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Monowave-Assisted Optimization of Transesterification for Sustainable Biodiesel Production from *Sterculia foetida* Oil

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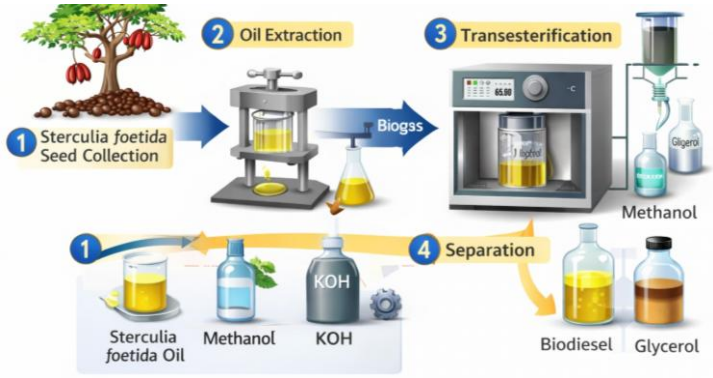
Graphical Abstract



Highlights

- Monowave irradiation enabled rapid biodiesel production from Sterculia foetida oil.
- Biodiesel yield ranged from 83.55 % to 97.22 % under optimized conditions.
- Optimal parameters were 55.45 % methanol, 0.884 wt% catalyst, 650 rpm, and 7.55 min.
- Maximum biodiesel yield of 97.4 % was achieved with high model accuracy (R^2 = 0.9857).
- The process significantly reduced reaction time while meeting ASTM D6751 and EN 14214 standards.

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ABSTRACT

This study presents a comprehensive optimization of biodiesel production from *Sterculia foetida* oil using monowave irradiation. Response Surface Methodology (RSM) based on a Box–Behnken design was employed to evaluate the effects of key process variables, including methanol-to-oil ratio, catalyst loading, reaction time, and irradiation conditions, on biodiesel yield. The results demonstrated that monowave irradiation significantly enhances heat transfer, accelerates reaction kinetics, and reduces processing time compared to conventional methods. Biodiesel conversion yields ranged from 83.55 % to 97.22 %, meeting the requirements of ASTM D6751 and EN 14214 standards. The developed quadratic model showed a high coefficient of determination (R^2 = 98.57 %), indicating strong model reliability. Analysis of variance (ANOVA) revealed that methanol-to-oil

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ratio, catalyst concentration, and irradiation time were the most significant factors affecting methyl ester yield. The optimal conditions were identified at a methanol-to-oil ratio of 55.45 %, catalyst concentration of 0.884 wt%, agitation speed of 650 rpm, irradiation time of 7.55 min, and reaction temperature of 100 °C, resulting in a maximum biodiesel yield of 97.4 %. These findings confirm that monowave-assisted transesterification is an effective, energy-efficient, and cost-effective approach for sustainable biodiesel production from non-edible feedstocks.

1. Introduction

The global energy demand continues to increase due to rapid industrialization, population growth, and economic development. Traditionally, this demand has been predominantly met by fossil fuels; however, environmental concerns such as greenhouse gas emissions, climate change, and resource depletion have accelerated the transition toward renewable energy systems. This transformation, often referred to as the "Grand Energy Transition," emphasizes the urgent need for sustainable, low-carbon, and environmentally friendly fuel alternatives [1–3]. Among various renewable energy options, biodiesel has gained considerable attention as a viable substitute for conventional diesel fuel due to its biodegradability, non-toxicity, and compatibility with existing diesel engines. Biodiesel is typically produced through the transesterification of triglycerides present in vegetable oils or animal fats with short-chain alcohols, such as methanol, in the presence of a catalyst [4–6]. However, the use of edible oils as feedstock has raised concerns regarding food security and cost, prompting increased research into non-edible and waste-derived feedstocks [7–9].

In this context, *Sterculia foetida* oil has emerged as a promising non-edible feedstock for biodiesel production due to its high oil content and favorable fatty acid composition. Unlike edible oils, *Sterculia foetida* does not compete with food resources, making it more suitable for sustainable biofuel production [10–12]. Previous studies have reported that oils derived from non-edible sources exhibit comparable physicochemical properties to conventional biodiesel feedstocks, while also contributing to waste valorization and environmental protection [13,14]. Despite these advantages, conventional transesterification processes are often limited by long reaction times, high energy consumption, and inefficient heat transfer. These limitations reduce overall process efficiency and increase production costs [15,16]. Therefore, alternative heating technologies have been explored to overcome these challenges. Among them, microwave and monowave irradiation have shown significant potential due to their ability to provide rapid, uniform, and volumetric heating [17–19].

