

# Production of Fatty Acid Methyl Ester Surfactants using Palm Oil with Various Reaction Temperatures and Duration

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## ABSTRACT

Most surfactants are made from petrochemicals, posing significant environmental concerns due to the non-biodegradable and non-renewable nature. To address this challenge, surfactants from biodegradable, non-toxic, and harmless materials are required, such as Fatty Acid Methyl Ester (FAME) derived from palm oil. Therefore, this research aimed to investigate the effect of reaction temperatures and durations as well as the interactions on the yield of FAME surfactants. The characteristics of the highest yield of FAME surfactants were also examined, including HLB (hydrophilic-lipophilic balance) value, surface tension, CMC (critical micelles concentration), density, and pH values. The study was conducted using Completely Randomized Factorial Design with three (3) factors, namely temperature (50 and 60 °C), reaction duration (60, 90, and 120 minutes), and the interaction. The results showed that the highest yield of 82.43% was produced at an interaction reaction temperature of 60 °C and a duration of 120 minutes. The characteristics of surfactants obtained were HLB value of 5.47, surface tension of 30.49 dyne/cm, capable of reducing surface tension by 73.20% (from 72.80 to 19.52 dyne/cm), CMC, density and pH values of 1.50% (v/v), 0.8757 g/cm<sup>3</sup>, and 6.86, respectively. These characteristics suggested that FAME has the potential for application as a water-in-oil (w/o) emulsifier. Moreover, the results could be applied to produce biodegradable surfactants using tropical oils through easy and simple technology.

**Keywords:** Characteristic; CMC (critical micelle concentration); FAME (fatty acid methyl ester); HLB; surface tension

## INTRODUCTION

Surfactants are unique chemical compounds characterized by both hydrophilic and lipophilic groups in a single molecule. This unique property enables the formation of a monolayer at interfaces and shows surface activity in heterogeneous mixtures. Although surfactants are commonly derived from petrochemicals, namely petroleum, the use of these resources proves challenging in terms of environmental degradation (non-biodegradable) and are categorized

as non-renewable resources (Akbari et al., 2018). Currently, surfactants sourced from renewable raw materials (RRMs) such as oleochemicals (plants, animals, and microorganisms) are being developed to meet consumer demand for more natural products. These materials conserve fossil resources and possess positive environmental impact, including easily degradable (biodegradable), non-toxic or with low toxicity, and harmless to human health. Among surfactants derived from oleochemicals, fatty acid methyl ester (FAME) is widely recognized due to the

distinctive characteristics (Berghuis et al., 2022; Rabiun et al., 2018).

FAME is obtained through a transesterification reaction between oil, serving as source of fatty acid, and methanol, facilitated by a catalyst. During this process, palm oil is selected as the raw material due to several attributes, including renewable natural resources, biodegradable, more economical, readily available, and a yield reaching 90.34% (Hutami and Ayu, 2015). The production of FAME can be influenced by several factors, such as the type of base catalyst, temperature, and reaction duration. Synthesis of FAME from palm oil using KOH and NaOH catalysts at temperatures of 40, 50, and 60 °C for 1, 2, and 3 hours has been conducted by Damayanti et al. (2013). The optimum synthesis using KOH catalyst was at 50 °C for 2 hours, and at 60°C for 1 hour during the use of NaOH catalyst. Jimmy et al. (2022) produced FAME from palm oil at a reaction temperature of 60°C for 1, 2, 3, and 4 hours using KOH and NaOH base catalysts. The results showed that the use of KOH catalyst was more effective, producing the highest yield at a reaction duration of 2 hours. The research showed also that an increase in reaction temperature led to a corresponding rise in effective collision frequency, along with the yield. However, at high temperatures ( $\geq 70$  °C), the yield decreased due to the reduction in methanol caused by evaporation (Prihanto et al., 2013). By using a longer reaction duration FAME yield increased due to a rise in the number of collisions between reactant molecules. An excessively long reaction duration of 3 hours was found to decrease the yield due to the formation of soap from the saponification reaction between residual free fatty acid and the catalyst (Mandei et al., 2020). Despite the numerous investigations, there is no information on the effect of reaction temperature (50 and 60 °C), reaction duration (60, 90, and 120 minutes), and the interactions on FAME production from palm oil with KOH catalyst. This information is needed to confirm the influence of temperature and reaction duration, serving as a basis for optimizing FAME from palm oil using KOH catalyst.

