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THE EFFECT OF ACIDIC AND ALKALINE CONDITIONS ON FREE FATTY ACID CONTENT DURING THE TRANSESTERIFICATION OF WASTE COOKING OIL

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ABSTRACT

Waste cooking oil is a waste material with significant potential for utilization as a biodiesel feedstock; however, its high free fatty acid (FFA) content can reduce the efficiency of the transesterification process. This study aimed to investigate the effect of acidic and alkaline conditions on the presence of free fatty acids during the transesterification of waste cooking oil using sodium hydroxide (NaOH) and potassium hydroxide (KOH) catalysts. Prior to the transesterification process, the waste cooking oil was filtered and heated at 60°C for 30 minutes to reduce its moisture content. Transesterification was carried out using methanol at a methanol-to-oil molar ratio of 6:1 and a catalyst concentration of approximately 0.25% of the oil weight. The reaction pH was varied at 6, 7, 8, 9, and 10, while the reaction temperature was maintained at 60°C for 1 hour. FFA content was analyzed before and after transesterification using an acid-base titration method with 0.1 N NaOH solution. The results showed that the initial FFA content of the waste cooking oil was 5.20%. The use of the NaOH catalyst resulted in final FFA contents of 3.00%, 2.55%, 1.90%, 1.75%, and 2.20% at pH 6, 7, 8, 9, and 10, respectively, corresponding to FFA reductions of 42.3%, 51.0%, 63.5%, 66.3%, and 57.7%. Meanwhile, the KOH catalyst produced final FFA contents of 2.80%, 2.35%, 1.70%, 1.60%, and 2.05%, with FFA reductions of 46.2%, 54.8%, 67.3%, 69.2%, and 60.6%, respectively. The reduction in FFA content increased with increasing pH and reached its optimum condition at pH 9 for both catalysts. At pH 10, a decrease in FFA reduction efficiency was observed, which was likely attributed to the occurrence of side reactions such as saponification. Overall, the KOH catalyst demonstrated superior performance compared to NaOH in reducing FFA content under all tested pH conditions. These findings indicate that mildly alkaline to moderately alkaline conditions, particularly at pH 9, represent the optimum conditions for reducing FFA content during the transesterification of waste cooking oil.

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INTRODUCTION

Used cooking oil is waste generated from cooking activities that can cause environmental pollution if not managed properly (Fakriah et al., 2022; Prasetyo, 2018). Disposing of used cooking oil directly into the environment can cause a decline in water and soil quality, and disrupt the balance of the ecosystem. On the other hand, used cooking oil still contains triglyceride components that can be used as an alternative raw material in biodiesel production. Utilizing this waste into biodiesel not only provides added economic value but also supports waste management efforts and the development of more environmentally friendly renewable energy (Arlofa & Rohanah, 2025; D. Ramadhani et al., 2024). Biodiesel production from used cooking oil is generally carried out through a transesterification process, namely the reaction between triglycerides and short-chain alcohols, such as methanol or ethanol, using a specific catalyst to produce methyl esters as the main product and glycerol as a byproduct (Jannah et al., 2025; Monde et al., 2022). This process is the most widely used method because it can produce biodiesel with high efficiency and relatively simple operating conditions. The success of the transesterification process is influenced by various factors, including the molar ratio of alcohol to oil, reaction temperature, reaction time, catalyst type and concentration, and the quality of the raw materials used.

In the transesterification process, one of the critical parameters determining the success of transesterification is the free fatty acid (FFA) content in used cooking oil (Andalia & Pratiwi, 2024; Haryani et al., 2025). High free fatty acid levels can lead to soap formation when using a base catalyst, ultimately reducing the yield and quality of the resulting biodiesel. One factor that plays a role in controlling the free fatty acid content during transesterification is the reaction pH (Akbar et al., 2025; Setiorini et al., 2023). Variations in pH can affect catalyst activity and the course of the transesterification reaction, thus affecting the final product, particularly the free fatty acid content (Ganisha et al., 2025; D. R. Ramadhani et al., 2025). Oils with high FFA content generally require pretreatment to reduce the acid number before transesterification using a base catalyst (Oko et al., 2021; Pratigto et al., 2019). The choice of catalyst significantly influences the transesterification process. NaOH and KOH are commonly used base catalysts because they have high catalytic activity and are relatively easy to obtain (Andalia & Pratiwi, 2024; Armah et al., 2025). However, these two catalysts have different characteristics in terms of solubility, reactivity, and tendency to form soap when reacting with free fatty acids (D. Ramadhani et al., 2024). Several studies have specifically examined the effect of the type of base catalyst on the quality of the resulting biodiesel. KOH is known to have a higher solubility in methanol than NaOH, resulting in faster and more homogeneous formation of methoxide ions as the active species in the transesterification reaction. This condition has the potential to increase the conversion of triglycerides to methyl esters and reduce the remaining FFA content in biodiesel products. Therefore, comparing the performance of these two catalysts is crucial in determining the most effective process conditions (Ganisha et al., 2025; Monde et al., 2022).

