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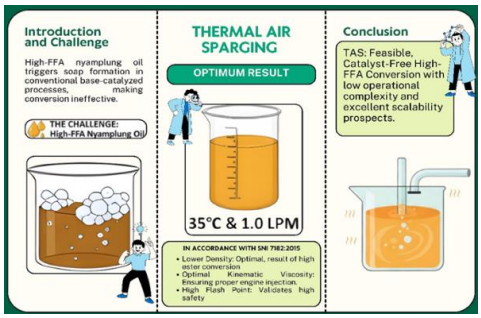
Optimization of biodiesel synthesis process from Nyamplung (*Calophyllum inophyllum*) oil using thermal air sparging method

Nasrul Ilminnafik[1]*, Dani Hari Tunggal Prasetiyo[1], Welayaturromadhona[2], Audiananti Meganandi Kartini[3], Bekti Palupi[4], Suyitno[5]

[1] Department of Mechanical Engineering, Universitas Jember, Jember 68121, **Indonesia**
[2] Department of Petroleum Engineering, Universitas Jember, Jember 68121, **Indonesia**
[3] Department of Environmental Engineering, Universitas Jember, Jember 68121, **Indonesia**
[4] Department of Chemical Engineering, Universitas Jember, Jember 68121, **Indonesia**
[5] Department of Mechanical Engineering, Universitas Sebelas Maret, Surakarta 57126, **Indonesia**
✉ nasrul.teknik@unej.ac.id



Highlights:

- Thermal Air Sparging (TAS) integrates heating and microbubble mixing for biodiesel synthesis.
- TAS provides an energy-efficient and low-pressure alternative for sustainable biodiesel production.
- Microbubble-assisted mixing enhances heat and mass transfer, improving biodiesel efficiency.

Abstract

This study investigated the optimization of biodiesel production from nyamplung (*Calophyllum inophyllum*) oil using a Thermal Air Sparging (TAS) reactor with NaOH catalyst. Crude oil containing 18.2% free fatty acids (FFA) underwent a two-stage acid esterification to reduce the acidity below the 2% threshold required for efficient alkaline transesterification. To assess the combined effects of thermal and hydrodynamic variables, a 3^2 full factorial experimental design with three replications was used, encompassing a reaction temperature of 30–40 °C and a hot air flow rate of 1.0–2.0 L min^{-1}. The produced biodiesel was characterized for density, kinematic viscosity, flash point, and heating value according to ASTM D4052, D445, D93, and D240, respectively. These values were then evaluated against SNI 7182:2015 specifications. Optimal operating conditions were achieved at 35 °C and a flow rate of 1.5 L min^{-1}. Reaction temperature emerged as the primary factor influencing biodiesel conversion, while hot air flow rate predominantly affected interfacial contact and residence time. Under these conditions, the biodiesel exhibited a density of 0.867 g·cm^{-3}, a kinematic viscosity of 4.67 cSt, a flash point of 183.2 °C, a calorific value of 10,283.20 cal·g^{-1}, and a conversion yield of 85.43%. This performance is attributed to the combined effects of heat transfer and dispersion driven by microbubbles, which continuously renew the interfacial surface area between the reactants and promote a more homogeneous reaction environment at atmospheric pressure and relatively low temperature. In contrast to mechanical stirring systems and other high-energy intensification methods, the TAS configuration allows for intensification of the thermofluidic process, reducing reaction time, stabilizing fuel properties, and lowering overall energy consumption. These results demonstrate the potential of TAS as a practical and scalable method for converting non-edible oils with high free fatty acid content into biodiesel through a low-energy process.

Keywords: Thermal air sparging; Nyamplung oil; Thermofluidic process intensification; Microbubble mixing; Energy-efficient transesterification

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1. Introduction

Global energy needs are currently dominated by fossil fuels, which account for more than 80% of global primary energy consumption by 2023 [1], [2], [3], [4]. High dependence on fossil fuels

Nomenclature

Symbol	Description	Unit	Symbol	Description	Unit
C	Viscometer calibration constant	$mm^2 \cdot s^{-2}$	U_c	Combined uncertainty	–
m	Mass of sample	g	V	Volume of biodiesel	cm^3
m_b	Mass of produced biodiesel	g	V_{NaOH}	Volume of NaOH used in titration	mL
m_o	Initial mass of oil	g	Y	Biodiesel yield	%
m_f	Mass of fuel in calorimeter	g	ρ	Density	$g \cdot cm^{-3}$
m_w	Mass of water in calorimeter	g	v	Kinematic viscosity	$mm^2 \cdot s^{-1}$
N	Normality of NaOH solution	–	ΔT	Temperature rise during calorimetric test	°C
Q_c	Heat correction for ignition wire and wick	J	FFA	Free fatty acid content	%
t	Flow time in viscometer	s	HHV	Higher heating value	$cal \cdot g^{-1}$

leads to increased emissions of carbon dioxide and fine particulate matter in the atmosphere, which directly contribute to global warming and environmental degradation This situation has encouraged various research initiatives to find alternative energy sources that are clean, renewable, and sustainable [5], [6]. Among various alternative energy sources, biodiesel is one of the most promising candidates because it has chemical characteristics similar to diesel, is easily biodegradable, has a high cetane number, and contains internal oxygen that can increase combustion efficiency [7], [8], [9], [10].

As a country with abundant tropical biodiversity, Indonesia has significant potential for biodiesel production from non-edible vegetable oils [11]. One promising feedstock is nyamplung oil (*Calophyllum inophyllum*), which can grow on marginal land with high oil productivity [12], [13], [14]. However, the main challenge in utilizing nyamplung oil lies in its high free fatty acid (FFA) content, which often exceeds 20%. This high FFA content is a major obstacle in the base-catalyzed transesterification process because it triggers a saponification reaction that consumes the catalyst and inhibits the formation of methyl esters [15], [16], [17]. In general, FFA levels above 2% can reduce conversion efficiency and cause difficulties in the product separation process [18], [19], [20]. Therefore, a biodiesel synthesis method is needed that is more adaptive to the characteristics of oils with high FFA without sacrificing process efficiency and product quality.

Several previous studies have explored non-catalytic and heat-intensive approaches to handle high-FFA oils. Makarevičienė *et al*. [21], Reported that uncatalyzed biodiesel synthesis under supercritical conditions was capable of converting high-FFA oils without the need for a pre-esterification step. Similarly, Asri *et al*. [22] studied the kinetics of non-catalytic transesterification reactions under subcritical and supercritical conditions and found that conversion efficiency was significantly affected by temperature and the molar ratio of alcohol to oil. Although promising, these methods require very high temperatures and pressures, high energy requirements, and specialized equipment, making them difficult to implement on a medium-scale or small-scale industry.

As a simpler and more efficient alternative, the Thermal Air Sparging (TAS) method was introduced. The principle of this method is based on the flow of hot air into a mixture of oil and methanol through a sparger pipe, which produces microbubbles. These bubbles act as both a heating medium and a mixing agent, increasing the efficiency of heat and mass transfer between the phases [23]. Unlike supercritical methods, microwave-assisted heating, or ultrasonic cavitation, TAS operates at low pressures and moderate temperatures, while still providing high mixing intensity [24], [25], [26]. In this study, the TAS process was combined with the use of a NaOH base catalyst to accelerate the formation of methoxide ions (CH_3O^-) and increase the conversion rate of triglycerides to methyl esters. This approach not only reduces energy requirements but also suppresses the formation of soap residue and simplifies the product phase separation [27], [28].

The performance of the TAS process is significantly influenced by reaction temperature and hot air flow rate, as these two parameters determine thermal stability, turbulence intensity, and the rate of methyl ester formation. Inappropriate parameter settings can lead to excessive methanol evaporation, incomplete reactions, or even thermal degradation of the product. However, research systematically examining the effect of temperature and hot air flow rate on the quality of biodiesel synthesized from nyamplung oil using the base-catalyzed TAS method is still very limited.

Based on this research gap, this study aims to analyze the effect of reaction temperature and hot air flow rate on the physical characteristics of biodiesel produced from nyamplung oil (*Calophyllum inophyllum*) using the Thermal Air Sparging method with a base catalyst. The main parameters analyzed include density, kinematic viscosity, flash point, and heating value, in

accordance with the SNI 7182:2015 quality standard. The results of this study are expected to determine the optimum operating conditions in the TAS process, as well as provide a scientific basis for the development of biodiesel production technology that is efficient, environmentally friendly, and suitable for application on a small and medium industrial scale.

