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Research Article

Integrated upgrading of waste cooking oil–derived biodiesel via reaction pathway selection, adsorption, and feedstock blending

Misri Gozan[a,b,c], Mochamad Yusuf Efendi[b], Andre Fahriz Perdana Harahap[b,c], Anusith Thanapimmetha[a], Maythee Saisriyoot[a], Pattarawit Krajomethong[a], Penjit Srinophakun[a]*

[a]*Department of Chemical Engineering, Faculty of Engineering, Kasetsart University, 50 NgamWongWan Road, Jatujak, Bangkok, 10900, Thailand*

[b]*Research Centre for Biomass Valorization, Faculty of Engineering, Universitas Indonesia, Kampus Baru UI, Depok, 16424, Indonesia*

[c]*Department of Chemical Engineering, Faculty of Engineering, Universitas Indonesia, Kampus Baru UI, Depok, 16424, Indonesia*

Abstract. Biodiesel production from waste cooking oil (WCO) offers environmental and economic advantages but is constrained by degraded feedstock quality and unstable fuel properties. This study evaluates an integrated upgrading strategy combining reaction pathway selection, adsorption-based purification, and feedstock blending to produce fuel-grade biodiesel. WCO was pretreated by moisture removal and acid esterification using H_2SO_4 to reduce free fatty acids. Transesterification was conducted in a temperature-controlled batch reactor at 60 °C using 6:1–12:1 methanol-to-oil mass ratios, 1.0–2.0 wt% KOH catalyst loadings, and 60–120 minute reaction times. The maximum FAME content achieved was 93.94 wt% at 6:1 methanol ratio, 1.5 wt% KOH, and 90 min reaction time. Post-reaction upgrading was performed via batch adsorption using activated carbon (0.5–2 wt%, 60 °C) and silica gel (3 wt%, 112 °C). Adsorption improved FAME content to 94–95 wt% (maximum 95.3 wt% with silica gel) and reduced acid value and carbon residue. However, oxidation stability decreased slightly after adsorption (from 4.20 to 4.05 h), indicating partial removal of natural antioxidants. GC–MS analysis confirmed the dominance of methyl esters and the reduction of minor impurity-related peaks after purification. To comply with multi-parameter fuel requirements, WCO was blended with palm olein prior to conversion at ratios of 100:0 to 10:90 (w/w). Full EN 14214 compliance (FAME $\geq$ 96.5 wt%) was achieved at $\geq$50% palm olein, with oxidation stability of 10.2 h at a 10:90 ratio. These findings demonstrate that adsorption enhances compositional purity, whereas feedstock blending is decisive for restoring oxidative stability and achieving robust, fuel-grade biodiesel.

Keywords: Waste cooking oil biodiesel; adsorption-based purification; esterification–transesterification; feedstock blending; fuel-grade biodiesel; process integration.

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1. Introduction

Biodiesel has been widely recognized as a renewable alternative to petroleum diesel due to its biodegradability, low sulfur content, favorable lubricity, and potential to reduce greenhouse gas emissions. In practical applications, biodiesel is predominantly used in blended form with petroleum diesel (BXX), making fuel quality, stability, and operability critical for large-scale deployment, particularly in countries with high blending mandates such as Indonesia (Paryanto *et al.,* 2019; Mansur *et al.,* 2019).

The sustainability and economic viability of biodiesel production are strongly influenced by feedstock selection. In recent years, increasing attention has been directed toward low-cost and waste-derived feedstocks, including waste cooking oil (WCO) and acid oil generated as a by-product of glycerol acidulation. Acid oil, which contains high levels of free fatty acids (FFA) and residual fatty acid methyl esters, is a technically and economically feasible biodiesel feedstock when appropriately processed via esterification routes (Myint *et al.,* 2023). These developments reflect a broader interest in valorizing degraded or secondary lipid streams to improve the overall sustainability of biodiesel production.

Among waste-derived feedstocks, WCO has attracted particular interest due to its wide availability, low cost, and environmental benefits associated with waste reduction. However, biodiesel derived from WCO often exhibits inferior quality compared to biodiesel produced from refined vegetable oils. Repeated heating during frying promotes oxidation, hydrolysis, and thermal degradation, resulting in elevated FFA content and the formation of polar and polymerized compounds. These characteristics complicate both biodiesel conversion and purification, making WCO-derived biodiesel more susceptible to quality degradation and operational challenges (Leung *et al.,* 2010; Sales *et al.,* 2022). Moreover, feedstock variability arising from frying history, contamination, and degradation severity can propagate into process instability and inconsistent fuel properties, shifting the challenge from conversion efficiency toward achieving consistent fuel-grade compliance (Uğuz *et al.,* 2019; Monika *et al.,* 2023; Serqueira *et al.,* 2021).

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* Corresponding author
Email: fengpjs@ku.ac.th (P. Srnophakun)

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From a process perspective, high-FFA WCO complicates catalytic route selection, as conventional base-catalyzed transesterification becomes prone to saponification and phase-separation problems in the presence of excessive FFA and water. To address this limitation, recent studies have explored bifunctional heterogeneous catalysts that simultaneously promote esterification and transesterification in a single-step, thereby simplifying processing and potentially reducing downstream purification requirements (Elias *et al.,* 2020). Complementary catalyst design strategies that enhance surface area and tailor acid–base functionality, including supported mixed oxides derived from locally abundant minerals or wastes, have also been reported as pragmatic approaches for WCO conversion with improved sustainability and cost-effectiveness (Mulyatun *et al.,* 2021).

Beyond reaction completion, biodiesel upgrading increasingly relies on post-reaction purification strategies and compositional control to satisfy multi-attribute fuel standards. Adsorption-based purification and dry-washing techniques have been widely documented to remove polar impurities, residual glycerides, soaps, and degradation products, often leading to improvements in FAME content and purity-related parameters. However, several studies caution that excessive adsorption may remove naturally occurring antioxidant species, thereby reducing oxidation stability, particularly for biodiesel derived from waste feedstocks (Osman *et al.,* 2024; Awogbemi *et al.,* 2025; Hosseinzadeh-Bandbafha *et al.,* 2022; Lopresto *et al.,* 2025a). Consequently, blending has emerged as a practical complementary strategy to restore oxidation stability and rebalance fatty acid composition. WCO–palm-based blending, in particular, has been repeatedly shown to improve oxidation stability and facilitate compliance with fuel standards (Çamur and Al-Ani, 2022; Kumar *et al.,* 2023). This integrated perspective also aligns with real-world operability concerns, where cold-flow behavior and precipitation risks are often governed by fuel composition and trace glyceride species rather than by conversion alone (Pradana *et al.,* 2024; Gozan *et al.,* 2022).

