

Optimization of continuous biodiesel production from refined palm oil using a rotating inert packed-bed reactor

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Abstract

This study investigates the application of a rotating inert packed-bed reactor (RIPB) for continuous production of biodiesel from refined palm oil (RPO) using sodium hydroxide (NaOH) as a homogeneous catalyst. The study examines the impact of catalyst loading, methanol-to-oil molar ratio, rotational speed and feed flow rate on biodiesel (or fatty acid methyl ester, FAME) yield. A central composite design (CCD) approach with response surface methodology (RSM) was used to identify the optimal conditions for achieving maximum biodiesel yield. A FAME yield as high as 97.9% was achieved at a NaOH loading of 1.258 wt.% of oil, a methanol-to-oil molar ratio of 5.5:1, a rotational speed of 1,280 rpm, a feed flow rate of 210 mL/min, and a reaction temperature of 60 °C. The performance of the RIPB was further compared with other intensified reactors including the rotating tube reactor, household hand blender, and spinning disc reactor. The RIPB demonstrated the short residence time (<5 s) and the high yield efficiency (20.9×10^{-4} g/J). The measured properties of the purified biodiesel were within their corresponding limits specified in EN 14214 and ASTM D6751. These results demonstrate the potential of the RIPB for intensified continuous biodiesel production.

Keywords: Biodiesel, Rotating packed bed reactor, Transesterification, Refined palm oil, Intensification reactor

1. Introduction

The demand for energy is constantly increasing, leading to the depletion of fossil fuels like oil and petroleum. Fuels that are renewable and sustainable are therefore needed. The low environmental impact of biodiesel and its high heating value have made it a promising alternative to petroleum and diesel fuel [1-4]. Industrial biodiesel is most frequently manufactured via transesterification, in which triglycerides react with a short-chain alcohol, typically methanol, to form fatty acid methyl esters (FAME) and glycerol [5-7]. The primary feedstocks used for biodiesel production are vegetable oils and animal fats. A wide range of catalysts has been employed for this reaction. Common catalysts include alkalis (e.g., NaOH, KOH, and CH_3ONa) and acids (e.g., H_2SO_4 , H_3PO_4 , and p-toluene sulfonic acid (PTSA)) [8]. Heterogeneous catalysts, such as metal oxides, sulfated zeolites, and hydrotalcite, are also used [9-11]. Homogeneous catalysts are commonly used because of their high catalytic activity and fast reaction rate. However, catalyst neutralization and additional downstream purification are generally required after the reaction. In contrast, heterogeneous catalysts are easier to separate from the reaction mixture, but their apparent reaction rates may be limited by additional solid-liquid mass-transfer resistance [7, 8].

In many practical systems, the overall rate of transesterification is strongly influenced by liquid-liquid mass transfer because vegetable oils and methanol are poorly miscible. Limited interfacial area and insufficient micromixing can therefore constrain conversion and productivity. Enhancing phase contact to reduce mass-transfer resistance is a key strategy to increase production rates, leading to higher biodiesel productivity, reduced production costs, and smaller reactor sizes [12]. Consequently, multifunctional reactor concepts, such as static mixer reactors [13], rotating tube reactors, hydrodynamic cavitation (HC) reactors [14, 15], reactive distillation (RD) [16], microreactors [17, 18], ultrasonic reactors [17], and microwave reactors [19, 20], have been explored and developed to overcome the heat and mass transfer limitations as well as thermodynamic constraints of biodiesel production. However, further improvement is still needed to achieve high FAME yield at short residence time under continuous operation.

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Among high-gravity contacting devices, the rotating packed-bed reactor (RPB) is a high-gravity technology that generates strong centrifugal fields through adjustable rotational speed [21]. The resulting thin films, droplets and intense turbulence within the packing can substantially improve mixing and mass transfer in multiphase systems. RPBs have been applied in diverse applications such as nanoparticle preparation [22, 23], gas–liquid contactors in absorption [24], and ozonation processes [25, 26]. From a manufacturing standpoint, conventional batch biodiesel production is often labor-intensive and time-consuming [27]. In contrast, continuous reactors allow for easy scaling up, which lowers production costs by increasing biodiesel production per unit of labor [28]. Recently, an RPB has been introduced for biodiesel production through K/ γ -Al₂O₃ catalyzed transesterification. The result showed that the RPB system achieved high FAME yield and less reactor volume for biodiesel production [21]. Although previous studies have demonstrated that rotating packed-bed reactors (RPB) can improve mass transfer and achieve high FAME yields, most systems require relatively high methanol-to-oil ratios and reactant recirculation to obtain high FAME yields. These operating conditions may increase process complexity and methanol recovery requirements. To facilitate single-pass continuous biodiesel production, a homogeneous catalyst that could be continuously introduced with the reactant feed was preferred. Therefore, NaOH was selected because it can be dissolved in the methanol feed and supplied independently of the packed bed. Nevertheless, effective liquid-liquid contacting remained essential because methanol and oil are poorly miscible. Therefore, the inert packed-bed elements were introduced into the rotating chamber solely as inert, non-catalytic contacting media to enhance liquid dispersion, interfacial renewal, and micromixing. The resulting configuration, in which NaOH provides the catalytic function in the liquid phase while the inert packing provides the contacting and mixing functions, is referred to as a rotating inert packed-bed reactor (RIPB).