Monowave irradiation, in particular, offers enhanced reaction kinetics by directly coupling electromagnetic energy with polar molecules in the reaction mixture, resulting in faster heat generation and improved mass transfer. This leads to shorter reaction times, higher conversion efficiency, and reduced energy consumption compared to

conventional heating methods [20,21]. Moreover, monowave-assisted processes have been widely applied in organic synthesis due to their reproducibility and ability to operate under controlled temperature and pressure conditions [22]. To further enhance process efficiency, statistical optimization techniques such as Response Surface Methodology (RSM) have been widely adopted. RSM enables the evaluation of multiple variables and their interactions simultaneously while minimizing the number of experimental runs required. This approach significantly reduces time, cost, and material usage while providing reliable predictive models for process optimization [23–25].

Therefore, this study aims to optimize the transesterification process of *Sterculia foetida* oil using monowave irradiation assisted by RSM. The effects of key parameters, including methanol-to-oil ratio, catalyst concentration, reaction time, and irradiation conditions, were systematically investigated to maximize biodiesel yield. The findings of this study are expected to contribute to the development of efficient, cost-effective, and sustainable biodiesel production technologies.

2. Materials and Methods

2.1 Materials

Sterculia foetida crude oil was procured from Cilacap, Central Java, Indonesia. All chemicals used in this study were of analytical grade. Methanol (ACS reagent, 99.9 %), sulfuric acid (>98.9 %), ortho-phosphoric acid (85 %), anhydrous sodium sulfate (99%), sodium hydrogen carbonate, and potassium hydroxide pellets (99 % purity) were obtained from standard commercial suppliers. The FAME standard mixture (C8–C24, 100 mg), methyl nonadecanoate (C19, ≥99.5 % purity), 1,2,4-butanetriol, glyceryl monononadecanoate (Mono C19), glyceryl dinonadecanoate (Di C38), and glyceryl trinonadecanoate (Tri C57) were supplied by Sigma-Aldrich through It Tech Research (M) Sdn Bhd. Phenolphthalein solution (1 % in ethanol) was purchased from Fluka Analytical.

2.2 Experimental Set-Up

The Sterculia feotida was first filtered to remove solid impurities and subsequently heated to eliminate residual moisture. The purified Sterculia foetida oil at a ratio of 7:3. The initial free fatty acid (FFA) content of the mixture was 8 %, which exceeds the acceptable limit for direct transesterification. Therefore, a pretreatment process consisting of degumming and esterification was carried out. Degumming was performed in a double-jacketed reactor (2 L) at 60 °C with an agitation speed of 1000 rpm for 30 min using 5 % (v/v) diluted ortho-phosphoric acid (20 %). Subsequently, esterification was conducted under similar reactor conditions at 60 °C and 1000 rpm for 2 h using 1 % (v/v) sulfuric acid (H_2SO_4) and 50 % (v/v) methanol-to-oil ratio. This process successfully reduced the FFA content to below 2 %, making the oil suitable for subsequent transesterification optimization. The optimization study was carried out using the Anton Paar Monowave 400 equipped with an Autosampler MAS 24. This high-performance monowave reactor is specifically designed for small-scale synthesis,

offering rapid and uniform heating, which makes it highly suitable for process optimization in biodiesel production.

2.3 Monowave Assisted Biodiesel Synthesis

The experimental design was used to investigate the optimization of converting the Sterculia feotida were shown as in Figure. 1. Monowave irradiation were used to convert the esterified Sterculia feotida to fatty acid methyl ester. The technical specification of the Anton Paar Monowave 400 with Autosampler MAS 24 was shown in Table 1.

Table 1. Technical specification of Anton Paar Monowave 400

Parameters	Monowave 400
Max. filling volume	20 ml for 30 ml Vial
Max. operation pressure	30 bar (435psi)
Max. IR temperature	300 °C
Max. fiber-optic temperature	300 °C
Max. power	850 W
Vial material	Borosilicate glass
Cap material	PEEK
Camera	Integrated
Autosampler MAS24	Yes
Seal material	Teflon-coated silicone

Figure 1. Anton Paar Monowave 400

The Anton Paar Monowave 400 with Autosampler MAS 24 are equipped with IR sensors for temperature controlling, pressure sensors to monitor the reaction in the closed vessels, and built-in magnetic stirrer to enable proper agitation. The monowave will allow these parameters were accurately measured and recorded throughout the experiment process. The heating profile in the monowave were divided into 3 steps: (1) heating the closed vessels to a required temperature (2) holding the temperature with required time and (3) cooling the closed vessels to cool temperature in order to stop chemical reaction. During the reaction, the power output was controlled based on the temperature set for the experiment. Power output will be automatically maintained and thoroughly check to avoid exotherms of the mixture during monowave irradiation.

