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Development of Analytical Method for Determination of Palm-Based Hydrotreated Vegetable Oil (HVO) in Diesel Blends Using Gas Chromatography: Preliminary Study

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ABSTRACT - Hydrotreated vegetable oil (HVO) is a renewable paraffinic biofuel derived from the catalytic hydrotreatment of triglycerides, particularly from palm oil. Because HVO's hydrocarbon structure is so similar to petroleum diesel, it can be mixed directly with regular diesel fuel. However, this structural similarity makes it difficult to accurately quantify HVO in diesel blends, which is essential for maintaining fuel quality and complying with regulations. In this study, gas chromatography–mass spectrometry (GC-MS) is used to identify the compounds, and gas chromatography–flame ionization detection (GC-FID) is developed to quantify biofuel HVO. The chromatographic profiles of diesel, HVO, and biodiesel display distinct hydrocarbon distributions. Two diagnostic peaks at retention times of 17.5–17.7 minutes, identified as heptadecane and 2,6,10,14-tetramethylpentadecane (pristane), are used for preliminary identification. The heptadecane peak, present in both diesel and HVO, is selected as a quantifier, while the heptadecane-to-pristane ratio of 1.25 in diesel is applied as a correction factor. The accuracy of the method is confirmed by a strong linear correlation ($R^2 = 0.9991$) for HVO concentrations ranging from 0 to 40% v/v. Recovery rates ranging from 97.0% to 102.2% further illustrate the reliability of the method for routinely analyzing the amount of HVO in diesel blends.

Keywords: Hydrotreated Vegetable Oil (HVO), diesel blend, gas chromatography, biofuel quantification, renewable fuel.

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INTRODUCTION

Indonesia has long held a leading role in the global palm oil industry, producing 51.58 million tons in 2020 alone. The commodity now makes up over 14% of the country's non-oil exports. This consistent increase suggests that demand is rising and that palm oil is becoming more and more important in a variety of industries, most notably as a raw material for biofuels. Fatty Acid Methyl Ester (FAME), also known as biodiesel, is produced in Indonesia from palm oil and is a popular renewable substitute for fossil diesel. (Aisyah & Wibowo, 2011; Silitonga et al., 2011). In general, FAME is biodegradable, non-toxic, sulfur-free, and typically has a higher cetane number than fossil diesel, making it an environmentally favorable fuel (Aisyah et al., 2016; Marchetti & Errazu, 2008).

Palm oil, in addition to being converted into FAME, also holds potential for transformation into various other biofuels through processes such as hydrogenation and co-processing (Holmgren et al., 2007). In particular, catalytic cracking routes have also demonstrated potential for producing hydrocarbon-rich biofuels from palm oil (Singh et al., 2015). Palm oil processed by the hydrogenation method can produce a fraction of hydrocarbons, equivalent to diesel oil, known as Hydrotreated Vegetable Oil (HVO) (Zikri et al., 2020). The hydrogenation process conducted with the stand-alone refinery began with the saturation of feedstock oil with hydrogen gas at high temperatures (>300°C), followed by the isomerisation and cracking processes to achieve the final product quality (ÉcoRessources Consultants, 2012). Unlike FAME, HVO consists primarily of paraffinic hydrocarbons, making it chemically similar to diesel fuel and offering superior combustion characteristics, lower emissions, and better storage stability (Chaudhuri, 2011; Szeto & Leung, 2022).

The similarity of carbon chains in HVO to those in diesel fuel has more potential as a mixture with diesel fuel compared to FAME (Orozco et al., 2017). These benefits have made HVO a promising alternative biofuel that has been incorporated into diesel as part of energy transition initiatives. However, precisely measuring the amount of HVO in diesel blends is a significant analytical challenge due to the chemical similarity between HVO and diesel.

