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Recent Advances in Biodiesel Production: Ultrasound-Assisted Interesterification of Palm Oil with Methyl Acetate

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Abstract. Currently, fossil fuels (non-renewable) are used continuously to meet bioenergy needs. Every year, there is an increase in bioenergy consumption, which will eventually lead to the depletion of fuel reserves. Therefore, it is necessary to have alternative and renewable fuels to substitute for the use of fossil fuels. One such application is the production of biodiesel, which serves as a substitute for diesel fuel. Biodiesel is made via the transesterification of triglycerides and methanol, with glycerol as a byproduct. The formation of glycerol holds no economic value and is deemed waste in biodiesel production, necessitating a separation process. Therefore, this research proposes an innovative method, specifically the non-alcoholic or interesterification reaction pathway, which involves three consecutive reversible reactions. In this reaction, methyl acetate is used to replace methanol, resulting in the production of triacetin instead of glycerol, which is completely soluble in biodiesel and offers a greater additional value than glycerol. To enhance the reaction rate and yield, potassium methoxide catalyst and ultrasound were used in this research. Meanwhile, to evaluate the influence of significant operational parameters on the interesterification reaction, experiments were carried out on different operating factors, namely a methyl acetate to oil molar ratio (3:1 to 25:1), reaction temperature (35 to 65 °C), catalysts (0.5 to 2% (w/w)), and interesterification time (1 to 30 minutes). It has been observed that the optimal yield is achieved at a 15:1 molar ratio, with a 1% catalyst amount, a reaction temperature of approximately 55 °C, and a reaction time of 5 minutes, resulting in a yield of 81.26%. Furthermore, a kinetic study was conducted to determine the activation of energy and rate constantly suitable for the second-order approximation. The reaction rate constant is 0.287 L/(mol.min) at an operational temperature of 55 °C, and the resulting activation energy is 50.50 kJ/mol.

Keywords: Biodiesel, Bioenergy, Interesterification, Kinetics, Ultrasound

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Currently, energy demand in Indonesia is increasing due to population growth, economic expansion, and technological advancements. The country's energy consumption is reliant on non-renewable fossil fuels, with annual increases in usage threatening to deplete these reserves (Moreau & Vuille, 2018). Consequently, it is imperative to develop alternative, environmentally friendly, and renewable fuels to replace fossil fuels. One promising alternative is biofuel derived from vegetable oil, such as biodiesel, which can serve as a substitute for diesel fuel (Chew and Bhatia, 2008; Tan *et al*., 2011a). By employing bioenergy technology, Indonesia has the potential to enhance its energy security and make a significant contribution to supplying environmentally friendly energy to local communities and the global market. In recent years, biodiesel has become one of the most promising energy sources. This is because biodiesel has biodegradable, non-toxic, and renewable properties, produces less carbon monoxide gas, is free of sulfur, and has a higher cetane number and flash point. Therefore, this biodiesel will become a more environmentally friendly fuel (Devita, 2015; Hadi, 2009; Hasan *et al*., 2012).

Indonesia, an agricultural nation situated on the equator, boasts abundant vegetable resources that can be extensively refined into biofuels, such as biodiesel. This renewable and abundant vegetable source will be extracted for oil and further processed into biodiesel. Various types of vegetable oils utilized in biodiesel production involve sunflower (Casas *et al*., 2011a; Stamenkovic *et al*., 2010a), soybean (Nunes and Castilhos, 2020a; Tang *et al*., 2013), peanut (Silveira Junior *et al*., 2016), coconut (Suryanto *et al*., 2015), palm (Ali and Tay, 2013a; Ansori and Mahfud, 2022a, 2021; Chew and Bhatia, 2008; Kareem *et al*., 2017a), jatropha (Badday et al., 2013a; Lin and Chen, 2017a), nyamplung *(Kusuma et al*., 2018a; Qadariyah *et al*., 2019), kapok seed (Suryanto *et al*., 2018), used cooking oil (Nurmawati *et al*., 2024) and others. One of the raw materials that has the potential to be developed into biodiesel is palm oil. Palm oil ranks among the most widely consumed and produced oils in Indonesia. In 2017, 41.98 million tons of palm oil (including palm kernel oil and crude palm oil) were produced in Indonesia, establishing it as the world's top producer of palm oil (Varqa, 2017).

Various research projects have been done on the derivation of palm oil to biodiesel synthesis (Ali and Tay, 2013b; Ansori and Mahfud, 2021; Chew and Bhatia, 2008; Kareem *et al*., 2017b). In general, there are two basic stages in the manufacture of biodiesel: esterification to form esters from fatty acids and transesterification to form biodiesel and glycerol (a side product) from triglycerides. When methanol is used as the reactant in esterification and transesterification reactions, it reacts with oil to form biodiesel (Badday *et al*., 2013b; Bankovic-Illic *et al*., 2012a; Lin and Chen, 2017b; Qadariyah *et al*., 2019). Biodiesel is typically manufactured through the transesterification of triglycerides and methanol, facilitated by the addition of a catalyst. However, this method has several weaknesses, including the slow mass transfer in the reaction caused by immiscible reactants (Sajjadi *et al*., 2015a). This causes the transesterification process to run slowly, resulting in a biodiesel yield that is less than optimal. Additionally, a separation and washing process must be implemented to distinguish between biodiesel and glycerol, a by-product. This is because the presence of glycerol can be regarded as an impurity that causes high density and has no economic value when mixed in biodiesel. The existence of the purification process certainly adds to the higher production costs in industrial processes (Calero *et al*., 2015; Kusuma *et al*., 2018b; Shikhaliyev *et al*., 2018).

