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Synthesis of Biodiesel from Feun Kase (*Thevetia peruviana*) Seed Oil Using NaOH Catalyst

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Abstract

The demand for biodiesel in the renewable energy sector continues to grow yearly. However, the majority of biodiesel sources currently still compete with the food sector. Feun Kase seeds contain high oil and do not compete with food, so they have prospects as a new source of biodiesel. This study aimed to find the optimal conditions for synthesizing biodiesel from Feun Kase seed oil, carried out through transesterification with four reaction variables: catalyst variation, time, temperature, and the molar ratio of oil/methanol. This study was equipped with parameter test data according to SNI (Indonesian National Standard) 7182:2015, also equipped with characterization using FTIR (Fourier Transform Infrared) and GCMS (Gas Chromatography Mass Spectroscopy). The highest biodiesel yield of 84.09% was obtained using optimum conditions of 1% NaOH catalyst, oil/methanol molar ratio of 1:6 at 70°C for 90 minutes of reaction. The test results of biodiesel parameters are density (851 kg/m^3), smoke point (6°C), kinematic viscosity (5.35 cSt); acid number (1.08 mg KOH/gr), saponification number (159.32 mg KOH/gr), iodine number (78.62 g I_2/100 g sample), flash point (165°C), and cetane number (62.86). FTIR analysis proved the presence of methyl esters with typical absorption at 1743 cm^{-1}, 1195.87 cm^{-1}, and 1436.97 cm^{-1}. GCMS characterization showed that Feun Kase biodiesel was dominated by methyl oleate (53.45%), methyl palmitate (27.05%), methyl stearate (10.96%), and methyl linoleate (6.29%).

1. Introduction

The world's demand for biodiesel is increasing. This can be seen in biodiesel production, which continues to increase by 20% every year [1]. A total of 46.8 billion liters were produced in 2020, with the most significant contributing countries being Indonesia (17%), the USA (14.4%), Brazil (13.7%), Germany (7.4%), France (5%), followed by the Netherlands (4.6%) [2]. As the world's largest biodiesel contributor, Indonesia has also experienced an increase in production, reaching 6.8 billion liters in 2015 [3] and 8 billion liters in 2020 [2]. Currently, the most widely used biodiesel sources are palm oil (Indonesia and Malaysia), soybean oil (The USA and Brazil), sunflower and rapeseed oil (Europe), coconut oil (Philippines), Jatropha and Mahua oil (India), Karanja oil (Australia), Neem oil (Bangladesh), and rubber seed oil (Brazil) [4, 5, 6]. However, the majority of vegetable oil sources are also food sources. This certainly triggers future price competition between the energy and food sectors [7]. Therefore, non-food sources of biodiesel are needed to avoid the problem of competition [8].

Feun Kase (*Thevetia peruviana*) is a widespread plant on the island of Timor-Indonesia and several other countries, such as India and Mexico. This plant produces 63% of vegetable oil from seeds [9]. Feun Kase is the name given by the Dawan tribe on the Timor-Indonesia island, also known as a poisonous plant. Thevetin A and thevetin B are cardiac glycosides contained in Feun Kase seed oil that have a harmful impact [10]. This makes Feun Kase seed oil unusable, so it is only used as an ornamental plant. Feun Kase oil from Gauhati University, India, has

been reported to contain fatty acids dominated by 43.72% oleic acid, 23.28% palmitic acid, 19.85% linoleic acid, 10.71% stearic acid and 2.41% arachidonic acid [11]. Meanwhile, palm oil contains oleic acid (39%), palmitic acid (45%), linoleic acid (11%), and stearic acid (4%) [12]. Feun Kase seed oil, which has fatty acid content similar to palm oil and does not compete with the food industry, can potentially be a promising source of biodiesel in the future.

The biodiesel synthesis is done through a transesterification reaction between triglyceride molecules and alcohol assisted by a catalyst. Alkaline catalysts, including NaOH, KOH, $NaOCH_3$, and $KOCH_3$, are currently the most frequently used in biodiesel [13]. Base catalysts such as NaOH have high catalytic activity in the transesterification reaction, which can give a reaction rate of 4000 times faster than acid catalysts [14]. Regarding temperature and time, Muthukumaran *et al.* [15] reported that the transesterification reaction of Madhuca Indica oil with a temperature of 60°C and reaction time of 90 minutes could give a yield of 88.71% biodiesel. In contrast, the transesterification of Karanja oil at a temperature of 70°C can yield 84% biodiesel [16]. Transesterification of sunflower oil at a temperature of 75°C with a NaOH catalyst also gave a high yield of 98% [17]. Regarding the oil/methanol molar ratio, a ratio of 1:6 is sufficient to provide a high product and is highly recommended when using methanol [18].

This study aimed to investigate the highest yield that can be achieved in synthesizing biodiesel from Feun Kase seed oil. Four important variables support the purpose of this research: catalyst, temperature, time, and the oil/methanol molar ratio, which can finally be formulated as optimal conditions. This research started with preparation, extraction, degumming, and transesterification and ended with biodiesel analysis. The analysis was conducted through parameter testing and characterization of biodiesel from the synthesis. Test parameters in this study include density, cloud point, kinematic viscosity, acid number, saponification number, iodine number, flash point, and cetane number, which can prove biodiesel quality from Feun Kase seed oil. Meanwhile, the characterization was performed using FTIR and GCMS to determine the methyl ester profile.

2. Methodology

This research was done in several steps, starting with sampling Feun Kase fruit, then preparation, extraction, degumming, transesterification, parameter testing, and characterization of biodiesel with GCMS and FTIR. The research was conducted at the University of Timor Laboratory. The parameter tests were carried out at the Center for Fuel Technology and Design Engineering – LIPI Serpong, while the FTIR and GCMS analyses were at the Laboratory of Advanced Minerals and Materials, State University of Malang.

2.1. Materials

Feun Kase seeds were collected from North Central Timor Regency, Indonesia. Solvents and other chemicals used were pro-analysis grades. The materials used were petroleum ether (Merck), H_3PO_4 (Merck), NaOH (Merck), KOH (Merck), methanol (Merck), filter paper (CV. Nurra Gemilang), distilled water (Purelizer), and anhydrous Na_2SO_4 (Merck).

2.2. Sampling

The black Feun Kase was collected from North Central Timor Regency, Indonesia. Feun Kase seeds were retrieved from the fruit after it had been peeled and then brought immediately to the lab for preparation.