Conventional biodiesel production, which relies on mechanical stirring, either with magnetic stirring or top stirring, is intrinsically limited by limited interfacial contact between the immiscible oil-alcohol phases and by non-uniform thermal distribution within the reaction medium. Consequently, the transesterification process is slow, often resulting in incomplete conversion and fluctuating fuel quality [29]. High-energy process intensification techniques, such as ultrasonic irradiation, microwave-assisted heating, and cavitation-based reactors, have demonstrated their ability to accelerate reaction kinetics and achieve biodiesel yields exceeding 95%. However, these approaches are generally associated with high energy requirements, complex reactor architectures, and high investment costs, which limit their practical scalability [30], [31]. Electrocatalytic transesterification has also emerged as an alternative pathway to increase reaction rates through the application of an external electric field; however, its development remains hampered by issues related to electrode durability and the lack of integration into sustainable and scalable reactor configurations [32].

In contrast to these approaches, the Thermal Air Sparging (TAS) method introduces a distinct thermofluidic mechanism in which heat transfer and mixing are simultaneously generated through the dispersion of hot microbubbles under atmospheric conditions. This configuration allows the formation of a dynamically renewed interfacial area while maintaining moderate thermal intensity and low energy consumption [33]. Despite these advantages, systematic studies elucidating the combined influence of thermal and hydrodynamic conditions of the gas flow on the conversion behavior of nyamplung oil with high free fatty acid content are still very limited. Therefore, this study aims to evaluate the interaction between reaction temperature and hot air flow rate through a factorial experimental framework and to establish TAS as a low-energy thermofluidic intensification pathway for efficient biodiesel production.

2. Method

This research was conducted through an experimental approach. The research was designed to identify and analyze the relationship between the main process variables of reaction temperature and hot air flow rate. The reaction temperature and hot air flow rate were used as treatments in the study of the characteristics of biodiesel synthesized from nyamplung oil (*Calophyllum inophyllum*). The reactor system used had a maximum capacity of 2 liters, made of corrosion-resistant stainless steel, with an inner diameter of 140 mm and a total height of 350 mm. The reactor was equipped with an 800 W electric air heater, an air compressor, and a digital flowmeter to regulate the hot air flow. Air was flowed through an insulated copper pipe to a porous sparger with a hole diameter of 0.5 mm installed at the bottom of the reactor. This design allows the formation of microbubbles that act as a heating medium and an efficient liquid interphase mixer.

Hot air was flowed into the reactor until it reached the desired operating temperature, then a mixture of esterified oil and methanol was introduced into the system with a molar ratio of oil to methanol of 1:6 (mol/mol The reaction was conducted at atmospheric pressure for 10 minutes, with varying reaction temperatures (30 °C, 35 °C, and 40 °C) and hot air rates of 1.0; 1.5; and 2.0 $L \cdot min^{-1}$, respectively, according to a two-way factorial design. The system was controlled using a digital PID controller to maintain temperature stability of ±0.5 °C throughout the process. The entire procedure was designed to replicate the results and meet fuel testing standards according to SNI 7182:2015. The SNI 7182:2015 standard can be seen in **Table 1**.

The research was conducted in three main stages, namely (1) pre-treatment of raw materials through degumming and esterification processes to reduce free fatty acid levels, (2) biodiesel synthesis using the Thermal Air Sparging (TAS) method assisted by a homogeneous NaOH catalyst, and (3) physical and thermal characterization of the resulting biodiesel to assess its compliance with the SNI 7182:2015 quality standard. These stages were carried out systematically to obtain a comprehensive understanding of the effect of operating conditions on the performance of the conversion process, while ensuring the traceability of experimental data and the validity of the analysis results.

Table 1. Biodiesel standards according to SNI 7182:2015

No.	Parameter	Standard	Unit
1	Density at 40 °C	850-890	$kg \cdot m^{-3}$
2	Kinematic viscosity at 40 °C	2.3-6.0	$mm^2 \cdot s^{-1}$
3	Flash point	Min. 100	°C

2.1. Materials and Sample Preparation

Nyamplung oil is obtained from the mechanical pressing of dried seeds that have been dried at 60 °C for 48 hours. Mechanical pressing of dried nyamplung seeds yielded an oil recovery of 49 wt% relative to the dry seed mass. This result demonstrates the substantial lipid potential of *Calophyllum inophyllum* as a non-edible biodiesel feedstock and aligns with values commonly reported in the literature (40–70 wt%). The high oil yield observed in this study provides adequate raw material for subsequent esterification and transesterification processes, thereby supporting the feasibility of the proposed TAS-based conversion pathway [34], [35]. The crude oil is filtered using Whatman No. 42 filter paper to separate solid particles. Supporting chemicals consist of phosphoric acid (H_3PO_4, 85%), sulfuric acid (H_2SO_4, 98%), and technical methanol (CH_3OH, 99.8%). All materials are used without additional purification. The research scheme for the synthesis of nyamplung biodiesel in this study can be seen in **Figure 1**.

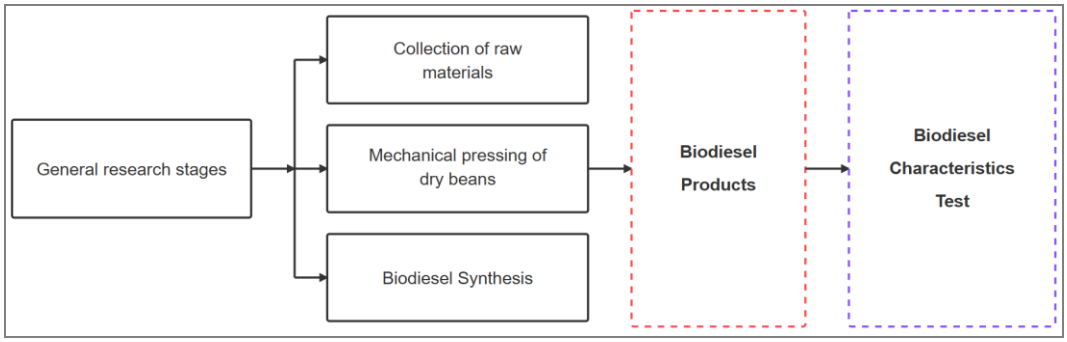

Figure 1. Research scheme for the synthesis of nyamplung biodiesel

2.2. Degumming Stage

The degumming stage was carried out to remove gum, phospholipids, and polar impurities that could inhibit the rate of chemical reactions. A total of 200 mL of crude nyamplung oil was put into a glass reactor equipped with a magnetic stirrer and a hotplate. H_3PO_4 solution was added as much as 2% of the oil mass, then the mixture was stirred at a speed of 600 rpm for 30 minutes at a temperature of 60 °C. After the process was complete, the mixture was allowed to settle until two layers formed. The upper layer (gum-free oil) was taken, washed with warm water, and dried at 105 °C for 20 minutes. The chemical reaction of gum removal through phospholipid decomposition can be observed in Eq. (1), where R-PO_4 represents the phosphate group in the oil bound to fatty acids.

$$\text{R-PO}_4 + \text{H}_3\text{PO}_4 \rightarrow \text{R-OH} + (\text{PO}_4\text{H})_2 \quad (1)$$

2.3. Free Fatty Acid Esterification Stage

The esterification stage is carried out to reduce the free fatty acid content in nyamplung oil from the degumming process so that it does not exceed 2%. The reaction is carried out between free fatty acids and methanol using sulfuric acid (H_2SO_4) as a homogeneous catalyst. This process takes place in a stainless steel reactor equipped with an air sparger at the bottom to produce microbubbles as a mixing medium and local heating. A total of 200 mL of degumming oil is mixed with methanol with a mass ratio of 1: 2 (oil: methanol, m/m). The H_2SO_4 catalyst is added as much as 1% of the oil mass. The process is run at a temperature of 35 °C for 10 minutes with a hot air flow rate of 1.5 L·min^{-1} to create micro-bubble turbulence that accelerates mass transfer. After the reaction is complete, the mixture is allowed to settle for ±8 hours until two layers are formed. The top layer (crude methyl ester) was removed and reprocessed in a second esterification step using identical conditions to ensure the FFA content dropped below 2%.

$$\text{R–COOH} + \text{CH}_3\text{OH} \xrightarrow[35\ ^\circ\text{C}]{\text{H}_2\text{SO}_4} \text{R–COOCH}_3 + \text{H}_2\text{O} \quad (2)$$

where R–COOH is free fatty acid, CH_3OH is methanol, and R–$COOCH_3$ is the methyl ester produced by the reaction. This step results in a significant reduction in FFA because free fatty acids are converted to esters through acid catalysis. The FFA content in the oil before and after esterification was analyzed using the acid-base titration method (ASTM D5555-95) with phenolphthalein as an indicator. The FFA value was calculated using Eq. (3).

$$\text{FFA}(\%) = \frac{V_{\text{NaOH}} \times N_{\text{NaOH}} \times 28.2}{m_{\text{sample}}} \quad (3)$$

where, V_{NaOH}= volume of NaOH solution used in titration (mL); N_{NaOH}= normality of NaOH solution; m_{sample}= mass of oil titrated (g); 28.2 is the conversion factor for oleic acid.

