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Enhancing Biodiesel Yield from Castor Seed Oil through Co-Solvent-Assisted Transesterification Using n-Hexane

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ABSTRACT: The growing demand for sustainable energy sources has increased interest in biodiesel as a renewable alternative to petroleum-based fuels. This study explored the effect of reaction time, molar ratio of methanol to oil, and catalyst concentration with the addition of co-solvent on the transesterification of castor seed oil, aiming to increase methyl ester yield. n-Hexane was chosen as the co-solvent due to its physical compatibility with methanol, thus facilitating reactant solubility and product separation. Using response surface methodology (RSM) with Minitab software, process parameters such as co-solvent ratio, reaction duration, and temperature were optimized. The optimized conditions (1:5 co-solvent ratio, 55°C, 20 min) resulted in a biodiesel yield of 98.86%, with GC-MS confirming a methyl ester content of 99.79%. The resulting biodiesel yield met the requirements of the Indonesian National Standard (SNI) as well as viscosity but had a heating value that was slightly below the standard range. These findings highlight the effectiveness of co-solvent integration in improving biodiesel production from vegetable oil feedstocks.

Keywords: biodiesel; Castor bean oil; co solvent; methyl ester; Response Surface Methodology (RSM)

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1. Introduction

The continuous rise in global population is directly correlated with increasing energy consumption, particularly in developing countries such as Indonesia. According to the Central Bureau of Statistics (2023), Indonesia's population is projected to exceed 330 million by 2050, which will significantly impact national energy demand. Concurrently, domestic fossil fuel reserves are depleting rapidly—from 7.5 billion barrels in 2019 to an estimated 1.137 billion barrels by 2024 (DEN, 2023; ESDM, 2024). This imbalance between growing demand and limited fossil fuel reserves calls for the urgent development of renewable, sustainable, and environmentally friendly energy alternatives. Among various options, biodiesel has emerged as a promising candidate due to its renewable origin, non-toxicity, biodegradability, and ability to reduce SOx, NOx, and CO_2 emissions (Mahmudah, 2023).

Biodiesel can be produced from a wide range of feedstocks including vegetable oils, animal fats, and waste oils. Castor bean oil, in particular, offers several advantages as a non-edible, high-yielding feedstock. Its high ricinoleic acid content and the ability of the castor plant to grow on marginal lands make it a valuable resource for sustainable biofuel production (Riani, 2018). Previous research has also highlighted the potential of Jatropha curcas, another non-edible oil source, but its high free fatty acid (FFA) content often necessitates a pre-treatment esterification step prior to transesterification (Maulida Astmaiya & Jalaluddin, 2023). To improve conversion efficiency, various enhancements have been proposed, one of ways is the addition of co-solvents to improve phase miscibility during transesterification.

Recent studies have demonstrated the benefits of co-solvent-assisted transesterification. Alhassan et al. (2014) reported that co-solvents such as diethyl ether and acetone improved biodiesel yield by enhancing reactant solubility and reducing reaction time. Similarly, Dianursanti et al. (2015) found that n-hexane significantly increased FAME conversion from microalgae when used in a 1:1 ratio with methanol. n-Hexane is particularly attractive due to its low boiling point (68°C), non-reactivity, and ease of separation from methanol, making it both efficient and economically viable (Daryono, 2020). Despite these promising outcomes, limited studies have examined the application of n-hexane as a co-solvent in castor oil-based biodiesel production, especially under systematically varied conditions using response surface methodology (RSM).

A review of the utility of castor oil as a high-potential feedstock for biodiesel is mentioned in the study of Nayak and Sahoo (2023) who investigated the physicochemical properties of castor oil, its cultivation benefits, and its compatibility with various transesterification strategies. Their analysis illustrated the suitability of castor oil for dry or marginal lands and its high ricinoleic acid content, which contributes to desirable fuel characteristics when properly processed. This supports the current research focus on castor oil as a sustainable and underutilized feedstock for biodiesel synthesis.

Therefore, this study aims to investigate the effect of n-hexane co-solvent addition on biodiesel production from castor bean oil. Using RSM, this research evaluates how variations in co-solvent ratio, reaction temperature, and time influence methyl ester yield. The novelty of this study lies in its integration of co-solvent-assisted transesterification with statistical optimization to enhance biodiesel yield from a non-edible and underutilized oil source. The findings are expected to support more efficient biodiesel production strategies, particularly in regions with abundant castor seed cultivation.

