

SYNTHESIS OF BIODIESEL FROM USED COOKING OIL USING COMPOSITE PHOTOCATALYST FROM MILKFISH BONES AND TiO₂

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Abstract: The increasing number of vehicles in Indonesia has increased air pollution and fuel consumption. The fuels commonly used come from fossil fuels, the availability of which is limited. One solution that can be applied is switching to alternative energy sources, such as biodiesel from vegetable oils, including cooking oil. This study investigated the effects of the reactant ratio, catalyst weight, UV exposure, and catalyst reusability on biodiesel yield and characteristics (density and viscosity) according to Indonesian national standards (SNI). The method used involves the impregnation of CaO catalysts with TiO₂ photocatalysts and simultaneous transesterification-esterification reactions for biodiesel production, with oil to methanol molar ratios of 1:6, 1:9, and 1:12 and catalyst weights of 3, 5, and 7%, respectively. The results revealed that the best product had a yield of 90.6%, a density of 882 kg/m³, and a viscosity of 2.45 mm²/s at a reactant ratio of 1:9 and a catalyst weight of 5%, with UV exposure for 4 hours at 65°C. XRD and EDS analysis revealed the presence of CaO-TiO₂ compounds in the synthesized photocatalyst, and GC-MS analysis revealed that the FAME (fatty acid methyl ester) content in the biodiesel was greater than 96%.

Keywords: Biodiesel; Photocatalyst; Cooking Oil; TiO₂; Milkfish Bone

Abstrak: Meningkatnya jumlah kendaraan di Indonesia telah meningkatkan polusi udara dan konsumsi bahan bakar. Bahan bakar yang biasa digunakan berasal dari fosil, yang ketersediaannya terbatas. Salah satu solusi yang dapat diterapkan adalah beralih ke energi alternatif, seperti biodiesel dari minyak nabati, termasuk minyak jelantah. Penelitian ini menyelidiki pengaruh rasio reaktan, berat katalis, paparan UV, dan penggunaan kembali katalis terhadap hasil dan karakteristik biodiesel (densitas dan viskositas) sesuai standar nasional Indonesia (SNI). Metode yang digunakan melibatkan impregnasi katalis CaO dengan fotokatalis TiO₂ dan reaksi transesterifikasi-esterifikasi simultan untuk produksi biodiesel, dengan rasio molar minyak terhadap metanol 1:6, 1:9, dan 1:12 dan bobot katalis 3, 5, dan 7%. Hasil penelitian menunjukkan bahwa produk terbaik memiliki hasil 90,6%, kerapatan 882 kg/m³, dan viskositas

2,45 mm²/s pada rasio reaktan 1:9 dan berat katalis 5%, dengan paparan UV selama 4 jam pada suhu 65°C. Analisis XRD dan EDS mengungkapkan adanya senyawa CaO-TiO₂ dalam fotokatalis yang disintesis, dan analisis GCMS menunjukkan bahwa kandungan FAME (*fatty acid methyl ester*) dalam biodiesel lebih dari 96%.

Kata kunci: Biodiesel; Fotokatalis; Minyak Jelantah; TiO₂; Tulang Ikan Bandeng

INTRODUCTION

The increasing number of vehicle users in Indonesia has increased fuel consumption. Most of the fuel comes from fossil fuels, so the need for fossil fuels is also increasing. Based on statistical data from the Special Task Force for Oil and Gas (SKK Migas) in February 2024, Indonesia's oil reserves were only approximately 4.7 billion barrels (Setiawan, 2024). Therefore, alternative energy sources are needed, such as biodiesel, a renewable fuel that is environmentally friendly and nontoxic, has a lower CO emission content, and has characteristics similar to those of diesel (Dai *et al.*, 2016). Biodiesel consists of methyl esters from the esterification or transesterification process of vegetable materials such as palm oil and animal fats. However, the development of biodiesel is hampered by the high cost of raw materials compared with that of fossil fuels. The high price of oil and fat results in high total production costs (Talha and Sulaiman, 2016). One alternative raw material that is cheaper and available in large quantities in Indonesia is cooking oil. The cooking oil used is no longer good

