

Enhanced Room-Temperature Transesterification of Palm Oil Using a DES K_2CO_3 -Glycerol Catalyst with Acetone as Co-Solvent

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ABSTRACT

Biodiesel production conventionally requires elevated temperatures (65–70 °C), resulting in high energy consumption. This study investigates a low-energy alternative by employing a deep eutectic solvent (DES) catalyst based on K_2CO_3 -glycerol for transesterification of palm cooking oil at room temperature (27–28 °C), assisted by acetone as a co-solvent to enhance phase miscibility. The effects of methanol concentration (25–35%), DES K_2CO_3 -glycerol catalyst loading (4–6%), and reaction time (2 and 4 h) were examined. Biodiesel properties—including density, viscosity, acid value, total glycerol, methyl ester content, and yield—were evaluated with reference to SNI 7182-2015 and ASTM D6751 standards. Increasing catalyst loading, methanol concentration, and reaction time generally reduced density, viscosity, and total glycerol while increasing methyl ester content. The most favorable operating conditions were obtained at 6% catalyst, 35% methanol, and 4 h reaction time, producing biodiesel with a density of 0.866 g/cm³, viscosity of 3.73 cSt, total glycerol of 0.404%, and methyl ester content of 95.7004%. While density and viscosity met SNI and ASTM specifications, the acid value and total glycerol did not fully comply, indicating the need for further catalyst and purification optimization. This work demonstrates the potential of DES K_2CO_3 -glycerol for room-temperature (27–28 °C) biodiesel synthesis and highlights key factors governing fuel quality in low-energy production routes.

1. Introduction

Concerns about increasing pollution, global warming, and the limited availability of fossil fuels have intensified research into renewable energy development. The continuous rise in fossil fuel consumption—from coal, oil, and natural gas - has led to resource depletion and elevated emissions, accelerating the global energy crisis. Persistent fossil fuel use also contributes substantially to atmospheric pollutants such as SO_2 , CO_2 , and CO, which are major drivers of global warming [1]. Consequently, the development of environmentally friendly, safe, and sustainable alternative fuels has become a priority for governments and industry.

Globally, biodiesel consumption in 2024 is projected to reach 44–45 million tons, aligning with the 50 billion liters of FAME biodiesel produced in 2023 [2]. Biodiesel is widely recognized as a renewable substitute or complement to diesel fuel and can be produced from vegetable oils through transesterification with methanol. Palm cooking oil is a particularly attractive feedstock due to its availability and potential to reduce CO_2 emissions, thereby mitigating climate impacts [3,4]. Biodiesel shares similar physical properties with diesel fuel and possesses a calorific value of approximately 37 MJ/kg, compared to 42.7 MJ/kg for diesel [5]. Through transesterification, triglycerides in vegetable oils are converted into fatty acid alkyl esters (biodiesel), with glycerol produced as a byproduct. This reaction proceeds through reversible steps involving triglycerides, diglycerides, monoglycerides, and methyl esters, facilitated by an external catalyst [6].

Alkaline catalysts such as KOH and NaOH are the most commonly used in biodiesel production. Recent studies have demonstrated that salts like

K_2CO_3 can enhance transesterification, particularly in oils such as jatropha [7]. Supported K_2CO_3 catalysts have also been shown to exhibit high catalytic activity [8]. Under typical operating temperatures of 50–80 °C, potassium and sodium methoxide formed in situ act as the active catalytic species. Mechanistically, base-catalyzed transesterification proceeds via nucleophilic attack of the alkoxide ion on the triglyceride carbonyl carbon, forming a tetrahedral intermediate that decomposes into methyl esters and glycerides [9,10]. Alkoxide catalysts, however, are highly sensitive to water and temperature, and elevated temperatures can increase soap formation. These limitations have motivated interest in glycerol-based media and deep eutectic solvents (DESs) as more stable catalytic environments [11].

Glycerol-based DESs are considered green solvents due to their low volatility, biodegradability, low cost, and potential as hydrogen-bond donors [12]. Compared with ionic liquids, DESs are also easier to synthesize [13]. DESs composed of phosphonium or ammonium salts combined with various hydrogen-bond donors have demonstrated catalytic activity in esterification and transesterification. For example, phosphonium DESs have effectively reduced free fatty acid levels in low-grade CPO [13], and choline chloride-based DESs have similarly achieved substantial FFA reduction [14]. Other studies have reported high FFA conversion using DES catalysts at elevated temperatures [15]. Despite these advances, most DES-based catalytic systems still operate above 100 °C.

