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## ARTICLE

### Synthesis of biodiesel via transesterification from waste fish oil and its application to a diesel agricultural engine

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#### ABSTRACT

This study addresses the growing need for sustainable energy sources by introducing a novel method for turning waste fish oil (WFO) into biodiesel. Applying sodium hydroxide (NaOH) and methanol as catalysts in the transesterification process, we achieved a conversion rate of over 96.5% in biodiesel synthesis from waste frying oil (WFO). The fatty acid methyl ester (FAME) produced was thoroughly analyzed using FT-IR and <sup>1</sup>H-NMR spectroscopy, confirming the successful conversion and molecular structure. The fuel exhibited physicochemical characteristics that meet the requirements of existing biodiesel standards (ASTM D-6751 and EN14214) and are similar to those of high-speed diesel. These features include a kinematic viscosity of  $3.05 \pm 0.02$  cSt at 40°C, a density of 0.872 g/cm<sup>3</sup>, and an acid value of 0.07 mg KOH/g. The effective use of biodiesel in a diesel farm engine highlights its potential for practical use, as it reduces waste and promotes sustainable fuel choices in the agricultural industry.

## 1. Introduction

The primary energy source worldwide mainly depends on fossil fuels, which are such as coal and crude oil, associated with significant environmental concerns such as air pollution, greenhouse gas emissions, waste disposal, and climate change (Dussadee et al., 2017; Pimpimol et al., 2022). These problems, namely the substantial carbon

footprint linked to transportation and related sectors, present a major risk to environmental well-being (Dussadee et al., 2022). As a reaction, there has been a significant transition towards sustainable energy sources, such as biofuels, which are increasingly acknowledged as essential in tackling the worldwide climate emergency (Junluthin et al., 2021; Saengsawang et al., 2022). Renewable energy production, which encompasses biofuels, is experiencing significant and continuous

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growth as the need to substitute traditional fuels becomes more intense (Kaewdiew et al., 2019; Kongchan et al., 2022). The quest for sustainable energy solutions has prompted the investigation of several alternatives that seek to mitigate the ecological consequences of fossil fuels and meet the growing worldwide energy demands. Fossil hydrocarbons are not only finite and diminishing but also expensive to extract. Consequently, there is an increasing fascination with utilizing vegetable oils and biomass to produce liquid biofuels. This transition signifies a viable and long-lasting option that has stimulated extensive investigation aimed at minimizing manufacturing expenses, lowering the release of greenhouse gases, and maximizing the efficient utilization of land and water resources (Inkerd et al., 2015).

Biodiesel is a liquid fuel that is a renewable energy and biofuel to diesel fuel derived from petroleum sources. It can be mixed with diesel used in diesel engines (Homdoun et al., 2020). The advantages of biodiesel are biodegradability, low emission of greenhouse gases such as CO, CO<sub>2</sub>, NO<sub>x</sub>, and SO<sub>x</sub>, excellent lubricity, low toxicity, and environmentally friendly. As a renewable and biodegradable fuel with a far lower environmental impact than conventional petroleum-based diesel, biodiesel has become an essential sustainable substitute. This eco-friendly fuel significantly reduces greenhouse gas emissions and minimizes the harmful effects of traditional diesel exhaust. With the rise in global energy consumption due to industrial expansion and population growth, it is crucial to shift towards renewable energy sources in order to address climate change and maintain environmental sustainability (Tongsiri et al., 2023). Biodiesel, which can be used with the current diesel infrastructure, is vital in this transition by reducing carbon emissions and improving global energy security.

The investigation of raw materials for biodiesel manufacturing is comprehensive, focusing on accessible resources such as vegetable oils, waste cooking oil, and animal fats, which have been historically used. Although traditional feedstocks are efficient, they pose several obstacles, such as high expenses, limited scalability, and ethical issues related to their impact on food availability (Albatayneh, 2023). These challenges have stimulated the pursuit of more sustainable and morally unequivocal alternatives. WFO, a residual product of the worldwide fish processing sector, presents a hopeful resolution within this framework. Fish, consumed globally due to its high nutritional value containing omega-3 fatty acids and vital vitamins, produces a considerable amount of byproducts, particularly WFO. Historically, the disposal of WFO has presented severe environmental difficulties.

The utilization of WFO in biodiesel production tackles the problem of waste disposal and significantly contributes to environmental management by transforming industrial waste into valuable biofuel. This practice demonstrates the core principles of the circular economy by improving the efficiency of resources and offering a sustainable solution to the waste generated as a result. The potential of WFO in the manufacture of biodiesel is substantial. However, the current research literature identifies several areas that require further investigation, namely, enhancing the production process's efficiency and improving the effectiveness of catalysts in the transesterification process.

