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Effect of Time and Different Catalysts on Biodiesel Production using Co-Solvent Method

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Abstract

A major problem in converting Crude Palm Oil (CPO) into biodiesel is typically high free fatty acid (FFA) content, which leads to low conversion efficiency and excessive soap formation during processing. Therefore, this study aims to reduce FFA content and improve biodiesel yield through esterification using an H_2SO_4 catalyst and methanol, which aims to convert free fatty acids into esters, followed by transesterification with the NaOH catalyst that aims to convert triglycerides into esters. Variations in catalyst concentrations are treated in order to compare the effectiveness of acid catalysts that work to reduce acid numbers (FFA%). The results of this study are samples with NaOH catalyst (120 minutes) produced biodiesel with the lowest ALB content (0.31%), compared with samples with KOH catalyst that had higher ALB levels (1.5% - 4.5%).

Keywords: *Biodiesel, Catalyst, CO Solvent, CPO, Free Fatty Acid (FFA)*

1. Introduction

In this modern era, the energy industry is undergoing significant changes towards the development of renewable energy sources. These changes are driven by a growing awareness of the negative impacts of fossil fuel use on the environment, including greenhouse gas emissions that contribute to global climate change. Biodiesel appears to be one of the most promising solutions to address these challenges.

Biodiesel is an alternative fuel produced from renewable natural resources, such as vegetable oil and animal oil. Its natural biodegradability, non-toxicity, and freedom from sulfur and aromatic compounds make biodiesel superior to conventional diesel fuel in terms of its environmental impact. Biodiesel can be used as a substitute for diesel fuel, because the physical and chemical composition of biodiesel and diesel fuel are not much different [1] [2]. Table 1 shows that exhaust emissions from biodiesel fuel are more environmentally friendly than emissions from fossil fuels.

Table 1. Comparison of Biodiesel and Diesel Fuel [3]

Criteria	Units	Biodiesel	Solar Diesel
SO_2	ppm	0	78
CO	ppm	10	40
NO	ppm	37	64
NO_2	ppm	1	1
O_2	(%-b)	6	6,6
Total Particulates	mg/Nm^3	0,25	5,6
Benzene	mg/Nm^3	0,3	5,01
Toluene	mg/Nm^3	0,57	2,31
Xylene	mg/Nm^3	0,73	1,57
Etilbenzene	mg/Nm^3	0,3	0,73

According to Andalia and Pratiwi [4], biodiesel has various advantages, such as a mixture of 20% biodiesel and 80% petroleum diesel can be used in unmodified diesel engines, half of the biodiesel industry can use recycled fats or oils, biodiesel is non-toxic, biodiesel has a high cetane number (above 100, compared to diesel fuel which only has a cetane number of 40), and the use of biodiesel can extend the life of diesel engines because biodiesel is smoother. Table 2 below shows the quality requirements for biodiesel.

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Table 2. Biodiesel Quality Requirements [5]

Test Parameters	Units, min/max	Requirements
Density at 40°C	kg/m^3	850-890
Kinematic Viscosity at 40°C	mm^2/s (cst)	2,3-6,0
Cetane Number	min	51
Flash Point	°c, min	100
Cloud Point	°c, max	18
Residu karbon : • in the original sample; or • in 10% of the distillation residue	%mass, max	0,05 0,3
Water and Sediment	%volume, max	0,05
Free Fatty Acid	mg-koh/g, max	0,5

In general, two types of reactions are used to produce biodiesel, namely esterification and transesterification reactions, each of which has different characteristics. The combination of these two reactions aims to increase the amount of biodiesel produced from the reaction [6]. Esterification and transesterification reactions occur when alcohol (such as methanol, ethanol, isopropanol, and others) interacts with crude oil. Methanol is the most commonly used type of alcohol in this reaction because it is cheap and easily obtained. This reaction has been widely used to convert triglycerides into esters and make crude oil thicker [7].

The transesterification reaction can be carried out using either heterogeneous or homogeneous catalysts. Homogeneous catalysis generally proceeds more rapidly and requires lower catalyst loading than the heterogeneous method. However, its main drawback lies in the difficulty of recovering and reusing the catalyst, which reduces process efficiency and makes the overall production less economical [7]. KOH and NaOH are catalysts commonly used as homogeneous base catalysts in the manufacture of biodiesel [8]. Figure 1 showing how transesterification reactions works.