Biodiesel is produced through esterification and/or transesterification reactions, in which triglycerides are converted stepwise into fatty acid methyl esters, forming diglycerides and monoglycerides as intermediates before glycerol is produced. Despite high conversion efficiencies, trace amounts of residual glycerides, particularly saturated monoglycerides, often remain in the final product. Numerous studies have demonstrated that even small concentrations of monoglycerides can significantly affect biodiesel properties, including viscosity, oxidation stability, cold-flow behavior, and precipitation tendency (Ghaizani *et al.,* 2018; Chupka *et al.,* 2014; Paryanto *et al.,* 2019). Due to their high melting points, saturated monoglycerides can crystallize above the cloud point, leading to precipitate formation during storage and handling, which may cause filter clogging and fuel instability (Mansur et al., 2019; Paryanto *et al.,* 2020). Recent work further indicates that precipitation behavior depends not only on monoglyceride concentration but also on blending ratio, temperature history, and overall fuel composition (Gozan *et al.,* 2022; Zuleta *et al.,* 2012).

To mitigate these challenges, integrated upgrading strategies are increasingly required. Two-step esterification–transesterification routes are commonly applied to high-FFA feedstocks to reduce soap formation and improve FAME yield (Atadashi *et al.,* 2010; Sales *et al.,* 2022; Hadiyanto *et al* 2020), while adsorption using materials such as activated carbon and silica gel has been explored to improve key fuel properties, including acid value, viscosity, and carbon residue (Paryanto *et al.,* 2020; Gozan *et al.,* 2022; Kpan *et al.,* 2019). Nevertheless, the potential trade-off between purification intensity and oxidation stability underscores the need for balanced, process-oriented evaluation (Zuleta *et al.,* 2012; Kpan *et al.,* 2019).

Despite extensive progress, many studies continue to address reaction optimization, purification, or blending in isolation, whereas industrial implementation requires simultaneous compliance across purity, stability, and operability under variable feedstock quality. It is important to distinguish between purity improvement and full fuel-grade compliance in the context of biodiesel upgrading. Improvements in fatty acid methyl ester (FAME) content, acid value, or carbon residue do not necessarily imply compliance with international biodiesel standards. In this study, adsorption-based purification is therefore evaluated primarily as a strategy for purity enhancement, while compliance with stringent fuel-grade specifications, particularly those defined in EN 14214, is assessed separately. By explicitly addressing the purification–stability trade-off and positioning the results within the broader WCO upgrading literature, this work provides process-relevant guidance for robust biodiesel quality control under heterogeneous waste-oil conditions.

2. Materials and methods

2.1. Materials

Waste cooking oil (WCO) was collected from a food processing facility operated by Charoen Pokphand Foods (Thailand). Approximately 30 L of WCO was filtered to remove suspended solids and subsequently heated to 105 °C for 1 h to remove residual moisture before biodiesel production. Fresh palm olein (27 L) was obtained commercially (Gaysorn Brand) and used as a blending feedstock.

Analytical-grade methanol (≥99.8%), potassium hydroxide (KOH pellets), and sulfuric acid (H_2SO_4, 98%) were used as catalysts and reactants. Activated carbon (decolorizing grade, Panreac) and silica gel 60 Å (40–60 µm) were used as adsorbents without further pretreatment. Activated carbon and silica gel were used as received, without further pretreatment or physicochemical modification. No additional characterization (e.g., BET surface area, pore size distribution, or SEM analysis) was performed in this study. The adsorption performance was therefore evaluated based on macroscopic fuel quality parameters rather than intrinsic adsorbent properties.

2.2. Biodiesel Production via Transesterification

In this study, the single-step process refers to direct base-catalyzed transesterification of waste cooking oil (WCO) using KOH as the catalyst. The two-step process involves sequential acid esterification followed by base-catalyzed transesterification to reduce the free fatty acid (FFA) content of WCO prior to the main transesterification step, thereby minimizing soap formation and improving FAME yield.

Base-catalyzed transesterification was conducted in a 500–1000 mL three-neck glass reactor equipped with a reflux condenser, thermocouple probe, and magnetic stirrer. The reactor was placed on a temperature-controlled hot plate. KOH pellets were first dissolved in methanol and then added to the reactor containing 250–500 g of preheated WCO at 60 °C. The reaction was carried out at methanol-to-oil mass ratios of 6:1-12:1, catalyst loadings of 1.0–2.0 wt% (based on oil mass), and reaction times of 60–120 minute as Ulukardesler (2023). Continuous stirring was applied throughout the reaction. Upon completion, the reaction mixture was transferred to a separating funnel and allowed to settle under gravity. The biodiesel phase was separated from the glycerol-rich phase, washed repeatedly with hot distilled water until the pH was neutral, and dried at 105 °C for 1 h to remove residual moisture (Degife *et al.,* 2015).

2.3. Adsorption-Based Purification

Batch adsorption experiments were conducted in a 600 mL beaker equipped with a thermocouple probe and magnetic stirrer. Biodiesel samples were heated to the desired temperature and maintained at that temperature during adsorption.

Activated carbon adsorption was performed at 60 °C with adsorbent loadings ranging from 0.5 to 2.0 wt% and contact times of 20–60 min. Silica gel adsorption was conducted under optimized conditions: 3 wt% adsorbent loading, 72 min contact time, and 112 °C. After adsorption, the biodiesel was separated from the adsorbent using vacuum filtration.

2.4. Feedstock Blending Strategy

To further improve biodiesel quality, WCO was blended with fresh palm olein at volumetric ratios ranging from 100:0 to 0:100. Biodiesel was produced from each blend under optimized transesterification conditions, and the resulting fuel properties were evaluated to assess compliance with commercial biodiesel standards.

