

# Effect of Concentration Catalyst in Production and Physicochemical Properties of Frying Cooking Oil and Neem Oil Mixture

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## ABSTRACT

Industrialization and the rapid growth of the population have resulted in a substantial increase in energy demand in recent years, along with adverse environmental impacts caused by the use of fossil fuels. The biodiesel production process in this study involves the utilization of feedstock with a mixture of frying cooking oil and neem oil. The study employs two methods: esterification using an acid catalyst ( $H_2SO_4$ ) and transesterification utilizing a base catalyst (KOH). The production process commences with the degumming process using  $H_3PO_4$ . Subsequently, the esterification process is conducted for 90 minutes at 1000 rpm. The transesterification process performed a KOH with varying concentrations (0.50%, 0.75%, 1.00%, and 1.50%) at a temperature of 60°C, a rotation speed of 1000 rpm, and 1.5 hours. The methyl ester has various characteristics, including viscosity, density, acid number, calorific value, oxidation, flash point, and iodine number. The findings reveal that the optimal yield value is obtained when using a catalyst concentration of 1.00 % KOH, resulting in a yield of 96.2%. Moreover, the biodiesel characteristic conducted on the mixture of frying cooking oil and neem oil showed that the biodiesel properties meet the requirements stipulated in the ASTM D6751 and EN 14214 standards for biodiesel. Notably, adding 1.00 % KOH to the mixture of frying cooking oil and neem oil significantly enhances the characteristic properties of the biodiesel, as evidenced by an oxidation stability of 10.5 hours, thereby making the energy content of the biodiesel comparable to diesel fuel.

*Keywords:* Alternative fuel, biodiesel, characteristics, waste cooking oil, neem oil

## 1. INTRODUCTION

Energy has always been the primary driving force facilitating human and societal growth; in recent decades, fossil fuels have been humanity's principal energy source (Das & Sharma, 2023). The pursuit of sustainable development and the need to reduce the environmental repercussions of fossil fuel consumption has driven interest in renewable energy sources such as biofuels (Shayanmehr et al., 2023). Bioethanol and biodiesel are gaining popularity as alternatives to petrol and diesel due to their advan-

tages as sustainable green energy sources (Farghali et al., 2023; Riyadi et al., 2023). The world is currently undergoing a fuel oil crisis. By 2050, fossil fuels will be exhausted. Many countries, including Indonesia, need more fuel oil (derived from fossil fuels). Indonesia, in particular, has imported significant amounts of fuel oil (particularly diesel fuel) for domestic consumption. Thus, biodiesel production has received much attention in recent years from scientists and researchers worldwide because of the increasing need for energy and increased awareness about the long-term negative impact of fossil fuels on the environment (Mahlia, Ismail,

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Hossain, Silitonga, & Shamsuddin, 2019).

Biodiesel is a mono-alkyl ester of long-chain fatty acids usually derived from vegetable oils or animal fats, is non-toxic, and has desirable fuel characteristics comparable to diesel oil (Riyadi et al., 2023). Because of its similar physicochemical, biodiesel is a feasible replacement for diesel fuel. Furthermore, when compared to fossil fuels, biodiesel is more environmentally benign. The combustion of fossil fuels emits sulfur dioxide (SO<sub>2</sub>), which contributes to high levels of air pollution (Mofijur, Atabani, Masjuki, Kalam, & Masum, 2013). The use of biodiesel in diesel engines can result in significant reductions in harmful pollutant emissions. Additionally, biodiesel has a more significant cetane number than petroleum-based diesel, which can increase ignition quality and fuel efficiency (Abukhadra & Sayed, 2018).

Indonesia has natural resource potential, and many researchers are exploring environmentally friendly alternative fuels, namely non-edible materials (Milano et al., 2018). Waste cooking oil (Topare & Patil, 2021; Zhang et al., 2023); Calophyllum inophyllum oil (Milano et al., 2022); Jatropha curcas oil (Riayat-syah et al., 2022); and insect oil (Kee et al., 2023). The non-edible raw materials are chosen to prevent worldwide difficulties with edible oil (palm oil), affecting the price of these raw materials (A. S. Silitonga et al., 2020). The high price of raw materials, especially vegetable oil, is a significant problem in commercializing biodiesel (Gupta & Rathod, 2018). Biodiesel production from frying cooking oil (FCO) is a cost-effective and waste-reduction solution. Many food processing companies, such as restaurants and post-processing facilities, produce FCO as a byproduct. It is frequently disposed of in landfills, which can harm the environment. Biodiesel manufacturing can assist in reducing the quantity of waste going to landfills while providing a renewable fuel source (Cordero-Ravelo & Schallenberg-Rodriguez, 2018). Thus, the cost of producing biodiesel synthesized from vegetable oil can be reduced by up to 70% if using FCO (Manikandan, Kanna, Taler, & Sobota, 2023; Putra, Nata, & Irawan, 2020). Using low-priced oils such as FCO will significantly reduce the cost of the final product (Putra et al., 2020). However, FCO has low physicochemical properties due to occurred reaction during frying among the components inside the oil or between oil components with the food contents (Milano et al., 2022). Biodiesel production have been investigated, and it has been elaborated that the type of oil mixture allows for improving biodiesel characteristics, such as viscosity and oxidation stability (Milano et al., 2016).

