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Determination of Hydrotreated Vegetable Oil (HVO) In Blended Diesel Fuel Using Calibration of Isooctane By GC-FID Measurement

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ABSTRACT - Hydrotreated vegetable oil (HVO) is emerging as a promising renewable fuel that is sharing similar chemical characteristics with fossil diesel, making it suitable as a blending component. However, this similarity is presenting challenges in distinguishing and quantifying HVO in diesel blends. The present study is focusing on developing a simple, cost-effective, and reliable method using gas chromatography with flame ionization detection (GC-FID) for determining HVO content in diesel mixtures. Two candidate markers, hexadecane ($C_{16}H_{34}$) and heptadecane ($C_{17}H_{36}$), are being evaluated based on linearity, detection limits, and accuracy. Calibration curves are being constructed using HVO–isooctane mixtures from 0 to 50% v/v HVO. The heptadecane peak is demonstrating superior performance with excellent linearity (R^2 = 0.9994), a low detection limit (1.77% v/v), and quantification limit (5.36% v/v). In contrast, the hexadecane peak is showing similar linearity but lower sensitivity. Accuracy tests are being conducted on diesel samples spiked with 10% HVO, showing recovery rates above 95% for both markers. Overall, heptadecane is proving to be a consistent and reliable marker for quantifying HVO in diesel blends using GC-FID.

Keywords: hydrotreated vegetable oil (HVO), diesel fuel, gas chromatography.

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INTRODUCTION

The growing global commitment to reducing carbon emissions has accelerated the development and adoption of renewable fuels(Azadi et al., 2017). Among these, Hydrotreated Vegetable Oil (HVO) has emerged as a promising alternative to fossil-based diesel. HVO is produced through catalytic hydrogenation of vegetable oils or animal fats, resulting in paraffinic hydrocarbons (mainly C10–C24) free from sulfur, oxygen, and aromatic compounds (Aatola et al., 2008, 2009; Ambaye et al., 2021; Ftturrahman et al., 2020; Kuronen et al., 2007; Vásquez et al., 2017). Its physicochemical properties, including high cetane number, thermal stability, and compatibility with conventional diesel engines, make it suitable for blending with petroleum diesel without requiring engine modifications (Ayu Bethari et al., 2016; d'Ambrosio et al., 2023; Zeman et al., 2019).

Despite its environmental advantages, the analytical quantification of HVO in diesel blends poses a significant challenge due to the compositional similarity between HVO and fossil diesel (Dobrzyńska et al., 2020). Conventional methods such as ASTM D6866, which rely on radiocarbon (^{14}C) analysis, are accurate but expensive and time-consuming (Funabashi et al. 2014; Noor 2020). Alternative techniques like Near-Infrared spectroscopy (NIR) (Alves & Poppi, 2016a; Vrtiška & Šimáček, 2016) and Gas Chromatography-Mass Spectrometry (GC-MS) coupled with multivariate analysis (e.g., Partial Least Squares Regression – PLSR) have demonstrated high accuracy ($R^2 > 0.99$) but often require complex chemometric modeling and robust calibration sets (Wang et al., 2021).

Gas Chromatography with Flame Ionization Detection (GC-FID) remains one of the most widely used techniques for hydrocarbon analysis due to its simplicity, cost-efficiency, and high sensitivity to combustible organic compounds. (Baldauf et al., 2017; de Aguiar et al., 2024; Liu et al., 2011) analyzes nonpolar and polar compounds in ultralow-sulfur diesel (ULSD) samples using two techniques: GC×GC-FID and ESI HR-MS. Chemometric methods were also used to study the storage stability of ULSD. The GC×GC-FID results identified and described the nonpolar compounds, including paraffins, mono- and dinaphthenes, olefins, and aromatics. However, existing GC-FID methods often quantify total hydrocarbon content using the area under a broad carbon range (e.g., C10–C28), without identifying or selecting specific peaks that uniquely represent HVO (U.S. EPA, 2015). This can lead to inaccurate quantification when overlapping signals from diesel components interfere with HVO-specific peaks.

To improve analytical selectivity and reliability, it is essential to identify a major peak as a well-resolved, repeatable chromatographic signal that correlates linearly with HVO concentration and is unaffected by diesel matrix components. The use of a major peak as the quantitative reference can simplify calibration, reduce analysis time, and improve method robustness without the need for internal standards or multivariate modeling.

METHODOLOGY

Materials and sample preparation

The instruments and materials used in this study included an Agilent 7820A gas chromatograph equipped with a flame ionization detector (GC-FID), Indonesian diesel fuel (cetane number 48), and palm oil-based hydrotreated vegetable oil (HVO) sourced from Indonesia.