2.5. Analytical Methods

Gas chromatography (GC–MS, Shimadzu GC-2010 PLUS) analysis was performed using a capillary column (30 m × 0.25 mm × 0.25 μm) suitable for fatty acid methyl ester (FAME) separation. The injector temperature was maintained at 250 °C. The oven temperature was initially set at 50 °C, then ramped at 10 °C min^{-1} to 280 °C and held for 10 min. Helium was used as the carrier gas at a constant flow rate of 1.0 mL min^{-1}. The mass spectrometer was operated in electron impact (EI) mode at 70 eV. The mass spectrometer was operated in electron impact (EI) mode at 70 eV. Mass spectra were recorded in the m/z range of 40–500. Compound identification was performed by comparing spectra with the NIST library. Acid value was measured according to ASTM D664, oxidation stability was determined following EN 15751, kinematic viscosity was measured using ASTM D445, and carbon residue was evaluated using ASTM D189.

3. Results and Discussion

3.1. Physicochemical Properties of WCO and Fatty Acid Composition of Palm Olein

To provide a baseline understanding of the feedstocks used in this study, the key physicochemical properties of waste cooking oil (WCO) and palm olein were first evaluated. These properties are critical because parameters such as acid value, viscosity, and density strongly influence both reaction behavior and downstream fuel quality. The comparative characteristics of WCO and palm olein are summarized in Table 1. As shown in Table 1, WCO exhibits a significantly higher acid value (5.21 mg KOH g^{-1}) compared to palm olein (0.56 mg KOH g^{-1}), confirming the degraded nature of WCO and the need for pretreatment prior to base-catalyzed transesterification. The higher viscosity of WCO also reflects the presence of oxidation and polymerization products formed during repeated frying. These differences highlight why WCO requires more careful selection of reaction pathways and post-reaction upgrading than fresh vegetable oil.

Table 1
Physicochemical properties of oil samples

Parameters	Unit	Waste Cooking Oil	Palm Olein
Acid value	mg KOH/g	5.21	0.56
Viscosity	cSt	43.4	40.22
Density	g/mL	889	897
Molecular weight	g/mol	866.22	855.13

Table 2
Quantification of fatty acids in oil samples

Fatty Acid	Structure	Waste Cooking Oil (%)	Palm Olein (%)
Saturated			
Lauric acid	C12:0	*ND*	0.17
Myristic acid	C14:0	0.33	0.53
Palmitic acid	C16:0	21.3	28.83
Stearic acid	C18:0	4.05	3.97
Arachidic acid	C20:0	0.29	0.31
Behenic acid	C22:0	0.25	0.15
Lignoceric acid	C24:0	0.2	0.12
Monounsaturated			
Palmitoleic acid	C16:1	0.6	0.37
Oleic acid	C18:1	32.14	40.26
Erucic acid	C22:1	0.09	*ND*
Polyunsaturated			
Linoleic acid	C18:2	37.66	23.8
Linolenic acid	C18:3	3.07	1.49

Note: ND = Not Detected

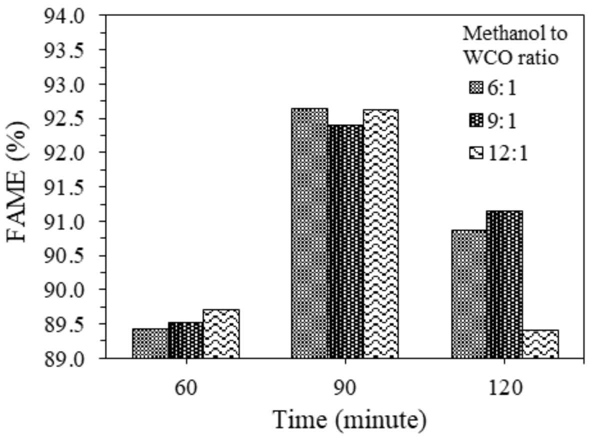

Fig 1. Effect of the methanol-to-waste cooking oil mass (w/w) ratio on the FAME at a temperature of 60 °C and 1.2 wt% KOH

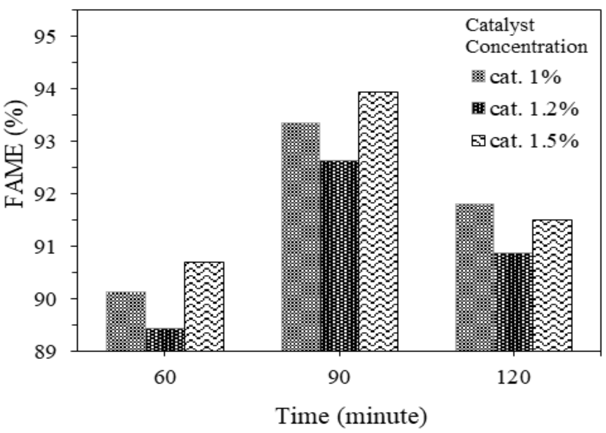

Fig 2. The effect of the concentration of potassium hydroxide on the FAME at a temperature of 60 °C

In addition to bulk physicochemical properties, the fatty acid composition of the oils plays a crucial role in determining biodiesel quality, particularly with respect to oxidation stability and cold-flow behavior. Therefore, the fatty acid profiles of WCO and palm olein were quantified and are presented in Table 2. Table 2 shows that WCO contains a higher proportion of polyunsaturated fatty acids, particularly linoleic acid (C18:2), compared to palm olein. This higher degree of unsaturation is known to reduce oxidation stability and increase susceptibility to degradation during storage. In contrast, palm olein exhibits a higher saturated and monounsaturated fatty acid content, which contributes to its superior oxidative stability. These compositional differences explain why blending WCO-derived biodiesel with palm olein-based biodiesel can effectively restore stability-related fuel properties.

3.2. Biodiesel Production from Waste Cooking Oil

3.2.1. Effect of Methanol-to-Oil Ratio and Reaction Time

The methanol-to-oil mass ratio and reaction time are two key parameters governing the position of the transesterification equilibrium and the rate of fatty acid methyl ester (FAME) formation. While the stoichiometric requirement is 3:1, excess methanol is commonly employed to shift the equilibrium toward ester production. However, the extent to which higher methanol-to-oil ratios enhance conversion depends strongly on the reaction time. Therefore, the combined effects of methanol-to-oil ratio and reaction time on FAME content were systematically evaluated, as presented in Figure 1. As shown in Figure 1, increasing the methanol-to-oil mass ratio from 6:1 to 12:1 improved FAME yield at shorter reaction times. At 60 min, the highest FAME content (89.71 wt%) was obtained at a mass ratio of 12:1, indicating faster equilibrium displacement at higher alcohol availability.