In this study, a rotating inert packed-bed reactor (RIPB) was designed for continuous biodiesel production from refined palm oil using a homogeneous NaOH catalyst. The effects of four independent variables including, catalyst loading, methanol-to-oil molar ratio, rotational speed and feed flow rate on FAME yield were investigated. A central composite design (CCD) combined with response surface methodology (RSM) was employed to determine the optimal conditions. The RIPB performance was further benchmarked against other intensified reactors in terms of residence time and yield efficiency.

2. Methodology

2.1 Materials

The RPO (OLEEN brand) was obtained from a local supermarket in Pathum Thani, Thailand. It had an average molecular weight of 847.3 g/mol. Methanol (99.8%) and methyl heptadecanoate (GC internal standard) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Sodium hydroxide (NaOH, 97.5%) and heptane (99.7%) were obtained from Carlo Erba Reagents (Val-de-Reuil, France).

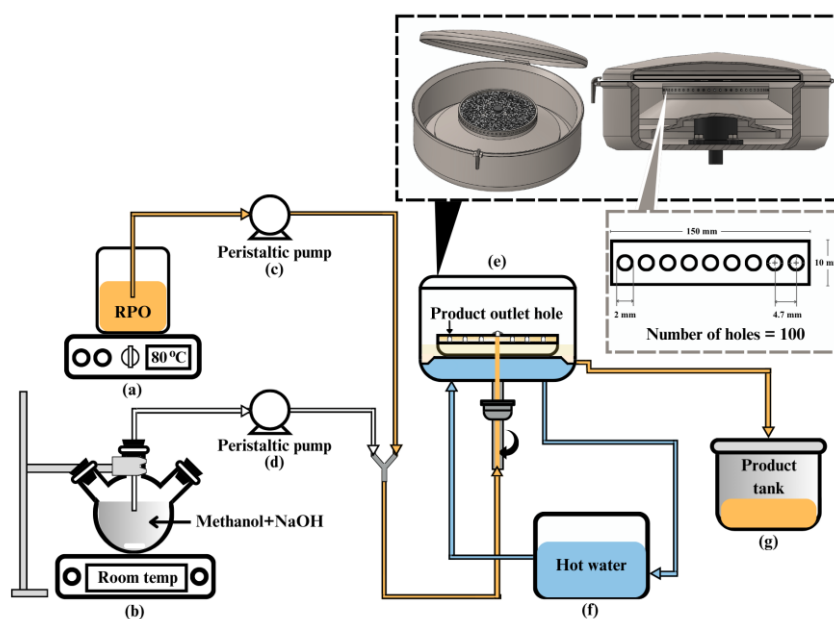


Figure 1 Schematic diagram of the RIPB. Components: (a) RPO feed tank and preheater, (b) 3-necked flask, (c, d) Peristaltic pumps, (e) RIPB, (f) Water bath, and (g) Product tank.

2.2 Experimental setup

Figure 1 shows a schematic of the experimental setup. The RIPB consisted of a packed-bed rotor, a motor, and a stationary cylindrical chamber. A stainless steel rotor disc had a diameter of 150 mm and a gap height of 10 mm. The inert packed bed was composed of cylindrical nylon elements (OD 5 mm, ID 3 mm, height 3 mm), with a total mass of 24.82 g, and was packed inside the rotating rotor chamber of the RIPB, as shown in Figure 1. It should be noted that the nylon packing elements were retained within the rotating chamber because their minimum external dimension (5 mm) was larger than the diameter of the product outlet holes (2 mm), thereby preventing the elements from leaving the chamber with the product stream. The inert packing promoted mixing between methanol and oil during continuous operation. For comparison, an experiment was also carried out without the inert packed bed. In this case, the nylon packing was removed from the rotating rotor chamber, while all other operating conditions, including catalyst loading, methanol-to-oil molar ratio, feed flow rate, and rotational speed, were kept the same. The reactant mixture passed through the rotating rotor chamber packed with inert nylon elements before leaving the reactor. The system temperature was maintained by

circulating hot water from an external water bath. The RPO was preheated at 80°C. The liquid feeds of RPO and methanol mixed with NaOH were continuously passed through the RIPB, while the rotor was operated at rotational speed ranging from 500 to 1,500 rpm. Since NaOH was dissolved in the methanol feed, the inert nylon packing functioned only as a mixing medium and did not contain catalyst particles. Two peristaltic pumps were used to introduce the substances into the reactor. The flow rate of the feed, molar ratio of methanol-to-oil, and the loading of NaOH catalyst were varied at 130-390 mL/min, 4.5-7.5, and 0.5-1.5 wt.% of oil, respectively. The residence time was estimated from the elapsed time between the injection of a sodium chloride (NaCl) tracer solution at the reactor inlet and first detectable conductivity response at the outlet. The measured residence time was less than 5 s. The reactor was operated at 60°C. After 20 min of continuous operation to ensure steady-state conditions, samples were collected and immediately quenched in an ice bath. The collected samples were then centrifuged at 3,000 rpm for 5 min to separate glycerol and methyl ester mixtures (biodiesel product). The FAME yield was determined using gas chromatography, and each experiment was conducted in triplicate.