The results of the FFA analysis are used as the basis for determining the success of the esterification stage; the process is declared successful if the FFA value is <2%, which indicates that the oil has met the requirements for the subsequent biodiesel synthesis process using the Thermal Air Sparging method.

2.4. Transesterification Reaction in Thermal Air Sparging (TAS) Method

The transesterification process is a key step in the conversion of triglycerides into methyl esters (biodiesel) and glycerol through the reaction between vegetable oil and alcohol. In this study, this stage was carried out using the Thermal Air Sparging (TAS) approach with the help of sodium hydroxide (NaOH) as a base catalyst. The selection of NaOH is based on its high ability to accelerate the formation of methyl esters through the ionization mechanism of methanol into methoxide ions (CH_3O^-), which then react with triglycerides to form glycerol and methyl esters. The transesterification process was carried out in a stainless steel reactor with a maximum capacity of 2 liters, an inner diameter of 140 mm, and a height of 350 mm. The system was equipped with an 800 W electric air heater, an air compressor, and a digital flowmeter to control the flow rate of hot air. Hot air was flowed into a fine-pored sparger with a hole diameter of 0.5 mm, thus forming microbubbles that function as a heating medium and dynamic stirrer in the oil-methanol liquid phase. The reactant mixture consisted of oil from the esterification stage and methanol with a molar ratio of 1:6 (mol/mol). NaOH catalyst was added at 1% of the oil mass, then the mixture was stirred using hot air (thermal sparging) for 10 minutes. The process was carried out at three temperature variations (30 °C, 35 °C, and 40 °C) and three variations of hot air flow rates (1.0 L·min^{-1}; 1.5 L·min^{-1}; and 2.0 L·min^{-1}). All experiments were carried out at atmospheric pressure conditions, with temperature control using a PID digital controller with an accuracy of ±0.5 °C. Mechanistically, the transesterification reaction takes place in three reversible stages, first the conversion of triglycerides to diglycerides, second diglycerides to monoglycerides, and third monoglycerides to glycerol. Each stage produces one methyl ester molecule as the main product. The overall reaction can be observed in Eq. (4).

$$C_3H_5(OOCR)_3 + 3CH_3OH \xrightarrow[\text{NaOH}]{\text{TAS}} 3RCOOCH_3 + C_3H_5(OH)_3 \quad (4)$$

where, $C_3H_5(OOCR)_3$: triglycerides in nyamplung oil; CH_3OH: methanol; $RCOOCH_3$: methyl ester (biodiesel); $C_3H_5(OH)_3$: glycerol (by-product).

After the process is complete, the reaction mixture is allowed to settle by gravity until two clearly separated layers are formed. The upper layer contains biodiesel, while the lower layer contains glycerol, residual catalyst, and a small amount of methanol. The biodiesel layer is then separated using a separating funnel, washed with warm water until the pH is close to neutral 7,

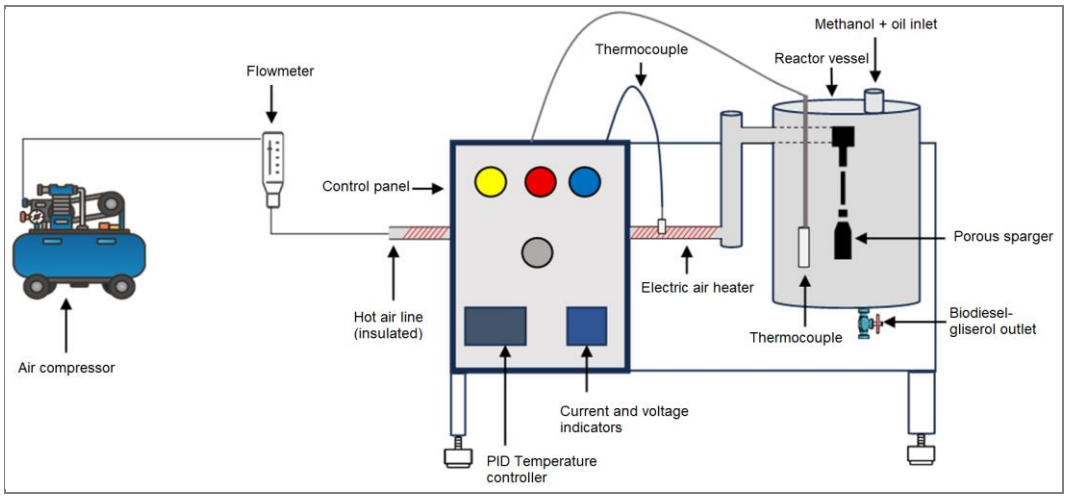

Figure 2. Schematic of thermal air sparging reactor system

and dried at 105 °C for five minutes to remove residual water. The dried biodiesel obtained is sto red in a closed container before testing its physical, thermal, and chemical properties. The thermal sparging scheme in the transesterification process of triglycerides into methyl esters and glycerol with the help of a NaOH base catalyst can be seen in **Figure 2**.

2.5. Experimental Design

This research design used a two-way factorial (3×3) approach to assess the effect of reaction temperature and hot air flow rate on the quality of biodiesel produced from nyamplung (*Calophyllum inophyllum*) oil. The two main factors studied were reaction temperature, namely 30 °C, 35 °C, and 40 °C, and hot air flow rate, respectively, of 1.0 L·min^{-1}; 1.5 L·min^{-1}; and 2.0 L·min^{-1}. These ranges were selected based on preliminary studies that demonstrated the thermal stability of the system and optimum reaction efficiency within these ranges. Each treatment combination was run for 10 minutes with an oil to methanol molar ratio of 1:6 (mol/mol) and the addition of 1% NaOH catalyst by oil mass. All experiments were conducted at atmospheric pressure, while other factors such as mixture volume, reaction time, and reactant ratio were kept constant to ensure uniformity of operating conditions. This approach allows a systematic analysis of the simultaneous influence of thermal and hydrodynamic variables on the performance of the Thermal Air Sparging process. Beyond assessing variable interactions, the factorial framework facilitates the identification of the primary factor influencing biodiesel conversion. Each treatment was repeated three times to ensure data reliability and minimize experimental deviations. The test results were then analyzed descriptively and comparatively, by examining the change patterns and consistency between biodiesel quality parameters including yield, density, kinematic viscosity, flash point, and heating value. Optimum operating conditions were identified based on the combination of treatments that produced biodiesel with physical and thermal characteristics closest to the SNI 7182:2015 standard specifications, while also demonstrating high process stability and maximum conversion efficiency. The experimental matrix of the 3×3 factorial design can be seen in **Table 2**.

Table 2. Experimental matrix of the 3×3 factorial design

Run	Reaction temperature (°C)	Hot air flow rate (L·min^{-1})
1	30	1.0
2	30	1.5
3	30	2.0
4	35	1.0
5	35	1.5
6	35	2.0
7	40	1.0
8	40	1.5
9	40	2.0

2.6. Calculation Equation

The properties of the biodiesel fuel produced in this study were analyzed using standard testing procedures. Density was measured according to ASTM D4052, kinematic viscosity according to ASTM D445, and flash point according to the Pensky–Martens closed cup method (ASTM D93). The calorific value was determined using an adiabatic bomb calorimeter according to ASTM D240. The measured values were then compared with the requirements specified in the Indonesian biodiesel standard SNI 7182:2015. A data reduction scheme was developed to quantitatively assess the conversion performance and quality of the produced fuel. Biodiesel yield serves as an indicator of the degree of conversion of triglycerides to methyl esters. Density and kinematic viscosity are used to characterize the flow and atomization behavior, which affect combustion performance. The calorific value represents the intrinsic energy content of the fuel. Collectively, these parameters provide a comprehensive basis for evaluating the effectiveness of the Thermal Air Sparging process. The mathematical expressions used for these calculations are presented in the following subsections.

