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Quality Assessment of Used Palm Cooking Oil Processed by Microfiltration Using Whatman Filter Papers with Different Pore Sizes

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Abstract. *Microfiltration is a preliminary treatment in the production of biodiesel. This method is characterized by its simplicity and its environmental friendliness. It can reduce foam production during the saponification process, which is generally concomitant with the esterification process. Furthermore, microfiltration process does not involve the use of chemicals. The micropore size of the filter greatly influences the quality of the biodiesel produced, given its impact on impurities present in the oil used. The present study sought to determine the effect of microporous membrane size on the quality of used frying oil. The treatment variables in this study were the micropore sizes of the Whatman filter paper used: 2.5 µm, 8 µm, 11 µm, 16 µm, 20 µm, and 25 µm. Meanwhile, the observation variables encompassed the free fatty acid (FFA) content, acid number, viscosity, and water content of the used cooking oil. The findings of the study indicate that the microfiltration treatment employing filter paper with 2.5 µm pore size yielded the best results, with free fatty acid (FFA) level, acid number, viscosity and water content of 0.00238%, 0.411 mg KOH/g, 3.2843 cSt and 0.00068%, respectively. In accordance with the stipulations outlined in the Indonesian National Standard (SNI) 7128:2015, the microfiltered oil obtained in this study is deemed suitable for use as a raw material in biodiesel production.*
Keywords: *used palm cooking oil; microfiltration; pore size; quality.*

Type of the Paper: Regular Article.

1. Introduction

Palm cooking oil is a food ingredient composed primarily of triglycerides derived from fractionated palm oil (refined, bleached, and deodorized palm oil) with or without additional food additives and contains vitamin A or provitamin A [1,2]. Disposing of used palm cooking oil improperly can be hazardous to the environment. This improper disposal can cause pollution and disrupt ecosystems [3]. To address this issue, the zero-waste program, an initiative aimed at reducing and managing waste in a manner that does not harm the environment while facilitating its reuse and/or transformation into new, useful products, may offer a viable solution. In the case of used palm cooking oil waste, for instance, it can be repurposed as a raw material for biodiesel production [4–6]. Biodiesel is a promising source of new renewable energies and a potential future substitute fuel for petroleum-based diesel. Triglycerides, a basic compound present in both plant

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and animal sources, reacts with alcohol to yield biodiesel. As stipulated by the National Standardization Agency of Indonesia, biodiesel is defined as a vegetable fuel in the form of fatty acid methyl esters (FAME) [7]. Biodiesel, alongside bioethanol, is a highly preferred variety of liquid biofuel due to its numerous advantageous characteristics. These include its capacity for natural degradation, its eco-friendliness, and its notable properties such as a high energy content, low sulfur levels, good lubrication qualities, and a high cetane rating [8]. However, certain products resulting from the cooking process can negatively affect the biodiesel production from used cooking oil, thereby necessitating pre-treatment. The esterification-transesterification method is the common approach in biodiesel production. Esterification, a pretreatment step in biodiesel production that utilizes an acid catalyst, presents several disadvantages. Chief among these is the potential for increased oil contamination, which can lead to maintenance challenges for the biodiesel. While esterification is a process that is only possible to undertake using an acid catalyst, transesterification can be conducted using either an acid or a base catalyst. Transesterification is susceptible to undesirable saponification reactions, especially in oils with high levels of free fatty acids and water. A potential alternative method to esterification that can reduce the acid number and free fatty acid levels is microfiltration [9].

Microfiltration is an alternative and highly effective method for reducing acid number and free fatty acids (FFA) levels in used palm cooking oil [10]. This filtration process aims to remove small particles and dissolved substances from cooking oil, such as proteins, carbohydrates, and free fatty acids, which can interfere with subsequent processing [11]. This process is a crucial initial step in the biodiesel production. Microfiltration is expected to improve the quality of the feedstock, separate impurities and small particles from the biodiesel, and aid in the purification of biodiesel. This process can reduce water loss and produce biodiesel with good physicochemical properties.