2.3 Analytical methods

The FAME yield was determined by gas chromatography according to EN 14103. To analyze the sample, gas chromatography was performed using a Perkin Elmer ZB5 capillary column (0.25 mm x 30 m). Helium was used as the carrier gas, and the oven temperature ramp program started at 150°C and continued for five minutes. The temperature was then raised to 170°C at a rate of 10°C/min, followed by a five-minute hold and finally reaching 220°C at 3°C/min. The injector and detector temperatures were set at 250°C. The methyl ester yield was determined using Equation (1) [29].

$$\% \text{FAME yield} = \frac{(\sum A) - A_{EI}}{A_{EI}} \times \frac{C_{EI} \times V_{EI}}{m} \times 100\% , \quad (1)$$

where $\sum A$ = Surface area (C14:0 - C24:1) of all peaks
 A_{EI} = Peak area of methyl heptadecanoate standard
 C_{EI} = Concentration of methyl heptadecanoate solution (mg/mL)
 V_{EI} = Volume of methyl heptadecanoate solution in mL
 m = Mass of the biodiesel sample (mg)

The biodiesel produced under the optimum operating condition was further characterized. Ester content was determined according to EN 14103, while glycerides (monoglyceride, diglyceride, and triglyceride) and glycerol (free glycerol and total glycerol) contents were determined according to EN 14105. Flash point, viscosity, and density were also determined. The measured biodiesel properties were compared with the specifications of EN 14214 and ASTM D6751.

2.4 Experimental design

The experiments of transesterification of RPO with methanol using the NaOH catalyst in the RIPB was designed using Center Composite Design (CCD) with Design Expert. The reaction parameters were selected based on the limitations of the experimental equipment, the properties of the substances involved, the desired product specifications, and preliminary experimental results. The range of values for each chosen parameter in the experimental design matrix is provided in Table 1. The specific independent variables chosen were catalyst loading (X_1), methanol-to-oil molar ratio (X_2), rotational speed (X_3), and feed flow rate (X_4). The FAME yield was defined as the dependent variable (also known as a response) in the CCD model. Each independent variable was assigned levels of -1, 0, and +1, as shown in Table 1.

Table 1 An overview of the variables used in CCD design

Symbols	Variable	Coded levels		
		-1	0	1
X_1	Catalyst loading (wt.% of oil)	0.5	1	1.5
X_2	Methanol-to-oil molar ratio (-)	4.5	6	7.5
X_3	Rotational speed (rpm)	500	1,000	1,500
X_4	Feed flow rate (mL/min)	130	260	390

Residence time was not varied independently in the CCD, instead, total feed flow rate (X_4) was included as an operating factor associated with the characteristic contact time in the continuous-flow reactor. The CCD experimental matrix is presented in Table 2. A total of 30 runs was required to determine how each independent variable interacted with each other. An analysis of variance (ANOVA) was used to determine the significance of the model.

Table 2 The response values and design matrix of the CCD

Run	Experimental variables				FAME yield (%)		
	X_1	X_2	X_3	X_4	Experimental	Predicted	Residual
1	0.5	4.5	500	130	65.83	64.30	1.53
2	1.5	4.5	500	130	86.10	86.15	-0.05
3	0.5	7.5	500	130	59.59	61.41	-1.82
4	1.5	7.5	500	130	85.90	86.06	-0.16
5	0.5	4.5	1500	130	72.00	74.01	-2.01

Table 2 (continued) The response values and design matrix of the CCD

Run	Experimental variables				FAME yield (%)		
	X ₁	X ₂	X ₃	X ₄	Experimental	Predicted	Residual
6	1.5	4.5	1500	130	96.35	95.73	0.62
7	0.5	7.5	1500	130	73.57	70.72	2.85
8	1.5	7.5	1500	130	93.70	95.22	-1.52
9	0.5	4.5	500	390	71.07	71.24	-0.17
10	1.5	4.5	500	390	80.96	82.02	-1.06
11	0.5	7.5	500	390	66.81	65.63	1.18
12	1.5	7.5	500	390	79.50	79.19	0.31
13	0.5	4.5	1500	390	74.60	72.64	1.96
14	1.5	4.5	1500	390	83.40	83.27	0.13
15	0.5	7.5	1500	390	64.98	66.61	-1.63
16	1.5	7.5	1500	390	80.30	80.04	0.26
17	0.5	6	1000	260	74.21	76.13	-1.92
18	1.5	6	1000	260	95.30	93.77	1.53
19	1	4.5	1000	260	88.60	89.54	-0.94
20	1	7.5	1000	260	87.03	86.48	0.55
21	1	6	500	260	88.10	87.86	0.24
22	1	6	1500	260	92.51	93.14	-0.63
23	1	6	1000	130	93.27	92.70	0.57
24	1	6	1000	390	87.61	88.58	-0.97
25	1	6	1000	260	92.10	92.32	-0.22
26	1	6	1000	260	91.80	92.32	-0.52
27	1	6	1000	260	94.00	92.32	1.68
28	1	6	1000	260	91.45	92.32	-0.87
29	1	6	1000	260	93.80	92.32	1.48
30	1	6	1000	260	92.00	92.32	-0.32

3. Results and discussion

3.1 Effect of inert packed bed

The biodiesel production in the RIPB was operated using a NaOH catalyst loading of 1 wt.% of oil, a methanol-to-oil molar ratio of 6:1, a feed flow rate of 260 mL/min, a rotational speed of 500 rpm and a temperature of 60°C. The residence time was less than 5 s under this operating condition. A 20-min start-up stabilization period was allowed before sample collection. Figure 2 compares the FAME yields obtained with and without inert packed bed. Under the same operating condition, the FAME yield increased from 74.3% without the inert packing to 88.1% with the inert packing. This improvement may be attributed to increased liquid dispersion, interfacial renewal, and micromixing within the rotating chamber.

