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Effects of Reaction Temperature and Phosphoric Acid Catalyst Volume on Free Fatty Acid Reduction in Used Cooking Oil via Esterification

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Abstract: Used cooking oil contains free fatty acids (FFAs), which can increase due to hydrolysis and repeated heating, thereby limiting its further use. This study descriptively evaluated the effects of reaction temperature (40 and 60 °C) and the volume of phosphoric acid catalyst (4 and 8 mL) on the reduction of free fatty acid (FFA) through esterification with methanol. Each experimental condition was conducted once using 100 mL of pretreated used cooking oil, 20 mL of methanol, and 4 or 8 mL of 85% H_3PO_4 for 60 minutes at 500 rpm. FFA content was determined by alkalimetric titration, while FTIR was used for supporting qualitative characterization. The density and viscosity of the new oil and used oil were also compared. The initial FFA content was 2.11%. After esterification, the FFA contents were 1.33%, 0.88%, 0.70%, and 0.33% under the conditions of 4 mL–40 °C, 4 mL–60 °C, 8 mL–40 °C, and 8 mL–60 °C, respectively. The FFA reduction was accompanied by calculated conversion values of 37.02%, 58.41%, 66.73%, and 84.55%, respectively, based on the reduction in acid value. The lowest FFA content was obtained at 60 °C with 8 mL of H_3PO_4. The FTIR spectra before and after treatment showed similar C=O and C–O ester bands because the triglycerides already contained ester groups; therefore, FFA titration was used as the primary indicator of esterification progress. Within the range of conditions tested, higher temperatures and catalyst volumes were associated with greater reductions in FFA.
Keywords: used cooking oil, esterification, free fatty acid, phosphoric acid, reaction temperature

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INTRODUCTION

Cooking oil is generally composed primarily of triglycerides, along with minor components such as mono- and diglycerides, water, and free fatty acids (FFA). During repeated frying, the oil undergoes hydrolysis, oxidation, and polymerization, which can alter its physical and chemical properties, including viscosity, density, and oxidative stability (Abrante-pascual, 2024; Hajek, 2021). One parameter commonly used to describe the degree of hydrolysis and deterioration in oil quality is the FFA content. An increase in FFA indicates a rise in fatty acids that are no longer bound as triglycerides and accompanied by an increase in the oil's acidity (Ferreira et al., 2021). For comparison, SNI 7709:2019 specifies a maximum FFA content of 0.3%, expressed as palmitic acid, for palm cooking oil (Nasional, 2019).

One way to reduce the free fatty acid (FFA) content in used cooking oil is through esterification, which is a reaction between FFA and methanol that produces methyl esters and water. In this reaction, H_3PO_4 acts as an acid catalyst that facilitates the esterification reaction. Reaction temperature affects the rate of esterification, while the amount of catalyst influences the rate of the reaction. Because esterification is a reversible equilibrium reaction, the conversion of FFA is also influenced by the equilibrium position. The use of excess methanol can favor ester formation, whereas the accumulation of water as a reaction product can shift the equilibrium toward the reactants and reduce conversion. Therefore, within an appropriate operating range, increasing the reaction temperature and catalyst amount can enhance FFA conversion, although the response may not always be linear (Putri et al., 2022; Supeno et al., 2023). Phosphoric acid (H_3PO_4) is one of the acids that can act as a homogeneous catalyst in oil-based reactions. H_3PO_4 has been reported as a catalyst in the transesterification of soybean oil(Encinar Maria et al., 2022), while super phosphoric acid, which is a more concentrated phosphoric acid system, has been used in the esterification of

palm fatty acid distillate, with the conversion being influenced by reaction temperature, methanol ratio, and catalyst amount (Metre & Nath, 2015).