2.4 Optimization for Production of Biodiesel

2.4.1 Temperature Parameter Determinant

The combination of closed vessel and monowave irradiance heating system will require a very short reaction protocols. Therefore, Arrhenius Law were employed in this studies in order to learn the conditions required to achieved some identical results with the model reaction that were used on a conventional heating systems. The Arrhenius Law declaimed that every 10 °C increases of reaction temperature will double the rate of reaction which means temperature of reaction is inversely proportional to the time of reaction. Hence, by increasing the temperature to 100 °C will reduce the reaction of the synthesis to 8 minutes where the reaction that conducted under a conventional heating required 60 °C with reaction time of 2 hr as shown in Table 2. The internal temperature and IR temperature are expected to be similar, as the methanol is a very high heating efficiency (tan δ) organic solvents that required to allowed a sufficient heating received during monowave irradiation.

$$k = \mathrm{A} \cdot e^{-E_a/RT} \quad (1)$$

In the Equation (1), k is the rate constant, A is the pre-exponential factor, E_a is the activation energy, R is the universal gas constant, and T is the reaction temperature.

Table 2. The temperature of reaction and time reaction calculated based on Arrhenius Law

Reaction Temperature	60 °C	70 °C	80 °C	90 °C	100 °C
Reaction Time	2 hr	1hr	30 min	15 min	8 min

2.4.2 Optimization Parameter Modelling Based on Statistical Design

The studied parameters were shown in Table 3 were methanol/oil ratio, concentration of the catalyst, speed of rotation of magnetic stirrer and lastly time of reaction as reaction temperature is inversely proportional to the time of reaction therefore, it has reduced the method optimization parameter and drastically reduce the total experimental run. The experiments were designed with 3 parts on the heating method to ensure the reaction is properly heated which is (1) heat to 100 °C in 2 minutes to conditioning the heating temperature of all run, (2) hold time was the studied time of reaction (D) and (3) cool down to temperature of 55 °C in order to stop the transesterification process. Consequently, Box-Behnken experimental design coupled with response surface methodology (RSM) resulted 24 experimental runs shown in Table

4 and these experimental are design to study the effect of each individual independent input variable. The response chosen was the experimental methyl ester yield conversion. Response surface regression was used to analysed the data collected by the following polynomial:

$$Q = c_o + \sum_{i=1}^{k} c_i Y_i + \sum_{i=1}^{k} c_{ii} Y_i^2 + \sum_{i,i>j}^{k} \cdot \sum_{i=1}^{k} c_{ij} Y_i Y_j + e \quad (2)$$

In the Equation (2), Q is the response variables (dependent variable), Y_i is the predictor variable, c_o is the constant, c_i is the coefficient first order model, c_ii is the quadratic coefficient of the ith factor; c_ij is interactions coefficients from classical linear models the for the interaction between the ith and jth factor, k is the number of factors studied and optimized in the experiment, and e is experimental values is attributed to the uncertainty of Q. Analysis of Variance (ANOVA) was carried out to evaluate the influence of all terms in the polynomial model with 95 % confidence interval. For the statistical optimization, the corresponding three-dimensional surface response plot was employed, that allowed the determination of the optimum operating parameters to maximise the conversion yield.

Table 3. Variables for optimization of the transesterification process parameters for the *Sterculia foetida* oil

Factor	Unit	Level		
		-1	0	1
(A) Methanol/oil ratio	v/v%	40	55	70
(B) Catalyst	w.t %	0.4	0.7	1
(C) Speed	rpm	600	800	1000
(D) Time of reaction	min	2	6	10

Table 4. Responses for optimization of transesterification process of 4 parameters and individual parameters consists of 3 levels for *the Sterculia Feotida*

Run	A Methanol/oil ratio (v/v%)	B Catalyst (wt.%)	C Speed (rpm)	D Time (min)	Experimental methyl ester yield (wt.%)	Predicted methyl ester yield (wt.%)
1	55	0.4	1000	6	92.22	92.05
2	55	1.0	1000	6	93.59	94.40
3	55	0.7	1000	10	93.62	93.67
4	55	1.0	800	10	92.35	92.26
5	55	0.4	800	2	89.13	89.01
6	70	0.7	800	10	91.23	91.27
7	70	1.0	800	6	92.04	91.45
8	55	0.7	800	6	94.53	94.99
9	55	0.7	800	6	95.22	94.99
10	55	0.7	800	6	95.32	94.99
11	70	0.4	800	6	88.80	89.04
12	70	0.7	1000	6	92.55	92.33
13	70	0.7	800	2	90.51	90.65
14	55	0.7	600	10	94.53	94.64
15	70	0.7	600	6	93.00	93.38
16	55	1.0	600	6	94.00	94.16
17	55	0.7	600	2	92.35	92.53
18	55	1.0	800	2	92.77	92.50
19	40	0.7	1000	6	90.00	89.41
20	55	0.7	800	6	94.75	94.99
21	55	0.7	1000	2	92.22	92.05
22	55	0.4	800	10	93.59	94.40
23	55	0.7	800	6	93.62	93.67
24	55	0.4	600	6	92.35	92.26