Considering that Indonesian crude palm oil consists mainly of triglycerides, there remains a need for transesterification catalysts derived from salt-based DESs that operate efficiently at milder conditions. DES K_2CO_3 -glycerol is a promising candidate, as potassium carbonate in glycerol offers environmental compatibility and glycerol is an abundant byproduct of biodiesel production [12].

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As an alkaline catalyst, DES K_2CO_3 -glycerol can promote faster transesterification even at lower temperatures by lowering the activation energy required [16].

Another key challenge in biodiesel synthesis is the immiscibility of methanol and oil, which limits reaction rates. Co-solvents such as acetone improve mutual solubility and enhance mass transfer between reactants, thereby accelerating conversion [17]. Given that traditional biodiesel production typically requires 65–70 °C [18], the development of a room-temperature (27–28 °C) process using acetone co-solvent could significantly reduce energy consumption and production costs.

Based on these considerations, this study examines the effects of methanol concentration, DES K_2CO_3 -glycerol catalyst loading, and reaction time on biodiesel production from palm cooking oil at room temperature (27–28 °C) with acetone as a co-solvent. The resulting biodiesel quality is evaluated according to the Indonesian National Standard (SNI 7182-2015) and ASTM D6751, focusing on density, viscosity, acid number, total glycerol, and methyl ester content. This work aims to assess the feasibility of using DES K_2CO_3 -glycerol catalysts to enable low-temperature, energy-efficient biodiesel production.

2. Materials and Methods

2.1. Materials

Palm cooking oil was used as the feedstock. Methanol (CH_3OH , p.a), glacial acetic acid (CH_3COOH , p.a), acetone (p.a), and distilled water were utilized throughout the experiment. The catalyst precursor consisted of potassium carbonate (K_2CO_3 , p.a) and industrial-grade glycerol, which were combined to synthesize the deep eutectic solvent (DES) K_2CO_3 -glycerol. Several analytical reagents—including KOH-ethanol solution, periodic acid solution, phenolphthalein indicator, starch indicator, and other standard solutions—were employed for determining total glycerol (TG), acid number, and related biodiesel parameters following FBI-A01-03, FBI-A02-03, and FBI-A03-03 methods.

2.2. Catalyst Preparation

The DES K_2CO_3 -glycerol catalyst was prepared by modifying the procedure of [12]. K_2CO_3 and glycerol were mixed in a molar ratio of 1:3.5. Glycerol was heated to 50–60 °C in an Erlenmeyer flask without stirring. Once the temperature reached 60 °C, K_2CO_3 was gradually added, and the mixture was stirred at 400 rpm while heating continued until 80 °C. The reaction time of 6 hours commenced when the mixture reached 80 °C. During the first 2 hours, stirring was maintained at 400 rpm, followed by a reduction to 200 rpm for the remaining 4 hours. The resulting DES K_2CO_3 -glycerol was stored and used as the catalyst for biodiesel production.

2.3. Transesterification Procedure

Transesterification was conducted using 100 g of palm cooking oil for each experimental run. The DES K_2CO_3 -glycerol catalyst was weighed according to the desired concentration (4%, 4.5%, 5%, 5.5%, or 6% w/w relative to oil). Methanol was prepared in three variations (25%, 30%, and 35% w/w relative to oil). Acetone was added as a co-solvent at 20% of the oil mass.

All components—oil, methanol, catalyst, and acetone—were placed in a sealed Erlenmeyer flask. The reaction was performed at room temperature (27–28 °C) with constant stirring at 300 rpm for either 2 hours or 4 hours. After the designated reaction time, glacial acetic acid equivalent to 80% of the catalyst mass was added to neutralize the alkaline DES K_2CO_3 -glycerol catalyst, and the mixture was stirred for an additional 5 minutes.

Following neutralization, stirring was stopped, and the mixture was transferred into a separation funnel and allowed to settle for 24 hours to enable phase separation. The lower glycerol layer was removed, weighed, and stored for further analysis. The upper biodiesel layer was collected for purification.

2.4. Purification of Biodiesel

The crude biodiesel was washed repeatedly with warm distilled water (40–50 °C) until the wash water became clear, indicating the removal of

residual catalyst, soap, and impurities. The washed biodiesel was then heated at 90–100 °C with gentle stirring for 1 hour to evaporate any remaining moisture. The final purified biodiesel product was weighed, stored in clean airtight glass bottles, and prepared for characterization.