However, extending the reaction time revealed a different trend. At 90 min, the maximum FAME content (92.64 wt%) was achieved at a methanol-to-oil ratio of 6:1, while further increases in the methanol ratio did not provide additional benefit. Prolonged reaction time (120 min) resulted in a decline in FAME content across all ratios, suggesting the occurrence of reverse reactions and increased solubility of glycerol in the ester phase. These results indicate that excessive alcohol and prolonged reaction time can negatively affect phase separation and product purity, consistent with previous findings on equilibrium reversal and glycerol retention. Overall, the optimal conditions for transesterification were identified as a methanol-to-oil ratio of 6:1 and a reaction time of 90 min, balancing conversion efficiency and product stability.

3.2.2 Effect of Catalyst Concentration

In addition to the methanol-to-oil ratio and reaction time, catalyst concentration plays a critical role in controlling the reaction rate, equilibrium position, and extent of triglyceride conversion during transesterification. An insufficient catalyst amount may limit the availability of active sites, whereas excessive catalyst loading can promote undesirable side reactions, particularly saponification in the presence of free fatty acids. Therefore, the effect of potassium hydroxide (KOH) concentration on FAME formation was systematically evaluated at different reaction times, as presented in Figure 2. As shown in Figure 2, increasing KOH concentration from 1.0 wt% to 1.5 wt% enhanced FAME formation, with the maximum value (93.94 wt%) obtained at 1.5 wt% after 90 min. This behavior can be attributed to the increased availability of active sites, which accelerates triglyceride conversion.

Nevertheless, further increases in catalyst concentration or extended reaction time led to reduced FAME content, likely due to soap formation and reverse reactions promoted by excess catalyst in the presence of free fatty acids. These findings confirm that catalyst dosage must be carefully optimized, particularly for degraded feedstocks such as waste cooking oil. Based on the results, 1.5 wt% KOH was selected as the optimal catalyst concentration.

3.3 Effect of Esterification Pretreatment

For high–free fatty acid feedstocks such as waste cooking oil, direct base-catalyzed transesterification often leads to soap formation and poor phase separation. To overcome this limitation, an acid esterification pretreatment is commonly used to convert free fatty acids into esters, thereby reducing the acid value prior to the main transesterification step. In this study, the effectiveness of sulfuric acid–catalyzed esterification at different acid concentrations was evaluated, and the resulting changes in acid value are shown in Figure 3.

Esterification effectively reduced the acid value from 5.21 mg KOH/g oil to below 1.0 mg KOH/g oil at a sulfuric acid concentration of 1.5 wt% (Figure 3), enabling subsequent transesterification without excessive soap formation. Despite this improvement, the two-step process did not yield a

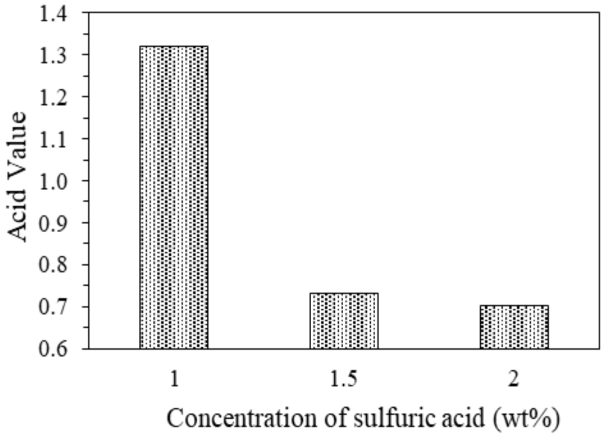

Fig 3. Acid value of waste cooking oil after esterification

significantly higher FAME content than the single-step process before purification. Instead, biodiesel produced via the two-step route exhibited slightly higher acid value and carbon residue, suggesting the presence of residual intermediates. These results highlight that while the two-step process improves process robustness for high-free-fatty-acid feedstocks, it does not inherently guarantee superior final fuel quality.

3.4. Adsorption-Based Upgrading: Effectiveness and Limitations

3.4.1. Effect of Adsorbent Type and Dosage

After reaction pathway selection, the quality of crude biodiesel remains limited by residual glycerides, soaps, and polar degradation products, which adversely affect fuel properties. To further improve biodiesel purity, adsorption was applied as a post-reaction upgrading step using solid adsorbents. The influence of activated carbon dosage on FAME content during biodiesel purification is presented in Figure 4.

Figure 4 illustrates the effect of adsorption on the reduction of polar impurities, particularly acid value, during the purification of WCO-derived biodiesel. The initial decrease in acid value reflects rapid surface adsorption of free fatty acids and polar degradation products, consistent with classical adsorption behavior governed by external mass transfer and the availability of active adsorbent sites. At higher adsorption levels, the rate of improvement gradually diminishes, suggesting the onset of pore blocking and partial saturation of adsorption sites. Such behavior is widely reported for adsorption systems involving complex liquid mixtures, where large molecular species can occupy pore entrances and hinder further adsorption of smaller compounds (Osman *et al.,* 2024; Awogbemi *et al.,* 2025). Similar equilibrium-type trends have also been observed in biodiesel purification studies using carbon-based adsorbents, indicating that adsorption performance is ultimately constrained by pore accessibility and adsorbent surface heterogeneity (Lopresto *et al.,* 2025a).

