrothermal Liquefaction and so on are complex and time-consuming [2]. So in this research, we attempt to solve this issue by using microwave irradiation. The potential applications of microwave heating in chemical industry processes are increasingly gaining attention, such as in the treatment of various waste streams, mineral processing, and energy production. Unlike traditional heating methods, microwave heating works by causing the dipole molecules inside the material to oscillate at high frequencies, generating "internal frictional heat" that raises the temperature of the material. This process eliminates the need for any heat conduction, Due to its unique heating characteristics, such as uniform bulk-phase heating and both thermal and non-thermal effects, microwave heati technology has been widely applied in laboratory scale intermittent and some batch production synthesis.

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Besides, specific microwave effects are those effects that cannot be (easily) emulated through conventional heating methods. Examples include: (i) selective heating of specific reaction components, (ii) rapid heating rates and temperature gradients, (iii) the elimination of wall effect [3]

In previous studies, we have focused on synthesize Bio-Diesel Fuel (BDF) by using ultrasonic and microwave methods. For virgin oil, it was found that the BDF yield ratio could reach 95% in a short time such as 10 mins. Besides, applying microwaves to BDF synthesis is regarded as a new method to increase BDF yield ratio. Unlike conventional heating methods, microwaves can heat the inside and outside of a heated object almost uniformly by a phenomenon called dielectric heating [4]. Expected benefits include significantly shortened reaction times, low-cost processes, and power savings [5]. Ikenaga et al. reported that a microwave BDF synthesis using lead oxide as a catalyst achieved a BDF yield ratio of about 90% [6] using homogeneous catalytic method.

However, it is not recommendable to use virgin oil to produce SAF, instead waste cooking oil (WCO) and other inedible vegetable oils are the best choices in terms of cost performance, Unlike virgin oil, waste cooking oil contains more free fatty acids, more water, and impurities that require pre-treatment to remove. Since there are few reports on using microwave irradiation to produce FAME, which is a key intermediate for SAF production from WCO, therefore in this study, we aimed to achieve a 95% synthesis rate within 10 minutes by using microwave-assisted FAME synthesis from waste cooking oil. FAME can later be possibly converted into SAF through isomerization processes and hydrodeoxygenation with less energy, which are beyond the scope of this study. The research included pre-treatment and neutralization, followed by FAME synthesis under various conditions. The results confirmed that a 95% synthesis rate was achieved within 10 minutes.

2. Principle of Experiment

2.1 SAF synthesis principles

Based on the chemical reaction equation shown in Fig. 1, fatty acid methyl ester (FAME) is synthesized by transesterifying waste cooking oil (triglycerides) with lower alcohols such as ethanol or methanol.

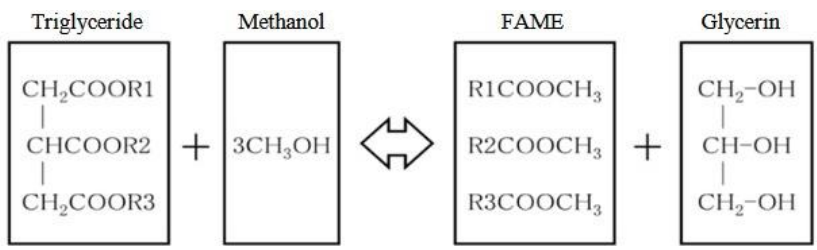

Fig. 1. The Chemical Reaction of FAME.

However, there are two important points to consider before synthesizing FAME. First, waste cooked oil contains a certain amount of water, so it is necessary to remove this water before starting the synthesis. If water remains, the transesterification reaction won't occur as expected, and instead, a saponification reaction might take place, leading to the formation of soap, as shown in Fig. 2. Second, compared to fresh oil, waste cooked oil contains more free fatty acids, which can hinder the transesterification reaction. To neutralize these acids, it's necessary to increase the amount of alkaline catalysts used. [7]

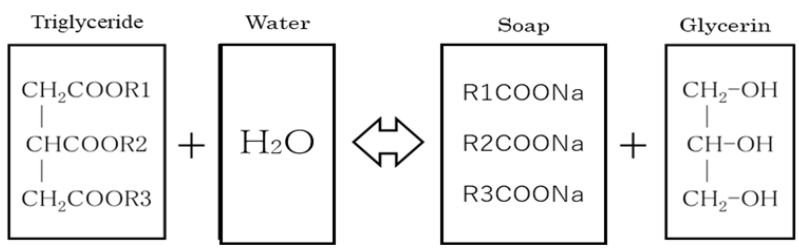

Fig. 2. The Formation of Soap.