2.5 Biodiesel Analysis

2.5.1 FAME Identification

Gas chromatography/ flame ionization detector (GC–FID) is an equipment used for determination of the ester content in fatty acid methyl ester (FAME) in the biodiesel for EN14103: 2011 method which also allows the determination of linolenic acid methyl ester content (C18:3). The EN14103: 2011 is the procedure that allows verifying the ester content of FAME is greater than 90 % (m/m) and the linolenic acid methyl acid content should be in between 1 to 15 % (m/m). This method is suitable for FAME, which contains methyl esters between C6 to C24. The GC was equipped with an Agilent HP Innowax column (polyethylene glycol (PEG) stationary phase, high polarity, 30 m x 0.25 mm x 0.25 um) and carrier gas used for this operating condition was helium gas. The analysis conditions were listed in the Table 5 and the analysed data were calculated by the following equation:

$$\text{FAME}=\frac{\sum \text{A} - \text{A}_{\text{EI}}}{\text{A}_{\text{EI}}} \times \frac{\text{W}_{\text{EI}}}{\text{W}} \times 100 \quad (3)$$

$$\text{L}=\frac{\text{A}_{\text{L}}}{\text{A}_{\text{EI}}} \times \frac{\text{W}_{\text{EI}}}{\text{W}} \times 100 \quad (4)$$

In the Equation (3) and Equation (4), FAME represent the fatty acid methyl ester content (% m/m), $\sum A$ is the total peak area from the methyl ester in C6:0 to C24:1, AEI is the peak area corresponding to methyl nanodecanoate (C19), WEI is the weight, in milligrams, of the methyl nanodecanoate (C19) being used as internal standard, W is the weight, in milligrams, of the sample, L represent linolenic acid methyl ester (% m/m), and AL is the peak area corresponding to the linolenic acid methyl ester.

Table 5. Operating conditions and standard needed for chromatographic–FID based on EN 14103:2011

Parameters	Specification
Capillary column	HP Innowax Column, 30 m x 0.25 mm x 0.25 um
Oven	60 °C, hold 2 min
	10 °C/min up to 200 °C, 0 min
	5 °C/min up to 240 °C, 0 min
	240 °C hold for 7 min
	Post run, 255 °C,0.5 min
Carrier gas	Helium
Helium pressure	70 kPa
Flow rate	1.5 ml/min
Split flow	100 ml/min
Split ratio	100:1.5
Injector temperature	250 °C
Detector temperature	250 °C
Type of injector	Split/splitless
Type of detector	Flame ionization detector (FID)

Parameters	Specification
Injection volume	1 µl
FID make up gas	Nitrogen
FAME standard	FAME mix C_8-C_{24},100 mg, SIGMA-ALDRICH
Internal standard	Methyl Nanodecanoate, C_{19}, min 99.5 % purity SIGMA-ALDRICH

2.5.2 Glycerol and Glycerides Identification

Agilent GC-Cool on column inlet was used to determine the glycerol, monoglyceride, diglyceride and triglyceride content in the methyl ester produced from W70CI30 biodiesel. To prepare the sample for the analysis, measure 50 mg of sample into a 12 ml amber vial, then add 40 ul of IS1, 100ul of standard glycerides stock solution (G3440-85018), 100ul of pyridine and lastly 100ul of MSTFA. Then closed the amber vial and shake vigorously to ensure the liquid is in homogeneous state. Store the amber vial in dark area at room temperature for 15 minutes. Then add 4 ml of the n-heptane into the solution by using a graduated cylinder and transfer 1.5ml of the solution into a GC amber vial. 1ul of the solution will be injected into the GC-FID cool on column for analysis and the analysed data were calculated by the following equation:

$$\text{Free Glycerol } \left(\frac{m}{m}\%\right), G=\left[a_G\left(\frac{A_G}{A_{EI1}}\right)+b_G\right]\times\left(\frac{M_{EI}}{m}\right)\times 100 \quad (5)$$

$$\text{Monoglycerides } \left(\frac{m}{m}\%\right), M=\left(\frac{A_{Mono}}{A_{MonoC19}}\right)\times\left(\frac{M_{MonoC19}}{m}\right)\times 100 \quad (6)$$

$$\text{Diglycerides } \left(\frac{m}{m}\%\right), D=\left(\frac{A_{Di}}{A_{DiC38}}\right)\times\left(\frac{M_{DiC38}}{m}\right)\times 100 \quad (7)$$

$$\text{Triglycerides } \left(\frac{m}{m}\%\right), T=\left(\frac{A_{Tri}}{A_{TriC57}}\right)\times\left(\frac{M_{TriC57}}{m}\right)\times 100 \quad (8)$$

$$\text{Total Glycerol } \left(\frac{m}{m}\%\right)=G+0.255M+0.146D+0.103T \quad (9)$$

Table 6. Operating conditions and standard needed for chromatographic FID for glycerol and glycerides identification based on EN 14105:2011