Consequently, the combination of the inert packed bed within the RIPB significantly enhances the contact of reactants, resulting in increased mass transfer efficiency. In addition, the mass transfer phenomenon in the liquid film is also dependent on various factors, including inert packing geometry, liquid properties, flow dynamics, and gravitational effects [30]. The utilization of inert packing within the rotor of the RIPB could increase the shear force of liquid, resulting in dispersion and micro-mixing to improve mass transfer [31]. The observed increase in FAME yield therefore indicates that the inert packing improved liquid–liquid contacting under the investigated conditions. Based on this result, the packed configuration was used in the subsequent CCD experiments.

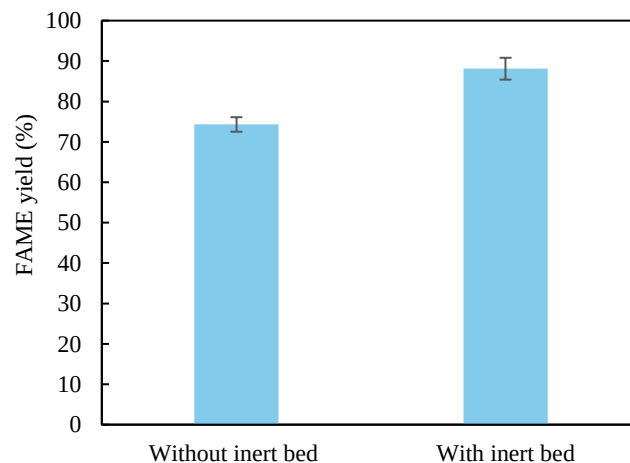


Figure 2 Comparison between RIPB with and without inert packed bed on biodiesel production.

3.2 Fitting of the model

To evaluate the response surface model and ANOVA, the experimental results from Table 2 were employed. Several models were fitted to the experimental results (as shown in Table 3), including linear, 2FI, quadratic, and cubic polynomial models. Based on the sequential model sum of squares and Fisher's F-test, the quadratic model was selected (p -value < 0.0001), indicating that the model was statistically significant. F-values and p-values were used to evaluate the significance of individual terms; a larger F-value and a smaller p-value indicate a more significant effect. At a 95% confidence level, terms with p -value < 0.05 were considered statistically significant (Table 4) [32]. The model showed a high R^2 of 0.9862, indicating that 98.62% of the response variability was explained by the model (only 1.38% remained unexplained). The predicted R^2 (0.9086) was in reasonable agreement with the adjusted R^2 (0.9733). The model also exhibited an adequate precision of 26.0805 (>4) and a low coefficient of variation (C.V.) of 2.08%, indicating good signal-to-noise ratio and satisfactory experimental reproducibility. In addition, the lack-of-fit was not significant ($p = 0.1007$), confirming that the quadratic model adequately represented the experimental data. The final quadratic model in terms of coded factors is given in Eq. (2):

$$Y = 92.32 + 8.82X_1 - 1.53X_2 + 2.64X_3 - 2.06X_4 + 0.6963X_1X_2 - 0.0350X_1X_3 - 2.77X_1X_4 - 0.1025X_2X_3 - 0.6825X_2X_4 - 2.08X_3X_4 - 7.37X_1^2 - 4.31X_2^2 - 1.82X_3^2 - 1.68X_4^2 \quad (2)$$

where Y is the FAME yield, and X_1 , X_2 , X_3 and X_4 are the coded variables. Among the linear main effects, catalyst loading (X_1) had the strongest influence on FAME yield ($F=467.74$), followed by rotational speed (X_3 , $F=41.91$), feed flow rate (X_4 , $F=25.49$), and methanol-to-oil molar ratio (X_2 , $F=14.05$). However, the significant X_1X_4 and X_3X_4 interaction terms and the significant X_1^2 and X_2^2 quadratic terms indicate that the response cannot be interpreted solely from the linear main effects. The individual and combined effects of these operating variables are discussed further in Section 3.3. The predicted FAME yields were in good agreement with the experimental values (Figure 3). The predicted responses were distributed close to the 45° identity line. The model exhibited a root-mean-square error of approximately 1.73%, and the maximum absolute residual was approximately 2.85%. It was indicated that the developed quadratic model adequately described the experimental data. This is consistent with the high R^2 value and the non-significant lack-of-fit shown in Table 4.

Table 3 The sum of squares of the sequential model

Source	Sum of squares	DF ^a	Mean square	F value	P value	Remarks
Mean	207700	1	207700			
Linear	1645.95	4	411.49	6.39	0.0011	
2FI	207.52	6	34.59	0.4688	0.8229	
Quadratic	1356.88	4	339.22	113.18	<0.0001	Suggested
Cubic	26.23	8	3.28	1.23	0.4006	Aliased
Residual	18.72	7	2.67			
Total	211000	30	7033.19			

Suggested model is indicated by bold words.