2.5. Biodiesel Characterization

Biodiesel quality parameters were analyzed to assess compliance with SNI 7182-2015 and ASTM D6751 standards. Density (g/cm^3) was measured at 40 °C using a pycnometer and viscosity (cSt) was measured at 40 °C using an Ostwald viscometer. Other characteristics of biodiesel such as acid number (mg-KOH/g), total glycerol (% mass), and methyl ester content (% mass) were determined following FBI-A01-03, FBI-A02-03, and FBI-A03-03 analytical procedures. Meanwhile, biodiesel yield (%) was calculated as the ratio of the mass of purified biodiesel to the initial mass of palm cooking oil. These analyses were performed for all combinations of catalyst amount, methanol percentage, and reaction time.

3. Results and Discussion

3.1. Effect of Catalyst Amount and Reaction Time on Biodiesel Density

Density is an essential parameter because it influences fuel atomization efficiency and overall engine performance [19]. As shown in Figure 1, the highest density (0.8814 g/cm^3) was obtained from the sample processed for 2 hours using 25% methanol and 4% catalyst, while the lowest density (0.8698 g/cm^3) was observed for the 4-hour reaction with 25% methanol and 6% catalyst.

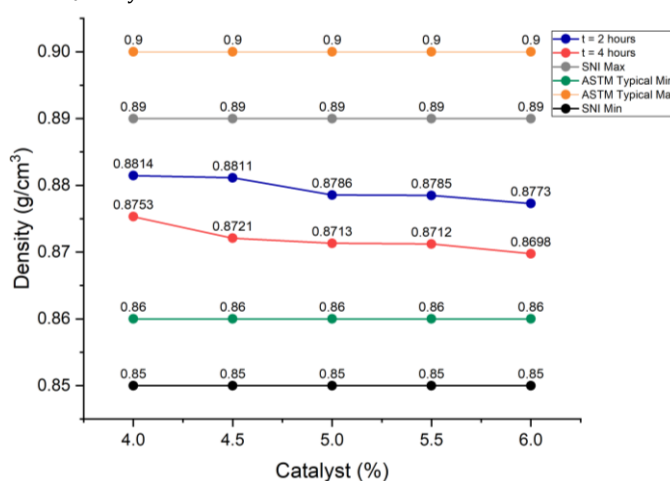


Figure 1. Effect of Catalyst amount and Reaction Time on Biodiesel Density

Density generally decreased with increasing catalyst loading and longer reaction time, indicating more complete transesterification and reduced residual glycerides. All samples remained within the SNI 7182:2015 and ASTM D6751 density ranges. Enhanced catalyst amount promotes faster triglyceride conversion, reducing residual glycerol and unreacted triglycerides, both of which contribute to higher density [20]. A longer reaction time similarly improves conversion efficiency, lowering the biodiesel density [21].

Biodiesel with excessively high density may cause poor atomization, extended ignition delay, and higher exhaust temperatures, which in turn can increase NO_x emissions and damage engine components [22,23]. The density values obtained in this study fall within acceptable ranges, indicating effective transesterification under the tested conditions.

3.2. Effect of Catalyst amount and Reaction Time on Biodiesel Viscosity

Viscosity determines fuel atomization and injection characteristics. High viscosity can hinder fuel flow and atomization, whereas excessively low viscosity may cause leakage in fuel injection pumps [24]. Figure 2 shows Viscosity decreased as catalyst concentration and reaction time increased, reflecting improved triglyceride conversion into simpler fatty acid methyl esters. All viscosity values satisfy SNI and ASTM standards. The highest viscosity (5.0417 cSt) was obtained with 25% methanol and 4% catalyst at 2 hours, while the lowest viscosity (3.7287 cSt) occurred with

35% methanol and 6% catalyst at 4 hours.