Parameters	Specification
Column	VF-5ht Ultimetal, 15 m x 0.32 mm id x 0.1-µm film
Oven	50 ^{0}C, hold 1 min
	15 ^{0}C/ min, 180 ^{0}C, 0 min
	7 ^{0}C/ min, 230 ^{0}C, 0 min
	10 ^{0}C/ min, 370 ^{0}C, hold 15 min
Carrier gas	Helium Gas
Helium pressure	80kPa
Flow rate	2.5 ml/ min
Injector temperature	track oven
Detector temperature	380 ^{0}C

Parameters	Specification
Type of injector	Cool on column
Type of detector	Flame ionization detector (FID)
Injection volume	1 uL
FID make up gas	Nitrogen
Internal Standard for Glycerol	1,2,4 - butanetriol
Internal Standard for Glycerides	Glyceryl monononadecanoate (Mono C_{19})
	Glyceryl dinonadecanoate (Di C_{38})
	Glyceryl trinonadecanoate (Tri C_{57})

In the Equation (5), (6), (7) and (8) a_G and b_G are the regression coefficients of the calibration function of glycerol, A_G is the peak area of glycerol, A_{EI1} is the peak of internal standard 1,2,4-butanetriol, M_{EI} is the weight, in milligrams, of internal standard 1,2,4-butanetriol, A_{Mono}, A_{Di}, and A_{Di} are the sum of the peak area of the monoglyceride, diglyceride and triglycerides respectively, $A_{MonoC19}$ is the area of the internal standard monoglycerides C_{19}, $M_{MonoC19}$ is the weight, in milligrams, of internal standard monoglycerides C_{19}, A_{DiC38} is the peak area of internal standard C_{38}, M_{DiC38} is the weight, in milligrams, of internal standard diglycerides C_{38}, A_{TriC57} is the area of the internal standard triglycerides C_{57}, M_{TriC57} is the weight, in milligrams, of internal standard triglycerides C_{57}. m is the weight, in milligrams, of the biodiesel sample.

2.5.3 Physiochemical Properties

The physiochemical properties of optimize *Sterculia Feotida* methyl ester by monowave produced from conventional equipment were also obtained and compared. The physiochemical properties such as kinematic viscosity, density, acid value, calorific value, oxidation stability, flash point, pour point, cloud point, cold filter plugging point, copper strip corrosion and Conradson carbon residue were determined according to the ASTM D6751 and EN 14214 standard then assessed with the physiochemical properties of diesel fuel.

3. Results and Discussion

3. 1 Parameter Control by Monowave Irradiation

The monowave reactor provides direct in-core heating to the reaction medium, enabling efficient and rapid energy transfer during the transesterification process. In this study, the esterified Sterculia foetida oil was introduced into a sealed glass vial, followed by the addition of pre-dissolved potassium hydroxide (KOH) in methanol and a magnetic stirring bar to ensure homogeneous mixing. The pre-dissolution of the catalyst in methanol plays a critical role in accelerating the reaction kinetics and ensuring uniform catalytic activity, thereby minimizing experimental variability given the short reaction duration. Upon completion of the reaction, a clear phase separation was

observed, where the upper layer consisted of fatty acid methyl esters (FAME), while the lower layer contained glycerol, excess methanol, catalyst residues, and other impurities. This indicates that the transesterification process was successfully completed within a significantly shorter time compared to conventional heating methods, which typically require several hours. The heating behavior of the reaction system followed a controlled three-stage profile were rapid heating to the desired reaction temperature, isothermal holding for a specified duration, and controlled cooling to a safe handling temperature. The variation in holding time was systematically investigated to evaluate its effect on biodiesel conversion yield. The monowave reactor is equipped with an integrated magnetic stirring system, which ensures uniform temperature distribution and prevents mass transfer limitations during the reaction. Additionally, the reactor automatically adjusts power input to maintain the set temperature, ensuring precise thermal control throughout the process. This dynamic power regulation minimizes the formation of localized hot spots and reduces the risk of uncontrolled exothermic reactions, thereby enhancing process safety and reproducibility. The monowave system demonstrates superior parameter control and efficiency, making it highly suitable for rapid optimization of biodiesel production.

3. 2 Optimisation of Methyl Ester Yield by Using Respone Surface Methodology

The experimental results obtained from 24 runs, along with the predicted values generated using Response Surface Methodology (RSM), are presented in Table 4. The methyl ester yield ranged from 83.55 % to 97.22 %, indicating a strong dependence of biodiesel conversion on the selected process variables. The experimental data were subjected to multiple regression analysis, and a quadratic model was identified as the most suitable to describe the relationship between the independent variables and the response. The quadratic regression equation for the methyl ester yield of *Sterculia foetida* oil in terms of coded variables is expressed as equation 10:

$$Y = 96.49 + 2.16A + 1.23B + 0.18C + 0.39D - 0.003AB - 0.70AC - 0.085AD - 0.057BC - 0.51BD - 0.66CD - 4.63A^2 - 2.28B^2 + 0.33C^2 - 1.56D^2 \quad (10)$$

where A, B, C, and D represent the methanol-to-oil ratio, catalyst concentration, agitation speed, and reaction holding time, respectively.