According to Ruszkowski et al. (2014), measurement of HVO percentage in diesel could become very important. However, existing analytical methods have limitations in terms of accuracy, practicality, or differentiation between bio-components. The existence of the measurement methods was established based on the C-14 isotope activity. Several methods have been developed and simplified, such as Liquid Scintillation Counting (LSC) and Accelerator Mass Spectrometry (AMS), in accordance with American Society for Testing and Materials (ASTM) D6866. However, the latter method has several deficiencies, including requiring a long time, being expensive, and having limited practicality. Besides, the method could not differentiate between the two bio components in the diesel fuel mixture (Alves, 2021; ASTM D6866, 2018; Berhanu et al., 2017; Bronić et al., 2017; Krištof & Logar, 2013; Norton & Woodruff, 2012; Oinonen et al., 2010; Varga et al., 2018).

The utilization of combined infrared spectrometry with chemometrics has been used to predict FAME concentrations and HVO in the mixture of diesel fuel included predicts the biodiesel's physical characteristics (Alves & Poppi, 2013a, 2013b, 2016; ASTM D7371, 2014; Balabin & Safieva, 2011; Baptista et al., 2008; Bellussi et al., 2016; Câmara et al., 2017; Cunha et al., 2020; EN14078, 2014; Fernandes et al., 2011; Palou et al., 2017; Rocha et al., 2012; Rohman et al., 2020; Velvarská et al., 2019; Vrtiška & Šimáček, 2016; Wikberg et al., 2021). The use of the infrared spectrometry method has limitations because it requires pre-processing modelling with the corresponding matrices; otherwise, it cannot be conducted, and the prediction of the results will not be accurate or may be incorrect. In comparison, gas chromatography-mass spectrometry (GC-MS) provides a high-resolution separation and molecular identification method for analyzing the composition of diesel fuel, biodiesel, and HVO (Baldauf et al., 2016; Cozendey et al., 2025; Desrina, 2010; Hariram et al., 2016; Sonthalia, 2019).

A standardized, reliable, and efficient test method for determining HVO content in diesel mixtures is still lacking. This study aims to develop and evaluate a gas chromatography-flame ionization detection (GC-FID) method for quantifying HVO in diesel blends, with GC-MS used for structural confirmation. This technique seeks to create a precise, practical, and repeatable methodology in order to bridge a gap in biofuel quality control and regulatory compliance by identifying significant hydrocarbon markers.

METHODOLOGY

Materials and sample preparation

Diesel fuel (cetane number 48), FAME, and palm-based hydrotreated vegetable oil (HVO) were obtained from The Testing Center for Oil and Gas "LEMIGAS", The Ministry of Energy and Mineral Resources, Indonesia. These samples were used to prepare diesel blends with HVO concentrations ranging from 0 to 40% v/v. Additionally, diesel blends with 0 to 40% v/v FAME were used to calculate the key peak ratio. All solvents and reagents used in the study were of analytical grade and obtained from Merck KGaA, Darmstadt, Germany.

Gas chromatography-flame ionization detection (GC-FID) analysis

GC-FID analysis was performed using an Agilent Technologies 7820A GC system (Santa Clara, CA, USA) equipped with a 5%-phenyl-methylpolysiloxane capillary column (30 m × 0.25 mm i.d., 0.25 µm film thickness, Agilent J&W Scientific, USA). Sample injection was conducted in split mode (split ratio 250:1, injection volume 1.0 µL) at an injector temperature of 280°C. The column oven temperature was programmed as follows: an initial hold at 60°C for 3 minutes, followed by a ramp to 300°C at a rate of 10°C/min, and then held for 5 minutes. Helium (99.999%) was used as the carrier gas at a constant flow rate of 1.0 mL/min. The FID detector was maintained at 300°C.

Gas chromatography-mass spectrometry (GC-MS) analysis

GC-MS analysis was performed using an Agilent 7890B GC system coupled with an Agilent 5977A mass spectrometer (Santa Clara, CA, USA). The same capillary column and temperature program were applied as in GC-FID analysis. The ionization source was operated at electron impact (EI) mode (70 eV), with an ion source temperature of 230°C and a transfer line temperature of 300°C. Data acquisition was conducted in full-scan mode (m/z 50–550). Mass spectral data were compared with the NIST17 MS database in order to identify the compound.

