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Dual-Waste Valorization: Sustainable Biodiesel Production from High-FFA Sludge Palm Oil Using Silica-Rich Water Treatment Sludge as a Low-Cost Catalyst Support

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ABSTRACT - To address feedstock costs and environmental sustainability, this study employes a dual-waste valorization strategy by converting high-free fatty acid (FFA) sludge palm oil (SPO) into biodiesel using silica-rich water treatment sludge from PDAM as a low-cost support for a heterogeneous K_2O/SiO_2 catalyst. Due to the high initial acidity of SPO, a two-step process is used: acid esterification to reduce FFA levels below 2%, followed by alkaline transesterification. Optimization of the reaction parameters identified the ideal conditions as 6% catalyst loading, a 15:1 methanol-to-oil molar ratio, and a 2-hour reaction time at 63°C. Under these conditions, the process achieves a biodiesel yield of 70.43% with a high methyl ester purity of 98.01%. The produced biodiesel meets the SNI 7182:2015 quality standards for density and acid number. Although the catalyst demonstrated stability over two consecutive cycles, its activity declined in the third cycle due to active-site leaching. This work confirms the viability of integrating industrial and municipal waste streams for sustainable and cost-effective bioenergy production.

Keywords: sludge palm oil, dual-waste valorization, water treatment sludge, heterogeneous catalyst, biodiesel

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INTRODUCTION

Indonesia has set a target to increase the use of renewable energy and reduce the use of fossil fuels by 2025, in accordance with the National Master Plan, as stipulated in Presidential Regulation No. 22/2017, and regulations issued by the Ministry of Energy and Mineral Resources. The target of achieving 23% renewable energy use by 2025 is equivalent to an annual energy consumption of 62.2 MTOE, of which 13.8 million kiloliters is expected to come from biofuels (Husada et al., 2023). Biofuels are fuels produced from biomass, such as bioethanol and biodiesel (Bera et al., 2020). Biodiesel is commonly used as a transportation fuel in Indonesia and is blended with diesel in certain proportions (Bethari et al., 2025). The use of biodiesel as an alternative fuel offers several advantages, including its production from local and renewable resources. The gradual incorporation of biodiesel into national fuel consumption can reduce dependence on imported fuel oil (Choirun Az Zahra et al., 2021). Biodiesel can be produced through the esterification and transesterification of vegetable oils or animal fats with alcohol under acidic or basic conditions, with the aid of a catalyst (Baptiste Nduwayezu et al., 2015). Feedstock accounts for 75–80% of biodiesel production costs; therefore, selecting economical feedstocks for biodiesel production is essential (Elgharbawy et al., 2021).

In Indonesia, large-scale biodiesel production commonly uses refined, bleached, and deodorized palm oil (RBDPO), a feedstock derived from the refining of crude palm oil (CPO) (Siregar et al., 2024). However, the use of vegetable oil as a biodiesel feedstock has certain drawbacks, such as high cost and potential disruption to the supply chain of vegetable oil as a food ingredient (Hidayat et al., 2018). As an alternative, palm oil sludge can be used as a biodiesel feedstock. Palm oil sludge is a byproduct resulting from oil loss during the CPO production process and is collected in palm oil mill effluent (POME) treatment ponds. The oil layer on the upper surface of POME can be separated and recovered as sludge palm oil. The amount of sludge palm oil obtained is approximately 1.5% of CPO production, with a free fatty acid (FFA) content of >20% and a moisture content of <2.5% (Chew et al., 2021; Muanruksa et al., 2019).

Hayyan et al. (2010) investigated biodiesel production from sludge palm oil using an esterification–transesterification method with a homogeneous catalyst. The esterification process was conducted using 0.75% p-toluene-4-sulfonic acid monohydrate (PTSA) as the catalyst, at a temperature of 60°C, a methanol-to-oil molar ratio of 10:1, and a reaction time of 60 minutes. This was followed by transesterification using 1% KOH as the catalyst at 60°C, with a methanol-to-oil molar ratio of 10:1 and a reaction time of 30 minutes, resulting in a yield of 76.62%. Abdullah et al. (2017) conducted a study using alum as the catalyst in the esterification process, followed by alkali-catalyzed transesterification. Esterification was performed using 6% alum as the catalyst at 60° C, a methanol-to-oil molar ratio of 20:1, and a reaction time of 3 hours. This was followed by transesterification using 1.5% KOH as the catalyst at a reaction temperature of 60°C for 1 hour, producing a yield of 93%. Loh et al. conducted research using an enzymatic process at 45°C, with a methanol-to-oil ratio of 5:1, using liquid lipase (Eversa Transform 2.0) as the enzyme catalyst, and a reaction time of 24 hours, yielding a biodiesel yield of 94% (Loh et al., 2021).