Based on the background above, this research aimed to investigate the influence of reaction temperature (50 and 60 °C), reaction duration (60, 90, and 120 minutes), and the interactions on FAME production from palm oil with KOH catalyst. Furthermore, FAME surfactants with the highest yield were characterized by measuring values of HLB (hydrophilic-lipophilic balance), surface tension, CMC (critical micelle concentration), density, and pH. The characterization was carried out to determine the application of FAME, which depends on the evaluated properties (Sampepana et al., 2015; Sarubbo et al., 2022; Shi et al., 2019; Wu et al., 2021).

## METHODS

### Materials

The materials used for FAME production included "KOI" brand palm cooking oil with a manufacturing date of December 20, 2020, obtained from one of the minimarkets in Pasuruan, East Java. Chemical reagents such as methanol, KOH,  $\text{MgSO}_4$ , and  $\text{NaHCO}_3$  were obtained from Merck (Darmstadt, Germany). The materials used for measuring surface tension, density, and pH included distilled water, FAME samples, and solutions of FAME with various concentrations, while all reagents used were of analytical grade.

### FAME Preparation

The production of FAME followed the method described by Damayanti et al. (2013). Briefly, 1% by weight of KOH catalyst was dissolved in 50 mL methanol, followed by the addition of 90.5 g palm. The mixture was refluxed at temperatures of 50 and 60 °C for 60, 90, and 120 minutes, which was left to stand for 2 hours in a separating funnel. After separation, the upper phase formed the methyl ester layer, which was washed using 50 mL of saturated  $\text{NaHCO}_3$  solution, and allowed to stand for 2 hours until separation. To the obtained upper phase, 1 g  $\text{MgSO}_4$  was added, centrifuged, and the collected supernatant constituted FAME. Quantification was carried out for methyl ester (ME) content using the formula based on the Indonesian National Standard (SNI) 7182:2015 on biodiesel (BSN, 2015) (Equation 1). FAME with the highest yield (determined using equation 2) was subjected to characterization in the subsequent stage.

$$ME \text{ levels} = \frac{100 (\text{saponification number} - \text{acid number} - 18.27(\text{total glycerol content}))}{\text{saponification number}} \quad (\text{Equation 1})$$

$$FAME \text{ Yield} = \frac{\text{Weight of supernatant}}{\text{Weight of raw materials (palm oil + catalys + methanol)}} \times ME \text{ levels} \quad (\text{Equation 2})$$

## Evaluation of FAME Surfactants Characteristics

### Surface tension test

Surface tension testing was conducted using the capillary tube method described by Wardana et al., (2019). Initially, density testing was performed on distilled water using a pycnometer (Iwaki, Japan). A total of 100 mL distilled water was poured into a petri dish with a diameter of 90 mm, followed by the insertion of a capillary tube with a diameter of 3 mm vertically and straight without touching the bottom of the dish. The rise of water in the capillary tube was measured at room temperature ( $25 \pm 2$  °C). Subsequently, testing was carried out on FAME solutions with concentrations

of 0.05%, 0.15%, 0.25%, 0.5%, 0.75%, 1%, 1.25%, 1.5%, 1.75%, and 2%, followed by surface tension calculation using Equation 3.

$$\text{Surface tension} = \frac{1}{2} \times r \times h \times \rho \times g \quad (\text{Equation 3})$$

Where:  $r$  = capillary tube radius (cm),  $h$  = height of rise (cm),  $\rho$  = liquid density ( $\text{g}/\text{cm}^3$ ), and  $g$  = gravity ( $\text{cm}/\text{s}^2$ ).

### CMC value

CMC value was determined by referring to Wardana et al. (2019), which included creating a graph between surfactants concentration (x-axis) and surface tension value (y-axis). The point where the addition of surfactants concentration reached a constant surface tension value showed CMC value in % (v/v).

### HLB value

HLB value was determined based on Wardana et al. (2019), with calculations using CMC values obtained through Equation 4.

$$\text{HLB} = 7 - 0,36 \ln [(100-\text{CMC})/\text{CMC}] \quad (\text{Equation 4})$$

### Density and pH

The density of FAME was determined using a pycnometer (Iwaki, Japan) based on the method described by Rahim and Prihatiningtyas (2017). Initially, an empty pycnometer was weighed, followed by the addition of FAME until full and sealed to prevent air escape. The pycnometer containing FAME was weighed, and the calculation was performed by comparing the difference in mass between the empty and pycnometer containing FAME.

