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The Duration of Sodium Methoxide Catalyst Impregnation in Methanol and Its Impact on the Reduction Monoglyceride Content in Biodiesel Production

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ABSTRACT

Biodiesel is one of the most promising alternatives to replace conventional fuel products, specifically diesel. The increasing use of biodiesel is currently a focus of many researchers aiming to improve its quality, one of which involves reducing the monoglyceride content. High monoglyceride content can cumulatively affect the performance of diesel engines. Several techniques are used to reduce the monoglyceride content in biodiesel, one of which is maximizing the transesterification reaction. This study focuses on the preparation stage before the transesterification reaction, where the treatment involves variations in the impregnation of sodium methoxide ($NaOCH_3$) in methanol (CH_3OH). The impregnation durations tested were 0, 10, 20, 30, and 40 minutes during chemical mixing. The transesterification process was then carried out at reaction temperatures of 62°C and 64°C. The results indicate that the duration of impregnation between sodium methoxide ($NaOCH_3$) and methanol (CH_3OH) affects the reduction of the monoglyceride content. The optimal condition identified in this study was an impregnation duration of 20 minutes at a reaction temperature of 62°C, which resulted in a monoglyceride content of 0.3629%.

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1. INTRODUCTION

In recent years, many countries have been concerned about the global energy situation, which still depends on the demand for fossil fuels. The declining availability of fossil fuels has prompted countries to seek

alternative energy sources as substitutes [1]. One viable alternative to replace fossil fuels is biodiesel, which is considered a promising option. The future potential of biodiesel is significant due to its renewable nature, safe and non-toxic production methods, and lower emissions compared to conventional fuels [2]. Biodiesel development is rapidly increasing, particularly in Indonesia. Indonesia is a country with significant biodiesel usage, supported by its abundant natural resources. This positions Indonesia as a hub for bioenergy, both locally and globally [3].

Biodiesel production in Indonesia has increased every year due to the blending rate (B) policy between biodiesel and conventional diesel fuel. In 2009, production was 190 thousand kiloliters (KL) at a B-2.5 blend, while in 2023, it reached 13.15 million kiloliters (KL) at a B-35 blend. Biodiesel production in Indonesia has followed the growing consumption of biodiesel. In 2009, biodiesel consumption was 119 thousand kiloliters (KL), and it continued to increase, reaching 12.24 million kiloliters (KL) in 2023 [4]. The potential of biodiesel presents a significant opportunity for Indonesia to achieve energy independence and reduce reliance on conventional fuels.

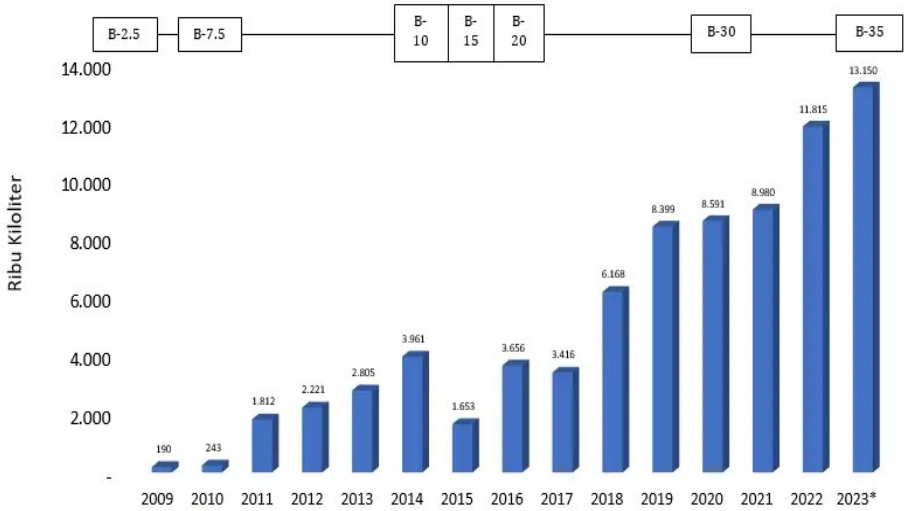


Figure 1. Graph of Biodiesel Production in Indonesia [4]