Additionally, alkaline catalysts are known to be effective in ester reactions, making them widely used in FAME synthesis. Fig. 3 shows the nucleophilic attack by a homogeneous alkaline catalyst. The methoxide ion, which is a catalyst component, attacks the carbonyl carbon of the vegetable oil, forming a tetrahedral intermediate that eventually produces fatty acid alkyl esters (SAF) and a glycerate anion. This anion reacts with alcohol to form diglycerides and methoxide ions. The methoxide ions can then be reused in the transesterification reaction, converting diglycerides to monoglycerides, and eventually, monoglycerides to glycerol. [8]

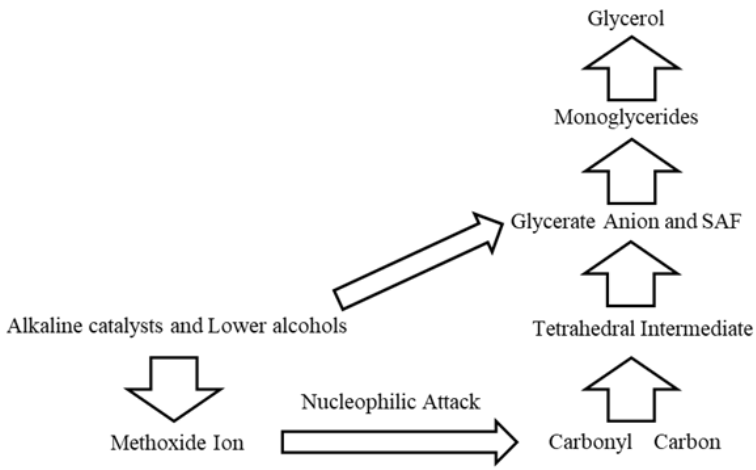

Fig. 3. Nucleophilic Attack by Homogeneous Alkaline Catalyst

2.2 Advantages of Microwave Technologies in FAME Synthesis

In the conventional FAME production methods, the reaction process typically takes 30 minutes to an hour, while the entire production process can take several hours or even days. To enhance production efficiency, microwave irradiation technologies have been proposed as more efficient methods for FAME synthesis. Unlike traditional heating methods that rely on external heat sources, microwave heating causes the object itself to generate heat. This brings several notable advantages. [9]

(1) Shorter Heating Time: Microwave heating directly heats the interior of the object, avoiding the slow temperature rise at the center seen in conventional heating, significantly reducing heating time.
(2) Uniform Heating: Microwaves can uniformly heat objects with complex shapes, preventing issues like overheating the exterior while the interior remains insufficiently heated, common in traditional heating methods.
(3) High Thermal Efficiency: Since microwaves primarily heat the object itself rather than the surrounding air or equipment, the thermal efficiency is much higher than that of traditional methods.
(4) Easy Control of Heating Power: The control of microwave heating power is very convenient, with power adjustments achievable via remote signals. Additionally, the rapid response of microwave heating control, with no heat lag, makes it suitable for automated production processes.