2.6.1. Biodiesel yield

Biodiesel yield is a quantitative parameter that indicates the success rate of the process of converting vegetable oil into methyl ester (biodiesel). The yield value is expressed as a percentage (%) and represents the ratio of the mass of biodiesel produced to the mass of the initial oil used in the reaction process. Mathematically, the yield can be calculated using Eq. (5) [36].

$$Y = \frac{m_b}{m_o} \times 100 \tag{5}$$

Eq. (5) is used to determine the total conversion efficiency of the transesterification process, both in the Thermal Air Sparging (TAS) system. The higher the Y value, the greater the fraction of oil successfully converted to methyl ester, thus indicating the effectiveness of the reaction process

and the efficiency of product separation. In practice, factors that influence the yield value include the molar ratio of methanol to oil, reaction temperature, hot air flow rate, and process duration. A high yield value (approaching 100%) indicates that most of the triglycerides have reacted to form biodiesel, while a low yield indicates that there are still remaining triglycerides, diglycerides, or monoglycerides in the oil phase that have not been completely converted. Measurement of the biodiesel mass (m_b) is carried out after the purification and drying stages of the product, to ensure that there is no influence of residual methanol or water on the calculation results. Thus, the biodiesel yield value reflects not only the results of the chemical reaction, but also the efficiency of the separation and purification stages carried out throughout the synthesis process.

2.6.2. Density (ρ)

Biodiesel density is a physical parameter that plays an important role in determining combustion characteristics and engine performance. The density value describes the mass of fuel per unit volume at certain temperature conditions, which directly affects the spraying process, atomization, and the amount of fuel injected into the combustion chamber. Density testing in this study was carried out at a temperature of 40 °C using a digital hydrometer in accordance with the ASTM D4052–19 standard, to obtain accurate results and can be compared with the SNI 7182:2015 specifications. The biodiesel density value was calculated using the following Eq. (6) [37].

$$\rho = \frac{m}{V} \tag{6}$$

where, ρ= biodiesel density (g·cm^{-3}); m= biodiesel mass (g); V= biodiesel volume (cm^3).

During the measurement, the biodiesel sample is placed in a calibrated graduated cylinder, and its density is read directly by a digital sensor after the system temperature stabilizes at 40 °C. The measurement results are then corrected against the reference temperature to eliminate the effect of volume changes due to thermal expansion. Theoretically, increasing temperature causes a decrease in density due to molecular expansion in the liquid phase. Therefore, controlling the test temperature is crucial to ensure representative and comparable measurement results between samples. The ideal biodiesel density value is generally in the range of 0.86–0.90 g·cm^{-3}, where deviations from this range may indicate the presence of impurities, methanol residue, or residual glycerol content that has not been completely separated during the purification stage. Thus, density measurement serves not only as a basic physical parameter but also as an indicator of the overall quality of the biodiesel synthesis results and the stability of its chemical composition.

2.6.3. Kinematic viscosity (v)

Kinematic viscosity is an important parameter that reflects a fluid's resistance to flow due to internal shear forces. This value significantly influences the performance of the fuel injection system, the atomization process, and the formation of the air-fuel mixture in the combustion chamber. In this study, viscosity testing was conducted using an Ostwald viscometer at 40 °C, following the ASTM D445–19 standard procedure, to ensure accurate measurement results comparable to international biodiesel specifications. The kinematic viscosity value (v) was calculated using Eq. (7) [38].

$$v = C \times t \tag{7}$$

where, C= viscometer calibration constant (mm^2·s^{-2}); t= time flows through glass (s).

The unit of kinematic viscosity is expressed in centistokes (cSt), where $1\ \text{cSt} = 1\ \text{mm}^2\cdot\text{s}^{-2}$. During the test, the biodiesel sample was placed inside a viscometer capillary tube submerged in a water bath at a constant temperature of 40 °C. The time required for the fluid to flow from the upper mark to the lower mark was recorded using a digital stopwatch with an accuracy of ±0.01 s. The value of the constant C was obtained through initial calibration using a standard fluid with known viscosity. Physically, an increase in viscosity indicates the presence of strong intermolecular cohesion forces, which can be caused by the length of the carbon chain, the number of double bonds, or the presence of residual glycerides in the biodiesel. Conversely, a viscosity that is too low indicates a high free methanol content. Therefore, this parameter not only functions as an indicator of the flow properties of the fuel, but also as a benchmark for the success of the purification process and the stability of the chemical structure of the resulting biodiesel.

2.6.4. Calorific value

Calorific value is a thermodynamic parameter that indicates the maximum amount of heat energy that can be released from a fuel during complete combustion. In the context of biodiesel, the calorific value plays an important role in determining energy efficiency, engine performance, and specific fuel consumption in the combustion system. The higher the calorific value of a fuel, the greater the energy produced per unit mass when burned. Calorific value measurements were carried out using an adiabatic bomb calorimeter with a test procedure referring to the ASTM D240–19 standard. The principle of this test is based on measuring the change in water temperature in a closed calorimeter system during complete combustion of a fuel sample under adiabatic conditions. The released heat energy is then calculated as the Higher Heating Value (HHV) using Eq. (8) [39].

$$\mathrm{HHV} = \frac{(m_w \times C_w \times \Delta T) - Q_c}{m_f} \tag{8}$$

where, m_w= mass of water in the calorimeter (g); C_w= heat capacity of water (4.186 J·g^{-1}·°C^{-1}); ΔT= temperature rise of water during combustion (°C); Q_c= heat correction for the ignition wire and wick (J); m_f= mass of the fuel sample (g).

A measured mass of biodiesel sample was placed in a crucible and then completely oxidized in a high-pressure oxygen atmosphere inside a sealed bomb chamber. The temperature rise of the water surrounding the bomb chamber was continuously recorded using a high-precision digital thermocouple. A correction value (Q_c) was applied to eliminate the heat contribution from the nichrome wire and wick material so that the calculation results only represent the energy released by the fuel. In general, the calorific value of biodiesel is slightly lower than that of fossil diesel fuel due to the internal oxygen content in the ester molecular structure. However, the more balanced distribution of carbon atoms and cleaner combustion make biodiesel competitive in terms of energy efficiency. The higher heating value (HHV) represents the total energy content of the fuel and was used in this study to compare the intrinsic energy potential of the produced biodiesel. For practical combustion systems such as engines and burners, the lower heating value (LHV) is more relevant because the water formed during combustion remains in the vapor phase and its latent heat is not recovered.

2.7. Data Validation

Each measurement in this study was repeated three times (triplicate) to obtain a representative average value and calculate the standard deviation as a measure of data dispersion. The combined uncertainty (U_c) is determined based on the square root of the sum of the individual uncertainties as stated in Eq. (9).

The magnitude of the experimental uncertainty for each measured parameter is presented in **Table 3**. The relatively low standard deviation and combined uncertainty values indicate that the measurements are highly repeatable and that random experimental errors have a negligible influence on the observed trends. In addition, the accuracy and resolution of the measuring instruments used in this study are summarized in **Table 4**. These specifications confirm that the selected instruments provide sufficient precision to detect changes in biodiesel properties resulting from variations in reaction temperature and hot air flow rate. Since the combined uncertainty values are much smaller than the variations between experimental conditions, the observed differences in biodiesel characteristics can be attributed to the process parameters rather than measurement limitations.

$$U_c = \sqrt{(u_1^2 + u_2^2 + \cdots + u_n^2)} \tag{9}$$

Table 3. Experimental uncertainty of measured parameters

Parameter	Standard deviation	Combined uncertainty (Uc)
Density (g·cm^{-3})	±0.002	±0.003
Kinematic viscosity (cSt)	±0.05	±0.06
Flash point (°C)	±0.8	±1.0
Calorific value (cal·g^{-1})	±35	±42
Yield (%)	±0.6	±0.7

Table 4. Accuracy of measuring instruments

Parameter measured	Instrument	Accuracy	Standard / model
Temperature	Digital thermocouple	±0.5 °C	—
Biodiesel density	Digital density meter	±0.001 g·cm^{-3}	ASTM D4052
Kinematic viscosity	Ostwald viscometer	±0.01 s (flow time)	ASTM D445
Flash point	Pensky–Martens apparatus	±1 °C	ASTM D93
Calorific value	Bomb calorimeter	±0.1%	ASTM D240

3. Results and Discussion

This study aims to examine the effect of reaction temperature and hot air flow rate on the quality of biodiesel synthesized from nyamplung oil using the Thermal Air Sparging (TAS) method. The quality parameters analyzed include density, kinematic viscosity, and flash point. Test data from various treatment combinations were compared to the biodiesel quality standards in SNI 7182:2015 and analyzed to identify optimum operating conditions.