Biodiesel research also support the strategic use of non-edible oils and environmentally friendly solvents to improve yield, efficiency, and sustainability. According to research, Verma et al. (2021) showed that deep eutectic solvent (DES) or hijang solvent, which contains a choline chloride-glycerol mixture, significantly increased biodiesel yield from used cooking oil. Under optimized conditions of methanol to oil molar ratio of 12:1, 65°C, and reaction time of 2 hours a biodiesel yield of 96.8% was achieved, highlighting the green potential of DES in transesterification. Similarly, in the study, Kumar et al. (2022) investigated heterogeneous catalysts derived from used egg shells and banana peel ash for non-edible oils such as jatropha and karanja. The optimal conditions involved a 9:1 methanol-oil ratio, 3 wt% catalyst loading, 60°C, and 90 min reaction time, which resulted in biodiesel yields above 93%. As for biodiesel research using co-solvents, Pathak and Singh (2023) compared several transesterification strategies and emphasized the efficiency of co-solvent-assisted processes, specifically using n-hexane and diethyl ether. In his study, using a methanol-oil molar ratio of 1:4 with 10% v/v n-hexane at 55°C for 30 minutes can increase the biodiesel yield to 95.2%, especially when applied to non-edible oils with high viscosity. The above study underscores the importance of integrating co-solvent or green solvent approaches with statistical optimization tools such as Response Surface Methodology (RSM) to develop biodiesel production methods that are not only high-yielding but also scalable and environmentally sustainable

2. Materials and Methods

The materials used in this study include castor seed oil sourced from Bandung, West Java, Indonesia. The alcohol used was methanol (Merck, ≥99.9% purity). The co-solvent employed was n-hexane (Merck, analytical grade/PA). Two types of catalysts were used: sulfuric acid (H_2SO_4, SmartLab, 96%) for the esterification stage, and sodium hydroxide (NaOH, Merck) for the transesterification stage.

2.1. Esterification

Esterification was carried out to reduce the free fatty acid (FFA) content of the raw castor seed oil, which initially exceeded 2%, making it unsuitable for direct transesterification (Megawati et al., 2022). The reaction involved mixing castor seed oil with methanol at a molar ratio of 1:7, using 30 grams of castor seed oil, H_2SO_4 96% as an acid catalyst at 0.5% w/w oil. The reaction was carried out at a constant temperature of 35°C for 90 minutes.

2.2. Transesterification

Transesterification refers to the process in which triglycerides react with alcohol to form esters and glycerol, thereby reducing the viscosity of vegetable oil to levels compatible with diesel engines (Al Ghifari & Samik, 2023). In this study, transesterification was carried out in two variations: without and with co-solvent.

2.2.1 Transesterification without co-solvent

The esterified castor oil was reacted with methanol at a molar ratio of 1:7, using NaOH as a base catalyst at 0.5% w/w oil. This transesterfication was carried out by dissolving NaOH with methanol mixed into the esterifirase product that had been heated to 35°C. The reaction was carried out at 35°C for 30 minutes with constant stirring. Upon completion, the mixture was allowed to settle, forming two distinct layers: an upper layer (containing methyl esters and residual methanol) and a lower layer (glycerol). The top layer is removed and distilled to remove excess methanol, resulting in the final biodiesel product.

2.2.2 Transesterification with co-solvent

In this modified method, n-hexane was used as a co-solvent to improve the solubility of methanol and triglycerides, thereby enhancing mass transfer and reaction efficiency (Mahlinda & Djafar, 2016). The transesterification was conducted using the same procedure as the conventional method, with the addition of n-hexane at varied weight ratios to methanol (1:1, 1:3, and 1:5 w/w). Reactions were conducted at three temperature levels (35 °C, 45 °C, and 55 °C) and three durations (10, 20, and 30 minutes).

Post-reaction, the mixture was allowed to separate into two layers. The upper layer was distilled to eliminate residual methanol, and the resulting biodiesel was collected and quantified based on volume and mass.

The biodiesel yield was calculated based on the mass of biodiesel product relative to the initial mass of oil used in the reaction. Specifically, the yield was determined using the following equation:

$$Yield\ (\%) = \frac{Mass\ of\ Biodiesel}{Mass\ of\ Oil} \times 100\% \qquad (1)$$

3. Results and Discussion

3.1. Response Surface Methodology (RSM) Analysis

The experimental data were analyzed using response surface methodology (RSM) to evaluate the effects of reaction time, temperature, and co-solvent ratio on biodiesel yield. RSM analysis facilitated the identification of the most influential variables and provided predictive insight into optimizing transesterification efficiency.

