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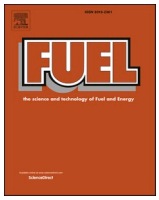

Full Length Article

# Forensic differentiation of Malaysia biodiesel and illicit fuels using GC-FID and GC–MS techniques

Mohd Rashidi Abdull Manap [a,b,*], Ananthy Retnam [c], Norizah Abdul Rahman [a,d], Nurul Ain Mohammed [a,c], Nurul Zulaikha Rozlan [a,c], Hui Juen Sew [a,c], Ramizah Azis [e], Noor Hazfalinda Hamzah [f], Philipp Weller [g]

[a] Department of Chemistry Faculty of Science Universiti Putra Malaysia, Serdang 43400 UPM Selangor, Malaysia
[b] Medical Microspectroscopy Research Group, Department of Experimental Medical Science, Lund University, Lund 22180, Sweden
[c] Environmental Quality Division, Drinking Water, Food and Environmental Safety Analysis Centre, Department of Chemistry Malaysia, Jalan Sultan, Petaling Jaya 46661, Selangor, Malaysia
[d] Laboratory of Nanomaterials Processing and Technology, Institute of Nanoscience and Nanotechnology, Universiti Putra Malaysia, UPM, Serdang 43400 Selangor, Malaysia
[e] Jabatan Pengajian Umum IKTBN Sepang, Bandar Baru Salak Tinggi, Sepang 43900 Selangor, Malaysia
[f] Forensic Science Program, Faculty of Health Sciences, Universiti Kebangsaan Malaysia, Bangi 43000 Selangor, Malaysia
[g] Institute for Instrumental Analysis and Bioanalytics, Faculty of Biotechnology, Technical University of Applied Sciences Mannheim, Paul-Wittsack-Str. 10, Mannheim 68163, Germany

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ABSTRACT

Illegal, unreported, and unregulated (IUU) fishing poses a serious threat to marine biodiversity and economic stability, particularly in coastal nations like Malaysia, where the smuggling of government-subsidized fuel is often linked to IUU activities. This study presents a forensic approach using gas chromatography-flame ionization detection (GC-FID) and gas chromatography-mass spectrometry (GC–MS) to chemically characterize and differentiate between legally distributed Malaysia biodiesel blends (B7 and B10) and illicit fuels seized from vessels involved in maritime violations. A total of 29 fuel samples from detained vessels in Kuala Terengganu and Mersing were compared with 20 reference biodiesel samples from major Malaysia fuel brands. GC-FID revealed hydrocarbon ranges of C10–C29 in biodiesel, with additional FAME peaks near n-C19 and n-C21, while IUU fuels showed wider ranges (C10–C33) but no FAMEs. GC–MS confirmed key biomarkers—bicyclic sesquiterpanes, adamantanes, isoprenoids, PAHs, and FAMEs—highlighting methyl palmitate (C16:0) and methyl oleate (C18:1) as diagnostic of biodiesel. Multivariate analyses (HCA and PCA) further separated B7, B10 and IUU samples, with subtle differences between B7 and B10 attributed to feedstock or blending variation. This study is the first to combine GC-FID, GC–MS, and chemometric analyses (PCA and HCA) into a forensic framework for differentiating Malaysian biodiesel blends (B7, B10) from illicit maritime fuels. By leveraging diagnostic biomarkers beyond FAMEs, the approach enables robust classification and provenance analysis. This integrated strategy provides evidential value for maritime law enforcement, advancing fuel forensics in Southeast Asia. These findings display the utility of chromatographic techniques in maritime law enforcement, enabling fuel source attribution and supporting legal proceedings. Despite promising results, limitations such as restricted sample coverage, lack of replicate analysis, and absence of a chromatographic fingerprint database highlight the need for further validation. The study advocates for the development of an integrated GC-based forensic framework to enhance Malaysia's capability in combatting fuel smuggling and IUU fishing activities.

## 1. Introduction

Illegal, unreported, and unregulated (IUU) fishing threatens marine biodiversity, weakens fisheries governance, and undermines sustainable resource management, with severe consequences for food security, economic stability, and ecosystem health in coastal nations such as

* Corresponding author at: Department of Chemistry Faculty of Science Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia.
*E-mail address:* rashidi.manap@med.lu.se (M.R. Abdull Manap).

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Malaysia [1,2]. The Malaysian Maritime Enforcement Agency (MMEA) has reported rising arrests of foreign vessels involved in IUU fishing and related crimes like fuel smuggling, which is fueled by regional price disparities and often supplies unauthorized vessels with subsidized Malaysian fuel [3,4]. Since these vessels may use illicit fuel, integrated forensic analyses are essential to link fuel evidence with maritime offenses, strengthen enforcement, and inform policies that disrupt logistical networks sustaining IUU operations. In this context, biodiesel—composed mainly of fatty acid methyl esters (FAMEs) derived from feedstocks such as vegetable oils, animal fats, waste cooking oils, and algal oils [5–8]—is of particular relevance. Its composition typically includes palmitic (C16:0), stearic (C18:0), oleic (C18:1), linoleic (C18:2), and linolenic (C18:3) acids, with pure biodiesel containing about 11 % oxygen by weight [9,10]. Southeast Asian countries rely heavily on palm oil for biodiesel, with Indonesia advancing B40 (40 % biodiesel, 60 % diesel) and B30D10 (30 % biodiesel + 10 % hydrogenated vegetable oil, 60 % diesel) blends [11], while Malaysia's nationwide commercial blends in 2021 were B10 EURO 5 (10 % palm methyl ester, 90 % diesel) and the higher-priced B7 EURO 5 (7 % palm methyl ester, 93 % diesel) [12]. Biodiesel blends such as B7 and B10, and illicit fuels associated with IUU fishing, can be chemically profiled and thus provide evidential linkages between seized fuel samples and maritime offenses.

Gas chromatography (GC) is a gold standard for differentiating fuel types, particularly diesel and biodiesel, by analyzing their complex chemical compositions with high resolution and precision. For biodiesel, GC is pivotal in determining FAME profiles, which influence cetane number and oxidation stability [13]. GC and liquid chromatography (LC) techniques also measure bound glycerol content without the need for derivatization [14]. Comprehensive two-dimensional GC (GC × GC) excels in analyzing commercial biodiesel-petroleum diesel blends in market, offering precise FAME quantitation even at low blend ratios [15], while standard gas chromatography-flame ionization detector (GC-FID) provides accurate detection of FAMEs in diesel fuel without complex sample preparation [16]. Complementary methods such as HPLC and high-performance size-exclusion chromatography (HPSEC) have also shown promise for quantifying FAMEs and detecting oxidation products without complex pre-treatment [17,18]. While GC-FID is mainly used in diesel fuel analysis, gas chromatography-mass spectrometry (GC–MS) is getting attention as this analysis provides more insight into the samples themselves through the sensitivity and precision, especially in forensic work. GC–MS provide details of chemical compounds profile, which is helpful in providing a complete picture of the samples and can serve as a confirmatory analytical tool, especially for unknown samples and/or compounds. The GC-FID is used as a screening tool to determine the hydrocarbon profile of the fuel samples, while GC–MS can be employed to probe into compound details and their ratios to distinguish the similarity and differences of fuel samples. Utilizing both GC-FID and GC–MS provide significant details of the fuel samples for decision making.

Despite advances in chromatographic techniques for fuel characterization, a critical research gap remains in integrating GC-FID and GC–MS into a comprehensive forensic framework tailored for maritime enforcement against IUU fishing and fuel smuggling. Existing studies largely focus on biodiesel blend composition but fall short of meeting evidentiary requirements such as validated, reproducible, and court-admissible protocols. In particular, the lack of an updated chromatographic database of commercial biodiesel blends, including Malaysia's B7 and B10, hinders real-time provenance analysis by enforcement agencies like the MMEA. While GC-FID is effective for quantifying FAMEs and GC–MS provides detailed molecular profiling, their combined application for classification and origin attribution of fuels in a forensic context remains underexplored in Southeast Asia. Developing an integrated GC-FID/GC–MS chemometric system and establishing chromatographic fingerprint libraries would enable robust discrimination between domestic and foreign biodiesel fuels, thereby enhancing Malaysia's capacity to link seized fuels to illicit maritime activities and support judicial proceedings.