The use of heterogeneous catalysts in SPO conversion remains limited despite their numerous advantages. Heterogeneous catalysts offer several benefits, including high mechanical strength, easy separation, and reusability. Most heterogeneous or solid catalysts consist of an active component and a support material (Kibar et al., 2023). The active site accelerates the reaction, while the support provides a large surface area for dispersing the active sites, thereby increasing the contact surface area (Mabate et al., 2023). Support materials typically consist of refractory oxides such as SiO_2, Al_2O_3, or TiO_2 (Mabate et al., 2023). Support materials typically consist of refractory oxides such as SiO_2, Al_2O_3, or TiO_2 (Munnik et al., 2015). Regional water company (PDAM) sludge is particulate matter formed during chemical coagulation and flocculation processes in drinking water treatment plants (Barakwan et al., 2019). In

drinking water treatment plants, this sludge is commonly disposed of in landfills or reused as a soil stabilizer, fertilizer, or clay substitute (Listiowati et al., 2021). The average daily sludge discharge from PDAM in Medan is 145.7 m^3/day, equivalent to 0.28% of the total daily clean water treatment discharge (Lukman et al., 2022; Rumapea & Harahap 2021). The chemical composition of dry PDAM sludge includes SiO_2 at 45–70%, Al_2O_3 at 15–35%, Fe_2O_3 at 2–5%, CaO at 0.5–1%, Na_2O at 0.1–1%, K_2O at 0.01–0.5%, and MgO at 0–0.7% (Ahmad et al., 2016; Barakwan et al., 2019; Hajar et al., 2014). Due to the high silica and alumina content of sludge from drinking water treatment plants, Pessoa Junior et al. (2020) investigated the use of water treatment sludge as a support for a heterogeneous acid catalyst by impregnating the treated sludge with H_2SO_4 to accelerate biodiesel synthesis from oleic acid feedstock. Using a methanol-to-oleic acid ratio of 20:1, a reaction time of 3 hours, and a reaction temperature of 100°C, they achieved an ester conversion of 97%. This study focuses on biodiesel production from sludge palm oil using H_2SO_4 as the catalyst in the initial esterification stage and a heterogeneous K_2O/SiO_2 catalyst derived from PDAM sludge in the subsequent transesterification stage. This approach is expected to reduce production costs and improve the efficiency of biodiesel production from sludge palm oil.

METHODOLOGY

Materials

Sludge palm oil (SPO) was obtained from the wastewater pond of the Pulau Tiga Palm Oil Mill, Aceh Tamiang Regency, Indonesia. PDAM sludge was obtained from the Medan Regional Water Company (PDAM). The chemicals used in this study were methanol, KOH, NaOH, and H_2SO_4, all of technical grade.

Raw material pre-treatment

A 1000 g SPO sample was preheated because it was semi-solid at room temperature (30 ± 2°C). Heating was performed at 60°C on a hot plate. The SPO was then filtered using filter paper to remove contaminants present in the sample.

Preparation of K_2O/SiO_2 catalyst from PDAM sludge

The silica-rich PDAM sludge was first dried in an oven at 105°C for 24 hours to remove free moisture. The dried sludge was then crushed and sieved to obtain a uniform particle size of 100 mesh. To extract silica and remove organic impurities, the sludge was subjected to acid washing with 1 M H_2SO_4, followed by washing with distilled water until a neutral pH was reached, and then dried. For the synthesis of the K_2O/SiO_2 catalyst, the treated silica support was impregnated with an aqueous potassium hydroxide (KOH) solution using the wet impregnation method. A specific amount of KOH was dissolved in distilled water to achieve a potassium loading of 3 wt%. The silica support was added to this solution and stirred continuously at 60°C for 24 hours until a slurry was formed. The resulting mixture was dried overnight in an oven at 80°C. Finally, the dried solid was calcined in a muffle furnace at 500°C for 4 hours to convert the potassium precursor into active K_2O species supported on the silica matrix.

SPO esterification

A A 500 g SPO sample was heated in a 1 L three-neck flask equipped with a reflux condenser. The operating conditions were maintained at 63°C, with a methanol-to-SPO molar ratio of 12:1, 1.5% H_2SO_4 as the catalyst, a reaction time of 2.5 hours, and a stirring speed of 400 rpm. The esterification products were separated into an upper layer consisting of ester and a lower layer consisting of water and other impurities. The ester was then separated from the water content by centrifugation and heated on a hot plate with stirring at 200 rpm, as water content can affect the yield. The esterification reaction involves the conversion of fatty acids into fatty acid alkyl esters (Fong et al., 2022), which can be represented by the following chemical Equation 1.

$$R - COOH + R' - OH \overset{\text{Catalyst}}{\rightleftharpoons} R - COOR' + H_2O \quad (1)$$

Transesterification

After the esterification process was completed, 30 g of the sample was placed in a 250 mL three-neck flask equipped with a reflux condenser and

heated to the reaction temperature of 63°C. In this stage, the transesterification reaction involves the conversion of triacylglycerol with an alcohol (methanol) in the presence of a catalyst to produce fatty acid alkyl esters (biodiesel) and glycerol as a byproduct (Fong et al., 2022). This chemical reaction can be expressed by the following Equation 2.