because it harms health, produces dangerous free radical compounds, and can cause cell damage. This oil can damage internal organs such as the liver and kidneys (Zhu *et al.*, 2021). Therefore, using cooking oil as a raw material for biodiesel is a good alternative in terms of the economy, and its availability is high. According to the National Team for the Acceleration of Poverty Reduction (TNP2K), in 2019, the amount of cooking oil used in Indonesia ranged from 6.46 – 9.72 million kiloliters (The Ministry of Energy and Mineral Resources, 2020). Owing to the abundance of this raw material, the use of cooking oil for biodiesel is a solution for processing this waste. The manufacture of biodiesel requires a suitable catalyst to achieve good results. The use of homogeneous catalysts such as NaOH is very sensitive to the presence of free fatty acids and air. The presence of these components causes soap formation, making separation and purification difficult (Abdullah *et al.*, 2017). Moreover, heterogeneous catalysts are easy to separate and can be reused after activation. In addition, heterogeneous catalysts can be synthesized from

environmentally friendly materials and are available in nature. Banten Province is famous for its milkfish products. This business produces a large amount of milkfish bone waste that has not been utilized properly. This study aims to use milkfish bone waste as a heterogeneous catalyst derived from CaO to produce biodiesel. Bones have previously been used as catalysts in biodiesel synthesis. Ayodeji *et al.* (2018) used cow bones to produce biodiesel from soybean oil, yielding 92.2% yield. Alsaiari *et al.* (2023) used fish bones to synthesize biodiesel from date seed oil and produced a yield of 89%. Another study used a mixture of fish and chicken bones to produce biodiesel from cooking oil, yielding 89.5% yield (Tan *et al.*, 2019). Although it results in high yields, the CaO derivative catalyst has weaknesses in terms of stability and stripping when reused. This stripping can cause deactivation. To overcome this problem, it can be done by supporting it on a carrier material or mixing it with other oxides (Lukić *et al.*, 2018). Therefore, milkfish bones were impregnated with TiO₂ as a photocatalyst to increase the biodiesel yield. TiO₂ was chosen as a photocatalyst because of its good performance, environmental friendliness, relative solid nature, and stability compared with other nanomaterials

(Adiwibowo *et al.*, 2022) (Jan *et al.*, 2022). In addition, the combination of CaO and TiO₂ can produce better yields than can the combination of only CaO or TiO₂ (Sisca *et al.*, 2021). This study used a composite photocatalyst of milkfish bones and TiO₂ with a heterogeneous catalyst from milkfish bones to increase the biodiesel yield.

METHOD

MILKFISH BONE PREPARATION

The fish bones separated from the head and tail were cleaned with water and boiled for 30 minutes. Then, the bones were soaked in acetone for 3×24 hours, with acetone replacement every 24 hours. This process was followed by drying the bones in the oven at 105°C for 2 hours. Once dried, the bones were crushed and filtered through a 200-mesh sieve.

SYNTHESIS OF CALCIUM OXIDE FROM MILKFISH BONES

Milkfish bone powder was then calcined to produce a CaO catalyst by heating it at 900°C in a furnace for 4 hours. The dried catalyst was stored in a silica gel container to prevent increased humidity. Next, the CaO catalyst was activated by immersing it in a KOH solution at a ratio of 1:8 and allowing it to sit for 24 hours at room temperature. The solution was then evaporated using a hotplate until the

mixture was dry. The mixture was then heated at 110°C for 4 hours and calcined at 500°C for 5 hours.

CAO CATALYST IMPREGNATION WITH TiO₂ PHOTOCATALYST

The activated CaO catalyst is then modified by combining the TiO₂ photocatalyst through the impregnation method. In this process, CaO was evenly mixed with TiO₂ at a ratio of 9:1, and then an aqueous mixture was added at a ratio of 1:2. This mixture was then dried in an oven at 110°C for 2 hours to remove the water content. The CaO-TiO₂ mixture that underwent the impregnation process was then activated by calcination at 400°C in a furnace for 5 hours.

CAO-TiO₂ CATALYST CHARACTERIZATION

The crystal phase and crystallinity of the catalyst were determined via X-ray diffraction (XRD) analysis with Cu K α radiation ($\lambda = 0.15406 \text{ \AA}$) at 40 kV and 30 mA, $2\theta=20-60^\circ$, and a scan speed of 4,000 degrees/min. After the peak value data were obtained, the degree of photocatalyst crystallinity was calculated using the Debye-Scherrer equation. The morphology and analysis of the material elements were carried out using the SEM-EDS analysis.

