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Improving the Environmental Performance of Palm Biodiesel via $AgNO_3$-Assisted Removal of Polyunsaturated Fatty Acids

Zuchra Helwani [1,*], Ida Zahrina [1], Said Zul Amraini [1], Rinaldi Idroes [2], Godlief Frederick Neonufa [3] and Sunarti Abd Rahman [4]

[1] Department of Chemical Engineering, Universitas Riau, Pekanbaru 28293, Indonesia; zuchra.helwani@lecturer.unri.ac.id (Z.H.); ida.zahrina@eng.unri.ac.id (I.Z.); saidzulamraini@lecturer.unri.ac.id (S.Z.A.)
[2] Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Syiah Kuala, Banda Aceh 23111, Indonesia; rinaldi.idroes@usk.ac.id (R.I.)
[3] Department of Agricultural Product Technology, Universitas Kristen Artha Wacana, Kupang, Indonesia; godliefneonufa@gmail.com (G.F.N.)
[4] Faculty of Chemical and Process Engineering Technology, Universiti Malaysia Pahang Al-Sultan Abdullah, Lebuhraya Persiaran Tun Khalil Yaakob 26300, Kuantan, Pahang, Malaysia; sunarti@umpsa.edu.my (S.A.R.)

***** Correspondence: zuchra.helwani@lecturer.unri.ac.id

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Abstract

Indonesian biodiesel products commonly exhibit low oxidative stability and high cloud points, which limit their performance and widespread use. These drawbacks are primarily due to the high content of polyunsaturated fatty acids (PUFAs) in the fatty acid methyl ester (FAME) mixture that constitutes biodiesel. A more suitable biodiesel composition includes higher proportions of saturated and monounsaturated fatty acids, which offer better combustion properties, higher cetane numbers, and greater resistance to oxidative degradation. In contrast, PUFAs promote oxidation reactions, resulting in fuel instability, increased sludge formation, and higher emissions of unburned hydrocarbons, negatively impacting both engine performance and the environment.This study investigates the use of silver nitrate ($AgNO_3$) as a selective extraction agent to remove PUFAs from palm oil-derived FAME. The goal is to identify the most effective biodiesel-to-$AgNO_3$ volume ratio for separating saturated fatty acid fractions from unsaturated ones, in order to produce a more stable and environmentally friendly biodiesel. Experimental results show that a 1:2 volume ratio significantly reduces the iodine number, from 57.22 to 47.38 g I_2/100 g sample, indicating a decrease in unsaturated compounds. Furthermore, oxidative stability improved from 11.18 hours to 11.69 hours after extraction. The removal of PUFAs not only improves the fuel's storage and combustion stability but also enhances its environmental profile. More stable biodiesel burns more completely, reducing emissions of particulate matter and greenhouse gases, and contributing to cleaner air and lower environmental impact. Thus, PUFA extraction using $AgNO_3$ presents a promising approach for improving the sustainability and performance of palm-based biodiesel fuels.

1. Introduction

Biodiesel is a renewable fuel composed primarily of fatty acid methyl esters (FAME). These fatty acids are classified into saturated fatty acids (SFA) and unsaturated fatty acids, which include monounsaturated fatty acids (MUFA) and polyunsaturated fatty acids (PUFA) [1]. Biodiesel with a high SFA content exhibits good oxidative stability and a

high cetane number. However, its high cloud point can lead to the formation of solid crystals at relatively low temperatures, potentially clogging fuel lines in engines.

While unsaturated fatty acids (UFA) contribute to a lower cloud point, the presence of PUFA, particularly methyl linolenate, significantly reduces oxidative stability. Biodiesel rich in PUFA is more susceptible to degradation, leading to increased acid values, water content, and the formation of sediments or sludge over time. These degradation products not only affect the efficiency and lifespan of engines but also contribute to environmental pollution through incomplete combustion and increased emissions of unburned hydrocarbons and particulate matter [2].

Biodiesel is compatible with compression-ignition (diesel) engines [3], and in many countries, it is blended with conventional petroleum diesel to reduce reliance on fossil fuels [4]. In tropical countries like Indonesia, national policy currently allows for a biodiesel blending rate of up to 30% (B30) [5].