Also, In the transesterification reaction for FAME preparation, alcohols have a strong polarity and are easily absorbed by microwave radiation, causing the dipoles to accelerate, move, and generate heat through friction. The rapid interaction between microwave energy and alcohol molecules generates microscopic heat in a short time, causing the alcohol to quickly heat up and reach its boiling point. In contrast, the longer fatty acid carbon chains in oils have weaker polarity, which means they hardly absorb microwaves. Therefore, in the ester exchange reaction between oils and alcohols, microwaves have a directional focusing effect, creating sustained localized overheating, which accelerates the catalytic reaction rate and effectively improves the FAME production efficiency. [10]

2.3 The influence of parameters on the synthesis reaction

The use of base catalysts for FAME synthesis is a common method, particularly with sodium hydroxide and potassium hydroxide. A suitable amount of base catalyst offers several advantages, such as mild reaction conditions and high catalytic activity, which can achieve high yield and reaction rates in a short period. However, when using waste

cooking oil as a feedstock, base catalysts are highly sensitive to the presence of free fatty acids (FFA) and water. Excessive use of base catalysts may lead to side reactions such as saponification, necessitating additional steps for separating catalytic impurities, ultimately increasing production costs. [11,12,13,14]

The amount of alcohol has a critical impact on FAME synthesis. Insufficient alcohol can prevent the transesterification reaction from proceeding, resulting in an incomplete reaction. Conversely, excessive alcohol usage may lead to a high content of unreacted alcohol in the synthesized FAME, ultimately reducing the FAME synthesis yield. [15]

The reaction temperature is a key factor influencing the yield of FAME production. Increasing the temperature can enhance the reaction rate by reducing the viscosity of the oil. However, if the temperature exceeds the optimal range, FAME production may decrease due to the accelerated saponification of triglycerides at higher temperatures. Typically, the transesterification reaction temperature should be lower than the boiling point of the alcohol to prevent its evaporation. The optimal temperature for FAME production generally falls within the range of 50 °C to 60 °C, depending on the type of catalyst and oil used. [16]

2.4 The properties of SAF and conventional Jet fuel

Main properties for SAF and conventional jet fuel are shown in Table 1. Conventional jet fuel follows the ASTM D1655 standard, while Sustainable Aviation Fuel (SAF) adheres to the ASTM D7566 standard. ASTM D1655 primarily covers conventional petroleum-based aviation turbine fuels, such as Jet A and Jet A-1, while ASTM D7566 covers SAF and other certified synthetic fuels, primarily including alternative fuels produced from renewable feedstocks (such as plant oils, waste fats, biomass, etc.) or synthetic processes. The D7566 standard allows certain types of synthetic fuels, after certification, to be considered equivalent in performance to D1655 fuels and, upon necessary certification, to be reclassified as D1655 fuels for use in existing aviation fuel infrastructure and aircraft. [17,18]

Table 1: The Properties for SAF and Conventional Jet Fuel

Property	**ASTM D7566 (SAF)**	**ASTM D1655 (Jet Fuel)**
COMPOSITION		
Acidity, total mg KOH/g, max	0.1	0.1
Aromatics: One of the following requirements shall be met:		
1.Aromatics, volume percent, max	25	25
2.Aromatics, volume percent, max	26.5	26.5
Sulfur, mercaptan, mass percent	0.003	0.003
Sulfur, total mass percent	0.3	0.3
Distillation temperature,ºC:		
10% recovered, temperature(T10), max	205	205
Final boiling point, temperature, max	300	300
Distillation residue, percent, max	1.5	1.5
Distillation loss, percent, max	1.5	1.5
Flash point,ºC, min	38	38
Density at 15ºC,kg/m^3	775 to 840	775 to 840
FLUIDITY		
Freezing point, ºC, max	-40 Jet A-1	-40 Jet A-1
viscosity -20ºC, mm^2/s,Max	8	8
COMBUSTION		
Net heat of combustion,MJ/kg, min	42.8	42.8
CORROSION Copper strip,2h at 100ºC, max	No. 1	No. 1
Lubricity, mm, max	0.85	0.85
Viscosity -40ºC, mm^2/s, max	12	12

3. Experiment

3.1 The pre-treatment and neutralization of waste cooking oil

Compared to virgin oil, waste cooking oil contains more free fatty acids and moisture, both of which can hinder the conversion process to FAME. Therefore, pre-treatment is necessary before using waste cooking oil for FAME production.

As for impurities-removing, filter was employed in the study.

