

Influence of Methanol Solvent and Alkali Catalyst on Biodiesel Production from Cottonseed Oil

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Abstract – This study investigates the influence of methanol solvent and alkali catalyst on biodiesel production from cottonseed oil. Utilizing local cottonseed oil from Ethiopia, the research focuses on optimizing the methanol-to-oil molar ratio and catalyst concentration to maximize biodiesel yield. The transesterification process was conducted with varying methanol-to-oil ratios (5:1, 6:1, 7:1) and sodium hydroxide (NaOH) concentrations (0.5 wt.%, 1 wt.%, 1.5 wt.%). Results indicated that a 6:1 methanol-to-oil ratio and 1 wt.% NaOH at 65 °C yielded an average biodiesel output of 98.25%. Methanol outperformed than ethanol and butanol by producing higher biodiesel yields. Besides, catalyst (NaOH) concentration is crucial for better yield, while deviations led to soap formation or incomplete reactions. In other words, moderate temperatures (55-65 °C) were seen optimal as higher temperatures (eg., 75 °C) caused methanol evaporation, reducing yield. Moreover, methanol's low cost, high reactivity, and ease of recovery, combined with NaOH's efficiency in catalyzing the reaction, were key factors in achieving high yields. This research underscores the importance of precise optimization in biodiesel production, contributing to sustainable energy solutions and promoting the use of regional agricultural resources. Future studies should explore pretreatment of cottonseed oil to further enhance the sustainability and economic viability of biodiesel production.

Keywords: Renewable feedstock; Transesterification; Optimization; Fatty Acid Methyl Esters; Sustainable biofuel; Cottonseed Oil.

Received: 15/04/2025 – Revised: 20/05/2025 – Accepted: 03/06/2025

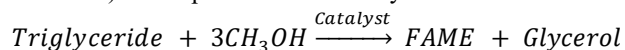
I. Introduction

The 21st century has witnessed unprecedented energy demands driven by industrialization, population growth, and urbanization. In 2024, fossil fuels, accounted for 81.5% of global primary energy consumption [1], are unsustainable due to finite reserves and severe environmental impacts. The combustion of petroleum diesel releases greenhouse gases (GHGs), including CO₂ (contributing to climate change) and particulate matter (linked to respiratory diseases) [2]. According to the Intergovernmental Panel on Climate Change [3], the transportation sector alone accounts for 24% of global CO₂ emissions, necessitating urgent transitions to renewable alternatives.

Biodiesel, a renewable, biodegradable, and non-toxic fuel derived from vegetable oils or animal fats, has

emerged as a viable substitute. Its chemical composition—fatty acid methyl esters (FAME)—ensures compatibility with conventional diesel engines without modification [4]. The global biodiesel market is projected to grow at a CAGR of 5.1% from 2023 to 2030, driven by policies like the EU Renewable Energy Directive (RED II) and U.S. Renewable Fuel Standard (RFS) [5]. Further, in recent years, advancements in production technologies have improved efficiency and scalability. Various methods such as high-shear mixing and ultrasonic reactors have been developed to enhance the transesterification process, reducing reaction times and increasing yields. These innovations are ensuring growing global demands for biodiesel, projected to reach approximately 277 million tons annually by 2050 [6].

Biodiesel is primarily produced via transesterification, where triglycerides in oils react with alcohol (e.g., methanol) in the presence of a catalyst:



Methanol is preferred over ethanol due to its lower cost (\$0.30/L), higher reactivity, and easier separation from glycerol [7]. However, it is essential to optimize the methanol-to-oil ratio in between 6:1-8:1, as excessive amounts can lead to increased glycerol solubility, complicates product separation and potentially lowers overall yield [8]. In other words, homogeneous alkali catalysts (NaOH, KOH) offer high conversion rates (>90%) but require feedstock with low free fatty acids (FFA < 2%) to avoid saponification [9] whereas heterogeneous catalysts (e.g., CaO, MgO) reduce soap formation but suffer from slower reaction rates [10].

Cottonseed oil (CSO), a byproduct of cotton farming, is an attractive biodiesel feedstock due to high oil content (18–25%) and global production of 5.3 million metric tons/year [11]. Additionally, non-edible status of CSO avoiding conflicts with food supply chains [12] while possessing lower cost (\$0.50/kg) compared to soybean (\$0.80/kg) or palm oil (\$0.70/kg) [5]. However, CSO's high FFA content (up to 5%) necessitates acid pretreatment (esterification) before alkali-catalyzed transesterification [2].