Neem is a tree in the family Malaiaceae that grows in different parts of Bangladesh and Indonesia. The neem (*Azadirachta indica* A. Juss, Meliaceae) is a plant originally from northeast India but is well adapted to regions such as America (Caribbean, central and south America), Asia (Nepal, Pakistan, Bangladesh, Sri Lanka, Myanmar, Thailand, Malaysia, Indonesia and Iran, China, Turkey, Indonesia), Africa (Cameroon), and even Queensland, Australia (Benelli et al., 2017). Its scientific name is '*Azadirachta indica*'. Neem tree reach 12 to 18 meters in height with a thickness of up to 1.8 to 2.4 meters (Kumar & Navaratnam, 2013; Michel et al., 2023). The total amount of neem oil (NO) varies from 40% to 50%, i.e., 7 to 8 kg of seed requirements produce one litre of neem oil, which has a high potential for biodiesel production (Kumar & Navaratnam, 2013; Michel, Aguilar-Zárate, Rojas, Martínez-Ávila, & Aguilar-Zárate, 2023). The total amount of neem oil (NO) varies from 40% to 50%, i.e., 7 to 8 kg of seed requirements produce one litre of neem oil, which has a high potential for biodiesel production (Benelli et al., 2017). Its oil has a higher molecular weight, viscosity, density, and flash point than diesel fuel. Neem oil is generally light brown to dark, bitter, and has a strong odour

that is said to combine the odours of peanuts and garlic (Anyá, Jack, & Osagie, 2016). Neem is mainly composed of triglycerides and a large number of triterpenoid compounds. It contains four significant saturated fatty acids, two of which are palmitic acid and two are stearic acid. It also contains polyunsaturated fatty acids such as oleic and linoleic acids (Boontawee, Nakason, Kaesaman, Thitithammawong, & Chewchanwuttiwong, 2017). Some of the physical properties like density and viscosity and heating of Neem oil are 929 kg/m<sup>3</sup> (at 15 °C) and 38.875 mm<sup>2</sup>/s (at 40 °C), respectively (Singh, Sharma, Soni, Sharma, & Kumari, 2019). Neem has mainly large amount of unsaturated part like oleic acid (25–54%) and linoleic acid (6–16%) and saturated parts have stearic acid (9–24%) (Ali, Mashud, Rubel, & Ahmad, 2013). A neem oil mixture is expected to improve the characteristic properties of FCO. Biodiesel fuel is produced via transesterification of refined mixture FCO and Neem oil using KOH catalysts. The nature of catalyst employed during transesterification reaction is crucial in converting triglycerides to biodiesel. As a result different catalysts have being explored for converting triglycerides to biodiesel fuel (Banga & Pathak, 2023). The catalysts usually employed to catalyse transesterification reaction are homogeneous catalysts and heterogeneous catalysts. Homogeneous alkaline catalysts such as NaOH, KOH, CH<sub>3</sub>ONa, and CH<sub>3</sub>OK are more often used in producing biodiesel (Banga & Pathak, 2023). In industrial-scale biodiesel production, the most common homogeneous base catalysts are usually KOH and NaOH. They have been shown to have high catalytic activity and are traditionally used commercially as low-cost catalysts (Riaz et al., 2022). Furthermore, homogeneous base catalysts are the most viable catalysts for mass production because the transesterification process using base catalysts is performed under low pressure and temperature conditions, conversion rate is faster in a short period of time, the conversion rate is outstanding with no intermediate steps, and the process is cost effective. (Benelli et al., 2017) used KOH in the transesterification of dry algae and achieved maximum biodiesel yields of 90.42%. Investigated the conversion of biodiesel from neem seeds oil with a KOH catalyst using response surface methodology statistical analysis. Therefore, this study investigates the production and characterization of biodiesel using a mixture FCO and neem oil using a catalyst KOH. The research's broad goals include lowering the cost of biodiesel production while maintaining the price of crude oil used for human consumption by employing less expensive raw materials as inputs. The novelty of this study is to procedures non-food oil raw materials into biodiesel it will give added value in replace fossil fuel consumption. On the other hand, producing biodiesel from a mixture FCO and neem seeds oil with a high yield value and to obtain the optimal methods and conditions of the biodiesel production process to deliver the best results and quality, the mixture of Neem oil is expected to be an additive to the FCO.

## 2. MATERIAL AND METHODS

### 2.1 MATERIAL

Frying cooking oil (Fig 1) was collected from households whereas crude neem oil (Fig 2) was purchased from Surabaya, East Java, Indonesia. The following chemicals were used for biodiesel production: methanol (purity: 99.9%, grade: ACS reagent), sulphuric acid (purity: > 98.9%), ortho-phosphoric acid (purity: 85%), anhydrous sodium sulfate (purity: 99%), sodium hydrogen carbonate (purity: > 99%), potassium hydroxide pellets (purity: 99%), Fluka® Analytical phenolphthalein solution (1% in eth-

anol). These chemicals were purchased from CV. Rudang Jaya, Medan, Indonesia.