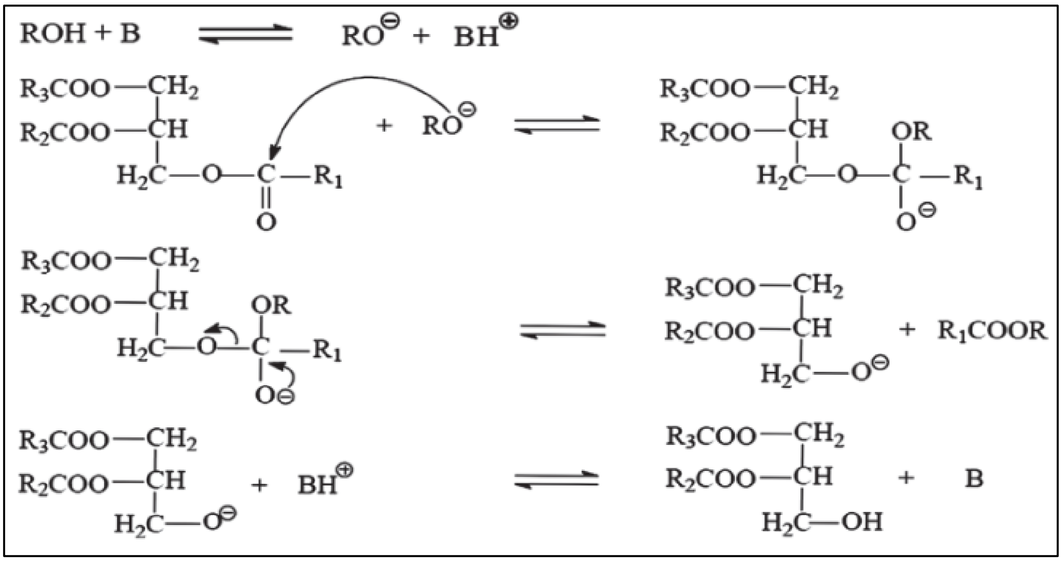

Fig 1. Base Transesterification Reaction Mechanism

A variety of edible and inedible fatty materials, including soybean oil, sunflower oil, rapeseed oil, jatropha curcas oil, cottonseed oil, waste oil, palm oil, etc., have been converted into biodiesel using co-solvents [9]. The low solubility of oil in alcohol causes the reaction to take a long time. Co-solvents are often used to increase the efficiency of the reaction between oil and alcohol [10]. N-Hexane was also chosen because it is easy to separate after the reaction due to its low boiling point (±68°C)[11].

Free fatty acids are straight-chain carboxylic acids with alcohol atoms, carbon 12 to 20. In general, these fatty acids are divided into two types, namely saturated fatty acids and unsaturated fatty acids. Factors causing relatively high free fatty acids in palm oil include palm fruits that are harvested untimely, delayed collection and transportation, and hydrolysis during the manufacturing process [12]. High free fatty acid levels can be reduced by pre-treating the raw materials and increasing the reaction temperature [13] [14].

This study aims to analyze the production of biodiesel from crude palm oil (CPO) through a combined esterification–transesterification process using methanol reactant. The objectives of this work are to evaluate the effect of free fatty acid (FFA) reduction during the pretreatment stage, to determine the performance of catalysts used in each reaction step, and to assess the role of n-hexane as a co-solvent in improving reaction efficiency. This study also seeks to characterize the biodiesel produced and compare its quality with standard biodiesel specifications.

2. Methods

In this study, there are two types of variables: fixed and variable. The fixed variables consist of CPO oil volume, methanol oil content ratio, catalyst weight percentage, and n-hexane solvent, while the variable variables consist of base catalyst and transesterification time. The base catalysts used in this study were NaOH and KOH, and the transesterification times were 1 hour, 2 hours, 4 hours, and 6 hours. This study used a method consisting of CPO preparation and analysis, triglyceride esterification and transesterification, and glycerol separation. The aim was to increase the yield of biodiesel produced. Research methods used in this study are listed below:

1. Literature Study
   Theoretical basis – theories and concepts in existing research related to the research to be conducted. The literature study examined biodiesel from several books, e-books, and related journals.
2. Data Collection
   This is a way to obtain the data needed by the author in this research, through several methods, including:
   - Observation Method, this is a method used to obtain data directly from the object being studied. This provides the necessary data.
   - Literature Research, data that we obtained through several book references in various places and sources related to the object being studied, which will later be useful for developing the results of observations and interviews.