While prior studies have explored CSO-based biodiesel, critical gaps remain methanol optimization, catalyst comparison and quality compliance. Most studies use a fixed 6:1 molar ratio while systematic evaluation of higher ratios (e.g., 8:1) is lacking [13]. In other words, NaOH and KOH are often tested in isolation, with no consensus on which performs better for CSO [9]. Additionally, few studies validate CSO biodiesel against ASTM D6751 standards for viscosity, cetane number, and oxidation stability [4].

Rashid et al. (2020) achieved 92% FAME yield from CSO using NaOH (1% wt.) and a 6:1 methanol ratio at 60°C [12]. while Leung et al. (2020) reported that 8:1 methanol ratio increased yield by 5–7% but required additional purification steps [7]. Further using NaOH catalyst favors of low cost (\$0.15/kg) and rapid reaction kinetics [12] despite of soap formation at FFA > 2%, complicating glycerol separation [5]. Whereas KOH has an advantage of formation of softer soap coupled with easier purification [9] despite of its higher cost (\$0.25/kg) and hygroscopicity (absorbs moisture) [2]. Further, acid pretreatment of cottonseed oil using H₂SO₄ reported to reduce FFAs to <1%, enabling efficient alkali catalysis [10]. Furthermore, two-step processes which

includes acid esterification followed by alkali transesterification improve yields to >95% [8].

However, the feedstock used in biodiesel production is crucial, as it constitutes a significant portion of production costs—often around 80%. Common feedstock includes vegetable oils (such as soybean and canola), animal fats, and recycled cooking oils. The choice of feedstock affects not only the cost but also the quality and yield of the biodiesel produced [14]. Thus, this study mainly addresses the variation in the yield of biodiesel by changing the methanol-to-oil ratios (5:1–7:1) and the alkali catalyst (NaOH) compositions (0.5–1.5% wt.) for transesterification using the local cottonseed oil as potential feedstock from Ethiopia under controlled conditions.

II. Materials and Methods

Methanol (CH₃OH) used as a solvent, Sodium hydroxide (NaOH) was applied as a catalyst, and Cottonseeds collected from the farmers of Jimma Zone in Ethiopia used as feedstock for oil extraction and for further biodiesel production process.

II.1. Cottonseed oil extraction and purification

About 3.5 kg of cottonseed sample were collected from farmer area in Jimma Zone South Western, Ethiopia and brought to a Chemical Reaction Engineering (CRE) laboratory, Jimma Institute of Technology (JIT), Department of Chemical Engineering for further processing. Then the sample were cleaned to remove all foreign materials such as foreign particles, sand particles, dirt, dust and washed thoroughly with tap water. Afterwards they were sun dried for one day and sent to Addis Mojo Oil Factory for extracting cottonseed oil using mechanical pressing machine. Finally, the product was separated from solid suspensions and impurities by using centrifuge at speed of 500 rpm for 20 minutes and about 0.985 kg crude cottonseed oil was extracted and stored at room temperature.

II.2. Alcohol-Catalyst Mixing

This typical process was carried out as depicted in Figure 1, by mixing sodium hydroxide (NaOH) solid pellets with pure methanol. Despite of its high toxicity, methanol is preferred in biodiesel production as it requires simple technology, possesses higher reactivity and lower cost [15]. Alkali hydroxide was first dissolved in the alcohol to produce alkoxide solution (alcohol-

catalyst mixture). Later methanol was mixed and stirred well until it completely dissolves with the catalyst using standard agitator before adding the oil. In 100 ml beakers, different molar ratios (5:1, 6:1, 7:1) of methanol to oil were charged to observe the impact of change in solvent to oil ration on biodiesel yield. Otherwise, another set of experiments were performed in 100 ml capacity glass beakers by changing the amount of catalyst (NaOH) as 0.5 wt.%, 1 wt.% and 1.5 wt.% while maintaining 6:1 methanol to oil ratio at a temperature of 65 °C.