While simple high temperature heating can remove the water of waste cooking oil, it may increase the free fatty acid content. In this experiment, the filtered waste oil was heated at 100ºC for 15 minutes, then was placed overnight to separate the oil and water.

For neutralizing waste oil, the catalyst amount was quantitively increased based on the pH value measured. 1 ml of settled oil with 10ml of isopropanol was mixed and stirred until the oil dissolves. Then, 0.1%NaOH solution was added until the pH reaches between 8 and 8.5, which helps determine the required catalyst amount for neutralizing 1 ml of oil. Finally, the NaOH amount needed for the entire experiment was calculated based on the oil volume.

3.2 FAME Synthesis Experiment and Conditions

A schematic of the microwave FAME synthesis system is shown in Fig. 4. Before microwave heating, NaOH must dissolve in methanol first. Then, the vegetable oil and methanol mixture are placed in a test tube and emulsified using a magnetic stirrer. The test tube is then placed in a microwave chemical reaction apparatus (Biotage's Initiator+) (Fig. 5) for heating, where the transesterification reaction takes place. The microwave chemical reaction apparatus can operate within an output range of 0-400 W using a 2.45 GHz magnetron. This device also supports precise temperature control over a range of 40-300°C. The heating rate can be adjusted between 2-5°C/sec, depending on the solvent and reaction conditions. The system specifications of the apparatus will show in Table 2.

The specific conditions for the FAME synthesis experiment are listed in Table3. In the microwave-assisted FAME synthesis experiments, only one reaction parameter, such as heating time, temperature of heat, NaOH content(catalyst), or methanol content, is adjusted in each experiment.

3.3 Verification of plain tube FAME Yield Ratio Measurement

In regard to the FAME yield ratio measurement, a Gas chromatography–mass spectrometry (GCMS, Shimadzu GCMS QP2020) shown in the Fig. 6 is employed to measure the yield ratio of FAME. In the measurement, a standard sample and FAME were quantitatively analyzed. As the analysis time progressed, various constitutions of FAME fatty acid methyl were detected, from which the FAME synthesis rate was calculated from the peak areas of the standard sample and FAME. The measurement conditions are shown in Table 4 and the formula for calculating the yield ratio is shown in equation (1). Also, for yield ratio measurement results, at least 3 experiments were repeatedly conducted for the same experimental conditions and all the experimental results shown in the following section were the average ones.

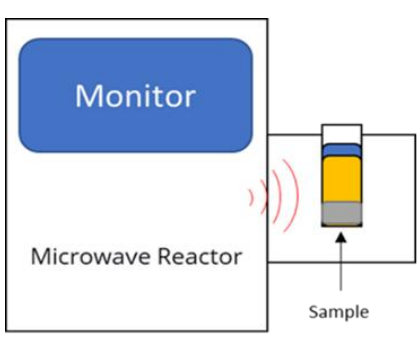

Fig. 4. Schematic of Microwave FAME Synthetic System

Fig. 5. Biotage's Initiator+

Table 2: System Specifications of Biotage's Initiator+

Model Name	Biotage's Initiator+
Temperature Range	40-300°C
Heating Rate	2-5°C/sec (varies with solvent and reaction conditions)
Maximum Irradiation Time	96 hours
Pressure Range	0-30 bar
Output Range	0-400 W (2.45 GHz magnetron)
Reaction Volume Range	0.2-20 mL
Power Supply	100-120V, 50/60 Hz (10 A)
Maximum Power Consumption	1100 VA

Table 3: FAME synthesis conditions

Tem. (℃)	Time (min)	WCO (ml)	Methanol (ml)	NaOH(Catalyst) (g)
100	10	4	1.5	0.07
100	10	4	1.5	0.06
100	10	4	1.5	0.05
100	10	4	1.5	0.04
100	10	4	1.5	0.03
100	10	4	1.5	0.02
100	10	4	0.5	0.06
100	10	4	1	0.06
100	10	4	2	0.06
100	2	4	1.5	0.06
100	4	4	1.5	0.06
100	6	4	1.5	0.06
100	8	4	1.5	0.06
40	10	4	1.5	0.06
60	10	4	1.5	0.06
80	10	4	1.5	0.06
120	10	4	1.5	0.06

Table 4. GCMS conditions.