The Analysis of Variance (ANOVA) results confirm that the developed quadratic model is highly significant, with an F-value of 68.69 and a p-value < 0.0001. The lack-of-fit value (F = 2.88, p = 0.1595) is not significant relative to the pure error, indicating that the model adequately fits the experimental data. The coefficient of determination (R^2 = 0.9857) shows that 98.57 % of the variability in methyl ester yield is explained by the model, demonstrating excellent agreement between predicted and experimental values. Furthermore, the predicted R^2 (0.9247) and adjusted R^2 (0.9713) are in close agreement, confirming the reliability of the model. The adequate precision value of 28.128, which is

significantly greater than 4, indicates a strong signal-to-noise ratio and validates the model for navigation within the design space. Among the investigated parameters, the methanol-to-oil ratio (A), catalyst concentration (B), and reaction time (D) were identified as significant factors ($p < 0.05$) affecting biodiesel yield. Interaction effects between methanol-to-oil ratio and agitation speed (AC), as well as agitation speed and reaction time (CD), were also found to be significant. The quadratic terms A^2, B^2, and D^2 further indicate the presence of curvature effects in the response surface, confirming the suitability of the quadratic model. In contrast, agitation speed (C) exhibited a comparatively minor and statistically insignificant effect on biodiesel yield ($p = 0.2509$). The perturbation analysis revealed that methanol-to-oil ratio, catalyst concentration, and reaction time have the most pronounced influence on methyl ester yield, as indicated by their steep response curves. In comparison, agitation speed showed a relatively gradual effect, confirming its lower significance. These observations highlight that precise control of reactant ratio, catalyst loading, and reaction duration is critical for achieving high conversion efficiency. The optimal conditions predicted by the model were a methanol-to-oil ratio of 55.45 % (v/v), catalyst concentration of 0.884 wt%, agitation speed of 650 rpm, and reaction time of 7.55 min, resulting in a maximum biodiesel yield of 97.4 %. These optimized parameters demonstrate the effectiveness of monowave-assisted transesterification in achieving high conversion efficiency within a significantly reduced reaction time. The statistical analysis confirms that the developed RSM model is robust, reliable, and suitable for predicting and optimizing biodiesel production from *Sterculia foetida* oil under monowave irradiation conditions.

The comparison between predicted and experimental biodiesel yields shows a close agreement within the range of 83.55 % to 97.22 %, confirming the reliability and accuracy of the developed quadratic model. The high correlation coefficient ($R^2 = 0.9857$) indicates that the model effectively represents the experimental data. The perturbation plot illustrates the sensitivity of each process parameter on the methyl ester yield. At the optimized conditions of 55.45 % methanol-to-oil ratio, 0.884 wt% catalyst concentration, 650 rpm agitation speed, and 7.55 min reaction time, the system achieved a maximum biodiesel yield of 97.4 %. Among the variables, methanol-to-oil ratio, catalyst concentration, and reaction time exhibit steeper slopes, indicating their stronger influence on biodiesel conversion, while agitation speed shows a comparatively minor effect.