Phosphoric acid (H_3PO_4) was selected as the acid catalyst in this study because phosphoric acid is among the inorganic acids used for acid-catalyzed processing of used cooking oil, particularly for feedstocks with high free fatty acid (FFA) content (Kumar et al., 2025). An 85% H_3PO_4 solution was used as the catalyst in this study. Catalyst volumes of 4 and 8 mL were selected to investigate the effect of catalyst amount on FFA reduction, while reaction temperatures of 40 and 60 °C were selected to evaluate the effect of temperature on the esterification process. Increasing the reaction temperature can enhance the esterification rate, while increasing the amount of acid catalyst can promote the reaction by increasing the availability of acidic catalytic species (Supeno et al., 2023). However, because esterification is a reversible equilibrium reaction, the effects of temperature and catalyst amount may not increase linearly under all operating conditions. Therefore, this study aims to descriptively evaluate the effects of reaction temperatures of 40 and 60 °C and H_3PO_4 catalyst volumes of 4 and 8 mL on FFA reduction. These changes were further supported by measurements of oil density and viscosity before treatment, as well as FTIR characterization before and after the esterification process.

METHOD

This laboratory study was conducted using two factors: reaction temperature (40 and 60 °C) and H_3PO_4 catalyst volume (4 and 8 mL), with a reaction time of 60 minutes for all treatments. Each treatment was conducted once, resulting in a total of four experimental runs. The study was carried out at the Joint Laboratory Building of Singaperbangsa University in Karawang from August 2025 to January 2026. The research stages included oil pretreatment, esterification, phase separation, washing, FFA content analysis, measurement of physical properties, and FTIR characterization, as summarized in Figure 1.

The used cooking oil was first subjected to pretreatment before being reacted with methanol in the presence of H_3PO_4 under the specified temperature and catalyst volume conditions. Following esterification, the reaction mixture was separated and washed before analysis. FFA content was determined to evaluate the reduction in free fatty acids, while density and viscosity were measured to provide preliminary information on the physical properties of the oil. FTIR characterization was performed to observe the functional groups before and after esterification. The results from the four treatment conditions were compared descriptively to identify trends in FFA reduction and changes in the measured properties resulting from variations in reaction temperature and H_3PO_4 catalyst volume.

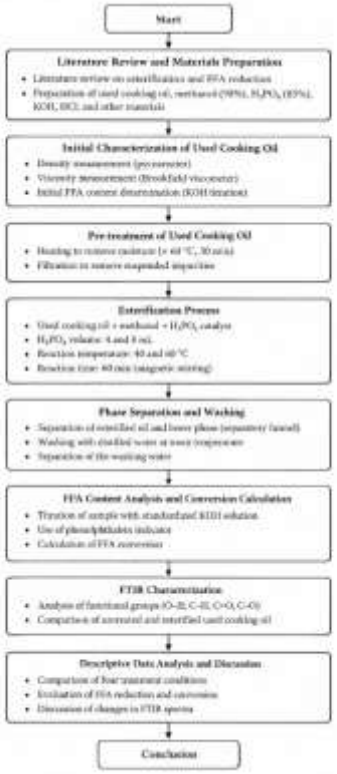

Figure 1. Research workflow for reducing FFA levels in used cooking oil

Density Testing

Density is a physical parameter that represents the ratio of the mass of a substance to its volume. In this study, density measurement was used as one of the physical parameters to compare the characteristics of fresh cooking oil and used cooking oil before the esterification process. Differences in density values can provide an indication of changes in the physical properties of the oil associated with repeated use and heating during the frying process. Density measurements were carried out using a pycnometer with a known and fixed volume, allowing the mass of the sample occupying the specified volume to be determined accurately. Before use, the pycnometer was cleaned and dried to minimize the influence of residual substances or water on the measurement results. The density of each sample was determined from the difference between the mass of the pycnometer filled with the sample and the mass of the empty pycnometer. This mass difference was then divided by the volume of the pycnometer used. The density was calculated using Equation (1) (Santoso et al., 2024).