Andalia and Pratiwi (2024) compared the performance of NaOH and KOH catalysts in biodiesel production from used cooking oil. The results showed that both catalysts were capable of producing biodiesel that met quality standards, but there were differences in product characteristics influenced by the properties of each catalyst. This research is supported by Ramadhani et al. (2024), who stated that catalyst type significantly influences biodiesel yield and final product quality, particularly regarding viscosity and acidity.

Free Fatty Acid (FFA) analysis in biodiesel is generally performed through acid-base titration using standard base solutions such as NaOH or KOH (Haryani et al., 2025;

Setiorini et al., 2023), where the reaction that occurs is FFA reacting with the base to form soap and water (FFA + NaOH/KOH → soap + water). The FFA value is very important in determining the success of the biodiesel production process, because if the FFA is less than 2%, the raw material is still suitable for direct processing through a transesterification reaction using a base catalyst (NaOH/KOH), whereas if the FFA is more than 2%, it will trigger a saponification reaction or soap formation which can reduce biodiesel yield and complicate the separation process between biodiesel and glycerol (Akbar et al., 2025; Arlofa & Rohanah, 2025).

Biodiesel research from used cooking oil has been extensively conducted, focusing on optimizing the molar ratio of alcohol to oil, catalyst concentration, reaction temperature, reaction time, and biodiesel yield. Furthermore, several studies have compared the use of base catalysts such as NaOH and KOH on the efficiency of converting triglycerides to methyl esters. However, most studies still use FFA levels only as an initial parameter to determine the suitability of raw materials before the transesterification process. To date, there are still limited studies that specifically examine the effect of acidity and alkalinity conditions of the reaction system on changes in FFA levels in the transesterification process of used cooking oil. Furthermore, information regarding the effectiveness of NaOH and KOH in reducing FFA levels at various pH conditions has not been comprehensively reported. The novelty of this study lies in examining the effect of variations in reaction pH (pH 6–10) on the presence of free fatty acids during the transesterification process of used cooking oil by comparing two commonly used base catalysts, namely NaOH and KOH. This study not only evaluates the reduction in FFA levels but also determines the optimum pH conditions and the most effective catalyst in suppressing the presence of FFA after the transesterification process.

METHODS

Research Design

This study used an experimental design with two main factors, namely the type of catalyst (NaOH and KOH) and variations in reaction pH. The pH factor consists of five treatment levels, namely pH 6, pH 7, pH 8, pH 9, and pH 10. The combination of these two factors resulted in ten treatments. Each treatment was carried out once so that a total of 10 experimental data were obtained. The used cooking oil sample used in each experiment was 50 ml. The volume of methanol used was determined based on the molar ratio of methanol to oil of 6:1. The NaOH and KOH catalysts were used with a concentration of 0.25% of the oil mass.

Preparation of Ingredients

The used cooking oil used in this study was first filtered to remove impurities and solid particles. After the filtering process, the oil was heated at 60 degrees Celsius for 30 minutes to reduce the water content that could interfere with the transesterification reaction. Next, a preliminary free fatty acid (FFA) analysis was performed using an acid-base titration method to determine the quality of the raw material prior to the reaction process. Oil with a high FFA content was still used in this study to determine the effect of reaction conditions on FFA reduction during the transesterification process.