Fig. 1 Frying Cooking Oil



Fig. 2 Neem Seeds Oil

## 2.2 EXPERIMENTAL SET-UP

The FCO and Neem Oil (NO) were prepared in different weight ratios of 90:10, 80:20, 70:30, and 60:40 w/w%. The crude oil was mixed by using a mechanical stirrer for 30 minutes at 60°C. Several properties such as Acid value, FFA, oxidation stability was evaluated using ASTM D6751 and EN 14214 standard before determining the best mixture. In comparison, determining FFA levels attempts to identify the level of free fatty acids in a mixture of FCO and NO to determine the oil's feasibility for producing biodiesel. The collected FCO was refined with filter paper to remove the solid impurities. The NO was mixed with FCO in a molar ratio of FCONO. Table 1 shows the values of acid, FFA, and oxidation with an increase of 10% in neem oil (NO).

Table 1 Properties of crude oil and mixture of crude oil

Crude oil and mixture crude oil	Acid value (mg KOH/g)	FFA (mg/g)	Oxidation stability (h)
FCO100	2.17	1.01	1.26
FCO90NO10	8.19	4.1	1.45
FCO80NO20	13.2	8.2	2.93
FCO70NO30	16.3	6.9	5.25
FCO60NO40	21.3	13.3	4.78
NO100	47.3	32.3	7.20

The oxidation stability of FCO90NO10 and FCO80NO20 oil mixtures is relatively low. Therefore, both oil mixtures are not recommended for further exploration. However, there is an increased oxidation stability for FCO70NO30 and FCO60NO40 oil blends, which is advantageous in selecting these oil blends for biodiesel production. The FFA content of the oil mixture was measured, and it was found that the FFA content was 9.92%, which was more than the permissible limit (2%) for the transesterification process (Milano et al., 2018). The mixture of crude oil show that the acid values for FCO70NO30 and FCO60NO40 are 16.3 mg KOH/g and 21.3 mg KOH/g, respectively; the acid values for both oil mixtures are high. The FFA content of the oil mixture was measured, and it was found that the FFA content was 6.9%, which was more than the permissible limit (2%) for the transesterification process (Milano et al., 2018). However, FCO60NO40 with 21.3 mg KOH/g requires two times the esterification process, which can increase the cost of biodiesel production. Therefore, the FCO70NO30 oil mixture was selected for this study, and the mixture was esterified and transesterified using a double jacket reactor glass (Milano et al., 2018).

## 2.2 BIODIESEL PRODUCTION

The frying cooking oil was refined using a filter to remove solid impurities and then heated to remove traces of water. Thus, the frying cooking oil was mixed with neem oil at a volume ratio 7:3. This oil sample was labeled as FC70NO30. The FFA content of the oil mixture was measured, and it was found that the FFA content was 6.9%, which was more than the permissible limit (2%) for the transesterification process (Milano et al., 2018).

### 2.2.1 Mixing and degumming process

The FCO and neem seed crude oils were mixed in order to improve the oxidation stability of the final product. In general, crude oils contain varying amounts of phosphatides (known as gums) and these phosphatides need to be removed from the oils since they are undesirable for biodiesel production. The process of removing phosphatides from crude oils is called degumming process. In this process, the FC70NO30 oil mixture was preheated at 60 °C for 15 min. The FC70NO30 oil mixture chosen, because this mixture has better properties compared to some other crude oil mixtures previously tested, as shown in Table 1. Following this, 5 vol. % of phosphoric acid (H<sub>3</sub>PO<sub>4</sub> 20%) was added into the oil mixture and heated at 60 °C over a period of 30 min with an agitation speed of 1000 rpm. This was followed by a simple filtration process for at least 2 h, in which the formation of phosphatides (gums) can be observed at the bottom of the flask. The gums were separated from the oil mixture and the oil mixture was washed several times with distilled water at 45 °C. The excess water was evaporated from the FC70NO30 oil mixture using a vacuum pump for 30 min in order to prevent oxidation of the oil mixture.

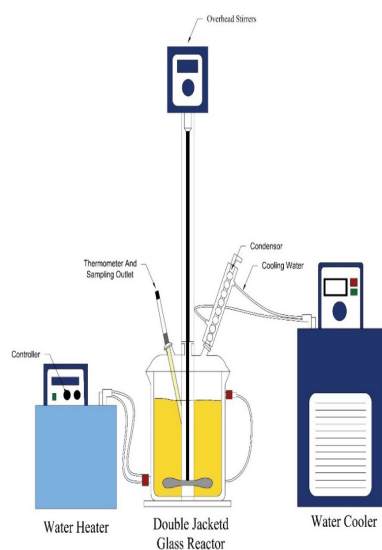
### 2.2.2 Esterification

The esterification process is carried out in two steps: esterification with an acid catalyst and transesterification with alkaline catalysts (KOH). The esterification process is carried out in two steps: esterification with an acid catalyst and transesterification with alkaline catalysts (KOH). The esterification process is also known as pre-treatment and the main purpose of this process is to reduce the amount of free fatty acids present in the oil mixture to less than 1%. In this process, 1% (v/v) of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)