^aDegree of freedom.

Table 4 ANOVA for the quadratic model based on the response surface

Source	Sum of squares	DF	Mean square	F-Value	p value	Remarks
Model	3210.36	14	229.31	76.51	<0.0001	significant
X_1	1401.85	1	1401.85	467.74	<0.0001	
X_2	42.11	1	42.11	14.05	0.0019	
X_3	125.61	1	125.61	41.91	<0.0001	
X_4	76.38	1	76.38	25.49	0.0001	
$X_1 X_2$	7.76	1	7.76	2.59	0.1285	
$X_1 X_3$	0.0196	1	0.0196	0.0065	0.9366	
$X_1 X_4$	122.99	1	122.99	41.04	<0.0001	
$X_2 X_3$	0.1681	1	0.1681	0.0561	0.8160	
$X_2 X_4$	7.45	1	7.45	2.49	0.1357	
$X_3 X_4$	69.14	1	69.14	23.07	0.0002	
X_1^2	140.72	1	140.72	46.95	<0.0001	
X_2^2	48.12	1	48.12	16.06	0.0011	
X_3^2	8.58	1	8.58	2.86	0.1113	
X_4^2	7.35	1	7.35	2.45	0.1381	
Residual	44.96	15	3.00			not significant
Lack of fit	39.02	10	3.90	3.28	0.1007	
Pure error	5.94	5	1.19			
Total	3255.32	29				

The statistical data provided shows an R^2 value of 0.9862 and an adjusted R^2 value of 0.9733, with a predicted R^2 value of 0.9086 and an adequate precision of 26.0805. The coefficient of variation (C.V.^a) is 2.08

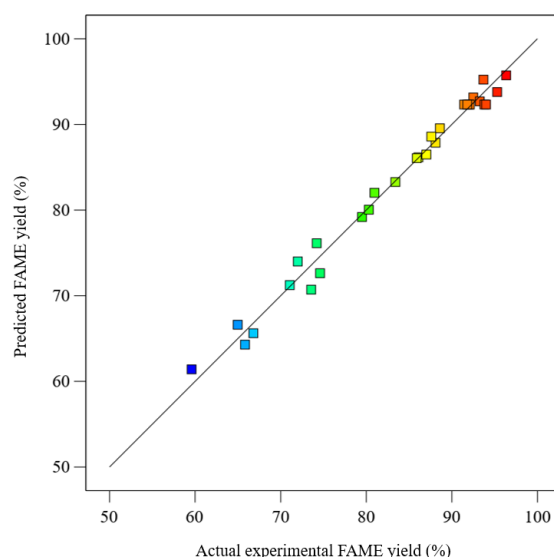


Figure 3 Predicted versus actual experimental values of FAME yield (%) from transesterification of RPO with methanol in RIPB.

3.3 Effects of operating parameters on FAME yield

Response surface plots were employed to visualize the effects of the four coded factors (X_1 - X_4 , catalyst loading, methanol-to-oil molar ratio, rotational speed and feed flow rate) on FAME yield in the RIPB system. Figures 4-7 present the three-dimensional response surfaces and corresponding contour plots, which facilitate identification of the region giving maximum predicted FAME yield. The 3D response surface plot in Figure 4 shows the impact of changes in catalyst loading (X_1) and methanol-to-oil molar ratio (X_2) on the variation of FAME yield. The FAME yield varied with NaOH loading between 0.5 and 1.5 wt.% of oil and a methanol-to-oil molar ratio ranging from 4.5 to 7.5. The lowest FAME yield was observed with 0.5 wt.% NaOH, indicating an incomplete reaction due to the minimal quantity of methoxide anion as an active site [12]. A higher catalyst amount leads to faster reactions, so increasing the catalyst amount from 0.5 to 1.1 wt.% of oil increased the FAME yield. However, further increasing catalyst loading (>1.1 wt.% of oil) may promote saponification, leading to soap formation and emulsion stabilization, which complicates phase separation and can reduce apparent biodiesel yield [12, 33]. With respect to methanol-to-oil ratio, the yield increased up to an intermediate value (approximately 5.6–5.8) and then slightly decreased at higher ratios. This behavior may be attributed to dilution of reactants at excessive methanol levels and increased separation burden downstream. Notably, although the surface shows curvature, the X_1X_2 interaction term was not statistically significant according to ANOVA (Table 4), indicating that the observed trend is primarily governed by the main effects and quadratic terms [33]. The stoichiometric methanol-to-triglyceride molar ratio for complete transesterification is 3:1. However, excess methanol is generally required in practice to shift the reversible reaction toward FAME formation and improve oil conversion. In this study, the optimum methanol-to-oil molar ratio was 5.503:1 corresponds to approximately 1.83 times the stoichiometric requirement. This value was lower than the 24:1 ratio employed in the heterogeneous, recirculating RPB system [21]. However, the differences among these values should not be attributed solely to reactor hydrodynamics because the studies also differed in catalyst type, feedstock, packing function, and operating mode.