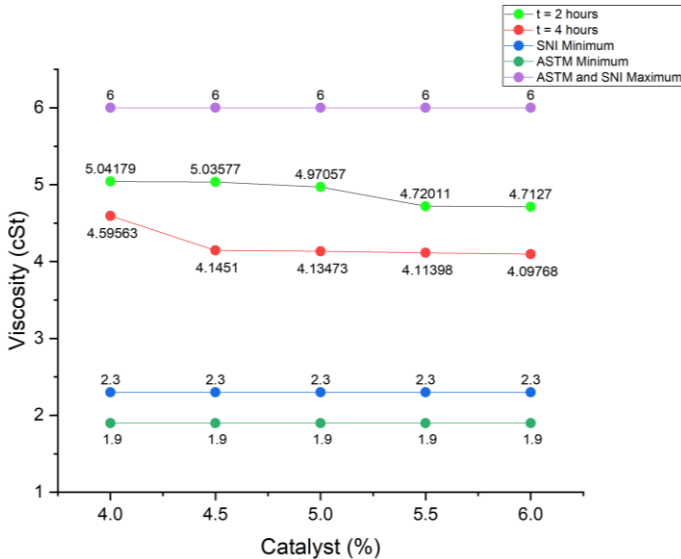


Figure 2. Effect of Catalyst amount and Reaction Time on Biodiesel Viscosity

The reduction in viscosity reflects enhanced triglyceride breakdown at higher catalyst amounts, yielding simpler fatty acid esters with lower molecular weights and viscosities [25]. Longer reaction times further promote ester formation, consistent with literature reporting viscosity decreases in extended transesterification [26].

3.3. Effect of Catalyst amount and Reaction Time on Biodiesel Yield

Biodiesel yield is influenced by reaction duration, catalyst amount, and methanol-to-oil ratio [27]. Yield increased with catalyst loading at 2 hours but decreased at higher catalyst levels during 4-hour reactions, likely due to soap formation and phase separation challenges. As shown in Figure 3, the highest yield was achieved at 4.5% catalyst for 4 hours, likely due to higher availability of active sites that promote faster reaction rates [28–30]. However, at 4 hours, excessive catalyst ($\geq 5.5\%$) decreased yield. This reduction is associated with soap formation from the alkaline DES K₂CO₃-glycerol catalyst, particularly when excess methanol is present [31,32]. Excess catalyst may also increase mixture viscosity and hinder mass transfer [33]. Additionally, imperfect phase separation may cause biodiesel to be entrained in crude glycerol during settling [34].

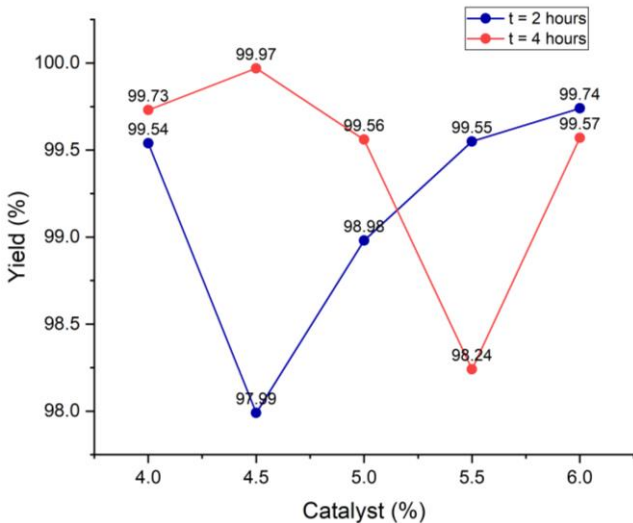


Figure 3. Effect of Catalyst amount and Reaction Time on Biodiesel Yield

3.4. Effect of Catalyst amount and Reaction Time on Acid Number

The acid number reflects the concentration of free fatty acids (FFA) in biodiesel and indicates fuel stability [35]. Acid number decreased with higher catalyst loading but increased with extended reaction time, partly due to oxidation, hydrolysis, and residual glacial acetic acid. All samples exceeded the maximum acid value permitted by SNI and ASTM (Figure 4).