In this research, pH of FAME was determined using a pH meter (WTW, Germany) according to the method described by Sutiko et al. (2020). FAME samples were placed in a glass beaker (Iwaki, Japan) and pH measurement was carried out using a pH meter.

### Research design

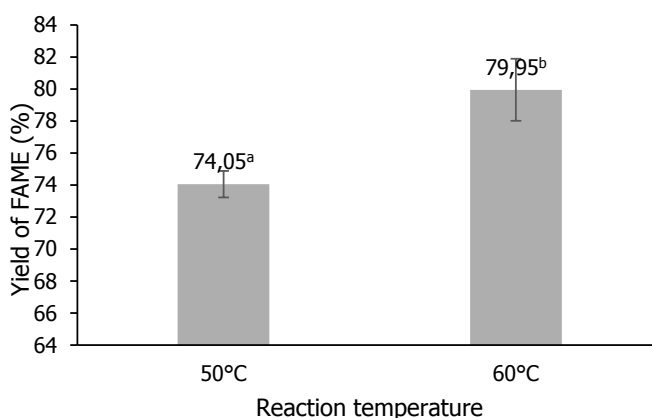
The research design used was Full Factorial Completely Randomized Design (FFCRD) with three (3) factors, namely temperature (50 and 60 °C), reaction duration (60, 90, and 120 minutes), and the interaction (temperature\*duration). Moreover, the interaction produced 6 levels of combinations, namely 50 °C for 60 minutes, 90 minutes, and 120 minutes, as well as 60 °C for 60 minutes, 90 minutes, and 120 minutes. Data were statistically analyzed using General Linear

Model (GLM) full factorial method at a significance level of  $p < 0.05$ . Further analysis was conducted using Duncan's Multiple Range Test (DMRT) and t-test at the same significance level to ascertain differences among reaction duration and temperature treatments. When there was a significant interaction effect, data analysis was conducted using Analysis of Variance (ANOVA) and DMRT for post hoc at a significance level of  $p < 0.05$ . Subsequently, samples with the highest yield were subjected to surfactants characteristic analysis with three repetitions.

## RESULTS AND DISCUSSION

### Effect of Reaction Temperature on FAME Yield

Figure 1 shows the influence of reaction temperature, where a significant increase in the FAME yield was observed when the temperature increased from 50 °C to 60 °C. According to Sipahutar and Tobing (2013), the yield increases as the reaction temperature rises because the conversion of methyl ester occurs perfectly when the reaction temperature reaches the boiling point of methanol at 65 °C. This shows that higher reaction temperatures can enhance the effective collision frequency between reactant molecules (palm oil and methanol), thereby increasing the rate of methyl ester formation, and reducing the required reaction time (Widyasanti et al., 2017). Lubes and Zakaria (2009) stated that the reaction should be conducted at the optimal temperature, lower than the boiling point of methanol (65 °C), to avoid a reduction in the amount of methanol available for reaction due to evaporation. When the reaction is carried out at



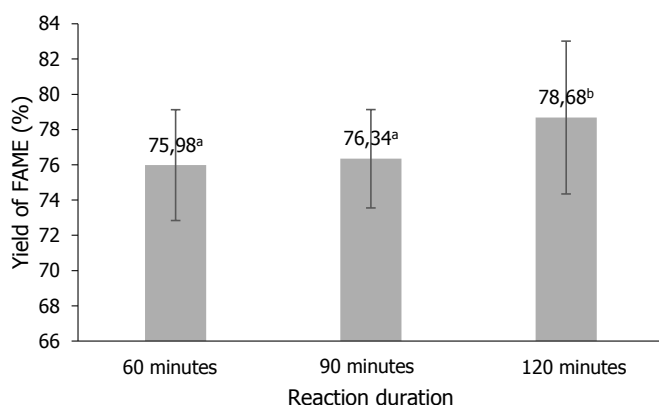
Description: numbers followed by different letters are significantly different at a significance level of  $p < 0.05$ ;  $n = 18$

Figure 1. Effect of reaction temperature on the average value of FAME yield

70 °C, methanol evaporates, resulting in a lower yield. Leung et al. (2010) and Leung and Guo (2006) stated that when the reaction temperature exceeded the optimum range (50–60 °C), a lower FAME yield was observed due to methanol evaporation, shifting the reaction towards saponification rather than transesterification. According to Chanakaewsomboon et al. (2020), undesired saponification reactions affect catalyst consumption, and phase separation, resulting in the formation of emulsions, capable of reducing FAME yield.