Quantification and calibration

Heptadecane ($C_{17}H_{36}$) and 2,6,10,14-tetramethylpentadecane, also known as pristane, were selected as key marker compounds for HVO quantification. A calibration curve was constructed using HVO-diesel blends at 5%, 10%, 15%, 25%, and 40% v/v HVO, with the heptadecane peak areas corrected using a heptadecane-to-pristane ratio of 1.25.

Linear regression analysis was applied to correlate corrected heptadecane peak areas with HVO concentration, and the method's accuracy was validated using recovery tests.

Statistical analysis

Linear regression analysis is a statistical method that can be conducted using Microsoft Excel. Data are expressed as mean ± 1.96 standard deviation, and the level of significance was set at 0.05. The method's results are reported with a 95% confidence level by following equation (1). Regression models were assessed with 95% CIs to determine the precision of estimates.

$$\mathbf{95\%\ C.I.\ of\ calculated\ y' =}$$

$$t_{n-2}S_{y|x}\sqrt{\left(\frac{1}{n}+\frac{(X-\bar{x})^2}{(n-1)S_x^2}\right)}\ ;\ \bar{x}=\frac{\sum x_i}{n} \qquad (1)$$

where: predicted value of y at x using 95% confidence level, is critical value from t-distribution with n-2 degrees of freedom, while is standard error of the estimate, n is number of sample, is mean of the x values, is sample variance of the x values, and X is specific valuef of x for which the prediction is made.

RESULT AND DISCUSSION

Figure 1 illustrates the processes of hydrogenation, decarboxylation, and hydrodeoxygenation that transform triglycerides into hydrocarbon products, like HVO, as explained by Sotelo-Boyas et al. (2011). This process creates free fatty acids by introducing hydrogen to long-chain fatty acids, which breaks down ester bonds. These fatty acids then undergo decarboxylation, removing CO_2, or decarbonylation, removing CO, to form shorter hydrocarbons. During the hydrodeoxygenation stage, oxygen atoms are removed as water (H_2O), resulting in the formation of saturated hydrocarbons.

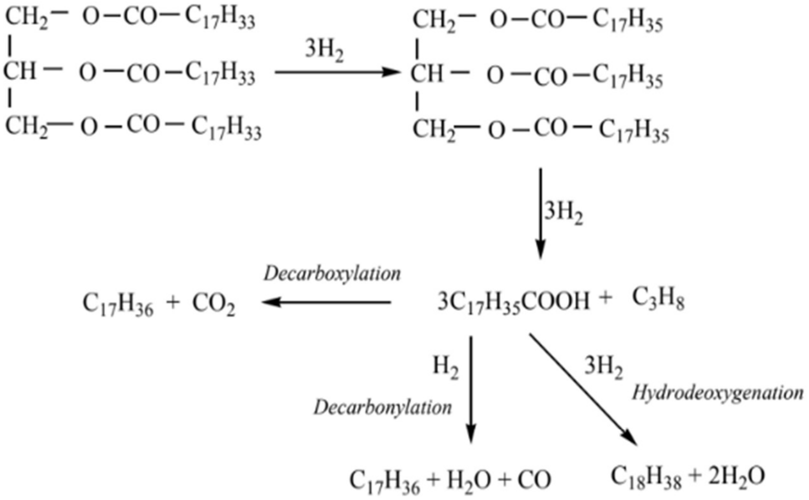

Figure 1. The conversion of fatty acid to HVO by the hydrogenation process.