From an environmental perspective, improving the quality of biodiesel is essential for maximizing its sustainability benefits [6]. Biodiesel is often promoted as a green alternative to fossil fuels due to its potential to reduce greenhouse gas (GHG) emissions, particularly carbon dioxide (CO_2), over its life cycle [7]. However, poor oxidative stability can negate these benefits by decreasing engine efficiency and increasing maintenance needs.

Although palm-based biodiesel is among the most widely used, it still has limitations [8]. Compared to pure Petro diesel (B0), palm biodiesel has significantly lower oxidative stability. This is largely due to the presence of oxygen in its molecular structure, which makes it susceptible to oxidative degradation, especially when the PUFA content is high. This degradation process further reduces the fuel's oxidative stability [9].

Improving oxidative stability by removing PUFA is essential not only for enhancing biodiesel's storage and combustion characteristics but also for supporting environmental sustainability goals. Biodiesel with improved oxidative stability burns more completely, producing fewer emissions and reducing environmental pollution. One effective method to remove PUFA involves extraction using silver nitrate ($AgNO_3$) solution [10]. However, previous studies have not thoroughly explored the optimal ratio of palm biodiesel to $AgNO_3$ solution for this extraction process. Our earlier research indicated that the most effective biodiesel-to-$AgNO_3$ ratio for removing polyunsaturated free fatty acids is 1:2, resulting in an iodine value of 47.38 g I_2/100 g oil [2].

Therefore, this study aims to determine the most effective volume ratio of $AgNO_3$ solution to biodiesel for separating saturated from unsaturated fatty acid fractions. The ultimate goal is to produce palm biodiesel with a lower iodine number, enhanced oxidative stability, and improved environmental performance.

2. Materials and Methods

Palm biodiesel was first obtained from PT Wilmar Bioenergy Indonesia. $AgNO_3$ is purchased from Merck (analytical grade), which is used to provide an $AgNO_3$ solution. The extraction process is carried out $AgNO_3$ solution. The variation of the ratio of biodiesel fuel volume with $AgNO_3$ solution studied in this study is 2:1, 1, 2, and 1:2.5. The raw material preparation stage consists of desiccating an indicator of MgSO4, using the FAME dehydration process (biodiesel), and manufacturing $AgNO_3$ solvent reagents.

During biodiesel extraction, Ag^+ ions form complexes with the double bonds in unsaturated fatty acids. This reaction reaches equilibrium in about three hours. Once equilibrium is achieved, the saturated fatty acid phase is separated from the aqueous phase containing the Ag^+-unsaturated complex. After separation, the methyl esters undergo washing and purification.

The procedure for analyzing iodine numbers using the Wijs reagent follows the standard procedure of AOCS Cd 1-25 [11], FAME composition analysis using GC-MS. Samples were diluted 20 times with hexane. The GC-MS used is the 2010 Shimadzu brand equipped with RTX-5 MS columns with dimensions of 30m x 0.25mm x 0.25µm with AOC-20i auto injector. The measurement method is as follows: as much as one microliter of the sample is injected into the GC with a split ratio of 1:100. The injector temperature is 250 °C. The oven temperature of the GC is programmed as follows: initially 40 °C, ramp 5 °C/min to 300 °C.

Oxidative stability was obtained according to the Rancimat method [12]. Around 3 g of the sample was placed in a test tube, bubbling synthetic air (10 Lh^{-1}, Linde) and heating the tube at 100 °C. After oxidizing the sample, the resulting steam passes through 50 mL of deionized water. The conductivity of this amount of water was measured. As the sample was oxidized, some products were developed, dissolving in water and increasing their conductivity. A Conductivity meter recorded conductivity (Crison EC-Meter GLP31+, Spain). Oxidative stability is expressed in hours, so the induction point should be calculated for this purpose.

Table 1. The original biodiesel composition (from PT. Wilmar Bioenergy Indonesia).

No	Fatty Acid	%-weight
1	Lauric Acid (C12:0)	0.68
2	Myristic Acid (C14:0)	2.15
3	Palmitic acid (C16:0)	39.17
4	Stearic Acid (C18:0)	7.18
5	Oleic Acid (C18:1)	42.41
6	Linoleic Acid (18:2)	5.87
7	Others	2.54
	Total	100

Table 2. The composition of saturated and unsaturated fatty acid fractions in the original biodiesel (from PT. Wilmar Bioenergy Indonesia).