Response surface methodology (RSM) with solvent-assisted transesterification to enhance biodiesel production was mentioned in the study of Faisal et al. (2021) who optimized the transesterification of palm oil using n-hexane as a solvent and achieved a biodiesel yield exceeding 96%

under moderate conditions (60°C, 40 min, 1% NaOH). Similarly, Atabani et al. (2020) applied RSM to the transesterification of waste oil and showed that the use of a methanol–n-hexane mixture significantly increased the conversion efficiency, achieving a yield of 98%. These findings strengthen the approach of the current study, where RSM was used to systematically evaluate the interactive effects of solvent ratio, temperature, and time on biodiesel yield from castor oil.

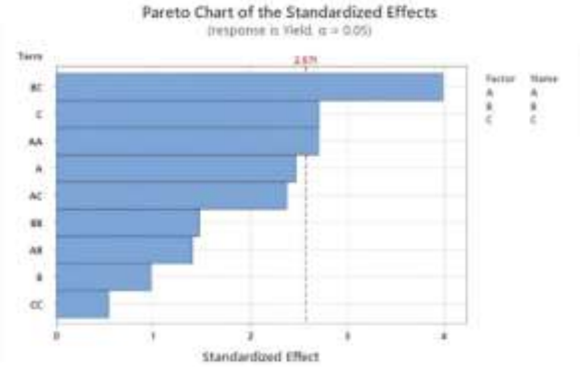

Figure 1. Pareto Chart of Variable Effects on Biodiesel Production

As shown in the Pareto chart (Figure 1), the co-solvent ratio and reaction time emerged as the dominant factors influencing methyl ester yield. In particular, the co-solvent variable exhibited a pronounced effect, underscoring its key role in enhancing the miscibility between methanol and oil phases. These observations are in strong agreement with previous findings by Alhassan et al. (2014), who demonstrated that co-solvent addition improved mass transfer and reaction kinetics during transesterification of cottonseed oil, and Dianursanti et al. (2015), who reported similar benefits using n-hexane in microalgae-based biodiesel systems.

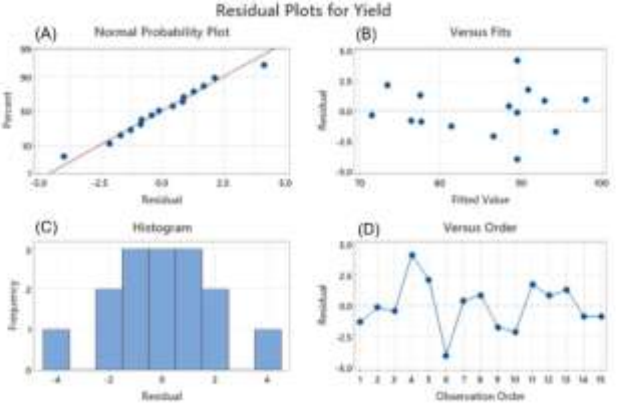

Figure 2. Residual plots on biodiesel production: (A) Normal probability plot, (B) Residuals versus fitted values, (C) Histogram of residuals, and (D) Residuals versus observation order

Figure 2 shows residual plots used to validate the statistical assumptions of the regression model. The normal probability plot reveals that residuals followed a near-normal distribution. The residuals vs. fitted plot and histogram support homoscedasticity and symmetry, while the versus order plot indicates independence. The R^2 value of 94.53% confirms strong model accuracy. Table 1 further presents p-values, with variable C (co-solvent ratio) showing significant influence ($p = 0.043$), supporting its critical role.

Table 1. Coded Coefficients RSM Analysis

Term	p-value
Constant	0.809
A	0.056
B	0.373
C	0.043
A*A	0.043
B*B	0.200
C*C	0.607
A*B	0.220
A*C	0.063
B*C	0.010

3.2. Effect of temperature on biodiesel yield

Figure 3 shows that increasing the reaction temperature from 35°C to 55°C led to a progressive increase in biodiesel yield, with the maximum yield observed at 55°C. This result highlights the importance of sufficient thermal energy in enhancing the reaction rate and promoting better miscibility between oil, methanol, and co-solvent. However, when the temperature exceeded 55°C, a decline in yield was observed. This can be attributed to the evaporation of methanol and n-hexane whose boiling points are 64.7°C and 68.7°C respectively resulting in a reduction of available reactants and disruption of the transesterification equilibrium. These findings are in agreement with Daryono et al. (2022), who observed a similar decline in yield when the reaction exceeded the boiling point of the solvents.