Triacylglycerol

$$+3R'\text{-}OH \overset{\text{Catalyst}}{\rightleftharpoons} \text{Fatty acid alkyl esters} + \text{Glycerol} \quad (2)$$

The methanol-to-SPO molar ratio was varied at 9:1, 12:1, 15:1, and 18:1; the concentration of the heterogeneous K_2O/SiO_2 catalyst derived from PDAM sludge was varied at 2%, 4%, 6%, and 8%; and the transesterification reaction time was varied at 1, 2, and 3 hours. The entire reaction was carried out at 63°C with stirring at 400 rpm. After the reaction was completed, the transesterification product was transferred to a separating funnel and allowed to stand for 12 hours, while the catalyst was separated using filter paper. After 12 hours, the upper layer, consisting of methyl ester, was separated from the lower layer, consisting of crude glycerol. The methyl ester was washed with 50 mL of hot water at 50°C until the mixture became clear. The methyl ester was then heated on a hot plate with stirring using a magnetic stirrer to reduce the remaining water content.

Analysis and characterization of biodiesel products

The physical and chemical properties of the biodiesel produced under the best conditions were analyzed, including density, viscosity, cloud point, acid value, flash point, and water content. Qualitative analysis of the biodiesel was performed using Fourier transform infrared spectroscopy (FTIR) to identify functional groups, while gas chromatography (GC) was used to determine the ester and glycerol content of the biodiesel derived from sludge palm oil. Catalyst reusability was evaluated by repeating the transesterification process using the recovered catalyst until the biodiesel yield decreased to below 40%.

RESULT AND DISCUSSION

Characteristics of sludge palm oil

The sludge palm oil used in this study was collected from the top layer of the liquid waste pond, and its characteristics are presented in Table 1. Based on the data above, sludge palm oil consists of 42.58% saturated fatty acids and 57.42% unsaturated fatty acids. High FFA levels and acid numbers can lead to soap formation during transesterification. Therefore, SPO esterification using a strong acid catalyst is required as a pretreatment for raw materials with high acid numbers, resulting in an efficient reduction in acidity. Specifically, the chemical properties of biodiesel are significantly influenced by the concentration of fatty acids it contains, whereas its physical properties depend on the fatty acid chain length and the number of double bonds (Folayan et al., 2019). Long-chain saturated fatty acids help increase the cetane number and oxidation stability, whereas unsaturated and short-chain fatty acids can improve viscosity and low-temperature flow performance, although these characteristics may not always be desirable. Biodiesel with a high content of saturated fatty acids tends to have a higher freezing point than biodiesel with a higher content of unsaturated fatty acids (Kumbhar et al., 2022). In this case, SPO, with relatively balanced unsaturated and saturated fatty acid contents, can be used as a raw material for biodiesel production, provided that it is protected from contamination and excess water, which may cause failure in the esterification–transesterification process.

Table 1. Characteristics of sludge palm oil based on GC analysis

Acid number	97.37
FFA (%)	48.02
Water content (%)	0.1
Fatty acid component	**Composition (%)**
Lauric acid ($C_{12:0}$)	0.08
Myristic acid ($C_{14:0}$)	0.96
Palmitic acid ($C_{16:0}$)	36.32
Palmitoleic acid ($C_{16:1}$)	0.15
Stearic acid ($C_{18:0}$)	4.86
Oleic acid ($C_{18:1}$)	42.63
Linoleic acid ($C_{18:2}$)	14.36
Linolenic acid ($C_{18:3}$)	0.15
Arachidic acid ($C_{20:0}$)	0.36
Eicosanoic acid ($C_{20:1}$)	0.13

The effect of catalyst amount on the SPO transesterification process

The differences in biodiesel yield across catalyst variations were clearly observed at different reaction times and catalyst loadings. The catalyst used in this reaction was a heterogeneous K_2O/SiO_2 catalyst derived from PDAM sludge. Base catalysts generally have higher catalytic activity than acid catalysts and are non-corrosive. In addition, heterogeneous base catalysts can be easily separated from the reaction mixture, thereby minimizing wastewater generation (Gardy et al., 2019; Maroa & Inambao 2021).

Figure 1 shows the relationship among reaction time, catalyst amount, and biodiesel yield. The graph presents biodiesel yields at catalyst concentrations of 2%, 4%, 6%, and 8%, with reaction times ranging from 60 to 180 minutes. The fixed variables in this experiment were a methanol-to-oil molar ratio of 15:1, a reaction temperature of 63°C, and a stirring speed of 400 rpm. At a catalyst concentration of 2%, increasing the reaction time from 60 to 180 minutes increased the biodiesel yield from 9.53% to 13.73%. The low yield was attributed to the small amount of solid catalyst used, which resulted in limited contact between the reactants and the catalyst. In general, reactions involving heterogeneous catalysts proceed slowly during the initial stages and require longer reaction times to achieve favorable product yields because the reaction mixture forms a three-phase system (Miyuranga et al., 2023; Yaghi et al., 2025; Yusuf et al., 2024). Therefore, using a catalyst concentration of 2% required 180 minutes to achieve the highest biodiesel yield under these conditions. At a catalyst loading of 6%, the biodiesel yield also increased up to a reaction time of 120 minutes. The biodiesel yield increased from 30.58% at 60 minutes to 71.50% at 120 minutes. However, at 180 minutes, the biodiesel yield decreased. This decrease may be attributed to the reversible nature of the transesterification reaction; prolonged reaction time after equilibrium has been reached may promote the reverse reaction and side reactions, including soap formation. Therefore, at a catalyst loading of 6%, a reaction time of 120 minutes was required to achieve a high biodiesel yield.

