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Research article

Enhancing biodiesel yield and purification with a recently developed centrifuge machine: A response surface methodology approach

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ABSTRACT

Biodiesel production processes, such as gravity settling, have limitations in terms of biodiesel yield, purification efficiency, operating time in the separation process, and more extensive equipment. Therefore, this study has focused on using a recently developed centrifuge machine for biodiesel separation to address these challenges due to its compact design, high efficiency, and simplicity. Additionally, this study aimed to optimize the separation efficiency of glycerol from biodiesel using a centrifuge machine, employing response surface methodology (RSM) with central composite design (CCD). The optimum conditions for separating glycerol from biodiesel via centrifuge machine are a rotation speed of 1800 rpm, a mixture flow rate of 192.25 ml/min, and a temperature of 55 °C, respectively. In optimum conditions, 94.52% separation efficiency was achieved. Biodiesel production can be improved, leading to higher yields and greater purity. The utilization of RSM proved valuable in determining the optimum conditions for separation. Furthermore, the machine successfully separated the biodiesel to meet ASTM D6751 and EN 14,214 standards. The results highlight the potential of the centrifuge machine for efficient and reliable biodiesel production, contributing to the advancement of the biodiesel industry.

1. Introduction

Alternative energy development to replace fossil fuels is becoming increasingly attractive because alternative energy has no negative environmental impact due to its potential to reduce greenhouse gas emissions [1,2]. One popular emerging energy alternative is biodiesel, a fuel from renewable resources such as vegetable oil, animal fat, algae, or used vegetable oil. It has similar combustion properties to diesel [3–6]. The production of biodiesel involves the transesterification of triglycerides with an alcohol in the presence of a catalyst. This process results in the formation of biodiesel and glycerol as major byproducts [7–9]. Separating these two components efficiently is important to obtain high-quality biodiesel that meets the quality standards for commercial use. However, glycerol can

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harm engines because of incomplete combustion [10]. Therefore, separating glycerol from biodiesel before usage is an important step.

Conventional separation methods, such as gravitational settling, have efficiency and time requirements limitations. In recent years, the development of centrifuge machines specifically designed for biodiesel separation has developed as a promising alternative [11]. These centrifuges use the principles of centrifugal force to enhance the separation of fluids of different specific gravity and accelerate particle sedimentation rates [12]. Centrifuges are used in biological medicine, agriculture, food, and so on [13–15]. Nevertheless, the application of centrifuges for separating glycerol from biodiesel is limited. The biodiesel separation process using centrifuge machines is necessary to understand the interactive effects of various parameters operation, which could influence the separation efficiency and overall biodiesel yield during centrifugation. One key attribute is the interaction between rotation speeds, mixture flow rates, and temperature [16,17]. Previous research has shown that creating relationships between factors can help increase productivity [18]. However, the relationship between these variables and their influence on separation efficiency has not been thoroughly investigated. Furthermore, determining the optimal values for these variables can lead to improved process efficiency, higher yields, and cost-effective biodiesel production. One approach that can be employed for this purpose is Response Surface Methodology (RSM).

RSM is a statistical experimental design and analysis technique widely beneficial in optimizing complex processes. It allows for the modeling and analysis of complex interactions among multiple variables and determines the optimal operating conditions for a desired response [19–21]. Consequently, this method has been considerably used for optimization to maximize or target the production of a special substance in several fields of study [22–25]. The response surface model can estimate the coefficients and assess the statistical significance of the variables and their interactions. Therefore, this research aims to investigate and establish the interaction relationship between rotation speed, mixture flow rate (biodiesel and glycerol), and mixture temperature to optimize biodiesel yield using the recently developed centrifuge machine.

2. Materials and methods

2.1. Production of biodiesel

The production of biodiesel was carried out using a 1 L capacity reactor. The catalyst was KOH, with a molar ratio of methanol to oil set at 9:1 (1% w/v) [26]. The mixture of methanol and KOH was added to the reactor, followed by the preheated oil at 60 °C. The stirring rate during the process was set to 500 rpm. The biodiesel production process remained in specific operating conditions, with a temperature of 60 °C and a reaction time of 45 min.

2.2. Experimental set-up

The crude biodiesel-glycerol mixture was separated using a recently developed centrifuge machine, as illustrated in Fig. 1. The warm tank was equipped with 0.8 kW heaters to maintain the desired temperature of the mixture. The centrifuge machine was equipped with a chemical pump with a capacity of 13.8 L/h and a 200 mm diameter and 50 mm height stainless steel cylindrical bowl.

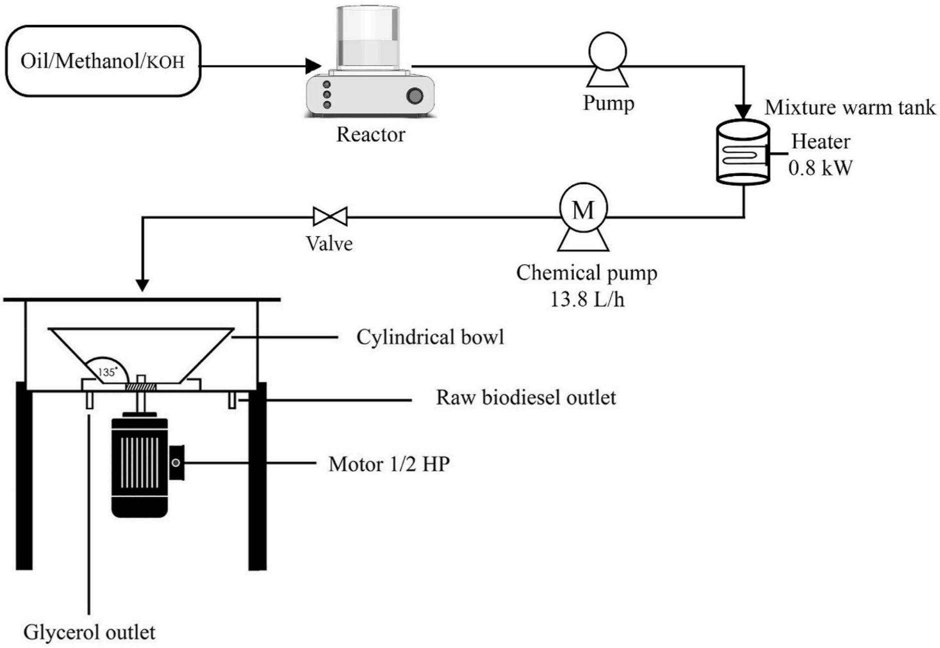


Fig. 1. Schematic diagram of a recently developed centrifuge machine.