was added into 500 ml of degummed FC70NO30 oil mixture. The esterification process was carried out over a period of 1.5 h using the following operating parameters: methanol-to-oil molar ratio: 50%, temperature: 60 °C and agitation speed: 1000 rpm. Once the reaction was complete, the products were poured into a separating funnel in order to separate excess methanol, H<sub>2</sub>SO<sub>4</sub> and impurities. The bottom layer containing excess methanol, H<sub>2</sub>SO<sub>4</sub> and impurities settles at the bottom of the separating funnel after 1h. Following this, the esterified FC70NO30 oil mixture was heated at 60 °C in a rotary evaporator under vacuum conditions for 1 h to remove methanol and water residues present in the oil mixture. The esterification process was used to reduce the FFA content in *Hevea brasiliensis* and *Ricinus* to less than 2 wt% prior to the transesterification process (A. Silitonga et al., 2016).

### 2.2.3 Transesterification

The esterified FC70NO30 oil mixture was measured and pre-heated to a temperature of 60 °C using a Hot Plate. Following this, catalyst with a percentage ratio (0.50%, 0.75%, 1.00%, 1.50%) wt.% of potassium hydroxide (KOH), which is an alkaline catalyst, was dissolved in methanol, noting that the methanol-to-oil ratio was 50%. This KOH–methanol solution was then added into the heated oil and the reaction continues over a period of 1.5 h. The oil mixture was stirred constantly at 1000 rpm using an overhead stirrer double jacket reactor during the transesterification process and the temperature was kept constant at 60 °C. Once the reaction was complete, the methyl ester (biodiesel) was poured into a separating funnel for 1 h in order to separate glycerol from the methyl ester. The excess methanol, glycerol and impurities contained in the high-density bottom layer were removed in this stage. Following this, the methyl ester (biodiesel) was poured into the rotary evaporator to remove methanol residues, and then washed with distilled water several times to remove entrained glycerol and impurities. In this process, 25% (v/v) of distilled water at 45 °C was sprayed over the surface of the methyl ester and stirred slowly. The methyl ester was further purified to remove excess water and methanol using a vacuum pump at 60 °C, and finally filtered using a filter paper. As shown in Fig 3 schematic diagram of the biodiesel production and Fig 4 esterification and transesterification process.



**Fig. 3 Schematic diagram of the biodiesel production**



**Fig. 4 Esterification and transesterification process**

### 2.2.3 PHYSICOCHEMICAL PROPERTIES AND YIELD BIODIESEL

The physicochemical properties were tested according to the ASTM D6751 and EN 14214 standards. Several tests, such as kinematic viscosity, density, flash point, heating value, acid value, iodine value, and oxidation stability. This study was conducted using the esterification transesterification method to obtain a high methyl ester yield. The methyl esters process was taken to calculate the amount of methyl ester yield produced (A. Silitonga et al., 2016). The methyl ester yield was calculated from esterification process until the methyl ester produced as follow equation 1:

$$\text{Methyl ester yield (\%)} = \frac{\text{Weight of methyl ester produced (g)}}{\text{Weight of oil used (g)}} \times 100\% \quad (1)$$

The methyl ester yield was calculated from the esterification process until the methyl ester was produced as follows equation 1.

## 3. RESULTS AND DISCUSSION

### 3.1 EFFECT OF CATALYST CONCENTRATION ON BIODIESEL YIELD

The methyl ester in this study uses KOH catalysts that vary in concentration. The higher methyl ester yield is expected to meet the quality requirements of ASTM or EN Standard. Figure 4 shows the ester yield versus the KOH catalyst concentration. The catalyst concentration varied from 0.50% to 1.50 wt%. With keeping other parameters constant. The higher methyl ester was produced at a 1.00 wt% KOH concentration variation with a 96.2%. The lower yield resulted in a variation in KOH concentration of 1.50 wt% with a yield of 78.3%. From the figure, the higher the concentration of KOH, the lower the toxicity. Therefore, increasing the amount of the catalyst in higher doses formed of soap lowers methyl ester (Sadaf et al., 2018). Soap formation will increase the solubility of the methyl ester produced in glycerine. As a result, an emulsion between the two phases will be formed, increasing the viscosity of the reactor and making the separation of the two phases more difficult. As a result, methyl ester yields are decreasing. Same as previous research, (Kusumo et al., 2020) concluded the effect of KOH loading (0.75 to 1.25 wt.%) on biodiesel yield was evaluated at methanol to oil ratio of 50 (w/w)%, with 15 min reaction time. It was shown that the biodiesel yield increases from 96.8 % to 98.72

% as the KOH loading increases from 0.75 to 1.0 wt.% and significantly decreased to 93.42 % afterwards. This could be explained that more triglycerides are formed in the saponification side reaction due to the excessive addition of KOH catalyst (Kusumo et al., 2020). It was found that the optimum catalyst loading in this study was 1.0 wt.% with biodiesel conversion of 96%.