Column Length (m)	30m
Column I.D. (mm)	0.25mm
Column Flow Rate (ml/min)	1.8ml/min
Carrier gas pressure (kPa)	100.2
Injector temperature (℃)	250℃
Detector temperature (℃)	250℃
Standard sample	Methyl Heptadecanoate

$$E = \frac{\sum A - A_{is}}{A_{is}} \times \frac{m_{is}}{m} \times 100 \quad (1)$$

The fatty acid methyl ester content E (yield ratio) obtained by GCMS is calculated by using Equation (1). Here, ∑A represents the total sum of the peak areas of the fatty acid components, A_{is} is the peak area of the internal standard, and m represents the mass of the fatty acid.

Fig. 6. Gas chromatography–mass spectrometry (GCMS, Shimadzu GCMS QP2020)

4. Results and Discussion

4.1 Effect of catalyst content on FAME yield ratio

The yield ratio of FAME was measured as the NaOH content as catalyst varied during the synthesis process. The catalyst content used is 0.02 g, 0.03 g, 0.04 g, 0.05 g, 0.06 g and 0.07 g while reacting temperature, reacting time, WCO and methanol was fixed to be 100℃, 10 min, 4 ml and 1.5 ml, respectively. The results are illustrated in Fig. 7, At a catalyst content of 0.07 g, the yield ratio was the highest, reaching 95.6%. As the catalyst content decreased to 0.06 g, the yield dropped to 90.5%. With further reductions to 0.05 g and 0.04 g, yields were relatively stable at 91.5% and 91.3% respectively. However, at lower catalyst levels of 0.03 g and 0.02 g, the yield significantly decreased to 80.1% and 75.9%. These results emphasized the importance of sufficient catalyst content for optimal FAME production.

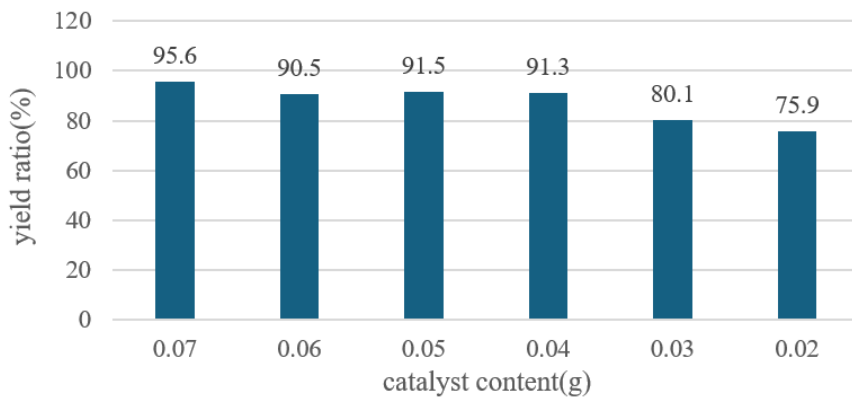

Fig. 7. FAME Yield Ratio by Changing Catalyst Content.

4.2 Effect of methanol content on FAME yield ratio

The impact of varying methanol content on the FAME yield ratio is illustrated in Fig. 8. The methanol content used is 0.5 ml, 1 ml, 1.5 ml and 2 ml while reacting temperature, reacting time, WCO and catalyst was fixed to be 100℃, 10 min, 4 ml and 0.06 g, respectively. The yield ratio west at 55.1% when using 0.5 ml of methanol. Increasing the methanol content to 1 ml resulted in a significant improvement, raising the yield to 84.2%. The highest yield was observed at 1.5 ml, reaching 90.5%. Since transesterifying reaction is a reversed reaction, with enough methanol, transesterifying reaction is possibly to produce FAME efficiently. However, further increasing the methanol content to 2 ml caused a decline in yield to 69.4%. These results indicate that while an optimal amount of methanol can enhance FAME production, excess methanol can have a detrimental effect.