A high level of FFA in used palm cooking oil poses a challenge to the biodiesel production process, particularly when base-catalyzed transesterification is employed. The presence of high levels of FFA can lead to undesirable saponification (soap formation), which reduces the yield of biodiesel and complicates the purification process [12]. The microfiltration process removes larger molecules, polymerized compounds, and suspended solids, contributing to the desirable viscosity of used palm cooking oil and facilitating its flow. The process can be used to remove water, for example, as water content in oil can cause a hydrolysis reaction [13]. This reaction produces free fatty acids due to the decomposition of oil or triglycerides. The free fatty acids that are formed can react further and form aldehydes and ketones, which will trigger rancidity in the oil [14]. This suggests that a high water content in oil can lead to a deterioration in its quality. In this context, microfiltration can function as a pre-treatment for used palm cooking oil, with the aim of improving its suitability for further processing. This treatment physically removes impurities, resulting in a reduction of acid numbers and FFA content (by preventing further degradation and removing associated particulates). Additionally, it decreases viscosity and reduces water content. The aim of this study is to examine the effect of the pore size of Whatman filter paper on the free fatty acid content, acid number, viscosity, and water content of used cooking oil subjected to microfiltration.

2. Materials and Methods

In this study, used palm cooking oil from frying crispy fried chicken was utilized as the raw material for microfiltration. Whatman filter papers with pore sizes of 2.5 µm, 8 µm, 11 µm, 20 µm, and 25 µm were employed. Whatman qualitative filter papers are characterized by their specific pore sizes that determine the levels of particle retention and the rates of flow. Smaller pores increase retention but decrease flow and filter more immpurities. The microfiltration process was assisted by a 2 CFM vacuum pump. The membrane process was executed with the vacuum pump, the flow rate of which was between 51 and 57 L/min. The partial pressure was set at 2 Pa, and the total pressure at 150 µPa for a duration of 45 minutes.

Initially, the used palm cooking oil was subjected to a standard filtration process to remove large impurities. Subsequently, microfiltration was conducted using Whatman filter paper. The microfiltration process in the present study and the residues filtered are illustrated in Fig. 1 below.

(a) (b)

Fig. 1. The microfiltration process (a) and the residue (b)

As demonstrated by Fig. 1 (b) provides a more detailed view of the filter paper used for microfiltration, with the presence of impurities in the oil evident on the filter paper. The study was conducted with three repetitions, and the analysis was carried out using a Randomized Complete Block Design (RCBD).

Fig. 2 presents a flowchart of the present study, starting with filtration, followed by microfiltration, and concluding with microfiltrated oil quality analyses. The observation variables in the study included acid number, free fatty acid content, viscosity, and water content of used palm cooking oil, both before and after microfiltration. To ascertain the existence of statistically significant differences in these parameters under various microfiltration conditions, an analysis of variance (ANOVA) was used. If the ANOVA indicated a significant difference, the Tukey's honestly significant difference (HSD) test would then be applied in order to identify which specific groups differ from one another. This process would provide a more detailed understanding of the effectiveness of the microfiltration process.