### Effect of Reaction Duration on FAME Yield

As shown in Figure 2, reaction duration of 60 and 90 minutes did not cause a significant increase in FAME yield, as similarly reported by Sipahutar and Tobing (2013). However, Eevera et al. (2009) produced FAME from various edible oils (coconut, palm, peanut, bran, and sesame oils) and non-edible oils (pongamia, cottonseed, and neem) using 1.5% (w/w) NaOH as catalyst, where a significant rise in yield was obtained when the reaction duration increased from 60 to 90 minutes. This difference was attributed to variations in the type and concentration of catalyst, as this research used 1% (w/w) KOH as catalyst. Generally, NaOH is more reactive than KOH due to the higher reactivity of sodium (Na) compared to potassium (K) (Mulana, 2011). According to Herawati et al. (2020) and Leung et al. (2010), the FAME yield decrease was attributed to the formation of soap (saponification) due to catalyst excess, which led to difficulties in separating FAME related to the formation of emulsified soap particles and water (Holilah et al., 2013; Leung et al., 2010).



Description: numbers followed by different letters indicate significantly different at a significance level of  $p < 0.05$ ;  $n = 18$

Figure 2. Effect of reaction duration on the average value of FAME yield

FAME yield significantly increased as reaction time changed from 90 to 120 minutes. Previous research established that longer reaction duration could increase the number of collisions between reactant molecules, thereby producing more products (Mandei et al., 2020). Similarly, previous studies that used watermelon seed oil and a catalyst of 0.19 g NaOH (Efavi et al., 2018), as well as virgin coconut oil (VCO) and a catalyst of 1.25% w/w NaOH (Mandei et al., 2020), reported increased FAME yield at reaction duration from 90 to 120 minutes. This showed that palm oil, watermelon seed oil, and VCO required a reaction duration of approximately 120 minutes for optimal FAME production. However, an extended reaction duration does not guarantee an increase in the amount of methyl ester produced because there is an optimal duration to achieve reaction equilibrium in producing FAME products (Mandei et al., 2020).

### Interaction between Temperature and Reaction Duration on FAME Yield

The statistical analysis showed that there was an interaction between temperature and reaction duration on FAME yield, as shown by a significance value of  $p = 0.000$ . The differences between the interaction temperature and reaction duration on FAME yield are presented in Table 1.

Table 1. Interaction of temperature and reaction duration on FAME yield

Interaction of temperature and reaction duration	Yield (%)
50 °C - 60 minutes	73,29±0,62 <sup>a</sup>
50 °C - 90 minutes	73,93±0,36 <sup>ab</sup>
50 °C - 120 minutes	74,93±0,43 <sup>b</sup>
60 °C - 60 minutes	78,67±0,59 <sup>c</sup>
60 °C - 90 minutes	78,75±0,05 <sup>c</sup>
60 °C - 120 minutes	82,43±0,06 <sup>d</sup>

Description: numbers followed by different letters are significantly different at a significance level of  $p < 0.05$ .

The interaction between temperature of 60 °C and a reaction duration of 120 minutes produced the highest FAME yield. This was due to the reaction temperature reaching the boiling point of methanol (<65 °C) and the optimal reaction duration, which increased the frequency as well as the number of effective collisions between reactant molecules to ensure perfect methyl ester conversion (Sipahutar and Tobing, 2013; Widyasanti et al., 2017).

### Characteristics of Fatty Acid Methyl Ester Surfactants with the Highest Yield

FAME surfactants with the highest yield were observed to appear golden yellow, as shown in Figure 3. Subsequently, the characteristics of these surfactants were determined, including surface tension value, CMC, HLB, density, and pH value.