2.3. Preparation

Feun Kase seeds were traditionally opened to obtain their kernel. This kernel was crushed in a blender and dried in an oven at 60°C for 4 hours [19]. The extraction process would be facilitated by crushing the kernel, and drying was intended to lower the water content. The dried Feun Kase kernel was stored in a closed container.

2.4. Extraction

The dried Feun Kase kernel was extracted to obtain the oil by maceration using petroleum ether solvent. The solvent was removed from the kernel after 60 minutes and separated from the oil by distillation. The oil obtained was filtered through filter paper, called crude oil.

2.5. Degumming

Crude oil (250 ml) was dissolved with 1% phosphoric acid in a separatory funnel with a ratio of 1:2 (v/v) at 70°C. After 24 hours, the oil was separated from the phosphoric acid solution and continued by evaporation on a hot plate at a minimum temperature of 100°C to free the oil from water molecules. Degumming was done to separate the gum and impurities contained in the oil. The degummed oil was then analyzed for acid number, %free fatty acid (FFA), density, viscosity, and cloud point tests [19].

2.6. Transesterification of Feun Kase oil

The transesterification reaction was conducted with four variables: variation of catalyst concentration, temperature, reaction time, and oil/methanol ratio. The amount of methanol and NaOH catalyst used in the reactor was adjusted according to the variations described as follows: the concentration variations of the catalyst used were 0.1% (0.25 g), 0.5% (0.13 g), 0.75% (0.19 g), 1% (0.25), 2% (0.5 g), and 5% (1.25 g) NaOH (w /w). Variations for the temperature variable were 35, 45, 55, 60, 70, and 90°C. For the time variable, variations of 30, 60, 90, 120, and 150 minutes were used. For the variable oil/methanol molar ratio, variations of 1:3, 1:6, 1:9, 1:12, and 1:15 were used, with the amount of methanol being 2.78, 5.57, 8.35, 11.14, 13.92 g, respectively.

A total of 5.57 g of methanol (molar ratio of 1:6), 0.25 g of NaOH catalyst (1%), and a magnetic stirrer were added to 25 g of crude oil which had been heated in the reactor (Duran bottle on a hot plate). The temperature was maintained at 60°C for 90 minutes. The above procedure is a standard procedure for all variables. As previously mentioned, a standard approach was employed for the variable catalyst concentration, which involved changing the concentration. This is also done for

temperature, time, and oil/methanol ratio variables. After the reaction was terminated, the mixture was poured into a separatory funnel and left for 90 minutes at room temperature to form two layers. The methyl ester layer (biodiesel) at the top was separated from the glycerol layer. This methyl ester layer was evaporated at a temperature above 65°C (to remove methanol) and then washed with warm water (40–50°C) until the pH of the wastewater became neutral (to remove the catalyst). The methyl ester was added with 1 g of anhydrous Na_2SO_4, then evaporated using a hot plate at a minimum temperature of 100°C to obtain water-free biodiesel. This biodiesel was stored in a closed container and ready for analysis. The biodiesel yield was calculated using equation (1) [20].

$$\text{Biodiesel yield} = \frac{\text{weight of biodiesel}}{\text{weight of oil}} \text{ x } 100\% \quad (1)$$

2.7. Feun Kase biodiesel analysis

Parameter tests were done to determine the quality of biodiesel, including density (SNI 7182:2015), cloud point (SNI 7182:2015), kinematic viscosity at 40°C (ASTM D445), acid number (ASTM D664), saponification number (SNI 7182: 2015), iodine number (SNI 7182:2015), flash point (ASTM D93), and cetane number that was calculated using equation (2) [21].

$$\text{CN} = 46.3 + \frac{5.458}{\text{SN}} - \text{IV} \quad (2)$$

where CN is the cetane number, SN is the saponification number, and IV is the iodine value.

The methyl ester profile was characterized using FTIR and GCMS. The state of the GCMS apparatus was as follows: Column Oven Temperature: 100°C, Injection Temperature: 250°C, Injection Mode: Split Flow, Control Mode: Linear Velocity Pressure (107.2 kPa), Total Flow: 433.8 mL/min, Column Flow: 1.43 mL/min, Linear Velocity: 44.5 cm/sec, Purge Flow: 3 mL/min, Split Ratio: 300.

3. Results and Discussion

3.1. Characteristics of Feun Kase seed oil

Feun Kase seed oil was degummed before being converted into biodiesel. The characteristics of degumming oil can be seen in Table 1, which can be concluded that degumming affected viscosity and acid number reduction.

Table 1. Characteristics of Feun Kase seed oil

Test Parameters	Crude oil	Oil after degumming
Density (kg/m³)	855	851
Cloud point (°C)	2	6
Kinematic viscosity (40°C) (mm²/s)	22.52	18.73
Acid number (mg KOH/gr)	4.91	3.23
Free fatty acids (%FFA)	2.47	1.62

3.2. Transesterification

The transesterification reaction in this study was conducted in a closed system reactor to avoid the loss of methanol molecules during heating. At the beginning of the reaction, it was seen that methanol was insoluble in oil. However, with the help of a magnetic stirrer and heating, methanol and oil can mix well. This transesterification reaction produces fatty acid methyl ester (FAME) products and glycerol [22]. Two layers were visible at the end of the reaction, with the top layer being methyl ester (pale yellow color) and the bottom layer being glycerol (dark yellow color) [23]. This is further reinforced by the fact that glycerol had a higher density (1260 kg/m³) than methyl ester (850–890 kg/m³). The transesterification process and the separation of methyl esters can be seen in Figure 1.

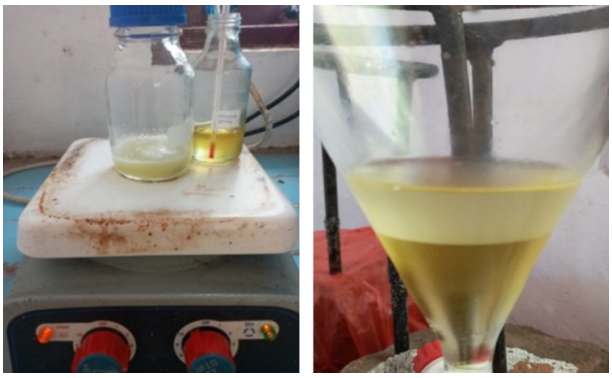


Figure 1. Transesterification process (left), methyl ester separation (right)

In almost all variables, the transesterification reaction was successfully conducted, as evidenced by the formation of a methyl ester layer. However, several variables did not perform well, such as the formation of 2 layers at 0.1% NaOH concentration variations where the methyl ester layer appeared to be relatively thin (0.11% yield), leading the crude oil layer to predominate at the separating funnel. The 5% NaOH concentration did not produce any layer. In this variable, there was 0% biodiesel yield since all the oil transformed into soap with a solid white texture. This happens because the excess amount of catalyst will form soap through the saponification reaction [24]. Three layers appeared in the variation of the oil/methanol ratio of 1:3, including methyl ester, glycerol, and soap. In this variable, the simultaneous transesterification and saponification reactions also resulted in the oil-producing soap, resulting in low biodiesel yields (52.07%). Hoda [25] discovered that an oil/methanol molar ratio of 1:3 would lead to a low yield.