COOKING OIL PRETREATMENT

Used cooking oil obtained from traders generally still contains residue from the frying process, so it must be separated before being used as a raw material for biodiesel. The separation process was carried out by filtration using filter paper to reduce the residue in the oil. After that, the cooking oil was further processed through purification by adsorption. Two hundred milliliters of used cooking oil was heated to 105°C, and then activated carbon was added while stirring with a magnetic stirrer for 60 minutes. The mixture was then filtered to separate the dissolved substances.

The oil that was pretreated to remove the frying residue and brownish color was then tested to determine the acid content. The determination of free fatty acid (FFA) levels in used cooking oil, both before and after purification, was carried out via the acid-base titration method according to the SNI 04-7182-2006 procedure of the Director General of Oil and Gas (AOCS CD 3d-63), which uses titration with 0.1 M NaOH. The formula for calculating the FFA level was as follows:

$$\%FFA = \frac{V_{NaOH} \times M_{NaOH} \times MW}{m_{sample} \times 1000} \times 100\%$$

where V_{NaOH} and M_{NaOH} are the volume (mL) and molarity (M) of NaOH used, respectively, MW is the molecular weight of the cooking oil used (g/mol), and

sample m is the mass of the cooking oil used (g).

BIODIESEL SYNTHESIS

The biodiesel was synthesized through the transesterification-esterification method in a photoreactor under UV light. This process involves heating at 65°C for 4 hours at a speed of 500 rpm, where a reaction occurs between methanol and used cooking oil at ratios of 1:6, 1:9, and 1:12. The CaO-TiO₂ photocatalyst was added to the cooked oil at proportions of 3, 5, and 7%.

The reusability test was carried out on a methanol:cooking oil ratio of 1:9 and a photocatalyst mass of 5% (the best yield from the previous variation). Reusability 1 used a fresh photocatalyst that had been used and recovered. Furthermore, the photocatalyst from reusability 1 was reused in reusability test 2 after the recovery process.

BIODIESEL PURIFICATION AND CATALYST RECOVERY

After the reaction, separation was performed via a Buchner funnel, a vacuum pump, and filter paper to separate the catalyst. After the catalyst was separated, the biodiesel that was still mixed with glycerol was separated using a separation funnel. The separated catalysts can be washed with aquadest and reused in

transesterification. The biodiesel was then washed with warm water, stirred, and left for 15 minutes for separation. The washing process was carried out twice. After that, the biodiesel was dried by heating at a temperature of 120°C to remove the remaining water, and then stored in a container filled with silica gel and dried for 15 minutes.

BIODIESEL CHARACTERIZATION

Density Test

The density test was carried out using a pycnometer at 40°C according to the ASTM D-1298 test standard. The pycnometer was rinsed with aqueducts and dried before use. An empty pycnometer is weighed first (m_1), filled with a sample and weighed again (m_2). The density value is calculated using a predetermined formula:

$$\rho = \frac{m_2 - m_1}{v}$$

where ρ is the density (g/mL), m_1 is the pycnometer mass (g), m_2 is the pycnometer mass + sample (g), and v is the sample volume (g).

Viscosity Test

Viscosity tests are carried out to determine the fluid characteristics that are resistant to flow. Viscosity testing was carried out using an Ostwald viscometer at 40°C using the ASTM D-1298 test standard. The

viscosity value was calculated using the following formula:

$$\mu = K \cdot t$$

where μ is the kinematic viscosity (mm^2/s), C is the viscometer constant (mm^2/s^2), and t is the time (s).

Methyl ester composition test

The methyl ester composition was determined using GC–MS analysis, which involves identifying the number and structure of compounds in the sample.