Over the years, there has been a growing interest in fuel analysis using gas chromatography and multivariate techniques. The chemical composition of fuel samples obtained by GC is often complex, and multivariate methods such as Hierarchical Cluster Analysis (HCA) [19], Principal Component Analysis (PCA) [20], and Discriminant Analysis (DA) [20] are commonly applied to extract meaningful information. HCA and PCA are unsupervised techniques widely used to explore clustering patterns and reduce dimensionality without prior class labels. While intrinsically PCA is not a classification algorithm, it remains a useful approach for identifying correlations or differences within complex datasets, and it is the most utilized pattern recognition tool for fuel analysis to distinguish sample classes [21,22]. Their utility has been demonstrated in diesel fuel studies, such as in Morocco [23], where PCA and PLS-DA of FTIR and GC–MS fingerprints successfully discriminated 80 diesel samples from four major suppliers, providing accurate classification and traceability, and in Taiwan [24], where PCA with diagnostic ratios effectively distinguished fresh and weathered diesel samples from two refinery companies.

Although multivariate techniques such as PCA and HCA have been applied to fuel studies, there are still very few references addressing their use for the identification of biomarkers beyond FAMEs and classification of biodiesels. Existing research has largely emphasized biodiesel composition and quality assessment, without integrating GC-FID, GC–MS, and chemometric methods into a unified forensic framework. In particular, while GC–MS combined with PCA has been employed for general fuel characterization, it has not yet been applied to the forensic differentiation of Malaysian biodiesel blends (B7 and B10) from illicit IUU fuels within an operational law enforcement context. This gap underscores the need for studies that not only characterize biodiesel but also translate analytical results into evidential tools for maritime enforcement.

This current study employs both GC–MS and GC-FID to determine the chemical profile of B7 and B10 biodiesel blends in Malaysia and IUU samples for classification and comparative analysis. GC-FID provides a hydrocarbon profile and additional peaks, while GC–MS enables detailed identification and quantification of hydrocarbons and FAME, the primary components of biodiesel. PCA was used to differentiate the different fuel types. By integrating both techniques, the study enhances the reliability of fuel characterization, supporting efforts to distinguish domestically distributed Malaysia fuel from foreign vessels seized in forensic cases related to IUU fishing activities within Malaysia waters.

## 2. Geographical and geological framework

### 2.1. Kuala Terengganu

Terengganu state is situated on the eastern coast of Peninsular Malaysia, and it has the longest coastline in Peninsular Malaysia, around 244 km facing the South China Sea waters [25]. Terengganu is known as one of the centres of abundance of underwater marine life [26]. Therefore, it is important to protect Malaysia's maritime borders against non-military threats, thefts, illegal activities, misuse of hubs, and other non-state entities [27]. The Malaysian Maritime Zone covers Malaysia's internal waters, territorial sea, continental shelf, exclusive economic zone, and Malaysian fisheries waters as well as the air space above these areas. One of the Jeti Pusat Tahanan Vesel (PTV) controlled by MMEA is located in Pulau Kambing, Kuala Terengganu. This enforcement agency has the power to expulse any vessel that is suspected to endanger the order and safety in the Malaysian Maritim Zones [28].

### 2.2. Mersing

In Johor, there are five development regions with distinct strengths and specializations [29]. In the east of Johor, Mersing is where the

majority of the fish are caught, compared to Kota Tinggi [30]. Mersing is located in the southeastern region of Peninsular Malaysia, approximately 136 km northeast of the state capital, Johor Bahru [31,32]. Mersing is well-known for its fishing industry, which contributes to the heavy boat traffic on the river. Furthermore, MMEA is also operated in Mersing and the office is located near the Mersing River in Johor, which is opposite the South China Sea.

## 3. Materials and methods

### 3.1. Sampling

Prior to sample collection, clean and dry glass bottles were pre-labelled with their corresponding lot numbers (see Supplementary Material). Each bottle was pre-rinsed with a small portion of the respective fuel sample, which was discarded to reduce contamination. Approximately 20 mL of sample was then collected per vessel. All IUU samples were sealed and stored at room temperature in the Universiti Putra Malaysia (UPM) laboratory. Analytical assessments of the 29 engine-derived samples were jointly conducted by the Department of Chemistry Malaysia and UPM. Both room temperature and low temperature storage are used in fuel studies. Biodiesel–diesel blends were commonly stored at room temperature (15 °C–25 °C), in clean, sealed glass containers protected from sunlight and moisture prior to analytical measurement [33,34]. Some studies also employ low–temperature storage (ca. 5 °C) to minimise degradation during long-term stability investigations [35]. In the present study, both low- and room-temperature storage were employed because the primary objective was to simulate realistic scenarios experienced by IUU maritime fuel samples during actual field conditions, rather than to assess long-term chemical stability.

A total of 29 fuel samples suspected to be associated with IUU activities were collected from seized vessels operating within Malaysian maritime waters, specifically in Kuala Terengganu and Mersing. They were collected directly from fuel filter. The sampling from Kuala Terengganu was conducted at the Jeti Pusat Tahanan Vessel in November 2021, following formal approval from the Head Office of MMEA, Putrajaya.

20 reference biodiesel samples comprising B7 (n = 7) and B10 (n = 13) blends were collected from commercial petrol stations in Seri Kembangan, Selangor [36]. The samples represented five major brands: BHPetrol, Petronas, Shell, Caltex, and Petron. Sampling was conducted with prior permission from the Ministry of Domestic Trade and Consumer Affairs Malaysia (KPDNHEP). All reference samples were transferred into amber glass bottles, registered with Jabatan Kimia Malaysia, and stored at 4 °C until analysis.

### 3.2. Sample analysis

Diesel samples were stored at 4 °C prior to analysis. The sample preparation and analysis were conducted in accordance with the BS EN 15522-2 standard) [37]. The methodology described in this manuscript is based on the method validation work instruction adopted for sample analysis. It is acknowledged that the BS EN 15522-2 standard outlines only the fundamental principles of sample preparation and analysis. Accordingly, the laboratory has validated this method to suit its specific instrumentation and analytical requirements.

#### 3.2.1. Chemicals

Acetone and *n*-hexane were obtained from Fisher Scientific (Loughborough, Leics, UK). α-Androstane and *p*-terphenyl-$d_{14}$ were purchased from Sigma-Aldrich (Burlington, USA). Dichloromethane was sourced from Merck (Darmstadt, Germany), while SINTEF oil was supplied by SINTEF (Norway, Europe).

#### 3.2.2. Sample preparation

All sample bottles and 10 mL volumetric flasks were rinsed three times sequentially with acetone, dichloromethane (DCM), and *n*-hexane to minimize contamination. A 20 µL aliquot of each biodiesel sample was accurately pipetted into a 10 mL volumetric flask and diluted to the mark with DCM.

For GC–FID analysis, 100 µL of the diluted sample was mixed with 10 µL of 100 ppm α-androstane solution, which served as the internal standard. DCM was used as the blank solution, and an *n*-alkane standard mixture (C10–C40) was employed as the calibration standard.

For GC–MS analysis, 200 µL of the diluted sample was mixed with 4 µL of 200 ppm *p*-terphenyl-$d_{14}$ solution, used as the internal standard. DCM served as the blank, and SINTEF oil was used as the standard reference material for compound comparison and calibration. Primarily, the biodiesel and IUU samples were prepared at a concentration of 10 mg/L. For GC-FID analysis, the concentration of both biodiesel and IUU samples was at 10 mg/L, while for GC–MS, the concentration was fixed at 3 mg/L.

### 3.3. Instrumentations

#### 3.3.1. Gas chromatography-flame ionization detection (GC-FID)

Biodiesel samples were prepared and analyzed using GC-FID to determine the hydrocarbon profile and *n*-alkane pattern. The analyses were performed using an Agilent 7890B GC system (Agilent Technologies, USA) equipped with a flame ionization detector and an Agilent 7693 autosampler. Chromatographic separation was achieved using an Elite-1 capillary column (30 m × 0.25 mm i.d., 0.25 µm film thickness).

The oven temperature was initially held at 35 °C for 1.5 min, ramped at 10 °C/min to 330 °C, and held isothermally for 10 min, resulting in a total run time of 40 min. The inlet temperature was maintained at 325 °C. All injections were carried out in splitless mode. Zero air (450 mL/min and hydrogen (40 mL/min) served as combustion gases, helium was used as the carrier gas, and nitrogen (25 mL/min) was used as the makeup gas.