$$\rho = \frac{m_1 - m_2}{v} \quad (1)$$

where ρ is the density (g/mL), m_1 is the mass of the pycnometer filled with the sample (g), m_2 is the mass of the empty pycnometer (g), and V is the volume of the pycnometer (mL)

Viscosity Testing

Viscosity is one of the physical parameters that describes the resistance of a fluid to flow. The viscosity value provides information about the flow characteristics of the oil and changes in its physical properties resulting from repeated use and heating. In this study, the dynamic viscosity (μ, cP) was measured directly using a digital viscometer for both fresh cooking oil and used cooking oil samples. The measurement was conducted to determine the internal resistance of the samples to flow under the specified measurement conditions. Subsequently, the kinematic viscosity (ν, cSt)

was determined from the ratio of dynamic viscosity to the density of each sample.
The kinematic viscosity was calculated using Equation (2), with the density values obtained from measurements using a pycnometer. The relationship between dynamic viscosity, kinematic viscosity, and density was used to provide a more comprehensive characterization of the flow properties of the oils. The calculation of kinematic viscosity followed the method reported by (Sánchez-rodríguez et al., 2025).

$$V = \frac{\mu}{\rho} \quad (2)$$

where ν is the kinematic viscosity, μ is the dynamic viscosity, and ρ is the density.

Tools and Material

The materials used were used cooking oil, 98% methanol, 85% phosphoric acid (H_3PO_4), 37% hydrochloric acid (HCl), potassium hydroxide (KOH), distilled water, and phenolphthalein indicator. Used cooking oil was used as the main raw material, while methanol and H_3PO_4 served as the alcohol and acid catalyst, respectively. KOH and phenolphthalein were used for FFA determination by titration. The equipment included an analytical balance, magnetic stirrer with a heating plate, thermometer, separating funnel, 25 mL Pyrex pycnometer, Brookfield DV-I viscometer (AMETEK), burette, glassware, and Shimadzu IRTracer-100 FTIR spectrometer. These instruments were used for sample preparation, reaction, physical property measurement, titration, and FTIR characterization.

Pre-Treatment of Used Cooking Oil

The sample used in this study was household used cooking oil generated from food frying activities. The sample was selected to represent used cooking oil that had undergone repeated heating and use at the household scale. Repeated use of cooking oil can cause changes in its characteristics due to exposure to heat during the frying process. The used cooking oil was subsequently collected and used as the raw material for the study after undergoing a pretreatment process.

Before the esterification process, 100 mL of used cooking oil was heated at 150 °C for 30 minutes and then filtered to remove water and suspended solids present in the sample. The heating step was performed to help reduce the water content before the oil was subjected to esterification, while filtration was carried out to separate impurities and solid particles remaining in the oil.

The heating and filtration steps were then repeated once under the same conditions to ensure more effective removal of water and impurities. After the second filtration, the sample was cooled to room temperature before being used in the esterification process. The pretreated sample was subsequently used as the raw material for all esterification treatments with the predetermined variations in reaction temperature and H_3PO_4 catalyst volume.

Esterification Process

A total of 100 mL of pretreated oil was mixed with 20 mL of 98% methanol and 85% H_3PO_4 according to the catalyst volume variations, namely 4 or 8 mL. The methanol and catalyst mixture was then combined with the pretreated oil and homogenized using a magnetic stirrer at a stirring speed of 500 rpm. Subsequently, the mixture was heated at the predetermined reaction temperature of 40 or 60 °C for 60 minutes. The stirring speed and reaction time were kept constant for all treatments, while the reaction temperature and H_3PO_4 catalyst volume were varied according to the experimental design. During the heating and stirring process, the reaction vessel was covered with aluminum foil to minimize methanol loss due to evaporation. After the esterification process was completed, the reaction mixture was first allowed to cool to a suitable condition for phase separation and then transferred into a separatory funnel. The mixture was

allowed to stand overnight to provide sufficient time for the components to separate into distinct phases. After the settling period, the mixture was expected to form two phases, which were subsequently separated and subjected to the washing and subsequent treatment steps.