3.1. Free Fatty Acid (FFA) Reduction

The nyamplung crude oil in this study contained 18.2% FFA. This clearly indicates its classification as a highly acidic feedstock, making it unsuitable for direct alkaline transesterification due to dominant saponification and poor phase separation. The first esterification step reduced the FFA to 9.20 ± 0.30%, indicating that the acid-catalyzed reaction proceeded effectively even in an immiscible methanol-oil system. However, this level remained above the critical threshold for base catalysis, reflecting equilibrium limitations caused by water formation during the reaction. The second esterification step further reduced the FFA to 1.89 ± 0.03%, corresponding to an overall reduction of 89.6%. Achieving this value is crucial, as it shifts the system from an acid-dominated regime to kinetically favorable conditions for alkaline transesterification. The low standard deviation obtained at this stage indicates stable hydrodynamic behavior and consistent thermal distribution throughout the process. The rapid free fatty acid conversion can be attributed to the thermofluidic mechanism of the Thermal Air Sparging (TAS) system. The heated microbubble dispersion continuously renews the liquid-liquid interface, intensifying mass transfer, and maintaining a uniform temperature field without mechanical stirring. These combined effects accelerate the diffusion of reactants toward the reactive zone and drive the reaction toward ester formation under relatively mild conditions. These results demonstrate that TAS-assisted esterification provides a controlled and efficient pretreatment pathway for nyamplung oil with high free fatty acid content, forming a stable, low-acidity intermediate that allows subsequent alkaline transesterification to proceed under optimal kinetic conditions. The reduction of FFA during the esterification process can be observed in **Table 5**.

Table 5. Reduction of FFA during the esterification process

No.	Stage	FFA (%) – Run 1	FFA (%) – Run 2	FFA (%) – Run 3	Average ± SD
1	Crude oil	18.2	-	-	18.2
2	After esterification 1	9.2	9.5	8.9	9.20 ± 0.30
3	After esterification 2	1.87	1.93	1.88	1.89 ± 0.03

3.2. Biodiesel Density Analysis

The density value of the biodiesel synthesized in this study was in the range of 0.867–0.888 g·cm^{-3}. The density value of the research results can be observed in **Figure 3** which fully meets the specification limits of SNI 7182:2015 (0.850–0.890 g·cm^{-3}). This range of values indicates that the process of converting triglycerides to methyl esters through the Thermal Air Sparging (TAS) method was stable under all tested operating conditions. Variations between treatments showed a significant effect of reaction temperature and hot air flow rate on the chemical conversion rate, which is directly related to the efficiency of methyl ester formation. The lowest density value, namely 0.867 g·cm^{-3}, was obtained at 30 °C and an air flow rate of 1.0 L·min^{-1}, indicating that the conversion of nyamplung oil to biodiesel took place most efficiently under these conditions. The lower density represents the dominance of the methyl ester fraction in the mixture, as biodiesel (FAME) naturally has a lower density than crude triglycerides.

This phenomenon can be explained by the balance between thermal energy and liquid phase dynamics. At low temperatures (30 °C), the formed hot air bubbles are micro-sized and move stably, increasing the contact time between the methanol and oil phases and allowing for a more complete transesterification reaction. Conversely, at higher temperatures, the increase in kinetic energy accelerates diffusion, but also increases methanol evaporation, reducing the amount of effective reactants in the system. When the temperature reaches 40 °C and the air flow rate increases to 2.0 L·min^{-1}, the density rises to 0.888 g·cm^{-3} due to excessive turbulence, which shortens the reactant residence time and reduces conversion efficiency.

Moderate thermal conditions between 30 °C and 35 °C have been shown to provide an optimal balance between reaction kinetics and mass transfer efficiency, reducing the likelihood of the formation of intermediate compounds such as diglycerides or monoglycerides. This is consistent with the findings of Hindarso *et al*. [40], who reported that the density of nyamplung

biodiesel ranged from 0.880–0.892 g·cm^{-3} when processed using a catalytic method. Therefore, the Thermal Air Sparging method has been shown to produce biodiesel with a narrower and more stable density range, indicating better thermal efficiency. The diffusion of microbubbles generated by the fine-pored sparger increases the contact area between the phases and maintains thermal uniformity throughout the reaction volume, thereby accelerating the conversion rate without causing thermal degradation.

A similar pattern was also observed by Sasmita *et al*. [41], who found that increasing the gas flow rate above the optimum limit can shorten the interphase contact time and reduce reaction efficiency due to the formation of an excessive aeration zone at the bottom of the reactor. Therefore, it can be concluded that the combination of 30 °C and 1.0 L·min^{-1} is the most ideal condition for producing low-density biodiesel, indicating maximum conversion, high thermal stability, and dominant methyl ester formation. Overall, these results confirm the superiority of the TAS method in creating homogeneous heat distribution and mixing without the need for mechanical stirring, while minimizing the risk of soap formation or residual glycerol as is common in conventional processes. The results of the biodiesel density test at various combinations of reaction temperature and hot air rate can be observed in **Figure 3**.

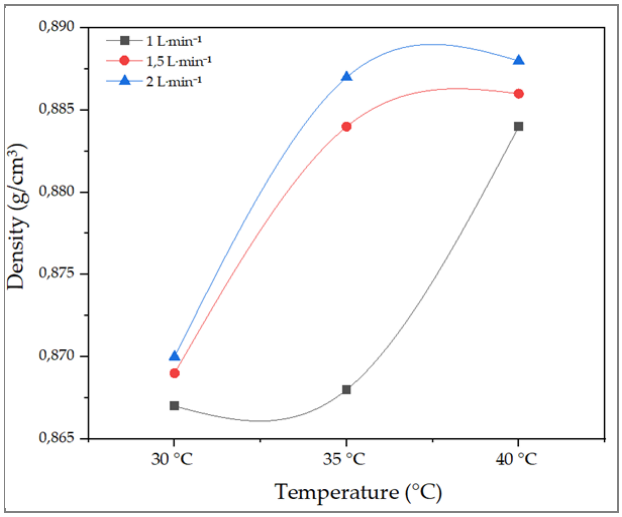

Figure 3. Results of biodiesel density tests at various combinations of reaction temperature and hot air rate

3.3. Biodiesel Viscosity Analysis

Figure 4 shows the results of biodiesel viscosity tests at various combinations of reaction temperatures and hot air rates. The kinematic viscosity values of the synthesized biodiesel ranged from 4.67 cSt to 8.97 cSt, where most samples at temperatures of 30 °C – 35 °C still met the SNI 7182:2015 specification limits (2.3–6.0 cSt). The viscosity increase pattern was clearly visible at 40 °C, especially at an air flow rate of 2.0 L·min^{-1} with a value reaching 8.97 cSt. This condition indicates that at high thermal intensity, the Thermal Air Sparging (TAS) system with a base catalyst began to experience phase imbalance due to excess methanol evaporation and the formation of intermediate compounds such as diglycerides and monoglycerides that were not fully converted. This phenomenon demonstrates the TAS system's high sensitivity to temperature and aeration fluctuations, where small changes in thermal conditions can affect the stability of the reaction kinetics and the fluid properties of the resulting biodiesel.