#### 3.3.2. Gas chromatography-mass spectrometry (GC–MS)

GC–MS analysis was performed to characterize both the diesel and FAMEs characteristics in the biodiesel samples. Analyses were conducted using an Agilent 7890B GC system coupled with a mass selective detector and the Agilent 7693 autosampler. An HP-5MS capillary column (30 m × 0.25 mm i.d., 0.25 µm film thickness) was employed for separation. The GC oven program commenced at 42 °C with an initial hold of 3.6 min, followed by a ramp of 5.48 °C/min to 330 °C, which was then held for 10 min. The total analysis time was 66 min. The inlet temperature was maintained at 325 °C, and all samples were introduced in splitless mode with helium as the carrier gas. Mass spectrometric detection was programmed for both SCAN and selected ion monitoring (SIM) modes as shown in Table S5. SCAN mode was used to generate the full biodiesel profile, whereas SIM mode was utilized for compound identification and quantification. For FAME detection, ion at $m/z$ 74 was monitored.

The BS EN 15522–2 method is a robust analytical approach for oil spill identification. It is designed for the analysis of various types of oils beyond diesel, including crude oil, heavy fuel oil, lubricating oil, and waste oil. These types of oils exhibit a high carbon number range; therefore, the GC–MS temperature program was configured to accommodate higher boiling compounds. Consequently, an extended run time was required to ensure adequate separation and detection of these components.

#### 3.3.3. Chemometric analysis

HCA was performed using OriginPro 2025b (OriginLab Corporation, USA) to classify Malaysia biodiesel blends (B7/B10) and illicit fuels (IUU) based on their chemical composition. Raw data was normalized to ensure equal weighting of all variables. HCA was performed using

Ward's linkage method and Euclidean distance to group fuels based on spectral similarity. The dendrogram was cut at a height of 0.5 to define clusters, with cophenetic correlation >0.9 confirming robustness.

For multivariate analysis, the peak height tables for 53 relevant compounds detected by GC–MS were used for principal component analysis (PCA). All data analysis was performed using Python 3.10 using scikit-learn and seaborn for plotting the data. The data were normalized and mean centered. A heatmap was generated from the 10 most relevant features obtained from the loadings plot.

## 4. Results and discussion

### 4.1. GC-FID screening

GC-FID was employed to examine the hydrocarbon composition of Malaysia biodiesel blends B7 and B10. Standard alkane mixtures (C10–C40) were analyzed concurrently as a reference to facilitate peak identification and quantification. In this study, five brands of biodiesel that are common in Malaysia namely BHPetrol, Caltex, Petron, Petronas and Shell, were sampled. The chromatographic profiles of biodiesel of B7 and B10 samples of different brands show that the predominant hydrocarbons ranged from *n*-C10 to *n*-C29, which is a typical hydrocarbon range for diesel. Together with this, additional peaks were observed to elute at *n*-C19 and *n*-C21. These additional peaks were observed to appear in all samples. These peaks that eluted near *n*-C19 and *n*-C21 were attributed to FAMEs. The intensity of FAME peaks for B10 was higher than B7 for all brands, which can be observed in Fig. 1. This is consistent with blend ratios where B10 consist of 10 % of FAME while B7 contain only 7 % of FAME. Chromatograms of the reference biodiesel B7 and B10 samples are shown in Fig. S2 and S3 in the Supplementary Material.

As for IUU samples, all samples showed a carbon range between C10 and C28, and in some samples extending up to C33, which indicates they are diesel fuel (Supplementary Material). Notably, no additional peaks corresponding to FAMEs were present between *n*-C19 and *n*-C21 in the IUU samples, indicating these samples were not biodiesels.

Fig. 1 shows the stacked chromatograms (B7, B10, and IUU), and it clearly illustrates the compositional differences between the samples. Both biodiesel B7 and biodiesel B10 exhibit prominent peaks corresponding to FAMEs C16 and C18. These FAME peaks serve as robust markers of Malaysia biodiesel in B7 and B10 blends.

The different diesel fuel samples can be differentiated by the shapes of the *n*-alkanes and the shape of the unresolved complex mixture (UCM) and their biomarker pristane and phytane ratios [38]. For biodiesel samples, all samples showed similar UCM and *n*-alkanes patterns. The UCM for all samples was found to be low, indicating the petrogenic

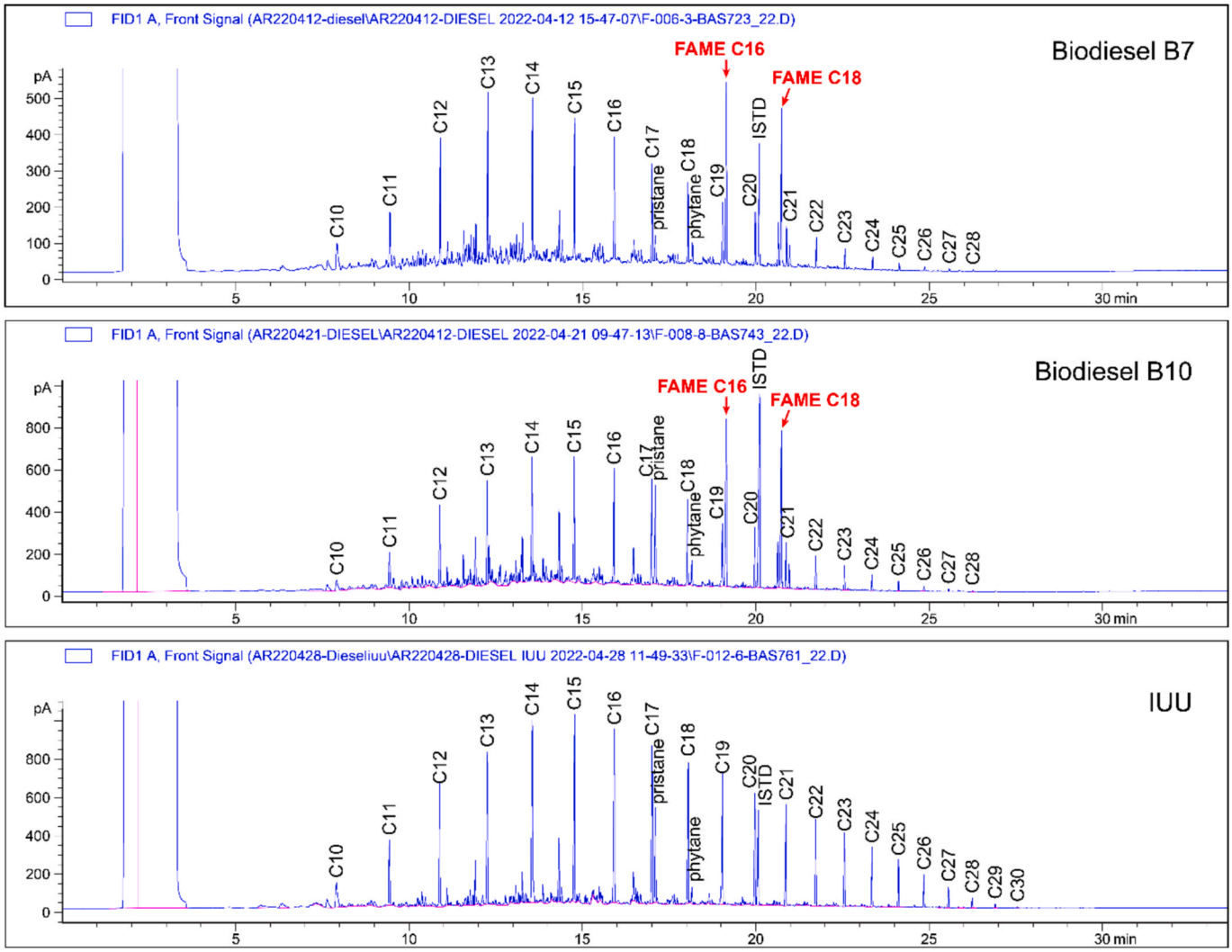


**Fig. 1.** A stacked GC-FID chromatogram for biodiesel B7, B10 and IUU samples. ISTD refers to androstane internal standard.

nature of the fuel samples. The *n*-alkane pattern for biodiesel showed similar pattern with the highest peak at *n*-C14 and *n*-C16 (Caltex), while for IUU samples, *n*-C14 was the highest peak in all samples. The isoprenoids ratios C17/pri, C18/phy and pri/phy were calculated for all samples (Table 1). In the biodiesel samples, the PHPetrol and Caltex biomarker pri/phy ratio was lower compared to Petron, Petronas and Shell. Meanwhile, for IUU samples, the ratios vary from the lowest (1.31) to the highest (6.16). These differences can be seen in the slope of the line above these compounds (Fig. 2).