Biodiesel yield

The yield percentage was calculated using the following formula:

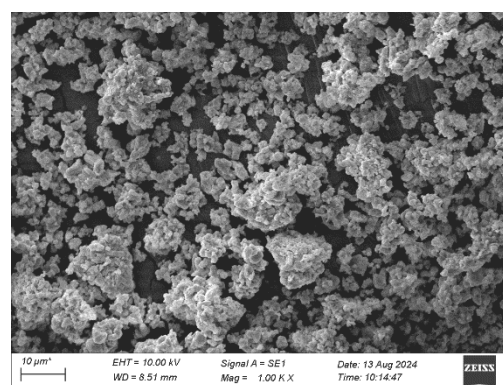
$$\%Yield = \frac{\text{biodiesel mass (gram)}}{\text{Oil mass (gram)}} \times 100\%$$

RESULTS AND DISCUSSION

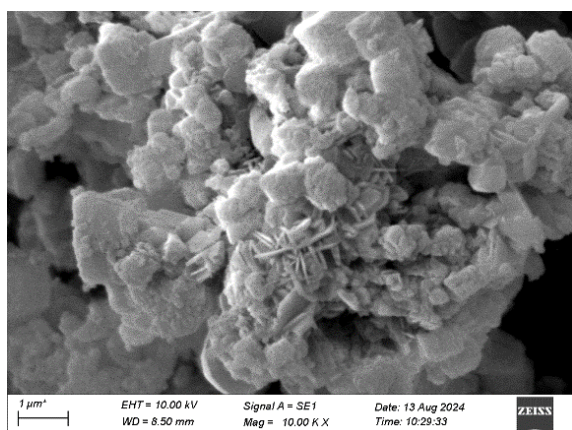
CHARACTERIZATION OF CAO-TiO₂ COMPOSITE PHOTOCATALYSTS

The SEM characterization results in Figure 1 show that the photocatalyst composite was irregularly shaped and that agglomerates formed. This irregular shape was caused by CaO being made with a top-down system, so obtaining uniform and regular results was difficult. The existing spherical form is TiO₂, similar to the initial form of TiO₂ P25. The results of the EDS analysis revealed the presence of Ca, Ti, O, and K, as expected.

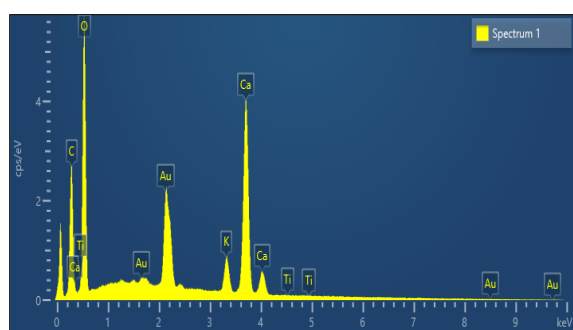
Figure 2 shows the XRD pattern of the CaO-TiO₂ photocatalyst. The sample has high crystallinity, as indicated by a narrow, sharp peak (Adiwibowo, Ibadurrohman and Slamet, 2018). The detected peaks were attributed to the diffraction of CaO, TiO₂ anatase and rutile phases and K₂O according to JCPDS standards Files No. 37-1497, 21-1272, 21-1276, and 77-2151, as shown in Table 1. The CaO activation process using KOH causes the appearance of K₂O diffraction peaks, which contribute to increasing the baseness of the catalyst and improving its performance as a whole. The presence of these four compounds indicates that CaO-TiO₂ synthesis was successful. The high crystallinity was directly proportional to the efficiency of the photocatalyst process. The average crystal size of the synthesized photocatalyst was 25.18 nm.



(a)



(b)



(c)

Figure 1. Characterization of CaO-TiO₂ composites for SEM analysis a) Magnified 1,000 times, b) 10,000 times, and c) EDS analysis