Quantification and calibration

Calibration standards were prepared by mixing isooctane and HVO at concentrations ranging from 0% to 50% v/v HVO with a total volume of 1.5ml, as shown in Table 1.

For sample analysis, HVO was blended at 10% v/v HVO into six different diesel fuel samples, each with a total volume of 10mL. Before injection, the mixtures were filtered using a 0.45 µm syringe filter and transferred into GC vials for analysis.

Table 1. HVO-Isooctane mixture composition

No	%v/v HVO	Volume Isooctane (ml)	Volume HVO (ml)
1	0	1.5	0
2	5	1.425	0.075
3	10	1.35	0.15
4	20	1.2	0.3
5	30	1.05	0.45
6	40	0.9	0.6
7	50	0.75	0.75

Statistical analysis

The concentration of HVO in the diesel blends was determined using the linear regression equation derived from the calibration curve. The equation used was:

$$X = \frac{(AX - A0) - b}{a} \tag{1}$$

where X is the concentration of HVO, AX is the peak area of x% v/v HVO, A0 is the peak area of 0 %v/v HVO, b is the Intercept from the calibration curve, and a is the slope.

RESULT AND DISCUSSION

The chromatograms of diesel fuel and Hydrotreated Vegetable Oil (HVO) are shown in Figure 1. As illustrated, the overall patterns of the two chromatograms appear highly similar, particularly across certain retention times. This similarity is expected, given that both fuels are composed mainly of paraffinic hydrocarbons, and it supports their compatibility when blended. However, this also creates a challenge in analytical separation making it more difficult to distinguish and quantify each component accurately when the two are mixed.

From the chromatographic profiles, two distinct peaks labeled as peak $C_{16}H_{34}$ (Hexadecane) and peak $C_{17}H_{36}$ (Heptadecane) were selected for further analysis. These peaks were chosen because they consistently appeared in both the diesel and HVO chromatograms, making them reliable reference points for samples containing both fuels. In addition, both peaks showed clear separation from neighboring signals and had symmetrical shapes, which are important for achieving accurate and repeatable integration during peak analysis. Palm oil is rich in palmitic acid and oleic acid (Sotelo-Boyas et al., 2012) , These are the main fatty acids that contribute to the composition of Hydrotreated Vegetable Oil (HVO) produced from this feedstock. During the hydrogenation process, oleic acid undergoes saturation of its double bond, and further reactions like hydrodeoxygenation or mild hydrocracking can result in the removal of oxygen groups or slight reductions in carbon chain length. These reactions primarily yield straight-chain alkanes with 16 or 17 carbon atoms, namely $C_{16}H_{34}$ (Hexadecane) and $C_{17}H_{36}$ (Heptadecane) (Sotelo-Boyas et al., 2012). When HVO is analyzed using GC-FID, these alkanes consistently appear as dominant peaks in the chromatogram. Their abundance and origin directly from the major components of palm oil make them excellent marker compounds for estimating HVO concentrations in diesel blends. Their presence reflects the chemical transformation of palm-based fatty acids during the refining process, offering both relevance and reliability for analytical purposes. The latest research utilized the heptadecane peak as the primary indicator to determine the HVO content in diesel blends, due to its presence in both diesel and HVO components (Bethari et al., 2025). The other research used the hexadecane peak to develop a predictive model for the determination of the HVO content in diesel blends (Cozendey et al., 2025).

To evaluate whether these peaks could be used for quantifying HVO in blends, we tested their linearity and accuracy. Calibration curves were prepared using known mixtures of diesel and HVO, and the response of each peak was monitored. Accuracy was then assessed by comparing the calculated HVO content in test samples against their known concentrations. These tests helped determine which peak yielded the most consistent and reliable results, providing insight into how well this peak-based approach could work for the routine analysis of HVO in diesel blends.