No	Raw Biodiesel	%-weight
1	Saturated FAME	49.18
2	Monounsaturated FAME	42.41
3	Polyunsaturated FAME	5.87
4	Others	2.54
	Total	100

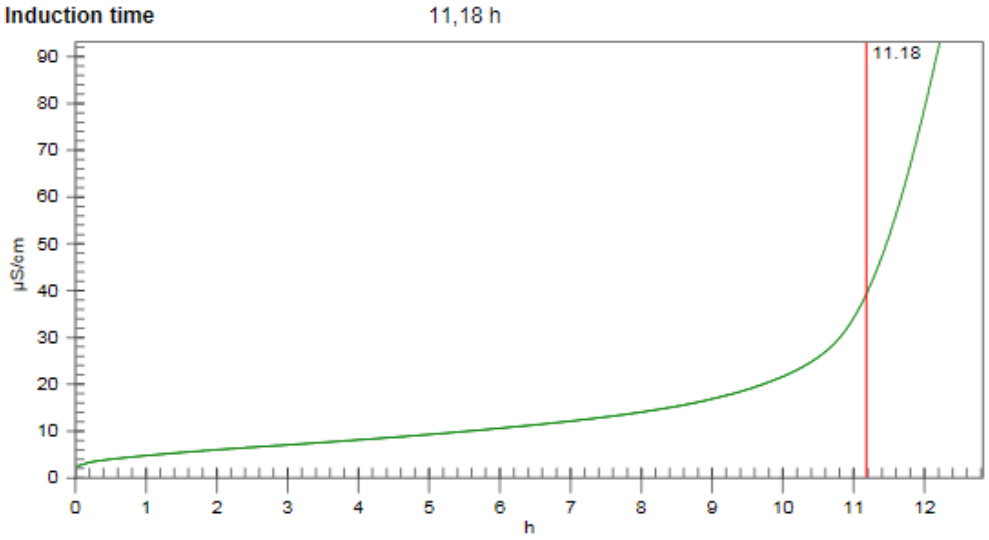

Figure 1. Graph of the oxidation stability of palm oil FAME (from Wilmar Bioenergy Indonesia Product).

3. Results and Discussion

3.1. Characterization Results FAME Raw material from Palm Oil

Fractionation stages about using biodiesel obtained from PT Wilmar as a sample of experimental raw materials. Analysis of the composition of FAME from palm oil used as raw material for this study is presented in Tables 1 and 2.

The fatty acid chains of palm oil (CPO) based biodiesel samples mainly contain C18 carbon atoms (C18:1 and C18:0). The content of linoleic (di-) unsaturated fatty acid, which is the most reactive to trigger oxidation reaction, was detected in insignificant amounts. Oxidative stability has been reported not to correlate with the total number of double bonds, but with the total number of bis-allylic sites (contained by polyunsaturated fatty acids) [13].

Tables 1 and 2 show that the biodiesel used as raw material for this study had a polyunsaturated fatty acid component of around 6% by weight. Meanwhile, the monounsaturated fatty acid component is around 42%. Cumulatively, the components of saturated fatty acids are relatively the same as those of unsaturated fatty acids. With the composition shown in Tables 1 and 2, the FAME iodine number (IN) obtained from palm oil is 57.22 g I_2/100 g sample, and oxidation stability as shown in Figure 1.

3.2 Results of Biodiesel Ratio to $AgNO_3$ Solution

The composition and iodine number (IN) of FAME are two main parameters used as a reference for detecting the

(a) (b) (c)

Figure 2. $MgSO_4$ (a), Wet $MgSO_4$ desiccant (b), Dry $MgSO_4$ desiccant (c).

Table 3. Biodiesel data before and after absorption.

Parameter	Initial Weight (gr)	Final Weight (gr)
Biodiesel	240	236.2

effectiveness of $AgNO_3$ absorbents against unsaturated fatty acid fractions.

3.2.1. Biodiesel Dehydration with $MgSO_4$

The stripping of water molecules from the biodiesel sample was carried out using $MgSO_4$, which had gone through a series of special experiments to maximize the absorption capacity of the surrounding water molecules. The $MgSO_4$ desiccant is white, as shown in Figure 2. The $MgSO_4$ (dry) was placed in a desiccator to absorb water from the biodiesel.

The results of the biodiesel dehydration experiment with $MgSO_4$ desiccant showed that the water absorption of biodiesel was around 1.58% by weight. Table 3 presents the data on the reduction in sample mass before and after being subjected to the absorbent. The difference in the initial and final biodiesel weights is calculated as the mass of water molecules released from the initial biodiesel.