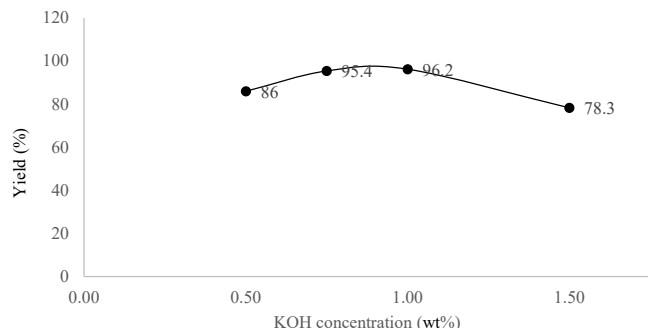


Fig. 5 Effect of KOH variation on methyl ester yield

### 3.2 CHARACTERISTICS OF METHYL ESTER

Table 2 shows that the properties of methyl ester were obtained within the limits specified by ASTM and EN Standard. Kinematic viscosity (ASTM D445 and EN ISO 3104), density (ASTM D4052 and EN ISO 3675), acid value (ASTM D664 and ISO 14104), and iodine value (AOSCS Cd 1–25). The acids and iodine values were tested using the elektrochemie hallae titrallion GmbH (ECH) titrator, the calorific value (ASTM D240) was tested with the Calorimeter C2000 Basic IKA, the oxidation (EN 14112 and EN ISO 1442) was tested with the oxidation stability tester RAPIDOXY 100 Fuel Anton Paar, and the Prenskey Martens tested the flash point (ASTM D-93 and ISO 22719).

Table 2 Properties of FCONO methyl ester

Properties	Unit	ASTM and EN standard				FCONO methyl ester			
		ASTM D6751	Standard method	EN 14214	Standard method	KOH 0.50 wt%	KOH 0.75 wt%	KOH 1.00 wt%	KOH 1.50 wt%
Viscosity 40 °C	mm <sup>2</sup> /s	1,9-6.0	D445	3.5-5.0	EN ISO 3104	4.561	4.601	4.547	4.641
Density 40 °C	kg/m <sup>3</sup>	850-890	D4052	860-900	EN ISO 3675	863	863.8	862.3	890.1
Acid value	mg KOH/g	0,5 (max)	D664	0.5 (max)	EN ISO 14104	0.21	0.23	0.17	0.52
Calorific value	MJ/kg	35 (min)	D240	-	-	38.77	39.75	41.35	40.35
Oxidation stability 110 °C	°h	3 (min)	EN 14112	6 (min)	EN ISO 14112	9.45	9.48	10.03	8.37
Flash point	°h	100-170	D93	101 (min)	EN ISO 22719	161	159.2	163	158.3
Iodine value	g-I <sup>2</sup> /100g	115	AOSCS Cd 1-25	-	-	98.2	104	110	105

### 3.3 KINEMATIC VISCOSITY

Viscosity kinematics is a number that expresses the amount of resistance in a liquid material to flow or the size of the shear resistance of a liquid material. Viscosity kinematics is one of the essential characteristics of diesel fuel because it affects the performance of the injectors in diesel engines. The measurement method used is ASTM D-445 using Anton Paar’s SVM 3001 Stabinger viscometer. The results of kinematics viscosity analysis on KOH concentration variations can be seen in Figure 5. The kinematic viscosity of the ASTM Standard (D-445) for diesel oil is in the range of 1.9–6.0 mm<sup>2</sup>/s, while the EN 14214 standard is in the range of 3.5–5.0 mm<sup>2</sup>/s. According to ASTM and EN standards, the kinematics of the methyl ester mixture of frying cooking oil and neem oil with variations in KOH concentration have qualified as biodiesel and diesel fuels. The lowest viscosity is at a KOH concentration of 1.00 wt%, 4.547 mm<sup>2</sup>/s, while the highest is found at a KOH catalyst concentration of 1.50 wt%, 4.641 mm<sup>2</sup>/s. The fuel must have a low viscosity to flow easily and be automated. This is due to rapid engine depletion, which requires rapid fuel injection.

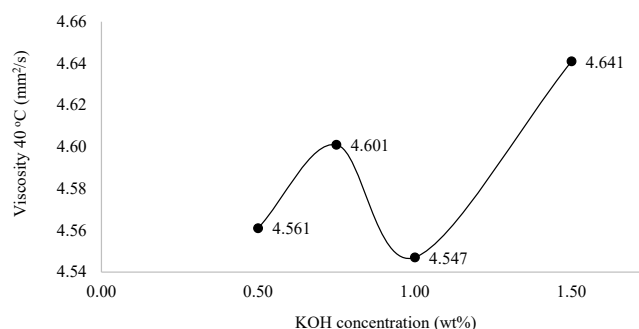


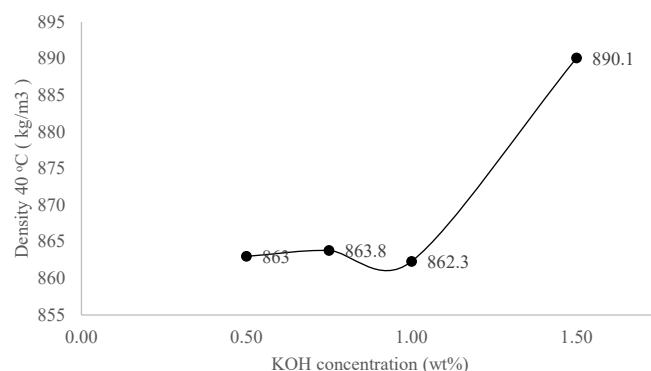
Fig. 6 Effect of KOH variation on kinematic viscosity