A 1/2 H P motor was employed to rotate the centrifuge bowl to drive the separation process.

2.3. Procedure for the separating experiment

In the biodiesel separation process using the recently developed centrifuge machine, a mixture contained in a warm tank was fed into the cylindrical bowl of the centrifuge. It was separated at various combinations of mixture temperature (55–65 °C), flow rate (150–250 ml/min), and revolution speed (1800–2400 rpm). The operating conditions for the temperature, flow rate, and revolution speed were controlled and varied within identified ranges according to the experimental design. After the mixture was fed into the cylindrical bowl, the centrifuge machine was activated, and the bowl was continually centrifuged for a specific duration of 30 s to obtain the maximum separated crude biodiesel. After centrifugation, the motor was stopped, and the separated crude biodiesel was collected and measured for each experimental run. Each procedure was conducted three times.

The separation efficiency of the developed machine was determined by using the following equation:

$$\eta = \frac{V_{B,out}}{V_B} \times 100 \tag{1}$$

Where η is the separation efficiency; $V_{B,out}$ is the separated crude biodiesel content (ml) and V_B is the initial crude biodiesel content (ml).

2.4. Statistical analysis

The response surface methodology (RSM) utilizes mathematical and statistical approaches to model and analyze a process for optimizing the conditions based on the minimized number of experiments in which the response of interest is affected by several independent variables [27]. The central composite design (CCD) is the most used response surface to design experiments because it is suitable for sequential experiments in obtaining appropriate information for testing lack of fit without many design points and is suitable for calibrating full quadratic models. This work has followed RSM and CCD to find the values of independent parameters for an optimized separation efficiency of the centrifuge machines for separating glycerol from biodiesel. Three factors, including the rotation speeds (X_1), the mixture flow rates (X_2), and temperature (X_3), where all factors are coded into three levels, are considered in this work. The factors, ranges, and levels of the investigated variables are listed in Table 1. The distance of the axial points from the center is coded as -1.682 ($-\alpha$) and $+1.682$ ($+\alpha$).

RSM, generated by Design-Expert 11 software, was employed to evaluate the experimental data to optimize conditions for the separation efficiency of glycerol from biodiesel via a centrifuge machine. This experiment required twenty runs to evaluate the pure error, including eight factorial points, six axial points, and six replicates at the center point. Table 2 shows the complete design matrix of CCD, including actual and coded independent variables. The data obtained were fitted with an empirical quadratic model as follows:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum\sum_{i<j} \beta_{ij} X_i X_j + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \varepsilon \tag{2}$$

Where Y is separation efficiency as the response, X_i, and X_j are independent variables, β_0 is the intercept, β_i is the first-order coefficient of the model, β_{ii} is the quadratic order coefficient of the model, β_{ij} be the coefficient of the interaction between i and j factors, k is the number of factors studied and optimized in the experiment, and ε is the experimental error attributed to the response variable.

2.5. Characterization of biodiesel

The biodiesel samples were evaluated for their physicochemical properties according to the standards set by ASTM D6751 and EN14214 [28]. The kinematic viscosity of the biodiesel samples was analyzed following ASTM D-445, employing a Cannon-Fenske Routine capillary viscometer. In the case of ester content, gas chromatography with a flame ionization detector (GC-FID), (Agilent (GC-Agro), USA) was employed for the analysis according to EN14103. Free glycerol and triglycerides in biodiesel were determined by GC-FID (Hewlett Packard, 6890, USA), according to BS EN14105, whereas the methanol content was determined by headspace gas

Table 1
RSM experimental design for three variables at three levels showing coded and uncoded values.

Variables presented in coded form						
Variables	Symbol	Level				
		$-\alpha$	−1	0	1	$+\alpha$
Rotation speeds, rpm	x_1	1595.46	1800	2100	2400	2604.54
Mixture flow rates, ml/min	x_2	115.91	150	200	250	284.09
Temperature, °C	x_3	51.59	55	60	65	68.41

Transformation of variable levels from coded (X) to uncoded was obtained as: $x_1 = 300X_1 + 2100$, $x_2 = 50X_2 + 200$, $x_3 = 5X_3 + 60$..

Table 2
Experimental and predicted separation efficiency of centrifuge for separating glycerol from biodiesel using CCD.

No.	Rotation speeds (x_1)		Flow rates (x_2)		Temperature (x_3)		Efficiency	Predicted Efficiency
	UC.	C.	UC.	C.	UC.	C.		
1	2100	0	200	0	60	0	90.29	90.3495
2	2100	0	200	0	60	0	90.21	90.3495
3	1800	−1	250	1	65	1	87.42	87.6735
4	2400	1	150	−1	55	−1	89.52	89.4257
5	1595.46	$-\alpha$	200	0	60	0	90.29	90.1235
6	2100	0	200	0	60	0	90.29	90.3495
7	2400	1	250	1	65	1	88.43	88.5581
8	2100	0	200	0	51.591	$-\alpha$	92.52	92.3845
9	2100	0	115.91	$-\alpha$	60	0	89.40	59.5749
10	2100	0	200	0	60	0	90.25	90.3495
11	2604.54	α	200	0	60	0	89.08	89.0214
12	1800	−1	250	1	55	−1	91.26	91.4527
13	2100	0	200	0	68.409	α	89.28	89.1904
14	2400	1	250	1	55	−1	90.29	90.5174
15	1800	−1	150	−1	65	1	89.85	89.7818
16	1800	−1	150	−1	55	−1	91.59	91.6210
17	2400	1	150	−1	65	1	89.44	89.4064
18	2100	0	284.09	α	60	0	88.52	88.7200
19	2100	0	200	0	60	0	90.57	90.3495
20	2100	0	200	0	60	0	90.45	90.3495

UC.: Uncoded value, C: Coded value.

chromatography with flame ionization detection (HS-GC/FID) based on BS EN14110. The acid value of biodiesel was determined using potentiometric titration (Titrando, Metrohm, USA), according to ASTM D664.