Therefore, to overcome the presence of glycerol, a new method is used that can produce biodiesel more effectively (without producing glycerol). The process involves an interesterification reaction (non-alcoholic method) comprising three consecutive reversible reactions. It entails the reaction between palm oil and methyl acetate in the presence of a catalyst, which involves three consecutive reversible reactions. In this reaction, methyl acetate is used to displace methanol, resulting in the generation of triacetin instead of glycerol, which is completely soluble in biodiesel and offers a greater additional value than glycerol. The presence of triacetin has no hostile impact on the end quality of the fuel mixture and still meets good quality standards for biodiesel fuel, even though the addition of triacetin exceeds 10% in weight. Adding triacetin to biodiesel is also hoped to lower the pour point and enable it to withstand frigid weather (Casas *et al*., 2011b, 2010; Medeiros *et al*., 2018a; Saka and Isayama, 2009a). Triacetin is also a mixed material used as a plasticizer for polymers and an agent for gelatinizing (Garcia *et al*., 2008; Maddikeri *et al*., 2013). The benefits of the product derived from the interesterification process in this research include the production of biodiesel and triacetin. Triacetin serves as both a biodiesel additive and an anti-knock agent, eliminating the need for additional separation processes.

The interesterification process in this research was conducted in a batch reactor. This is because the batch process does not require a lot of equipment, and it is easier to control the reaction than the continuous process. Additionally, the selection of energy sources to be utilized in the biodiesel production process is also crucial, as the production process must consider the effectiveness of the energy sources employed. Various techniques have been employed in biodiesel production to enhance reaction speed and yield, such as using catalysts *(*Ali & Tay, 2013b; Ansori *et al.,* 2019; Casas *et al.,* 2011d; Medeiros *et al.,* 2018b; Tang *et al*., 2013), supercritical conditions (without catalyst) *(*Saka and Isayama, 2009b; Tan *et al*., 2011b), enzymes (Kareem *et al*., 2017b; Subhedar and Gogate, 2016a), ultrasound (Badday *et al*., 2013b; Deshmane *et al*., 2009a; Hingu *et al*., 2010; Kashyap *et al*., 2019c; Mahamuni & Adewuyi, 2009a; Medeiros *et al*., 2018b), microwave (Ansori *et al*., 2019; El Sherbiny *et al*., 2010; Lin & Chen, 2017b), and others (Muharja *et al*., 2024) . One alternative technology that has been widely developed for the manufacture of biodiesel through interesterification reactions is the chemical interesterification method, which utilizes a catalyst and ultrasonic waves. In interesterification, the process differs from transesterification in that one ester exchanges its alcohol group with another ester. This exchange causes the mixture of reactions to transition from polar to non-polar, leading to partial insolubility of the potassium methoxide catalyst (Casas *et al*., 2011b). Additionally, the reactants that do not mix in the interesterification reaction can be facilitated by the mixing process using ultrasound. The use of ultrasound will help emulsify immiscible reactants in the presence of microturbulence, ensuring that the reactants and catalysts are well mixed (Badday *et al*., 2013b; Bankovic-Illic *et al*., 2012b).

By using ultrasonic waves, the waves propagate through the water medium, leading to bubble growth (nucleation), which enables the use of water as a reagent medium. The enormous energy of ultrasound due to cavitation, thermal (high local system temperature and pressure), and mechanical effects will increase the reaction rate (Badday *et al.*, 2013b; Deshmane *et al.*, 2009b; Sajjadi *et al.*, 2015b). Furthermore, ultrasound can be utilized across numerous types of reactions, including catalytic, ion exchange, enzyme-catalyzed, and biphasic (Sancheti and Gogate, 2017), and also interesterification reactions (Kashyap *et al.*, 2019a; Maddikeri *et al.*, 2013b; Medeiros *et al.*, 2018b; Subhedar & Gogate, 2016b). Heating in the interesterification process using ultrasonic wave radiation exhibits different characteristics compared to conventional heating. This is because the ultrasound method can enhance reaction kinetics, particularly in slow reactions, thereby reducing the reaction time required and increasing the yield of biodiesel. Several research results have shown that heating using ultrasonic wave radiation in organic chemical synthesis needs a shorter time than conventional heating (Ansori and Mahfud, 2022b; Kashyap *et al.*, 2019c; Maddikeri *et al.*, 2013; Subhedar and Gogate, 2016b). Therefore, the use of ultrasonic waves and potassium methoxide catalyst in the interesterification reaction is the most effective method to accelerate the interesterification process, increase the reaction rate, reduce energy consumption, and minimize catalyst usage, thereby producing a higher yield of biodiesel.

To enhance the effectiveness of biodiesel production through interesterification using ultrasound, a biodiesel yield model is developed to determine the optimal kinetic model for ultrasound-assisted interesterification (UAI). The kinetic model is important because it can be employed to forecast biodiesel yield before the interesterification process is conducted and to design an effective control system in many industrial biodiesel production applications (Casas et al., 2011c; Kashyap *et al.*, 2019c; Maddikeri *et al.*, 2013). Thus, this research introduces a green chemistry approach by replacing methanol with methyl acetate, thereby minimizing toxicity and generating triacetylglycerol as a valuable byproduct instead of glycerol. Additionally, the application of ultrasound cavitation enhances energy efficiency, achieving high biodiesel yields (90–93%) at ambient temperatures and within shorter reaction times (30–40 minutes), compared to conventional reactions requiring 60–80°C and 2–4 hours. To maximize the benefits of ultrasound in biodiesel production, a kinetic study was conducted to model biodiesel yield under ultrasound-assisted interesterification (UAI) conditions. This modeling is essential for predicting biodiesel production outcomes and designing effective control strategies in industrial applications. The study also compares two simplified kinetic models to determine the most suitable representation of UAI kinetics, validated through rate constants, coefficients of determination, and calculated activation energy values.