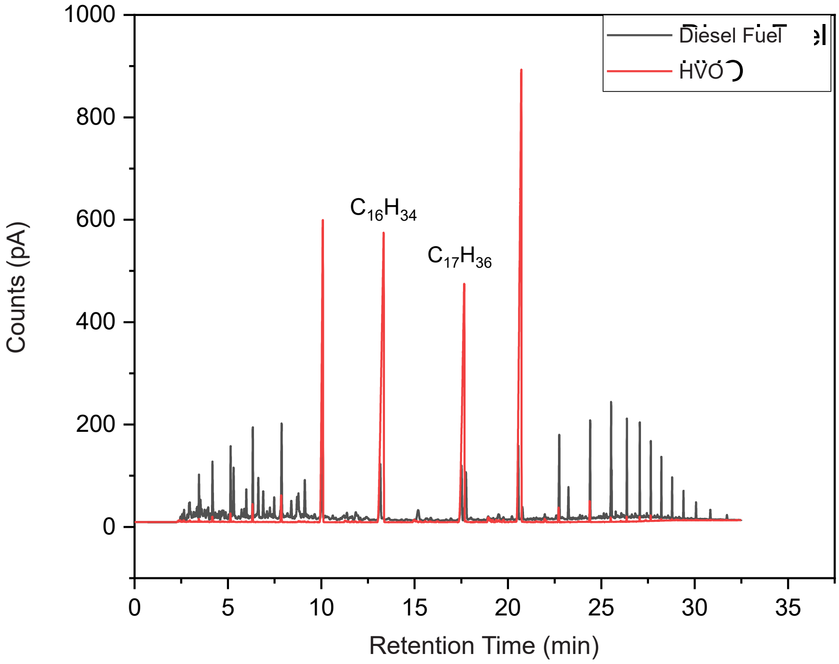

Figure 1. Chromatograms of HVO and diesel fuel.

After selecting the chromatographic peaks for HVO quantification, the next step was to evaluate their analytical performance in terms of linearity, as well as the limit of detection (LOD) and limit of quantification (LOQ). This evaluation was essential to identify which peak could provide the most accurate and reliable results for measuring HVO content.

To develop the calibration curves, a series of HVO-isooctane mixtures was prepared with HVO concentrations ranging from 0% to 50% v/v HVO. Isooctane was chosen as the solvent because it does not naturally appear in either diesel or HVO. Isooctane was selected as the calibration solvent in this study due to its compatibility with both the chemical nature of diesel and HVO. As a hydrocarbon compound derived from petroleum, isooctane shares similar physicochemical properties with fossil diesel, including non-polarity and volatility. These characteristics allow it to effectively dissolve HVO, which is also composed primarily of saturated hydrocarbons with similar chain lengths. The use of isooctane ensures good miscibility with HVO without introducing interfering compounds that might overlap with diesel components in the chromatographic analysis. Furthermore, isooctane is not naturally present in either fossil diesel or HVO, making it an ideal background solvent that does not contribute to target peaks during GC-FID detection. Its use enables the construction of accurate calibration curves solely based on the concentration of HVO, ensuring that the quantification reflects only the target analyte. This approach simplifies the calibration process and enhances the precision of HVO measurement in various diesel blend compositions.

The linearity of each peak was assessed by plotting the detector response (peak area) against the known concentrations of HVO. Linear regression was then used to generate calibration equations, and the coefficient of determination (R^2) was calculated to assess the fit of the data to a straight line. A strong linear correlation is important for ensuring that the method can reliably predict HVO content across different concentrations.

As illustrated in Figure 2, the hexadecane peak shows a clear and strong linear relationship between the peak area and the concentration of HVO in the sample. The calibration curve displays excellent linearity, with a correlation coefficient (R^2) of 0.9994, indicating that the peak response increases consistently with increasing HVO concentration. From the calibration curve, the following linear regression equation was obtained: $y = 40.032x - 6.7796$. Using this calibration, the Limit of Detection (LOD) and Limit of Quantification (LOQ) were also calculated. The result values were LOD:1.84% v/v HVO and LOQ: 5.57% v/v HVO.