2.6. The conventional biodiesel separation method

The purpose of comparing the physicochemical properties of this biodiesel layer, obtained through the gravitational settling method, with the biodiesel obtained using the centrifuge method was to evaluate the effectiveness of the centrifuge-based separation in achieving improved quality and purity of the biodiesel product.

The crude biodiesel-glycerol mixture, produced under the same conditions described earlier using the recently developed centrifuge machine, underwent a subsequent separation step. In this step, gravitational settling was employed to separate the glycerol from the biodiesel layer. After the transesterification process, the mixture was transferred into a separatory funnel. The mixture was allowed to settle for 24 h [29]. The remaining biodiesel layer was used to measure the physicochemical properties compared to crude biodiesel obtained by separation with the centrifuge method.

3. Results and discussion

3.1. Optimization of operation parameters of centrifuge machine by RSM

This research employed mathematical models based on linear, two-factor interaction, and quadratic terms to fit the experiment response. Considering the F value, P-value, and R^2 shown in Table 3, the quadratic model was chosen as the best.

The quadratic polynomial model in terms of coded factors for the factors affecting the efficiency of the centrifuge for separating glycerol from biodiesel is given in Eq. (3).

$$\widehat{Y} = 90.35\text{-}0.3277X_1 - 0.3280X_2 - 0.9496X_3 + 0.3150X_1X_2 + 0.4550X_1X_3 - 0.4850X_2X_3 - 0.2524X_1^2 - 0.5088X_2^2 + 0.1771X_3^2 \quad (3)$$

The quality and fitness of the model were evaluated using an analysis of variance (ANOVA) test, employing the least squares method. The results of the ANOVA test are presented in Table 4. It determined the significant fitness and the effect of individual terms of the model. Moreover, it determined their interaction with the separation efficiency of the centrifuge. The lack of fit test showed a P value of 0.5001, much higher than 0.05. Therefore, the lack of fit is not insignificant at a 95% confidence level, and this model is developed very well. The quadratic model in Eq. (3) with an F-value of 145.11 and a P-value of <0.0001 indicates that it is significant

Table 3
Lack of fit test for evaluating the best fit with experimental data.

Source	Sum of Square	DF	Mean Square	F-value	P-value	R^2
Linear	9.64	11	0.8762	46.15	0.0003	0.6105
Linear + interaction	5.31	8	0.6632	34.93	0.0006	0.7838
Quadratic	0.0948	5	0.0190	0.9998	0.5001	0.9928

Table 4
Analysis of variance (ANOVA) for response surface quadratic model.

Source	Sum of Square	DF	Mean Square	F-value	P-value	Degree of significance
Model	24.79	9	2.75	145.11	<0.0001	significant
Speed, X_1	1.47	1	1.47	77.23	<0.0001	significant
Flow rate, X_2	1.47	1	1.47	77.41	<0.0001	significant
Temperature, X_3	12.32	1	12.32	648.71	<0.0001	significant
X_1X_2	0.7938	1	0.7938	41.81	<0.0001	significant
X_1X_3	1.66	1	1.66	87.24	<0.0001	significant
X_2X_3	1.88	1	1.88	99.12	<0.0001	significant
X_1^2	0.9184	1	0.9184	48.38	<0.0001	significant
X_2^2	3.73	1	3.73	196.49	<0.0001	significant
X_3^2	0.4521	1	0.4521	23.81	0.0006	significant
Residual	0.1899	10	0.0190			significant
Lack of Fit	0.0949	5	0.0190	0.9998	0.5001	not significant
Pure Error	0.0949	5	0.0190			
Total	24.98	19				

at a 95% confidence level. The coefficient of variation (CV) is 0.1532, which is less than 10, demonstrating a high degree of precision and a good deal of reliability with the experimental values, providing the coefficient of determination (R^2) of 0.9924. This implies that Eq. (3) could account for 99.24% of the variation in the efficiency for separating glycerol from biodiesel. Adjusted R^2 and predicted R^2 were also considered in this research, showing 0.9856 and 0.9649, respectively. The high value of both R^2 and adjusted R^2 justifies an excellent correlation between the independent variables and supports the high significance of the model.

The actual separation efficiency compared to the predicted separation efficiency was demonstrated in Fig. 2, in which an acceptable correlation was obtained between predicted and actual data on the efficiency of the centrifuge for separating glycerol from biodiesel. In addition, the line of (y = x) remarked an excellent model fitting with experimental results. The distribution of residual values to compare the predicted (model) and the observed (experimental) ones was also examined in this work. The normal probability plot of the residuals illustrates the adequacy of the models. The residuals should fall close to the diagonal line, as shown in Fig. 3(a), which implies normal distribution. The outlier t-plot of the response of interest in Fig. 3(b) shows no data point outside the threshold boundary of ±4.15. All fitted models are consistent with the experimental data. The random scatter of the residuals in Fig. 3(c) indicates that the suggested models are appropriate for interpreting the process and applying them to experimental data. Cook's distance is used to find influential of a data point. Fig. 3(d) shows that no data point unduly influences the estimated regression coefficient or, in turn, the fitted values.

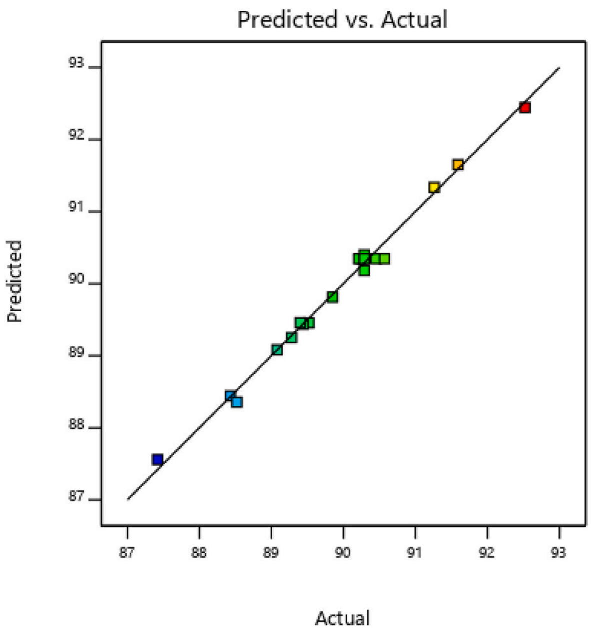


Fig. 2. Experimental versus predicted separation efficiency of centrifuge for separating glycerol from biodiesel.