Separation and Washing Process

After the settling process was completed, the bottom phase, which contained most of the excess methanol, water, and acid catalyst, was slowly separated from the oil phase using a separatory funnel. The separated oil phase was then subjected to a washing step to reduce the residual reactants, catalyst, and impurities carried over from the esterification process. Washing was performed three times using 100 mL of hot distilled water at 70 °C for each washing step. In each step, distilled water was added to the separatory funnel containing the oil phase, and the mixture was gently shaken to promote contact between the oil and aqueous phases. The mixture was then allowed to stand until two distinct phases were formed again. The lower aqueous phase was slowly drained, while the oil phase was retained for the subsequent washing step. The washing process was carried out to reduce residual methanol, H_3PO_4, and other impurities remaining in the oil phase after the esterification and initial phase separation processes. The washing step was repeated until the aqueous phase appeared clear, with a total of three washing cycles performed according to the established procedure. After all washing steps were completed, the oil phase was separated and used for further analysis.

Standardization of KOH and Analysis of FFA Content

A 0.1 N KOH solution was prepared by dissolving 5.61 g of KOH in distilled water to a final volume of 1 L. A nominal 0.1 N HCl solution was prepared by diluting 0.83 mL of 37% HCl to a final volume of 100 mL. The normality of the KOH solution was determined by titration against the 0.1 N HCl solution using phenolphthalein as an indicator. The titration was performed in triplicate, and the determined KOH normality was used in the calculation of the FFA content.

A 10 g sample of the esterified oil was mixed with 25 mL of methanol and then heated at 50 °C for 30 minutes. The sample was titrated using the standardized KOH solution with phenolphthalein as an indicator until a stable pink color appeared. The FFA content, expressed as palmitic acid, was calculated using Equation (3) (Helwani et al., 2025).

$$\text{FFA (\%)} = \frac{25.6 \; x \; N \; x \; V}{W} \qquad (3)$$

where V is the volume of KOH (mL), N is the normality of KOH (N), 25.6 is the equivalent mass factor of palmitic acid, and w is the sample mass (g). The FFA conversion was calculated using Equation (4) and (5) (Al-sakkari et al., 2020).

$$\text{FFA Conversion (\%)} = \frac{AV_0 \; x \; AV_t}{AV_0} \quad \text{x } 100\% \qquad (4)$$

where AV_0 is the initial acid value and AV_t is the acid value after esterification.

FTIR Characteristic Analysis

FTIR spectroscopy was used to compare the functional group characteristics of the used cooking oil before esterification and the treated oil after the process. The analysis was performed using a a Shimadzu IRTracer-100 instrument over a wavenumber range of 500–4000 cm^{-1}, with a resolution of 2 cm^{-1} and 45 scans. The FTIR spectra of each sample were compared to observe changes or differences in the absorption characteristics after the esterification process. The spectral interpretation focused on the absorption regions of O–H, aliphatic C–H, carbonyl C=O, and C–O functional groups associated with the organic structure of the oil. These absorption regions were examined based on their wavenumber positions and the characteristics of the bands appearing in the spectra of each sample. The interpretation was conducted with reference to the

principles of FTIR spectral interpretation for organic materials as described by (Nandiyanto, 2019).

RESULT AND DISCUSSION

Density and Viscosity Testing

A comparison of physical properties shows that used cooking oil has a density of 0.91 g/mL, which is higher than that of new cooking oil at 0.89 g/mL. Dynamic viscosity also increased from 55 to 59 cP, while kinematic viscosity increased from 61.85 to 65.09 cSt (Table 1). This trend is consistent with changes in oil resulting from repeated heating, during which oxidation and polymerization reactions can form compounds with higher molecular weights and polarity, thereby increasing viscosity and potentially affecting density (Abrante-pascual, 2024; Maduelosi et al., 2024). Since the measurement temperature and instrument uncertainties were not presented as separate analyses, the density and viscosity data in this study were interpreted as descriptive comparisons.