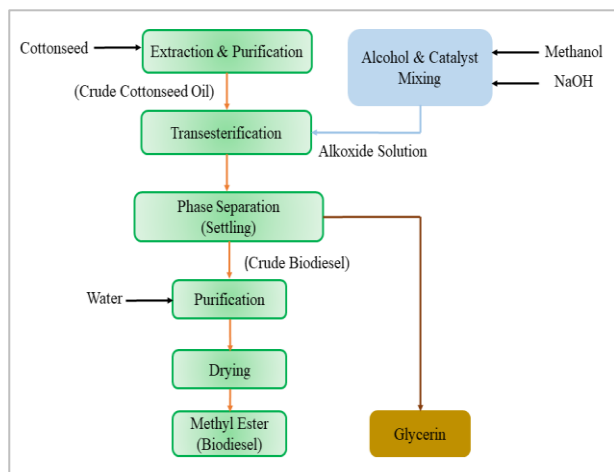


Figure 1. Schematic diagram of biodiesel production from cottonseed oil

II.3. Biodiesel Preparation

The chemical reaction takes place when the solution of sodium methoxide prepared by dissolving of catalyst (NaOH) in methanol. And it is added to a 20 ml of purified warm cottonseed oil, the mixture is poured into a reaction vessel in water bath, with defined agitation throughout the reaction using a standard agitator, where the reaction was maintained at 65°C with residence time of 30 min to allow the transesterification to proceed. The mixture was transferred to a settling vessel and allowed to stay overnight for the reaction to be completed. Once the reaction accomplished, there exists two separate layers of methyl ester of cottonseed oil and the glycerol phase (much denser) at the bottom. In a well completed reaction, glycerol begins to separate immediately when stirring and heating of the mixture is stopped with two-layers being separated by means of a separating funnel.

II.4. Transesterification reaction

The transesterification (alcoholysis), is a chemical reaction between of triglyceride (fat or oil) with an alcohol in the presence of a catalyst to form esters and

glycerol [16]. A triglyceride has a glycerine molecule as its base with three long chain fatty acids attached. Methyl esters are the product of the reaction when methanol is the alcohol used in the process and it is called methanolysis [15]. The stoichiometric transesterification requires, 3:1 molar ratio of alcohol to triglyceride to complete the reaction, while the associated reaction produces 3 mole of fatty acids methyl esters (FAME) and 1 mole of glycerin. The amount of alcohol, catalyst, reaction temperature and time, alcohol-to-oil ratio and rate of mixing are the main factors that affect transesterification reaction [17-18]. As depicted in Figure 2, in the presence of NaOH catalyst, crude cottonseed oil reacts with methanol to produce methyl esters of fatty acids (biodiesel) and glycerol.

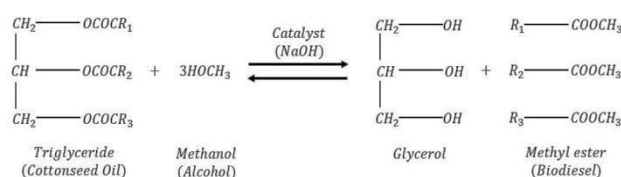


Figure 2. Transesterification reaction with methanol for biodiesel production

II.5. Separation and Purification of the Products

After the reaction is completed, the two phases are separated by decanting. Draw off glycerol from the bottom and biodiesel (methyl ester) from the top of the settling vessel. To recover high quality, the methyl ester layer (rich in biodiesel) was washed with warm distilled water to remove by-products (impurities) such as residual catalyst (NaOH), remnants of alcohol (un-reacted), glycerin or soaps, which can damage engine components, reduce fuel lubricity and cause injection cooking and other deposits [15]. Finally, the biodiesel portion was heated on a heating mantle to dry and remove the remaining water molecules from it. The heavier, co-product (glycerol) may be sold as it is or may be purified for use in other industries like pharmaceutical, and cosmetics [16].

III. Results and Discussion

The alkali-based catalyzed transesterification process was carried out at different catalyst concentrations and methanol to oil molar ratios to obtain optimal yield of methyl esters of cottonseed oil. It was found that optimized variables of 6:1 methanol to oil molar ratio, 1 % NaOH concentration (wt.%), 65 °C reaction

temperature and 30 min reaction time offered the maximum methyl ester yield (98.9%).

III.1. Effect of catalyst concentration on biodiesel production

In this experiment, different catalyst concentrations as shown in Figure 3, used were impacted on the percentage of yield of biodiesel. At constant temperature (65 °C) and at a molar ratio of methanol to oil of 6:1, by increasing catalyst (NaOH) concentrations from 0.5 to 1 wt.%, the percentage of yield of biodiesel production increased from 71.6% to 98.9% (Figure 4). Higher concentrations of catalyst decreased the reaction yield, with the yield dropping to 77.4% at 1.5 wt.% NaOH. So, this result indicates that the 1 g of NaOH gave the optimum yield of biodiesel for the transesterification reaction.