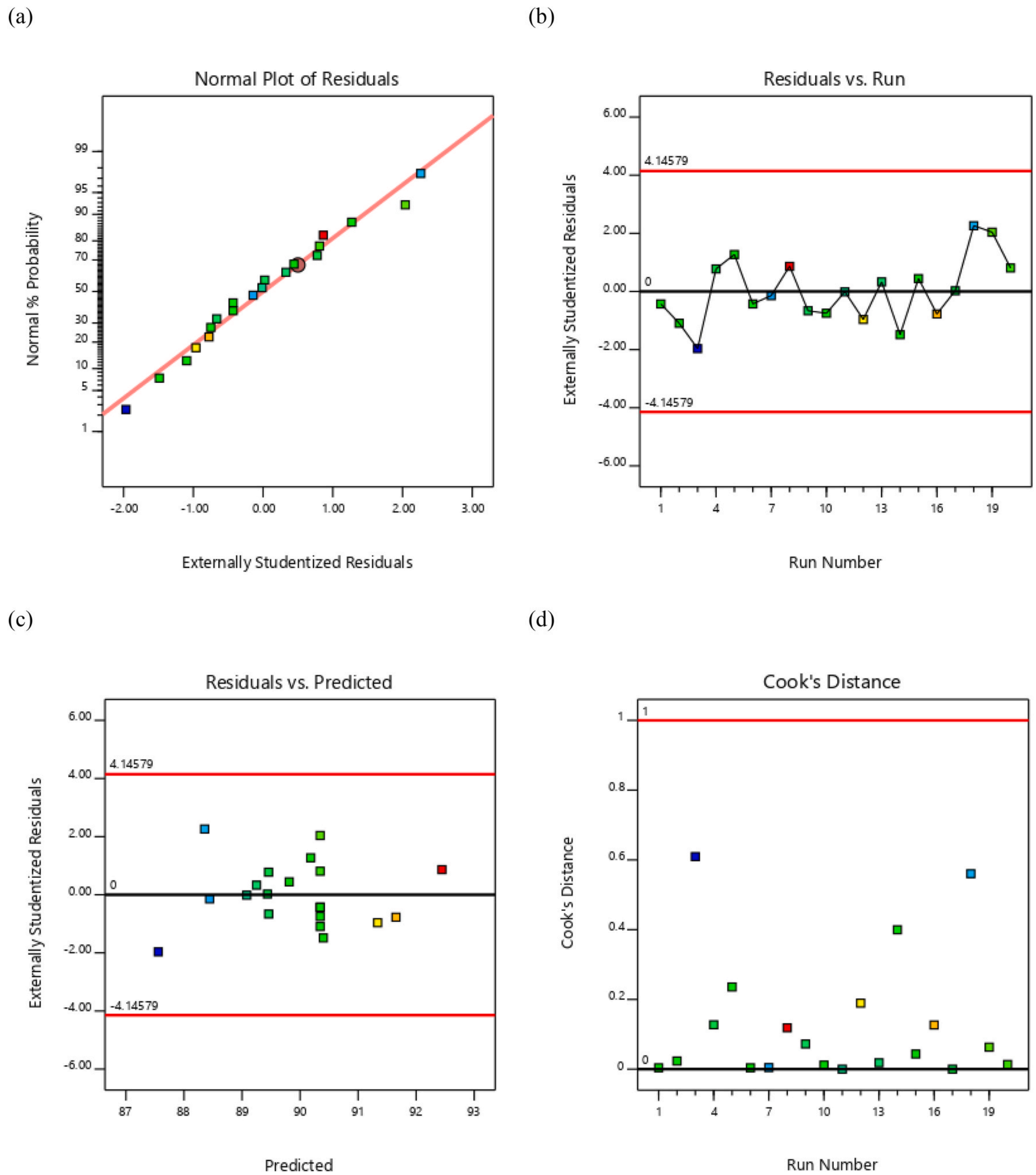


Fig. 3. Residual plots for separation efficiency of centrifuge for separating glycerol from biodiesel.

3.2. Individual effects of variables

Fig. 4 demonstrates the effect of process variables such as rotation speeds, mixture flow rates, and temperature on the separation efficiency of the centrifuge for separating glycerol from biodiesel. The individual parameter effect on the separation efficiency was determined by keeping other variables constant at hold value (0,0,0) in coded form. The variable with a steeper slope has a more significant influence on the separation efficiency of the centrifuge than the flatter slope.

With increasing the rotation speeds (X_1) from 1800 rpm to 2400 rpm, the separation efficiency of the centrifuge was decreased. The high rotation speed is directly proportional to an increasing centrifugal force of glycerol, which drives the glycerol out of the cylindrical bowl into the outer container; accordingly, this can decline separation efficiency. Additionally, the operating speed range from 2100 to 2400 rpm is excessively high, which has the emulsion on crude biodiesel-glycerol mixture [30]. The intense mechanical forces at such speeds can create a stable emulsion by shearing the biodiesel and glycerol into fine droplets that are uniformly dispersed and

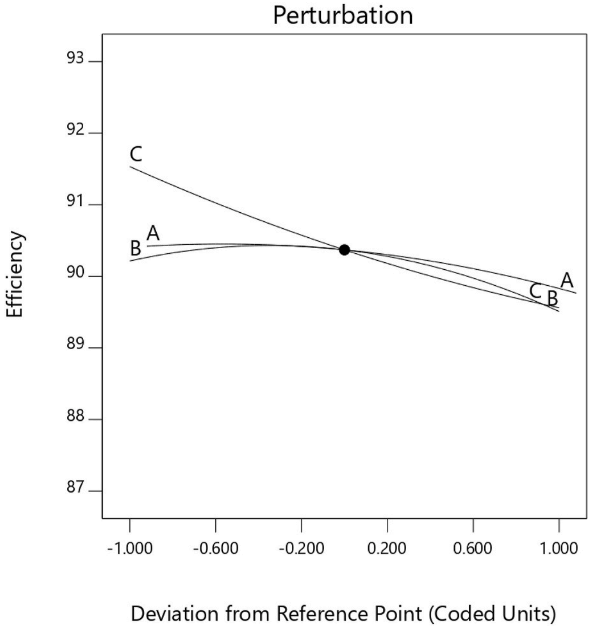


Fig. 4. Effect of process parameters on the separation efficiency of centrifuge for separating glycerol from biodiesel.