MATERIALS AND METHOD

Materials and Catalyst

The raw material, palm oil, for this research was sourced from a local market in Surabaya. The chemicals used, methyl acetate and methanol, are of analytical purity (Merck KGaA, 99.9%), and phosphoric acid (ortho, 85%) was obtained from Brattaco Chemicals

& Co. The catalyst employed was 99% potassium methoxide (Zhishang Chem).

Experimental Procedure

The materials for the ultrasound-assisted interesterification method in biodiesel production were outlined in a prior study (Ansori & Mahfud, 2021). The variable used in this research is the methyl acetate-to-oil mole ratio (3:1 to 25:1). The two reactants were put into a three-neck flask and mixed. The mixture was then warmed to the specified temperature (35-65 °C) using a heating mantle. Once the temperature stabilized, a mixture containing methanol and potassium methoxide at a specific amount of catalysts (0.5-2% (w/w)) was introduced into the flask.

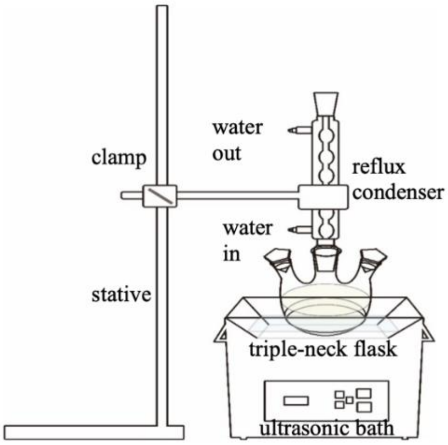

Fig. 1: Schematic apparatus for UAI

The flask containing the reactants and catalysts was then placed in the ultrasound-assisted interesterification apparatus (Figure 1) for the planned reaction times (1-30 minutes). At the end of each reaction period, the reaction was stopped by adding 0.1 mL of phosphoric acid. The product mixture was transferred to a separatory funnel and washed with 20 mL of warm distilled water, resulting in two distinct layers. The upper layer was collected into a beaker and dried in an oven at approximately 110 °C. The final phase involved analyzing the obtained product, with the biodiesel yield determined using Eq. (1).

$$\text{Yield} = \frac{\text{product mass X \% purity level}}{\text{palm oil mass}} \text{x } 100\% \quad (1)$$

Kinetic Modeling

The interesterification process consists of three sequential and reversible reactions. Triglycerides are transformed into monoacetin-diglycerides, then into diacetin-monoglycerides, and finally into triacetin, with methyl ester molecules being released at each step (Casas *et al.*, 2011b). This author (Casas *et al.*, 2011d) has proposed a kinetics model to describe the triglyceride interesterification reaction, using methyl acetate. The author's approach (Casas *et al.*, 2011d) suggests that if an excessive methyl acetate composition is used, it can be hypothesized that the forward reaction follows pseudo-first-order kinetics, while the reverse reaction adheres to second-order kinetics. Likewise, (Narvaez *et al.*, 2007) also stated that if an excess of methyl acetate is used in an interesterification reaction, the rate constant (k) for the reverse reaction is presumed to be zero. Consequently, the interesterification, which was originally reversible, can then be viewed as an irreversible reaction. The constant of reaction rate is defined by the Arrhenius equation, which is expressed as follows:

$$k(T) = \left(Ae^{-Ea/R.t}\right) \quad (2)$$

Where, Ea is the activation energy in joules per mole (J/mol), A represents the pre-exponential factor or frequency factor, R is the universal gas constant (8.314 J/(K·mol)), $k(T)$ is the rate constant dependent on temperature (mol/L), and T is the absolute temperature in kelvin (K).

The provided Equation represents a first-order kinetic model (Stamenkovic *et al.*, 2010b).

$$\frac{dX_A}{dt} = r_A = k_1(1 - X_A) \quad (3)$$

Upon integration, Eq. (3) yields Eq. (4):

$$-\ln(1-X_A) = k_1 t + C \quad (4)$$

Here, X_A denotes the biodiesel yield with purity, t is the reaction time (min), r_A is the triglyceride reaction rate (mol/L.min), k_1 is the kinetic constant (L/mol.min), and C is the integration constant. Plotting $-ln(1-X_A)$ against t should result in a straight line if the model is accurate, with k_1 as the slope and C as the y-intercept.

The second-order kinetic model is described by Eq. (5) (Casas *et al.*, 2011d):

$$-\frac{dC_{TG}}{dt} = r_{TG} = k_2.C_{TG}{}^2 \quad (5)$$

Where, r_{TG} is the reaction rate (mol/L.min), C_{TG} is the molar concentration of triglycerides (mol/L), k_2 is the kinetics constant (L/mol.min), and t is the interesterification time (min). The kinetic model of a second-order reaction is proposed as Eq. (6):

$$\frac{1}{C_{TG0}} = k_2.t + \frac{1}{C_{TG}} \quad (6)$$

Where, C_{TG0} is the beginning of the triglyceride's molar concentration (mol/L). The mapping of $\frac{1}{C_{TG}}$ against t will form a straight line if the model is correct. The slope value will be a k_2 value.