At catalyst concentrations of 4%, 6%, and 8%, biodiesel yields showed similar trends over reaction times of 60 to 180 minutes. A decreasing trend in biodiesel yield was observed from 71.50% to 65.42% at a catalyst concentration of 6% and from 61.52% to 54.57% at a catalyst concentration of 8% as the reaction time increased. This phenomenon may be caused by the excessive addition of heterogeneous catalyst, which increases the viscosity of the reaction mixture and mass-

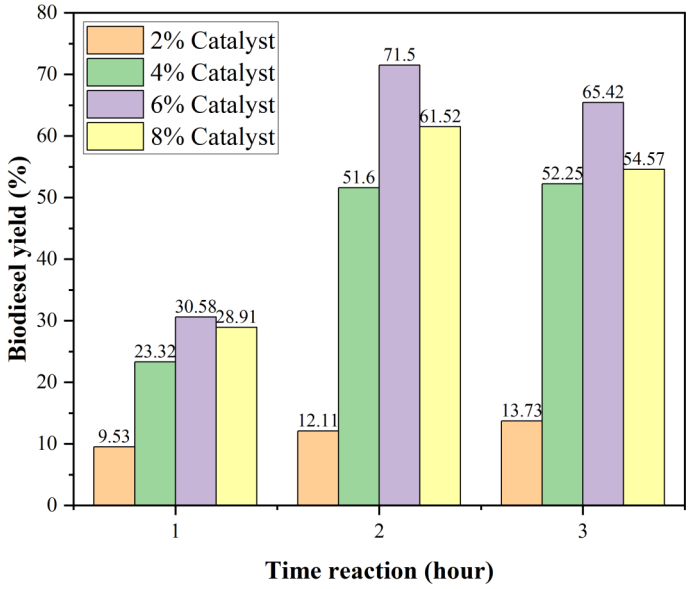

Figure 1. The effect of reaction time and catalyst amount on SPO biodiesel yield (Reaction conditions: methanol-to-SPO molar ratio of 15:1, temperature 63°C, and stirring speed 400 rpm). The maximum yield of 71.50% was achieved with a 6% catalyst loading at 2 hours.

transfer resistance between the reactants and the catalyst, thereby reducing biodiesel production (Yusuf et al., 2024). This is also supported by the lower biodiesel yield obtained at an 8% catalyst concentration compared with that obtained at 6% under the same reaction time.

Catalyst loadings of 6% and 8% required 120 minutes to produce high biodiesel yields. Therefore, an appropriate reaction time and catalyst amount are required, considering both the effect of reaction time on the transesterification reaction and the effect of catalyst amount on mass-transfer resistance between the reactants and the catalyst, to achieve a high biodiesel yield (Yusuf et al., 2024). Based on the relationship between reaction time and catalyst amount, the highest biodiesel yield was 71.50%, obtained using 6% catalyst and a reaction time of 120 minutes.

The effect of different methanol molar ratios on the transesterification of SPO

As shown in Figure 2, the biodiesel yield increased with increasing reaction time until it reached a maximum point at 2 hours, after which it decreased. The graph shows that the biodiesel yield increased from a reaction time of 1 hour to 2 hours for each methanol-to-oil molar ratio and decreased at a reaction time of 3 hours for all reactant molar ratios of 9:1, 12:1, 15:1, and 18:1. This occurred because a short reaction time caused incomplete conversion of triglycerides into methyl esters, resulting in a low yield (Hazrat et al., 2023). At the beginning of the transesterification reaction, SPO and methanol, in the presence of a heterogeneous catalyst, are dispersed slowly to form an oil–alcohol mixture (Trejo-Zárraga et al., 2018). Subsequently, the transesterification reaction proceeded rapidly until the optimum biodiesel yield was achieved. Furthermore, the transesterification reaction is reversible; therefore, once the optimum yield conditions are reached, increasing the reaction time does not further improve the yield if equilibrium has been attained and may even promote the reverse reaction, thereby decreasing the methyl ester yield (Farouk et al., 2024). This explains the decrease in yield across the variations used.

The effect of varying the reactant molar ratio on biodiesel yield is also evident in Figure 2. The graph shows that, at a fixed reaction time, increasing the methanol-to-oil molar ratio increased the biodiesel yield up to a molar ratio of 15:1, after which the yield decreased at a molar ratio of 18:1. The addition of excess methanol aims to shift the reaction equilibrium toward product formation, thereby increasing the methyl ester yield. However, an excessively high methanol-to-oil molar ratio may dissolve glycerol as a byproduct along with excess methanol, thereby