3.3. Effect of the variables on biodiesel yield

3.3.1. Effect of NaOH catalyst concentration on biodiesel yield

Biodiesel yield data with various concentrations of NaOH catalyst can be seen in Figure 2. The biodiesel production using 0.1% NaOH catalyst concentration was only 0.11%, indicating that the 0.1% concentration is insufficient for converting oil into methyl esters. Hsiao *et al.* [26] reported that using 0.2% NaOH catalyst only produces a 16.3% biodiesel yield, while 1% NaOH could provide a 90.2% biodiesel yield. Moreover, using a catalyst concentration that is too high will prevent the formation of methyl esters. This can be seen as a 5% NaOH concentration results in a 0% biodiesel yield,

meaning that no methyl esters are produced. In addition, this 5% NaOH formed soap at the end of the transesterification reaction, thus indicating a saponification reaction. According to Efavi *et al.* [27], using too much NaOH catalyst creates soap and reduces biodiesel yield. The 1% NaOH catalyst concentration gave the highest biodiesel yield of 81.48%. This is in line with the results of Dhoot's research that the 1% (w/w) NaOH catalyst concentration with *Thevetia peruviana* oil resulted in the highest yield [28]. Therefore, it can be concluded that the best catalyst concentration for biodiesel synthesis from Feun Kase oil is 1% NaOH.

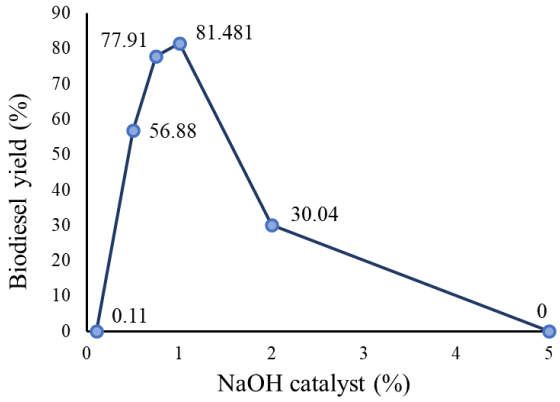


Figure 2. The yield of biodiesel with variations in NaOH catalyst concentration with an oil/methanol molar ratio of 1:6, 90 minutes, and 60°C

3.3.2. Effect of reaction temperature on biodiesel yield

Biodiesel yield data with temperature variations can be seen in Figure 3. The transesterification reaction at 35°C gave a biodiesel yield of 30.95%. This indicates that the oil cannot be optimally converted into methyl esters at low temperatures. High temperatures are also not ideal for the transesterification reaction. This can be seen from the decrease in yield at a temperature of 90°C by 59.73%.

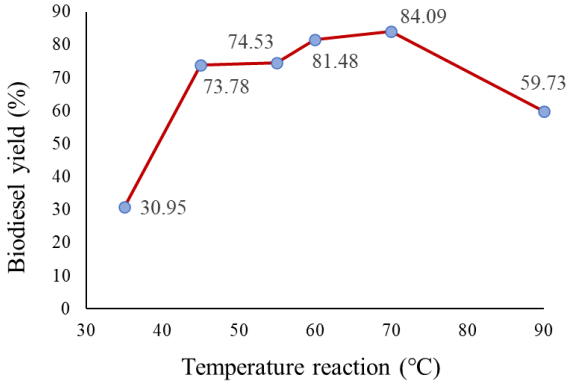


Figure 3. The yield of biodiesel with variations in temperature with 1% NaOH catalyst concentration, 90 minutes, and oil/methanol molar ratio of 1:6

Transesterification or methanolysis of vegetable oils is usually carried out near the boiling point of methanol. If the temperature is too high, a saponification process will begin before the transesterification reaction is finished [29]. The data proves that the temperature is sufficient to influence biodiesel yield. The highest yield (84.09%) was achieved in this study with synthesis at 70°C. Therefore, it can be said that 70°C is the ideal temperature for producing biodiesel from Feun Kase oil.

3.3.3. Effect of reaction time on biodiesel yield

Biodiesel yield data with variations in time can be seen in Figure 4.

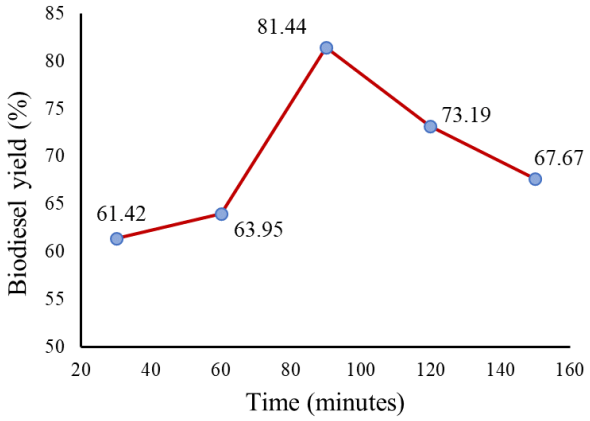


Figure 4. The yield of biodiesel with time variation with 1% NaOH catalyst concentration, 60°C temperature, and oil/methanol molar ratio of 1:6

Despite not being at its maximum, the transesterification reaction can produce a high yield of 61.42% in only 30 minutes, meaning that the chemical equilibrium cannot be shifted towards the methyl ester in 30 minutes. Increasing the transesterification reaction time will increase the conversion to methyl ester [28]. However, a prolonged reaction time can also lower biodiesel production, as evidenced by the decreasing biodiesel yield at a reaction time of 120 and 150 minutes. This is because transesterification is reversible; continuing the reaction for a more extended period leads to oil reformation [30]. This information suggests avoiding a prolonged reaction time because it could be costly and time-consuming for the biodiesel industry. The data proves that the reaction time affects the biodiesel yield. The highest yield of 81.44% can be achieved using a reaction time of 90 minutes during synthesis. Therefore, it can be concluded that the best time for synthesizing biodiesel from Feun Kase oil is 90 minutes.