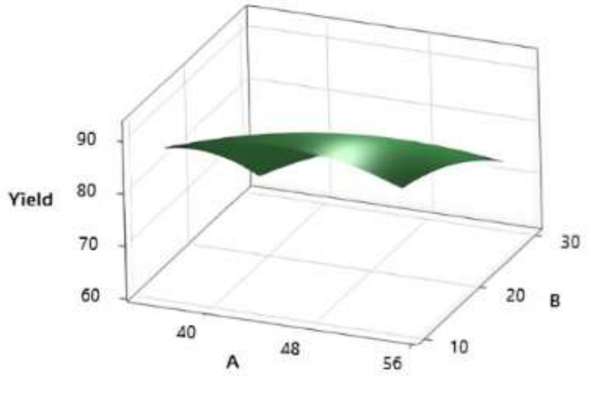

Figure 3. Surface plot of temperature (A, °C) and time (B, min) on biodiesel yield (%)

3.3. Effect of time on biodiesel yield

As shown in Figure 4, Biodiesel yield increased with reaction time, with the highest yield achieved at 20 minutes. This trend indicates that the transesterification reaction approaches equilibrium around this time, allowing maximum conversion of triglycerides into methyl esters. Extending the reaction time beyond 20 minutes, however, resulted in a noticeable decrease in yield. In addition to the possible hydrolysis of methyl esters back into free fatty scid at longer reaction times, this backward reaction may be promoted by the presence of residual water in the system arising from insufficient drying of oil or non-anhydrous methanol as well as by excessive catalyst concentration, which may trigger side reactions such as saponification that reduce biodiesel yield. The observation aligns with findings by Daryono et al. (2022), who reported a similar decrease in yield after the optimal time was surpassed, emphasizing the importance of controlling reaction duration to avoid backward reactions and product degradation.

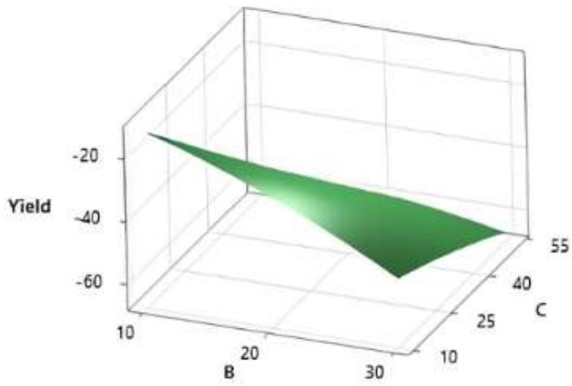

Figure 4. Surface plot of time (B, min) and temperature (C, °C) on biodiesel yield (%)

3.4. Effect of Methanol-to-Co-Solvent Ratio on Biodiesel Yield

Figure 5 illustrates the interaction between temperature and co-solvent weight on biodiesel yield. The results demonstrate a strong positive correlation: as the weight of n-hexane co-solvent increased, biodiesel yield also improved. The optimal yield was observed when the co-solvent weight reached 52.5 grams, under a reaction temperature of 55°C. This indicates that the co-solvent plays a crucial role in enhancing the efficiency of the transesterification process, particularly at elevated temperatures.DES

The increase in biodiesel yield with higher co-solvent concentration can be attributed to improved solubility and miscibility between methanol and triglycerides. N-Hexane improved the miscibility between methanol and triglycerides and accelerates mass transfer, allowing for more efficient contact between reactants and more complete conversion to methyl esters. These findings are consistent with the study by Muyassaroh et al. (2012), which reported that conventional transesterification without co-solvent resulted in a methyl ester yield of 55.11% after two minutes, whereas the addition of a co-solvent at a 2:1 ratio with methanol increased the yield significantly to 94.79%.

This study confirms that the strategic use of co-solvent not only enhances reaction kinetics but also significantly improves overall biodiesel productivity. The positive trend observed in the surface plot reinforces the importance of optimizing co-solvent ratios as a key parameter in biodiesel synthesis.

Table 2. Comparison of biodiesel yield, feedstock, reaction conditions, and co-solvent use between the present study and selected previous works.