The lowest viscosity value was recorded at 30 °C and an air flow rate of 1.0 L·min^{-1}, at 4.67 cSt, representing the most thermofluidically stable operating conditions. Under these conditions, the microbubbles formed by the fine-porous sparger have a relatively uniform diameter and a moderate rise rate, creating even heat distribution and homogeneous interphase mixing. This mechanism enhances molecular diffusion between oil and methanol, accelerates the formation of methoxide ions (CH_3O^-) from NaOH, and promotes more efficient conversion of triglycerides to methyl esters. Consequently, the resulting biodiesel has a low viscosity, a homogeneous chemical structure, and good thermal stability. In contrast, the significant increase in viscosity at 40 °C and high air flow rates reflects excess thermal energy and excessive turbulence, which accelerate the evaporation of methanol before the reaction reaches equilibrium. As a

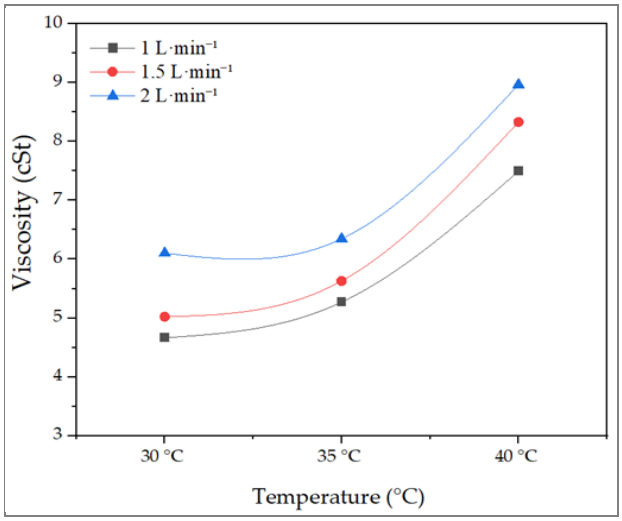

Figure 4. Results of biodiesel viscosity tests at various combinations of reaction temperature and hot air rate

result, some triglycerides do not fully react, resulting in a heavy fraction with long carbon chains and high intermolecular cohesion. This increase in the heavy fraction contributes to the greater flow resistance of biodiesel. A similar mechanism was described by Vallinayagam *et al*. [42], who stated that the efficiency of a base-catalyzed transesterification reaction is highly dependent on the balance between heating and mixing intensity. An imbalance in either factor has been shown to result in residual glyceride molecules, which increase the final viscosity of the product.

The consistency of these results was also confirmed by Permanasari *et al*. [43] and Woo and Kim [44], who confirmed that biodiesel viscosity will remain within the optimum range if thermal and hydrodynamic parameters are carefully controlled. In this study, conditions at 30 °C with an air flow rate of 1.0 L·min^{-1} – 1.5 L·min^{-1} proved most effective, as they provided a balance between activation energy and contact time without triggering thermal degradation or methanol loss. This ratio allowed the reaction to proceed homogeneously, resulting in maximum conversion and stable viscosity.

Overall, the increase in viscosity under extreme conditions indicates an imbalance between reaction kinetics, mass transfer, and methanol volatility in the base-catalyzed TAS system. Therefore, controlling the temperature and hot air flow rate is key to maintaining biodiesel viscosity within the standard specification range. Controlled operating conditions not only ensure complete methyl ester formation but also maintain the flow characteristics and thermal stability of the biodiesel, thus supporting more efficient and environmentally friendly combustion performance.

3.4. Biodiesel Flash Point Analysis

The results of biodiesel flash point tests at various reaction temperature and hot air flow rates can be seen in **Figure 5**. The flash point of the synthesized biodiesel ranged from 175.5 °C to 187.0 °C, with a tendency to increase with increasing reaction temperature and hot air flow rate. The highest value was 187.0 °C at 40 °C and a hot air flow rate of 2.0 L·min^{-1}, while the lowest value was 175.5 °C at 30 °C and 1.0 L·min^{-1}. All test results were well above the minimum limit of SNI 7182:2015 (≥100 °C), indicating that the biodiesel synthesized using the Thermal Air Sparging (TAS) method with a base catalyst has a high level of thermal safety and good storage stability.

Although hot air containing oxygen was introduced into the reactor during the TAS process, oxidative degradation of biodiesel was considered negligible. This is because the reaction was conducted at relatively low temperatures (30–40 °C) and a short processing time (10 minutes), which are far below the temperature range typically associated with significant thermal oxidation of fatty acid methyl esters. The high flash point values and the stable viscosity obtained under optimum operating conditions indicate that no severe oxidation or polymerization occurred. Therefore, the presence of oxygen in the injected air did not adversely affect biodiesel quality. The use of air instead of an inert gas was intentionally selected to maintain a simpler, low-cost, and energy-efficient process configuration.

An increase in flash point generally correlates with a decrease in residual methanol content in the biodiesel. The combination of higher reaction temperatures and intense hot aeration accelerates the evaporation of methanol from the liquid phase, thereby reducing fuel volatility and increasing the initial ignition temperature. This phenomenon is highly advantageous, considering that the presence of free methanol in biodiesel often poses a fire risk and reduces the thermal stability of the final product. Therefore, the increasing flash point trend in the TAS system indicates that the hot air-based heating mechanism can effectively act as an in-situ drying process that occurs simultaneously with the transesterification reaction.

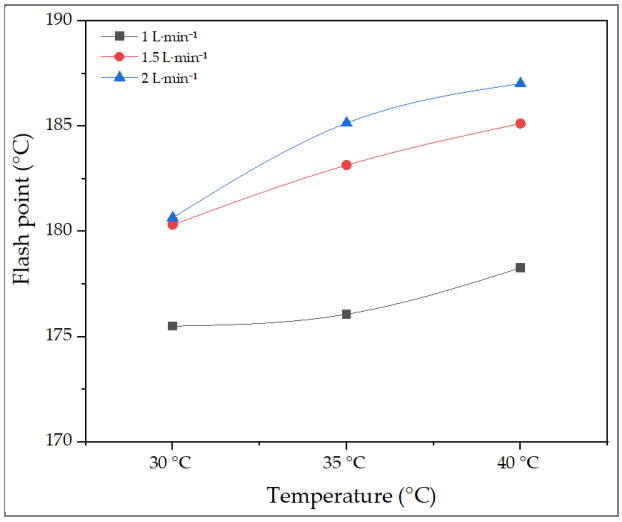

Figure 5. Results of biodiesel flash point tests at various combinations of reaction temperature and hot air rate

However, the results also indicate that a high flash point does not always correlate with chemical conversion efficiency. At 40 °C and 2.0 L·min^{-1}, even though the flash point reached its highest value (187.0 °C), the viscosity increased to 8.97 cSt. This indicates that excessively rapid methanol evaporation can reduce the

effectiveness of methoxide ion (CH_3O^-) formation, resulting in incomplete conversion of some triglycerides and diglycerides to methyl esters. As a result, longer-chain intermediates are formed in greater quantities, increasing the flow resistance of the biodiesel without reducing its ignition temperature. Therefore, a balance between heating and aeration intensity is required to achieve the optimum between solvent evaporation efficiency and stable reaction kinetics.

This finding is consistent with the report by Arumugam and Ponnusami [34], who stated that biodiesel produced from non-catalytic processes tends to have a higher flash point than catalytic methods because it does not contain base residues or heavy solvents that can reduce thermal stability. However, the results of this study indicate that the TAS method with a base NaOH catalyst can produce an even higher flash point than the non-catalytic system under optimum conditions. This is due to the combination of catalytic effects and aerodynamic heating, which increases methanol evaporation efficiency without causing thermal degradation. For comparison, Nolhakim *et al*. [45] reported that catalytically produced waste cooking oil-based biodiesel has a flash point in the range of 150 °C–168 °C, much lower than the results of this study. These differences reinforce the evidence that the TAS method offers advantages in terms of product purity and thermal safety, while reducing the need for further drying steps after the reaction is complete. From a thermofluidic perspective, the flash point increase phenomenon is also influenced by the distribution of microbubbles generated by the fine-pored sparger. Under moderate aeration conditions (1.0 L·min^{-1} – 1.5 L·min^{-1}), the surface area of air in contact with the reaction mixture increases significantly, allowing for more efficient heat transfer and gradual evaporation of methanol. This process promotes natural drying of the biodiesel without disturbing the liquid phase equilibrium [34], [35], [46]. However, at air rates above 1.5 L·min^{-1}, excessive turbulence introduces mechanical disturbances that inhibit chemical conversion, as reflected by an increase in viscosity and a slight decrease in yield.