Transesterification is the chemical process used in biodiesel production to convert oil triglycerides into fatty acid methyl esters (biodiesel) and glycerol. This process requires the use of an alcohol such as methanol and a catalyst. Various scientific and technological improvements have been applied to improve the efficiency and

sustainability of biodiesel production. Sodium hydroxide (NaOH) is a notable catalyst choice because it is cost-effective and can efficiently promote the reaction at moderate settings. This reduces energy consumption and improves the environmental impact of biodiesel synthesis. The biodiesel produced from transesterification reaction from WCO, animal oil, and vegetable oil is the attention of source raw materials. Fish is widely consumed food around the world and has high nutritional value of omega-3 fatty acids, Eicosatetraenoic acid (EPA), Docosahexaenoic acid (DHA), Vitamins (D and B2 riboflavin) and is rich in calcium and phosphorus. Therefore, the different components of fish were used for medicinal consumption, human consumption, and other purpose.

A thorough examination of the physicochemical characteristics of biodiesel, including kinematic viscosity, density, acid value, cloud point, and pour point, is crucial for evaluating its appropriateness for diesel engine use and guaranteeing adherence to strict global fuel regulations. However, some of the fish parts are discarded, such as the caudal fin, anal fin, pelvic fin, dorsal fin, eye, and WFO. The WFO is a raw material in the transesterification reaction. The reaction between a triglyceride in raw materials and alcohols, mostly with methanol [9]. Zabeti et al. (2009) studied biodiesel reaction without a catalyst, but it requires a high temperature of 250 – 400 °C and 35 – 60 MPa pressure. Common catalysts are NaOH or potassium hydroxide (KOH), which are low-cost and readily available. In addition, it activates the reaction in weak conditions (Kushwaha et al., 2023). Wong et al. (2023) reported that the raw material as a cooking oil with higher free fatty acid 1 wt% and sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) was used to accelerate the reaction.

Nawaz et al. (2023) utilized vegetable oil as raw material, which contains higher free fatty acids, about 6 wt%. However, the homogeneous catalysts make it challenging to separate the glycerine phase product, biodiesel, and wastewater purification to remove the catalyst from biodiesel. Recently, NaOH or KOH has been a common catalyst widely used in the transesterification reaction of biodiesel from vegetable oil, soybean oil, and waste frying oil. In 2011, potassium hydroxide/bentonite was used as a catalyst for the transesterification reaction of palm oil to biodiesel (Kushwaha et al., 2023). The present work uses WFO as a raw material in the transesterification process. The physicochemical properties of the resulting biodiesel were characterized by kinematic viscosity, density, acid value, cloud point, and pour point, which were employed to investigate the fuel quality generated.

This study investigates the utilization of NaOH in the transesterification of WFO, with the primary objective of optimizing the procedure to attain biodiesel of superior quality. It seeks to offer an in-depth understanding of the viability of WFO as a raw material for biodiesel production and the effectiveness of NaOH as a catalyst. The findings of this research will contribute to expanding the use of WFO in the biofuel sector. Through this investigation, we aim to contribute substantially to renewable energy, specifically biodiesel production. The study addresses the current deficiencies in utilizing WFO and emphasizes this strategy's environmental and economic advantages. This research has the potential to develop a biodiesel manufacturing process that is both sustainable and economically feasible. This might promote the widespread use of biodiesel and contribute to the global shift towards a more sustainable energy system.

## 2. Materials and methods

### 2.1 Materials

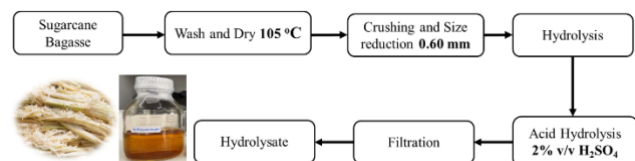
Waste fish oil (WFO) has an acid value of 0.07 mg KOH g<sup>-1</sup> and was purchased from commercial sources in the Thai market. Sodium hydroxide (NaOH) and Potassium hydroxide (KOH) were purchased from Fluka. Methanol (99.5%), acetone (99%), and hexane (99%) were obtained from Fluka.

### 2.2 Sample preparation

The waste fish was cooked at 100 °C for 30 minutes, resulting in a liquid and solid slurry mixture. The slurry was then precipitated for 1 h using Earth's gravity, and the supernatant solution was filtered into a basket, giving rise to a liquid-phase raw material.