Based on the GC-FID results, the major difference in the samples was that the biodiesel samples were indicated by the presence of the FAME compounds, while all IUU samples were found to be diesel, given that there was no detectable presence of FAME compounds. Further insight into the hydrocarbons of the samples indicated some similarities and differences among the biodiesel and IUU samples, as can be seen in the distribution of *n*-alkanes and isoprenoids, pristine and phytane. Further detailed analysis by GC–MS is required to understand the similarity and differences between the biodiesel and IUU samples.

**Table 1**
Summary of ratios C17/pri, C18/phy and pri/phy for Malaysia biodiesel and IUU samples.

| Biodiesel type | Brand | Ratio | | |
|---|---|---|---|---|
| | | C17/pri | C18/phy | pri/phy |
| B7 | BH Petrol | 3.91 | 4.11 | 1.29 |
| B10 | BH Petrol | 3.30 | 4.16 | 1.55 |
| B7 | Caltex | 3.91 | 3.75 | 1.10 |
| B10 | Caltex | 3.40 | 3.70 | 1.25 |
| B10 | Caltex | 3.75 | 3.84 | 1.20 |
| B7 | Petron | 1.24 | 3.53 | 3.48 |
| B7 | Petron | 1.19 | 3.56 | 3.43 |
| B10 | Petron | 1.15 | 3.32 | 3.66 |
| B10 | Petron | 1.10 | 4.02 | 3.98 |
| B10 | Petron | 1.16 | 3.69 | 3.85 |
| B7 | Petronas | 1.15 | 3.74 | 3.76 |
| B7 | Petronas | 1.01 | 3.00 | 3.61 |
| B10 | Petronas | 1.03 | 3.36 | 3.77 |
| B10 | Petronas | 1.11 | 3.32 | 3.62 |
| B10 | Petronas | 1.00 | 3.23 | 3.82 |
| B10 | Petronas | 1.51 | 3.29 | 3.56 |
| B7 | Shell | 1.31 | 3.55 | 3.16 |
| B10 | Shell | 1.13 | 4.10 | 4.11 |
| B10 | Shell | 1.11 | 3.45 | 3.53 |
| B0 | IUU | 1.86 | 7.94 | 4.83 |
| B0 | IUU | 2.08 | 5.99 | 3.32 |
| B0 | IUU | 1.91 | 6.41 | 3.86 |
| B0 | IUU | 1.81 | 8.16 | 5.12 |
| B0 | IUU | 1.80 | 8.19 | 5.22 |
| B0 | IUU | 2.94 | 3.37 | 1.31 |
| B0 | IUU | 1.85 | 8.71 | 5.40 |
| B0 | IUU | 3.01 | 5.73 | 2.32 |
| B0 | IUU | 2.69 | 6.11 | 2.66 |
| B0 | IUU | 1.94 | 4.59 | 2.83 |
| B0 | IUU | 2.02 | 7.08 | 3.88 |
| B0 | IUU | 1.87 | 6.74 | 4.22 |
| B0 | IUU | 4.20 | 4.49 | 1.26 |
| B0 | IUU | 1.63 | 8.14 | 5.65 |
| B0 | IUU | 1.72 | 6.90 | 4.72 |
| B0 | IUU | 2.80 | 5.68 | 2.30 |
| B0 | IUU | 1.93 | 8.73 | 5.01 |
| B0 | IUU | 2.28 | 6.94 | 3.69 |
| B0 | IUU | 1.85 | 4.98 | 3.24 |
| B0 | IUU | 2.84 | 5.87 | 2.35 |
| B0 | IUU | 1.66 | 8.75 | 6.16 |
| B0 | IUU | 1.63 | 8.27 | 5.78 |
| B0 | IUU | 2.25 | 5.04 | 2.51 |
| B0 | IUU | 1.89 | 8.22 | 5.24 |
| B0 | IUU | 3.14 | 5.91 | 2.17 |
| B0 | IUU | 1.57 | 3.33 | 2.33 |
| B0 | IUU | 1.66 | 4.50 | 3.17 |
| B0 | IUU | 2.03 | 3.87 | 2.32 |
| B0 | IUU | 2.35 | 2.84 | 1.51 |

### 4.2. GC–MS confirmatory analysis

To further characterize the biodiesel B7/B10 and IUU samples, GC–MS was utilized, with analysis focused on the identification and quantification of hydrocarbons and FAME compounds. Selected ion monitoring (SIM) mode was employed to enhance sensitivity and selectivity of compounds.

In this study, a total of 55 individual hydrocarbon compounds belonging to five groups were analysed. Out of these, only a handful of compounds, such as biomarkers bicyclic sesquiterpanes, adamantane, isoprenoids, PAH compounds (e.g. C1-fluoranthene/pyrene, C1-dibenzothiophene) and FAMEs showed significant differences between biodiesel and IUU samples. Figs. 3 and 4 shows the ion chromatograms of biodiesel (B7 and B10) and IUU samples of isoprenoids (*m/z* 113), bicyclic sesquiterpanes (*m/z* 123), FAMEs (*m/z* 74), PAHs-dibenzothiophenes (*m/z* 198), PAHs-fluoranthenes/pyrenes (*m/z* 216), and adamantane (*m/z* 135).

Biomarker bicyclic sesquiterpanes (BS) are lower molecular weight biomarkers, usually found ambiguously in light fuel oils, such as diesel. There are nine commonly used bicylic sesquiterpane compounds, namely $C_{14}H_{26}$-bicyclic sesquiterpane (BS1), $C_{14}H_{26}$-bicyclic sesquiterpane (BS2), $C_{15}H_{28}$-bicyclic sesquiterpane (BS3), $C_{15}H_{28}$-bicyclic sesquiterpane (BS4), $C_{15}H_{28}$-8β(H)-drimane (BS5), $C_{15}H_{28}$-bicyclic sesquiterpane (BS6), $C_{16}H_{30}$-bicyclic sesquiterpane (BS8), $C_{16}H_{30}$-sesquiterpane (BS9), and $C_{16}H_{30}$-8β(H)-homodrimane (BS10). In this study, notable presence of BS1, BS3, BS4, BS5, BS6, BS8, BS9 and BS10 was found in both biodiesel and IUU. However, in IUU samples, the presence of BS2 was observed, which is not found in biodiesel samples. The presence of BS2 in IUU samples differentiates IUU samples from biodiesels. To confirm the differences between the biodiesel and IUU samples, diagnostic ratios of the BS were calculated. The compounds with a signal-to-noise ratio above 3 (S/N > 3) were integrated and calculated. The selection of compounds for ratio calculation is based on method BS EN 15522–2. The ratios BS1/BS2, BS5/BS6 and BS8/BS10 showed significant differences between the biodiesel and IUU samples (Table 2). Other ratios, such as BS4/BS5 and BS8/BS9, did not show significant differences between biodiesel and IUU samples.

Further investigation into biomarker compounds isoprenoids (*m/z* 113) shows the presence of isoprenoids: farmesane (i-C15), i-C16, norpristane (i-C18), pristane (i-C19) and phytane (i-C20). All of these compounds showed a similar pattern in both biodiesel and IUU samples, except that pristane in IUU samples showed higher intensity compared to biodiesels, indicating the IUU samples are different from the biodiesels. However, the ratios of C17/pri, C18/phy and pri/phy did not show any significant differences among the samples.

Adamantane (*m/z* 135) is a biomarker compound which is commonly used in the identification of light fuel oil, such as diesel. In this study, all samples showed the presence of adamantanes. However, compound 1-methyladamantane (1-MAdam) and 2-methyladamantane (2-MAdam) were more abundant in IUU samples than in biodiesel, providing a clear distinction between the two. The ratio for 1-MAdam/1,2-DMAdam further confirms the differences between biodiesel and IUU samples.