3.3.4. Effect of oil/methanol molar ratio on biodiesel yield

The yield data of biodiesel with various molar ratios between oil and methanol can be seen in Figure 5. The transesterification reaction at a ratio of 1:3 gave the lowest yield of 52.07%. Stoichiometrically, 1 mole of oil reacts precisely with 3 moles of methanol to produce 3 moles of methyl ester (biodiesel) [18]. However, the methanol ratio must be higher in practice than the stoichiometric ratio to increase the collision between methanol and oil and generate a high yield [31]. This can be seen in the comparison data of 1:6 and more, which gives a higher yield, where the oil/methanol ratio of 1:6 is sufficient to produce a high yield of 73.59%. Researchers also observed the formation of soap in the transesterification reaction at a ratio of 1:3. This proves that the small amount of methanol, as well as the low

solubility of oil and methanol, causes the NaOH catalyst to react with free fatty acids to form soap [29], considering that the free fatty acid content in this study was also relatively high (4.91 mg KOH/gr).

The most frequently used oil/methanol ratio is 1:6 [13]. According to Hsiao *et al.* [26], the ratio of 1:6 greatly enhanced biodiesel yield, but the ratio of 1:12 was not particularly significant. This is because the amount of methanol is sufficient to dissolve the catalyst and support the transesterification reaction. In this study, biodiesel synthesis with an oil/methanol ratio of 1:6 had the highest yield of 73.59%. Therefore, this leads to the conclusion that a maximum reaction can be achieved with a ratio of 1:6, whereas a larger ratio does not significantly improve reaction performance.

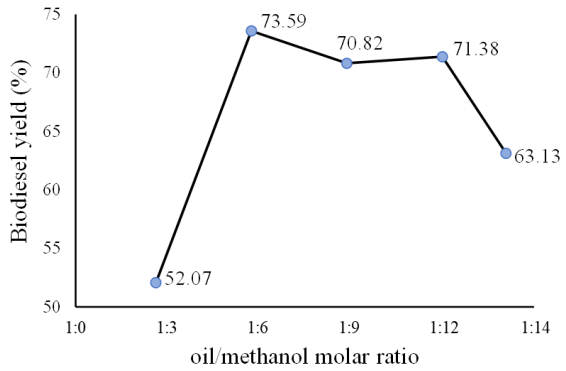


Figure 5. The yield of biodiesel with various molar ratios with 1% NaOH catalyst concentration, 60°C, and 90 minutes

3.4. Feun Kase Biodiesel Analysis

The analysis of biodiesel in this study was performed with several test parameters according to SNI 7181:2015. Parameters tested include density, cloud point, kinematic viscosity, acid number, free fatty acid (FFA), iodine number, saponification number, flash point, and cetane number. Biodiesel has a pale yellow color floating on top of the glycerol layer at the end of the reaction. Additionally, compared to crude oil, biodiesel has lower flash point and viscosity characteristics [32]. Feun Kase biodiesel can be seen in Figure 6, and Table 2 shows the results of parameter tests comparing Feun Kase biodiesel and its crude oil.

Figure 6. Crude oil (left) and Feun Kase biodiesel (right)

The results of the Feun Kase biodiesel parameter test can be seen in Table 2. The researcher added Feun Kase crude oil analysis data in Table 2 for comparison. According to the analysis, Feun Kase biodiesel conformed with SNI 7181:2015, excluding the acid number.

Table 2. Feun Kase biodiesel parameter test results

Test Parameters	Crude oil	Biodiesel Feun Kase	SNI 7181:2015
Density (kg/m³)	855	851	850-890
Cloud point (°C)	2	6	Max. 18
Kinematic viscosity (40°C) (mm²/s)	22.52	5.53	2.3-6
Acid number (mg KOH/gr)	4.91	1.08	Max. 0.8
Free Fatty Acids (%FFA)	2.47	0.54	-
Iodine number (g I_2/100 gr)	137.44	78.62	Max. 115
Saponification number (mg KOH/gr)	165.65	159.32	-
Flash point (°C)	324	165	Min. 100
Cetane number	50.55	62.86	51

Information on the acid number is intended to reveal the potential for corrosion, which can reduce the shelf life of the vehicle's fuel tank [33]. The acid number of biodiesel with a value of 1.08 mg KOH/gr has exceeded the SNI limit. The high acid number in biodiesel comes from the high acid number of crude oil. This indicates that Feun Kase crude oil contains a significant level of free fatty acids, considering that esterification was not performed throughout this study. Cai *et al.* [34] produced biodiesel from used cooking oil with an acid number of 124.9 mg KOH/g. Cai *et al.* [34] reported that biodiesel production with two reaction steps (esterification followed by transesterification) could give a 93.1% biodiesel yield. Therefore, performing esterification before biodiesel synthesis is strongly advised.

Density, viscosity, flash point, iodine number, and cetane number are the most frequently investigated biodiesel parameters because they significantly affect combustion, performance, and emissions in diesel engines [35]. Density and viscosity greatly affect the quality of atomization, droplet size, and penetration of biodiesel through the injection system. Therefore, the density standard has been determined, namely 850–890 kg/m³, and the density of Feun Kase biodiesel (851 kg/m³) has complied with SNI. Panchal stated that biodiesel from *Thevetia peruviana* from India only has a density of 840 kg/m³ [36].

Feun Kase's biodiesel viscosity (5.53 mm²/s) has met the standard limits assigned by SNI. This viscosity measurement parameter has a considerable effect on the combustion performance of the engine. Higher viscosity (over 6 mm²/s) will lead to slow-burning of the fuel, raise the temperature of the diesel engine [37], and also cause problems in exhaust emissions caused by incomplete combustion [38]. The viscosity of biodiesel in this study was thicker than biodiesel from *Thevetia peruviana* from India, which was 4.45 mm²/s [36]. Although the viscosity of the biodiesel in this study was not significantly different from that of the biodiesel made in Nigeria using *Thevetia peruviana*. Yarkasuwa *et al.* [39] reported that the viscosity of biodiesel from Nigeria was 5.10 mm²/s, which also meets SNI.

The flash point of Feun Kase biodiesel (165°C) also met SNI. The actual flash point has an indirect effect on engine performance. However, information on these parameters is used for safe storage and transportation requirements in compliance with flammable materials regulations [40]. The flash point in this study was similar

to the flash point of *Thevetia peruviana* biodiesel from Nigeria, which was 175°C [36].