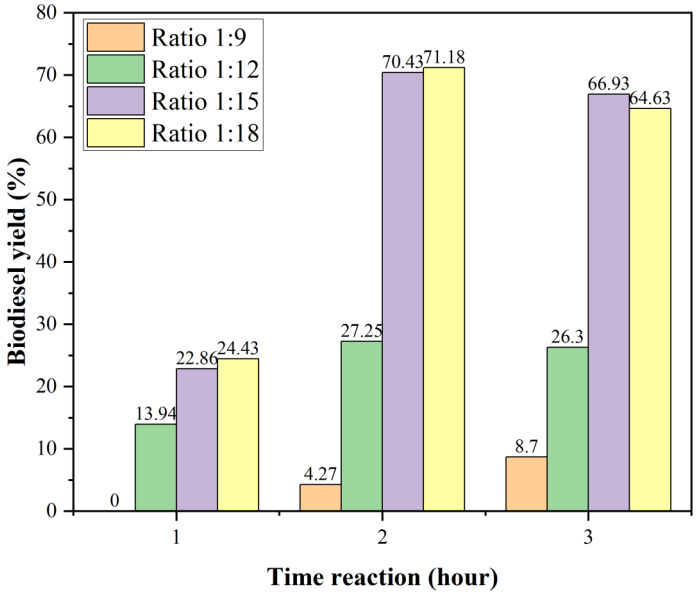

Figure 2. The relationship between reaction time, reactant molar ratio, and biodiesel yield at a reaction temperature of 63°C, catalyst concentration of 6%, and stirring speed of 400 rpm

inhibiting the interaction among methanol, SPO ester, and the catalyst. Moreover, the polar hydroxyl groups in methanol may interact with emulsifying compounds, making it more difficult to separate biodiesel from the reaction mixture and consequently reducing the methyl ester yield (Enguilo Gonzaga et al., 2021).

The best conditions obtained in this study were a reaction temperature of 63°C, catalyst loading of 6%, a methanol-to-SPO molar ratio of 15:1, and a reaction time of 2 hours, which produced a biodiesel yield of 70.43%. This result was lower than that reported by Hayyan et al. (2010), who obtained a yield of 76.62% using a homogeneous alkali catalyst in a two-step reaction process.

Effect of catalyst concentration on methyl sster content

The results of biodiesel production from SPO using a heterogeneous K_2O/SiO_2 catalyst derived from PDAM sludge, with catalyst concentration as the main variable, are presented in Figure 3. Increasing the catalyst concentration enhanced methyl ester formation up to an optimum concentration of 6%, beyond which the methyl ester content slightly decreased. At a low catalyst loading of 2%, the limited number of active sites was insufficient to fully convert triglycerides into methyl esters within the reaction time, resulting in low yield and methyl ester content. This behavior is consistent with previous reports stating that catalyst loading must be optimized to balance reaction rate and side reactions (Alagha et al., 2024; Amizan et al., 2024; Aseibichin et al., 2024).

The decrease in biodiesel methyl ester content observed when using an 8% catalyst loading was attributed to limited mass transfer and the high viscosity of the reaction mixture. These conditions weakened the mixing of reactant components, resulting in decreased methyl ester conversion. This mass-transfer process occurs during the methoxide/alkali formation stage of the transesterification reaction. When interacting with a heterogeneous catalyst, methoxide forms on the catalyst surface. This process depends on the effectiveness of mass transfer from methanol to the heterogeneous catalyst, which may reduce the reaction yield. At high catalyst concentrations, conversion can decrease because the catalyst active sites may become blocked (Okechukwu et al., 2022).

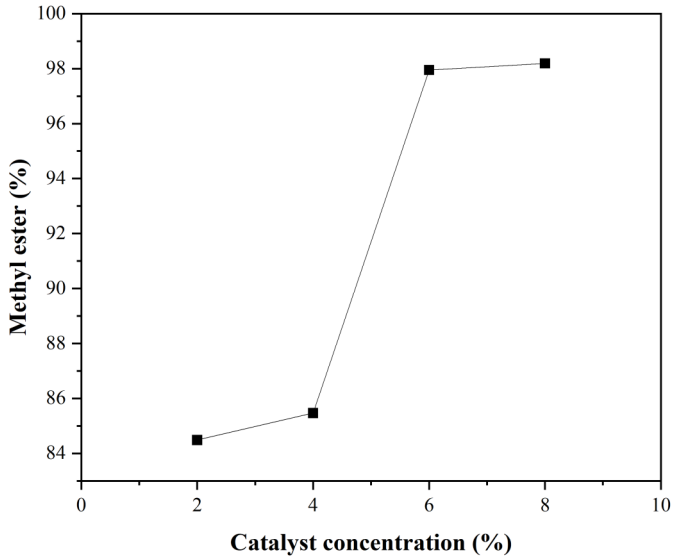

Figure 3. The relationship between catalyst concentration and methyl ester content under the conditions of a methanol-to-oil molar ratio of 15:1, reaction time of 2 hours, reaction temperature of 63°C, and stirring speed of 400 rpm. (Note: In this study, biodiesel yield refers to the total mass percentage of the final product phase obtained relative to the initial mass of sludge palm oil. Conversely, methyl ester content denotes the actual analytical purity, expressed as the percentage of fatty acid methyl esters in the final product, as quantified by gas chromatography (GC)).