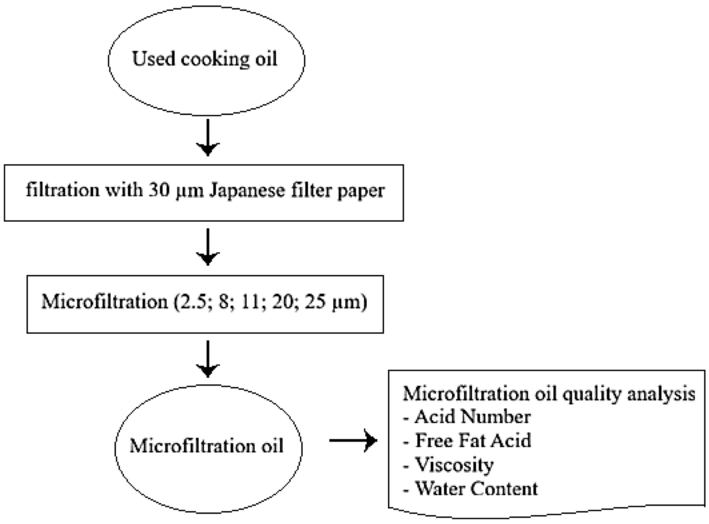

Fig. 2. Flowchart of the microfiltration process employed for used cooking oil

2.1. Analysis Methods

2.1.1. Acid Number

Acid number is a measure of the amount of KOH in milligrams needed to neutralize the free fatty acids present in 1 g of sample [7]. The acid number analysis using titration method is in accordance with Indonesian National Standard (SNI) 7182:2024. A 3-g oil sample was weighed, and then 50 mL of 96% ethanol were added. The sample was heated at 50°C for 10 minutes. Subsequently, 1% phenolphthalein indicator was incorporated and a titratation was conducted with 0.1 N KOH. The titration was deemed complete when a pink hue manifested [7]. The calculation of acid number was performed in accordance with Eq. (1).

$$Acid\ Value = \frac{A\ x\ N\ x\ 56{,}1}{sample\ mass} \quad (1)$$

Description:

A	Volume of KOH used
N	KOH normality
56,1	KOH molecular weight

2.1.2. Free Fatty Acid Content

The method for analyzing free fatty acid content was the same as the method for analyzing acid number, utilizing titration as outlined by the National Standardization Agency of Indonesia [1]. However, a different equation was employed to quantify the results. Free fatty acid percentage (%FFA) expresses the concentration of free fatty acids in the oil as a percentage by weight, calculated as if all free fatty acids were a specific predominant fatty acid. The free fatty acid as palmitic acid content was calculated using the following Eq. (2).

$$\%FFA = \frac{V.KOH\ x\ N.KOH\ x\ oil\ m.w}{sample\ mass\ x\ 1000} x100\% \tag{2}$$

Description:

V KOH	Volume of KOH used	
Oil m.w	Molecular weight of oil	(palmitic acid = 25.6)
N KOH	KOH normality	

2.1.3. Viscosity

Viscosity analysis was performed using an Oswald viscometer, an instrument that is well-suited for measuring the viscosity of the resulting sample [15]. The analysis involved measuring the time required for the liquid to flow through a capillary tube from point *a* to point *b*. The liquid was inserted into an Oswald viscometer, which was then placed on a thermostat. The liquid was sucked into the viscometer using a pump until it reached the mark *a*. The liquid was allowed to flow downwards, and the time required from *a* to *b* was recorded using a stopwatch. The viscosity was measured using Eq. (3).

$$\eta = \frac{\eta_0 \cdot d \cdot t}{d_0 \cdot t_0} \tag{3}$$

Description:

η	Dynamic viscosity of the sample fluid (poise)
η_o	Viscosity of the reference fluid (poise)
t_o	Flow time of reference fluid (s)
t	Flow time of sample fluid (s)
do	Density of distilled water (g/mL)
d	Sample density (g/mL)

Dynamic viscosity was converted to kinematic viscosity using Eq. (4).

$$v = \frac{\mu}{\rho}, v(mm^2/s) = v(m^2/s) \times 1{,}000{,}000 \tag{4}$$

Description:

ν	Kinematic viscosity (m^2/s)
μ	Dynamic viscosity (Pa.s)
ρ	Palm oil density (kg/m^3)

Given that 1 m is equivalent to 1000 mm, it follows that 1 m² is equivalent to (1,000 mm)², or 1,000,000 mm².