Therefore, the conditions of 35 °C and 1.5 L·min^{-1}, which produce a flash point of 183.2 °C, can be considered the most balanced operating point. Under these conditions, methanol evaporation efficiency, thermal stability, and chemical conversion reach optimum equilibrium. Overall, the results of this study prove that the Thermal Air Sparging method with a base catalyst is not only effective in increasing the efficiency of the transesterification reaction, but also superior in terms of thermal safety and fuel storage quality. The high flash point value is a strong indicator that the resulting biodiesel has minimal methanol content, is free from catalyst residues, and is stable against thermal degradation, making it a worthy candidate for application as an environmentally friendly alternative fuel.

3.5. Biodiesel Calorific Value Analysis

The calorific value of biodiesel synthesized using the Thermal Air Sparging (TAS) method can be observed in **Figure 6**. In this discussion, the calorific value refers to the higher heating value (HHV), which is used to compare the intrinsic energy content of the produced biodiesel rather than to represent the actual efficiency of practical combustion systems. The results of this test ranged between 9,132.05–10,283.20 cal·g^{-1}, indicating that the energy contained in the fuel from the conversion of nyamplung oil has good thermal characteristics and is competitive compared to conventional biodiesel. The highest value was obtained at a temperature of 35 °C and an air rate of 1.5 L·min^{-1}, which was 10,283.20 cal·g^{-1}, while the lowest value was recorded at 40 °C and 2.0 L·min^{-1}, which was 9,132.05 cal·g^{-1}. The pattern of changes in calorific value showed that increasing the reaction temperature to a moderate limit contributed positively to increasing combustion energy, while excessively high thermal conditions actually reduced efficiency due to phase instability disturbances and excessive methanol evaporation. The increase in heating value up to 35 °C can be attributed to the increased efficiency of the transesterification reaction, where methyl ester formation proceeds more completely and results in a more homogeneous molecular

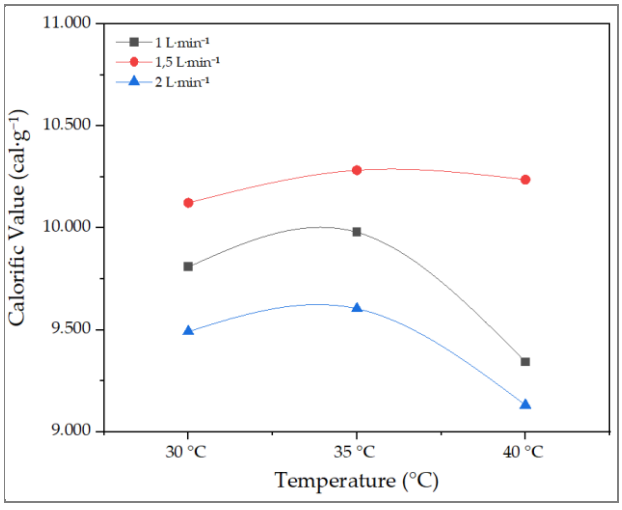

Figure 6. Results of biodiesel calorific value tests at various combinations of reaction temperature and hot air rate

composition. Biodiesel rich in methyl esters has a balanced proportion of carbon and hydrogen atoms and a lower oxygen content than the initial triglyceride, thus producing higher heat energy when burned. However, at 40 °C, the heating value decreases because some of the lighter fractions, particularly residual methanol, volatilize too quickly, while the ester formation reaction has not yet reached full equilibrium. This condition encourages the formation of intermediate compounds such as monoglycerides and diglycerides, which have lower energy content, thus reducing the overall HHV value.

In addition to temperature, the hot air flow rate also plays a significant role in the final result. An air flow rate of 1.5 L·min^{-1} produces the highest heating value at all temperature variations because it provides optimal heat transfer and gas diffusion without causing excessive turbulence. In contrast, an air flow rate of 2.0 L·min^{-1} tended to produce lower heating values, indicating that excessive aeration could destabilize the two-phase system and shorten the effective contact time between methanol and oil. This imbalance resulted in incomplete conversion of some triglyceride molecules to methyl esters, resulting in a decrease in the energy content per unit mass of fuel. These results also demonstrate a strong correlation between heating value and flash point. Conditions with a high flash point, 183.2 °C at 35 °C – 1.5 L·min^{-1} indicate a low residual methanol content, resulting in a significant increase in heating value. This correlation indicates that the TAS system is capable of not only producing an efficient conversion reaction but also an effective solvent evaporation process. This phenomenon is consistent with the results of Hassan and Smith [47], who emphasized that non-catalytic reaction systems based on thermal aeration are highly dependent on the balance between thermal energy and mass transfer, as small disturbances in either aspect can reduce conversion efficiency and combustion energy output. Overall, the combination of 35 °C temperature and 1.5 L·min^{-1} hot air flow rate was identified as the optimum condition because it produced the highest Higher Heating Value and stable thermal consistency. This condition not only showed efficient conversion performance but also produced biodiesel with clean combustion potential and high thermal stability, indicating a higher intrinsic energy content of the fuel. Thus, the TAS method has great potential to be implemented on a small to medium industrial scale, especially for the production of high FFA oil-based biodiesel that requires a simple, efficient, and environmentally friendly process.

3.6. Biodiesel Yield Analysis

The results of the yield test in this study can be observed in **Figure 7**. The results showed that the yield of biodiesel synthesized using the Thermal Air Sparging (TAS) method with a NaOH base catalyst was in the range of 78.20%–85.43%, indicating a high conversion efficiency of triglycerides to methyl esters. The highest value was achieved at a temperature of 40 °C and a hot air rate of 1.0 L·min^{-1}, namely 85.43%, while the lowest value was obtained at 30 °C and 2.0 L·min^{-1}, namely 78.20%. This variation indicates that the yield is significantly influenced by the combination of reaction temperature and hot air rate, where increasing the temperature within the optimum limit accelerates the reaction, while excessive air turbulence actually decreases the conversion efficiency. The increase in yield up to a temperature of 40 °C can be explained by the thermal effect on the kinetics of the base-catalyzed transesterification reaction. Higher temperatures increase the kinetic energy of the molecules and accelerate the formation of methoxide ions (CH_3O^-) from NaOH and methanol. These ions react with triglycerides to form methyl esters and glycerol through a stepwise mechanism (triglyceride → diglyceride → monoglyceride → glycerol). In the context of a Thermal Air Sparging system, the heated air flow acts as both an energy transfer medium and a fluid agitator, thus enhancing the contact between the liquid phases. This combination of thermal and mechanical effects accelerates mass transfer between the methanol and oil phases, increasing the reaction rate and overall conversion.

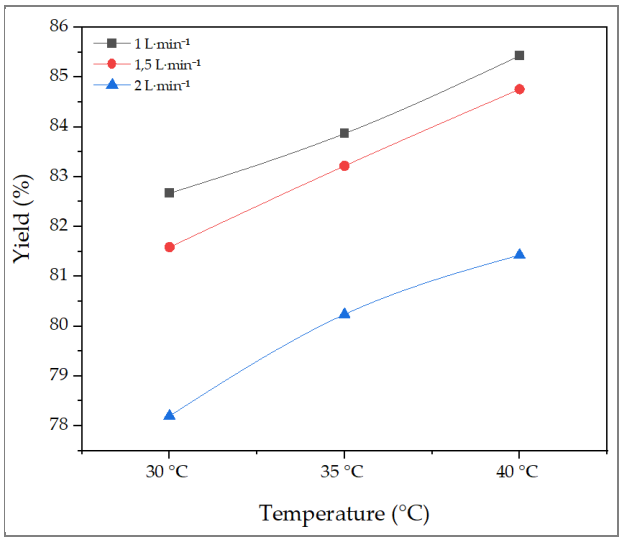

Figure 7. Results of biodiesel yield tests at various combinations of reaction temperatures and hot air rates

However, increasing the air flow rate above the optimum limit (2.0

L·min^{-1}) causes premature evaporation of methanol and the formation of excessive turbulence, which reduces the effective contact time between the liquid phases. As a result, some of the triglycerides do not have time to fully react, and the yield decreases. The optimum conditions of 40 °C and 1.0–1.5 L·min^{-1} demonstrated that the TAS system is capable of achieving high conversions without the need for extreme temperature increases. This confirms that the heat distribution through stable microbubbles allows the reaction to proceed homogeneously and in a controlled manner, while avoiding thermal degradation of the methyl ester [48].