Table 1. Density and viscosity of new and used cooking oil measured at room temperature

Sample	Density (g/mL)	Dynamic viscosity (cP)	Kinematic viskosity (cSt)
New Cooking Oil	0,89	55	61,85
Used Cooking Oil	0,91	59	65,09

FFA Content Testing

The initial FFA content of the used cooking oil was 2.11%. After esterification using H_3PO_4, the FFA content decreased under all tested combinations of reaction temperature and catalyst volume, as presented in Table 2. The decrease in FFA content indicates a change in the free fatty acid content after the oil underwent the esterification treatment. The FFA values obtained after esterification varied among the different combinations of reaction temperature and H_3PO_4 catalyst volume. The variations in temperature and catalyst volume resulted in different levels of FFA reduction, allowing the trend in FFA content changes under the applied reaction conditions to be observed. The initial and post-esterification FFA values were subsequently compared to describe the changes in free fatty acid content for each treatment.

Table 2. FFA content and conversion rate after esterification

Treatment Variations	FFA after esterification (%)	Initial FFA (%)	Initial Acid Value (%)	Acid Value After Esterification	FFA Conversion (%)
4 mL 40 °C	1,33	2,11%	4,65	2,92	37,02
4 mL 60 °C	0,88			1,93	58,41
8 mL 40 °C	0,70			1,54	66,73
8 mL 60 °C	0,33			0,71	84,55

At a catalyst volume of 4 mL, increasing the temperature from 40 to 60 °C reduced the FFA content from 1.33% to 0.88%. At a catalyst volume of 8 mL, the same temperature increase reduced the FFA content from 0.70% to 0.33%. An increase in the H_3PO_4 volume from 4 to 8 mL was also accompanied by a decrease in FFA content at both temperatures. Thus, the 8 mL–60 °C conditions yielded the lowest residual FFA content and the highest reported conversion, namely 84.55%.

In addition to the effect of temperature, increasing the H_3PO_4 catalyst volume from 4 to 8 mL was also accompanied by a decrease in FFA content at both reaction temperatures. At 40 °C, the FFA content decreased from 1.33% with 4 mL of catalyst to 0.70% with 8 mL of catalyst. At 60 °C, the FFA content similarly decreased from 0.88% to 0.33% when the catalyst volume was increased

from 4 to 8 mL.

Thus, both increasing the reaction temperature and increasing the H_3PO_4 catalyst volume showed a trend toward lower FFA content within the range of conditions tested.

The combination of 8 mL H_3PO_4 and 60 °C resulted in the lowest residual FFA content, at 0.33%, compared with the other treatments. This condition also produced the highest FFA conversion obtained in the study, namely 84.55%. These results indicate that the combined increase in reaction temperature and catalyst volume within the tested treatment range was associated with a greater reduction in FFA content. However, this result only identifies the treatment that produced the lowest FFA content and the highest conversion among the conditions evaluated and therefore cannot be used to establish an optimum condition beyond the experimental range. Furthermore, because the results were presented without standard deviations and no inferential statistical tests were performed, the differences in FFA content among the treatments were interpreted as descriptive trends. Therefore, the results cannot be used to conclude that the differences among treatments were statistically significant; rather, they describe the observed pattern of FFA content changes in response to the variations in reaction temperature and H_3PO_4 catalyst volume applied in this study.