Eq. (6) can be reorganized for conversion (yield of biodiesel with purity) and will become Eq. (7) *(Maddikeri et al.*, 2013):

$$\frac{X}{(1-X)} = C_{TG0}.k_2.t + C \quad (7)$$

Where X is the conversion of triglycerides (yield of biodiesel with purity) to t (time), and C is an integral constant. In the interesterification process, the triglyceride concentration can be determined using biodiesel yield information and Eq. (6). If the model holds, plotting $\frac{X}{(1-X)}$ against $t$will result in a linear relationship. The slope represents the k_2 value, and the intercept represents the Cvalue. Eq. (2) can be reformulated to:

$$ln(k) = \frac{-Ea}{RT} + ln(A) \quad (8)$$

Plotting $ln(k)$ against $\frac{1}{T}$ allows us to determine the slope and intercept. The slope aligns to $\frac{-Ea}{R}$, enabling the calculation of the activation energy.

RESULTS AND DISCUSSION

Temperature Profile in the Reactor

To investigate the effect of the amount of ultrasonic wave energy emitted by the ultrasound-assisted interesterification method on biodiesel production, examining the temperature rise per unit of time is necessary. It should be noted that this review of the temperature profile only considers the use of optimal parameters for the variables employed, specifically a methyl acetate-to-oil molar ratio of 15:1 and a 1% concentration of catalyst (w/w) for 60 minutes. The temperature profiles of various powers are presented in Figure 2.

Figure 2 shows that the temperature increases linearly over time during the interesterification process. The longer reaction time will result in a smaller temperature rise every minute. In addition, the heating rate is also studied by regressing each line, yielding the equation y = 0.5669x +

304.04. Here, the heating rate is the first derivative of the relationship between temperature and time, so that the heating rate β=dT/dt is obtained at 0.5669 K/min.

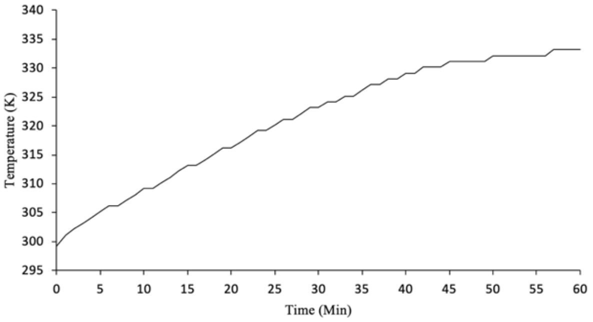

Fig. 2: Temperature profile in UAI

Effect of Molar Ratio on Yield

The quantity of methyl acetate utilized in the reaction will affect the yield of biodiesel obtained. Typically, an excess of methyl acetate is used to enhance the reaction to form the desired product. Where less methyl acetate is used, the biodiesel production cost will be reduced. To determine the optimal molar ratio of methyl acetate to oil, an experiment was conducted with varying molar ratios, including 3:1, 6:1, 9:1, 12:1, 15:1, 20:1, and 25:1.

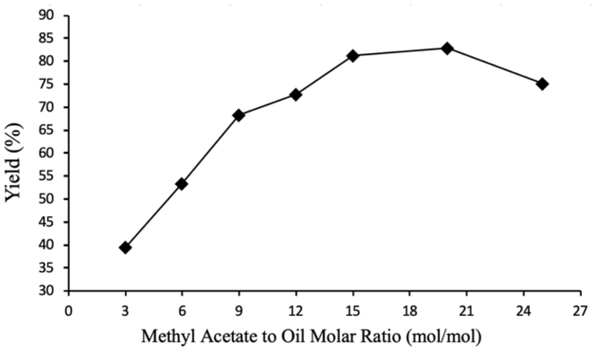

Fig. 3: Effect of reactant ratio on yield (1%wt, 55 °C, 5 min)

Figure 3 shows that a higher molar ratio yields increased amounts. This increase in ratio enhances the interaction frequency between reactants, facilitating the reaction towards product formation and improving the conversion. Nevertheless, this rate of increase plateaus at a molar ratio of 20:1, beyond which the yield starts to decline. The decline is attributed to the dilution of products at higher molar ratios, which may initiate the reverse reaction and reduce biodiesel conversion, as noted by the authors (Tan *et al.*, 2011b). Similarly, another author (Visioli *et al.*, 2018) also reported that excess methyl acetate could contribute to low reagent concentrations in the medium and hinder reaction progress. In addition, the low ester yield was caused by the presence of unreacted methyl acetate, which limited the reaction balance. This is according to the research of Subhedar and Gogate (2016b), which explains that a greater molar ratio led to a greater biodiesel conversion, for example, at a molar ratio of 9:1; however, a further increase resulted in a lower biodiesel yield. Likewise, the research conducted by Ribeiro *et al.* (2018) reported that using a higher molar ratio would yield increased levels of FAME and triacetin. They then opted to explore further under the optimal condition of a 20:1 molar ratio, as increasing it to 40:1 did not result in a significant increase in the levels of methyl esters and triacetin obtained. Moreover, they observed that the molar ratio had a small effect on the expected rate of reaction. With a higher molar ratio, there is increased dilution of the reagents, which in turn reduces the rate of interesterification. The use of ultrasound in interesterification required less excess methyl acetate to achieve high yields, with a yield of 96.1% obtained during a 3-hour interesterification (Subhedar and Gogate, 2016b). Therefore, the optimal molar ratio was 15:1, yielding 81.26% at a temperature of 55 °C with a 1% catalyst amount (w/w) and a 5-minute interesterification time. The use of a large methyl acetate molar ratio will only increase the production cost.