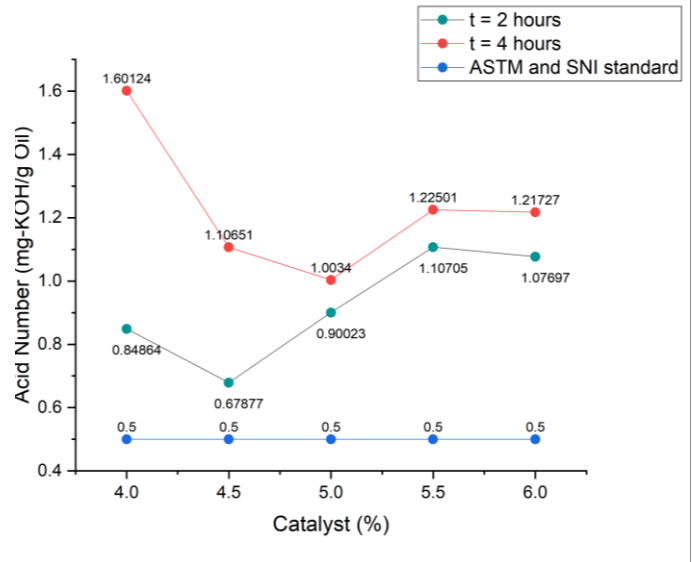


Figure 4. Effect of Catalyst amount and Reaction Time on Biodiesel Acid Number

Higher acid values at extended reaction times may be attributed to oxidation or hydrolysis during washing and heating, allowing fatty acids to regenerate [36]. Furthermore, glacial acetic acid added for catalyst neutralization may contribute to elevated acid values if not completely removed during washing [37]. Thus, measured acid levels may reflect residual acetic acid rather than FFA content alone.

High acidity can accelerate corrosion and form deposits in engine components [38,39]. Optimization of washing steps or reduction of acetic acid dosage may be necessary to lower the acid number.

3.5. Effect of Catalyst amount and Reaction Time on Total Glycerol

Total glycerol represents the sum of free glycerol and bound glycerol in mono-, di-, and triglycerides and is a critical indicator of biodiesel purity [40,41]. Total glycerol declined steadily with increasing catalyst concentration and reaction duration, indicating improved conversion and phase separation (Figure 5). However, all samples remained above the 0.24% limit specified by SNI and ASTM.

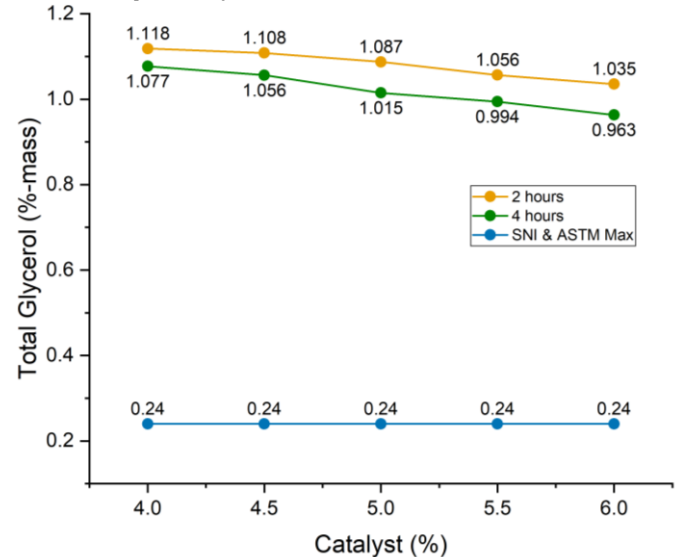


Figure 5. Effect of Catalyst amount and Reaction Time on Total Glycerol Content in Biodiesel

Higher catalyst loading promotes more complete transesterification and reduces remaining intermediate glycerides [42,43]. Longer reaction times similarly enhance ester formation and facilitate phase separation [43]. High glycerol content can cause engine deposits and unstable combustion [44].

3.6. Effect of Catalyst amount and Reaction Time on Methyl Ester

Content

According to the standards set forth in SNI-7182:2015, the biodiesel produced must have a minimum methyl ester content of 96.5%. The level of methyl ester is a key indicator of the effectiveness of the biodiesel production process. Figure 6 illustrates the levels of methyl esters observed in this study, indicating that none of the biodiesel samples met the SNI 7182:2015 standards. However, the ASTM D6751 standard was created to accommodate various production methods and feedstocks that are prevalent in the U.S., primarily focusing on soybean and waste cooking oils. By not exclusively identifying "methyl" as the only acceptable ester, the standard permits the inclusion of other alcohols (such as ethanol), provided that the final product fulfills the overall quality and performance criteria specified.

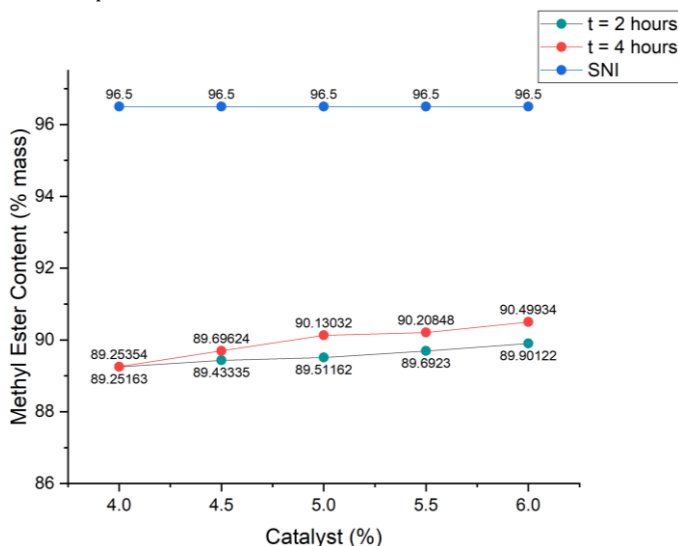


Figure 6. Effect of Catalyst amount and Reaction Time on Methyl Ester Content of Biodiesel