Figure 3. Methyl esters of cottonseed oil at varied concentrations of NaOH catalyst.

III.2. Effect of different methanol to oil molar ratio on biodiesel production

In the transesterification process of cottonseed oil, different methanol to oil molar ratio was studied, for instance, 5:1 (sample 1), 6:1 (sample 2), and 7:1 (sample 3) and their derived methyl esters (Figure 5). The reaction was carried out at a catalyst (NaOH) concentration of 1 wt% and a temperature of 65 °C for 30 min. The results are represented in Figure 6. The results show that increasing methanol to oil molar ratio from 5:1 to 6:1 increased the yield of biodiesel production. It is seen (Figure 6), that percentage of yield of biodiesel reduces at 7:1 oil to methanol molar ratio. However, oil to methanol molar ratio of 6:1 gave the optimum yield of biodiesel among them. The reaction yield was 97.6 % for 6:1 of oil to methanol molar ratio.

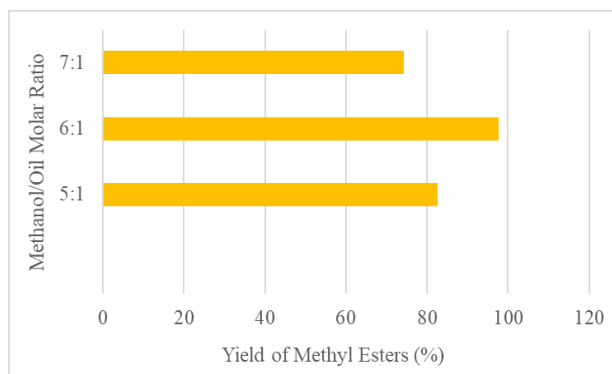


Figure 4. Effect of catalyst concentration on biodiesel yield



Figure 5. Methyl esters of cottonseed oil at varied molar ratios of methanol to oil

III.3. Comparison of the yield with earlier work

Yield is defined as the ratio of methyl esters produced to initial oil weight. The influence of solvent type, solvent-oil molar ratio, reaction temperature, and catalyst concentration on the yield of biodiesel from cottonseed oil are demonstrated by Table 1. The highest yield (98.9 wt. %) was achieved in this study using methanol at a 6:1 molar ratio, 65 °C, and 1% NaOH catalyst. This outperformed previous studies, including Rashid et al. [12], who reported a 96.9% yield using sodium methoxide (0.75%) under similar conditions, and Fan et al. [17], who observed a 92.72% yield with 1% NaOH at 55 °C.

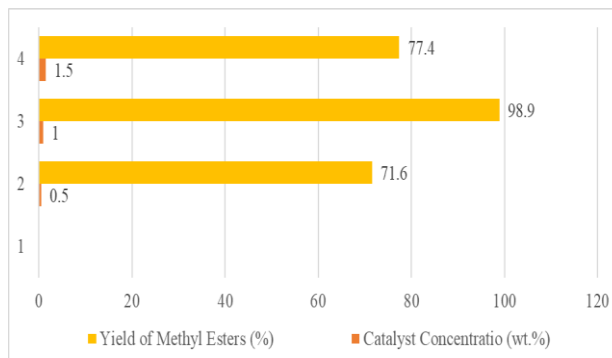


Figure 6. Effect of methanol to oil molar ratio on biodiesel yield

The molar ratio of alcohol to oil significantly impacted biodiesel yield. Increasing the methanol-to-oil ratio from 5:1 to 6:1 in this study improved the yield from 82.7% to 97.6%, while a further increase to 7:1 reduced the yield to 74.3%, likely due to excessive alcohol diluting the reaction mixture. Similar trends were observed by Patil et al. [19], where a higher ethanol-to-oil ratio (9:1) enhanced yield compared to 3:1. However, excessively high ratios may hinder separation efficiency.

Further, catalyst concentration played a critical role, with 1% NaOH yielding optimal results (98.9%). Deviations from this concentration—either lower (0.5%) or higher (1.5%)—resulted in reduced yields (71.6% and 77.4%, respectively). Fan et al. [17] also noted a sharp decline in yield (10%) when NaOH was increased to 2%, likely due to soap formation. The absence of catalyst (0% NaOH) resulted in negligible conversion, emphasizing its necessity for transesterification.