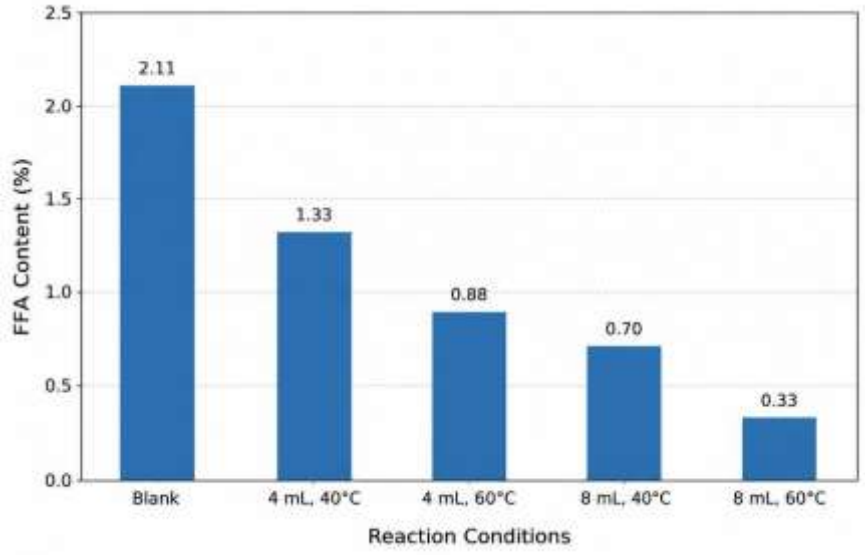

Figure 2. FFA content of used cooking oil before and after the esterification process

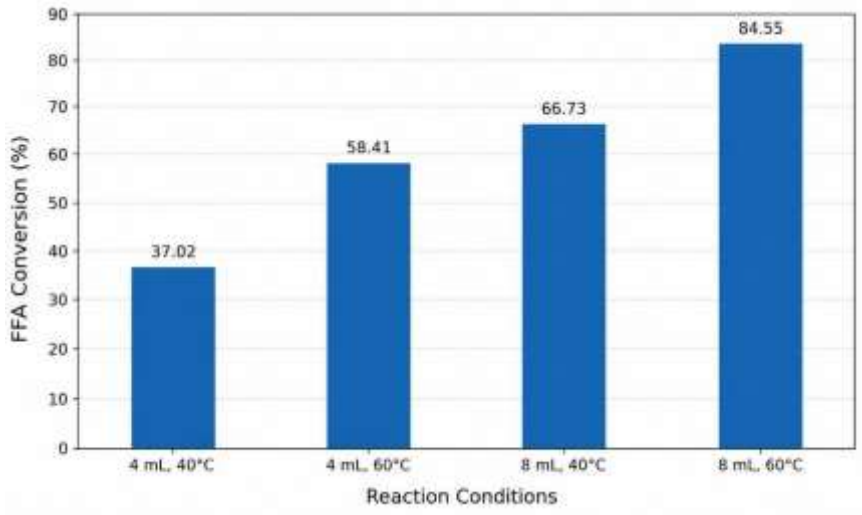

Figure 3. FFA Conversion at Different Combinations of Temperature and Catalyst Volume

The decrease in FFA concentration observed at higher temperatures can be attributed to an increase in the esterification reaction rate due to higher kinetic energy and intermolecular collision frequency. Putri et al., (2022) also reported an increase in the FFA esterification rate at higher temperatures. On the other hand, an increase in catalyst volume provides a greater number of acid species that can facilitate the protonation of the carbonyl group, thereby accelerating ester formation under the conditions studied. These results are consistent with previous studies reporting that catalyst amount is one of the key parameters in the esterification of used cooking oil and feedstocks with high FFA content (Megawati et al., 2021; Supeno et al., 2023). The 8 mL–60 °C conditions in this study should be described as the best conditions within the tested range, rather than as optimal conditions, since only two temperature levels and two catalyst volume levels were evaluated. Furthermore, the reaction temperature of 60 °C is close to the boiling point of methanol; therefore, controlling methanol loss becomes important if the reaction temperature is increased further.