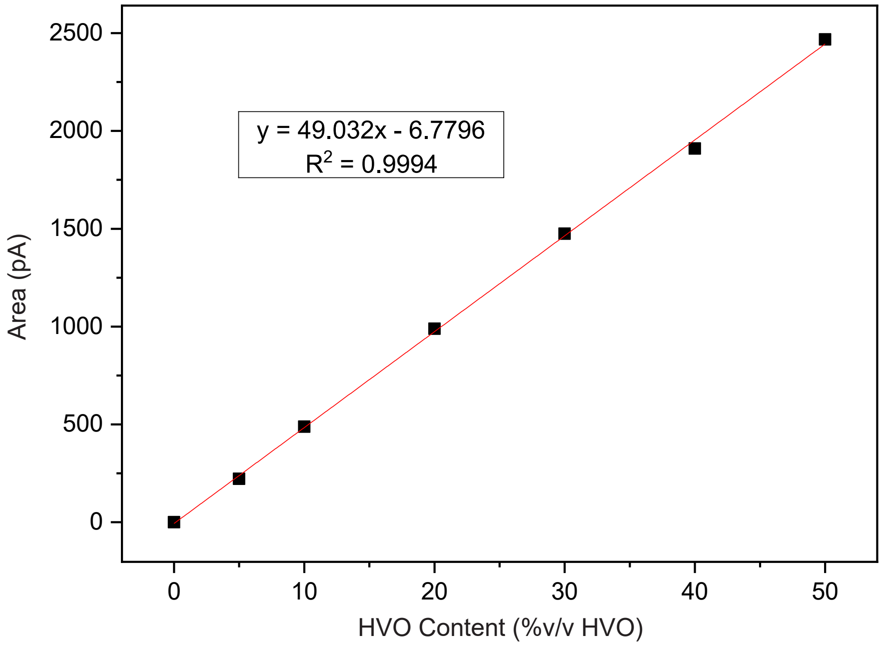

Figure 2. Linearity curve of hexadecane peak area to % HVO in diesel blends.

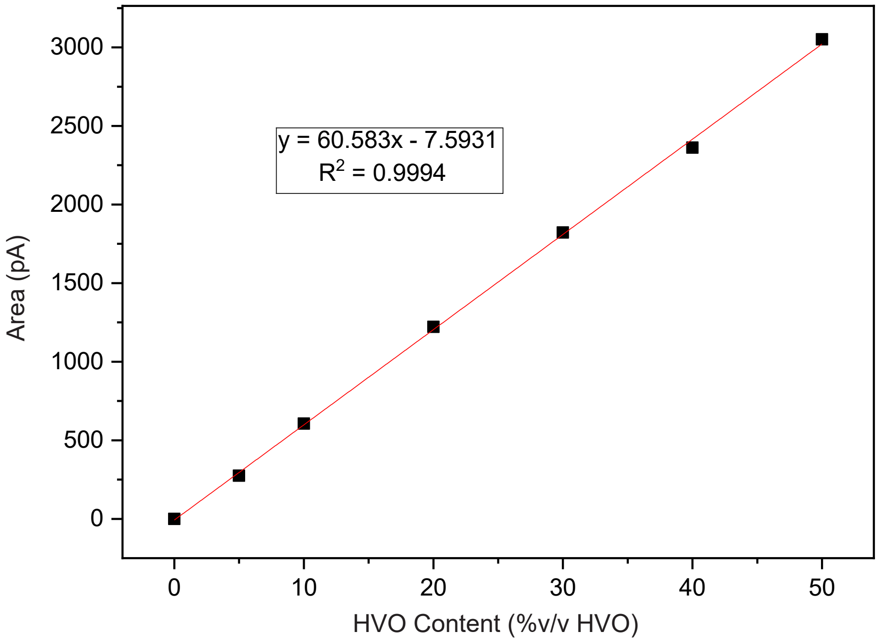

Figure 3. Linearity curve of heptadecane peak area to % HVO in diesel blends.

As shown in Figure 3 The linearity of the heptadecane peak shows the same correlation between the HVO concentration and peak response compared to the hexadecane peak. The coefficient of determination (R^2) is 0.9994 its better than the previous research 0.991 gas chromatography coupled to mass spectrometry (Cozendey et al., 2025). From the calibration curve, the following linear regression equation was obtained: y = 60.583x – 7.593. LOD and LOQ value from these curves are: 1.77 % v/v HVO and 5.36 % v/v HVO. Based on the data presented in Figure 2 and Figure 3, it is evident that the calibration curve for the heptadecane peak demonstrates better sensitivity compared to that of the heptadecane peak. This is supported by the higher slope and lower Limit of Detection (LOD) and Limit of Quantification (LOQ) obtained for heptadecane, indicating a more sensitive and consistent analytical response (Harvey, 2000).

From Figure 2 and Figure 3. The linearity data obtained in this study demonstrate excellent correlation for determining HVO concentrations above 1% v/v. Moreover, the linear trend continues consistently across the tested concentration range, suggesting that the method remains robust even at higher HVO levels. This indicates that the calibration model is not only suitable for low to moderate HVO blends but can also be extended to accurately quantify pure HVO (100% v/v) if necessary. The present study demonstrates an improvement over previous research, which was limited to quantifying a maximum of 20% v/v HVO using a near-infrared spectroscopy (NIR)(Wikberg et al., 2021). In practical terms, this means that the analytical method developed is versatile and reliable for a wide range of HVO concentrations, including scenarios where diesel fuel is fully replaced by renewable HVO. This flexibility enhances the applicability of the method in various industrial and regulatory settings where HVO content may vary depending on fuel specifications or policy requirements. Following this evaluation, an accuracy test was conducted to assess how well the established linear calibration curve could predict HVO content in real samples. This test involved the analysis of six different diesel fuel samples spiked with 10% v/v HVO. The results of the accuracy test are summarized in Table 2 and Table 3, and provide insight into the reliability and practical applicability of the method in routine HVO determination.