The effect of transesterification reaction time on mono-, di-, and triglyceride levels

Figure 4 shows the SPO transesterification reaction process at 63°C over reaction times of 60, 120, and 180 minutes, using a 6% heterogeneous K_2O/SiO_2 catalyst derived from PDAM sludge, a methanol-to-oil molar ratio of 15:1, and a stirring speed of 400 rpm. In the first hour, the methyl ester content increased significantly to 84.34%. It then continued to increase and reached near equilibrium at 2 hours, with a methyl ester content of 97.88%. At 3 hours, only a slight increase to 98.02% was observed. As the methyl ester content increased significantly with transesterification time, the glyceride content decreased. This was due to the conversion of intermediate products, such as monoglycerides and diglycerides, during the reaction (Trejo-Zárraga et al., 2018). The triglyceride concentration decreased as the reaction progressed, reaching 0.29% after 3 hours.

The concentrations of monoglycerides and diglycerides also decreased as the transesterification reaction time increased, reaching 1.36% and 5.86%, respectively, in the first hour. These values then decreased significantly up to the third hour, with monoglycerides reaching 0.31% and diglycerides reaching 1.35%. This occurred because the transesterification reaction in SPO does not proceed instantaneously but takes place through several consecutive stages (Trejo-Zárraga et al., 2018).

The sequential nature of the transesterification process involves the conversion of triglycerides into diglycerides, monoglycerides, and, eventually, methyl esters and glycerol (Barbosa et al., 2022). This stepwise conversion can be represented by the following consecutive reactions (Equation 3).

$$\text{Triglyceride} + \text{ROH} \rightleftharpoons \text{Diglyceride} + \text{R'CO}_2\text{R}$$

$$\text{Diglyceride} + \text{ROH} \rightleftharpoons \text{Monoglyceride} + \text{R'CO}_2\text{R}$$

$$\text{Monoglyceride} + \text{ROH} \rightleftharpoons \text{Glycerol} + \text{R'CO}_2\text{R} \quad (3)$$

Because triglycerides act as the primary starting reactants in this stepwise conversion, their concentration decreases continuously as the reaction proceeds. Consequently, the final triglyceride level of 0.29% was lower than that of the intermediate diglycerides and monoglycerides once the reaction approached equilibrium. In the transesterification reaction, the reactants initially form a two-phase liquid system. In this state, the reaction is controlled by diffusion or mass transfer, and only limited diffusion occurs between the two immiscible phases, leading to a slow initial reaction. When esters are formed, they act as mutual solvents for the reactants, thereby forming a mixed liquid system (Barbosa et al., 2022;

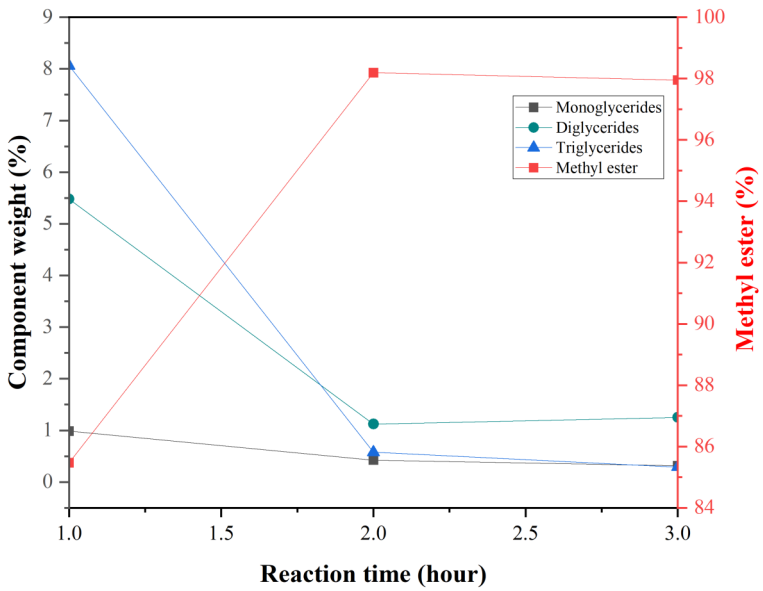

Figure 4. The effect of transesterification reaction time on mono-, di-, and triglyceride levels at a reaction temperature of 63°C, catalyst loading of 6%, methanol-to-oil molar ratio of 15:1, and stirring speed of 400 rpm

Darnoko & Cheryan 2000). This causes the SPO transesterification reaction to proceed progressively, whereby longer reaction times promote greater triglyceride conversion until equilibrium is reached.

Effectiveness of heterogeneous catalyst reuse on biodiesel yield

Reusability is one of the key economic factors in the use of heterogeneous catalysts for transesterification reactions. In this study, the catalyst was reused without reactivation. After the transesterification reaction was completed, the filtered catalyst was washed, dried, and weighed to determine its weight loss. The residual catalyst weight was then calculated and adjusted relative to the amount of SPO used to ensure uniform yield calculations. The relationship between catalyst reuse and biodiesel yield was observed under the best operating conditions: a reaction temperature of 63°C, a reaction time of 2 hours, a methanol-to-oil molar ratio of 15:1, and a catalyst loading of 6%.