The data listed consists of data sheets (base design) for batch reactors. In this simulation, the case studies conducted are:
1. Analyzing Biodiesel from CPO raw materials.
2. Comparing variations in NaOH and KOH catalysts in the transesterification stage with the %FFA results in Biodiesel.
3. Obtaining the relationship between Esterification and Transesterification.
4. Obtaining variations in time to increase the effectiveness of biodiesel production.

The variables in this study are as follows:
1. Fixed Variables
   Fixed variables are variables that are conditioned to be the same and assumed to be constant throughout the research process. The fixed variables in this study are catalyst concentration, methanol to oil weight ratio, and the volume of CPO used in each experiment.
2. Dependent Variables
   Dependent variables are the variables that are the objective of this study. The dependent variables in this study are FFA%, FAME, yield, pH, and temperature.
3. Independent Variables
   Independent variables are variables that influence dependent variables. In this study, the independent variables are basic

3. Result and Discussion

Table 3. Free Fatty Acid Content in Biodiesel

Sample	Catalyst	Reaction Time (minute)	FFA (%)
Sample 1	NaOH	120	0,31
Sample 2	KOH	60	2,25
Sample 3	KOH	120	4,5
Sample 4	KOH	240	1,5
Sample 5	KOH	360	1,8

The best free fatty acid value was shown in sample 1 with a free fatty acid value of 0.31%. Sample 1 had a catalyst variation in the form of sodium hydroxide (NaOH) and an operating time of 120 minutes. SNI 7182-2015 explains that the maximum free fatty acid content in biodiesel is 0.5% [5]. High free fatty acid values can make biodiesel corrosive and cause scaling when applied to engines [15]. High free fatty acid values were observed in samples 2 to 5, with free fatty acid values ranging from 1.5% to 4.5%. These samples underwent transesterification with potassium hydroxide (KOH) catalyst for 60 minutes, 120 minutes, 180 minutes, and 240 minutes. High free fatty acid values can cause saponification reactions during transesterification, which can prevent the transesterification reaction from proceeding optimally [16].

Table 4. Yield of Biodiesel

Sample	Catalyst	Reaction time (minute)	Volume (mL)	Weight (gram)
Sample 1	NaOH	120	114	92
Sample 2	KOH	60	163	130,6
Sample 3	KOH	120	164	135,5
Sample 4	KOH	240	155	125,5
Sample 5	KOH	360	188	150

Biodiesel with a reaction time above 120 minutes showed fewer results compared to those below 120 minutes, which is in line with the theory explained by Rahkadima [17], where the transesterification reaction is reversible, so that reaction times exceeding the optimum limit will cause the reaction to reverse, resulting in the formation of biodiesel that can be broken down again. Sample 5 experienced an increase due to suboptimal stirring during the reaction. The stirrer used in sample 5 for 360 minutes was smaller than those used in the other samples. More optimal mixing can increase biodiesel conversion rates [18].

Biodiesel with Potassium Hydroxide (KOH) catalyst produces a greater volume than Sodium Hydroxide (NaOH) catalyst. The greater volume is due to KOH having higher solubility in methanol than NaOH, which allows for more efficient methoxide formation and accelerates the transesterification reaction [19].

4. Conclusion

Samples with NaOH catalyst (120 minutes) produced biodiesel with the lowest ALB content (0.31%), in accordance with SNI 7182:2015 standard which sets the maximum ALB limit in biodiesel at 0.5%. Samples with KOH catalyst had higher ALB levels (1.5% - 4.5%), especially at a reaction time of 120 minutes. This indicates that the use of KOH in transesterification without prior esterification can cause saponification reactions, which inhibit the optimal formation of biodiesel. Biodiesel with a reaction time above 120 minutes showed fewer results than those below 120 minutes, because the transesterification reaction is reversible, so that a reverse reaction can occur when the reaction time exceeds the optimal limit.

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