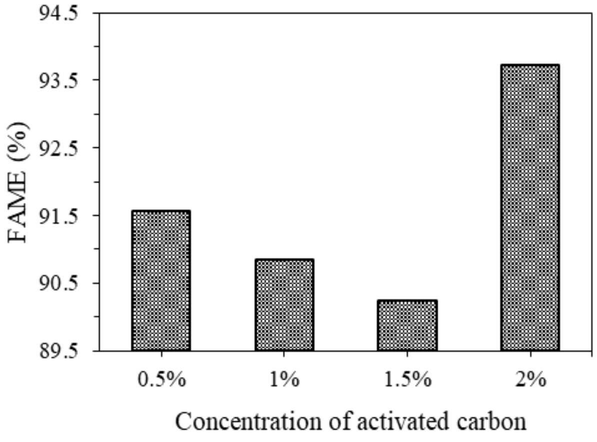

Fig 4. Effect of concentration of activated carbon on the FAME at a temperature of 40 °C

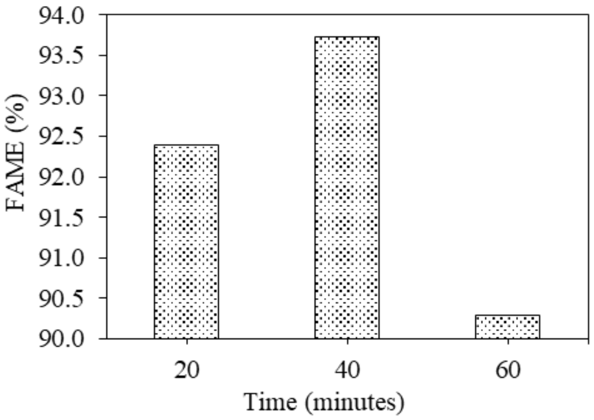

Fig 5. Effect of time on the FAME at a concentration of activated carbon 2 wt%

In addition to adsorbent dosage, contact time is another critical parameter that governs adsorption efficiency and the extent of impurity removal during biodiesel purification. At shorter contact times, adsorption is dominated by rapid surface interactions, whereas prolonged exposure may lead to site saturation and equilibrium effects that limit further improvement. Therefore, the influence of contact time on FAME content at a fixed activated carbon concentration was evaluated, as shown in Figure 5.

Figure 5 compares the adsorption performance of activated carbon and silica gel, highlighting the superior effectiveness of activated carbon in removing polar, high-molecular-weight compounds, such as carbon residue. This behavior can be interpreted in terms of differences in pore structure and adsorption equilibrium. Activated carbon typically exhibits a broader pore-size distribution and a higher surface area, facilitating multilayer adsorption and stronger interactions with large molecular species. In contrast, silica gel has a more uniform pore structure, which may limit its capacity to accommodate bulky degradation products and promote earlier pore blocking (Osman *et al.,* 2024; Lopresto *et al.,* 2025a). Moreover, adsorption systems involving complex organic mixtures often approach a quasi-equilibrium state, in which further impurity removal becomes thermodynamically unfavorable due to a balance between desorption and adsorption and the competitive occupation of active sites (Awogbemi *et al.,* 2025). These equilibrium constraints explain the diminishing marginal improvement observed at higher purification levels and underscore the intrinsic trade-off between adsorption efficiency and achievable fuel quality.

As shown in Figures 4 and 5, activated carbon exhibited optimal performance at a dosage of 2 wt% and a contact time of 40 min, resulting in an increase in FAME content to 94.20 wt%. Lower dosages were insufficient to remove impurities, while prolonged contact time led to partial desorption, reducing FAME content. Silica gel demonstrated a greater effect on FAME improvement at higher temperatures but showed limited effectiveness in reducing acid value. These results indicate that

Table 3
Properties of biodiesel before and after adsorption by activated carbon and silica gel

Properties	Unit	Transesterification			Two-step Process			ASTM D6751	EN 14214	Compliance with	
		Control	AC	SG	Control	AC	SG	Limit	Limit	ASTM	EN
FAME	wt%	93.94	94.2	94.97	93.69	94.42	95.30	≥96.5	≥96.5	No	No
Acid value	mg KOH/g	0.45	0.43	0.45	0.48	0.45	0.476	≤0.50	≤0.50	Yes	Yes
Viscosity	mm^2/s at 40°C	4.53	4.5	4.5	4.5	4.51	4.505	1.9–6.0	3.5–5.0	Yes	Yes
Carbon residue	wt%	0.59	0.39	0.41	0.64	0.49	0.454	<0.05[b]	0.3[b]	No	No
Oxidation Stability	h	4.20	4.05	3.44	3.16	3.15	2.4	≥3	≥6	Yes	No

Notes: Control = Unpurified biodiesel; AC = Adsorption by Activated Carbon; SG = Adsorption by Silica Gel.
[a]ASTM D6751 does not specify a minimum FAME content.; EN 14214 serves as the benchmark for full fuel-grade compliance, while ASTM D6751 is used for comparison against minimum operability requirements.
[b]Different method: ASTM tests the whole biodiesel sample using the Micro Carbon Residue (ASTM D4530) method, while EN tests the heaviest part (10% residue) from the distillation process of the sample (EN ISO 10370).

adsorbent type and operating conditions strongly influence purification efficiency.

3.4.2 Impact of Adsorption on Fuel Quality and Oxidation Stability

To evaluate the effectiveness of adsorption as a post-reaction upgrading strategy, key biodiesel quality parameters were analyzed before and after purification using activated carbon (AC) and silica gel (SG). These parameters represent critical indicators of fuel purity, stability, and compliance with international standards. The comparative effects of both adsorbents under transesterification and two-step production routes are summarized in Table 3. Table 3 shows that adsorption improved FAME content and reduced acid value and carbon residue in both reaction pathways. In the transesterification route, activated carbon increased FAME from 93.94 wt% (control) to 94.28 wt%, while silica gel increased FAME to 94.97 wt%. Under the two-step process, FAME increased from 93.69 wt% (control) to 94.42 wt% with activated carbon and reached 95.30 wt% with silica gel. These results indicate that silica gel exhibits slightly stronger selectivity for the removal of residual glycerides and polar fractions, which contribute to incomplete ester purity.

In terms of acid value reduction, activated carbon consistently demonstrated superior performance, lowering the acid value more effectively than silica gel in both production routes. This may be attributed to the broader pore-size distribution and greater surface heterogeneity of activated carbon, which enhances the adsorption of free fatty acids and polar degradation products. Similarly, carbon residue was reduced after adsorption treatment, although the reduction remained more strongly associated with purification efficiency rather than feedstock composition at this stage.

Despite improvements in FAME purity, adsorption treatment resulted in a slight decrease in oxidation stability. For the transesterification route, oxidation stability decreased from 4.20 h (control) to 4.05 h with activated carbon and to 3.44 h with silica gel. A similar trend was observed in the two-step process. This behavior is likely related to the partial removal of naturally occurring antioxidant species during adsorption. Although specific antioxidant compounds were not quantified in this study, the trend aligns with previous reports that adsorption can reduce both impurities and stabilizing phenolic components in waste-derived biodiesel.