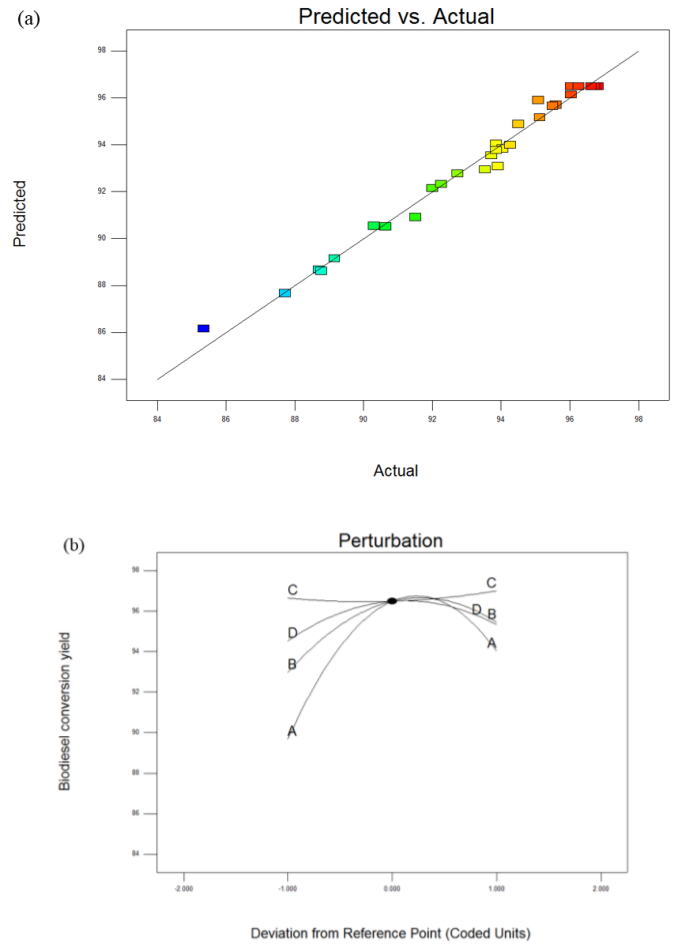

Figure 4. (a) Comparison between predicted and experimental biodiesel conversion yield (83.55 %–97.22 %), demonstrating strong agreement of the quadratic model (R^2 = 0.9857); (b) perturbation plot showing the influence of methanol-to-oil ratio (A), catalyst concentration (B), agitation speed (C), and reaction time (D) on methyl ester yield at optimized conditions of 55.45 % (v/v), 0.884 wt%, 650 rpm, and 7.55 min, resulting in a maximum yield of 97.4 %

3. 3 Effect on Methyl Ester Conversion Yield

3.3.1 Effect on Methanol-to-Oil Ratio

The methanol-to-oil ratio is a critical parameter influencing biodiesel conversion efficiency in the transesterification process. In this study, the methanol-to-oil ratio was varied from 40 % to 70 % (v/v), while other variables were maintained near their central levels. The results demonstrate that the methyl ester yield increased with increasing methanol content, reaching an optimum at approximately 55.45 % (v/v), where the maximum biodiesel yield of 97.4 % was achieved. Beyond this optimum, a slight decline in yield was observed, although the values remained within the experimental range of 83.55 % to 97.22 %. This trend is attributed to the role of methanol in shifting the reversible transesterification reaction toward ester formation, consistent with Le

Chatelier's principle. Insufficient methanol limits triglyceride conversion, whereas excessive methanol increases glycerol solubility in the biodiesel phase, thereby hindering phase separation and reducing apparent yield [27,33]. Moreover, in a closed monowave system, methanol evaporation is minimized, which further emphasizes the importance of precise methanol dosage for maximizing conversion efficiency. Similar observations have been reported in previous studies on biodiesel optimization using response surface methodology [34,35].

3.3.2 Effect of Catalyst Concentration

The effect of catalyst concentration was investigated within the range of 0.4–1.0 wt% using potassium hydroxide (KOH) as a homogeneous catalyst. The results indicate that biodiesel yield increased with increasing catalyst concentration, reaching an optimum at 0.884 wt%, corresponding to the maximum yield of 97.4 %. Further increases in catalyst loading resulted in a slight decrease in yield. This behavior is associated with the catalytic role of KOH in accelerating the transesterification reaction by enhancing the formation of methoxide ions, which promote triglyceride conversion [15,34]. However, excessive catalyst concentration leads to saponification reactions, resulting in soap formation that complicates product separation and reduces effective biodiesel yield [30]. Therefore, an optimal catalyst concentration is essential to balance reaction kinetics and product purity. These findings are in agreement with previous studies reporting similar trends in alkali-catalyzed biodiesel production [31,32].

3.3.3 Effect of Agitation Speed

Agitation speed was varied to evaluate its influence on methyl ester yield. The results show that agitation has a relatively minor effect compared to other process parameters. The optimal condition was observed at 650 rpm, where the biodiesel yield reached its maximum value of 97.4 %. Increasing agitation speed beyond this point did not significantly improve the yield and, in some cases, caused slight fluctuations within the range of 83.55 % to 97.22 %. Although agitation speed was statistically insignificant ($p > 0.05$), it plays an important role in enhancing mass transfer between immiscible phases (oil and methanol), ensuring uniform mixing, and maintaining temperature homogeneity within the reaction system [27]. Adequate agitation facilitates interfacial contact between reactants, thereby supporting efficient transesterification. However, excessive agitation may lead to vortex formation or emulsification, which can negatively affect phase separation and yield. Similar observations have been reported in previous biodiesel studies [33].

3.3.4 Effect of Reaction Time

Reaction time is a crucial parameter affecting biodiesel conversion. The results indicate that methyl ester yield increased with increasing reaction time, reaching an optimum at 7.55 min, where the maximum yield of 97.4 % was achieved. Extending the reaction time beyond this optimum resulted in a slight decline in yield. This phenomenon can be explained by the rapid attainment of reaction equilibrium under

monowave irradiation. The enhanced heating efficiency and uniform energy distribution accelerate reaction kinetics, allowing high conversion to be achieved within a short time frame [17,20]. Prolonged reaction time may promote reverse reactions, such as hydrolysis of esters, or lead to thermal degradation, thereby reducing overall yield [29]. The ability to achieve high conversion within minutes clearly demonstrates the superiority of monowave-assisted transesterification compared to conventional heating methods, which typically require significantly longer reaction times [18,28].