Feun Kase biodiesel iodine number (78.62 g I_2/100 gr) also met SNI. The iodine number indicates the tendency of the oil to oxidize and polymerize, thus forming deposits on the engine. The higher the iodine number, the more deposits stick to the machine [41]. Therefore, the maximum iodine number determined by SNI is 115 g I_2/100 gr.

The cetane number of Feun Kase biodiesel (62.86) has also met SNI. The cetane number in this study was obtained by calculating the saponification and iodine numbers. The cetane number is mainly responsible for the engine's ignition delay (the time between injection and fuel combustion). The higher the cetane number, the better because the ignition delay time will be shorter and reduce knocking [42]. In short, the ignition delay will also accelerate the heat dissipation rate, so the engine does not require high heat during the combustion process [43]. Therefore, the high cetane number will make the engine run smoothly and quietly [44]. This study's cetane number of biodiesel was higher than that of *Thevetia peruviana* biodiesel from Nigeria. Yarkasuwa *et al.* [39] revealed that Nigerian biodiesel had a cetane number of 58.97.

3.5. Characterization of methyl esters

3.5.1. FTIR analysis

FTIR analysis in this study aimed to identify functional groups in Feun Kase biodiesel. The FTIR spectra of Feun Kase biodiesel in this study can be seen in Figure 7 or the supplementary file for more details. The synthesized biodiesel can be seen from the presence of methyl esters from the absorptions. The absorption of the ester group was seen at a wavenumber of 1743 cm^{-1} which showed C=O stretching and O-CH_3 stretching at 1170.79 cm^{-1} and 1195.87 cm^{-1} [45]. The spectra also show CH_3 asymmetric stretching at 1436.97 cm^{-1} and O-CH_3 stretching at 1195.87 cm^{-1}. Torres *et al.* [46] explained that the FTIR spectra at wavenumbers around 1195 cm^{-1} and 1436 cm^{-1} are the main differences between methyl ester and oil spectra, where both wavenumbers only appear in methyl ester spectra.

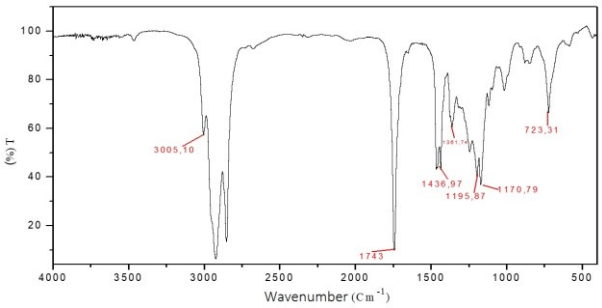


Figure 7. FTIR spectra of Feun Kase biodiesel

The same thing was also reported by Rabelo *et al.* [47] that CH_3 asymmetric stretching at 1436.97 cm^{-1} only appeared in the methyl ester spectra. The regions of 1425-1447 cm^{-1} and 1188-1200 cm^{-1} are typical absorptions of methyl esters (unavailable in the oil spectra), which prove the transformation from oil to biodiesel [48, 49]. The success of biodiesel synthesis in this study was also supported by the absence of wavenumbers in the absorption region of 1370-1400 cm^{-1} for O–CH_2 (glycerol groups from tri-, di-, monoglycerides) and 1075-1111 cm^{-1} for O–CH_2-C, where the uptakes indicate the presence of oil molecules [50].

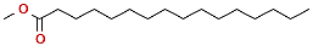
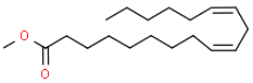
Wavenumbers of 2924.09 cm^{-1} and 2854.74 cm^{-1} confirmed the presence of stretching (aliphatic) C-H groups, which were strengthened by a typical bending C-H absorption at 1436.97 cm^{-1}. The wavenumber of 723.31 cm^{-1} also proves the presence of $-(CH_2)_n-$, which indicates the presence of long-chain alkane hydrocarbons. The peak of 3005 cm^{-1} indicates the presence of =C-H stretching, which demonstrates that Feun Kase biodiesel also contains carbon double bonds (alkenes) in the aliphatic chain [45].

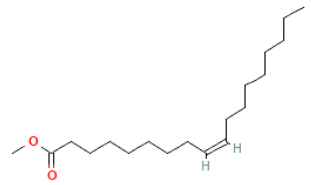
3.5.2. GCMS analysis

GCMS analysis in this study was used to identify the methyl ester profile of Feun Kase biodiesel. GCMS mass spectra of Feun Kase biodiesel can be seen in the supplementary file for more details. The GCMS mass spectra of Feun Kase biodiesel showed four peaks with a large percentage of area, namely the retention time of 29.121, 35.395, 35.574, and 36.308, while the other peaks only have an area percentage below 1%.

Feun Kase methyl ester profile showed in Table 3. It is revealed that the methyl ester profile of Feun Kase is primarily dominated by methyl oleate (53.45%), followed by methyl palmitate (27.05%). When compared to other research, methyl palmitate (45%) and methyl oleate (39%) were the two main components of the palm oil-derived biodiesel [12], used cooking oil from Arabia was predominated by methyl palmitate (36.9%), and methyl oleate (31.6%) [51], Indonesian coconut oil was dominated by methyl laurate (32.4%) and methyl oleate (3.9%) [52], sunflower oil from Iraq was predominated by methyl linoleate (63.4%) and methyl oleate (24.6%) [53], methyl linoleate (45.59%) and methyl oleate (16.36%) were the two main components of the Chinese soybean oil [54], whereas *Thevetia peruviana* oil from India dominated by methyl oleate (43.72%) and methyl palmitate (23.28%) [11]. With the above comparison, Feun Kase biodiesel contains the highest methyl oleate (53.45%) among all biodiesel resources.

Table 3. Feun Kase methyl ester profile

Methyl Esters	Molecular Formula	Retention Time	% Area
Methyl palmitate 	$C_{17}H_{34}O_2$	29.121	27.05
Methyl linoleate 	$C_{19}H_{34}O_2$	35.395	6.29
Methyl oleate	$C_{19}H_{36}O_2$	35.574	53.45

 Methyl stearate	$C_{19}H_{38}O_2$	36.308	10.96

The number of double bonds is an essential factor affecting the oxidation rate: the more double bonds, the more susceptible it to oxidation [41]. However, the presence of double bonds in biodiesel also positively impacts decreasing viscosity (more liquid), so it can improve biodiesel performance at low temperatures. Unsaturated fatty acid methyl ester (FAME) has a lower viscosity than saturated FAME. This can be explained by considering molecular geometry. Almost all FAMEs with pi bonds always have a cis configuration, so these molecules do not stack effectively. In other words, the cis configuration prevents the sp2 carbon of neighboring molecules from approaching, so the intermolecular interactions in unsaturated FAME are weaker than in saturated FAME [55].