Biodiesel production in Indonesia has kept pace with the growing consumption of biodiesel. In 2009, biodiesel consumption was 119 thousand kiloliters (KL), and it continued to rise, reaching 12.24 million kiloliters (KL) in 2023 [4]. The potential of biodiesel presents a significant opportunity for Indonesia to achieve energy independence and reduce reliance on conventional fuels. Biodiesel can be produced from vegetable oils with a lower free fatty acid (FFA) content, such as Refined Bleaching Palm Oil (RBDPO). It is generally produced through a transesterification process using a base catalyst (alkali) [5]. Biodiesel is a mixture of alkyl esters produced from fatty acids and used as a substitute for conventional diesel fuel. It can be used in its pure form or blended with diesel, as it mixes well with it. The biodiesel mixture is referred to as Bxx, where 'xx' indicates the proportion of biodiesel to diesel. In biodiesel production, raw materials in the form of triglycerides are required, which can be sourced from both vegetable oils and animal fats. Triglycerides are compounds formed from glycerol molecules bonded with three molecules of either saturated or unsaturated fatty acids [6]. One example of a raw material commonly used in the biodiesel industry is refined, bleached, and deodorized palm oil (RBDPO). This raw material is primarily favored due to its abundant availability [7].

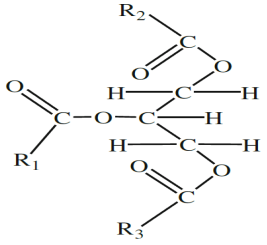


Figure 2. Chemical Structure of Triglycerides [8]

In the biodiesel production process, alcohol is required to break the mono-alkyl ester bond of long-chain fatty acids. The most commonly used alcohol for this reaction is methanol (CH_3OH), which results in the formation of methyl ester. Methanol is preferred over other alcohols due to its higher reactivity, owing to its short carbon chain [9]. The biodiesel production process using a transesterification reaction requires a catalyst to accelerate the attainment of activation energy, allowing the reaction to occur more quickly [10]. Catalysts commonly used in the biodiesel industry are classified as homogeneous catalysts, where the catalyst phase is the same as the reactant phase during the reaction. Homogeneous catalysts are still considered the preferred choice because they are more reactive, operate at relatively low temperatures, and require less time to achieve a complete reaction [11]. The amount of catalyst used in the reaction is generally smaller compared to heterogeneous catalysts. Despite their advantages, homogeneous catalysts have some disadvantages, such as sensitivity to water content and free fatty acids (FFAs) in raw materials, which can lead to saponification as a side reaction. Transesterification reactions typically use homogeneous base catalysts (alkali), such as sodium hydroxide (NaOH), potassium hydroxide (KOH), potassium methoxide ($KOCH_3$), and sodium methoxide ($NaOCH_3$). Sodium-based catalysts are preferred over potassium-based catalysts due to their better solubility in methanol and the higher yields they produce [12].

The use of homogeneous catalysts remains the primary choice in the biodiesel industry. The most commonly used homogeneous base catalyst is sodium methoxide, due to its highly active properties, which facilitate better process separation after the reaction. This catalyst is widely utilized in the Indonesian biodiesel industry [13]. Sodium methoxide is produced through two methods, one of which involves reacting sodium metal with methanol. However, this production process can be quite dangerous. An alternative method is to react sodium hydroxide with methanol, with water as a byproduct [14]. The presence of water in sodium methoxide can cause a reverse reaction, forming methanol and sodium hydroxide. This water content leads to the deactivation of sodium methoxide as a catalyst [15]. Catalyst deactivation will affect the transesterification reaction, leading to a reduced biodiesel yield, lower ester content, and an increased number of unreacted glycerides.

$$CH_3OH + NaOH \leftrightarrow NaOCH_3 + H_2O$$

Figure 3. Sodium Methoxide Synthesis Reaction Mechanism [16]

Sodium methoxide used as a catalyst in the biodiesel industry has an alkalinity of 25-30% in methanol. The synthesis of sodium methoxide typically uses sodium hydroxide as a precursor on an industrial scale due to its low cost and low toxicity. Figure 3 illustrates the exothermic reversible reaction involved in the synthesis of sodium methoxide. The production of sodium methoxide using sodium hydroxide has a disadvantage, as it produces water, which can lead to catalyst deactivation. Several factors, such as water, free fatty acids (FFAs), or peroxides, can cause the deactivation of sodium methoxide. The water content in the catalyst is a major issue, as stoichiometrically, 0.1% of the catalyst is deactivated by just 0.033% water [15].