FTIR Characterization Test

FTIR was employed as a qualitative characterization method to compare the functional groups present in the used cooking oil before treatment with those in the four samples after the esterification process. This analysis was conducted to observe changes in the functional group characteristics of the samples before and after treatment based on the position and intensity of the absorption bands. The comparison of the FTIR spectra was used as supporting data to identify functional groups associated with the molecular structure of the oil and to observe spectral changes following the esterification process. In addition, the FTIR analysis provides information on changes in the chemical characteristics of the oil that may occur during the esterification process, particularly through differences in the absorption intensity and position of the characteristic bands. The spectra obtained from each treatment were compared with the spectrum of the untreated used cooking oil to evaluate the changes in the functional group characteristics under different reaction conditions. Thus, the FTIR results complement the FFA conversion analysis by providing qualitative evidence of changes in the chemical structure of the oil after esterification. The FTIR spectra of the untreated used cooking oil and the four samples after esterification are presented in Figures 4 and 5.

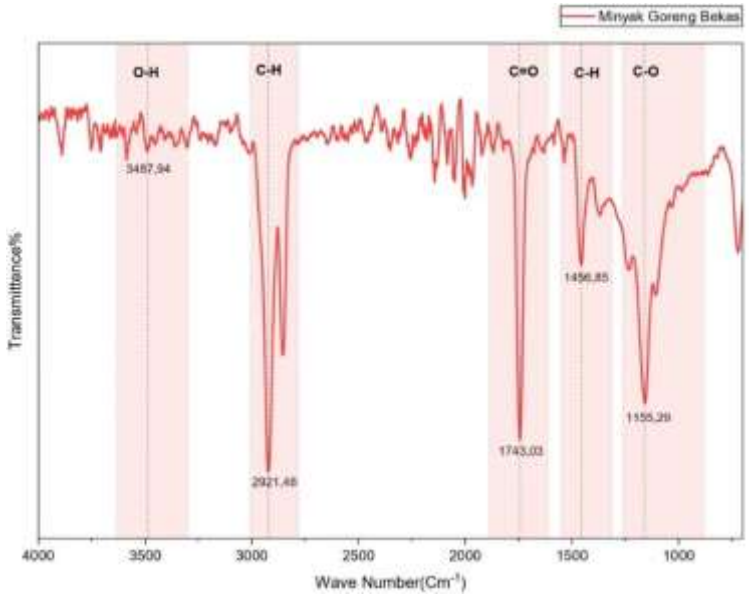

Figure 4. FTIR Spectrum of Used Cooking Oil Before Esterification

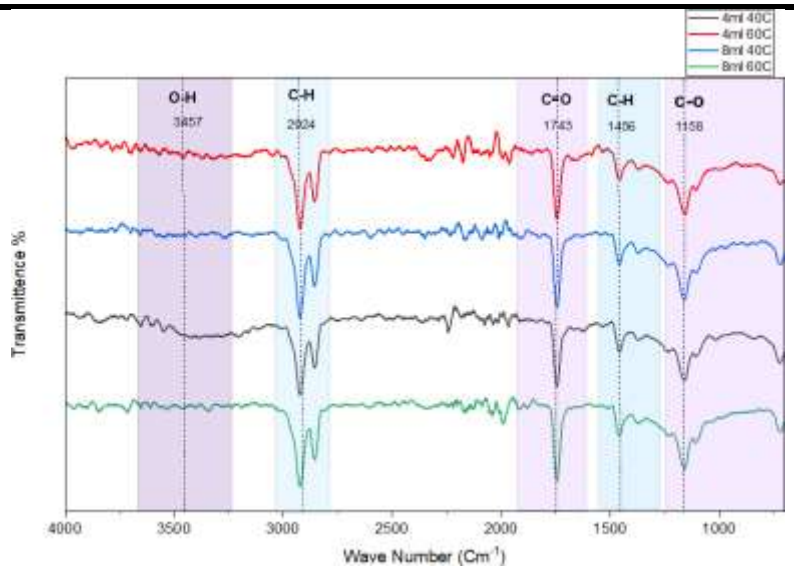

Figure 5. FTIR Spectrum of Used Cooking Oil After the Esterification Process

The FTIR spectra of the used cooking oil after esterification showed absorption bands corresponding to the main functional groups commonly observed in triglyceride-based oils. As presented in Table 3, the O–H absorption bands were observed at 3412, 3452, 3441, and 3343 cm^{-1} for the 4 mL–40 °C, 4 mL–60 °C, 8 mL–40 °C, and 8 mL–60 °C treatments, respectively. The C=O absorption bands were observed at 1742–1744 cm^{-1}, while the C–O absorption bands appeared at 1155–1159 cm^{-1}. In addition, the aliphatic C–H stretching band was observed at approximately 2924 cm^{-1}. These absorption bands are within the characteristic regions of O–H (3200–3570 cm^{-1}), C–H (2850–3000 cm^{-1}), C=O (1725–1750 cm^{-1}), and C–O (1000–1300 cm^{-1}) reported for triglyceride-based oils (Nandiyanto, 2019).

The C=O and C–O absorption bands were observed within relatively narrow wavenumber ranges among the treatments, indicating that these functional groups remained present under the different reaction conditions. Meanwhile, the O–H absorption band showed greater variation in its position, ranging from 3343 to 3452 cm^{-1}. These variations in the absorption-band positions may reflect differences in the chemical environment of the functional groups resulting from the different reaction temperatures and H_3PO_4 catalyst volumes used during esterification.