Palm-based oil predominantly contains palmitic acid ($C_{16}H_{32}O_2$), oleic acid ($C_{18}H_{34}O_2$), stearic acid ($C_{18}H_{36}O_2$), and myristic acid ($C_{14}H_{28}O_2$) as its main components. These feedstocks lead to the production of HVO, which consists primarily of saturated hydrocarbons, such as heptadecane ($C_{17}H_{36}$), octadecane ($C_{18}H_{38}$), hexadecane ($C_{16}H_{34}$), and pentadecane ($C_{15}H_{32}$). Specifically, octadecane and heptadecane are formed through the hydrogenation of oleic acid and stearic acid, while hexadecane and pentadecane result from the hydrogenation of palmitic acid.

Figure 2 presents the chromatograms of diesel fuel, HVO, and blended diesel fuel obtained by mixing 40% v/v HVO into diesel fuel. Among the three chromatograms, diesel exhibits a complex profile with numerous peaks, indicating a wide variety of hydrocarbons, including aromatics, paraffins, and sulfur-containing compounds. Nevertheless, HVO's chromatogram has fewer, sharper peaks than that of diesel or blended diesel, suggesting that its composition is simpler and mostly composed of paraffinic hydrocarbons with little aromatics and contaminants. The chromatogram of blended diesel shows a hybrid profile that blends elements of HVO and diesel. The blend's heterogeneous chemical composition is reflected in the visible peaks from both fuels. Which hydrocarbons are more noticeable in particular samples can be determined by differences in peak intensities and retention times. Finding the primary peaks is crucial for figuring out the mixture's composition.

Determined by GC-MS, the chromatogram of HVO reveals four major components: peak a corresponds to pentadecane, peak b to hexadecane, peak c to heptadecane, and peak e to octadecane. Those peaks confirm that the HVO samples used in this research are sourced from palm oil. These peaks also appear in the diesel chromatogram, demonstrating that HVO can be effectively used as a diesel blend.

One potential first step in determining the composition of the diesel fuel mixture would be to concentrate on the two peaks with retention around 17.5 to 17.7 minutes. These peaks, labeled as peak c and peak d in Figure 2, provide valuable insight. Notably, peak d does not show up in the HVO chromatogram, making it specific to diesel and its blends. The close retention times of these two peaks suggest that they are composed of chemically similar compounds. In general, quantifying the two peaks using an internal standard may be necessary to introduce additional internal standards, as they are specific to different analytes, to improve accuracy. In this research, we found that the two key peaks with nearby retention times can be helpful for quantification without the need for an additional internal standard.