Comparison of PAH compounds cluster C1-fluoranthenes/pyrenes (*m/z* 216), which comprises of 6 compounds from different compound classes of aromatic hydrocarbons, namely 1-methylpyrene (1-MPy), 2-methylpyrene (2-MPy), 4-methylpyrene (4-MPy), 2-methylfluoranthene (2MFl), Benzo(b + c)fluorene (B(b + c)]F) and Benz(a)fluorene (BaF). They are relatively stable and used to compare diesel samples. In both biodiesel and IUU samples, a significant presence of 2-MPy, 4-MPy and 1-MPy was detected, but with no notable presence of B(b + c)F. Remarkably, 2-MFl was detected in biodiesel, while BaF was observed only in IUU fuel. When comparing the relative abundance of these compounds in all samples, 4-MPy was the base peak (100 %), 2-MPy was at 73 % in biodiesel and slightly lower at 61 % in IUU samples, while 1-MPy was relatively similar in both types of samples at 59 % in biodiesel

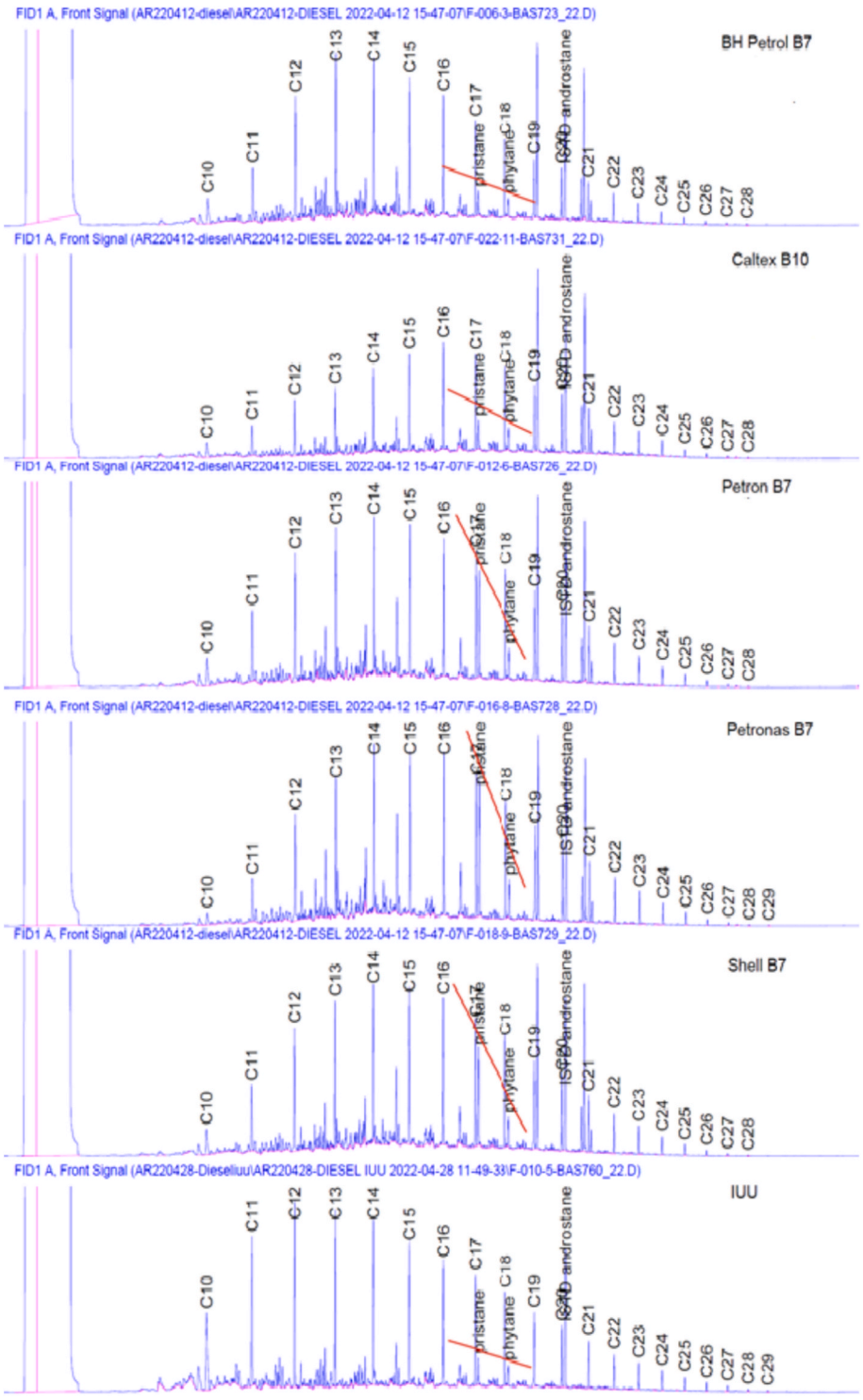


**Fig. 2.** Chromatogram of biodiesel and IUU samples shows the different line slope above the biomarkers pristane and phytane.

and 51 % in IUU samples. Compound BaF was not detectable in biodiesel, while at 16 % in IUU and for 2-MFl at 28 % in biodiesel but not detected in IUU samples (Fig. S5). To confirm these differences, various ratios were examined. Ratios 2-MFl/4-MPy, 2-MPy/4-MPy and BaF/4-MPy confirm the significant differences among biodiesel and IUU samples.

Sulfur-containing PAHs compound cluster, C1-dibenzothiophenes (*m/z* 198) comprises 3 compounds, namely 4-methyldibenzothiophene (4-MDbt), 2-methyldibenzothiophene + 3-methyldibenzothiophene (2-MDbt + 3-MDbt) and 1-methyldibenzothiophene (1-MDbt). All these compounds were abundant in IUU samples but not in biodiesels, with a relative abundance of 1-MDbt at 27 % relative to 4-MDbt, and further confirmed by the high ratio of 4-MDbt/1-MDbt in IUU samples. This indicates that the biodiesel samples, which are predominantly Euro 5, have a low sulfur content. This is in line with Malaysia's policy encouraging the adaptation of Euro 5 standards for diesel fuel vehicles [39].