### 2.3 Transesterification reaction

Biodiesel was synthesized in a three-neck round bottom batch reactor equipped with a condenser for cooling and a thermocouple to check temperature. The reaction conditions were designed by mixing methanol to waste fish oil with a molar ratio of 12: 1 and 1 wt.% catalyst to oil and stirring continued at 300 rpm, 65 °C for 1 h. After the reaction, the mixture was separated into biodiesel and glycerin using the reported method (Rochat et al., 2016). To monitor the reaction, 0.5 – 1.0 mL of the reaction mixture was collected at 0 min, 10 min, 30 min, and 1 h for sample analysis.



**Figure 1.** Flow diagram of the experiments for biodiesel synthesis by Methanolysis using NaOH common catalyst

## 3. Results and Discussion

### 3.1 Investigation of transesterification mechanism

The transesterification reaction mechanism is an essential and crucial stage in manufacturing biodiesel. Scientists have suggested a potential mechanism for the transesterification reaction, which occurs through a common nucleophilic substitution reaction (Ramaraj et al., 2016; Saengsawang et al., 2020). This procedure frequently employs NaOH as a catalyst. The reaction is initiated by NaOH, which acts as a proton acceptor, removing a proton (H<sup>+</sup>) from methanol and producing a methoxide anion (CH<sub>3</sub>O<sup>-</sup>). The methoxide anion functions as a potent nucleophile.

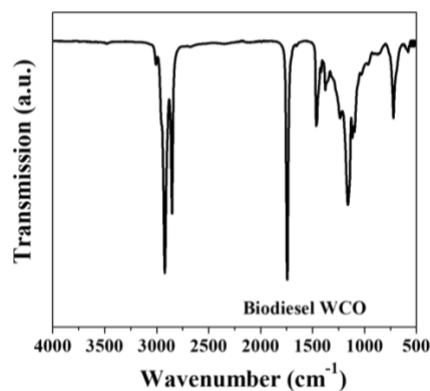
The methoxide anion subsequently reacts with the electrophilic carbonyl carbon group in the triglycerides, resulting in the predominant production of biodiesel and the minor creation of glycerol. Thus, forming the methoxide anion is the crucial step in this mechanism (Waidee et al., 2018). The hypothesized mechanism and the crucial role of the methoxide anion were extensively examined and confirmed based on the documented literature, guaranteeing the precision and

dependability of the transesterification process. The comprehensive comprehension of the transesterification mechanism highlights the significance of the nucleophilic substitution reaction in transforming waste oils into biodiesel, showcasing the effectiveness of NaOH as a catalyst in this procedure.

### 3.2 Fourier transform infrared (FT-IR) analysis

The FTIR spectra of the biodiesel sample obtained from waste fish oil, which included an excess of methanol, displayed multiple distinctive IR bands, suggesting the existence of specific functional groups linked to biodiesel. The presence of aliphatic hydrocarbons was indicated by observing the C-H stretching peak within the 2700 - 2900 cm<sup>-1</sup> range. The existence of hydrocarbons is further supported by the appearance of the C-H bending peak within the range of 1350 -1500 cm<sup>-1</sup>. Furthermore, a prominent C-O stretching peak was observed within the 1050 – 1100 cm<sup>-1</sup> range, suggesting the presence of esters.

The prominent C=O stretching peak at 1650 cm<sup>-1</sup> confirmed the presence of FAME, the main product of the transesterification procedure used to make biodiesel. This peak serves as a definitive indication of the efficient transformation of discarded fish oil into biodiesel, as depicted in Figure 2. The FT-IR results conclusively indicate that biodiesel is the primary product generated throughout the reaction, highlighting the efficiency of the transesterification process. The thorough examination conducted using FT-IR spectra confirms the successful synthesis of biodiesel from waste fish oil, emphasizing the presence of specific functional groups indicative of biodiesel. This verifies the transesterification process and affirms the feasibility of utilizing waste fish oil as a raw material for biodiesel synthesis.