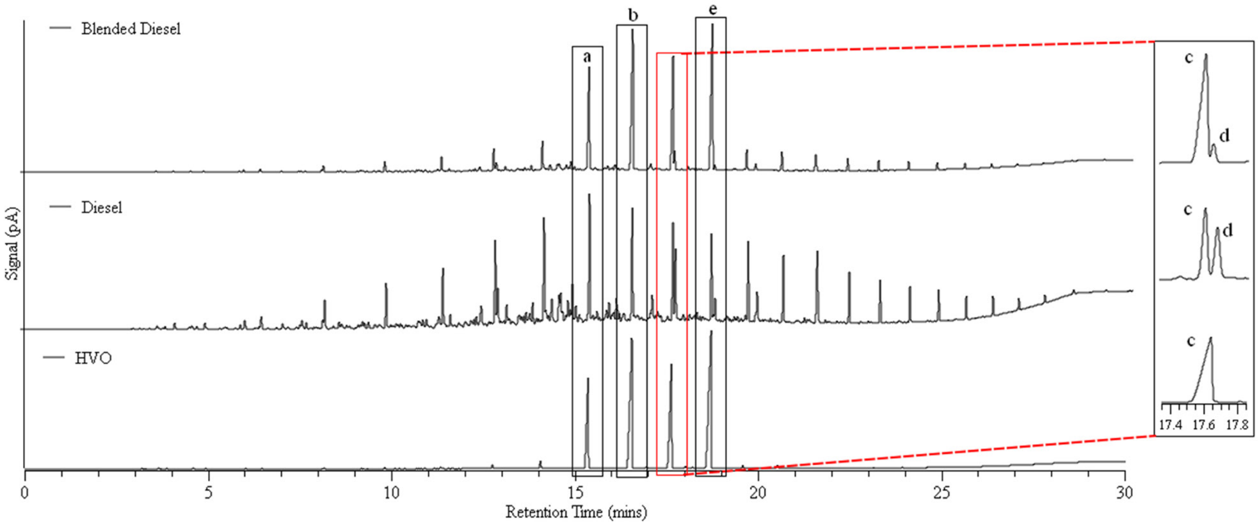

Figure 2. Chromatograms of HVO, diesel fuel, and blended diesel fuel.

Figure 3 presents the mass spectral analysis of peak c, which is identified as heptadecane ($C_{17}H_{36}$) with a molecular ion peak at m/z 240.27 and followed by a fragment at m/z 197.21, corresponding to $C_{14}H_{30}$, resulting from the cleavage of a $C_3H_7^+$ (m/z 43.10) fragment. Additional peaks are observed due to the sequential expulsion of CH_2 units from C_{14} to C_3.

Figure 4 presents the mass spectral analysis of peak d, which is identified as 2,6,10,14-tetramethylpentadecane, known as pristane, with a molecular ion peak at m/z 268.52 and followed by a fragment at m/z 196.09, corresponding to $C_{14}H_{29}^+$, resulting from the cleavage of a $C_5H_{11}^+$ (m/z 71.07) fragment. Furthermore, the cleavage of a CH group is responsible for a peak at m/z 183.16 that corresponds to $C_{13}H_{28}^+$. The sequential expulsion of CH_2 units from C_{13} to C_3 is the cause of the other peaks that are seen. These results verify that the two mass spectra for pristane and heptadecane match those in the NIST database.

In the chromatogram of blended diesel, heptadecane and pristane are present. The heptadecane peak originates from both diesel and HVO. As the HVO volume increases in the blended diesel, the heptadecane peak intensifies, while the portion of heptadecane derived from diesel decreases. Logically, heptadecane and pristane sourced from diesel, should maintain the same ratio, even when the diesel is diluted. To verify this, we prepared a mixture of diesel and FAME, which is already used as a diesel blend in Indonesia. Since FAME has a different molecular structure from diesel, its chromatographic peaks do not interfere with those of diesel. The chromatogram of FAME, shown in Figure 5 and identified by GC-MS, reveals four major peaks: peak W corresponding to methyl tetradecanoate, peak X to hexadecenoic acid, peak Y to 9-octadecanoic acid, and peak Z to methyl stearate. These compounds are methyl ester derivatives of myristic acid, palmitic acid, oleic acid, and stearic acid, confirming the primary components of biodiesel derived from palm-based feedstock. Notably, there are no detectable peaks for heptadecane or pristane. This FAME's peak indicate that the two peaks observed within the retention time range of 17.5–17.7 minutes are exclusive to diesel.

By varying the volume of FAME in diesel fuel within the range of 0 to 40%, the peak areas of heptadecane and pristane were determined, as shown in Table 1.

Table 1. Measured peak area and ratio

%v/v FAME	Area (pA*s)		Ratio
	Heptadecane	Pristane	
0	1,169.78	941.98	1.24
10	1,092.63	871.18	1.25
20	976.74	777.98	1.26
30	840.01	672.28	1.25
40	717.80	573.36	1.25
	Mean		1.25 ±0.01