The presence of transesterification processes in biodiesel production causes the viscosity value of the vegetable oil to be reduced and fulfil the range of ASTM and EN standards for biodiesel. In the transesterification process, where fatty acids react with the KOH catalyst and form soap, With the existence of soap produced in the production of biodiesel, it results in the surface volt-

age of biodiesel becoming high, and when the surface tension is high, it is difficult to break down the molecules of the compound; this is related to the degree of stiffness of the biodiesel compounds. The increasing number of KOH catalysts will also cause the methanolysis process to accelerate so that the viscosity value increases, which is not suitable for the engine diesel (Milano et al., 2018). According to the result (Wedler & Trusler, 2023), the higher the conditions of viscosity, the higher the flow capacity, as it will affect the injector's performance on the diesel engine. Increasing viscosity can affect good lubrication in fuel. The conditions of viscosity, pressure, injection, and the size of the injector hole will affect fuel atomization. High viscosity will make the atomized fuel into more significant drops with high momentum and tend to collide with the cylinder wall, which is relatively cooler, and cause flame extinction, increased deposit, fuel spray penetration, and engine emissions. Then, kinematic viscosity gradually increases with increasing catalyst concentration. According to (Pali, Sharma, Kumar, & Singh, 2021), the viscosity of the produced biodiesel decreases with increasing reaction yield. The inadequate amount of catalyst in reaction medium for low catalyst concentrations (e.g., 0.50% of potassium hydroxide) reduces yield of the transesterification reaction, and increases kinematic viscosity of produced biodiesel. Moreover, the use of higher catalyst concentration develops yield of the transesterification reaction, and thus the viscosity of the produced biodiesel decreases. On the other hand, if excess catalyst concentration is used, the yield of the transesterification reaction decreases and the viscosity of the produced biodiesel increases because of formation of fatty acid salts (soap), decrease in activity of catalyst and difficulty in separation of glycerol (Mandari & Devairai, 2021), in the next stage of the study, catalyst concentration of 1.00% giving the lowest viscosity was kept constant and the other parameters were changed. (Syarif, Yerizam, Yusi, Kalsum, & Bow, 2020) result it was found that an increase in the concentration of catalyst used would increase the yield of biodiesel WCO products. The highest yield was obtained at the use of a 1% concentration of 86.95%. The higher the concentration of catalyst KOH in solution, the smaller the activation energy of a reaction (Sharma, Kodgire, & Kachhwaha, 2020). The use of high catalyst concentrations increases the rate of transesterification reaction between triglyceride molecules with methanol and reduces the activation energy needed to achieve the transesterification reaction so that the conversion of oil to biodiesel is increased. On the other hand, the addition of catalysts by 1.2% resulted in a decrease in yield. The use of excess catalyst concentrations can increase saponification reactions and reduce yield production (Syarif et al., 2020).

### 3.4 DENSITY

Density properties determine the fuel feasibility level in the diesel engine. The measurement method used is ASTM D-4052 using Anton Paar's SVM 3001 Stabinger viscometer. The density measurement results at varying catalyst concentrations are presented in Figure 6.



**Fig. 7 Effect of KOH concentration on density**

Figure 4 can show that the weight of the methyl ester frying cooking oil and neem oil, with the addition of the variation in KOH concentration, is increasing. The highest density is at a concentration of KOH 1.50 wt%, which is 890.1 kg/m<sup>3</sup>, and the lowest is found in the 1.00 wt% KOH catalyst, which is 862.3 kg/m<sup>3</sup>. Excessive catalysts can result in absorption reactions and the presence of residual deodorant reactions that cause increased density (Vital-López et al., 2023). This allows the presence of detoxifying substances such as potassium soap and glycerol. These fatty acids are not converted into methyl esters, water, residual hydroxide, residual methoxide, or residual methanol that, cause the mass of the methyl ester to be more significant. On the contrary, if the use of a base catalyst with a small concentration causes the mass of the kind of methyl ester to be low (Manikandan et al., 2023). According to research (Ahmad, Yadav, Singh, & Pal, 2023) influence of the catalyst amount on the density of the methyl ester shows a decrease as the catalyst amount increased. On the other hand, viscosity decreased up to 1.0% KOH concentration for the biodiesel and after that it was almost constant.