Table 3. Observed FTIR Absorption Bands of Used Cooking Oil after Esterification

Gugus Fungsi Pita Serapan	**O-H (3200 – 3570 cm^{-1})**	**C=O (1725 – 1750 cm^{-1})**	**C-O 1000 – 1300 cm^{-1}**
4 mL 40 °C	3412	1742	1156
4 mL 60 °C	3452	1743	1155
8 mL 40 °C	3441	1743	1158
8 mL 60 °C	3343	1744	1159

The relatively small shifts in the absorption bands among the treatments indicate variations in the functional groups during the esterification process. The C=O band showed only a slight shift from 1742 to 1744 cm^{-1}, while the C–O band varied from 1155 to 1159 cm^{-1}. The O–H band showed greater variation, ranging from 3343 to 3452 cm^{-1} among the treatments. Meanwhile, the C–H stretching band was observed at approximately 2924 cm^{-1}, corresponding to the aliphatic hydrocarbon chains of the oil. These variations may be associated with differences in reaction temperature and H_3PO_4 catalyst volume. However, the FTIR spectra alone do not provide definitive confirmation of methyl ester formation. The FTIR results, together with the reduction

in FFA content, support the occurrence of chemical changes associated with the esterification process.

CONCLUSIONS

The density and viscosity of used cooking oil were higher than those of new cooking oil, indicating changes in its physical properties due to repeated use. These measurements were used for preliminary characterization and were not directly used to determine the success of the esterification process. The FFA content decreased from 2.11% to 1.33%, 0.88%, 0.70%, and 0.33% under the conditions of 4 mL–40 °C, 4 mL–60 °C, 8 mL–40 °C, and 8 mL–60 °C, respectively. The use of 8 mL H_3PO_4 at 60 °C resulted in the lowest FFA content (0.33%) and the highest FFA conversion of 84.55%. The FFA conversion was calculated using the reduction in acid value as the basis of the calculation. Within the tested range, increasing the reaction temperature and H_3PO_4 catalyst volume was associated with greater FFA reduction. FTIR analysis showed O–H, C–H, C=O, and C–O absorption bands with small variations among the treatments, providing supporting qualitative evidence of chemical changes during esterification. Since the C=O and C–O bands were also present before esterification, FTIR was used as supporting qualitative characterization, while FFA analysis was used as the primary basis for evaluating FFA reduction. The lowest FFA content of 0.33% was close to the maximum FFA limit of 0.30% specified by SNI 7709:2019 for palm cooking oil; however, this comparison was limited to the FFA parameter and does not indicate overall compliance with edible-oil standards.

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