3.2.2. Biodiesel Extraction Using $AgNO_3$

The method applied for this biodiesel fractionation is the extraction method using $AgNO_3$ solvent. The separation progress with the above method is determined based on the iodine number (IN) of the saturated and unsaturated biodiesel obtained. The expectation at this fractionation stage is to obtain saturated biodiesel with an IN range of 30-40 and unsaturated biodiesel with IN> 70. Three series of biodiesel extraction experiments have been conducted with different volume ratios of biodiesel to $AgNO_3$. These three series of experiments are part of an effort to find the most appropriate volume ratio of biodiesel: $AgNO_3$ to produce biodiesel grouping with IN that meets the above expectations.

Table 4 presents the results of GC-MS analysis of the composition of feed and FAME products after extraction with several variations of the volume of biodiesel and AgNO3 solutions.

The data in Table 4 show the progress of the separation of saturated and unsaturated biodiesel by the extraction method using $AgNO_3$ solvent. In three series of extraction experiments, there seems to be a significant increase in saturated fatty acids C16, along with a decrease in monounsaturated fatty acids (C18:1). This phenomenon indicates that the extraction process using silver nitrate solvent is going quite well, although not yet at maximum, because some unsaturated fatty acids are combined with the product claimed as saturated fatty acids after separation.

Table 4 also shows that under 1:2 ratios of the FAME and solvent of $AgNO_3$, 100% of C18:2 in methyl palm could be reduced to 0%. The result proves that methyl linolenic (C18: 2) adsorbs most strongly on the AgNO3 in a 1:2 ratio of FAME with $AgNO_3$ solvent. Ghebreyessus et al. [13] have reported that Methyl linolenate (18:3) adsorbs most strongly, followed by methyl linoleate (18:2), on the $AgNO_3/SiO_2$. The achievement of C18: 2 to 100% depletion results in enrichment in the C18: 1 fraction of about 2.41% and the C16: 0 fraction of about 3.31% by weight. So, about 2.41% of C18:2 is converted to C18:1 by breaking one of the bonds (ligands) in the double bond position, and the simultaneous capture of hydrogen is thought to originate from water molecules bound to the $AgNO_3$ solvent. While the phenomenon of increasing about 3.31% by weight of C16: 0 is likely to be partly C18: 2 because after undergoing the process of hydrogenation of a double bond, a breakdown of the molecular chain

Table 4. Comparison of raw biodiesel composition with biodiesel extraction products.

No	Fatty acids	%-weight			
		Raw biodiesel	Saturated Biodiesel		
			Ratio 2:1	Ratio 1:2	Ratio 1:2.5
1	Lauric Acid (C12:0)	0.68	1.22	0.73	0.75
2	Myristic Acid (C14:0)	2.15	3.47	2.34	2.23
3	Palmitic Acid (C16:0)	39.17	42.73	42.48	41.37
4	Linoleic Acid (C18:2)	5.87	4.27	0	0.03
5	Oleic Acid (C18:1)	42.41	38.4	44.82	41.48
6	Stearic Acid (C: 18:0)	7.18	6.42	7.19	7.25
7	Others	2.54	3.49	2.44	6.89
	Total	100	100	100	100

Table 5. Composition of saturated and unsaturated biodiesel after extraction.

No.	Volume Ratio of Biodiesel to $AgNO_3$ Solution	Composition (%-area)			Total
		Saturated Biodiesel	Unsaturated Biodiesel	Others	
1	Raw biodiesel	23.31	70.7	5.99	100
2	2:1	53.84	42.67	3.49	100
3	1:2	52.74	44.82	2.44	100
4	1:2.5	51.6	41.51	6.89	100

structure of fatty acid esters takes place to form a short-chain saturated fatty acid ester (C16: 0). This phenomenon has been reported by Pullen and Saeed [14] that in some cases, oxidation results in the chemical structure of biodiesel breaking apart to form shorter chain acids and aldehydes.

Table 5 presents further analysis of the extracted saturated biodiesel, using GC-MS chromatogram data to classify the total saturated and unsaturated biodiesel fractions.