Compared to the conventional method without aeration, the base-catalyzed TAS system showed a yield increase of several percent. This is due to the synergistic effect between the chemical reaction and fluid dynamics, where the microturbulence of the heated air helps expand the contact surface between the phases, reducing diffusion resistance, and accelerating the overall reaction. Furthermore, the reaction under moderate thermal conditions reduces the possibility of soap formation (saponification), thus facilitating the separation of biodiesel and glycerol [12], [49]. Overall, the results of this study indicate that the combination of 40 °C and an air flow of 1.0 L·min^{-1} is the optimum operating condition for achieving the highest conversion efficiency with good product purity. The TAS system with a NaOH base catalyst has been shown to be effective in improving transesterification process performance by increasing thermal homogeneity and mass transfer efficiency [35]. Therefore, this approach has great potential for development in the production of high-FFA biodiesel oils such as nyamplung, especially on a medium scale that requires a fast, efficient, and environmentally friendly process.

A comparison of the primary effects of the two operating variables shows that reaction temperature exerts a greater influence on biodiesel yield than hot-air flow rate. Reaction temperature directly controls the kinetics of methoxide ion formation and accelerates the conversion of triglycerides to methyl esters. In contrast, hot air flow rate mainly affects the system's hydrodynamic properties, particularly the interfacial contact area and the effective residence time of reactants. Excessive aeration reduces contact time and leads to premature methanol evaporation, which decreases overall conversion efficiency. The highest biodiesel yield observed in this study was 85.43%, achieved at a reaction temperature of 40 °C and a hot air flow rate of 1.0 L·min^{-1}. This condition provides the optimal balance between reaction kinetics and mass transfer under mild thermal conditions.

To further assess the effectiveness of the proposed reactor configuration, the biodiesel yields and fuel properties obtained in this study were compared with previously reported results from studies using various feedstocks, catalysts, and reactor systems. The comparison focused on the optimum operating conditions for each process to ensure a fair evaluation of conversion efficiency, thermal requirements, and product quality. The results of this comparative analysis are presented in **Table 6**.

Table 6. Comparison of biodiesel production methods and fuel properties with previous studies

Feedstock	Method / Reactor	Catalyst type	Temp (°C)	Time	Yield (%)	Density (kg·m^{-3})	Viscosity (mm^2·s^{-1})	Flash point (°C)	Study
Nyamplung oil	Thermal Air Sparging (microbubble reactor)	Homogeneous NaOH	30–40	10 min	85.43	867	4.67	183.2	This study
Nyamplung oil	Conventional transesterification	Heterogeneous (modified zeolite)	60	5 h	88.19	887	5.63	121	[40]
Waste cooking oil	Conventional transesterification (RSM optimized)	Homogeneous NaOH	60–65	1–2 h	91.30	870–890*	4–6*	–	[10]
Waste cooking oil	Conventional transesterification	Heterogeneous K_2CO_3/Al_2O_3	65	2 h	87.57	–	–	–	[45]

Table 6 demonstrates that conventional catalytic transesterification of nyamplung oil typically requires elevated reaction temperatures and extended reaction times to achieve biodiesel yields exceeding 85%. For example, the heterogeneous catalytic system described by Hindarso *et al*. [40] produced 88.19% biodiesel at 60 °C over 5 hours, while the current TAS system achieved a comparable conversion within 10 minutes at 30–40 °C. A similar pattern is evident in waste cooking oil–based biodiesel, where yields above 90% are attained only under higher thermal conditions and prolonged processing times. Beyond reduced thermal severity, the biodiesel produced in this study exhibits a higher flash point than several previously reported catalytic systems, suggesting more effective in-situ methanol removal and enhanced fuel purity. These results indicate that the

thermofluidic mechanism, generated by the dispersion of heated microbubbles, facilitates simultaneous heat transfer and interfacial renewal. This intensifies the transesterification process without increasing reactor complexity or energy consumption. Achieving high conversion under atmospheric pressure, at low temperature, and with a short reaction time constitutes a significant advantage for decentralized and medium-scale biodiesel production from a process engineering standpoint.

3.7. Study Limitations and Future Work

Although the Thermal Air Sparging (TAS) system demonstrates good performance in enhancing biodiesel conversion at relatively low temperatures and short reaction times, several limitations inherent in the current experimental framework need to be critically addressed to avoid overgeneralizing the findings. The first limitation relates to the evaluation of oxidative effects during the TAS process. In this study, the presence of oxygen in the injected heated air was indirectly assessed through viscosity stability, flash point, and heating value. While these parameters indicate that severe thermal degradation does not occur within the applied operating range, they do not provide a direct measure of oxidative stability. Parameters such as peroxide value, anisidine value, or induction time were not determined, and therefore, the long-term resistance of the produced biodiesel to thermo-oxidative degradation during storage cannot be quantitatively determined.

The second limitation relates to the reactor scale. All experiments were conducted in a laboratory-scale system with a working volume of 2 L, where heat distribution and microbubble dispersion tend to remain relatively uniform. At larger processing volumes, bubble coalescence, a non-uniform temperature field, and increased mass transfer barriers can alter the hydrodynamic behavior of the system and, consequently, affect conversion efficiency. For this reason, the results of this study should be interpreted as an assessment of process intensification at a laboratory scale rather than a direct representation of industrial performance. Another aspect worth noting is the limited range of process variables. The optimization focused solely on the interaction between reaction temperature and hot air flow rate, while other parameters known to influence transesterification kinetics, such as the methanol-to-oil molar ratio, catalyst loading, sparger configuration, and reaction duration, were kept constant. This approach was deliberately adopted to isolate the thermofluidic contribution from the TAS mechanism; however, it also means that the global optimum operating conditions of the process have not been fully explored.

From a fuel characterization perspective, the analysis was limited to the main physicochemical properties required by SNI 7182:2015. While these parameters are sufficient to verify the suitability of the fuel, they do not provide detailed insight into its molecular composition. The absence of fatty acid methyl ester (FAME) profiling, glyceride content analysis, and impurity quantification makes it difficult to establish a direct correlation between the observed thermophysical behavior and the underlying reaction rates at the molecular level.

Therefore, future investigations should address three key aspects. First, a dedicated oxidative stability assessment is needed to clarify the long-term effects of hot air blowing on biodiesel durability. Second, scale-up studies accompanied by energy efficiency analysis and process intensification are needed to verify whether the thermal and hydrodynamic advantages observed at the laboratory scale can be maintained at higher production capacities. Third, multivariable optimization combined with detailed compositional analysis and engine performance testing will enable a more comprehensive evaluation of the TAS method, not only as a synthesis technique but also as an integrated approach linking reaction engineering, fuel quality, and practical combustion performance.

4. Conclusion

The results indicate that the Thermal Air Sparging (TAS) reactor achieved a biodiesel conversion yield of 85.43% within 10 minutes under atmospheric pressure and relatively low temperatures (30–40 °C). This outcome represents a significant reduction in processing time compared to mechanically stirred transesterification systems, which typically require longer reaction durations to achieve similar conversion levels. Although supercritical and other high-energy intensification techniques reported in the literature can achieve yields exceeding 90%, these methods require substantially higher temperatures and pressures, greater energy input, and more complex reactor designs. In contrast, the TAS system in this work produced biodiesel that fully satisfied the SNI 7182:2015 specification, with a density of 0.867 g·cm^{-3}, a kinematic viscosity

of 4.67 cSt, a flash point of 183.2 °C, and a heating value of 10,283.20 cal·g^{-1} under the optimum condition of 35 °C and 1.5 L·min^{-1}. The ability to obtain high conversion and stable fuel properties simultaneously under mild thermal conditions confirms that the thermofluidic mechanism generated by heated microbubble dispersion effectively intensifies heat and mass transfer without mechanical agitation. Compared with mechanically stirred reactors and energy-intensive intensification methods such as ultrasonic, microwave-assisted, and supercritical processes, the TAS configuration offers a more practical, energy-efficient approach for converting high-FFA non-edible oils into biodiesel. This advantage is attributed to its atmospheric operation, reduced reaction time, simplified system design, and lower thermal requirements.

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Authors' Declaration

Authors' contributions and responsibilities - The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation and discussion of results. The authors read and approved the final manuscript.

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Availability of data and materials - All data is available from the authors.

Competing interests - The authors declare no competing interests.

Additional information – No additional information from the authors.

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