Figure 5 shows that, in its initial use, the PDAM sludge-derived K_2O/SiO_2 catalyst produced a biodiesel yield of 70.43%. However, the heterogeneous K_2O/SiO_2 catalyst derived from PDAM sludge exhibited relatively low catalytic stability upon reuse, with the biodiesel yield decreasing from 70.43% in the first cycle to 43.36% in the third cycle. This decline, particularly from the second to the third cycle, was likely due to the loss of potassium species during the catalyst washing step. The leaching of K^+ may weaken or disrupt the interaction between potassium and the silica support, leading to the formation of surface intermediates such as KOH and potassium diglyceroxide. These species can block or mask active sites, thereby hindering effective contact between the reactants and the catalyst, which is a well-known deactivation mode for potassium-based solid bases in liquid-phase biodiesel systems (Hafriz et al., 2022; Li et al., 2025; Sádaba et al., 2015). In addition, the K_2O/SiO_2 catalyst is sensitive to air and moisture because silica readily adsorbs water. Exposure to O_2, H_2O, and CO_2 from the air can generate inactive carbonate or hydroxide species that block active basic sites (Mandari & Devarai 2022; Sádaba et al., 2015). The loss or transformation of these active potassium species can modify the catalyst structure and reduce its basicity, leading to lower catalytic activity upon reuse (de Oliveira et al., 2022; Hafriz et al., 2022; Li et al., 2025). Residual oil or heavy organic species remaining on the catalyst surface

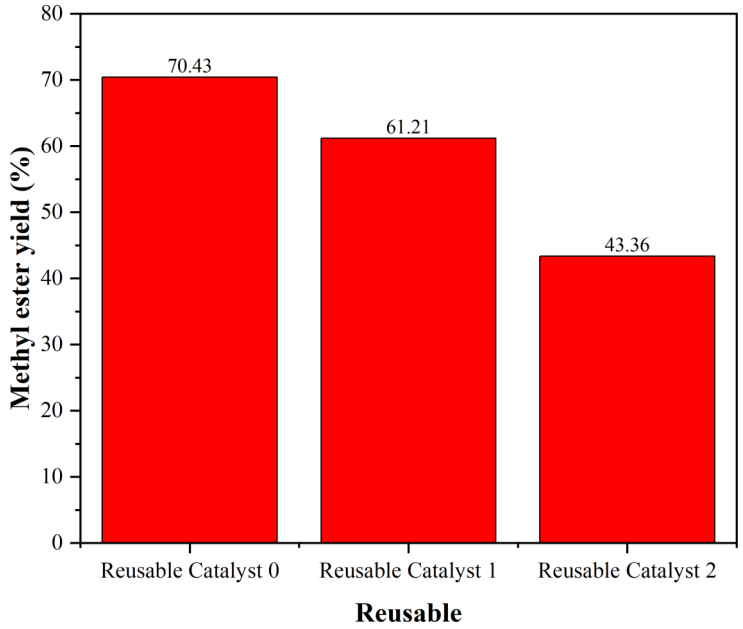

Figure 5. The effect of reusing the K_2O/SiO_2 catalyst derived from PDAM sludge on methyl ester yield. The catalyst exhibited decreasing stability upon reuse,with the yield dropping from 70.43% in the first cycle to 43.36% in the third cycle.

after the reaction can further foul the pores and active sites, compounding deactivation through surface coverage and fouling, which is a common problem in heterogeneous biodiesel catalysts (Argyle & Bartholomew 2015; Ishak et al., 2021; Lin et al., 2022).

Characteristics of SPO biodiesel

The viscosity of diesel fuel indicates its lubricating properties; fuels with relatively high viscosity tend to provide better lubrication. Another effect of viscosity is related to the fuel's ability to atomize when injected into the combustion cylinder. Fuel with relatively lower viscosity atomizes more easily and flows more readily. As shown in Table 2, the viscosity values of SPO biodiesel produced at a reaction time of 2 hours and a methanol-to-oil molar ratio of 15:1 exceeded the SNI 7182:2015 standard range of 2.3 –6.0 cSt. However, relatively high viscosity, when still close to the acceptable threshold, may contribute to lubrication and help reduce engine wear when blended with diesel fuel. When biodiesel density exceeds the specified limit, incomplete conversion during oil processing may occur (Aworanti et al., 2019). Biodiesel with a density exceeding the standard limit is not recommended for use in diesel engines because it can increase engine abrasion, produce excessive emissions, and potentially damage engine components. The density characteristics of the SPO biodiesel in this study remained within the SNI test range of 850–890 kg/m³ and were therefore suitable for use (Wirawan & Prayana Trisna, 2025). The FTIR analysis of the biodiesel is shown in Figure 6. The spectrum shows the presence of C –O bond vibrations associated with ester groups at 1169 cm^{-1} and 1124 cm^{-1}. In addition, no hydroxyl group absorption was observed at 3100–3500 cm^{-1}, indicating the absence of residual fatty acids and suggesting that the transesterification reaction proceeded as expected.