difficult to separate. Moreover, the heat generated at these high speeds can alter the physical properties of the mixture, further stabilizing the emulsion and preventing effective separation. As a result, it became difficult to separate layers between crude biodiesel and glycerol. Furthermore, the residence time of the mixture within the centrifuge may be reduced at higher rotation speeds. Insufficient residence time can limit the settling of the heavier glycerol phase, leading to incomplete separation. It is crucial to determine the suitable rotation speed to maximize the efficiency of the centrifugal force for separation and minimize the negative consequences of excessive speed, such as emulsion formation and reduced residence time. Therefore, the rotation speed impacts the centrifugal force experienced by the mixture, which in turn influences the settling and separation of the biodiesel and glycerol phases. Adjusting the rotation speed can help optimize the separation process and enhance the efficiency of the centrifuge.

In addition, the separation efficiency of the centrifuge was increased as the flow rate (X_2) increased from 150 ml/min to 200 ml/min. A rise in flow rates is a direct function of swirl velocity related to the centrifugal force increases [31]; thus, the crude biodiesel and glycerol were separated. However, a further increase in flow rates from 200 ml/min to 250 ml/min leads to decreased separation efficiency. The crude biodiesel cannot be separated before exiting into the outlet channel because of the short residence time in the cylindrical bowl and the high flow rate [32]. The shorter contact time between the mixture and the centrifuge components hinders the efficient separation of the two phases.

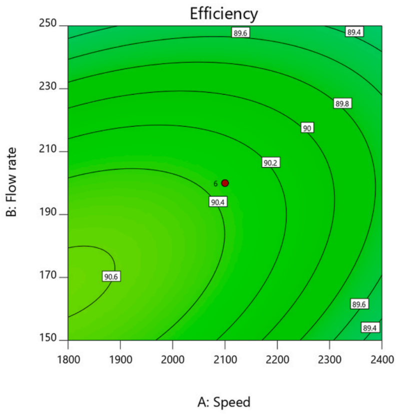
Furthermore, the separation efficiency of the centrifuge was found to decrease with increasing temperature (X_3). A further increase in the temperature from 62 °C to 68 °C leads to decreased efficiency caused by the vaporization of methanol in crude biodiesel at a temperature above its boiling point [33]. The analysis revealed that the rotation speeds (X_1), the mixture flow rates (X_2), and temperature (X_3) have a significant influence on the efficiency of the centrifuge for separating glycerol from biodiesel by their low P-values. By controlling these variables, it becomes possible to achieve higher separation efficiencies and improve the overall performance of the centrifuge system.

According to Eq. (3), the positive terms indicate that as the independent variable increases, the response variable also increases, whereas the negative terms suggest that the response variable tends to decrease as the independent variable increases. The finding indicated that the temperature (X_3) had the highest effect on the efficiency of the centrifuge for separating glycerol from biodiesel (effect of $X_3 = -0.9496$), followed by the mixture flow rates (X_2), and rotation speeds (X_1). The mixture flow rates (X_2), and rotation speed (X_1) equally affect the separation efficiency (effect of $X_2 = -0.3280$ and effect of $X_1 = -0.3277$). The rotation speeds (X_1), the mixture flow rates (X_2), and temperature (X_3) show a negative effect on the separation efficiency. Therefore, higher rotation speeds, flow rates, and temperatures influence separation efficiency, which is reduced the efficiency of the centrifuge for separating glycerol from biodiesel.

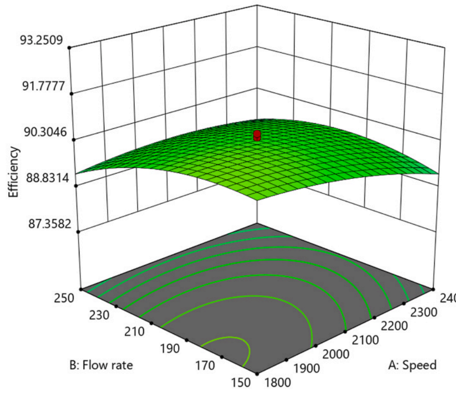
3.3. Interaction relationship between studied variables

The interactive effects of the process variables on the separation efficiency were investigated. Referring to Eq. (3), it was observed that the interaction rotation speeds (X_1) with mixture flow rates (X_2) and, as well as interaction rotation speeds (X_1) with temperature (X_3), and interaction mixture flow rates (X_2) with temperature (X_3) were all significant (effect $X_1X_2 = 0.3150$, effect $X_1X_3 = 0.4550$, and effect $X_2X_3 = -0.4850$).

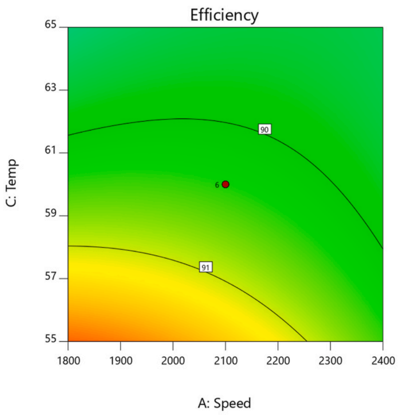
(a) Contour plot showing the interaction of rotation speeds and the mixture flow rates.



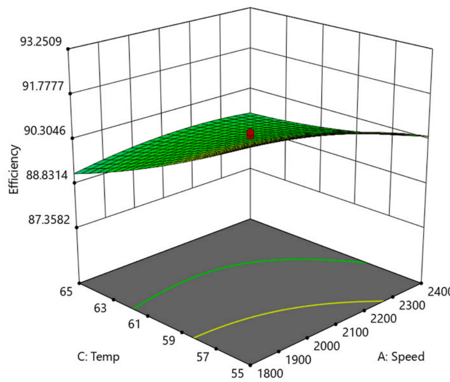
(b) Surface plot showing the interaction of rotation speeds and the mixture flow rates.