### 3.5 ACID VALUE

The acid value is used to measure the amount of free fatty acids present in the oil. The measurement methods used are ASTM D664 and EN ISO 14104 using the Elektrochemie Halle GmbH (ECH) Titrator. Where the ASTM and EN standards for acid numbers are maximums of 0.5 mg KOH/g. The acid value measures the amount of free fatty acids present in the oil. The measurement methods used are ASTM D664 and EN ISO 14104 using the Elektrochemie Halle GmbH (ECH) Titrator. The ASTM and EN standards for acid numbers are maximums of 0.5 mg KOH/g. The number of crude oil acids and the comparison of the KOH catalyst concentration to methyl ester can be seen in Figure 7. High acidity is a sign that methyl ester still contains free fatty acids. Methyl ester contains cyclopropenoid fatty acids that will polymerize, resulting in diesel engine injectors being blocked above 0.5 mg KOH/g (Milano et al., 2018). This level of acidity is related to the storage capacity and the level of corrosion. The magnitude of the number of acids can also be affected by the storage method, which can cause hydrolysis (Boro, Verma, Chettri, Yata, & Verma, 2022). This shows that FCONO methyl ester has a lower acid content than FCO, which meets the requirements given in the standard test method EN 14103:2011.

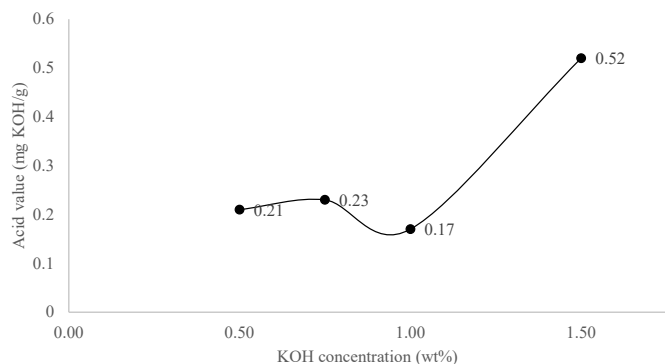


Fig. 8 Effect of KOH concentration on acid value

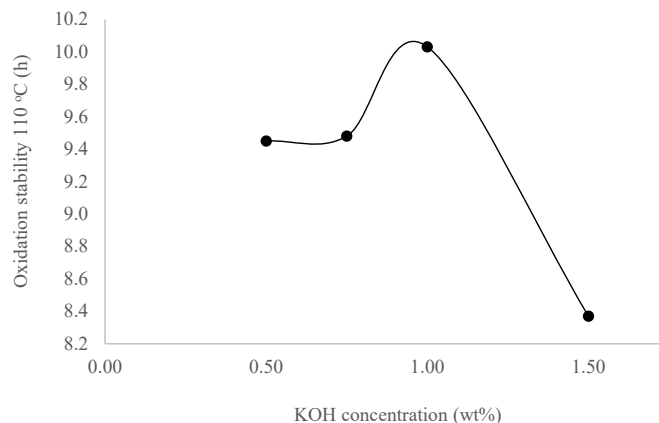


Fig. 9 Effect of KOH concentration on oxidation stability

### 3.6 OXIDATION STABILITY

Oxidation stability is an important feature as it shows the rate of fuel degradation, which plays an important role for fuel storage, handling, and transportation (Milano et al., 2022). The measurement method used is ASTM D6751 and EN ISO 14214 using the oxidation stability tester with Rapidox 100 Fuel by Anton Paar. FCO, FCONO methyl ester, and diesel fuel oxidation stability results can be seen in Table 4. It showed that the oxidation stability at 110 °C of biodiesel FCO is 3.11 hours, higher than the minimum value specified in ASTM D6751 (3 hours). In contrast, the oxidation stability for biodiesel FCONO KOH is 0.50 wt%, 0.75 wt%, 1.00 wt%, and 1.50 wt% (9.45, 9.48, 10.03, and 8.37) hours, respectively. These four KOH concentration variations meet ASTM and EN standards. However, the FCO biodiesel oxidation stability does not meet the requirements of EN 14214, which specifies the minimum limit of 6 hours of oxidation stability. Oxidation stability increased from the concentration of the KOH catalyst of 0.50 wt% to the catalyst of 1.00 wt% and decreased when the catalyst concentration reached 1.50 wt%. Adding neem oil (NO) to FCO by converting it into methyl ester has significantly improved oxidation stability. This suggests that the oxidation stability is quadrupled by mixing two types of raw materials, and FCONO oxidation stability is close to diesel fuel. If the fuel oxidation stability is low, it will decrease the characteristic properties of the fuel in which the acid value increases, which will cause the engine component to become corrosive and the kinematic viscosity to increase (Milano et al., 2018). Based on the results, it is seen that the mixture of frying oil with neem oil significantly improves the stability of methyl ester oxidation.