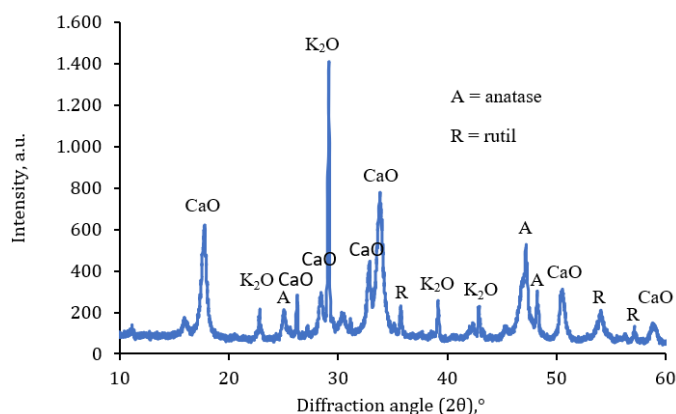


Figure 2. CaO-TiO₂ Photocatalyst XRD Pattern

Compound	Data	CaO JCPDS File No. 37- 1497	TiO ₂ anatase JCPDS File No. 21- 1272	TiO ₂ rutile JCPD S File No. 21- 1276	K ₂ O JCPDS File No. 77- 2151
CaO	17.78	17.4			
K ₂ O	22.82				23.04
Anatase	24.99		24.8		
CaO	26.25	26.3			25.88
CaO	28.42	28.13			
K ₂ O	29.15				29.3
CaO	32.89	32.2			
CaO	33.83	33.5			
Rutile	35.72			35.6	
K ₂ O	39.14				39.5
K ₂ O	42.92				41.9
Anatase	47.21		47.6		
Anatase	48.23		48.05		
CaO	50.53	50.5			
Rutile	54.06			54.00	
Rutile	57.13			56.1	
	58.79	58.3			

COMPARISON OF BIODIESEL YIELD USING CAO AND CAO/TIO2

Our preliminary study revealed that a methyl ester yield using CaO alone was 66.7%, whereas the combination of CaO-TiO₂ under the same conditions was 90.6%. This increase in catalyst activity is due to the reduction of the CaO catalyst by TiO₂ electrons that move to the catalyst surface (Rodríguez and Perillo, 2015). This increases the electron density at the CaO basic site, accelerating the biodiesel formation reaction.

EFFECT OF THE FEED RATIO ON BIODIESEL YIELD

The biodiesel synthesis process is a reversible reaction that requires the addition of a certain amount of methanol

to achieve optimal results. To optimize the results, various methanol ratios were used (1:6, 1:9, and 1:12). Figure 3 shows that the yield increases at a ratio of 1:9 but decreases when the ratio exceeds 1:9. Too much methanol can make efficient contact between the catalyst and the oil difficult. According to Sisca et al. (2021), excessive alcohol can complicate the heating and elimination process, so the proper molar ratio must be maintained to obtain a high conversion yield. In addition, the reaction can be reversed if the oil:methanol ratio is too high, thus lowering the biodiesel yield (Guo et al., 2021). At a molar ratio of 1:9, the yield obtained was 88.75%, with a catalyst weight of 3%. To improve the biodiesel yield, maintaining the oil:methanol molar ratio was essential. A study by Kalos et al., (2024) also reported that the best molar ratio was 1:9.

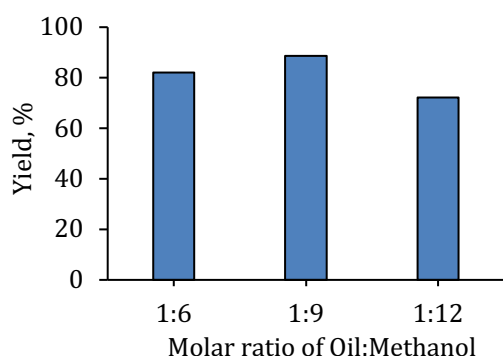


Figure 3. Effect of Molar Ratio on Biodiesel Yield.

EFFECT OF CATALYST WEIGHT ON BIODIESEL YIELD

Figure 4 shows how the use of catalysts affects the yield of biodiesel produced. Catalysts with 5% catalyst mass/oil mass increased biodiesel production to a maximum of 90.4%. An increase in the number of catalysts increases the number of active sites of the reaction. However, too many catalysts can increase the viscosity of the reactant mixture, inhibit the progress of the reaction, and make it challenging to handle the product (Ni et al., 2019). In addition, excessive use of catalysts causes reactants and products to be absorbed by the catalyst, resulting in aggregations that reduce the total number of active sites where the reaction occurs (Wang et al., 2014).