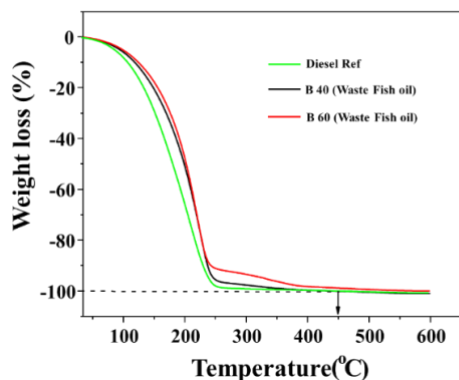


**Figure 2.** The FTIR spectra of biodiesel from waste fish oil

### 3.3 TG/DTA analysis

The biodiesel samples, specifically B40 and B60 mixtures derived from WFO, were further characterized through thermogravimetric analysis (TG) and differential thermal analysis (DTA). These analyses assist in comprehending the modifications in the physical and chemical properties of materials that occur during the heating or cooling process (Alves et al., 2022). Insights into a material's thermal stability and composition are available through the measurement of weight change by TG. The TG analysis of the biodiesel samples in this study demonstrated a weight loss pattern that was substantially similar to that of conventional diesel, suggesting that the thermal decomposition

behavior was comparable. The biodiesel blends and standard diesel also exhibited similar patterns when using DTA, which captures the temperature difference between a sample and a reference under controlled conditions.



**Figure 3.** TG/DTA curves of diesel standard, diesel B40, and B60 from waste fish oil

Figure 3 shows that the biodiesel samples (B40 and B60) and regular diesel saw a 5% decrease in weight at temperatures of 80°C, 95°C, and 100°C. This further corroborates the efficacious synthesis of biodiesel from waste frying oil. The TG/DTA curves for diesel standard, B40, and B60 mixtures from WFO show a 5% decrease in weight at particular temperatures: 80°C for B40, 95°C for B60, and 100°C for standard diesel. As the thermal degradation patterns closely

resemble those of standard diesel, these observations substantiate the successful formation of biodiesel from WFO. The thermal transition and weight loss patterns are consistent, indicating that the biodiesel samples have thermal stability comparable to conventional diesel (Anand, 2018). They are appropriate as alternative fuels. Therefore, the TG/DTA analysis confirms that the B40 and B60 biodiesel samples derived from WFO exhibit thermal properties that are comparable to standard diesel, thereby validating the efficacy of the transesterification process. The analysis further substantiates the potential of these biodiesel mixtures as viable alternatives to conventional diesel fuels, illustrating their suitability for practical applications.

### 3.4 Analysis of the physical and chemical characteristics of the produced biodiesel

A comprehensive analysis was conducted to examine the physicochemical characteristics of biodiesel products, with particular emphasis on important factors, including kinematic viscosity, density, acid value, cloud point, and pour point. These qualities are essential for diesel engine applications and were assessed using the ASTM D-6751 and EN14214 standard techniques. The analysis revealed that the biodiesel generated possessed the subsequent characteristics: a kinematic viscosity of  $3.05 \pm 0.02$  cSt at 40°C, a density of  $0.872 \text{ g/cm}^3$ , an acid value of  $0.07 \text{ mg KOH/g}$ , a cloud point of  $7^\circ\text{C}$ , and a pour point of  $3^\circ\text{C}$ . The values were comparable to those of standard biodiesel, as indicated in Table 1. The Physicochemical properties of biodiesel products were studied using the kinematic viscosity, density, acid value, cloud point, and pour point. These properties are pivotal in diesel engine applications and are evaluated following the ASTM D-6751 and EN14214 standard methods.

**Table 1.** The physicochemical properties of biodiesel products (B0 std. – B100)

Biodiesel	Kinematic viscosity ( $\text{m}^2\text{s}^{-1}$ )	Density ( $\text{g m}^{-3}$ )	Acid value ( $\text{mg KOH g}^{-1}$ )	Cloud point ( $^\circ\text{C}$ )	Pour point ( $^\circ\text{C}$ )
B0 (std.)	$2.73 \pm 0.02$	0.822	0.09	3	<-5
B20	$3.70 \pm 0.04$	0.832	0.01	4	<-5
B40	$3.46 \pm 0.07$	0.839	0.02	4	<-5
B60	$3.50 \pm 0.02$	0.851	0.03	5	0
B80	$3.24 \pm 0.04$	0.860	0.04	6	1
B100	$3.05 \pm 0.02$	0.872	0.07	7	3
High-speed diesel	$3.2 \pm 0.2$	-	<0.02	-5	<-15 to -5
ASTM D-6751	1.9 - 6.0	-	$\leq 5$	-3 to 12	-15 to +16
EN14214	3.5 - 5.0	-	$\leq 5$	-	-

We found that the kinematic viscosity  $3.05 \pm 0.02$  °C (cSt) at 40 °C, density  $0.872 \text{ ag m}^{-3}$ , acid value  $0.07 \text{ mg KOH g}^{-1}$ , cloud point  $7^\circ\text{C}$  and pour point  $3^\circ\text{C}$  of Biodiesel products (B0 – B100). The biodiesel properties were similar to the standard observed in Table 1.