2.1.4. Water Content

The gravimetric method was used to analyze water content in the cooking oil samples [16]. A total of 2 g of cooking oil samples were weighed and placed in a porcelain cup of known weight. Then, the porcelain cup containing the cooking oil sample was subjected to an oven temperature of 100°C until the oil weight was constant. Subsequent to heating process, the porcelain cup was re-weighed to determine the quantity of water lost during the heating process. The water content (in percentage) of the analyzed cooking oil sample was calculated using Eq. (5).

$$\%water\ content = \frac{m_1 - m_2}{m_0} x100\% \quad (5)$$

Description:
m_o Weight of empty cup
m_1 Weight of cup and initial sample
m_2 Weight of cup and sample after drying

3. Results and Discussion

The effectiveness of a membrane is influenced by a number of factors, including its state, the elements with which it interacts in the feed, the velocity, temperature, pressure, and the composition of the membrane itself. Membranes can be categorized into two types: organic and inorganic. The present study examined the microfiltration process using cellulose membrane or filter paper. Microfiltration is a preliminary treatment that aims to reduce the impurities present in used palm cooking oil, thereby rendering suitable for processing into biodiesel through the transesterification process. In several studies, microfiltration was conducted using ceramic membranes, but in the present study, cellulose membranes were utilized. The purification of vegetable oil by means of a cellulose membrane is a noticeably uncomplicated process with the potential to offer numerous advantages [17]. These include the saving of energy, as the process is carried out at low temperatures, and the possibility of true stage cancellation. It thus provides a promising alternative to conventional methods for achieving eco-friendly and cost-effective technical operations [10].

The used palm cooking, having undergone microfiltration, was subjected to a testing including the assessment of its acid number, free fatty acid content, viscosity and water content. The results of this assessment, along with the established standard, are presented in Table 1. As shown in Table 1, the utilization of filter paper with a 2.5 µm pore size resulted in the production of oil that met the standards for both acid number and free fatty acid parameters. All filter papers yielded oil with water content that met the standard.

Table 1. Quality of used palm cooking oil after microfiltration

Pore Size of Filter Paper	Acid Number	Free Fatty Acid	Viscosity	Water Content
μm	mg KOH/gram	%	cSt	%
Without microfiltration	0.9144	0.6982	5.4017	0.0022
2.5	0.4108	0.2384	3.2843	0.0006
8	0.5222	0.3230	3.9591	0.0007
11	0.6160	0.4766	4.0939	0.0010
20	0.6524	0.5285	4.5094	0.0011
25	0.7276	0.6296	4.6971	0.0015
Standard	0.5	0.3	-	0.05

3.1. Acid Number

The data presented in Table 1 shows a decrease in the acid number of used palm cooking oil following microfiltration. The filter paper with a pore size of 2.5 μm produced the lowest acid number. This result is attributable to the enhanced effectiveness of filter paper with smaller pores in filtering solid particles [18]. The smaller the pores of the filter paper used, the more solid particles are retained, including those capable of increasing the acid number.

The maximum limit for the acid number of used palm cooking oil to be processed by transesterification into biodiesel is 0.5 mg KOH/g, in accordance with the biodiesel standards set out in SNI 7182:2024 regarding Biodiesel for Diesel Fuel Mixture [7]. In the present study, the lowest average of acid number recorded was 0.4108 mgKOH/g, achieved through microfiltration treatment using filter paper with a 2.5 μm pore size. This acid number meets the requirement for the acid number limit of used palm cooking oil to be processed into biodiesel through the transesterification process. The transesterification process will reduce the acid number of the oil to a greater extent, thus producing biodiesel of a higher quality [19].