Figure 5 shows the response surface for catalyst loading (X_1) and rotational speed (X_3). Increasing rotational speed from 500 to 1,500 rpm improved the FAME yield. The significant positive linear effect of rotational speed indicated that increasing the rotational speed from 500 rpm generally increased the predicted FAME yield, although the benefit became less pronounced toward the upper end of the investigated range. Higher rotational speed may increase centrifugal acceleration and local liquid shear within the inert packed bed, thereby promoting liquid-film breakup, dispersion, interfacial renewal, and micromixing between methanol and oil. Because these hydrodynamic quantities were not measured directly, the observed yield improvement is interpreted as being consistent with enhanced liquid-liquid contacting rather than as direct evidence of an increased mass-transfer coefficient. Although the response surface shows curvature, the ANOVA results indicate that the X_1X_3 interaction was not significant (Table 4), suggesting that rotational speed and catalyst loading mainly influenced the FAME yield through their individual (main) effects rather than their interaction.

Figure 6 presents the interaction between catalyst loading (X_1) and feed flow rate (X_4). In this range, catalyst loading exerted a pronounced effect on the FAME yield, whereas the effect of feed flow rate was comparatively smaller. The ANOVA results confirmed a significant X_1X_4 interaction (Table 4), implying that the influence of flow rate depends on catalyst loading. This behavior is consistent with changes in residence time and mixing intensity at different operating conditions. Figure 7 presents the interaction between rotational speed (X_3) and feed flow rate (X_4). Increasing the rotational speed improved the FAME yield, whereas increasing the feed flow rate slightly reduced the FAME yield. The higher rotational speed enhanced liquid dispersion and micromixing within the inert packed bed, resulting in improved mass transfer between methanol and oil. In contrast, increasing the feed flow rate reduced the residence time of the reactants in the reactor, thereby limiting the extent of the transesterification reaction and slightly decreasing the FAME yield. The ANOVA results confirmed a significant X_3X_4 interaction (Table 4), indicating that the effect of feed flow rate on FAME yield depended on the rotational speed. This behavior is consistent with the combined effects of residence time and mixing intensity under different operating conditions.

Numerical optimization predicted a maximum FAME yield of 97.2% at a NaOH loading of 1.258 wt.% of oil, a methanol-to-oil molar ratio of 5.503:1, a rotational speed of 1,283 rpm, and a feed flow rate of 210.5 mL/min. Validation experiments were subsequently conducted at the practically rounded conditions of 1.258 wt.% NaOH, a methanol-to-oil molar ratio of 5.5:1, a rotational speed of 1,280 rpm, and a feed flow rate of 211 mL/min. The experimental FAME yield was 97.9%, compared with the model-predicted value of 97.2%, corresponding to an absolute deviation of 0.7%. This agreement confirms the adequacy of the developed model for navigating the operating window and identifying high-yield conditions.

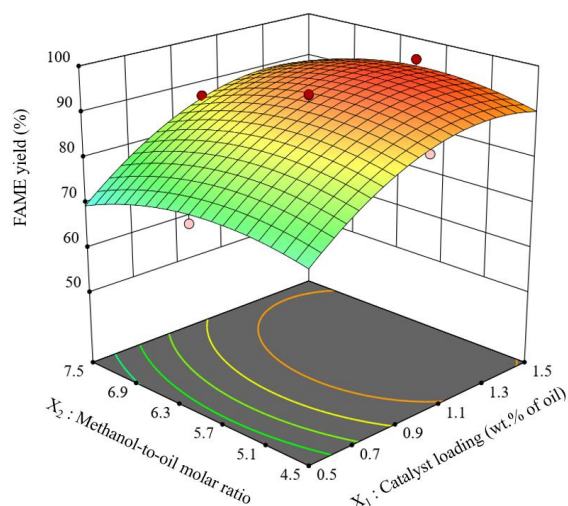


Figure 4 Response surface plot for transesterification of RPO as functions of catalyst loading (X_1) and methanol-to-oil molar ratio (X_2).

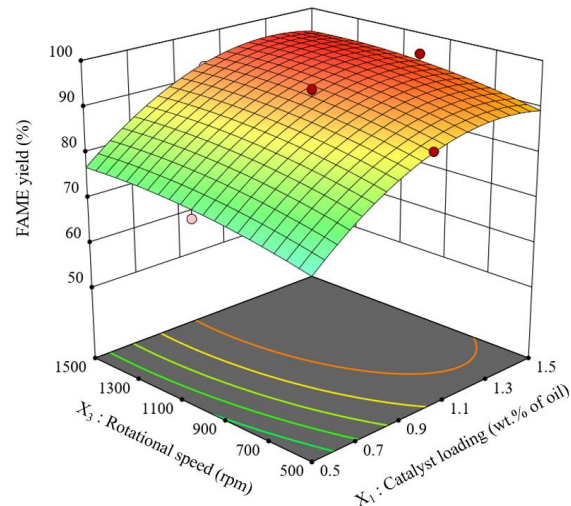


Figure 5 Response surface plot for transesterification of RPO as functions of catalyst loading (X_1) and rotational speed (X_3).