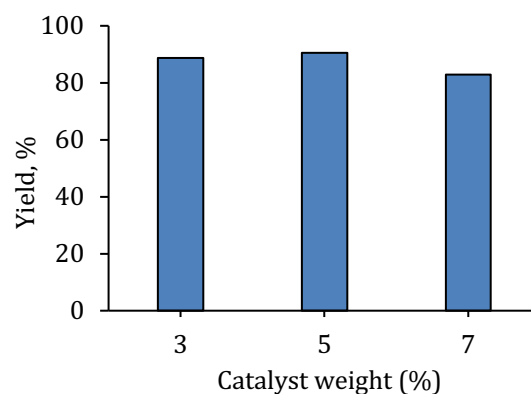
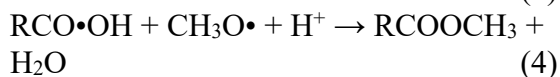
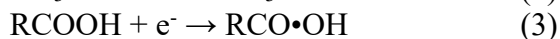
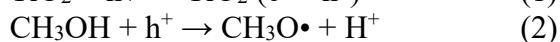
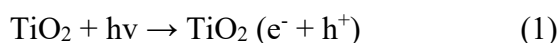


Figure 4. Effect of Catalyst Weight on Biodiesel Yield.

EFFECT OF UV LIGHT ON BIODIESEL YIELD

Figure 5 shows that UV-assisted biodiesel synthesis results in higher yields than does the process without UV light, with a maximum yield of 90.6% when UV light is used and 78.67% without UV light. This suggests that UV light was essential for increasing the activation of TiO_2 . In addition to being able to function as a catalyst, when TiO_2 is irradiated by light with energy equal to or greater than the energy of the band gap, it will produce excited electrons and positive holes (eq. 4). These electron-hole pairs react with methanol and oil to produce radical compounds and hydrogen ions (eqs. 5 & 6). These three compounds from this reaction then interact to form biodiesel and water (eq. 7) and increase the biodiesel yield. The reactions that may occur are as follows:



A study by Mohamad et al., (2017) also reported that activating the CaO-TiO_2 catalyst with UV light produces higher yields.

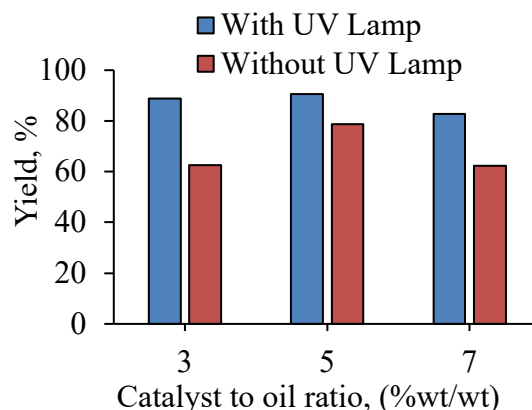


Figure 5. Effect of UV Light on Biodiesel Yield.

EFFECT OF CATALYST REUSE ON BIODIESEL YIELD

Figure 6 shows that the heterogeneous catalyst CaO-TiO_2 can be reused in the synthesis process. The reuse of this catalyst results in lower yields than those of new catalysts. This can be due to leaching from K or obstruction of the active site on the catalyst surface by the product or reactant (Khatibi et al., 2021). In addition, damage to the active surface of the catalyst due to the change in Ca ions and contamination of moisture and CO_2 from the air during the filtration process also decreases catalytic activity (Sudsakorn et al., 2017).

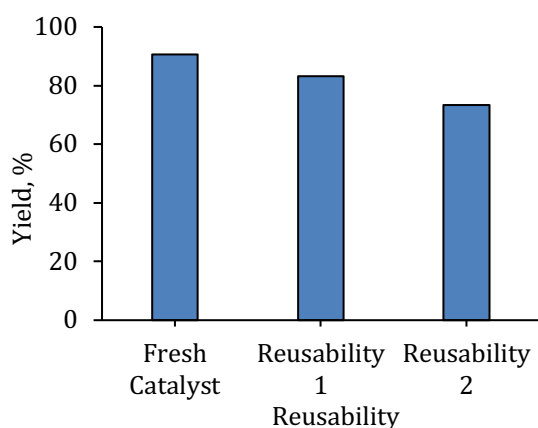


Figure 6. Effect of Catalyst Reusability on Biodiesel Yield

BIODIESEL CHARACTERIZATION

Density

Density is an essential characteristic of biodiesel. Vehicle engines are designed to work on specific air–fuel ratios so that improper density will lead to inefficient combustion and lower engine efficiency. High-density biodiesel can undergo incomplete combustion, whereas low-density biodiesel is highly volatile. The density is directly proportional to the volumetric fuel heating value, which means that the greater the density is, the greater the fuel energy content carried into the engine (Sheet, 2018).