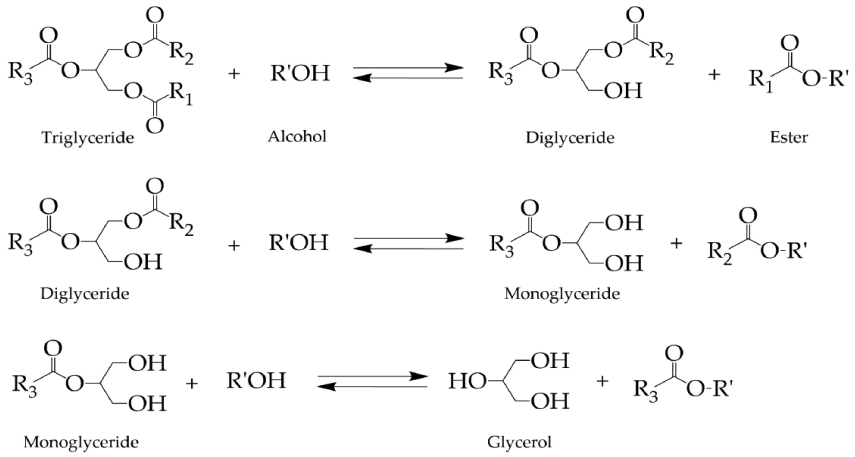


Figure 4. Mechanism of Transesterification Reaction of Triglycerides with Alcohol [17]

The transesterification reaction involves the reaction of triglycerides with alcohol using a base catalyst to produce methyl esters and glycerol. This reaction occurs in several stages, where the triglycerides in fatty acids are first converted into diglycerides, then into monoglycerides, and ultimately into ester and glycerol molecules. The stoichiometric ratio of methanol to triglycerides in this reaction is 3:1. However, in practice, this ratio is increased to 6:1 to ensure that the reaction equilibrium favors the production of the desired product [13]. In the transesterification reaction using an alkali catalyst, triglycerides and alcohol must be in anhydrous conditions to prevent the partial occurrence of saponification, which produces soap. The presence of soap consumes the catalyst, reduces its effectiveness, increases viscosity, promotes gel formation, and makes it difficult to separate glycerol at the end of the reaction [21]. The transesterification process is influenced by several factors such as transesterification temperature where the transesterification reaction is an endothermic reaction. The reaction requires heat with a temperature that should not be too low or too high where the optimal temperature needed to change triglycerides is in the range of 60-70 ^{o}C [17].

One of the main focuses in improving biodiesel quality today is reducing its glyceride content. Glycerides are considered impurities that can negatively affect the performance of diesel engines. In particular, monoglycerides are highly undesirable because their accumulation can lead to the formation of deposits in the engine [18]. Improving quality by reducing the monoglyceride content in biodiesel can be seen through the blending rate (B) policy, where in 2009 (B-2.5) the monoglyceride content was 0.80%, and in 2025 (B40), it is expected to be reduced to a maximum of 0.50% [19]. Research conducted by M Fuad et al (2022) [20] used a distillation technique with 1 and 30 column plates to reduce the monoglyceride content in biodiesel. The results obtained were 0.17% wt and 0.29% wt, respectively. However, the distillation process for reducing the monoglyceride content requires high temperatures (240-325°C), which demands significant energy. The optimum transesterification reaction process presents an alternative for reducing the glyceride content in biodiesel. Continuous reduction of the monoglyceride content is essential for obtaining high-quality biodiesel products. This forms the basis for researchers to investigate how the impregnation time of sodium methoxide in methanol influences the reduction of monoglyceride content in biodiesel products.

2. RESEARCH METHOD

This research was conducted at PT. XYZ, a company located in Riau, Indonesia, engaged in biodiesel processing. The study aims to demonstrate that the duration of sodium methoxide ($NaOCH_3$) and methanol (CH_3OH) impregnation before reacting with Refined Bleaching Palm Oil (RBDPO) can reduce the monoglyceride content in biodiesel products. The materials required for this study include RBDPO, produced by the company itself, with the following quality specifications: FFA 0.082%, moisture and impurities 0.08%, color 2.2/22.0 R/Y; methanol (CH3OH) with a moisture content of 0.1%; sodium methoxide catalyst (NaOCH3) with moisture content of 0.2% and alkalinity of 30%; hydrochloric acid (HCl) 3.0%; and process water (demineralized water). Additionally, various equipment is needed for biodiesel production and analysis, including a three-neck boiling flask, magnetic stirrer, reflux condenser, hot plate stirrer, erlenmeyer flask, beaker glass, separating funnel, whatman filter paper, thermometer, viscometer, pycnometer, vacuum dryer, and gas chromatography (GC) equipment.