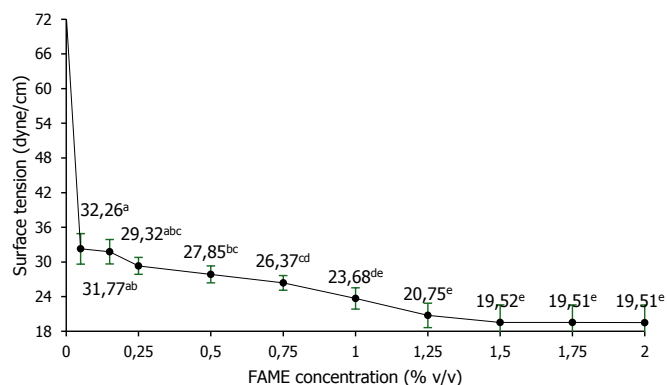
Figure 3. Appearance of FAME produced from palm oil at a temperature of 60 °C and a reaction duration of 120 minutes

### FAME Surface Tension

Based on the surface tension test, FAME with the highest yield showed a surface tension of  $30.49 \pm 0.98$  dyne/cm. Similarly, Freitas et al. (2011) and Phankosol et al. (2014) reported surface tension values of palm oil methyl ester at 31.89 dyne/cm and 30.13 dyne/cm, respectively. Generally, optimal surface-active biosurfactants typically have interfacial tension of less than 35 dyne/cm, such as sophorolipid (33 dyne/cm), rhamnolipid (30 dyne/cm), and surfactin (27 dyne/cm) (Akbari et al., 2018; Sarubbo et al., 2022). The surface tension value of FAME produced in this research was comparable to some commercial materials, such as methyl ester sulfonate (MES) at 30.13 dyne/cm and sodium lauryl sulfate (SLS) at 30.20 dyne/cm (Sampepana et al., 2015). This showed the potential of FAME surfactants developed in this research for commercial development.

### CMC (Critical Micelle Concentration)

CMC is an essential physicochemical parameter effective in characterizing pure surfactants regarding surface activity and ability to form aggregates (Perinelli et al., 2020; Shi et al., 2019). Specifically, CMC value is determined by observing the changes in physical and chemical properties of a solution that occur after the addition of surfactant at various concentrations. These



Description: 10 samples of FAME solution at various concentrations with 3 repetitions at each concentration

Figure 4. Effect of various concentrations of FAME surfactant on water surface tension. Numbers followed by the same letter are not significantly different ( $p < 0.05$ )

properties, which include conductivity, surface tension, osmotic pressure, and turbidity, pass through sudden changes after micelle formation (Karimi et al., 2015). CMC shows the minimum concentration of surfactants required to achieve the lowest surface tension. Furthermore, it is reached when surfactants start forming micelles, as shown by the absence of a further decrease in surface tension. In practical applications, surfactants with low CMC values are considered more advantageous than those with higher values (Sarubbo et al., 2022; Shi et al., 2019).

Figure 4 shows the reduction in surface tension of water after the addition of various concentrations of FAME. Based on the results, CMC was reached when the surface tension of water remained constant at  $19.52 \pm 3.05$  dyne/cm with a concentration of 1.50% (v/v). When the surfactant concentration is above the CMC, it shows constant surface tension activity because micelles begin to form, giving rise to a surfactant layer on the water surface (Belhaj et al., 2019). FAME surfactants obtained in this research showed the potential to reduce the surface tension of water by 73.20%, from 72.80 dyne/cm to 19.52 dyne/cm. This is similar to the ability of commercial glycerol esters (palmitate, stearate, oleate), which can reduce surface tension by 59–79% (Wardana et al., 2019). The ability to reduce surface tension is also found in several biosurfactants, such as surfactin (62.91%), rhamnolipid (58.79%), and sophorolipid (54.67%) (Sarubbo et al., 2022). Other surfactants capable of reducing surface tension include tert-butyl glucoside (37.26%), SDS (sodium dodecyl sulfate) (46.43%), and CPB (cetylpyridinium bromide) (47.94%) (Elarbi et al., 2020; Pawignya et al., 2018).

Table 2. CMC and HLB values for several surfactants

Surfactants	CMC	HLB	Source
Metil ester sulfonat (MES)	2,22%	13,36	Sampepana et al. (2015)
Commercial glycerol palmitate	0,50%	5,09	Wardana et al. (2019)
Commercial glycerol stearate	0,50%	5,09	Wardana et al. (2019)
Commercial glycerol oleate	0,50%	5,09	Wardana et al. (2019)
FAME	1,50%	5,47	Research results

According to Peltonen et al. (2001), CMC value increases along with the hydrophilic properties of surfactants and decreases as the hydrophobic properties of surfactants rise. The change in CMC value depends on the hydrophobicity of surfactants, which is the length of hydrophobic tail (Perinelli et al., 2020). This is consistent with HLB values of surfactants in Table 2, where those with a higher HLB value means it has a more significant number of hydrophilic groups compared to their lipophilic groups, so it has a higher CMC, and vice versa. According to Sarubbo et al. (2022), surfactants with lower CMC values are more advantageous for applications. This shows that FAME produced has the potential for development, as CMC value varies between the commercial surfactants.