Table 2 shows that the density characteristics of the biodiesel produced under UV light followed the Indonesia National Standard (SNI 7182:2015), i.e., 850–890 kg/m³, indicating oil conversion after biodiesel synthesis. The best results were obtained at a molar ratio of 1:9 with a

catalyst weight of 5%, which reached a density of 882 kg/m³. The use of UV light improves the performance of the photocatalyst to produce a density that corresponds to the SNI range, as in a previous study by Adiwibowo et al. (2023), which showed that the density of biodiesel synthesized with UV light corresponds to Indonesian national standards.

Table 2. Biodiesel densities at various variations

Catalyst	Mol ratio	Catalyst weight, %	UV light	Density, kg/m ³
New	1:6	3	With	889,9
New	1:9	3	With	886,0
New	1:12	3	With	882,5
New	1:9	3	With	886,0
New	1:9	5	With	882,0
New	1:9	7	With	888,1
New	1:9	3	Without	893,0
New	1:9	5	Without	899,0
New	1:9	7	Without	896,0
New	1:9	5	With	882,0
Reusability 1	1:9	5	With	892,4
Reusability 2	1:9	5	With	899,0

Viscosity

Viscosity is one of the critical parameters in determining the quality of biodiesel. A viscosity that is too high can decrease the efficiency of fuel spraying, causing excessive smoke in the exhaust (Hamid et al., 2023).

Table 3. Viscosity of biodiesel at various variations

Catalyst	Mol ratio	Catalyst weight, %	UV light	Viscosity mm ² /s
New	1:6	3	With	2,71
New	1:9	3	With	2,58
New	1:12	3	With	2,49
New	1:9	3	With	2,58
New	1:9	5	With	2,45
New	1:9	7	With	2,26
New	1:9	3	Without	2,58
New	1:9	5	Without	3,39
New	1:9	7	Without	3,52
New	1:9	5	With	2,45
Reusability 1	1:9	5	With	2,45
Reusability 2	1:9	5	With	3,09

In Table 3, the viscosity value of the biodiesel produced partially meets the SNI 7182:2015 specification, which is in the range of 2.3–6.0 mm²/s. However, according to the Indonesian national standard (SNI), one sample does not meet the minimum viscosity standards. This can occur because the biodiesel produced still contains impurities, such as byproducts in the form of glycerol or reactants that do not fully react and separate.

GC–MS analysis

Table 4 shows that the synthesized biodiesel has various types of methyl esters, with the main components of biodiesel being methyl oleate (50.49%), methyl palmitate (33.81%), and methyl stearate (8.78%). This result was in line

with previous research (2021), where cooking oil from palm oil was dominated by oleic acid and palmitic acid.

Table 4. GC–MS Biodiesel analysis

No	Area, %	Components
1	0,57	Dodecanoic acid, methyl ester
2	2,65	Tetradecanoic acid, methyl ester
3	0,12	Pentadecanoic acid, methyl ester
4	0,79	9-Hexadecenoic acid, methyl ester
5	33,81	Methyl palmitate
6	0,09	cis-10-Heptadecenoic acid, methyl ester
7	0,28	Heptadecanoic acid, methyl ester
8	50,49	Methyl oleate
9	8,78	Methyl stearate
10	0,64	cis-11-Eicosenoic acid, methyl ester
11	1,25	Eicosanoic acid, methyl ester
12	0,22	Docosanoic acid, methyl ester
13	0,22	Tetracosanoic acid, methyl ester
31 - 55%		Low
0 - 30%		Very Low

CONCLUSION

The larger the ratio of oil:methanol and catalyst weight is, the greater the yield produced until a certain point where the yield decreases with increasing ratio of oil:methanol and catalyst weight. The use of UV light increases the yield of biodiesel produced. CaO-TiO₂ catalysts can be reused by decreasing the yield with each reuse. The best product yield was 90.6%, the density was 882 kg/m³, and the viscosity was 2.45 cSt at a reactant ratio of 1:9 catalyst weight of 5% with the use of UV light for 4 hours at a temperature of 65°C. The results of the XRD test revealed that there was a CaO-TiO₂ compound.

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