Importantly, while adsorption increased FAME to approximately 94–95 wt%, this level remains below the EN 14214 minimum requirement of 96.5 wt%. Therefore, adsorption alone is insufficient to achieve full compliance with fuel-grade standards. These findings highlight the inherent trade-off between compositional purity and oxidation stability, and they justify the need for an additional upgrading strategy, such as feedstock blending, to achieve simultaneous compliance with multiple fuel-quality parameters.

To further examine the compositional profile of the purified biodiesel, gas chromatography–mass spectrometry (GC–MS) analysis was performed on the adsorption-treated sample. The analysis was conducted under standard GC–MS conditions for fatty acid methyl ester (FAME) identification, allowing separation and detection of methyl ester components based on retention time and mass spectra. The resulting chromatogram provides qualitative insight into the dominant ester species present after adsorption-based purification.

As shown in Figure 6, the chromatogram of adsorption-treated biodiesel is dominated by well-defined peaks corresponding to common fatty acid methyl esters, including methyl palmitate (C16:0), methyl stearate (C18:0), methyl oleate (C18:1), methyl linoleate (C18:2), and minor long-chain esters such as methyl arachidate (C20) and methyl behenate (C22:0). The strong and sharp peaks within the retention time range of approximately 9–15 minutes indicate a high proportion of FAME species. Only minor low-intensity signals are observed outside the primary FAME region, suggesting effective removal of polar impurities and non-ester fractions through adsorption treatment.

The predominance of methyl ester peaks in Figure 6 supports the numerical increase in FAME content reported after

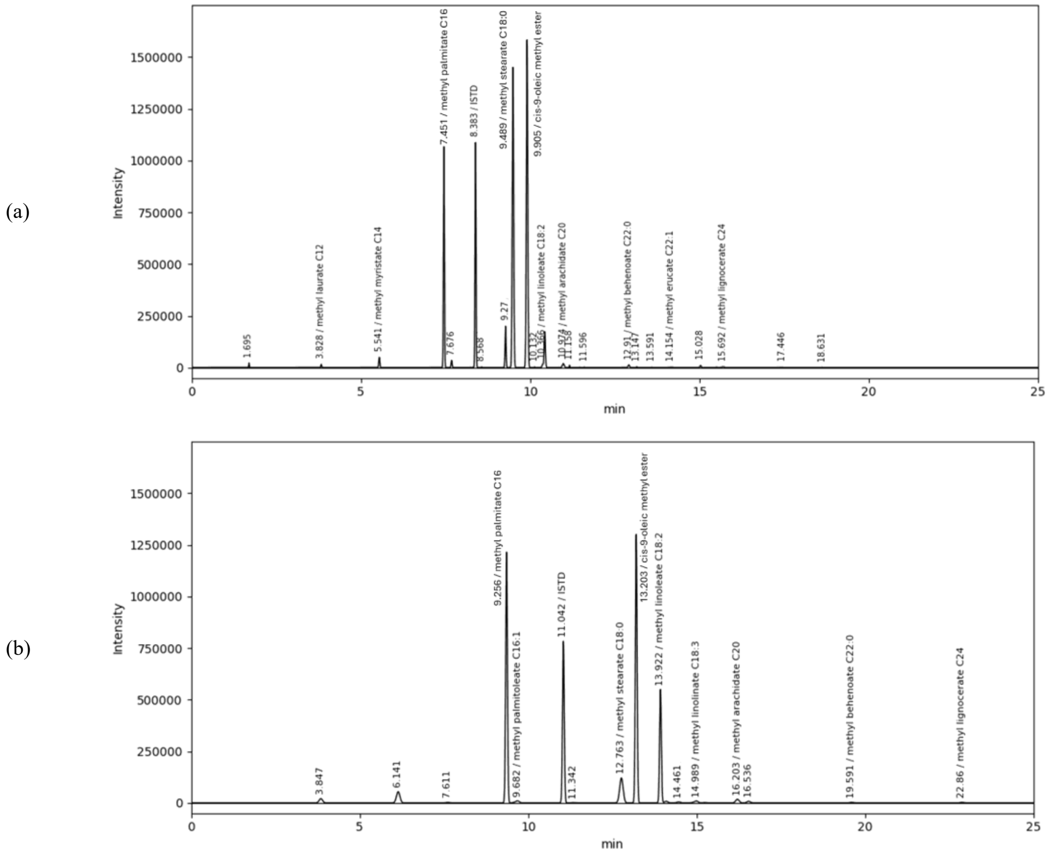

Fig 6. GC–MS chromatograms of (a) unpurified biodiesel, and (b) two-step transesterification and adsorption biodiesel. The unpurified sample exhibits additional low-intensity peaks corresponding to residual impurities and non-FAME compounds, while the treated sample shows enhanced dominance of FAME components.

adsorption. The reduction of minor impurity-related peaks qualitatively indicates the removal of residual glycerides, polar degradation products, and other non-volatile fractions. However, while adsorption enhances compositional purity, the slight decrease in oxidation stability observed after treatment may be attributed to the concurrent removal of naturally occurring antioxidant species present in degraded waste cooking oil. This suggests that adsorption improves fuel cleanliness but may alter the balance of stabilizing compounds, thereby contributing to the purification–stability trade-off. The chromatographic profile thus reinforces the process-level findings by providing molecular-level confirmation of compositional refinement after adsorption.

Several studies have demonstrated that adsorption and water- or dry-washing steps can reduce oxidation stability by removing phenolic compounds, tocopherols, and other naturally occurring antioxidants present in degraded lipid feedstocks (Hosseinzadeh-Bandbafha *et al.,* 2022; Osman *et al.,* 2024; Awogbemi *et al.,* 2025; Lopresto *et al.,* 2025a). These compounds, although present in trace amounts, play a disproportionate role in delaying peroxide formation during accelerated oxidation testing. Consequently, purification strategies that aggressively remove polar species may inadvertently reduce oxidation stability, particularly in waste-cooking-oil–derived biodiesel.