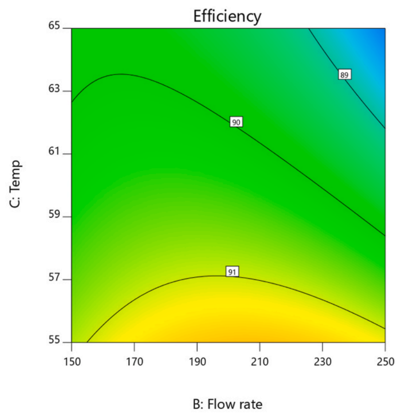
(c) Contour plot showing the interaction of rotation speeds and temperature.



(d) Surface plot showing the interaction of rotation speeds and temperature.



(e) Contour plot showing the interaction of the mixture flow rates and temperature.



(f) Surface plot showing the interaction of the mixture flow rates and temperature.

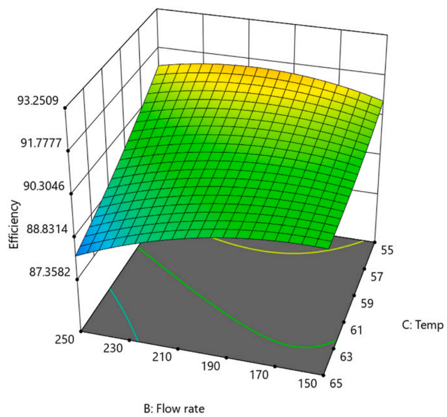


Fig. 5. Contour and surface plot of the combined effect of rotation speeds (X_1), the mixture flow rates (X_2), and temperature (X_3).

The contour plot and response surface plot, depicted in Fig. 5, illustrate the graphical representation of the interactive effects observed in the study. According to the three variables in the model, these plots were organized, each with two target variables, whereas the other held constant at zeros in their coded values. The interaction of rotation speeds (X_1) and mixture flow rates (X_2) on the separation efficiency of centrifuge for separating glycerol from biodiesel at a temperature of 60 °C are presented in Fig. 5(a) (contour plot) and Fig. 5(b) (3D surface plot). The result reveals that the separation efficiency of the centrifuge shows an upward trend with decreasing rotation speeds and mixture flow rates. When the mixture flow rates are lower, the centrifugal force is still reduced, although it is utilized more efficiently. This is because the lower flow rate decreases the chances of emulsification, which ultimately helps with the separation process. The relationship between higher flow rates and the driving force is complex. At first, increasing flow rates might increase the impact of the centrifugal force by providing more contact with the centrifugal field, which may improve separation efficiency. However, beyond a certain point, further increases in flow rate can lead to decreased efficiency due to reduced residence time and increased turbulence, which can disrupt the stratification necessary for effective separation. The separation efficiency is directly related to the centrifugal force generated in a centrifuge. Lower rotation speeds result in reduced centrifugal force, allowing for gentler separation. This downward force can help prevent emulsification and enhance glycerol separation from biodiesel. Moreover, a longer residence time in the centrifuge bowl provides more time for the separation process, allowing for improved separation efficiency. Additionally, the separation efficiency is enhanced by minimizing turbulence. These might be reduced turbulence that can hinder the separation process by disturbing the stratification of the biodiesel and glycerol phases. This suggests that the interaction between rotation speeds and mixture flow rates has significantly improved separation efficiency in the centrifuge process.

Fig. 5(c) and (d) are presented the contour and surface plot for the interaction effect between rotation speeds (X_1) and temperature (X_3) toward separation efficiency at a 200 ml/min flow rate. The result illustrated that the separation efficiency of the centrifuge decreases with increasing rotation speeds and temperature. The high F-values in the ANOVA results indicate a significant interaction effect between the rotation speeds and temperature (Table 4). Additionally, the high temperature may favor the triglycerides saponification in the sample, which creates an emulsion, causing an effect in the separation [34]. Nevertheless, the additional reason for lower separation efficiency is the volatilization of methanol. It might, therefore, be concluded that temperature is a significant factor in the separation process of biodiesel production. Moreover, higher temperatures can enhance the miscibility of biodiesel and glycerol, resulting in reduced phase separation. The increased thermal energy can promote molecular interactions and solubility between the two components, making it more difficult for the centrifuge to separate them effectively.

The simultaneous effect of mixture flow rates (X_2) and temperature (X_3) on the separation efficiency at a rotation speed of 2100 rpm are demonstrated in Fig. 5(e) (contour plot) and Fig. 5(f) (3D surface plot). The result indicated high separation efficiency at higher flow rates and lower temperatures. From the ANOVA results in Tables 4 and it was observed that there is a significant interaction effect between the flow rates and temperature. In addition, separation with a centrifuge at lower temperatures increased the viscosity. It lowered the solubility of the glycerol phase in the biodiesel phase, causing the distributed crude biodiesel-glycerol mixture for easier separation. This result is consistent with a previous study that used ceramic membranes to separate glycerol from biodiesel at low temperatures [35]. Besides, the biodiesel and glycerol phases exhibit a greater density difference at lower temperatures, which is beneficial for stratification and separation. As temperature decreases, the density of each phase increases differently, with glycerol density increasing more than biodiesel. This enhanced density difference at cooler temperatures aids in the gravitational settling of glycerol, facilitating clearer phase demarcation and more efficient separation. The combined effects of increased flow rates and decreased temperatures include enhanced driving force for separation, improved phase stratification due to the greater density difference, and reduced turbulence, which collectively lead to a significant improvement in the separation efficiency of biodiesel and glycerol.

3.4. Optimization and validation

The optimum conditions of three process parameters were predicted by solving the regression equation. The process variables were optimized to maximize the efficiency of the centrifuge for separating glycerol from biodiesel using the RSM within the variable range under study. The optimized parameters were obtained as follows: rotation speeds (X_1) = 1800 rpm, the mixture flow rates (X_2) = 192.25 ml/min, and temperature (X_3) = 55 °C. Based on these parameters, the model estimated the separation efficiency of the centrifuge at 92.02%. Confirmatory experiments were conducted to validate the model with the obtained optimum conditions. The average separation efficiency among the three parallel experiments was 94.52 ± 0.24%, which reasonably agrees with the predicted values (92.02%). This indicates that the established model is valid for separating glycerol from biodiesel via centrifuge in this study

Table 5
Comparison of biodiesel characteristics obtained by separation with centrifuge and other methods.