Effect of the Amount of Catalyst on Yield

In a study aimed at determining the optimal conditions for a chemical reaction, it is crucial to investigate how the concentration of the catalyst (in this case, the amount of palm oil) affects biodiesel yield. The use of catalysts significantly impacts production costs, as they accelerate the reaction rate by lowering activation energy during the formation of methyl esters. To assess this effect, catalyst amounts of 0.5%, 1%, 1.5%, and 2% (w/w) were tested with reactant molar ratios of 9:1, 15:1, and 20:1.

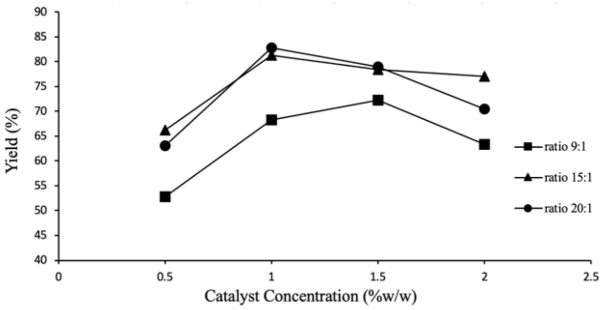

Fig. 4: Effect of catalyst on the biodiesel yield (55 °C, 5 min)

As shown in Figure 4, the increase and decrease in yield with the methyl acetate to oil molar ratio exhibit a similar pattern, especially at molar ratios of 15:1 and 20:1. In contrast, the molar ratio of 9:1 displays a distinct pattern. However, the biodiesel yield obtained at a 20:1 or 15:1 ratio is much higher than that obtained at a 9:1 ratio. At molar ratios of 15:1 and 20:1, the highest biodiesel yields were achieved with a 1% (w/w) catalyst amount, reaching 81.26% and 82.85%, respectively. Conversely, at a molar ratio of 9:1, the highest yield of 72.29% was obtained with a catalyst amount of 1.5% (w/w). Increasing the catalyst amount from 0.5% to 1% across all molar ratios led to an increase in biodiesel yields. However, for molar ratios of 15:1 and 20:1, yields tended to decrease as the catalyst amount increased from 1% to 2%. For the 9:1 molar ratio, increasing the amount of catalyst from 1% to 1.5% increased yield, but yield decreased with the amount of catalyst above 1.5%. This is coherent with the study by Maddikeri *et al.* (2013). They have demonstrated that a 1% concentration of potassium methoxide catalyst can increase conversion to 90%. Still, a supplementary concentration of catalysts of 1 to 1.25% didn't result in a meaningful improvement in the yield of biodiesel. Meanwhile, if the concentration used is smaller (0.5%), it will result in a low yield of biodiesel due to incomplete conversion of oil into biodiesel. Likewise, the study reported by Medeiros *et al.* (2018b) found that a catalyst amount of 1% resulted in the greatest triglyceride conversion, with a value of 94.64%, in the interesterification process of cotton oil. Otherwise, an elevation of the catalyst amount exceeding 1.17% did not result in a meaningful increase in biodiesel conversion. Similarly, the research that has been reported by (Kashyap *et al.*, 2019a) related to the interesterification process of Karanja oil with a catalyst concentration of 0.5-1.5%wt using ultrasound. They reported that the optimal yield value was obtained at a catalyst concentration of 1%wt, corresponding to 91%.

Thus, catalyst addition in the interesterification process can increase the yield of biodiesel obtained. Moreover, it was also found that the most optimal amount of catalyst was 1% (w/w), where further increasing the catalyst concentration did not increase the conversion but rather reduced the yield of biodiesel obtained (Ansori and Mahfud, 2022b). In addition, the fewer catalysts used, the more economical the production cost will be. According to El-gendy *et al.* (2015), a high concentration of catalyst can slow down the reaction rate and cause resistance to mass, thereby reducing biodiesel conversion and yield.

Effect of Temperature on Yield

Temperature is a necessary factor in an interesterification reaction. An optimal temperature is necessary because a reaction has a specific optimal temperature. Where a temperature that is too low can prevent a reaction from occurring, if the temperature is too high, it will result in the evaporation of the reactants used, yielding an optimal yield. To investigate the influence of temperature on biodiesel yield, temperature variables of 35, 45, 55, and 65 ^{o}C were used, along with reactant molar ratios of 15:1 and 20:1.