Another important compound (and compound of interest) that

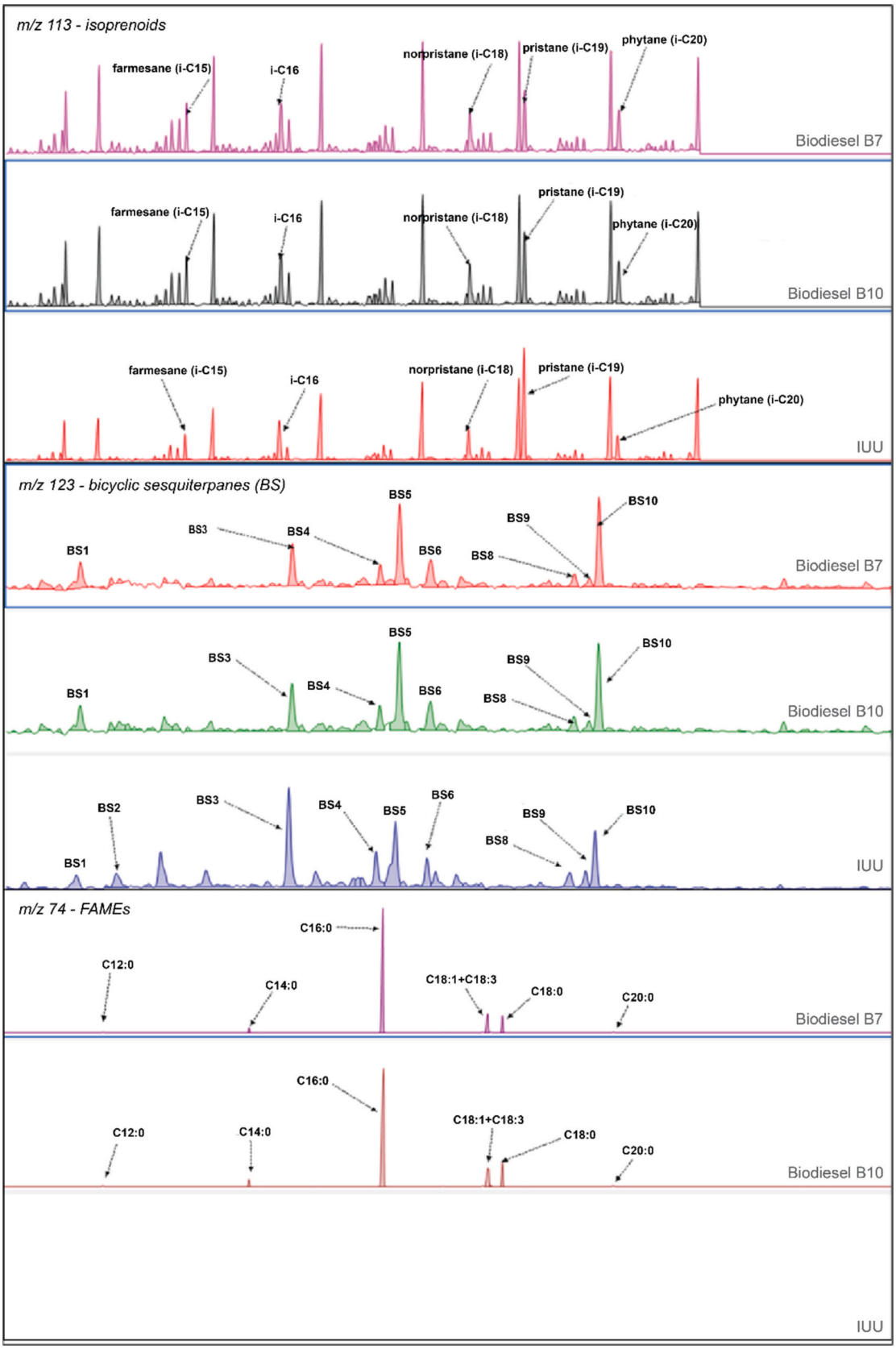


**Fig. 3.** Ion chromatograms for biomarker isoprenoids (*m/z* 113), bicyclic sesquiterpanes (BS; *m/z* 123) and FAMEs (*m/z* 74).

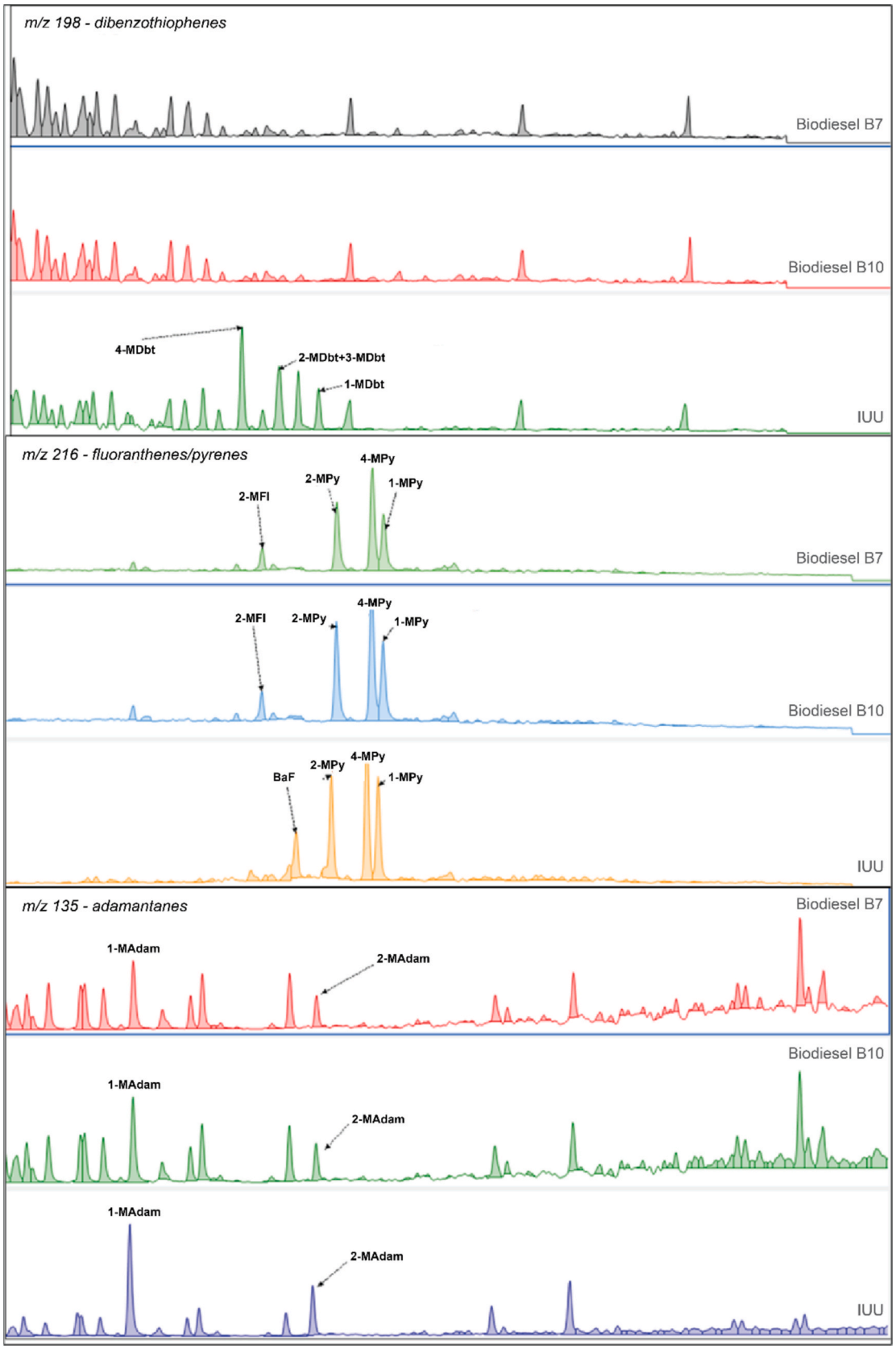


**Fig. 4.** Ion chromatograms for biomarker PAHs-dibenzothiophenes (*m/z* 198), PAHs-fluoranthenes/pyrenes (*m/z* 216) and adamantanes (*m/z* 135).

**Table 2**
Ratios of various compounds in biodiesel and IUU samples.

| Ratio | B7 | B10 | IUU |
|---|---|---|---|
| BS1/BS2 | 1.74 | 1.43 | 0.84 |
| BS4/BS5 | 0.29 | 0.25 | 0.26 |
| BS5/BS6 | 3.01 | 2.99 | 0.41 |
| BS8/BS9 | 1.08 | 1.12 | 1.00 |
| BS3/BS5 | 0.53 | 0.52 | 2.82 |
| BS8/BS10 | 0.14 | 0.17 | 3.05 |
| *n*-C17/pri | 4.88 | 4.24 | 4.42 |
| pri/phy | 0.97 | 1.09 | 1.04 |
| *n*-C18/phy | 4.06 | 4.10 | 4.08 |
| 1-MAdam/1,2-DMAdam | 1.40 | 1.52 | 2.11 |
| 4-MDbt/1-MDbt | nd | nd | 43.8 |
| 2-MFl/4-MPy | 0.22 | 0.21 | 0.15 |
| BaF/4-MPy | 0.03 | 0.06 | 0.77 |
| 2-MPy/4-MPy | 0.66 | 0.69 | 0.38 |
| 1-MPy/4-MPy | 0.55 | 0.55 | 0.55 |
| FAME C16:0/C18:0 | 7.21 | 4.71 | nd |
| FAME C12:0/C16:0 | 0.01 | 0.01 | nd |
| FAME C14:0/C16:0 | 0.04 | 0.06 | nd |
| FAME C18:2/C18:0 | 0.06 | 0.04 | nd |
| FAME C18:1 + C18:3/C18:0 | 1.10 | 0.76 | nd |

Note: nd – not detected.

showed significant differences between biodiesel and IUU samples is FAME. FAME compounds were explored using *m/z* 74 for both biodiesel and IUU samples. In biodiesel samples, FAME C14:0, FAME C16:0, FAME C18:0, FAME C18:1, FAME C18:3 and FAME C20:0 were detected at significant levels, while absent in IUU samples. The presence of the various FAMEs relative to FAME C16:0 revealed that C18:1 + C18:3 and C18:0 were at similar levels at 15 % and 14 % respectively, whereas C14:0 was present only at 4 %. The ratios for various FAME compounds were calculated to probe the differences among the samples. The ratios for IUU samples indicated as not detected (nd), given their presence was not at detectable levels. Meanwhile, these ratios in biodiesel samples are of interest. The saturated FAME ratio C16:0/C18:0 and FAME C18:1 + C18:3/C18:0 were significantly higher in biodiesel B7 samples compared to B10. This indicates there might be differences between biodiesel B7 and B10 based on FAME content.