Table 2. % recovery of six different diesel samples using the peak of heptadecane

No. Sampel	Theoretical Concentration (%v/v HVO)	Measured Concentration (% v/v HVO)	% Recovery
1	10	10.21	102.08
2	10	10.33	103.32
3	10	9.48	94.83
4	10	10.28	102.81
5	10	10.29	102.88
6	10	9.95	99.51

Based on the data presented in Table 2 and Table 3, the average recovery values for HVO quantification using the heptadecane and hexadecane peaks were 102% and 101%, respectively. These high recovery rates indicate that both peaks provide excellent accuracy in determining HVO concentrations in diesel fuel blends. The results suggest that the analytical method is highly reliable and capable of producing consistent and precise measurements. Among the two, the heptadecane peak yielded slightly superior accuracy, making it the preferred marker for quantifying HVO content. This reinforces the suitability of using these hydrocarbon peaks—particularly heptadecane—for accurate and robust analysis of HVO in complex fuel matrices using GC-FID.

Table 3. % Recovery of six different diesel samples using the peak of hexadecane

No. Sampel	Theoretical Concentration (%v/v HVO)	Measured Concentration (% v/v HVO)	% Recovery
1	10	10.09	103.96
2	10	10.15	104.52
3	10	9.27	95.99
4	10	10.03	103.33
5	10	9.80	101.11
6	10	9.85	101.63

The method developed in this study has demonstrated strong potential for application in determining HVO concentrations in diesel fuel blends, supported by excellent performance in key validation parameters, linearity, limit of detection (LOD), limit of quantification (LOQ), and accuracy. The calibration curves showed highly linear responses across the tested concentration range, with coefficients of determination (R^2) consistently close to 1. This indicates the method's reliability for quantifying HVO over a wide range, including low and high concentrations. Additionally, the low LOD and LOQ values highlight the method's sensitivity, allowing for the detection of even trace amounts of HVO in diesel. Accuracy testing conducted on multiple samples further confirmed the method's reliability, with recovery rates exceeding 95%, suggesting minimal deviation from the actual values. The previous study used infrared NIR spectroscopy combined with multivariate analysis and reported a REP of 2% (Alves & Poppi, 2016b). There are several reasons why this error value was lower compared to the present work. First, in this study, six different diesel samples with a single concentration variation were used, whereas the previous study utilized only one type of diesel sample with various concentration levels. Taken together, these findings confirm that the GC-FID method, utilizing selected hydrocarbon peaks, is not only simple and cost-effective but also robust and suitable for both routine quality control and research applications in the monitoring of HVO in blended diesel fuels.

CONCLUSION

This study is successfully developing a straightforward and reliable method using GC-FID to measure the amount of Hydrotreated Vegetable Oil (HVO) in diesel fuel blends. Two peaks, hexadecane and heptadecane, are being assessed as potential markers, and the results are clearly showing that the heptadecane peak is offering better overall performance. It is demonstrating excellent linearity ($R^2 = 0.9994$), along with low detection (1.77% v/v HVO) and quantification limits (5.36% v/v HVO). When tested across six different diesel samples containing 10% HVO, the method is producing recovery rates above 95%, confirming its accuracy and consistency. In comparison, the hexadecane peak is showing the same linearity ($R^2 = 0.9994$) but lower sensitivity, making it less suitable for precise quantification. Overall, the use of the heptadecane peak is providing a practical and cost-effective approach for routine HVO analysis in diesel blends. The method is simple to apply, does not require complex instrumentation, and is being considered highly suitable for regular use in quality control laboratories. These findings are supporting the integration of reliable and efficient tools for tracking renewable fuel content, an important step toward ensuring fuel quality and advancing the global transition toward sustainable energy sources.

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GLOSSARY OF TERMS

Symbol	Definition	Unit
HVO	Hydrotreated Vegetable Oil	
ASTM	American Society for Testing and Materials	
NIR	Near-Infrared Spectroscopy	
GC-MS	Gas Chromatography-Mass Spectrometry	
GC-FID	Gas Chromatography-Flame Ionization Detection	
LOD	Limit of Detection	
LOQ	Limit of Quantification	
REP	Relative Error of Prediction	%
PLSR	Partial Least Squares Regression	
%v/v	Precentage Volume	
$C_{16}H_{34}$	Hexadecane	
$C_{17}H_{36}$	Heptadecane	
X	The concentration of HVO	
AX	The peak area of x% v/v HVO	
$A0$	The peak area of 0 %v/v HVO	
b	The Intercept from the calibration curve	
a	The slope from the calibration curve	

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