Properties (Unit)	Centrifuge machine	Gravitational settling	Ultrafiltration [7]	ASTM D6751-12	EN 14214:2012
Yield (%)	94.5	83.3	–	–	–
Kinematic viscosity (mm^2/s)	4.86	4.87	4.85	1.9-6	3.5–5.0
Density (g/cm^3)	0.85	0.88	0.87	–	0.86–0.90
Ester content (%mass)	98.14	97.68	97.5	–	>96.5
Free glycerol (%mass)	0.014	0.017	0.011	<0.02	<0.02
Triglyceride content	0.19	0.23	–	–	<0.20
Methanol content	0.12	1.96	–	<0.20	<0.20
Acid value (mg KOH/g)	0.128	0.157	1.26	<0.50	<0.50

and confirms that the RSM is an effective method for determining optimum conditions. The biodiesel yield and purity can be increased by optimizing the operational parameters using RSM, resulting in higher-quality biodiesel with improved market value and environmental benefits. These will contribute to the advancement of biodiesel production by enhancing the understanding of the centrifuge-based separation process.

3.5. Comparison of biodiesel characteristics with different separation methods

Both separated biodiesel obtained by the recently developed centrifuge machine at optimum conditions and gravitational settling were subjected to further purification. The purification process involved washing the separated biodiesel with distilled water to remove any remaining impurities. Subsequently, the physicochemical properties of biodiesel samples were characterized and compared to those obtained through centrifugation using a conventional centrifuge machine. Table 5 presents the results of the

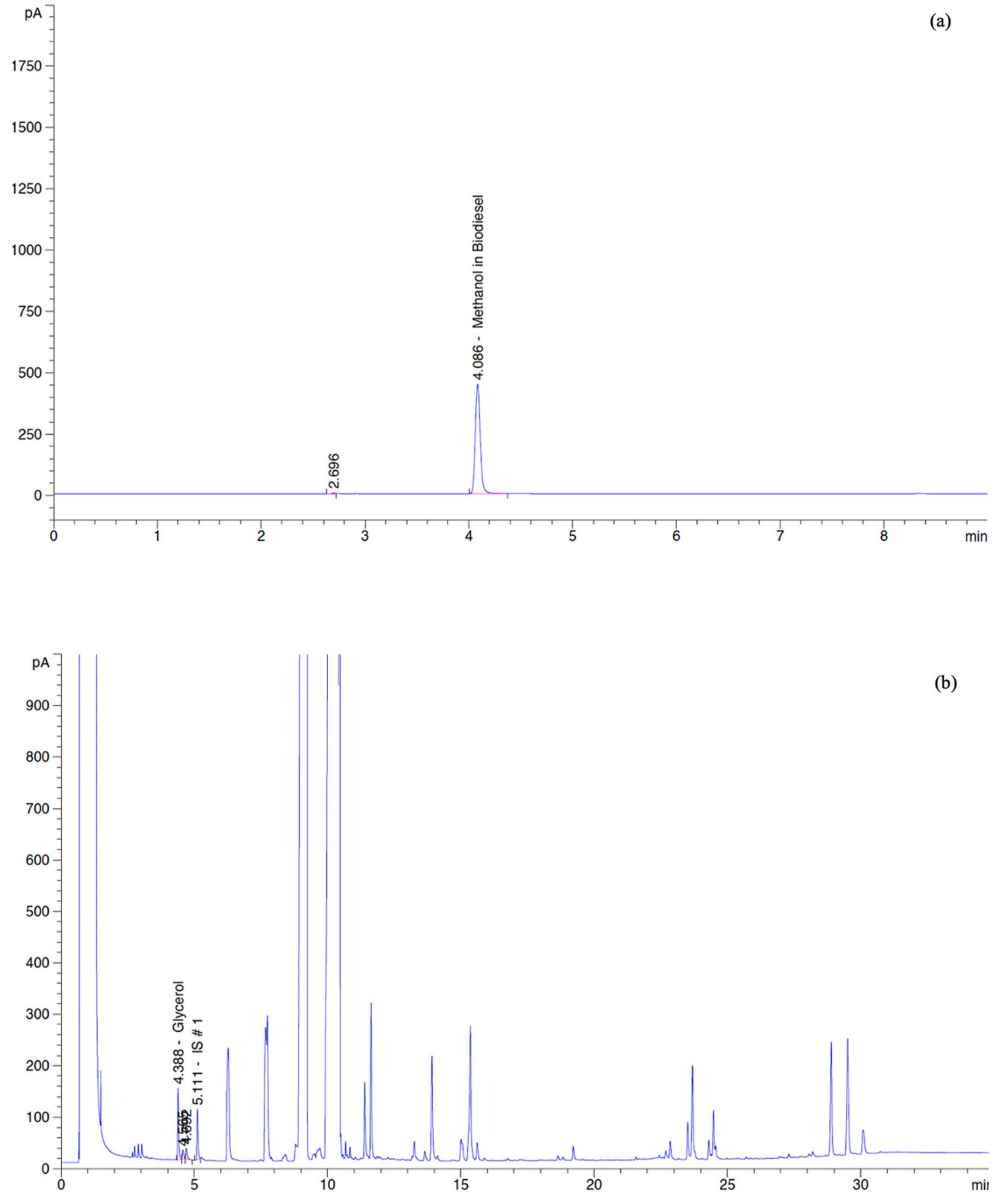


Fig. 6. GC chromatograms of biodiesel sample showing the peaks for (a) methanol and (b) glycerol.

physicochemical property analysis for both sets of biodiesel samples. Gas Chromatography (GC) analysis provided a more granular view of these properties, with the centrifuge-processed biodiesel showing a reduced methanol peak at 4.086 min and a lower free glycerol peak at 4.385 min (Fig. 6), indicative of a cleaner separation process compared to gravitational settling. The chromatograms also displayed a series of sharp peaks between 14 and 22 min, corresponding to the fatty acid methyl esters (FAMEs), suggesting a high purity of the biodiesel consistent with quality standards. The results demonstrate that the physicochemical properties of the biodiesel obtained through centrifugation using the recently developed machine closely align with the biodiesel standards.