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In comparison, biodiesel from fish oil is very susceptible to oxidation because it contains molecules with double bonds of more than three [41]. Feun Kase biodiesel is dominated by methyl oleate, which only has one double bond, so it can be said that Feun Kase biodiesel has good oxidation stability and, at the same time, has a positive impact on viscosity.

4. Conclusion

A series of results of this study concluded that the optimum conditions for biodiesel synthesis from Feun Kase seed oil were at 1% NaOH catalyst, 70°C, 90 minutes, and an oil/methanol molar ratio of 1:6. The maximum biodiesel yield achieved in this study was 84.09%. Parameter tests of Feun Kase biodiesel were density (851 kg/m^3), cloud point (6°C), kinematic viscosity (5.35 mm^2/s), acid number (1.08 mg KOH/gr), iodine number (78.62 g I_2/100 gr), saponification number (159.32 mg KOH/gr), flash point (165°C), and cetane number (62.86). In addition to the acid number, the quality of biodiesel from Feun Kase seed oil has complied with SNI 7182:2015. FTIR analysis proved the presence of a methyl ester structure at typical absorptions of 1743 cm^{-1}, 1195.87 cm^{-1}, and 1436.97 cm^{-1}. GCMS results showed that biodiesel from Feun Kase seed oil was dominated by methyl oleate (53.45%), methyl palmitate (27.05%), methyl stearate (10.96%), and methyl linoleate (6.29%).

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References

[1] N. D. Avarskii, V. V. Taran, K. N. Gasanova, A. N. Osipov, E. A. Silko, V. R. Gumerov, World biodiesel market and optimal scenarios for biodiesel production and use in Russia, *International Journal of Innovative Technology and Exploring Engineering*, 8, 12, (2019), 4026-4038 http://doi.org/10.35940/ijitee.L3625.1081219

[2] Ren21, *Renewables 2021 Global Status Report*, REN21 Secretariat, Paris, 2021

[3] Andika Pambudi, Eka Puspitawati, Nursechafia Nursechafia, The determinants of biodiesel export in Indonesia, *Signifikan: Jurnal Ilmu Ekonomi*, 8, 2, (2019), 207-216 https://doi.org/10.15408/sjie.v8i2.10961

[4] Dong-Shik Kim, Mohammadmatin Hanifzadeh, Ashok Kumar, Trend of biodiesel feedstock and its impact on biodiesel emission characteristics, *Environmental Progress & Sustainable Energy*, 37, 1, (2018), 7-19 https://doi.org/10.1002/ep.12800

[5] Ayhan Demirbas, Progress and recent trends in biodiesel fuels, *Energy Conversion and Management*, 50, 1, (2009), 14-34 https://doi.org/10.1016/j.enconman.2008.09.001

[6] Digambar Singh, Dilip Sharma, S. L. Soni, Sumit Sharma, Pushpendra Kumar Sharma, Amit Jhalani, A review on feedstocks, production processes, and yield for different generations of biodiesel, *Fuel*, 262, 116553, (2020), 1-15 https://doi.org/10.1016/j.fuel.2019.116553

[7] Amela Ajanovic, Biofuels versus food production: does biofuels production increase food prices?, *Energy*, 36, 4, (2011), 2070-2076 https://doi.org/10.1016/j.energy.2010.05.019

[8] Simla Tokgoz, Wei Zhang, Siwa Msangi, Prapti Bhandary, Biofuels and the future of food: competition and complementarities, *Agriculture*, 2, 4, (2012), 414-435 https://doi.org/10.3390/agriculture2040414

[9] S. A. Ibiyemi, V. O. Fadipe, O. O. Akinremi, S. S. Bako, Variation in oil composition of *Thevetia peruviana* Juss' yellow oleander' fruit seeds, *Journal of Applied Sciences and Environmental Management*, 6, 2, (2002), 61-65 https://doi.org/10.4314/jasem.v6i2.17178

[10] K. Singh, K. K. Agrawal, V. Mishra, S. M. Uddin, A. Shukla, A review on: *Thevetia peruviana*, *International Research Journal of Pharmacy*, 3, 4, (2012), 74-77

[11] Dibakar Chandra Deka, Sanjay Basumatary, High quality biodiesel from yellow oleander (*Thevetia peruviana*) seed oil, *Biomass and Bioenergy*, 35, 5,

(2011), 1797-1803
https://doi.org/10.1016/j.biombioe.2011.01.007

[12] Khairul Azly Zahan, Manabu Kano, Biodiesel production from palm oil, its by-products, and mill effluent: A review, *Energies*, 11, 8, (2018), https://doi.org/10.3390/en11082132

[13] Mohd Nurfirdaus Bin Mohiddin, Yie Hua Tan, Yee Xuan Seow, Jibrail Kansedo, N. M. Mubarak, Mohammad Omar Abdullah, Yen San Chan, Mohammad Khalid, Evaluation on feedstock, technologies, catalyst and reactor for sustainable biodiesel production: A review, *Journal of Industrial and Engineering Chemistry*, 98, (2021), 60-81 https://doi.org/10.1016/j.jiec.2021.03.036

[14] I. M. Rizwanul Fattah, H. C. Ong, T. M. I. Mahlia, M. Mofijur, A. S. Silitonga, S. M. Ashrafur Rahman, Arslan Ahmad, State of the art of catalysts for biodiesel production, *Frontiers in Energy Research*, 8, 101, (2020), https://doi.org/10.3389/fenrg.2020.00101

[15] Chandrasekaran Muthukumaran, Ramachandran Praniesh, Periyasamy Navamani, Raghavan Swathi, Govindasamy Sharmila, Narasimhan Manoj Kumar, Process optimization and kinetic modeling of biodiesel production using non-edible *Madhuca indica* oil, *Fuel*, 195, (2017), 217-225 https://doi.org/10.1016/j.fuel.2017.01.060

[16] P. K. Srivastava, Madhumita Verma, Methyl ester of karanja oil as an alternative renewable source energy, *Fuel*, 87, 8-9, (2008), 1673-1677 https://doi.org/10.1016/j.fuel.2007.08.018