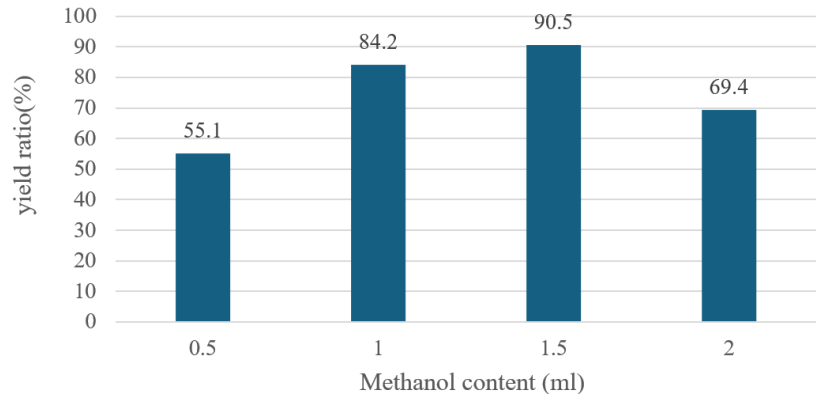

Fig. 8. FAME Yield Ratio by Changing Methanol Content.

4.3 Effect of heating time on FAME yield ratio

Fig. 9 shows the influence of varying heating times on the FAME yield ratio. The heating time used is 2 min, 4 min, 6 min, 8 min and 10 min while reacting temperature, WCO, methanal and catalyst was fixed to be 100℃, 4 ml, 1.5 ml and 0.06 g, respectively. The yield was lowest at 52.6% with a heating time of 2 minutes. As the heating time increased to 4 minutes, the yield improved significantly to 81.8%. The highest yield of 91.6% was achieved at 6 minutes. Generally speaking, the longer the reaction time, the more SAF will be produced. However, extending the heating time further led to a decrease in yield, with 70.1% at 8 minutes and 90.5% at 10 minutes. These results suggest that there is an optimal heating time to maximize FAME yield, under or beyond which the SAF yield ratio may drop.

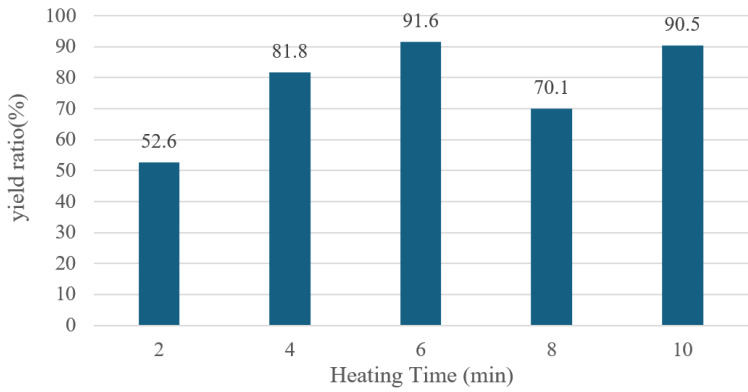

Fig. 9. FAME Yield Ratio by Changing Heating Time.

4.4 Effect of temperature of heat on FAME yield ratio

Fig. 10 shows the impact of different temperatures of heat on the FAME yield ratio. The reacting temperature used is 40℃, 60℃, 80℃ and 100℃ while reacting time, WCO, methanal and catalyst was fixed to 10 min, 4 ml, 1.5 ml and 0.06 g, respectively. The highest yield of 90.5% was achieved at 100ºC, while the lowest yield was 70% at 60ºC. At 40ºC, the yield ratio was 88.3%, and it slightly decreased to 87.3% at 120ºC. Interestingly, the yield dropped to 72.7% at 80ºC, indicating that 100ºC is the most effective temperature for maximizing FAME production. The irregular FAME yield ratio may be influenced by the changed dielectric loss efficient that is related to the changed reaction temperature, which led to a changed FAME yield ratio.