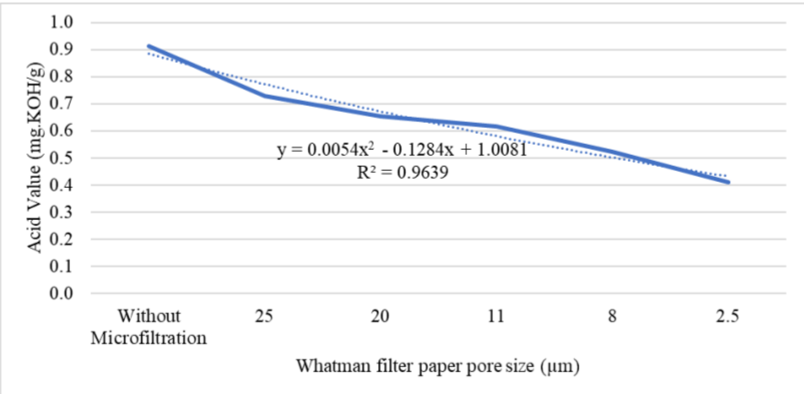

Fig. 3. Trendline of the acid number of used palm cooking oil

Fig. 3 shows a trendline for the acid number of microfiltrated oil. The trendline equation, $y = 0.0054x^2 - 0.1284x + 1.0081$, indicates a relationship between treatment and the results. Fig. 3 also shows an R^2 value of 0.9639, signifying that the treatment affected the acid number by 96.39%. The remaining 3.61% can be attributed to other factors, such as water content in oil, operating temperature, contact time between oil and filtration media, and the type of filtration media used.

3.2. Free Fatty Acid

Free fatty acids are an indicator of oil quality, with high levels of these acids potentially indicating oil damage or hydrolysis during processing [20,21]. The transesterification reaction requires oil of the utmost purity, characterized by a free fatty acid content that is less than 2% [22]. According to Ketaren (1986), the maximum level of free fatty acids in oil that can be processed for transesterification to obtain biodiesel is 1%, but according to SNI 7709:2019, the maximum is 0.3% [1,23]. The results the study, as presented in Table 1, demonstrate that the utilization of filter paper of a specific size in the microfiltration process exerts a highly significant effect on the free fatty acid content of the oil produced.

The decrease in the levels of free fatty acid in used cooking oil following microfiltration is due to the reduction in particles, both dirt and contaminants. These particles can instigate oxidation and hydrolysis reactions, which are the main causes of the increase in free fatty acids. Free fatty acids (FFAs) are typically composed of 8–22 carbon atoms and numerous forming double bonds. Because of their chemical structure, FFAs posses a high degree of reactivity, readily undergoing a variety of reactions, including esterification, oxidation, and hydrolysis [24]. In a process of esterification, they react with glycerol, resulting in the formation of triglycerides, which are the primary constituents of fats, oils, and grease (FOG). This reaction has a major effect on properties such as pH, melting point, viscosity, and reactivity [25]. The data presented in Table 1 indicates that the lowest free fatty acid content, 0.2384%, was obtained in microfiltration using 2.5 µm pore size filter paper, while the highest free fatty acid content, 0.6296%, was obtained in microfiltration using 25 µm pore size filter paper. It can thus be concluded that microfiltration treatment using filter paper with a 2.5 µm pore size can reduce the free fatty acid level of used cooking oil to a level that is suitable for further processing to obtain biodiesel by transesterification.

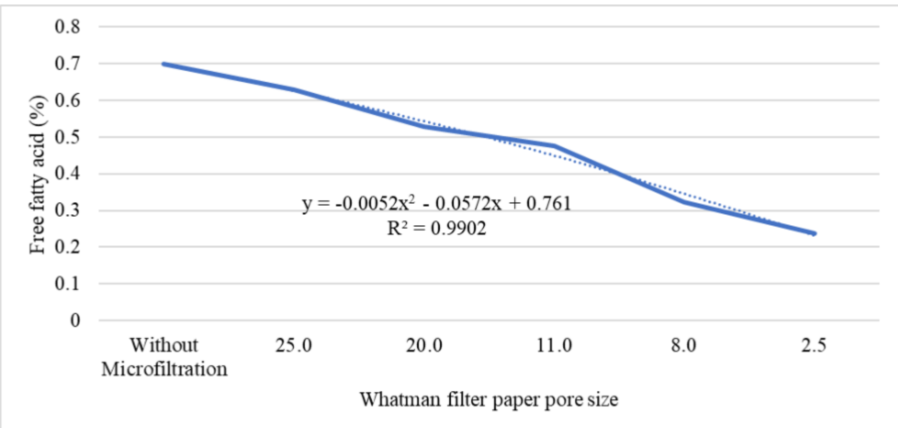

Fig. 3. Trendline of the free fatty acid content of used palm cooking oil

Fig. 3 shows a trendline for the free fatty acid content of microfiltrated oil. The trendline equation, $y = -0.0052x^2 - 0.0572x + 0.761$, indicates a relationship between treatment and the results. Fig. 3 also shows an R^2 value of 0.9902, indicating that the treatment affected the free fatty acid content by 99.02%. The remaining 0.98% can be attributed to other factors, such as water content, lipase enzyme content, separation efficiency, operational conditions, and temperature.