[17] Vlada B. Veljković, Ana V. Veličković, Jelena M. Avramović, Olivera S. Stamenković, Modeling of biodiesel production: Performance comparison of Box–Behnken, face central composite and full factorial design, *Chinese Journal of Chemical Engineering*, 27, 7, (2019), 1690-1698 https://doi.org/10.1016/j.cjche.2018.08.002

[18] Idris Atadashi Musa, The effects of alcohol to oil molar ratios and the type of alcohol on biodiesel production using transesterification process, *Egyptian Journal of Petroleum*, 25, 1, (2016), 21-31 https://doi.org/10.1016/j.ejpe.2015.06.007

[19] Jefry Presson, Yohana Ivana Kedang, Suwari Suwari, The Physicochemical Profile of Feun Kase (*Thevetia Peruviana*) Oil As A New Feedstock For Renewable Energy, *Savana Cendana*, 6, 01, (2021), 11-15 https://doi.org/10.32938/sc.v6i01.1238

[20] R. E. E. Ana Godson, G. Udofia Bassey, Characterization of oil and biodiesel produced from *Thevetia peruviana* (yellow oleander) seeds, *International Journal of Sustainable and Green Energy*, 4, 4, (2015), 150-158

[21] José Luis Guil-Laynez, José Luis Guil-Guerrero, Álvaro Guil-Laynez, Bioprospecting for seed oils in tropical areas for biodiesel production, *Industrial Crops and Products*, 128, (2019), 504-511 https://doi.org/10.1016/j.indcrop.2018.11.044

[22] Puneet Verma, M. P. Sharma, Review of process parameters for biodiesel production from different feedstocks, *Renewable and Sustainable Energy Reviews*, 62, (2016), 1063-1071 https://doi.org/10.1016/j.rser.2016.04.054

[23] Jesús Andrés Tavizón-Pozos, Gerardo Chavez-Esquivel, Víctor Alejandro Suárez-Toriello, Carlos Eduardo Santolalla-Vargas, Oscar Abel Luévano-Rivas, Omar Uriel Valdés-Martínez, Alfonso Talavera-López, Jose Antonio Rodriguez, State of art of alkaline earth metal oxides catalysts used in the transesterification of oils for biodiesel production, *Energies*, 14, 4, (2021), https://doi.org/10.3390/en14041031

[24] A. Suryanto, Suprapto Suprapto, Mahfud Mahfud, Production of Biodiesel from Coconut Oil Using Microwave: Effect of Some Parameters on Transesterification Reaction by NaOH Catalyst, *Bulletin of Chemical Reaction Engineering & Catalysis*, 10, 2, (2015), 162-168 https://doi.org/10.9767/bcrec.10.2.8080.162-168

[25] N. Hoda, Optimization of biodiesel production from cottonseed oil by transesterification using NaOH and methanol, *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects*, 32, 5, (2010), 434-441 https://doi.org/10.1080/15567030802612168

[26] Ming-Chien Hsiao, Peir-Horng Liao, Nguyen Vu Lan, Shuhn-Shyurng Hou, Enhancement of biodiesel production from high-acid-value waste cooking oil via a microwave reactor using a homogeneous alkaline catalyst, *Energies*, 14, 2, (2021), 1-11 https://doi.org/10.3390/en14020437

[27] J. K. Efavi, D. Dodoo-Arhin, Dindomah Kanbogtah, V. Apalangya, E. Nyankson, E. K. Tiburu, B. Onwona-Agyeman, A. Yaya, The effect of NaOH catalyst concentration and extraction time on the yield and properties of *Citrullus vulgaris* seed oil as a potential biodiesel feed stock, *South African Journal of Chemical Engineering*, 25, 1, (2018), 98-102 https://doi.org/10.1016/j.sajce.2018.03.002

[28] S. B. Dhoot, D. R. Jaju, S. A. Deshmukh, B. M. Panchal, M. R. Sharma, Extraction of Thevetia peruviana seed oil and optimization of biodiesel production using Alkalicatalyzed methanolysis, *Journal of Alternate Energy Sources and Technologies*, 2, 2, (2011), 8-16

[29] Andrew C. Eloka-Eboka, Ogbene Gillian Igbum, Freddie L. Inambao, Optimization and effects of process variables on the production and properties of methyl ester biodiesel, *Journal of Energy in Southern Africa*, 25, 2, (2014), 39-47

[30] Achanai Buasri, Nattawut Chaiyut, Vorrada Loryuenyong, Phatsakon Worawanitchaphong, Sarinthip Trongyong, Calcium oxide derived from waste shells of mussel, cockle, and scallop as the heterogeneous catalyst for biodiesel production, *The Scientific World Journal*, 2013, 460923, (2013), 1-7 https://doi.org/10.1155/2013/460923

[31] Jin-Suk Lee, Shiro Saka, Biodiesel production by heterogeneous catalysts and supercritical technologies, *Bioresource Technology*, 101, 19, (2010), 7191-7200 https://doi.org/10.1016/j.biortech.2010.04.071

[32] Dennis Y. C. Leung, Xuan Wu, M. K. H. Leung, A review on biodiesel production using catalyzed transesterification, *Applied Energy*, 87, 4, (2010), 1083-1095 https://doi.org/10.1016/j.apenergy.2009.10.006

[33] Fabiano Barbieri Gonzaga, Sidney Pereira Sobral, A new method for determining the acid number of

biodiesel based on coulometric titration, *Talanta*, 97, (2012), 199-203 https://doi.org/10.1016/j.talanta.2012.04.017

[34] Zi-Zhe Cai, Yong Wang, Ying-Lai Teng, Ka-Man Chong, Jia-Wei Wang, Jie-Wen Zhang, De-Po Yang, A two-step biodiesel production process from waste cooking oil via recycling crude glycerol esterification catalyzed by alkali catalyst, *Fuel Processing Technology*, 137, (2015), 186-193 https://doi.org/10.1016/j.fuproc.2015.04.017

[35] M. S. M. Zaharin, N. R. Abdullah, G. Najafi, H. Sharudin, T. Yusaf, Effects of physicochemical properties of biodiesel fuel blends with alcohol on diesel engine performance and exhaust emissions: A review, *Renewable and Sustainable energy reviews*, 79, (2017), 475-493 https://doi.org/10.1016/j.rser.2017.05.035

[36] Balaji M. Panchal, Sanjay A. Deshmukh, Munish R. Sharma, Biodiesel from *Thevetia peruviana* seed oil with dimethyl carbonate using as an active catalyst potassium-methoxide, *Sains Malaysiana*, 45, 10, (2016), 1461-1468