The created biodiesel complied with the ASTM D-6751 and EN14214 standards, demonstrating qualities within the specified ranges. More precisely, the kinematic viscosity of B100, measured at  $3.05 \pm 0.02$  cSt, is within the ASTM range of 1.9 - 6.0 cSt. Additionally, the acid value of B100, determined to be  $0.07 \text{ mg KOH/g}$ , is significantly lower than the maximum permitted limit of  $5 \text{ mg KOH/g}$ . Furthermore, the cloud and pour point parameters guarantee the practicality of the biodiesel in different temperature environments (Aitlaalim et al., 2020). The results indicate that biodiesel derived from waste fish oil has comparable physicochemical characteristics to conventional biodiesel, affirming its compatibility for utilization in

diesel engines. The strict adherence to set standards confirms the high quality and dependability of biodiesel (Adeoti et al., 2015), thereby establishing it as a practical substitute for traditional diesel fuel.

### 3.5 Application of Biodiesel in agricultural engine

The efficacy of the synthesized biodiesel in diesel agricultural engines underwent extensive testing, resulting in highly positive outcomes (Table 1). The experiment revealed that the engine exhibits remarkable efficiency when fuelled by biodiesel, especially in mitigating detrimental emissions. The biodiesel engine substantially reduced CO and  $\text{CO}_2$  gas emissions, illustrating biodiesel's environmental advantages compared to traditional diesel fuel.

Biodiesel use decreases greenhouse gas emissions, helps combat climate change, decreases air pollution, and reduces health hazards.

The enhanced combustion qualities of biodiesel result in more thorough fuel burning, reducing pollutants and improving fuel efficiency. Efficient combustion and atomization of biodiesel in the engine reduce the number of unburned hydrocarbons and particulate matter, resulting in a more environmentally friendly. Moreover, the enhanced combustion efficiency of biodiesel adds to the extended lifespan of the engine (Bibin et al., 2023). Optimizing the combustion process minimizes the accumulation of carbon deposits in the engine, resulting in less damage to engine parts and decreased maintenance expenses.

The exceptional lubricating properties of biodiesel also minimize friction between the various components in motion, significantly prolonging the engine's biodiesel performance in conventional diesel

fuel without substantially altering current diesel engines. The flexibility to adjust is essential for agricultural applications, where the most important factors are the reliability and durability of the engine.

The presentation, depicted in Figure 4, highlights the practical advantages of using biodiesel in agricultural environments. The statement emphasizes the capacity of biodiesel to sustain engine performance while offering environmental and economic benefits. The utilization of waste fish oil as a source of biodiesel has been demonstrated to be a feasible and enduring substitute for traditional diesel fuel, facilitating the shift towards renewable energy sources in the agricultural sector (Homdoun et al., 2020).



**Figure 4.** Demonstration of biodiesel in diesel agricultural engine

To summarize, using biodiesel in agricultural engines provides several advantages, such as decreased emissions of greenhouse gases, enhanced engine performance and durability, and decreased maintenance expenses. The benefits of biodiesel make it an appealing choice for farmers seeking to embrace more environmentally friendly methods. This study's favorable results support the broader adoption of biodiesel in agricultural machinery, advancing environmental conservation and ensuring long-term energy sustainability in the agricultural industry.

#### 4. Conclusion

This study successfully produced biodiesel from waste fish oil by transesterification, utilizing NaOH as the catalyst. The reaction optimization involved combining methanol and waste fish oil in a molar ratio of 12:1, using 1 wt% NaOH, and applying heat to the mixture at 65°C for 1 hour. The waste fish oil was efficiently transformed into biodiesel with this procedure. The biodiesel's physicochemical qualities were comprehensively examined, affirming its appropriateness as an alternative to diesel fuel. In addition, we showcased the utilization of the produced biodiesel in a diesel agricultural engine, proving its comparable efficiency to regular diesel. Moreover, this alternative fuel exhibited the advantages of decreased emissions of CO and CO<sub>2</sub>, as well as enhanced engine durability resulting from optimized combustion and reduced carbon buildup. These findings suggest that increasing biodiesel production to a pilot-scale level may be possible. This

highlights the practicality of using waste fish oil as a renewable, eco-friendly fuel option. The findings of our study confirm the feasibility of utilizing waste fish oil as a viable source for biodiesel production. This is a practical approach to decrease dependence on fossil fuels and foster the use of renewable energy in multiple industries, including agriculture.

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