3.5 Feedstock Blending as a Complementary Upgrading Strategy

While adsorption significantly improves several purity-related properties, it was insufficient to achieve full compliance with EN 14214, particularly in terms of oxidation stability and FAME content. To address this limitation, feedstock blending was investigated as a complementary upgrading strategy. Fresh palm olein was progressively blended with waste cooking oil (WCO)–derived biodiesel to rebalance the fuel composition and restore key quality attributes. The effect of volumetric blending ratio on FAME content, acid value, carbon residue, oxidation stability, and EN 14214 compliance is summarized in Table 4. As shown in Table 4, increasing the palm olein fraction systematically improved both FAME content and oxidation stability. At a WCO:palm olein ratio of 50:50, the FAME content reached 96.5 wt%, which meets the EN 14214 minimum requirement. Further increases in palm olein content (25:75 and 10:90) enhanced both FAME purity and oxidation stability, confirming that feedstock composition plays a decisive role in achieving fuel-grade biodiesel from degraded waste oils.

Notably, oxidation stability increased from approximately 4 h for neat WCO-derived biodiesel to values exceeding 8 h after blending, demonstrating that palm olein effectively compensates for the loss of natural antioxidants caused by adsorption-based purification. This confirms blending as a critical “second lever” for restoring stability in waste-derived biodiesel.

In addition to oxidation stability and FAME content, feedstock blending also reduced carbon residue. As the palm olein fraction increased, carbon residue decreased from 0.45 wt% for neat WCO biodiesel to below 0.2 wt% at high palm olein proportions. This indicates that carbon residue is influenced by both purification efficiency and feedstock composition, with

Table 4
Effect of feedstock blending on biodiesel quality

WCO:Palm Olein (w/w)	FAME content (wt%)	Acid value (mg KOH g^{-1})	Carbon residue EN (wt%)	Oxidation Stability (h)	EN 14214 compliance
100 : 0	94.2	0.45	0.45	4.05	No
75 : 25	95.3	0.38	0.39	6.20	No
50 : 50	96.5	0.31	0.29	≥ 8	Yes
25 : 75	97.1	0.27	< 0.2	≥ 9	Yes
10 : 90	97.8	0.24	< 0.2	≥ 10	Yes

blending providing an additional pathway for improving combustion-related properties.

Overall, blending enabled simultaneous restoration of oxidation stability, enhancement of FAME content to ≥96.5 wt%, and reduction of carbon residue. These results demonstrate that feedstock blending offers a practical, flexible, and industrially relevant strategy to address the inherent limitations of degraded waste cooking oil.

3.6 Purification–Stability Trade-Off and the Role of Feedstock Composition

Several recent studies have emphasized that biodiesel derived from waste cooking oil exhibits inherently variable quality due to differences in fatty acid composition, thermal degradation, and the presence of oxidation products formed during frying operations (Uğuz *et al.,* 2019; Monika *et al.,* 2023; Serqueira *et al.,* 2021). These factors not only influence transesterification efficiency but also govern downstream fuel properties, including oxidation stability, cold-flow behavior, and storage performance. As a result, biodiesel upgrading strategies must address both impurity removal and compositional effects, rather than focusing solely on conversion efficiency.

Adsorption-based purification and dry-washing techniques have been widely reported as effective methods for reducing residual glycerides, free fatty acids, soaps, and polar degradation products in biodiesel, particularly when water-based washing is undesirable (Osman *et al.,* 2024; Ferrusca *et al.,* 2023; Awogbemi *et al.,* 2025). The improvements in FAME content, acid value, and carbon residue observed in the present study after adsorption using activated carbon and silica gel are therefore consistent with the broader literature. However, several authors have also reported that excessive adsorption may reduce oxidation stability, as adsorbents can remove not only undesirable impurities but also naturally occurring antioxidant compounds retained in waste-derived feedstocks (Hosseinzadeh-Bandbafha *et al.,* 2022; Lopresto *et al.,* 2025b). This mechanism provides a theoretical explanation for the observed decline in oxidation stability after adsorption in this study and highlights a necessary trade-off between fuel purity and oxidative durability.

To overcome this limitation, blending has increasingly been recognized as a powerful and practical design variable for controlling biodiesel quality. Studies on waste cooking oil biodiesel blended with more saturated biodiesel fractions, such as palm-based biodiesel, consistently report substantial improvements in oxidation stability due to reduced unsaturation and a more balanced fatty acid profile (Çamur and Al-Ani, 2022; Kumar *et al.,* 2023). Similar trends have been observed in investigations focusing on cold-flow properties and precipitation behavior, where feedstock composition and monoglyceride content play dominant roles in fuel operability (Pradana *et al.,* 2024; Gozan *et al.*, 2022; Wahyono *et al,* 2022). The significant improvement in oxidation stability achieved through palm olein blending in the present study, therefore, aligns well with these findings. It supports the conclusion that feedstock composition control is often more decisive than purification intensity for meeting fuel-grade specifications.

Taken together, these observations reinforce the need for an integrated upgrading strategy when processing heterogeneous waste-derived oils. While adsorption-based purification is effective for improving purity-related parameters, blending provides a complementary lever to address composition-driven properties such as oxidation stability and low-temperature performance. Similar conclusions have been reached in recent reviews and comparative studies, which emphasize that no single upgrading method can simultaneously optimize all biodiesel quality attributes for waste-derived feedstocks (Monika *et al.,* 2023; Osman *et al.,* 2024; Awogbemi *et al.,* 2025). The integrated framework adopted in this study is therefore consistent with current scientific understanding and offers a practical pathway for robust biodiesel production under variable feedstock conditions.

The superior performance of activated carbon compared to silica gel in reducing acid value and carbon residue can be explained by well-established differences in adsorbent structure reported in the literature. Activated carbon typically exhibits a significantly higher specific surface area and a broader pore-size distribution, which enhances its capacity to adsorb free fatty acids and polar compounds. In contrast, silica gel possesses a more uniform pore structure and a lower surface area, which may limit its effectiveness in removing larger degradation products from waste-derived biodiesel. While no direct adsorbent characterization was conducted in this study, the observed trends are consistent with previously reported adsorption behavior for similar systems (Osman *et al.,* 2024; Awogbemi *et al.,* 2025; Lopresto *et al.,* 2025a).

Detailed physicochemical characterization of the adsorbents, including BET surface area, pore volume, and surface morphology, was beyond the scope of the present study and is recommended for future work to further elucidate structure–performance relationships in adsorption-based biodiesel purification.