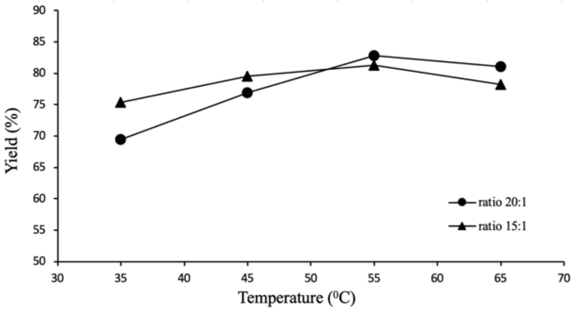

Fig. 5: Effect of temperature on the biodiesel yield (1%wt, 5 min)

Another variable that affects the yield of biodiesel is temperature. Based on Figure 5, the greater the reaction temperature, the greater the yield obtained. Escalating the reaction temperature from 35 to 55°C resulted in higher biodiesel yields. The elevated temperature promotes more frequent contact between reactants, thereby accelerating the reaction rate and increasing the conversion of oil into biodiesel. Moreover, the high operating temperature can reduce the level of the cavitation effect (Hingu *et al.*, 2010; Maddikeri *et al.*, 2013). Meanwhile, the research by Kashyap *et al.* (2019c) suggests that an increased temperature substantially lowers the surface tension and viscosity of the liquid, thereby facilitating the occurrence of the cavitation process. It was found that the ideal operational temperature for the interesterification process using ultrasonic waves was 50°C.

However, raising the temperature from 55 to 65°C may result in a decrease in the biodiesel yield. This is because the evaporation of methyl acetate leads to its loss, filling the condenser or reflux, which reduces the association of oil and methyl acetate. This reduced contact diminishes the reaction rate and prevents the optimal conversion of reactants into products. Additionally, the oil that has been converted into biodiesel can undergo further reactions at increasing temperatures and increasing pressures (Ansori and Mahfud, 2022b; El Sherbiny *et al.*, 2010; Medeiros *et al.*, 2018b). According to the research conducted by Saka and Isayama (2009b), they reported lower FAME yields at 380 ^{o}C compared to 350 ^{o}C due to the damage of unsaturated fatty acids at higher temperatures. Meanwhile, according to Casas *et al.* (2011b), who converted sunflower oil into biodiesel using a potassium methoxide catalyst, a temperature of 50 ^{o}C was chosen as the operating condition. Therefore, the most optimal temperature is 55 ^{o}C because it produces an optimal biodiesel yield (81.26%) for a 15:1 molar ratio with a 1% wt amount of catalyst during an interesterification time of 5 minutes. Keep in mind that higher temperatures can reduce the time it takes to achieve optimum conversion.

Effect of Reaction Time on Yield

In a study aimed at determining the optimal conditions for a chemical reaction, it is essential to determine the impact of interesterification time on yield, as this is related to the process's effectiveness. To investigate the effect of interesterification time on biodiesel yield, interesterification times ranging from 1 to 30 minutes were used at temperatures of 35 °C and 55 °C. The last variable that affects the yield of biodiesel is reaction time (interesterification time), which is shown in Figure 6.

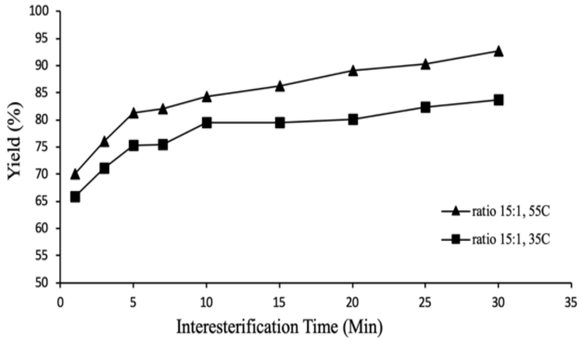

Fig. 6: Effect of time on yield (15:1, 1%wt)

From Figure 6, both temperatures exhibit a similar trend, where longer reaction times result in enhanced biodiesel yield. However, the most substantial increase in biodiesel yield occurred within the 1 to 5 minutes. This is because the longer the interesterification time, the more triglycerides will react with methyl acetate, resulting in a higher yield of biodiesel. According to the research carried out by Medeiros *et al.* (2018b), the interesterification process occurs rapidly when using a catalyst and ultrasound, with the reaction converting triglycerides into biodiesel at a rate of up to 50% within 1 minute. Meanwhile, for a period of 5 to 10 minutes, the yield of biodiesel decreased, specifically from 81.26% to 84.34% (at 55 °C) and from 75.32% to 79.43% (at 35 °C). Moreover, with extended times from 10 until 30 minutes, there is a noticeable increase in biodiesel yield at 55°C compared to 35°C. Specifically, at 30 minutes, the biodiesel yield reached 93.15% at 55°C compared to 83.65% at 35°C.

According to Figure 6, the biodiesel yield remains stable between an interesterification time of 10 to 30 minutes, particularly at 35°C. This suggests that biodiesel production reaches nearly equilibrium within this timeframe, making additional time less effective in increasing biodiesel yield (Ansori *et al.*, 2019; Kusuma *et al.*, 2018b). This aligns with the theory that describes the interesterification reaction as reversible. Where the reaction can shift towards the reactants (to the left side) when they have achieved equilibrium, so it will reduce the yield produced because the reaction speed is getting smaller due to the decrease in reactants. However, suppose the interesterification reaction is conducted at a shorter time. In that case, it has been worried that the yield of biodiesel obtained is small due to incomplete reactions or increased formation of monoglycerides and diglycerides (Casas *et al.*, 2011d, 2011b; Kusuma *et al.*, 2018b). The impact of using ultrasound as a heating method in biodiesel production has been widely investigated. Ultrasonic waves can accelerate organic synthesis much faster than conventional heating by enhancing the reaction rate (Kashyap *et al.*, 2019c; Maddikeri *et al.*, 2013; Medeiros *et al.*, 2018b; Sajjadi *et al.*, 2015b). Therefore, it can be explained that the interesterification process using ultrasound is more about time selectivity (the time used) to produce optimal biodiesel.