3.4 Model Validation

The Design expert software predicted an optimal condition for all factors that are meant to influence the methyl ester yield. The predicted optimal condition for methanol to oil ratio was 59.60 % (v/v %), catalyst concentration of 0.774 % (v/v %), agitation speed of 600 rpm and lastly the holding time of reaction at 7.15 min that will compromise for a maximum methyl ester yield of 97.40 %. In order to validate the regression model, experimental work based on the optimal conditions were conducted, five confirmatory experiments were carried out and the results of individual biodiesel conversion yield was shown in **Error! Reference source not found.**. The average of the five confirmatory e xperiments on the methyl ester yield was 97.65 % which is above the predicted methyl ester yield and their error was 0.26 % compared with the predicted value. As the results, the RSM with Box-behken experimental design is an effective tool for predicting the maximised methyl ester yield conversion by providing optimal conditions for a transesterification process.

Table 7. Experimental validation of the regression model under optimised conditions.

Methanol to oil ratio (v/v %)	Catalyst (v/v %)	Speed (rpm)	Time (min)	Biodiesel conversion yield	
				Predicted	Experimental
59.60	0.774	600	7.15	97.4	97.57
59.60	0.774	600	7.15	97.4	97.40
59.60	0.774	600	7.15	97.4	97.65
59.60	0.774	600	7.15	97.4	97.37
59.60	0.774	600	7.15	97.4	98.33

3.5 Properties of The Optimized *Sterculia Feotida* biodiesel

The physiochemical properties of the optimised blended biodiesel were determined and compare with other result shown in Table 8. The optimised blended biodiesel produced was compared with the biodiesel produced from conventional method. The results show that both blended biodiesel produced are compatible with the ASTM D6751 standard and EN 14214 standard.

Table 8. Physiochemical properties of *Sterculia feotida* biodiesel with ASTM and EN standards

Properties	Unit	Biodiesel				Diesel	Biodiesel Sterculia Feoitda	Optimized biodiesel *Sterculia feotida* Monowave	*Millettia pinnata* biodiesel [37]
		ASTM D6751	Test method	EN14214	Test method				
Kinematic viscosity at 40 °C	mm^2/s	1.9 to 6.0	D 445	3.5 to 5.0	EN ISO 3104	2.90	5.28	4.72	4.2
Density at 15 °C	kg/m^3	880	D 127	860 to 900	EN ISO 3675	842.5	875.3	860.1	860.0
Acid Value	mgKOH/g	0.5max	D 664	0.5max	EN 14104	0.015	0.55	0.46	0.14
Calorific Value	MJ/kg	-	D 240	-	-	45.361	40.91	41.35	39.94
Flash point	°C	100 to 170	D 93	101 min	EN ISO 22719	75.5	162.5	160.5	130
Copper strip corrosion	-	3 max	D 130	-	-	1a	1a	1a	-
FAME content	% (m/m)	-	-	90 min	EN 14103:2011	-	91.22	98.25	-
Linolenic-AME content	% (m/m)	-	-	1-15	EN 14103:2011	-	0.58	0.41	-
Monoglycerides	% (m/m)	-	-	0.8	EN 14105	-	0.288	0.255	-
Diglycerides	% (m/m)	-	-	0.2	EN 14105	-	0.135	0.045	-
Triglycerides	% (m/m)	-	-	0.2	EN 14105	-	0.215	0.161	-
Free glycerol	% (m/m)	0.02 max	D 6584	0.02	EN 14105	-	0.065	0.015	-
Total glycerol	% (m/m)	0.24 max	D 6584	0.25	EN 14105	-	0.184	0.113	-

4. Conclusion

This study demonstrates that monowave-assisted transesterification is a highly effective approach for enhancing biodiesel production from *Sterculia foetida* oil. The application of Response Surface Methodology (RSM) enabled systematic evaluation of key process variables and confirmed that reactant ratio, catalyst loading, and reaction time are the dominant factors governing conversion efficiency, while agitation plays a secondary role. The developed statistical model exhibited strong predictive capability, indicating its suitability for process optimization and scale-up considerations. The integration of monowave irradiation significantly improves reaction kinetics through rapid and uniform heating, allowing efficient conversion within a substantially reduced processing time compared to conventional methods. In addition, the biodiesel produced meets international fuel quality standards, confirming the viability of *Sterculia foetida* oil as a sustainable non-edible feedstock. This study highlights the potential of monowave technology as an energy-efficient and scalable process intensification strategy for biodiesel production, contributing to the advancement of sustainable biofuel technologies and supporting the transition toward low-carbon energy systems.

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CRediT Authorship Contribution Statement

Zaki Aqila and Zhafa Aulia Pawisty: Conceptualization, Methodology, Investigation, Data Curation, Writing Original Draft, Writing – Review & Editing, and Visualization; Adri Rakha Sebayang: Investigation, Data Curation and Formal Analysis; Dodi Tri Nugraha Winandar and Syarafina: Methodology, Formal Analysis; Deswita and Fayaz Hussain: – Review & Editing and Supervision.

Conflicts of Interest

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

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