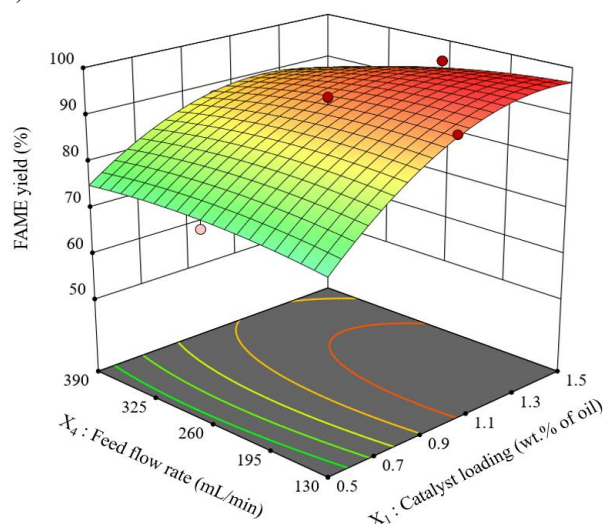


Figure 6 Response surface plot for transesterification of RPO as functions of catalyst loading (X_1) and feed flow rate (X_4).

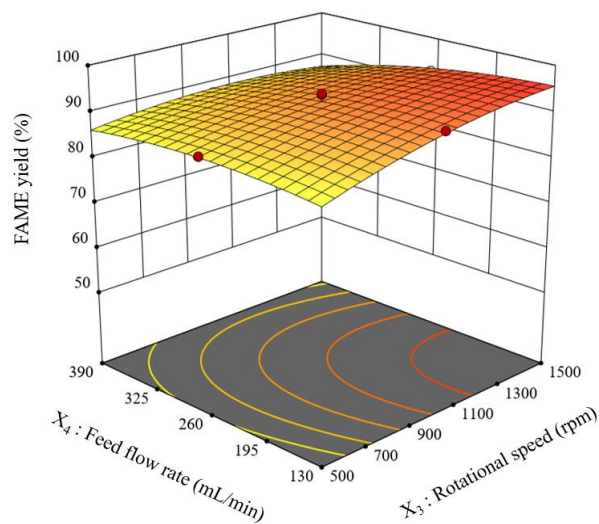


Figure 7 Response surface plot for transesterification of RPO as functions of rotational speed (X_3) and feed flow rate (X_4).

3.4 Biodiesel analysis

Selected properties of the purified biodiesel produced under the validated optimum operating condition were determined to evaluate the quality of the resulting product. The validation condition was a NaOH loading of 1.258 wt.% of oil, a methanol-to-oil molar ratio of 5.5:1, a rotational speed of 1,280 rpm, a feed flow rate of 211 mL/min, and a reaction temperature of 60 °C. The measured properties included ester content, monoglyceride, diglyceride, triglyceride, free glycerol, total glycerol, flash point, kinematic viscosity at 40 °C, and density at 15 °C. As shown in Table 5, the purified biodiesel had an ester content of 97.9% by mass. The monoglyceride, diglyceride, triglyceride, free glycerol, and total glycerol contents were below their corresponding maximum limits. The measured flash point, kinematic viscosity, and density were also within their applicable specification ranges. Accordingly, all properties measured in this study were within the corresponding limits specified in EN 14214 and ASTM D6751.

Table 5 Biodiesel properties of the product obtained under the optimum operating condition.

Property	Specification	This work	Unit
Ester content	96.5 (min.)	97.9	% m/m
Monoglyceride	0.80 (max.)	0.55	% m/m
Diglyceride	0.20 (max.)	0.18	% m/m
Triglyceride	0.20 (max.)	0.079	% m/m
Free glycerol	0.02 (max.)	<0.001	% m/m
Total glycerol	0.25 (max.)	0.11	% m/m
Flash point	130 (min.)	251	°C
Kinematic viscosity at 40 °C	1.9-6.0	4.82	mm ² /s
Density at 15 °C	860-900	892	kg/m ³

min. = minimum requirement; max. = maximum allowable value. The specification values are based on EN 14214 and ASTM D6751.

3.5 Performance comparison with different intensified reactors

Recently, several intensified reactors have been used for continuous biodiesel production. Table 6 summarizes the biodiesel production performance in terms of biodiesel yield and yield efficiency of various reactor types, and their optimal operating conditions. The yield efficiency is one of the parameters used to evaluate the energy efficiency of biodiesel production. It is defined as the rate of biodiesel production per rate of energy consumption. Under the validated optimum conditions, the RIPB achieved a FAME yield of 97.9% at a NaOH loading of 1.258 wt.% of oil, a methanol-to-oil molar ratio of 5.5:1, a rotational speed of 1,280 rpm, a feed flow rate of 211 mL/min, and a reaction temperature of 60 °C. The estimated residence time was less than 5 s, and the reported yield efficiency was 20.9×10^{-4} g/J. The RIPB yield efficiency was lower than the value of 37.5×10^{-4} g/J reported for the rotating tube reactor [12] but higher than the values of 18.0×10^{-4} and 13.7×10^{-4} g/J reported for the household hand blender [34] and spinning-disc reactor [33], respectively. Therefore, the yield efficiency of the RIPB was within the range reported for the selected intensified reactors. Chen et al. [21] reported a FAME yield of 98.5% using a conventional rotating packed-bed reactor (RPB) with a K/ γ -Al₂O₃ catalyst. However, their system employed a methanol-to-oil molar ratio of 24:1 and a reaction-mixture recirculation time of 60 min in an RPB configuration. In the present study, NaOH was first dissolved in methanol and continuously fed into the RIPB. The reactant mixture then passed through the rotating rotor chamber containing inert nylon packing, which promoted mixing between methanol and oil and enhanced mass transfer. A FAME yield of 97.9% was achieved at a methanol-to-oil molar ratio of 5.5:1 and a residence time of less than 5 s. Overall, the RIPB showed comparable performance to other intensified reactors while operating with very short residence time under continuous operation.