3.3. Viscosity

Used palm cooking oil that has been microfiltrated has a lower viscosity than pre-filtrated oil. This is due to the removal of suspended solid particles and some microorganisms in the used oil through the microfiltration process, which is based on the principle of physical separation. However, dissolved components such as oil and fat remain in the oil [26]. Consequently, following microfiltration, the oil will be clearer and free of particles that could potentially increase its viscosity. A study of the quality of palm oil using viscosity testing and refractive index analysis indicates that fresh palm oil has the highest values for both indicators, while palm oil that has been used twice has the lowest value [15]. Diesel engines are incompatible with direct vegetable oils (DVO) due to their viscous nature [27,28].

As illustrated in Table 1, treatment using filter paper with a 2.5 µm pore size resulted in used cooking oil with the lowest level of viscosity (3.2843 cSt), while treatment using filter paper with a 25 µm pore size produced oil with the highest level of viscosity (4.6971 cSt). Moreover, as demonstrated in Table 1, the application of microfiltration treatments, employing filter papers of varying pore sizes, resulted in a significant variation in the viscosity levels of the microfiltrated cooking oil. The viscosity of used cooking oil is not a primary determinant of its suitability for transesterification [8]; however, excessively high viscosity can impede the efficiency of the transesterification process by complicating mass transfer and reactions. On the other hand, cooking oil with insufficient viscosity (i.e., a thin consistency) may contain high levels of free fatty acids (FFA), which also can impede the transesterification process and compromise the quality of the resulting biodiesel.

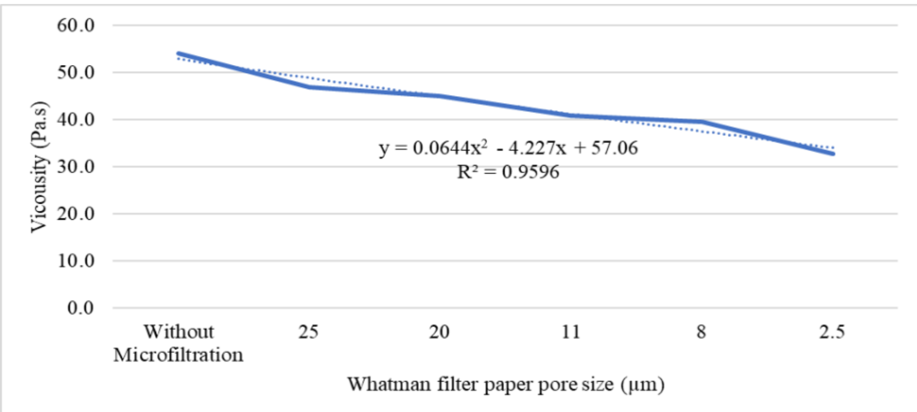

Fig. 4. Trendline of the viscosity of used palm cooking oil

Fig. 4 presents a trendline for the viscosity of microfiltrated oil. The trendline equation is $y = 0.0644x^2 - 4.227x + 57.06$, indicating a relationship between treatment and the results. Fig. 4 also presents an R^2 value of 0.9596, signifying that the treatment affected the viscosity by 95.96%. The remaining 4.04% can be due to factors such as water content, treatment temperature, oil type, and chemical reactions that transpired during the frying process.

3.4. Water Content

Water content is a critical parameter in the processing of oil. Excess water can induce undesirable chemical reactions, compromise the quality of oil product, and even result in equipment damage [29,30]. Membrane-based separation has been reported by several studies as an effective separation method. This method has been shown to exhibit several advantages, including minimal power consumption, highly refined filtered output, adaptable unit size, the capacity to reuse concentrated materials in certain systems, and the elimination of the need for chemical or media replacement [31].