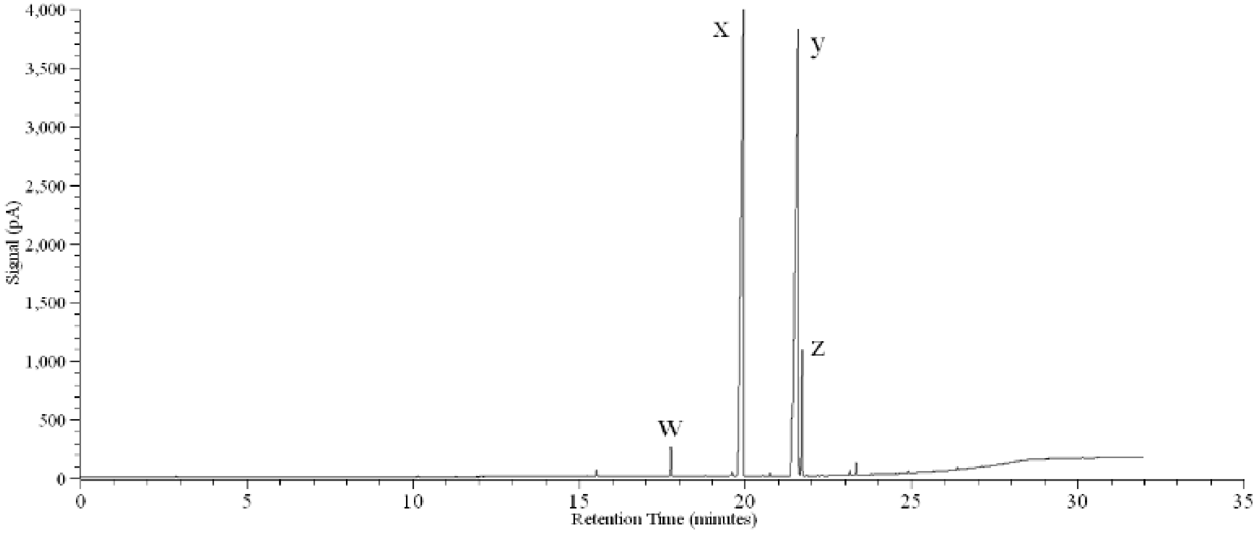

Figure 5. Chromatogram of FAME

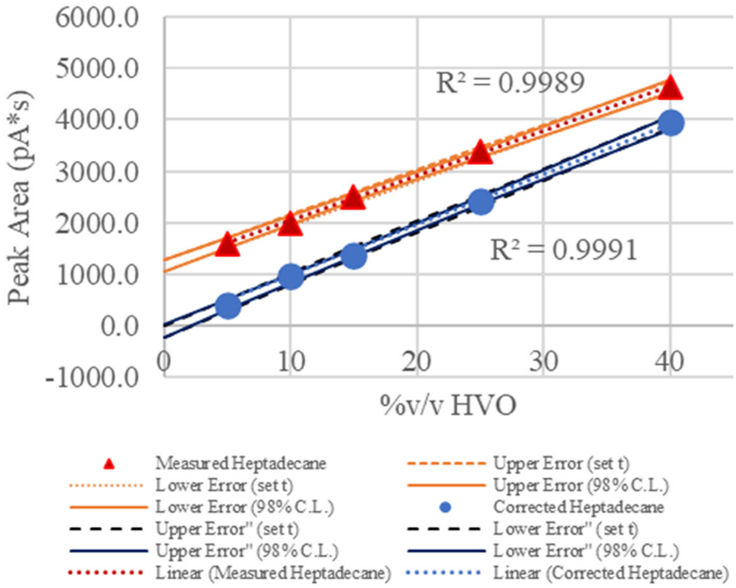

Figure 6. Calibration curve of measured and corrected heptadecane peak area to %HVO

The calculations confirm that the ratio of heptadecane to pristane in diluted diesel remains consistent, as predicted. A ratio of 1.25 is established to calculate the corrected heptadecane originating from HVO in blended diesel with HVO variations ranging from 5 to 40%v/v, as shown in Table 2.