Study	Feedstock	Co-Solvent (Type, Ratio)	Reaction Conditions	Yield (%)
This study	Castor oil	n-Hexane (1:5 w/w methanol)	35,45, and 55°C, 10,20, and 30 min,	98.86
Muyassaroh et al., 2012	Castor oil	TetraHydrofuran (0:1, 1:1, and 2:1 w/w methanol)	55°C, 2,4,6,8, and 10 min	94.79
Daryono et al., 2022	Palm oil	None	60 min, base catalyst	90.60
Faisal et al., 2021	Palm oil	n-Hexane	60°C, 40 min	>96.00
Pathak & Singh, 2023	Various non-edible oils	n-Hexane (10% v/v)	55°C, 30 min	95.20

The effectiveness of this method can be further appreciated when compared to previous studies. Muyassaroh et al. (2012) reported a biodiesel yield of 94.79% using castor oil with a 2:1 methanol-to-co-solvent ratio under conventional conditions.

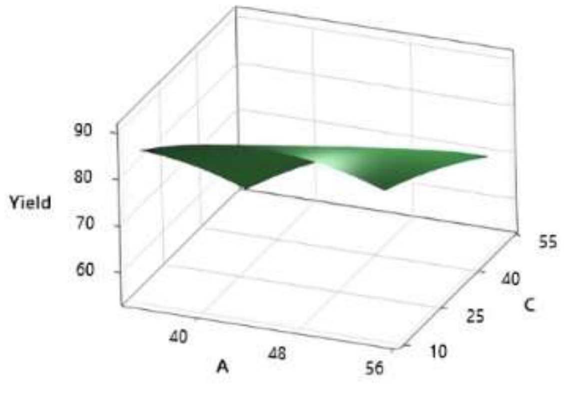

Figure 5. Surface plot of temperature (A, °C) and time (C, min) on biodiesel yield (%)

In contrast, Daryono et al. (2022) obtained a lower yield of 86.3% from palm oil without any co-solvent, even with a longer reaction time of 60 minutes. In the present study, a higher yield of 98.86% was achieved using castor oil and a 1:5 methanol-to-n-hexane ratio at only 20 minutes and 55°C. This result highlights the significance of optimizing both co-solvent ratio and reaction parameters to improve biodiesel production efficiency

3.5. Characterization of Biodiesel

Under optimal conditions (55°C, 20 minutes, 1:5 methanol-to-co-solvent ratio), biodiesel yield reached 98.86%. GC-MS analysis (Figure 6) revealed methyl ricinoleate as the dominant compound (84.76%), followed by 6-octadecenoic acid methyl ester (11.44%), with overall methyl ester content reaching 99.79% (Figure 7).

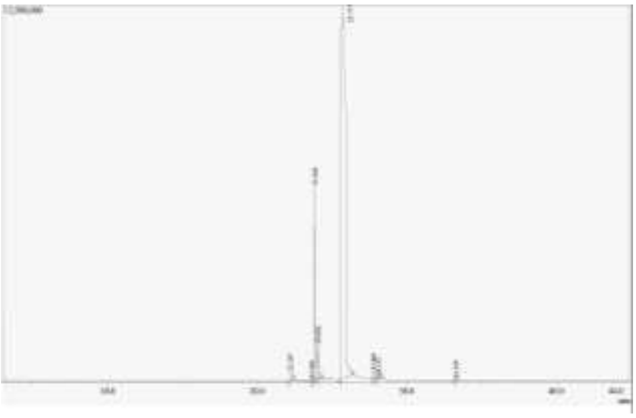

Figure 6. GC-MS Chromatogram of Biodiesel using co-solvent

Table 2 shows the GC-MS chromatogram of the biodiesel sample transesterified from castor oil. The analysis showed that the main peak was detected at a retention time of 25.717 minutes, which was identified as methyl ester of ricinoleic acid, the main compound in castor oil. This peak has an area proportion of 84.76%, indicating that the majority of the components in biodiesel are methyl esters of ricinoleic acid. In addition, there are several minor peaks, such as at a retention time of 23.894 minutes which was identified as 6-octadecenoic acid, methyl ester (an isomer of methyl oleate) with a percentage of 11.44%, and at 24.096 minutes octadecanoic acid, methyl ester (methyl stearate) was detected with an area of 1.91%. These spectra indicate that the biodiesel produced is dominated by methyl ester compounds of long-chain fatty acids, which is the result of successful transesterification.