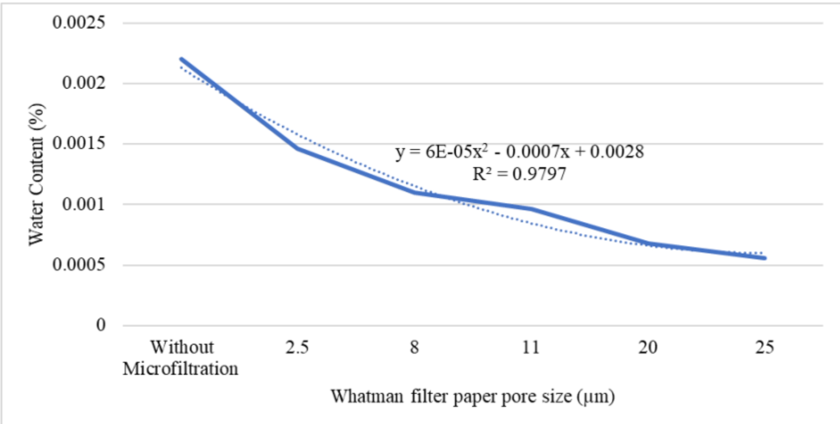

Fig. 5. Trendline of the water content of used palm cooking oil

According to SNI 7182:2015, the maximum water content permissibe for oil transesterification is less than 0.05%, whereas according to SNI 3172:2002, the acceptable range is 0.1–0.3% [32–34]. The water content of the microfiltrated oil in the present study is presented in Table 1. The lowest water content, 0.0006%, was obtained by microfiltration using a 2.5 µm Whatman filter paper, while the highest water content, 0.0015%, was obtained by microfiltration using a 25 µm Whatman filter paper. Despite the resulting variation in water content, the results of the ANOVA data analysis demonstrated no significance difference between the treatments employing Whatman filter papers with varying pore size. As demonstrated by the study data and with referrence to Lestari et al. [33], it is possible to further process the microfiltrated oil into biodiesel through the transesterification process. The utilization of Whatman filter paper, made from cellulose, for microfiltration has been shown to remove water-in-oil emulsions. This finding is consistent with the findings of related studies by Wang et al. and Xu et al. [35,36]. The greater the number of emulsions filtered during the microfiltration process, the lower the water content in the oil after microfiltration. This is due to the microfiltration process being designed to remove particles and liquid droplets, including water in oil-in-water emulsions, from a solution or mixture [26,37].

Fig. 5 presents a trendline for the water content of microfiltrated oil. The trendline equation is $y = 0.00006x^2 - 0.0007x + 0.0028$, indicating the relationship between treatment and the results. Fig. 5 also shows an R^2 value of 0.9797, indicating that the treatment affected the water content by 97.97%. The remaining 2.03% can be attributed to other factors, such as initial oil characteristics and microfiltration process conditions.

4. Conclusion

In the present study, the microfiltrated, used palm cooking oil of the highest quality was obtained with an acid number, free fatty acid content, viscosity, and water content of 0.4108 mg KOH/g, 0.2384%, 3.2843 cSt, and 0.0006%, respectively. This oil was obtained through microfiltration using a 2.5 µm Whatman filter paper. It is suitable for use in the transesterification process, which is a method of converting oil into biodiesel.

Abbreviations

Not applicable

Data Availability Statement

Data supporting this study will be made available on request.

Credit Authorship Contribution Statement

Poppy Diana Sari: conceptualization, methodology, conceptual, translator, writing and editing. **Rukmi Sari Hartati**: conceptual, methodology, supervision, validation. **Syamsudduha Syahrorini**: visualization, formal analyses, validation. **I Nyoman Setiawan**: methodology, supervision, validation. **Rahmah Utami Budiandari**: writing and editing, software, data curation, translator, validation. **I Wayan Sukerayasa**: data curation, formal analysis, validation. **Muhammad Ainnur Rafdiansyah**: Resources, project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Declaration of Use of AI in the Writing Process

Nothing to disclose.

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