In order to reduce the mineral oil consumption, diesel is blended with a certain amount of biodiesel. Biodiesel is a common term used for FAMEs in plant and animal glycerides. The transesterification of fats yields primarily a mixture of methyl palmitate (FAME C16:0), methyl stearate (FAME C18:0) and unsaturated C18 FAMEs, including methyl oleate (FAME C18:1), methyl linoleate (FAME C18:2), and methyl

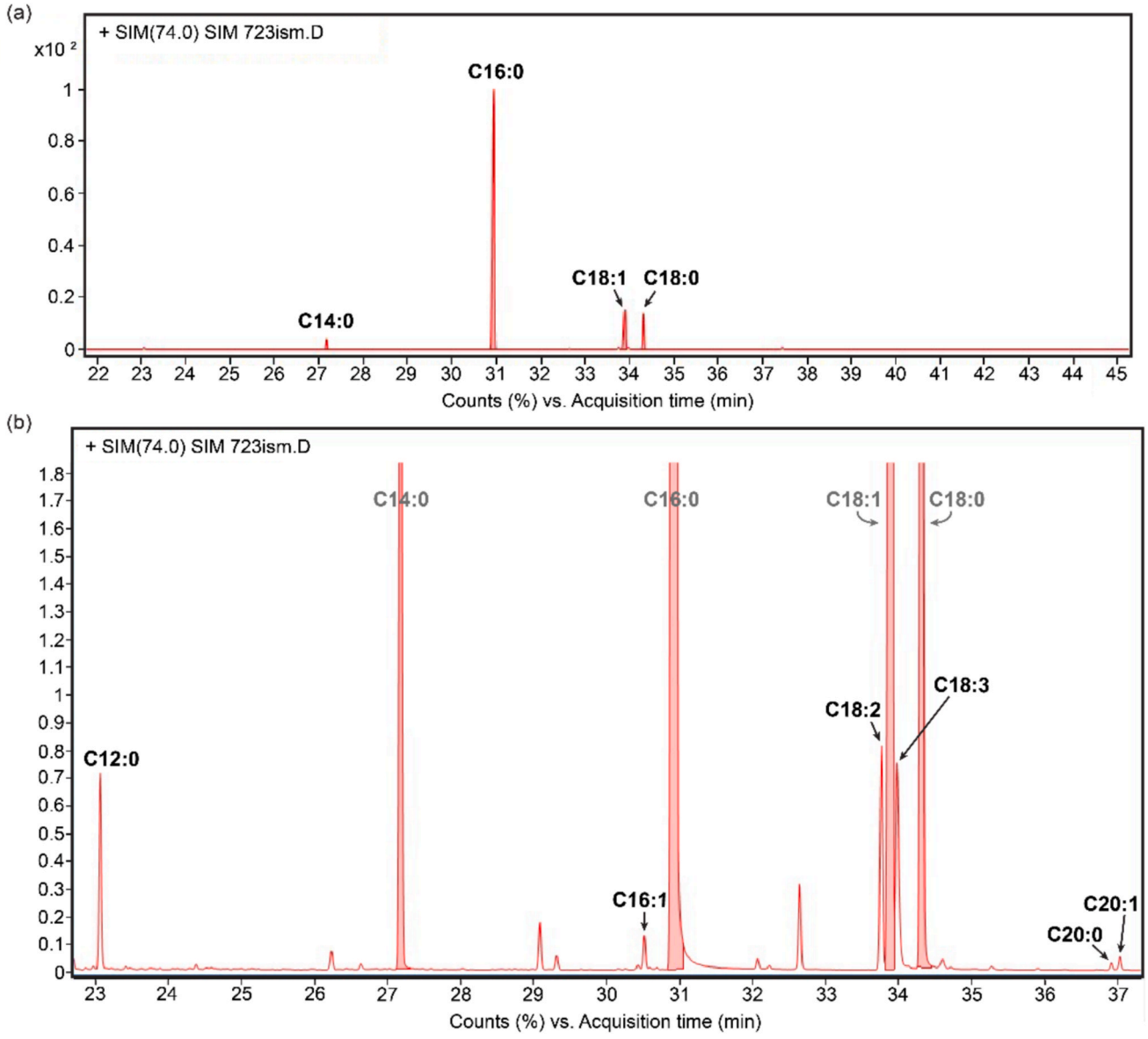


**Fig. 5.** GC–MS chromatogram of FAME (*m/z* 74) in Malaysia biodiesel. (a) shows normalized chromatogram with dominant FAME peaks. (b) presents a zoomed-in view of the minor peaks (non-normalized) for clarity.

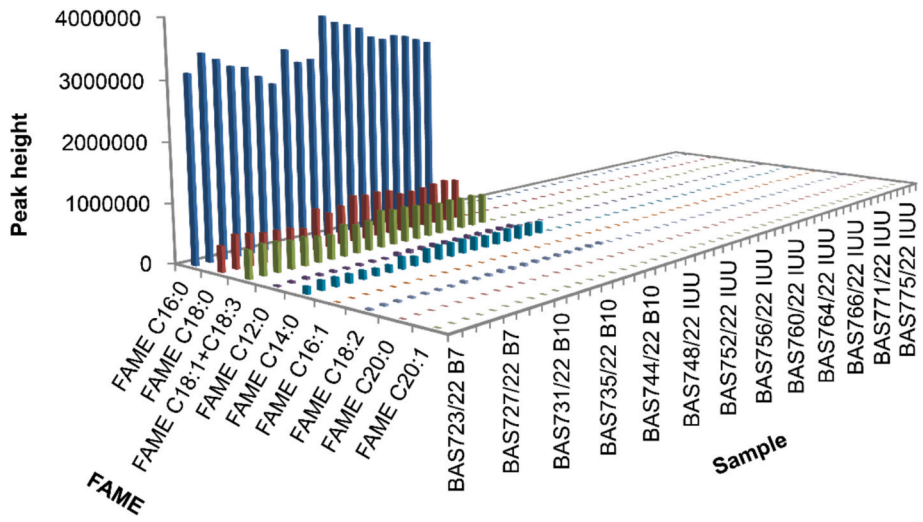


**Fig. 6.** FAMEs content in biodiesel (B7 and B10) and IUU samples.

linolenate (FAME C18:3). The sources of FAMEs in biodiesel can be plant-based, such as soybean oil [40] and canola oil [41], or derived from used animal fats and waste frying oils [42]. In the current work, a total of 10 distinct FAMEs were identified in the C12–C20 range across the biodiesel samples, with FAME C16:0 and FAME C18:1 as the dominant species (Fig. 5). The retention time of FAME C16:0 eluting at approximately 30 min and FAME C18:1 at 33 min agree with the literature [43]. The major FAME compounds were C14:0, C16:0, C18:0, and C18:1, while those present in lower abundance included C12:0, C18:2, and C18:3. Trace levels of FAME C16:1, FAME C20:0, and FAME C20:1 were also detected. For quantification purposes, FAME C18:1 and FAME C18:3 were combined as FAME C18:1 + FAME C18:3 due to the proximity.

Fig. 6 shows the composition of prominent FAME content in biodiesel (B7 and B10) and IUU samples. A total of 6 distinct FAMEs were identified, with FAME C16:0 and FAME C18:0 emerging as the dominant species, followed by FAME C18:1 + C18:3. Saturated FAMEs C16:0 and C18:0 were found to be dominant in biodiesel feedstock originating from animal or mix oil [7,44,45], while soybean is high in FAME C18:2 [46] and canola is dominated by FAME C18:1 [47]. Therefore, it can be suggested that the feedstock for Malaysia biodiesel B7 and B10 originated from animal or waste frying oil [7,44,45].