Table 2. Physical properties of SPO biodiesel

Catalyst concentration (%)	Kinematic viscosity at 40°C (cSt)	Density at 40? (kg/m^3)	Acid number (mg KOH/g)	Methyl ester content (%)
2	6.35	880	0.42	84.49
4	6.93	875	0.42	85.47
6	6.91	875	0.44	98.01
8	7.12	876	0.45	97.95

Table 3. SPO biodiesel specifications

Characteristics of SPO Biodiesel		SNI 7182:2015	
		Unit	Limit
Methyl ester content	98.01	%	96.5
Density at 40°C	875	kg/m^3	850-890
Viscosity at 40°C	6.96	mm^2/s (cSt)	2.3-6.0
Acid number	0.44	mg-KOH/g, max	0.5
Cloud point	15.20	°C, max	18
Flash point	174	°C, min	100
Monoglycerides	0.42	%mass, max	0.8
Diglycerides	1.12	%mass, max	-
Moisture content	0.11	%volume, max	0.05

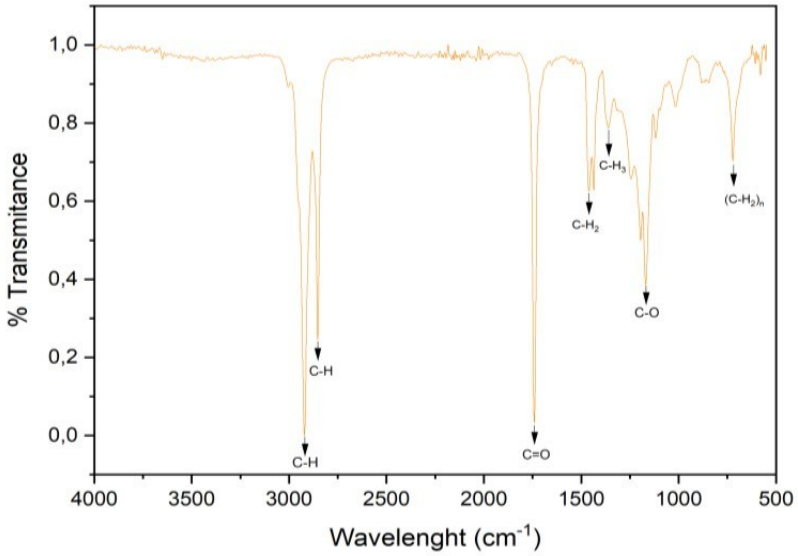

Figure 6. FTIR results of SPO biodiesel under optimum conditions (reaction time of 2 hours, catalyst loading of 6%, reaction temperature of 63°C, and stirring speed of 400 rpm). The spectrum confirms a successful transesterification reaction, indicated by the presence of C–O bond vibrations associated with ester groups (1169 cm^{-1} and 1124 cm^{-1}) and the absence of hydroxyl group absorption (3100–3500 cm^{-1}) from residual fatty acids.

Table 3 shows the specifications of biodiesel produced from SPO under optimum and economically favorable conditions, namely a biodiesel yield of 70.43%, a transesterification time of 2 hours, a catalyst loading of 6%, a reaction temperature of 63°C, a methanol-to-oil molar ratio of 15:1, and a stirring speed of 400 rpm.

CONCLUSION

This study successfully demonstrated a dual-waste valorization strategy by converting high-FFA sludge palm oil (SPO) into biodiesel using a low-cost heterogeneous catalyst supported by silica-rich water treatment sludge (PDAM sludge). The two-step process effectively treated SPO with an initial FFA content of 48.02% and produced biodiesel with a yield of 70.43% under optimal conditions: 6% catalyst loading, a 15:1 methanol-to-oil molar ratio, and a reaction time of 2 hours at 63°C. The synthesized biodiesel exhibited high quality, with a methyl ester content of 98.01%, exceeding the minimum requirement of 96.5%. Key physicochemical properties, including density (875 kg/m^3) and acid number (0.44 mg KOH/g), fully complied with the Indonesian National Standard (SNI 7182:2015). Although the moisture content (0.11%) exceeded the maximum limit of 0.05% and the viscosity (6.96 cSt) was slightly above the standard limit, these results suggest that the produced biodiesel may be more suitable for use as a blend with petrodiesel, such as B30 or B40, to improve atomization in engines. Future pilot-scale studies should incorporate extended vacuum drying to optimize moisture reduction. In terms of catalyst stability, the heterogeneous K_2O/SiO_2 catalyst derived from PDAM sludge maintained high activity for two consecutive cycles but became deactivated in the third cycle, with the yield decreasing to 43.36%, due to active-site leaching and pore blocking. Future work should focus on optimizing the calcination temperature or exploring surface modifications to enhance the stability of the potassium–silica interaction. Overall, utilizing PDAM sludge as a catalyst support offers a promising and sustainable pathway to reduce biodiesel production costs while simultaneously managing two industrial waste streams.

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DECLARATIONS

Author contributions

M. Anshari, M. Rivani, M.E.H. Nasution, M.A. Nasution, and T.R.F. Sinuhaji contributed equally as the main contributors to this paper. All authors read and approved the final manuscript.

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Competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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During the preparation of this work, the authors used ChatGPT to check for writing errors. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

GLOSSARY OF TERMS AND SYMBOLS

Terms & Symbols	Definition	Unit
CPO	Crude Palm Oil	-
FFA	Free Fatty Acid	-
FTIR	Fourier Transform Infrared Spectroscopy	-
GC	Gas Chromatography	-
PDAM	Medan Regional Water Company	-
PTSA	p-Toluene-4-sulfonic acid monohydrate	-
RBDPO	Refined, bleached, and deodorized palm oil	-
SNI	Indonesian National Standard	-
SPO	Sludge Palm Oil	-

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