Transesterification Process

The transesterification process was carried out by mixing 50 ml of used cooking oil with 12.7 mL of methanol with a molar ratio of 6: 1 (methanol:oil). Sodium hydroxide or potassium hydroxide catalyst was added as much as 0.1125 grams (0.25% by weight of oil) according to each treatment. Variations in acidity and alkalinity conditions were adjusted to pH 6, pH 7, pH 8, pH 9, and pH 10 using buffer solutions according to the needs of the reaction system. pH measurements were carried out before the transesterification process (methanol + buffer + catalyst), the reaction mixture was put into the reactor and stirred

constantly at a temperature of 60 degrees Celsius for 1 hour. After the reaction was complete, the mixture was left to stand until two layers formed, namely biodiesel on the top and glycerol on the bottom. The biodiesel layer was then separated and washed using warm water to remove residual catalyst, methanol, and other impurities.

FFA (Free Fatty Acid) Analysis

Free fatty acid levels were analyzed before and after the transesterification process using an acid-base titration method. A 5-gram sample was weighed and dissolved in 50 mL of 96 percent alcohol. The solution was then heated to 40°C to ensure homogeneity, followed by two to three drops of phenolphthalein indicator. Next, titration was performed using 0.1 N sodium hydroxide solution until a color change occurred, indicating the endpoint of the titration. The reaction was a neutralization between the free fatty acids and the base.

$$R–COOH + NaOH \rightarrow R–COONa + H_2O$$

FFA levels are calculated and expressed as oleic acid using the equation:

$$FFA\ (\%)=(V \times N \times 28{,}2)/W$$

Description:
V = volume of NaOH (mL)
N = normality of NaOH (0,1 N)
W = sample mass (gram)
28,2 = factor for oleic acid

RESULT

This study analyzed the FFA content of biodiesel produced from the transesterification process using an acid-base titration method. In this study, the used cooking oil used had an initial free fatty acid (FFA) content of 5.20%, which is theoretically above the optimal limit for a base-catalyzed transesterification process. However, this study still conducted a direct transesterification process using NaOH and KOH catalysts without an initial esterification step. This was done to evaluate the extent to which varying reaction conditions, particularly pH, could affect the reduction of FFA content and to observe the reaction characteristics under conditions of high FFA feedstock. This approach is based on the consideration that in practical field conditions, used cooking oil is often used directly without a complex pretreatment process. Therefore, this study also aims to provide a realistic picture of the performance of base catalysts under conditions of high FFA feedstock. Although theoretically FFA levels >2% have the potential to cause saponification reactions, the results show that under certain conditions (especially pH 8–9), the transesterification process can still proceed with a significant reduction in FFA content. This indicates that optimizing reaction conditions can reduce the negative impact of high FFA on soap formation, although it cannot completely eliminate it. The measurement data obtained are presented in Table 1 and Table 2 below.

Table 1. Testing of FFA levels before and after the transesterification process using NaOH catalyst

pH Reaction	Initial FFA Content (%)	Final FFA Content of NaOH (%)	FFA reduction of NaOH (%)
6	5.20	3.00	42.3
7	5.20	2.55	51.0
8	5.20	1.90	63.5
9	5.20	1.75	66.3
10	5.20	2.20	57.7

Table 2. Testing of FFA levels before and after the transesterification process using KOH catalyst

pH Reaction	Initial FFA Content (%)	Final FFA Content of KOH (%)	FFA reduction of KOH (%)
6	5.20	2.80	46.2
7	5.20	2.35	54.8
8	5.20	1.70	67.3
9	5.20	1.60	69.2
10	5.20	2.05	60.6

Research data show that reaction pH significantly influences the reduction of Free Fatty Acid (FFA) levels during the transesterification of used cooking oil using both NaOH and KOH catalysts (Ganisha et al., 2025; Monde et al., 2022). The initial FFA content of the oil remained at 5.20%, then varied depending on the reaction pH conditions used. At pH 6, both NaOH and KOH catalysts performed less than optimally, as indicated by relatively high final FFA levels of 3.00% (NaOH) and 2.80% (KOH), respectively, and reduction percentages of 42.3% and 46.2%, respectively. This indicates that the acidic environment deactivates part of the base catalyst with FFA, thereby preventing optimal methyl ester formation (Oko et al., 2021; Pratigto et al., 2019).