Kinetic Analysis

In the production of biodiesel using ultrasound-assisted interesterification (UAI), the yield of biodiesel obtained increases rapidly at the beginning and then decreases with increasing interesterification time.

Differences in the yield of biodiesel produced can be illustrated by plotting the yield against interesterification time. This can then be used to identify and study the kinetic model of biodiesel production through interesterification reactions. This kinetics model is used to analyze the best results from the research conducted, specifically the methyl acetate-to-oil molar ratio of 15:1 for 30 minutes, using a catalyst-to-oil concentration of 1% (w/w) at different temperatures, namely 35 °C and 55 °C. It should be noted that this analysis was conducted by incorporating a thermocouple into a triple-neck round-bottom flask to maintain a constant temperature.

The reaction kinetics were analyzed using conversion data or biodiesel yield with purity versus time, and the rate constant value was adjusted to determine the rate constant value at different temperatures using the kinetic models of first and second order. The assumption of an irreversible reaction by adding excess methyl acetate allows for a lower triglyceride concentration when equilibrium is reached in the interesterification, thus analogous to the irreversible reaction (Maddikeri *et al.*, 2013).

Studying the 1st model of the interesterification reaction, it can be done by plotting between $-\ln(1-X)$ against t, and the second-order kinetic model it can be done by plotting $X/(1-X)$ versus t which is presented in Figure 7. Furthermore, linearization of the plot is carried out to obtain slope and intercept values to be applied to investigate the value of k_1 for 1st-order, as well as on the 2nd model to determine the value of k_2. To determine a model that can represent the experimental results of the interesterification process, the R^2 and Root Mean Square Deviation (RMSD) values of each kinetic model used can be examined. The kinetic model is said to represent the experimental results well if it has an R^2 value close to 1 and the smallest RMSD value.

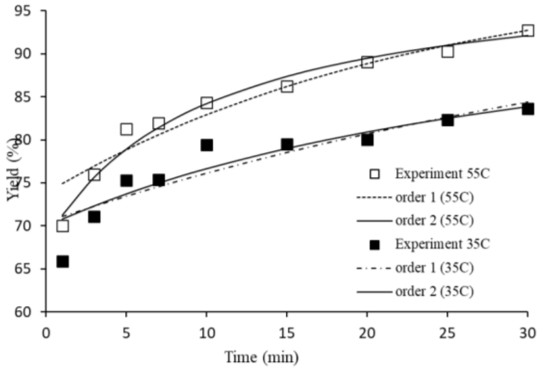

Fig. 7: Comparison of experiment data and 1st and 2nd models for reactants molar ratio 15:1 and 1%wt catalyst.

From Figure 7, the slope value of each resulting line represents the reaction rate constant at the corresponding temperature. To calculate the reaction rate constant value on the 1st order (k_1), use the slope value generated by the regression equation in Figure 7(a) and use Eq. (4).

Meanwhile, to calculate the second-order reaction rate constant value (k_2), the formula in Eq. (7) and the slope value obtained from the regression equation in Figure 7(b) are used. The reaction rate constant (k), slope, intercept, correlation coefficient (R^2), and RMSD value obtained from the plot above are shown in Table 1.

As shown in Figure 7 and Table 1, the second-order kinetic model for the interesterification reaction at 35 °C using a UAI method has a higher coefficient of determination (R^2 = 0.9119) and a lower RMSD value than the first model. This suggests that the second model provides a better fit to the experimental results of the interesterification reaction in biodiesel production. Specifically, at a temperature of 55°C, the second model exhibits a higher coefficient of determination (R^2 = 0.9691) compared to the first model (R^2 = 0.9581). Additionally, the root mean square deviation (RMSD) value of 1.0446 for the second model is lower than that of the first model (RMSD = 1.9801). Therefore, it can be concluded that the second model provides a better fit for the interesterification reaction, as indicated by its higher coefficient of determination (R^2) compared to the first kinetic model. Furthermore, Figure 7(c) also indicates that the second model yield is more in line with the experimental yield than the first model, both at 35 °C and 55 °C. This explains that the interesterification reaction of palm oil and methyl acetate using ultrasound follows the second model well (Casas *et al.*, 2011d; Kashyap *et al.*, 2019c; Maddikeri *et al.*, 2013). In general, the two kinetic models are quite good at representing the model.

Table 1. Kinetic models constants obtained

Reaction temperature	**Kinetic Models**			
	First-order		Second-order	
35°C	Slope	0.0213	Slope	0.0954
	k_1	0.0213	K_2	0.0863
	R^2	0.8569	R^2	0.9119
	RMSD	2.2447	RMSD	2.0265
55°C	Slope	0.0428	Slope	0.3172
	k_1	0.0428	K_2	0.2871
	R^2	0.9681	R^2	0.9691
	RMSD	1.9801	RMSD	1.0446