This research began with the impregnation process of the sodium methoxide catalyst ($NaOCH_3$) and methanol (CH_3OH) in a chemical mixture, with varying impregnation duration of 0, 10, 20, 30, and 40 minutes. Additionally, the reaction was conducted at temperatures of 62°C and 64°C. The sodium methoxide catalyst ($NaOCH_3$) used was 1.8% wt, and the methanol reactant (CH_3OH) was 19.54% wt. The catalyst and reactant mixture were then proportionally introduced into the first and second reactors, where they reacted with RBDPO. The chemical composition in the first reactor accounted for 60%, while the second reactor contained 40% of the total reactants and catalysts used.

A total of 250 grams of RBDPO will be placed into the first reactor, where it is then mixed with the catalyst and methanol, comprising 60% of the total amount used in the chemical impregnation process. In the first reactor, the reaction will be carried out for 2 hours at a temperature of 62°C, with stirring at 350 rpm using a magnetic stirrer. Once the reaction time is complete, the separation process will be carried out in the first separator to separate the light phase (crude FAME) from the heavy phase (glycerol). The light phase will then

be transferred to the second reactor, where 40% of the total impregnated material from the chemical mixing will be added. The transesterification reaction in the second reactor will proceed for 2 hours, maintaining a reaction temperature of 62°C and stirring at 350 rpm using a magnetic stirrer. After the reaction time is complete, the separation process will be continued in the second separator to obtain the light phase (crude FAME) and the heavy phase (glycerol).