The data in Table 5 show that the total fraction of saturated biodiesel derived from the initial biodiesel extraction product with silver nitrate solvent is in the range of 51 - 54%, while the total unsaturated biodiesel is in the range of 41 - 45%. Thus, it can be observed that the total saturated biodiesel from the extraction product has increased significantly compared to the initial biodiesel. Conversely, the total unsaturated biodiesel from the extraction products have decreased significantly compared to the initial biodiesel.

The results of the initial iodine measurement for biodiesel were 57.22 g I_2/100 g of the sample. At the same time, biodiesel products after extraction are 47.38 g I_2/100 g of samples. There was a decrease in the initial iodine biodiesel number of around 9.84 points or 17.2%. These results indicate that there has been a reduction in unsaturated fatty acids followed by an increase in saturated fatty acids in biodiesel extraction products. Thus, the ratio of 1:2 for the volume of biodiesel with $AgNO_3$ solution is the most effective for depleting the polyunsaturated fatty acid fraction inside biodiesel.

3.3 Determination of Oxidation Stability According to Ratio of Biodiesel Volume: $AgNO_3$ (1 2)

After finding an effective ratio of 1:2 for the volume of biodiesel with $AgNO_3$ solution in the extraction process, oxidation stability is observed and measured on extraction products subject to a 1:2 ratio. Oxidation stability is one of the most important properties of fatty acid alkyl esters (biodiesel fuel) and primarily affects the stability of biodiesel during extended storage [14]. Several series of measurements of oxidation stability have been carried out repeatedly to observe the effects of oxygen during the extraction process, the effects of water molecules, and Ag ion residues in biodiesel products (extraction results) on the stability of oxidation. The measurement results to prove the effects mentioned above on oxidation stability, respectively shown in Figures 3, 4, 5, and 6. Figure 3 shows a graph of the biodiesel test during the extraction process, which is not exposed to nitrogen to expel oxygen. It can be seen in Figure 3 that the oxidation stability of biodiesel products only reached 4.5 hours or decreased significantly, which was 6.68 hours compared to the initial biodiesel. The low oxidation stability proves that biodiesel oxidation degradation occurred during extraction. Thus, the presence of oxygen in the extractor chamber during the extraction process has led to contact between oxygen and biodiesel and changed the resistance of biodiesel fuels to the degradation process.

Figure 4 shows that the presence of water molecules in the extraction process chamber has contributed to the degradation of biodiesel fuels. Compared to the original biodiesel (which has an oxidation stability of 11.18 hours), there has been a decrease in the value of oxidation

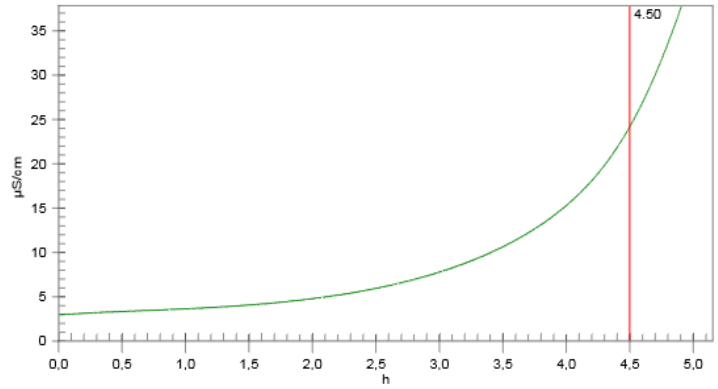

Figure 3. Effects of oxygen on the oxidation stability of biodiesel.

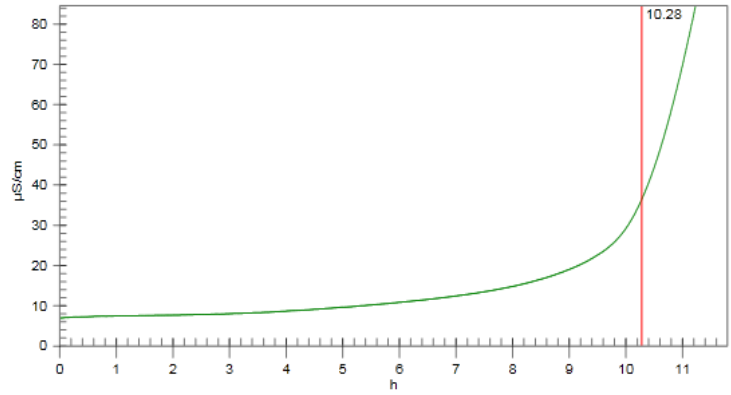

Figure 4. Effect of water molecules on the oxidation stability of biodiesel.