**Calculated by Ms. Excel (*) k_1 (min^{-1}); k_2 (L/(mol.min)*

According to Table 1, the second model exhibits a higher R^2 value and a lower RMSD value than the first kinetic model; therefore, the interesterification reaction kinetic model is better represented by the second model. Additionally, it was observed that the second-order kinetic rate constant value, calculated using Eq. (7), increased from 0.086 to 0.287 L/(mol·min) as the temperature rose from 35 to 55 °C during the interesterification process. Where an increase in temperature causes increased molecular agitation, so that the collisions that occur also increase, and the reactions that occur are faster. Furthermore, it will increase the reaction rate due to the high turbulence generated in the reaction mixture caused by the cavitation effect (Kashyap *et al.*, 2019c; Maddikeri *et al.*, 2013; Sajjadi *et al.*, 2015b). The results of reaction rate constants by some authors (Maddikeri *et al.*, 2013) in the interesterification process of waste cooking oil and methyl acetate using ultrasound show that an increase in temperature from 30°C to 50°C can result in a rate constant value increase from 0.22 to 0.93 (L/mol.min). Meanwhile, another author (Kashyap *et al.*, 2019c) also reported that increasing the reaction temperature increases the rate constant value, specifically by 0.16 (L/mol.min) and at 30 °C and by 0.36 (L/mol.min) at 50 °C. Likewise, this author (Medeiros et al., 2018b) also reported their research results regarding the rate constant value, which increased from 0.424 (L/mol.min) at 30°C to 2.122 (L/mol.min) at 50°C. The kinetics of this interesterification reaction were found to correspond to a second-order mechanism (Casas *et al.*, 2011d) in the interesterification process of sunflower oil using conventional heating (Maddikeri *et al.*, 2013) and on the waste cooking oil using the ultrasound method.

Using the rate constants value presented in Table 1, and temperature controlled at 35°C and 55°C, the activation energy value can be attained by plotting the data from the reaction rate constant value vs the inverse of temperature, as shown in Figure 8. The slope value of this line (Figure 8) indicates a ratio between universal gas constant (R) and activation energy ($-E_a$), whereas linear coefficients represent the pre-exponential factor. Therefore, by performing the calculations according to Eq. (8), the activation energy is 50.5034 kJ/mol, and the pre-exponential factor value is 3.14 x 10^7 L/(mol.min). The value of activation energy for palm oil interesterification using ultrasound falls within the range of activation energy values reported by some authors (Casas et al., 2011d) in their studies on sunflower oil interesterification kinetics. Where the activation energy value ranges from 24.318 to 88.551 kJ/mol. Meanwhile, Maddikeri *et al.* (2013) also reported a higher activation energy of 58.170 kJ/mol, and another author (Medeiros *et al.*, 2018b) reported an even higher activation energy of 65.541 kJ/mol. In contrast, lower activation energy values have been reported by others (Kashyap *et al.*, 2019c), specifically 32.68 kJ/mol and 47.46 kJ/mol (Kashyap *et al.*, 2019a). The Arrhenius equation generated to obtain the rate constant of the interesterification reaction of palm oil and methyl acetate using ultrasound is given by Eq. (9):

$$k = 3{,}14\text{x}10\text{\^{}}7\ \exp(-\frac{50{,}5034}{RT}) \tag{9}$$

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CONCLUSIONS

This study evaluated the performance of ultrasound-assisted interesterification (UAI) for converting palm oil into biodiesel. The results demonstrated that higher molar ratios of reactants and elevated reaction temperatures significantly enhanced biodiesel yield. The optimal operating conditions were identified as a methyl acetate-to-oil molar ratio of 15:1, 1% catalyst loading (w/w), a temperature of 55 °C, and an interesterification time of 5 minutes, under which a maximum yield of 81.26% was achieved. The cavitation effect induced by ultrasound effectively overcomes mass transfer limitations, thereby accelerating the interesterification reaction and reducing overall energy consumption. As a result, this method contributes to lower operational costs by minimizing the required amounts of reactants, temperature, catalyst, and reaction time. The kinetic analysis revealed that a second-order kinetic model best fits the experimental data, offering a higher coefficient of determination (R^2) and a lower root mean square deviation (RMSD) compared to the first-order model. In the second-order kinetics, the reaction rate constant increased with temperature, reaching 0.287 L/(mol·min) at 55 °C, with an activation energy of 50.5034 kJ/mol. Overall, the use of methyl acetate and potassium methoxide in the UAI process demonstrates a promising, efficient, and sustainable approach for biodiesel production from palm oil. Future research should explore the use of other renewable feedstocks, investigate the reusability and stability of heterogeneous catalysts under ultrasound conditions, and assess the techno-economic feasibility and life cycle impacts of scaling up the UAI process.

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NOMENCLATURE

c_p : specific heat at constant pressure [J kg^{-1} K^{-1}]

D : diameter [m]

I : thermal inertia [J m^{-2} K^{-1} s$^{-1/2}$]

k : thermal conductivity [W m^{-1} k^{-1}]

n : normal vector

Nu_{θ} : local Nusselt number

$\underline{Nu}$: average Nusselt number

p : pressure [Pa]

Re : Reynolds number

T : absolute temperature in kelvin (K)

t : time (s)

k(T) is the rate constant dependent on temperature (mol/L),

k_1 : 1st order reaction constant (min^{-1});

k_2 : 2nd order constant (L/(mol.min)

X : Reaction conversion (-)

R : universal gas constant (8.314 J/(K·mol)

E_a : activation energy in joules per mole (J/mol)

A : pre-exponential factor or frequency factor

C : molar concentration (mol/L),

C_{TG} : molar concentration of triglycerides (mol/L),

C_{TG0} : Initial molar concentration of triglycerides (mol/L),

r_{TG} : reaction rate (mol/L.min),

R^2 : coefficient of determination

RSMD: root mean square deviation

x, y : Cartesian coordinate (m)

ρ : density (kg m^{-3})

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