### HLB (Hydrophilic-Lipophilic Balance)

HLB is the ratio between the hydrophilicity and hydrophobicity contained surfactants, expressed in the range of value 1–20, serving as an indicator of surfactants properties. Generally, surfactants with HLB values of 1.5–3 are used as antifoaming agents, 3–6 for water-in-oil (w/o) emulsifiers, which include glycerol, propylene glycol fatty acid, polyglycerol, and sorbitan fatty acid. Other functions include surfactants with 7–9 as wetting and spreading agents, 13–15 as detergents, 12–16 as oil-in-water (o/w) emulsifiers (proteins, phospholipids, potassium, sodium salts, and alginates), with 15–18 as solvents or solubilizing agents (Nakama, 2017; Ng and Rogers, 2018; Reningtyas and Mahreni, 2015).

The calculated HLB value for FAME was  $5.47 \pm 0.04$ , showing the presence of more lipophilic groups than hydrophilic, and potentially used as a water-in-oil (w/o) emulsifier. The use of FAME can replace commercial surfactants with similar HLB value, such as sorbitan esters (Span 40 (6.7), Span 60 (4.7), Span 80 (4.3)) and commercial glycerol esters (palmitate, stearate, oleate: 5.09) (Melo-Espinosa et al., 2015; Wardana et al., 2019).

### Density

Density is an essential parameter for surfactants due to the relevance in the application. Generally, surfactants require a density value with a small difference compared to solvent or product, to facilitate mixing and maintain the stability of emulsion (Uzwatania et al., 2017). In this research, FAME has a density of  $0.876 \pm 0.002$  g/cm<sup>3</sup>, consistent with a study of Ghazanfari et al. (2017), which reported that the density of FAME from palm oil ranges from 0.843 to 0.890 g/cm<sup>3</sup>. The investigation conducted by Hutami and Ayu (2015) also produced FAME with a density of 0.872 g/cm<sup>3</sup>. However, these FAME values have a lower density compared to commercial surfactants, such as sorbitan and glycerol esters, ranging from 0.941 to 1.041 g/cm<sup>3</sup>.

### pH

A neutral pH is essential in FAME to avoid taste alteration in food products (Nawangasasi, 2017). In this research, FAME produced has a pH of  $6.86 \pm 0.07$ , which is categorized in pH range (5-7.7) of some commercial surfactants commonly used in food products, such as Tween 80 and 60.

Based on the similar characteristics with sorbitan and glycerol esters, FAME can also be applied as emulsifier in several products. Specifically, FAME is used in chocolate products, ice cream, flavored-filled candies, and margarine (Awuchi et al., 2020; Barišić et al., 2019; Chen, 2015; Partridge et al., 2019). Since emulsifiers require a neutral pH, so there is no influence on the taste of food products when added as a food additive. However, the application of FAME with a low pH as an emulsifier tends to affect the taste of the food product, resulting in slight acidity (Kinyanjui et al., 2003; Mcglynn, 2016).

### CONCLUSION

In conclusion, this research showed the significant influence of reaction temperature, reaction duration,

and their interaction on the yield of FAME surfactants produced from palm oil using KOH as catalyst. The reaction conditions at 60 °C for 120 minutes resulted in the highest yield, reaching 82.43±0.06%. FAME surfactant produced under these conditions had an HLB value of 5.47±0.04, surface tension of 30.49±0.98 dyne/cm, and could reduce the surface tension of water from 72.80 dyne/cm to 19.52 dyne/cm (73.20%) at CMC of 1.50% (v/v), with a density of 0.876±0.002 g/cm<sup>3</sup> and pH value 6.86±0.07. These characteristics showed the potential for development as biodegradable surfactants using renewable materials, possessing characteristics comparable to some commercial surfactants. Moreover, further research was recommended including validation tests to confirm the presence of methyl ester compounds in FAME. Other tests and analyses should also be carried out to explore the potential application of FAME as emulsifier in a w/o (water in oil) emulsion system, including the characteristics and stability.

## CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest.

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