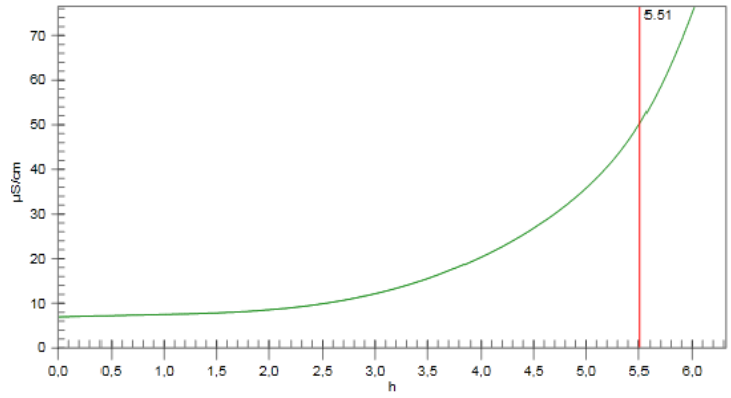

Figure 5. Effect of Ag ion residues on biodiesel oxidation stability.

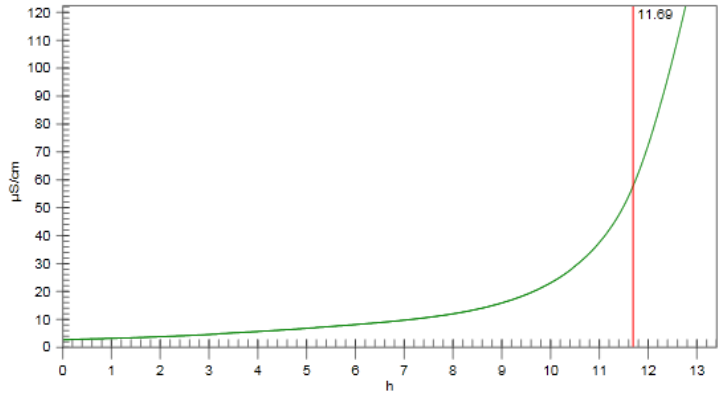

Figure 6. Graph of the stability of the oxidation of biodiesel extraction products after demetalization and dehydration.

stability of about 0.90 hours on biodiesel fuel contaminated with water. The contact between biodiesel and water molecules causes the hydrolytic oxidation process [15] in biodiesel, which decreases oxidation stability. According to Pullen and Saeed [14], hydrolysis from contact with water or moisture in tanks and fuel lines can degrade biodiesel fuel properties.

Figure 5 shows that biodiesel contamination by Ag ion residue, which is still left behind due to the imperfect leaching process, has a significant degradation effect on biodiesel fuel. The effect was observed in the decreased oxidation stability of biodiesel fuel by 5.67 hours compared to the original biodiesel.

Figure 6 shows that the biodiesel fuel product, which is extracted with nitrogen during the extraction process, is completely washed until all Ag ions are completely drained and completely dried, giving a higher oxidation stability (0.51 hours) than the initial biodiesel. These results prove that when the whole series of production processes runs well, biodiesel fuel without the fraction of polyunsaturated fatty acids is more stable than biodiesel that still contains polyunsaturated fatty acids. The high oxidation stability also shows that the ratio of 1:2 to the volume of biodiesel with $AgNO_3$ solution is most effective for depleting polyunsaturated fatty acids during the initial biodiesel extraction process.

4. Conclusions

The removal of polyunsaturated fatty acids from palm biodiesel can be effectively achieved through extraction using a silver nitrate ($AgNO_3$) solution. This study found that the optimal volume ratio of biodiesel to $AgNO_3$ for depleting the C18:2 (linoleic acid) fraction is 1:2. Eliminating the C18:2 fraction led to a relative increase in the concentrations of C18:1 (oleic acid) and C16:0 (palmitic acid), both of which contribute to improved oxidative stability. As a result, the iodine value of the biodiesel was significantly reduced, indicating a lower degree of unsaturation. This reduction enhances fuel stability and minimizes the risk of oxidation-related degradation. Biodiesel with reduced C18:2 content demonstrates better storage characteristics and combustion efficiency, ultimately supporting lower emissions and a more environmentally sustainable alternative to conventional diesel fuels.

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