This non-compliance may be attributed to an inefficient catalyst that hinders the conversion of triglycerides into methyl esters [45]. There is a close, inverse correlation between the levels of methyl ester and total glycerol content. As the total glycerol content diminishes in biodiesel, the methyl ester concentration rises. Glycerol is a byproduct generated during the transesterification process that yields biodiesel. These findings suggest that elevated levels of glycerol are indicative of inadequate conversion of triglycerides into fatty acid esters in the biodiesel [46].

According to Figure 6, a rise in the quantity of catalysts generally leads to an increase in the content of methyl esters. This occurs because catalysts enhance the transesterification reaction, leading to a reduction in the overall glycerol concentration in biodiesel and consequently raising the levels of methyl esters. A catalyst amount of 6% with 30% methanol resulted in the highest methyl ester concentrations, achieving 89.9012% after 2 hours and 90.4993% after 4 hours of reaction time. The increased amounts of catalysts in the biodiesel transesterification process elevates methyl ester levels up to a specific threshold. Catalysts function by speeding up reactions and enhancing the reaction rate, which allows for the production of a greater amount of biodiesel compared to the triglycerides present in the oil [47].

3.7. Effect of Methanol Concentration on Biodiesel Density

Methanol acts as both a reactant and a solvent that enhances system homogeneity [48]. Figure 7 shows that Density decreased with increasing methanol concentration, reflecting enhanced solubility and more complete transesterification. All values met SNI and ASTM density standards. Excess methanol improves solubility and accelerates transesterification, reducing unreacted glycerides and lowering density [49].

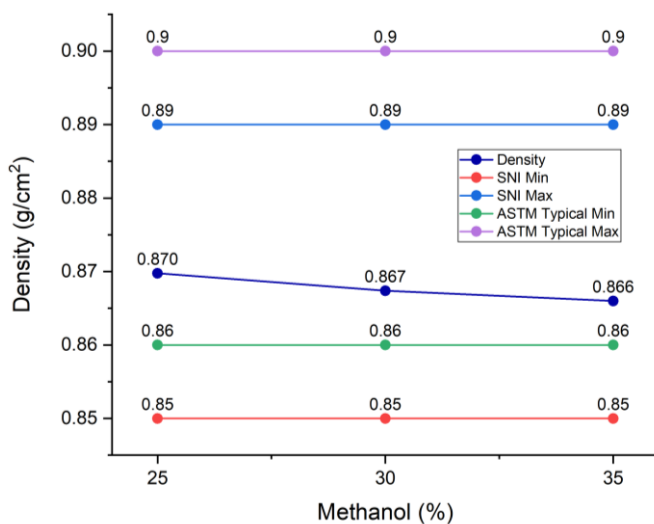


Figure 7. Effect of Methanol Concentration on Biodiesel Density

3.8. Effect of Methanol Concentration on Biodiesel Viscosity

Higher methanol concentrations reduced viscosity due to improved reactant miscibility and esters formation. All viscosity values complied with SNI and ASTM specifications. Figure 8 shows that the lowest viscosity (3.7287 cSt) was observed with 35% methanol and 6% catalyst. Enhanced methanol availability improves mixing and solubilization of triglycerides, supported by acetone co-solvent and DES K2CO3-glycerol catalyst, which contribute to better reaction kinetics [50,51].