Temperature variations influenced reaction efficiency, with 55–65 °C being optimal. Higher temperatures (75 °C) reduced yields, possibly due to methanol evaporation [17]. Nnaenezie [20] reported an 85% yield with ethanol at 45 °C, suggesting solvent-dependent temperature optima. Moreover, this study highlights methanol's superiority over ethanol and butanol in biodiesel production, attributed to its higher reactivity and lower steric hindrance. The findings align with Rashid et al. [12] and Moawia et al. [21], who achieved high yields (96.9% and 88.6%, respectively) using methanol with specialized catalysts.

Table 1. Comparison of this study with other studies in terms of the Yield of Biodiesel at different operating conditions

Solvent	Solvent to oil molar ratio	Reaction Temperature (°C)	Catalyst (wt. %)	Yield of Biodiesel (wt. %)	Reference
Ethanol	3:1	65	NaOH (1 %)	58.33	Patil et al., [19]
	6:1			71	
	9:1			75	
Methanol	2:1	55	NaOH (1 %)	56.48	Fan et al., [17]
	6:1	35	NaOH (1 %)	89.13	
	6:1	55	NaOH (0 %)	0	
	6:1	55	NaOH (1 %)	92.72	
	6:1	55	NaOH (1 %)	89.36	
	6:1	55	NaOH (2 %)	10	
	6:1	75	NaOH (1 %)	82.04	
	10:1	55	NaOH (1 %)	87.61	
Methanol	7:1	55	NaOH (1 %)	82.5	Nnaenezie [20]
Ethanol	18:1	45	NaOH (1 %)	85	
Butanol	19:1	40	NaOH (0.5%)	75	
Methanol	1:33	60	Alkaline biopolymer (2.5 %)	88.6	Moawia et al., [21]
Methanol	6:1	65	Sodium methoxide (0.75 %)	96.9	Rashid et al., [12]
Methanol	5:1	65	NaOH (1 %)	82.7	This study
	6:1			97.6	
	7:1			74.3	
Methanol	6:1	65	NaOH (0.5%)	71.6	This study
			NaOH (1 %)	98.9	
			NaOH (1.5%)	77.4	

IV. Conclusions

This study demonstrates the significant impact of methanol solvent and alkali (NaOH) catalyst on biodiesel production from cottonseed oil extracted from local feedstocks from Ethiopia. By optimizing the methanol-to-oil molar ratio and catalyst concentration experimentally under constant temperature at 65 °C, the research achieved an average biodiesel yield of 98.25% with a 6:1 methanol-to-oil ratio and 1 wt.% NaOH. Deviations from these conditions—such as higher or lower catalyst concentrations, excessive alcohol ratios, or elevated temperatures—resulted in reduced efficiency, highlighting the delicate balance required for optimal transesterification. Methanol outperformed ethanol and butanol, likely due to its higher reactivity and lower steric hindrance in the reaction. Besides, catalyst concentration is crucial for better yield, while deviations led to soap formation or incomplete reactions. Further, methanol's low cost, high reactivity, and ease of recovery, along with NaOH's efficiency, were crucial in achieving high yields. In other words, moderate temperatures (55-65 °C) were seen optimal as higher temperatures (eg., 75 °C) caused methanol evaporation, reducing yield. Additionally, the methanol-to-oil ratio of 6:1 was seen as ideal, with both lower and higher ratios decreasing efficiency.

The findings underscore the importance of precise optimization in biodiesel production to enhance yield and efficiency. Utilizing local cottonseed oil not only supports sustainable energy production but also promotes the use of regional agricultural resources. This research contributes valuable insights into the biodiesel production process, highlighting the need for balanced reaction conditions. Future studies should explore alternative feedstocks and catalysts to further improve the sustainability and economic viability of biodiesel production.

Acknowledgements

Authors would like to express their deepest gratitude to HOD, School of Chemical Engineering for untiring endeavour in establishing Chemical Engineering laboratories at Jimma Institute of Technology, Jimma University. Authors extend their thankfulness to Dr. Sujana Ramesh for her consistent patronage in proof reading and editing this document.

Declaration

- The authors declare that they have no known financial or non-financial competing interests in any material discussed in this paper.
- The authors declare that this article has not been published before and is not in the process of being published in any other journal.
- The authors confirmed that the paper was free of plagiarism

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