Table 4 Oxidation stability of crude oil, methyl ester and diesel fuel

Properties	Diesel	FCO	NO	FCONO methyl ester			
				KOH 0.50 wt%	KOH 0.75 wt%	KOH 1.00 wt%	KOH 1.50 wt%
Oxidation (hours)	15.2	3.11	7.32	9.45	9.48	10.03	8.37

### 3.7 FLASH POINT

The flash point is a number that indicates the lowest temperature of the oil fuel, which will occur for a moment when the flame is approached. Measurement of flame point using ASTM D-93 and EN ISO 22719 methods with a Pensky Martens Flash Point Tester. Figure 9 shows the FCONO flash point with KOH 0.50 wt%, 0.75 wt%, 1.00 wt%, and 1.50 wt%, respectively is 161 °C, 159.2 °C, 163 °C, 158.3 °C which is much higher than diesel (75.5 °C). The KOH 0.50 wt%, 0.75 wt%, and 1.00 wt% methyl ester flash point meets the requirements of ASTM D6751 and EN 14214, which stipulate that the respective fuel flash point must be in the range of 100–170 °C and more than 101 °C. The methyl ester produced exceeds the ASTM standard minimum limit. Therefore, it mentioned that the flash point value obtained meets the standard. If the flash point value is too high, it will be so hard to light that it will require more energy. The flash point is necessary for considering the safety of oil fuels against fire hazards, so high flame points are highly recommended. However, if too high, more than 176.67 °C is close to the point of the flash of the lubricant oil for the engine (Yusoff et al., 2022). Based on (Syarif et al., 2020) it is found that the acid number is influenced by the concentration of the catalyst used. The lowest acid number is in products with a catalyst concentration of 1%, 0.439 mgKOH / gr while the highest acid number is in products with a catalyst concentration of 1.4%, which is 0.711 mg-KOH / gr. The optimum concentration of catalyst causes the free fatty acids that react with the catalyst to increase so that the number of acidic products becomes lower (Adhikesavan, Ganesh, & Augustin, 2022). The use of an excess catalyst will form fat-free soap so that it will complicate the purification process.

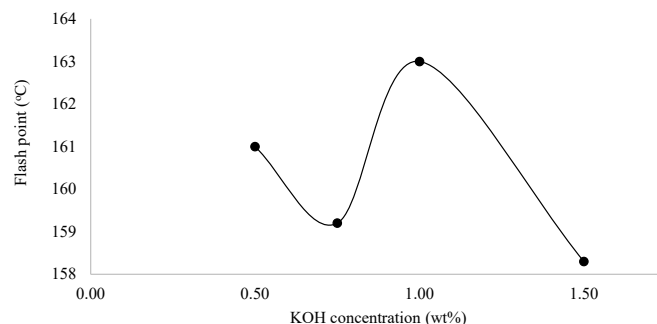


Fig. 10 Effect of KOH concentration on flash point

### 3.8 IODINE VALUE

The amount of iodine is used to determine the saturation in fatty acids. The amount of iodine in this methyl ester is 98.2 g I<sub>2</sub>/100g, 104 g I<sub>2</sub>/100g, 110 g I<sub>2</sub>/100g, and 105 g I<sub>2</sub>/100g, while the maximum standard set by ASTM for biodiesels is 115 g I<sub>2</sub>/100g. Overall, this methyl ester has met the standard for iodine value in Figure 10. Methyl ester with high iodine content is more easily oxidized when in contact with air (Barabás & Todoruț, 2011). The high iodine content will tend to polymerize if deposits form on the nozzle injector and the piston ring at the start of combustion. In addition, the higher the saturation, the higher the cloud point, and the lower the point of depletion. However, there is a negative effect, which is the possibility of free fatty acid formation (Mallah & Sahito, 2020).

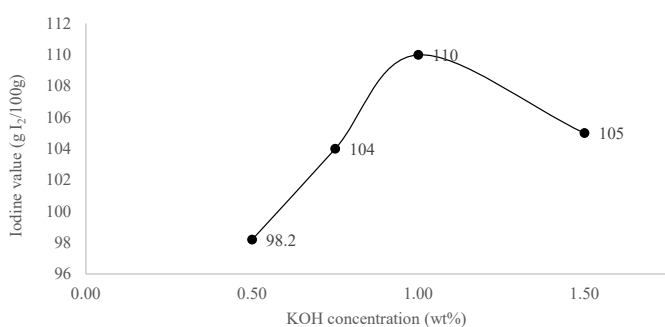


Fig. 11 Effect of KOH Concentration on Iodine value

### 4. CONCLUSIONS

In the current study, biodiesel production from a mixture of FCO and NO in various proportions was assessed utilising a green and scalable technique, and it was discovered that a lower acid value of 0.17 mgKOH/g and a high yield of 96.2% were obtained. Thus, the high methyl ester from the combination FCO and NO transesterification method demonstrates that mixture oil can be used as a feedstock for biodiesel production with less pollution concerns than petroleum diesel while lowering FAME production costs. The physicochemical properties of the produced biodiesel show that it meets ASTM criteria and may thus be utilised as a diesel alternative. The systematic application of various parametric optimisation techniques could be efficient in reducing the cost production costs and improving the stability of the oxidation of the methyl ester. This is due to oxidation stability being an important property because it shows the rate of fuel degradation, which plays an essential role in fuel storage, handling, and transportation. Future developments in this work could include investigating different biomass sources as precursors for catalyst synthesis, as these materials are abundant and inexpensive.

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