The close agreement between the properties of the centrifuged biodiesel and the biodiesel standards suggests that the recently developed centrifuge machine effectively separates glycerol from biodiesel while maintaining the desired biodiesel quality. This indicates the successful optimization of the centrifuge system using response surface methodology and highlights its potential for enhancing biodiesel yield and purity. Compared to the research in the literature using the membrane technology [36], the ester content of each separation method was greater than the minimum required for marketing at 96.5%. Also, the free glycerol was below 0.02% in mass, which was further confirmed by the absence of significant peaks for glycerol in the GC analysis of the centrifuged samples. Additionally, the methanol contents of the sample obtained by the centrifuge machine are below those prescribed by the standard, as evidenced by the smaller methanol peaks in the GC chromatograms. The separated biodiesel with centrifuging had a lower methanol content than the settling method. The low content of methanol in separated biodiesel with centrifuging might suggest that this could be the advantage for the recovery of methanol in biodiesel production. Other physicochemical properties of biodiesel from centrifuging are also better than the settling method, such as lower acid value and kinematic viscosity.

Additionally, separating biodiesel using the settling method provides at least ten biodiesel volumes to come out with glycerol [37]. Considering that the separation of biodiesel using the settling method can be replaced by the recently developed centrifuge machine, the biodiesel using the centrifuge in this study has the benefit of a low amount of biodiesel coming out with glycerol after the separation process, achieving a high yield of biodiesel and providing a short time for the production of biodiesel. The GC data, with no significant peaks detected beyond the FAME region, support the assertion of improved separation efficiency by the centrifuge. Samples of separated biodiesel using a centrifuge machine and gravitational settling are shown in Fig. 7. As these samples pass through the centrifuge machine at an optimum condition, that removed substance provides biodiesel in a clear amber-yellow color [38]. The separated biodiesel using a centrifuge machine exhibited a brighter color than the separated biodiesel with gravitational settling. This might suggest that the separation of biodiesel from glycerol using a centrifuge machine was efficient. Moreover, the achievement of this study might be helpful to opportunities for process optimization in small and medium-sized enterprises (SMEs). The enhanced separation efficiency provided by the centrifuge machine also carries environmental benefits, such as minimizing waste and reducing the glycerol-contaminated biodiesel volume, thereby decreasing methanol content. This process follows severe environmental standards, potentially decreasing the requirement for chemical additions and improving the quality and quantity of biodiesel produced. promoting a more sustainable industry practice. Applying bio-adsorbents for post-separation purification presents a significant opportunity to further reduce environmental impacts. This promising area of research could improve product recovery and address environmental concerns. The findings of this study provide an important foundation for future research, particularly in bio-adsorbent utilization, indicating significant progress in biodiesel production technology.

4. Conclusion

The separation process is an essential step in biodiesel production. The recently developed centrifuge machine separated glycerol from biodiesel and improved biodiesel yield and purity. The CCD-based RSM method was employed to design the experiments and determine the optimal conditions of the developed machine for separating glycerol from biodiesel. The statistical test (ANOVA) illustrated a good agreement between experimental and predicted data with an R^2 of 0.9924. The optimized operating parameters, confirmed by an independent experiment, were achieved at rotation speeds of 1800 rpm, mixture flow rates of 192.25 ml/min, and operating temperature of 55 °C. The separation efficiency based on RSM under optimum conditions was 94.52%. Therefore, using a centrifuge machine under optimum conditions, the separation process led to a high biodiesel yield and complied with the ranges specified for biodiesel. This study showed that using the centrifuge machine, the CCD-based RSM is a valuable tool for optimizing the operating parameters for separating glycerol from biodiesel. Importantly, a recently developed centrifuge machine might be used to separate glycerol from biodiesel instead of the gravitational settling method for SMEs because of its easy-to-use, low operating time, compact structure, and high efficiency for the separation process in biodiesel production. These results might contribute to the advancement of sustainable energy solutions and the wider adoption of biodiesel as an alternative fuel source.

Data availability

The authors declare that the data supporting the findings of this study are available within the article. The raw/derived data supporting the findings of this study are available from the corresponding author at request.

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(a) Biodiesel obtained by centrifuging (b) Biodiesel obtained by setting gravity

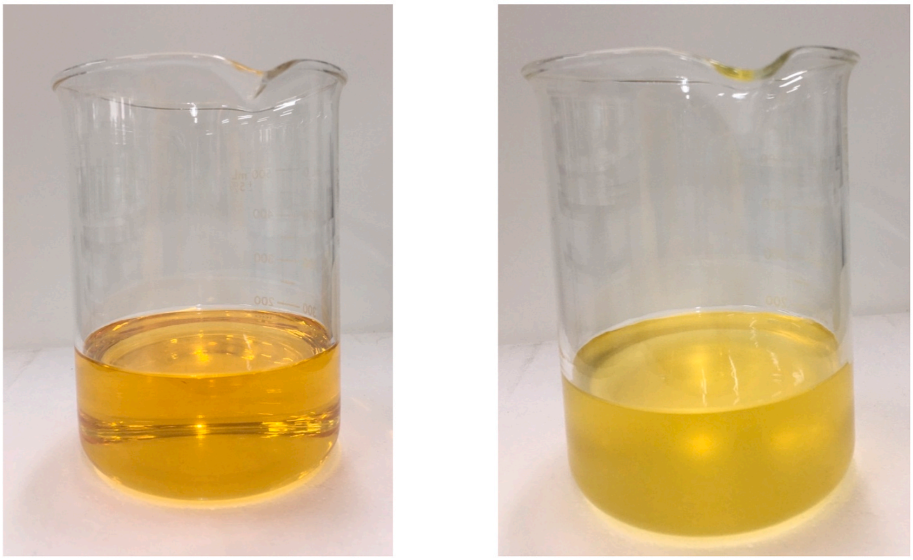

Fig. 7. Sample of biodiesel obtained by different separation methods.

CRediT authorship contribution statement

Warunee Limmun: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation. **Thatchapol Chungcharoen:** Writing – review & editing, Methodology, Funding acquisition, Conceptualization. **Chaiwat Rattanamechaiskul:** Visualization. **Kittisak Phetpan:** Writing – review & editing, Visualization. **Wanida Limmun:** Writing – review & editing, Validation, Formal analysis, Data curation.

Declaration of competing 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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