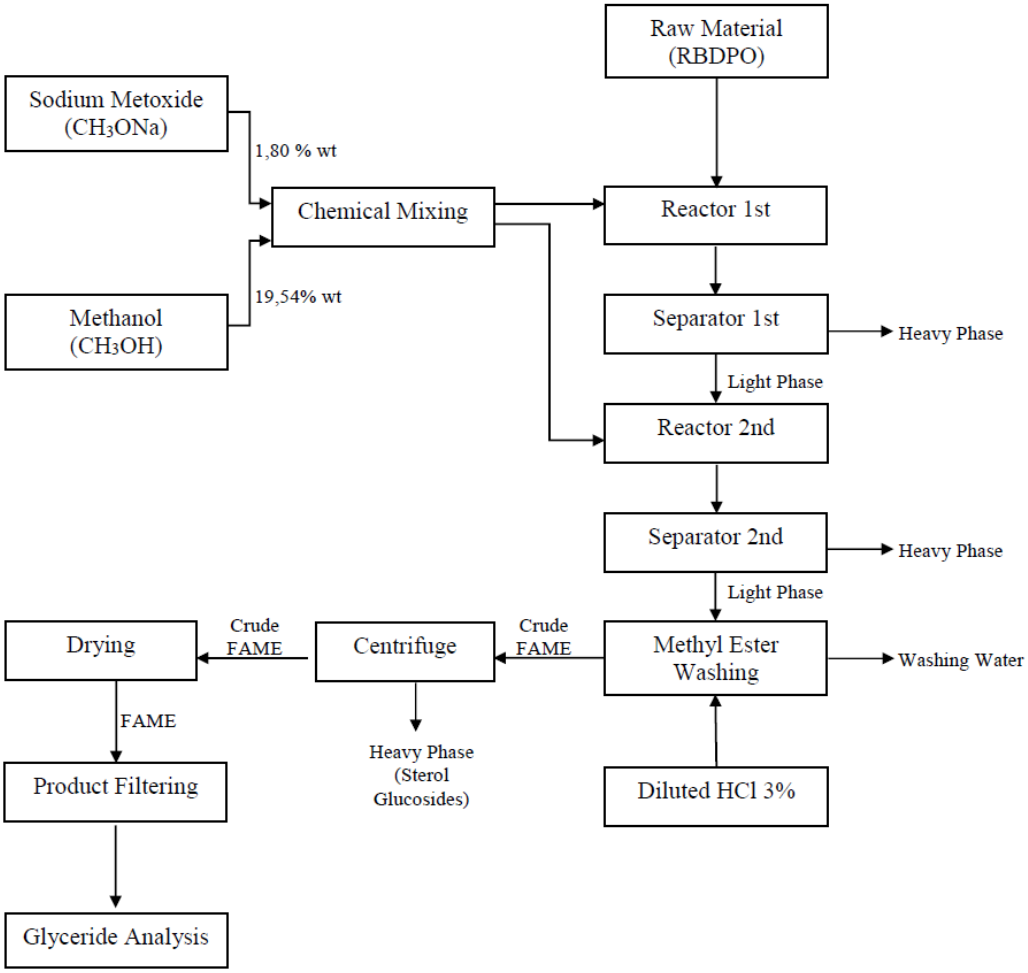


Figure 5. Flowchart of Biodiesel Production Process (FAME) Using Transesterification Method

Crude FAME from the second separator is then treated with 3.0% HCl to adjust the pH to 2.0-3.0. This step neutralizes the sodium methoxide catalyst ($NaOCH_3$) and the methanol reactant (CH_3OH) still present in the crude FAME. The addition of HCl also helps prevent emulsion formation and facilitates the separation of impurities such as sterol glucosides during the washing process with demineralized water. The washing process continues until the pH reaches 6.0-7.0. The washed crude FAME is then placed in a centrifuge, rotating at 6000 rpm, to separate it from impurities and excess water. After centrifugation, the crude FAME is transferred to a vacuum dryer, and the dried product is filtered using Whatman filter paper. The final product, biodiesel (FAME), will then undergo glyceride analysis using gas chromatography (GC). The specifications of biodiesel are presented in the Table 1.

Table 1. Biodiesel Fuel Specifications and Testing Methods [19]

No	Characteristics	Unit	B30		B40		Test Method
			Min	Max	Min	Max	
1	Density at 40 °C	Kg/m^3	850	890	850	890	SNI 7182:2015, ASTM D4052
2	Viscosity at 40 °C	mm^2/s	2.3	6.0	2.3	6.0	SNI 7182:2015,

							ASTM D445
3	Cetane Number		51	-	51	-	SNI 7182:2015, ASTM D613
4	Flash Point	°C	130	-	130	-	SNI 7182:2015, ASTM D93
5	Copper Strip Corrosion		Class 1			Class 1	SNI 7182:2015, ASTM D130
6	Carbon Residue	%m	-	0.3	-	0.3	SNI 7182:2015, ASTM D4530
7	Distillation (90% evaporation vol)	°C	-	360	-	360	SNI 7182:2015, ASTM D1160
8	Sulphated Ash Content	%m	-	0.02	-	0.02	SNI 7182:2015, ASTM D874
9	Sulfur	mg/kg	-	10	-	10	SNI 7182:2015, ASTM D5453
10	Phosphorus	mg/kg	-	4	-	4	SNI 7182:2015, AOCS Ca 12-55
11	Acid Value	mg KOH/g	-	0.4	-	0.4	SNI 7182:2015, ASTM D664
12	Free Glycerol	%m	-	0.02	-	0.02	SNI 7182:2015, ASTM D6584
13	Total Glycerol	%m	-	0.24	-	0.24	SNI 7182:2015, ASTM D6584
14	Ester Content	%m	96.5	-	96.5	-	SNI 7182:2015
15	Iodine Value	%m	-	115	-	115	SNI 7182:2015, AOCS Cd 1-25
16	Ranchimat Oxidation Stability	minutes	-	660	-	720	SNI 7182:2015, EN 15751
17	Monoglycerides	%m	-	0.525	-	0.500	SNI 7182:2015, ASTM D6584
18	Color	-	-	3	-	3	ASTM D1500
19	Water Content	ppm, max	-	340	-	320	ASTM D6304
20	CFPP (Cold Filter Plugging Point)	°C	-	15	-	15	D 6371
21	Metal I (Na+K)	mg/kg	-	5	-	5	EN 14108, EN 14109, EN 14538, AAS, ICPS
22	Metal II (Ca+Mg)	mg/kg	-	5	-	5	EN 14538, AAS, ICPS
23	Total contaminants	mg/liter	-	20	-	20	ASTM D6217

3. RESULTS AND DISCUSSION

3.1 Characteristics of Biodiesel

The biodiesel produced in this study was subsequently analyzed to assess whether its specifications met the established standards. Table 2 presents several analyses determining the characteristics of the produced biodiesel, as follows:

Table 2. Biodiesel Analysis Results Specifications

No	Characteristics	Unit	Testing Results	Evaluation of Results
1	Density at 40 ° C	Kg/ m^3	861	Accepted
2	Viscosity at 40 ° C	m m^2 /s	4.55	Accepted
5	Cloud Point	° C	15	Accepted
8	Water and Sediment	%v	0.002	Accepted
13	Acid Value	mg KOH/g	0.321	Accepted
14	Free Glycerol	%m	0.00001	Accepted
15	Total Glycerol	%m	0.1138	Accepted
16	Ester Content	%m	98.33	Accepted
18	Ranchimat Oxidation Stability	minutes	890	Accepted
19	Monoglycerides	%m	0.3629	Accepted
20	Color	-	1.5	Accepted
21	Water Content	ppm, max	211	Accepted

Additionally, the glyceride components of the biodiesel were analyzed using gas chromatography. This analysis identifies the types of monoglycerides, diglycerides, and triglycerides present in the biodiesel, along with the quantity of each component. Figure 6 presents the results of the glyceride analysis of the produced biodiesel, as follows:

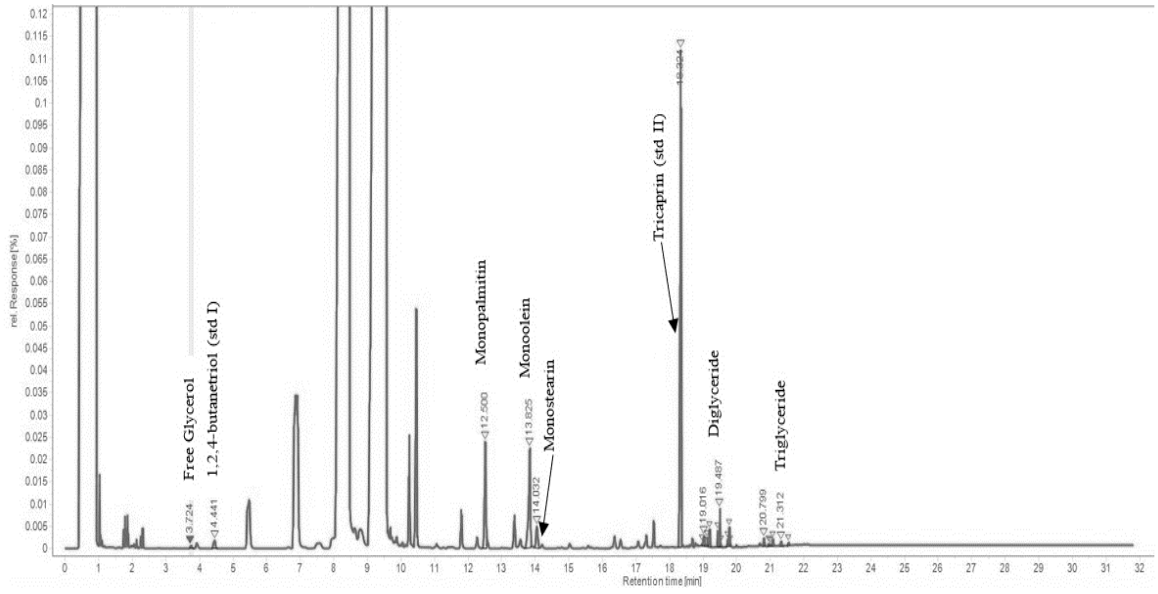


Figure 6. Results of the Biodiesel Component Analysis by Gas Chromatography (GC)

The results of the biodiesel component analysis using gas chromatography reveal several components, including glycerin, monoglycerides, diglycerides, and triglycerides. Additionally, the data also show the quantity of each component present in the biodiesel. Table 3 presents the components at each retention time, as follows:

Table 3. Results of Biodiesel Component Analysis Based on Standard Retention Time

Component	Retention time (minutes)
Free Glycerol	3.724
1,2,4-butanetriol (std I)	4.441
Monopalmitin	12.500
Monoolein	13.825

Monostearin	14.032
Tricaprin (std II)	18.324
Diglycerides	19.487
Triglycerides	21.312