As the pH increases toward neutral to slightly alkaline conditions (pH 7–8), the effectiveness of the transesterification reaction increases significantly. At pH 7, FFA reduction reaches 51.0% (NaOH) and 54.8% (KOH), while at pH 8, it increases to 63.5% (NaOH) and 67.3% (KOH), with lower final FFA levels, respectively. This indicates that in neutral to slightly alkaline conditions, the catalyst works more effectively due to the formation of methoxide ions, which play a crucial role in accelerating the transesterification reaction, thus optimizing the conversion of free fatty acids to methyl esters (FAME) (Armah et al., 2025; Jannah et al., 2025).

The optimum conditions for both catalysts were found at pH 9, where the FFA reduction reached the highest values of 66.3% for NaOH and 69.2% for KOH, with the lowest final FFA levels of 1.75% and 1.60%, respectively. This indicates that the alkaline conditions at this pH strongly support the maximal transesterification reaction. Thermodynamically, this condition indicates a lower activation energy and a shift in the reaction equilibrium toward ester formation, resulting in higher conversion (Ganisha et al., 2025; D. Ramadhani et al., 2024; Trisnaliani et al., 2018).

However, when the pH was further increased to pH 10, the effectiveness of both catalysts decreased. This was evident in the increase in the final FFA content to 2.20% (NaOH) and 2.05% (KOH), with the percentage reductions decreasing to 57.7% and 60.6%, respectively. This decrease in performance was due to the dominant side reaction, saponification, which is the formation of soap resulting from the reaction between the base catalyst and free fatty acids or triglycerides. The resulting soap can inhibit phase separation between biodiesel (FAME) and glycerol, and consume some of the catalyst, thereby reducing reaction efficiency (Akbar et al., 2025; Setiorini et al., 2023). Furthermore, the hygroscopic nature of KOH, particularly the presence of water in the system, can trigger hydrolysis reactions that regenerate FFA (Andalia & Pratiwi, 2024; Haryani et al., 2025).

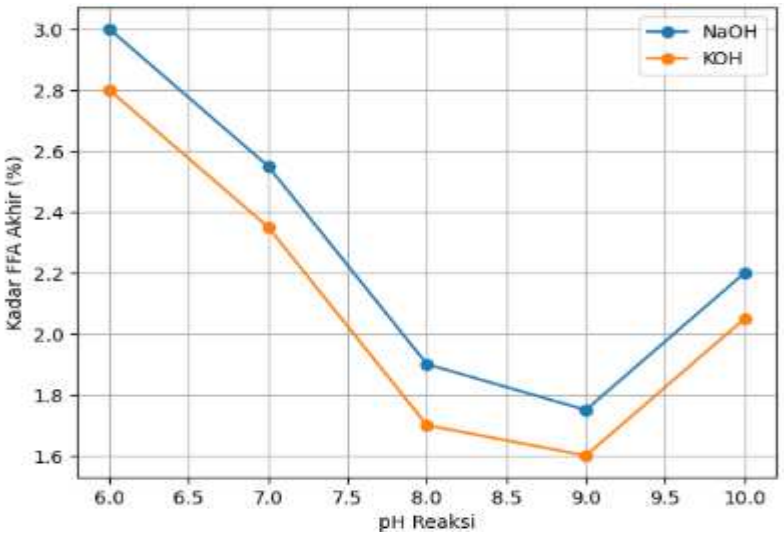

Figure 1. Graph of FFA content analysis against pH variations

Overall, the results of this study confirm that controlling the reaction pH is a key factor in the transesterification process. Both NaOH and KOH catalysts showed a similar pattern, namely increasing reaction efficiency until reaching optimum conditions at pH 9, then decreasing at too high a pH. In this case, KOH showed slightly better performance than NaOH, indicated by lower final FFA levels at each pH variation (Monde et al., 2022; D. Ramadhani et al., 2024). Thus, proper pH control, especially in the moderately alkaline range (pH 8–9), is crucial to maximize the conversion of FFA to biodiesel and minimize side reactions that can degrade product quality (Arlofa & Rohanah, 2025).

CONCLUSION

Differences in reaction pH indicate a decrease in FFA levels in the transesterification process using NaOH and KOH catalysts. Optimal conditions were obtained at pH 9, with the highest FFA reduction and the lowest final FFA levels. However, at pH levels that were too low or too high, reaction efficiency decreased. In general, KOH performed slightly better than NaOH.

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