4.5 Discussion

According to literature review, a 2011 paper using traditional heating and stirring methods achieved biodiesel yields of 87.4%, 89%, and 88.3% under optimal conditions (methanol/oil molar ratio of 6:1, catalyst concentration of 1.25%,

65°C, 2 hours, 150 rpm). A 2022 study also used traditional heating but with a high-temperature treated scallop shell catalyst soaked in methanol for 4 hours as the catalyst, yielding nearly 90%. A 2008 study using palm kernel oil achieved a 96% yield with optimized ethanol to oil ratio, 60°C, 120 minutes, and 1% KOH catalyst. [19,20,21]

By comparing the reported yield ratios and reaction time with those obtained in this paper, it was confirmed that by using microwave the reaction time could be drastically decreased. As for yield ratio, since the physical properties of the WCO collected are little bit different with each other due to the different situation where virgin oil was used, the final yield ratio of FAME would always appear to be the different values. This would be the main reason for the uncertainty of the FAME synthesis experiment. Although FAME itself does not meet ASTM D7566 specifications, achieving a high FAME yield is crucial because it serves as a feedstock for subsequent hydrodeoxygenation and isomerization to produce SAF. Therefore, microwave-assisted FAME synthesis can be considered a promising upstream process for the future large-scale production of SAF.

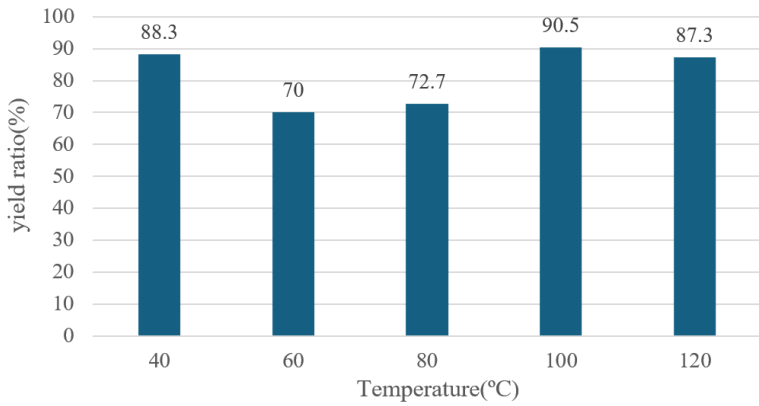

Fig. 10. FAME Yield Ratio by Changing Temperature of Heat.

4.6 Cost of this research

All costs are calculated using the maximum values for estimation. The machine's maximum output is 0.4 kWh, and the electricity base cost is 31 yen/kWh, resulting in an electrical cost of 2.06 yen per experiment lasting 10 min. For the alkaline catalyst costs, 500g of sodium hydroxide costs 1000 yen, and each experiment only uses 0.05 g to 0.07 g, amounting to approximately 0.1 to 0.14 yen. In Japan, the price for collected WCO is zero, so the cost for 5ml is 0 too. Regarding methanol, 1000ml costs 500 yen, and each experiment uses 1 ml to 2 ml, equivalent to a cost of approximately 0.5 to 1 yen. In conclusion, the total cost per experiment is approximately 3.2 yen, which is equivalent to around 0.02 USD.

5. Conclusion

In this research, microwave irradiation was applied to FAME synthesis from WCO. As a series of experiments under different conditions, some conclusions can be obtained as follows:

(1) Synthesis reaction time is less than that of conventional heating method:
(2) The highest FAME yield of 95.6% was achieved with a catalyst content of 0.07 g;
(3) Other conditions for FAME yield ratio over 90% are listed as follows:
   1) Heating at 100°C for 10 minutes with 1.5 ml of methanol and catalyst amounts of 0.04 g, 0.05 g, 0.06 g, 0.07 g
   2) Heating at 100°C for 6 minutes with 1.5 ml of methanol and 0.06 g of catalyst.

As for our future work, a new flow-type microwave reactor is under construction by which continuous mass production of FAME can be possibly realized and FAME yield ratio will be measured.

Acknowledgments

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