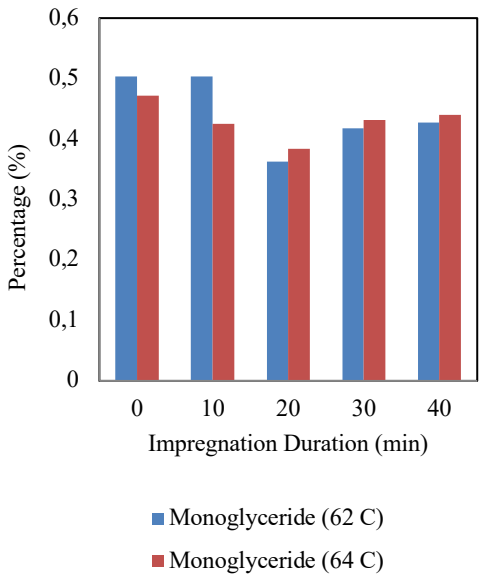
Along with displaying the components at specific retention times, the analysis also indicates the quantity of each component, as shown in Table 4 below.

Table 4. Results of the Glyceride Component Percentage Analysis in Biodiesel

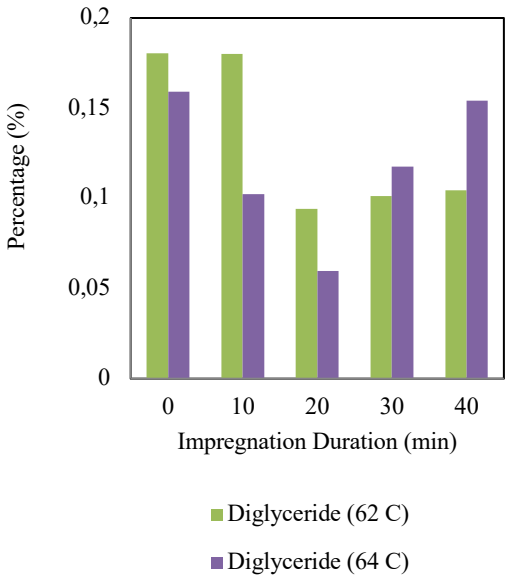
Temperature Reaction	Duration Impregnation	Glyceride Component		
		Monoglycerides	Diglycerides	Triglycerides
62 ° C	0	0.5039	0.1803	0.0755
	10	0.5030	0.1800	0.0810
	20	0.3629	0.0942	0.0723
	30	0.4175	0.1014	0.0670
	40	0.4277	0.1045	0.0678
64 ° C	0	0.4715	0.159	0.0878
	10	0.4253	0.1023	0.0630
	20	0.3839	0.0596	0.0479
	30	0.4315	0.1177	0.0710
	40	0.4401	0.1542	0.0511

3.2 Effect of Sodium Methoxide ($NaOCH_3$) Impregnation Duration in Methanol (CH_3OH) on the Glyceride Components of Biodiesel

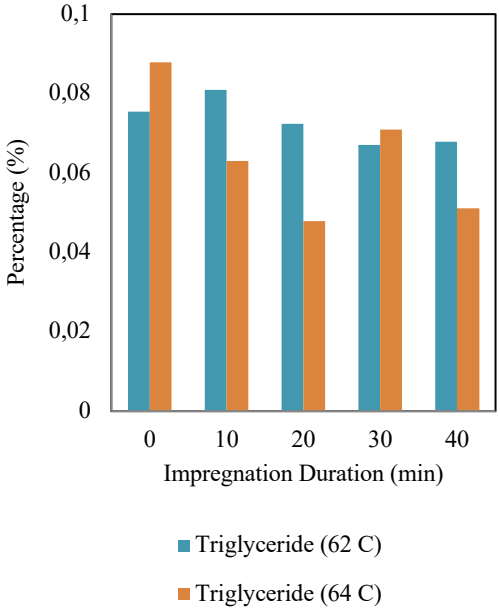
The study's results demonstrate the effect of the sodium methoxide ($NaOCH_3$) impregnation duration in methanol (CH_3OH) on the glyceride components in biodiesel. The impregnation duration was divided into five treatments: 0, 10, 20, 30, and 40 minutes, followed by reactions at temperatures of 62°C and 64°C.