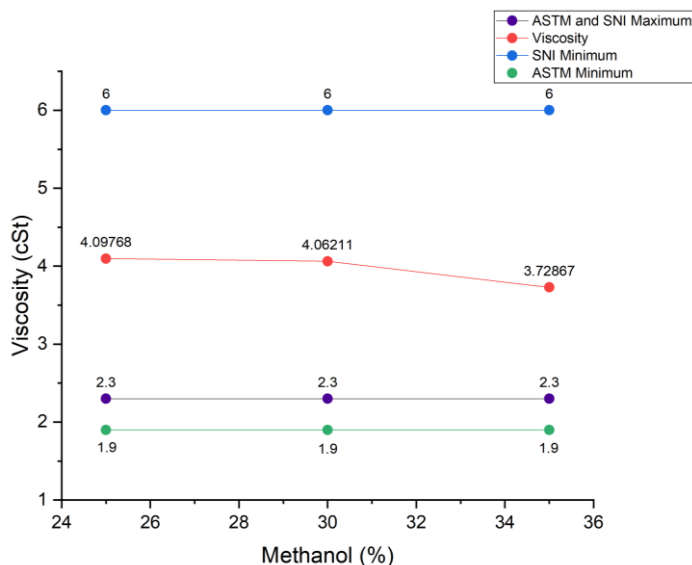


Figure 8. Effect of Methanol Concentration on Biodiesel Viscosity

3.9. Effect of Methanol Concentration on Biodiesel Yield

Figure 9 indicates that Increasing methanol concentration produced a slight decrease in yield, attributed to glycerol solubility and potential reverse reaction. Differences among the three methanol levels were relatively small. Excess methanol enhances glycerol solubility in biodiesel, making separation more difficult and reducing the apparent biodiesel mass [52]. Excess methanol may also shift the reaction backward (reverse transesterification) during prolonged reaction [53,54].

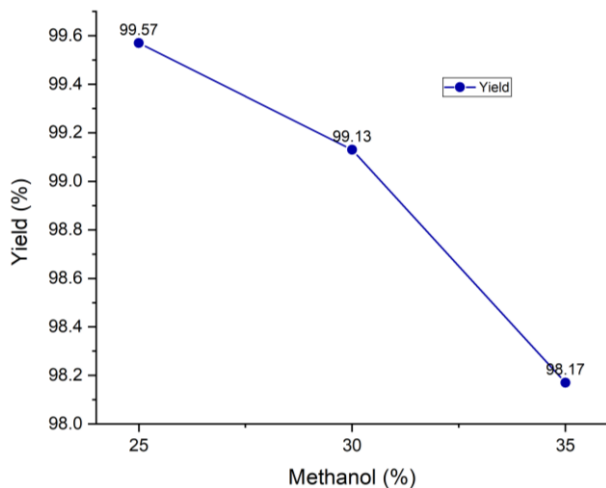


Figure 9. Effect of Methanol Concentration on Biodiesel Yield

3.10. Effect of Methanol Concentration on Acid Number

As shown in Figure 10, Acid number decreased with increasing methanol concentration, though none of the samples met SNI or ASTM limits, likely due to residual acetic acid or incomplete removal of free fatty acids [55]. Excessive or insufficient methanol can reduce reaction efficiency, necessitating optimization [26].

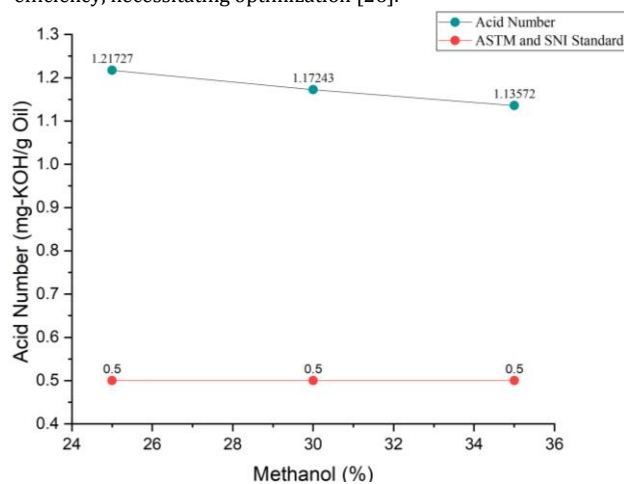


Figure 10. Effect of Methanol Concentration on Acid Number

3.11. Effect of Methanol Concentration on Total Glycerol

Figure 11 shows that total glycerol decreased substantially with increasing methanol concentration, with the lowest value occurring at 35% methanol. Nonetheless, all samples exceeded the SNI/ASTM threshold of 0.24%. The lowest value (0.404%) occurred at 35% methanol with 6% catalyst, consistent with faster methanolysis and improved glycerol-ester phase separation [36].