Table 2. Measured and corrected peak area

%v/v HVO	Area (pA*s)		
	Heptadecane	**Pristane**	**Corrected Heptadecane**
5	1602.5	962.7	399.1
10	1999.2	820.0	974.2
15	2512.9	915.0	1369.1
25	3399.8	790.7	2411.4
40	4625.9	525.2	3969.5

Calibration curves are plotted using both measured heptadecane and corrected heptadecane against the %v/v HVO in the mixture, as shown in Figure 6. The graph is presented with a 95% confidence level to show accuracy. The HVO-diesel blends exhibit excellent linear regression with corrected heptadecane peak area showing an R^2 value of 0.9991, while the R^2 measured heptadecane has an R^2 value of 0.9989. The higher R^2 value from the corrected heptadecane graph explains the greater variance in the data, as its mean heptadecane peak is linearly related to having a higher HVO-sourced heptadecane content than diesel-sourced heptadecane in the blend. curve of measured and corrected heptadecane peak area to %HVO

The regression is used to calculate the recovery of a simulated sample containing 30 %v/v HVO in a diesel blend. The simulated sample result is 29.28 ± 0.99, obtained using the measured heptadecane graph, with a recovery range of 94.3 –100.9%. A better narrow result of the recovery range, 97.0 – 102.2%, is obtained using the corrected heptadecane graph, whereas the result for the simulated sample is 29.77 ± 0.90. In conclusion, this method demonstrates high potential for determining HVO palm-based diesel blend. For the routine determination of HVO content, this GC-FID-based method offers a substantially faster, more economical, and operationally simpler approach than radiocarbon-based techniques like AMS and LSC. Additionally, it does not require costly equipment or complicated sample preparation, which makes it especially appropriate for extensive laboratory applications in Indonesia. Also, to further verify its applicability, it is necessary to test varying HVO and diesel sources to confirm the robustness of this method.

CONCLUSION

The range of HVO in blended diesel (0–40% v/v) exhibits excellent linear regression with the corrected heptadecane peak area, yielding an R^2 value of 0.9991. The percentage of HVO can be calculated based on the difference in percentage volume (v/v) of diesel in the total mixture. Since this method is significantly faster, cost-effective, and operationally simpler, and is applicable for determining HVO in diesel mixtures, it is recommended that the next step in research focuses on optimizing the method and evaluating its analytical performance, including variations in diesel and HVO sources. Furthermore, this method has high potential to be implemented as an Indonesian National Standard, fulfilling a critical gap in biofuel quality control for regulatory purposes.

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GLOSSARY OF TERMS

Symbol	Definition	Unit
FAME	Fatty Acid Methyl Ester / biodiesel	
HVO	Hydrotreated Vegetable Oil	
LSC	Liquid Scintillation Counting	
AMS	Accelerator Mass Spectrometry	
ASTM	American Society for Testing and Materials	
GC-MS	Gas Chromatography-Mass Spectrometry	
GC-FID	Gas Chromatography-Flame Ionization Detection	
EI	Electron impact	
Pristane	2,6,10,14-tetramethylpentadecane	
t_{n-2}	Critical value from t-distribution with n-2 degrees of freedom	
$S_{y\|x}$	Standard error of the estimate	
n	Number of samples	
C.I.	Confidence level	%
$\bar{x}$	Mean of the x values	
S_x^2	Sample variance of the x	
X	Specific value of x	
CO_2	Carbon dioxide	
CO	Carbon monoxide	
H_2O	Water	
$C_{16}H_{32}O_2$	Palmitic acid	
$C_{18}H_{34}O_2$	Oleic acid	
$C_{18}H_{36}O_2$	Stearic acid	
$C_{14}H_{28}O_2$	Myristic acid	
$C_{17}H_{36}$	Heptadecane	
$C_{18}H_{38}$	Octadecane	
$C_{16}H_{34}$	Hexadecane	
$C_{15}H_{32}$	Pentadecane	

Symbol	Definition	Unit
$C_{15}H_{32}$	Pentadecane	
%v/v	Percentage volume	
m/z	Mass-to-charge ratio of an ion	
NIST	National Institute of Standards and Technology	

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