Based on the detailed probe into the profile of ion chromatograms of hydrocarbon and FAME compounds, together with the ratio analysis, confirmed that IUU samples and biodiesel samples were significantly different. However, the differences between the biodiesels B7 and B10 cannot be established based on the hydrocarbon and FAME profiles, as well as the ratios. The same can be said for IUU samples, where the differences among the IUU samples cannot be established based on the chromatogram and ratios. As such, to statistically confirm the differences between biodiesel and IUU and further probe into the differences between the biodiesel (B7 and B10) and IUU samples, multivariate analysis was employed.

### 4.3. *Multivariate analysis*

To further explore the similarity and differences among the biodiesel and between IUU samples, multivariate analysis HCA and PCA were employed.

As depicted in Fig. 7(a), the HCA dendrogram reveals that biodiesel

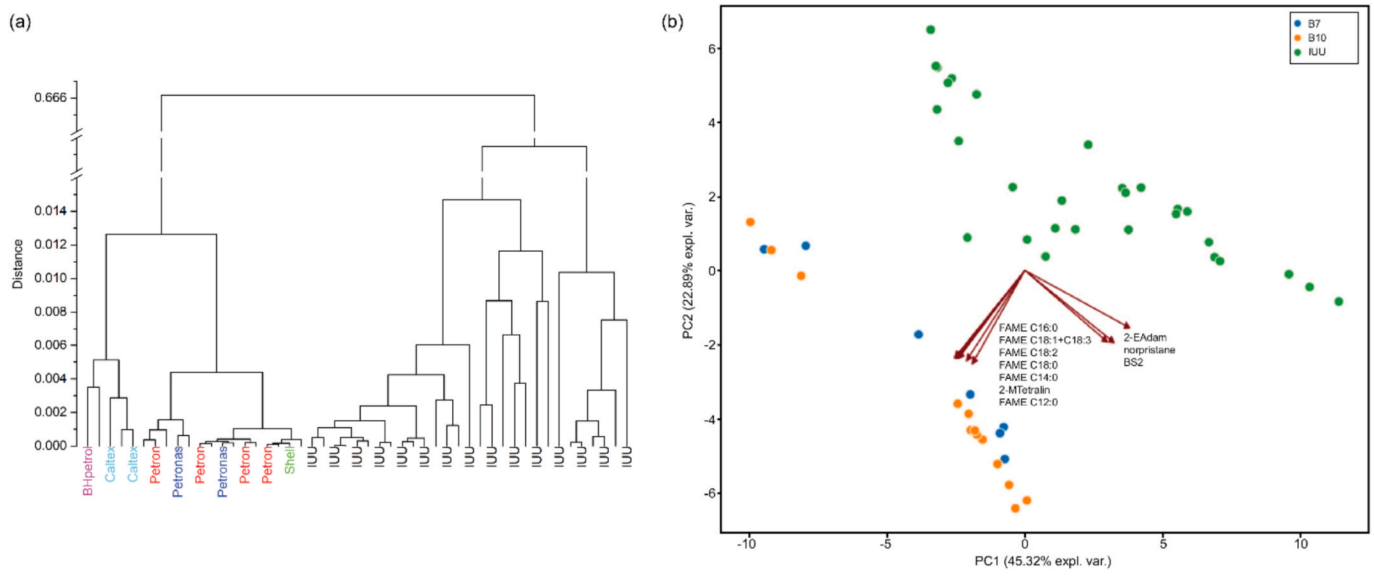


**Fig. 7.** (a) Dendogram of biodiesel and IUU samples. (b) PCA score biplot from PC1 vs PC2 of B7, B10 and IUU samples. The top ten relevant variables for the differentiation of B7 and B10 vs. IUU samples are depicted.

blends B7 and B10 form tight clusters, which suggests their group are nearly identical. IUU samples form a separate branch, confirming they are chemically different.

In order to further assess this clustering, PCA was also performed to support the HCA. Fig. 7(b) shows the scores plot of the first two principal components in the form of a biplot, which additionally depicts the vectors for the ten most relevant variables that differentiate Malaysia B7 and B10 samples from the IUU samples. Besides the FAME compounds, hydrocarbon compounds such as 2-methyltetralin (2-MTetralin), 2-ethyladamantane (2-EAdam), norpristane and BS2 were found to contribute to the differences between biodiesel and IUU samples. A heatmap, based on the PCA of all samples, using only the top ten variables, is shown in Fig. S6 in the Supplementary Material. The observed differences in B7, B10, and IUU samples, particularly in the FAME profiles, are likely attributable to variations in feedstock types [7], mineral oil composition, or blending formulation [8,48]. The PCA biplot with the top ten variables to discriminate between B7 and B10 is shown in Fig. S7.

Overall, both HCA and PCA analyses demonstrate the feasibility of distinguishing B7 and B10 biodiesel types sold in Malaysia to IUU samples based on their chemical compositions. However, the multivariate HCA and PCA analysis could not distinguish differences between biodiesel B7 and B10 based on FAME or hydrocarbon composition.

## 5. Conclusion

In summary, the integration of GC-FID and GC–MS chromatographic methods allows the profiling of both hydrocarbon and FAME compounds in forensic differentiation between biodiesel from IUU samples. These findings support the application of chromatographic-based methods in maritime law enforcement, enabling the identification and differentiation of fuel origin and enhancing efforts to combat fuel smuggling associated with IUU fishing activities. GC-FID screening analysis demonstrated that both biodiesel B7 and B10 hydrocarbons ranged from C10 to C29, while the IUU samples had a hydrocarbon range between C10 to C33. The hydrocarbon profiles of biodiesel samples showed additional peaks which were absent in IUU samples. The additional peaks in biodiesel samples highlighted the presence of FAME compounds. Furthermore, ratios of isoprenoids C17/pri, C18/phy and pri/phy were further helpful in distinguishing the biodiesel and IUU samples. The GC–MS analysis is used to further probe into specific compounds that can distinguish biodiesel and IUU samples. The ion chromatograms revealed five hydrocarbon biomarker groups: BS, adamantanes, isoprenoids, FAME and PAH compounds (C1-dibenzothiophenes and C1-fluoranthenes/pyrenes), which showed characteristic differences between biodiesel and IUU samples, further confirmed by their compound ratios. The FAME content in the biodiesel samples revealed ten distinct FAMEs, with methyl palmitate (C16:0) and methyl oleate (C18:1) as the dominant constituents and again with near to no presence in IUU samples. The composition of FAME revealed that the Malaysia biodiesel feedstock is from animal and/or waste frying oil.

Both chromatographic techniques were found to be useful in investigating the differences between biodiesel and IUU samples based on the hydrocarbon profile and marker compounds. However, the gas chromatographic technique alone could not explore the differences between the biodiesels B7 and B10. Multivariate statistical techniques were employed to distinguish further the differences between biodiesel and IUU samples and to probe the differences among biodiesels. Multivariate statistical analysis, HCA and PCA, effectively classified B7 and B10 samples into two distinct clusters, underscoring the compositional differences between these blends. This integrative chromatographic approach, together with a multivariate approach, supports maritime enforcement operations by providing a scientific tool that authenticates fuel origin and differentiates seized fuels from domestically distributed biodiesel. Despite the promising findings, this study is not without limitations. A limited sample scope, as only selected brands of Malaysia biodiesel were analyzed, and a small number of IUU samples, limiting the generalizability of the findings. Additionally, the study did not consider the impact of temporal degradation on fuel composition, nor did it employ multiple multivariate models for enhanced classification. These factors suggest that while the findings are promising, further validation and methodological refinement are necessary for broader forensic and regulatory applications.

## CRediT authorship contribution statement

**Mohd Rashidi Abdull Manap:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Ananthy Retnam:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Norizah Abdul Rahman:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Investigation, Formal analysis, Data curation, Conceptualization. **Nurul Ain Mohammed:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Resources, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Nurul Zulaikha Rozlan:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Resources, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Hui Juen Sew:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Resources, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Ramizah Azis:** Writing – review & editing, Writing – original draft, Project administration, Methodology, Investigation. **Noor Hazfalinda Hamzah:** Writing – original draft, Funding acquisition. **Philipp Weller:** Writing – original draft, Validation, Methodology, Data curation.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fuel.2025.137037.

## Data availability

Data will be made available on request.

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