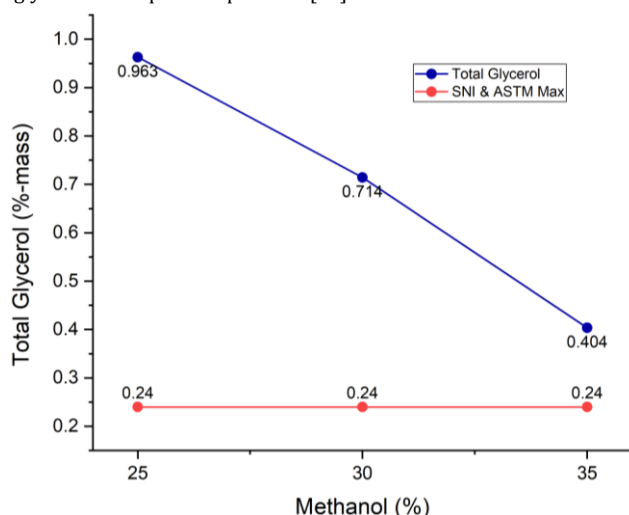


Figure 11. Effect of Methanol Concentration on Total Glycerol in Biodiesel

3.12. Effect of Methanol Concentration on Methyl Ester Content

Methyl ester content increased with methanol concentration, reaching a maximum at 35% methanol, although still below the SNI requirement of $\geq 96.5\%$ (Figure 12). Lower ester content reflects incomplete conversion or the presence of impurities such as residual glycerol [56,57]. Optimal methanol usage is therefore critical for achieving high methyl ester levels.

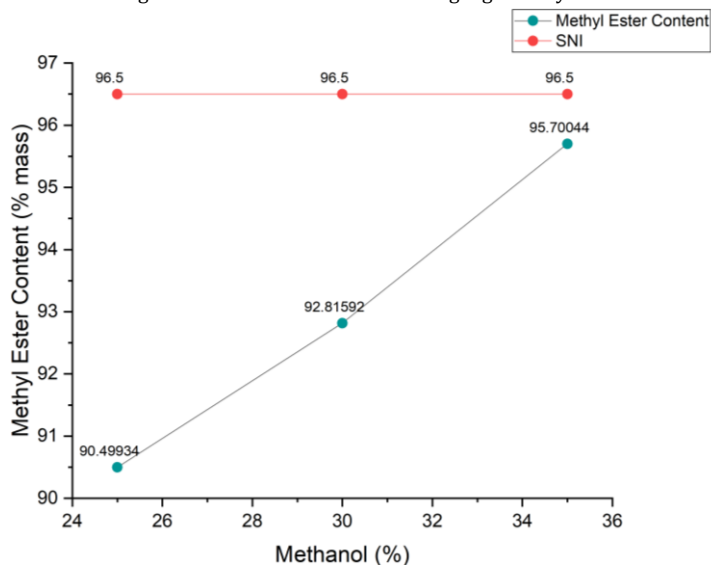


Figure 12. Effect of Methanol Concentration on Methyl Ester Content

4. Conclusion

This study investigated biodiesel production from palm cooking oil at room temperature (27-28 °C) using a DES K_2CO_3 -glycerol catalyst and acetone as a co-solvent. Variations in catalyst amount, methanol percentage, and reaction time were evaluated with respect to key biodiesel quality parameters defined by SNI 7182-2015 and ASTM D6751.

Increasing the catalyst amount and reaction time generally reduced density, viscosity, and total glycerol levels while increasing methyl ester content, indicating improved transesterification efficiency. Methanol concentration also played a significant role, where higher methanol levels enhanced ester formation and reduced density, viscosity, and acid number. Across all conditions tested, the most favorable biodiesel properties were obtained using 6% catalyst, 35% methanol, and a reaction time of 4 hours. Under these conditions, the biodiesel achieved a density of 0.866 g/cm^3 , viscosity of 3.72867 cSt , total glycerol content of 0.404% , and methyl ester content of 95.7004% .

While density and viscosity met SNI and ASTM specifications, the acid number and total glycerol did not fully comply with the required limits, indicating incomplete reaction or insufficient purification. These findings suggest that although the DES K_2CO_3 -glycerol catalyst demonstrates promising catalytic activity at room temperature (27-28 °C), further optimization—particularly in catalyst formulation, methanol removal, and biodiesel washing—remains necessary to meet full biodiesel quality standards.

Overall, this research highlights the potential of DES K_2CO_3 -glycerol catalysis combined with acetone co-solvent to enable low-energy biodiesel production. Future investigations should focus on catalyst recyclability, minimizing soap formation, enhancing purification strategies, and evaluating the economic feasibility of room-temperature (27-28 °C) biodiesel processes.

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