3.7 Integrated Process Perspective and Design Implications

The results demonstrate that no single upgrading strategy is sufficient to address all quality constraints associated with biodiesel derived from waste cooking oil. Reaction pathway selection ensures process feasibility, adsorption improves fuel purity but introduces stability trade-offs, and feedstock blending provides a practical route to restoring fuel performance. The integrated application of these strategies offers a process-relevant framework for biodiesel upgrading under variable feedstock conditions. To further contextualize the present results, a comparison with representative studies on biodiesel derived from waste or degraded lipid feedstocks is presented in Table 5.

Table 5
Positioning of the present study relative to selected studies on the quality improvement of waste/waste cooking oil biodiesel

Reference	Feedstock and strategy	Key reported outcomes	Positioning relative to this work
This study (adsorption stage)	Waste cooking oil biodiesel; adsorption using activated carbon or silica gel	FAME increased to 94–95 wt%; acid value and carbon residue decreased; oxidation stability reduced to 3.40–4.05 h	Demonstrates that adsorption improves purity-related parameters but may compromise oxidation stability due to the removal of antioxidant species
This study (blending stage)	Waste cooking oil blended with palm olein before biodiesel production	Oxidation stability increased up to >10 h at high palm-olein fraction; FAME ≥ 96.5 wt%	Shows blending as the most effective strategy for achieving oxidation stability and fuel-grade compliance
Rahadianti *et al.*. (2018)	Waste cooking oil biodiesel via alkali transesterification	Viscosity ~5.9 cSt; acid value ~0.26 mg KOH g^{-1}	The present work achieves lower viscosity while maintaining a comparable acid value, highlighting trade-offs between feedstock quality and upgrading strategy.
Mansur *et al.*. (2019)	FAME–diesel blends; precipitation behavior analysis	Precipitation is strongly linked to monoglyceride content and blending ratio	Supports the present emphasis on impurity control and blend composition
Çamur and Al-Ani (2022)	Waste frying oil biodiesel blended with palm-based biodiesel	Oxidation stability improved to meet EN 14214 (≥8 h) at higher palm fractions	Consistent with the blending-driven stability improvement observed in this study
Gozan *et al.*. (2022)	Prediction and mitigation of precipitate formation in B20 fuels	Fuel stability is governed by monoglyceride content and feedstock composition	Reinforces the integrated process perspective adopted in this work

Previous studies have consistently shown that biodiesel quality, particularly oxidation stability and precipitation tendency, is strongly influenced by residual glycerides, free fatty acids, and fatty acid composition, rather than by conversion efficiency alone (Knothe, 2005; Paryanto *et al.,* 2019). In the present study, adsorption using activated carbon and silica gel effectively improved biodiesel purity, as indicated by increased fatty acid methyl ester (FAME) content and reduced acid value and carbon residue. However, oxidation stability decreased after adsorption, particularly when silica gel was used. This behavior is consistent with the established understanding that adsorption may remove not only undesirable polar impurities but also naturally occurring antioxidant compounds, thereby reducing oxidative resistance despite higher apparent fuel purity (Knothe and Razon, 2017; Zuleta *et al.,* 2012). By contrast, blending waste cooking oil biodiesel with palm-olein-derived biodiesel resulted in a substantial improvement in oxidation stability (Silviana *et al*, 2022). Feedstock blending with palm olein represents an economically pragmatic alternative to extensive purification or the use of synthetic antioxidants. Although palm olein is a higher-quality feedstock than waste cooking oil, blending does not require additional unit operations or energy input beyond conventional mixing. In regions where palm-based oils are widely available, blending offers a cost-effective strategy to recover fuel-grade compliance from degraded waste streams, particularly when compared to repeated adsorption or chemical additive approaches.

Similar trends have been reported in previous studies, in which blending waste-derived biodiesel with more saturated biodiesel fractions was identified as an effective strategy to meet stricter oxidation-stability requirements, such as those specified in EN 14214 (Çamur and Al-Ani, 2022; Gozan *et al.,* 2022). While several studies have focused either on reaction optimization, purification, or blending strategies in isolation, the present work demonstrates that no single approach is sufficient to address all quality constraints associated with degraded feedstocks. Instead, the integrated application of reaction pathway selection, adsorption-based purification, and feedstock blending provides a more robust and flexible pathway toward producing fuel-grade biodiesel under variable feedstock conditions.

From an energy perspective, adsorption using silica gel requires heating to approximately 112 °C to ensure effective removal of polar impurities. While this condition is feasible at the laboratory scale and is commonly reported in the literature, it introduces an additional thermal energy demand that may reduce overall process efficiency at a large scale. In contrast, adsorption using activated carbon can be conducted under milder conditions, i.e. 60 °C, making it more attractive from an energy-consumption standpoint for practical implementation.

Overall, the comparison confirms that while adsorption-based purification enhances biodiesel purity, control of feedstock composition through blending is the dominant factor governing oxidation stability, and the integrated application of multiple upgrading strategies is required to achieve robust fuel-grade biodiesel from degraded waste oils. From an integrated process viewpoint, the results suggest that upgrading strategies should not be evaluated solely on reaction yield or impurity removal, but also on their combined energy, operational, and economic implications. In this context, feedstock blending emerges as a low-energy, low-complexity lever that complements adsorption-based purification, aligning with industrial constraints on cost and operability.

Direct identification and quantification of antioxidant species (e.g., total phenolic content or HPLC-based profiling) were beyond the scope of this study. Future work combining adsorption performance with targeted antioxidant analysis would provide deeper mechanistic insight into the observed trade-off between purification and oxidation stability.

4. Conclusions

This study demonstrates that adsorption using activated carbon and silica gel effectively improves FAME purity and reduces acid value and carbon residue in WCO-derived biodiesel. Silica gel achieved slightly higher FAME levels (up to 95.3 wt%), whereas activated carbon showed a stronger reduction in acid value. However, adsorption alone did not meet the EN 14214 minimum FAME requirement of 96.5 wt% and resulted in a slight decrease in oxidation stability, indicating a trade-off between purification and stability. GC–MS analysis qualitatively confirmed the predominance of methyl ester species after adsorption. Full compliance with EN 14214 was achieved only through feedstock blending with palm olein, which restored oxidation stability and increased FAME content above 96.5 wt%. The integrated upgrading framework highlights that process-level control, rather than reaction optimization alone, is critical for producing robust biodiesel from heterogeneous waste-derived feedstocks.

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