[37] Tikendra Nath Verma, Pankaj Shrivastava, Upendra Rajak, Gaurav Dwivedi, Siddharth Jain, Ali Zare, Anoop Kumar Shukla, Puneet Verma, A comprehensive review of the influence of physicochemical properties of biodiesel on combustion characteristics, engine performance and emissions, *Journal of Traffic and Transportation Engineering (English Edition)*, 8, 4, (2021), 510-533 https://doi.org/10.1016/j.jtte.2021.04.006

[38] Ertan Alptekin, Mustafa Canakci, Characterization of the key fuel properties of methyl ester–diesel fuel blends, *Fuel*, 88, 1, (2009), 75-80 https://doi.org/10.1016/j.fuel.2008.05.023

[39] Chindo Istifanus Yarkasuwa, Danbature Wilson, Emmanuel Michael, Production of biodiesel from Yellow Oleander (*Thevetia peruvian*) Oil and its Biodegradability, *Journal of the Korean Chemical Society*, 57, 3, (2013), 377-381 https://doi.org/10.5012/jkcs.2013.57.3.377

[40] Jorge Henrique Faber Boog, Eva Lúcia Cardoso Silveira, Lilia Basilio De Caland, Matthieu Tubino, Determining the residual alcohol in biodiesel through its flash point, *Fuel*, 90, 2, (2011), 905-907 https://doi.org/10.1016/j.fuel.2010.10.020

[41] James Pullen, Khizer Saeed, An overview of biodiesel oxidation stability, *Renewable and Sustainable Energy Reviews*, 16, 8, (2012), 5924-5950 https://doi.org/10.1016/j.rser.2012.06.024

[42] M. U. Kaisan, F. O. Anafi, J. Nuszkowski, D. M. Kulla, S. Umaru, Exhaust emissions of biodiesel binary and multi-blends from Cotton, Jatropha and Neem oil from stationary multi cylinder CI engine, *Transportation Research Part D: Transport and Environment*, 53, (2017), 403-414 https://doi.org/10.1016/j.trd.2017.04.040

[43] Paramvir Singh, S. R. Chauhan, Varun Goel, Assessment of diesel engine combustion, performance and emission characteristics fuelled with dual fuel blends, *Renewable Energy*, 125, (2018), 501-510 https://doi.org/10.1016/j.renene.2018.02.105

[44] Gerhard Knothe, Andrew C. Matheaus, Thomas W. Ryan III, Cetane numbers of branched and straight-chain fatty esters determined in an ignition quality tester*, *Fuel*, 82, 8, (2003), 971-975 https://doi.org/10.1016/S0016-2361(02)00382-4

[45] Asep Bayu Dani Nandiyanto, Rosi Oktiani, Risti Ragadhita, How to read and interpret FTIR spectroscope of organic material, *Indonesian Journal of Science and Technology*, 4, 1, (2019), 97-118 https://doi.org/10.17509/ijost.v4i1.15806

[46] Alicia Torres, Beatriz Fuentes, Karina E. Rodríguez, Andrea Brito, Laura Díaz, Analysis of the Content of Fatty Acid Methyl Esters in Biodiesel by Fourier-Transform Infrared Spectroscopy: Method and Comparison with Gas Chromatography, *Journal of the American Oil Chemists' Society*, 97, 6, (2020), 651-661 https://doi.org/10.1002/aocs.12350

[47] Sabrina N. Rabelo, Vany P. Ferraz, Leandro S. Oliveira, Adriana S. Franca, FTIR analysis for quantification of fatty acid methyl esters in biodiesel produced by microwave-assisted transesterification, *International Journal of Environmental Science and Development*, 6, 12, (2015), https://www.doi.org/10.7763/IJESD.2015.V6.730

[48] Morgana Rosset, Oscar W. Perez-Lopez, FTIR spectroscopy analysis for monitoring biodiesel production by heterogeneous catalyst, *Vibrational Spectroscopy*, 105, 102990, (2019), 1-6 https://doi.org/10.1016/j.vibspec.2019.102990

[49] Muhammad Waseem Mumtaz, Ahmad Adnan, Farooq Anwar, Hamid Mukhtar, Muhammad Asam Raza, Farooq Ahmad, Umer Rashid, Response surface methodology: An emphatic tool for optimized biodiesel production using rice bran and sunflower oils, *Energies*, 5, 9, (2012), 3307-3328 https://doi.org/10.3390/en5093307

[50] Naresh N. Mahamuni, Yusuf G. Adewuyi, Fourier transform infrared spectroscopy (FTIR) method to monitor soy biodiesel and soybean oil in transesterification reactions, petrodiesel- biodiesel blends, and blend adulteration with soy oil, *Energy & Fuels*, 23, 7, (2009), 3773-3782 https://doi.org/10.1021/ef900130m

[51] R. Abd Rabu, I. Janajreh, D. Honnery, Transesterification of waste cooking oil: Process optimization and conversion rate evaluation, *Energy Conversion and Management*, 65, (2013), 764-769 https://doi.org/10.1016/j.enconman.2012.02.031

[52] Kamisah Delilawati Pandiangan, Wasinton Simanjuntak, Transesterification of coconut oil using dimethyl carbonate and TiO_2/SiO_2 heterogeneous catalyst, *Indonesian Journal of Chemistry*, 13, 1, (2013), 47-52 https://doi.org/10.22146/ijc.21325

[53] Maimoonah Kh. Qasim, Modified nanostructure MgO superbasicity with CaO in heterogeneous transesterification of sunflower oil, *Egyptian Journal of Chemistry*, 62, 3, (2019), 475-485 https://dx.doi.org/10.21608/ejchem.2018.4321.1386

[54] Balaji Panchal, Tao Chang, Shenjun Qin, Yuzhuang Sun, Jinxi Wang, Kai Bian, Optimization of soybean oil transesterification using an ionic liquid and methanol for biodiesel synthesis, *Energy Reports*, 6, (2020), 20-27 https://doi.org/10.1016/j.egyr.2019.11.028

[55] Luis Felipe Ramírez-Verduzco, Javier Esteban Rodríguez-Rodríguez, Alicia del Rayo Jaramillo-

Jacob, Predicting cetane number, kinematic viscosity, density and higher heating value of biodiesel from its fatty acid methyl ester composition, *Fuel*, 91, 1, (2012), 102-111
https://doi.org/10.1016/j.fuel.2011.06.070