(A)



(B)



(C)

Figure 7. Effect of Impregnation Duration on Component (A) Monoglyceride (B) Diglyceride (C) Triglyceride Biodiesel

Figure 7 illustrates the monoglyceride, diglyceride, and triglyceride components at each impregnation duration, specifically from 0 to 20 minutes, for reaction temperatures of 62°C and 64°C, where these components predominantly decreased in percentage in biodiesel. However, with an impregnation duration of 20 to 40 minutes at reaction temperatures of 62°C and 64°C, the monoglyceride, diglyceride, and triglyceride components in biodiesel showed a predominant increase. The optimal impregnation duration in this study was found to be 20 minutes. At a reaction temperature of 62°C, the monoglyceride component was 0.3629%, the diglyceride component was 0.0942%, and the triglyceride component was 0.0723%. At a reaction temperature of 64°C, the monoglyceride component was 0.3839%, the diglyceride component was 0.0596%, and the triglyceride component was 0.0479%. The monoglyceride standard provided by K. EBTKE (2024) [19] The percentage is 0.50%, indicating that the results obtained under the optimal conditions with an impregnation duration of 20 minutes at reaction temperatures of 62°C and 64°C have met the biodiesel specifications for a B40 blending ratio. The impregnation process of sodium methoxide ($NaOCH_3$) in methanol (CH_3OH) aims to increase the concentration of sodium methoxide ($NaOCH_3$), thereby enhancing the efficiency of the transesterification reaction. However, the use of technical-grade sodium methoxide ($NaOCH_3$) in the biodiesel industry has certain disadvantages, particularly due to its water content. This water can cause side reactions that affect both the catalyst compound and the transesterification process. The presence of water in sodium methoxide ($NaOCH_3$) leads to a reversible reaction that produces methanol (CH_3OH) and sodium hydroxide ($NaOH$). This water content causes catalyst deactivation, reducing the activity of sodium methoxide ($NaOCH_3$) as a catalyst when introduced into the reactor [15]. The use of larger amounts of methanol (CH_3OH) in the impregnation of sodium methoxide ($NaOCH_3$) helps to regenerate sodium hydroxide ($NaOH$) and maintain the reaction equilibrium, promoting the formation of sodium methoxide ($NaOCH_3$). This aligns with Le Chatelier's principle, which states that in an equilibrium reaction, the addition of a reactant will shift the equilibrium toward the product side [22]. The characteristics of the reaction mechanism for the formation of sodium methoxide ($NaOCH_3$) include exothermic reactions, where the reaction releases heat to the environment, and it is also a reversible reaction.

The impregnation duration of sodium methoxide ($NaOCH_3$) in methanol (CH_3OH) for up to 20 minutes yielded optimal results. A longer impregnation process allows more time for methanol (CH_3OH) and sodium hydroxide ($NaOH$) to react, forming sodium methoxide ($NaOCH_3$) and water. However, when impregnation exceeds 20 minutes, the glyceride content increases. This occurs because the extended reaction

time enables water and sodium methoxide ($NaOCH_3$) to react again, producing methanol (CH_3OH) and sodium hydroxide (NaOH)

The reaction temperature also affects the transesterification process. Since the transesterification reaction is endothermic, it requires heat to accelerate the reaction. The lower the reaction temperature, the slower the reaction rate, due to the diffusion barrier between the reactants [23]. High reaction temperatures should also be avoided, as they can slow down the reaction rate due to the volatile nature of methanol (CH_3OH). Methanol has an evaporation temperature of 63-64°C, which can cause the formation of a bubble phase (gas) during the transesterification reaction. This phase difference between the reactants complicates the process of breaking the alkyl group from fatty acids (triglycerides), as it becomes difficult to achieve homogeneity. In general, higher reaction temperatures are more effective in increasing the activity of the sodium methoxide ($NaOCH_3$) catalyst, as they accelerate molecular movement, leading to more frequent collisions and a faster reaction [24].

The results of the research showed that the quality of the glyceride produced at a temperature of 62°C was better than at 64°C. The optimal result in this study was achieved with an impregnation duration of 20 minutes at a reaction temperature of 62°C, which produced a better monoglyceride component compared to the reaction temperature of 64°C. This is because the higher temperature causes a phase change in methanol (CH_3OH), reducing the number of reactants available to react with RBDPO, which in turn affects the equilibrium of the chemical reaction.

4. CONCLUSION

This study shows that the impregnation duration of sodium methoxide ($NaOCH_3$) in methanol (CH_3OH) influences the reduction of monoglyceride components in biodiesel products. The optimal impregnation duration was found to be 20 minutes, with a transesterification reaction temperature of 62°C, resulting in a monoglyceride component of 0.3629%. The research findings align with the standard results provided.

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