Table 3 presents the calorific values, showing that biodiesel with co-solvent had higher energy content (8762.47 cal/g) than biodiesel without (7131.77 cal/g), though both remain below the SNI standard of 10160–11000 cal/g. According to EN 14214, the minimum acceptable calorific value is around 35 MJ/kg (approximately 8370 cal/g), which means that the biodiesel produced with co-solvent meets the EN standard but does not fulfill the stricter requirement set by SNI. The ASTM D6751 standard does not explicitly define calorific value limits but ensures quality through performance-based tests.

Table 2. Content produced by biodiesel using co-solvent

Peak	Name	R. (min)	Area (%)	Height (%)	A/H
1	Hexadecanoic acid, methyl ester (CAS)	22.247	0.78	1.48	6.60
2	1 3-Hexyloxacyclotridec-10-en-2-one	23.698	0.10	0.37	3.28
3	6-Octadecenoic acid,methyl ester , (Z)- (CAS)	23.894	11.44	31.10	4.61
4	Octadenoic acid, methyl ester (CAS)	24.096	1.91	5.66	4.23
5	Methyl ester of Ricinoleic Acid	25.717	84.76	59.44	17.86
6	9-Octadenoic acid, methyl ester, (E)- (CAS)	27.801	0.61	1.32	5.84
7	Octadecanoic acid, 9,10-dihydroxy- ,methyl ester (CAS)	28.143	0.27	0.38	9.05
8	2,6,10,14,18,22-Tetracosahexaene,2,6,10,15,19,23-hexamethyl (CAS)	33.329	0.11	0.25	5.63
	Sum		100	100	

In terms of biodiesel yield, the transesterification process using co-solvent achieved a methyl ester content of 99.79%, surpassing the minimum requirement of ≥96.5% as set by both SNI 7182:2015 and EN 14214. Although ASTM D6751 does not directly specify a required percentage for methyl ester content, such a high yield aligns well with ASTM quality expectations, as it indicates efficient conversion and minimal impurities. By contrast, biodiesel without co-solvent generally showed lower yield and quality.

Additionally, viscosity results indicated that biodiesel using co-solvent had improved flow properties. With a measured viscosity of 2.4193 cSt, it met the SNI range of 2.3–6.0 cSt and also satisfied the ASTM D6751 range of 1.9–6.0 cSt. However, it fell slightly below the EN 14214 requirement, which specifies a narrower range of 3.5–5.0 cSt. Meanwhile, biodiesel without co-solvent had a lower viscosity of 1.887 cSt, falling below the acceptable ranges of all three standards. The raw castor oil exhibited a very high viscosity of 131.85 cSt. These results are consistent with Handayani & Susilo (2014), who reported that co-solvent use significantly reduces biodiesel viscosity and improves fuel quality.

Table 3. The Calorific Value Analysis and FAME yield of Biodiesel

Samples	Caloric value (cal/g)	FAME yield (%)	SNI Caloric ≥10,160	ASTM standard

			& FAME ≥96.5%)	FAME ≥96.5%)
Biodiesel using co-solvent	8762.47	99.79	Not compliant (caloric <10,160)	Satisfies the FAME requirement (≥96.5%)
biodiesel without co-solvent	7131.76	66.67	Not compliant (both below standard)	Not Satisfies FAME (≥96.5%)

4. Conclusions

This study confirms that the strategic addition of n-hexane as a co-solvent significantly improves the transesterification process in biodiesel production from castor seed oil. By enhancing the solubility between methanol and triglycerides, the co-solvent approach allows for more efficient reaction kinetics and higher product quality. This aligns with the study's objective to develop a more effective method for increasing biodiesel yield and purity using a non-edible, renewable feedstock.

Compared to conventional processes, this method operates under relatively mild conditions while achieving superior performance, including improved calorific value and fuel viscosity. These advantages make the approach not only technically feasible but also potentially scalable for decentralized or resource-limited biodiesel production systems.

This work contributes to the growing body of knowledge by demonstrating how process intensification through co-solvent integration can optimize biodiesel quality. Future research should focus on evaluating the environmental and economic implications of upscaling this method, exploring the use of alternative green co-solvents or catalysts, and adapting the approach for continuous-flow reactor systems to enhance industrial applicability.

Statement

During the preparation of this work the authors used ChatGPT 4o in order to improve English language and proofread the text. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

CRediT authorship contribution statement

Annisa Yasyifa: Investigation, Data curation, Resources, Writing – original draft.
Ro Atin: Formal analysis, Visualization, Validation, Writing – review & editing.
Alit Istiani: Writing – review & editing, Revision support
Heni Anggorowati: Conceptualization, Methodology, Supervision, Writing – review & editing.

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.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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