Table 6 Performance comparison of various continuous intensified reactors for transesterification

Reactor	Feedstock	Operating conditions	Residence Time (min)	FAME yield (%)	Yield efficiency ($\times 10^{-4}$ g/J)	Ref.
Rotating inert packed-bed reactor	Refined palm oil	Methanol-to-oil molar ratio = 5.5:1 NaOH loading = 1.258 wt.% of oil Rotational speed = 1,280 rpm Total feed flow rate = 211 mL/min Temperature = 60 °C	< 5 s	97.9	20.9	This study
Rotating tube reactor	Refined palm oil	Methanol-to-oil molar ratio = 6:1 Catalyst = 1 wt.% NaOH Tube rotational speed = 1,000 rpm Total feed flow rate = 30 mL/min Temperature = 30°C (ambient)	11.23	97.5	37.5	[12]
Rotating packed bed	Soybean oil	Methanol-to-oil molar ratio = 24:1 Catalyst = 7.05 wt.% K/ γ -Al ₂ O ₃ Rotor speed = 900-1500 rpm CSTR stirring speed = 800 rpm Circulation flow rate = 250 mL/min Temperature = 60°C	60*	98.5	-	[21]
Household hand blender	Refined palm oil	Methanol-to-oil molar ratio = 6:1 Catalyst = 0.8 wt.% NaOH Total feed flow rate = 12 mL/min Maximum impeller speed = 10,640 rpm Temperature = 60°C	< 16.7	96.6	18.0	[34]
Spinning disk reactor	Refined palm oil	Methanol-to-oil molar ratio = 6:1 Catalyst = 1 wt.% NaOH Rotational speed = 1,000 rpm Total feed flow rate = 260 mL/min Temperature = 60°C	< 1	97.0	13.7	[33]
Static mixers tubular reactor	Soybean oil	Methanol-to-oil molar ratio = 6:1 Catalyst = 2 wt.% KOH Static mixer packing dual-size stainless steel spheres (2.5 and 1.0 mm) Total feed flow rate = 6.10 mL/min Temperature = 60°C	< 1	93.7	-	[35]
Packed tubular reactor	Soybean oil	Methanol-to-oil molar ratio = 6:1 Catalyst = 2 wt.% KOH Dual-size stainless steel sphere packing (2.5 and 1.0 mm) Oil flow rate = 4.85 mL/min Methanol flow rate = 1.25 mL/min Total feed flow rate = 6.10 mL/min Temperature = 60°C	< 1	98.2	-	[36]
Oscillatory flow reactor	Rapeseed Oil	Methanol-to-oil molar ratio = 6:1 Catalyst = 1.5 wt.% KOH Oscillatory reynolds number = 160 Net reynolds number = 3.48 Temperature = 60°C	5	97.0	-	[37]
Centrifugal contactor separator	Sunflower oil	Methanol-to-oil molar ratio = 6:1 Catalyst = 1 wt.% NaOCH ₃ Rotational frequency = 30 Hz (1800 rpm) Oil flow rate = 12.6 mL/min Methanol flow rate = 3.15 mL/min Total feed flow rate = 15.75 mL/min Temperature = 75°C	11.43	96.0	-	[38]

*Reaction time in a recirculating RPB system, rather than the single-pass residence time.

4. Conclusions

In this study, a rotating inert packed-bed reactor (RIPB) containing hollow cylindrical nylon elements was evaluated for the single-pass continuous transesterification of refined palm oil using NaOH as a homogeneous catalyst. Under the same operating conditions, the addition of the inert packing increased the FAME yield from 74.3% to 88.1%. This improvement was consistent with enhanced liquid–liquid contacting, dispersion, and micromixing within the rotating chamber. Response surface methodology combined with central composite design (RSM-CCD) was employed to evaluate the effects of the operating parameters and determine the optimum conditions for biodiesel production. Numerical optimization predicted a maximum FAME yield of 97.2% at a NaOH loading of 1.258 wt.% of oil, a methanol-to-oil molar ratio of 5.503:1, a rotational speed of 1,283 rpm, a feed flow rate of 210.5 mL/min, and a reaction temperature of 60 °C. Experimental validation conducted at a NaOH loading of 1.258 wt.% of oil, a methanol-to-oil molar ratio of 5.5:1, a rotational speed of 1,280 rpm, a feed flow rate of 211 mL/min, and a reaction temperature of 60 °C produced a FAME yield of 97.9% with a residence time of less than 5 s. The biodiesel produced under the optimum operating conditions satisfied the fuel quality specifications of EN 14214 and ASTM D6751. The RIPB significantly reduced the residence time and exhibited high yield efficiency, demonstrating its potential as an efficient reactor for continuous biodiesel production.

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