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Forensic identification of adulterated petroleum fuels (diesel, Pertalite, and Pertamax) through GC-MS fingerprinting and physicochemical analysis

Sofia Anita*, M Zikri Arafi
Department of Chemistry, Universitas Riau, Pekanbaru 28293, Indonesia

ABSTRACT

A Fuel adulteration is a form of petroleum product fraud that can cause significant economic losses to consumers and adversely affect vehicle engine performance. This study aims to investigate the chemical composition and determine the characteristics of adulterated diesel, Pertalite, and Pertamax fuels. A qualitative analytical approach was employed by comparing adulterated fuel samples with their corresponding unadulterated counterparts. Gas chromatography-mass spectrometry (GC-MS) was used to identify the chemical constituents, while flash point and viscosity measurements were performed to evaluate their physicochemical properties. The GC-MS results revealed variations in alkane chain distributions and the presence of sulfur-containing compounds, aromatic hydrocarbons, naphthalene, and benzene derivatives. The identified hydrocarbon profiles indicated the presence of kerosene and diesel fuel components in the adulterated samples. Furthermore, the physicochemical characterization revealed differences in flash point and viscosity between pure and adulterated fuels. The reference flash point specifications for diesel, Pertalite, and Pertamax were 52°C, 23.5°C, and 27.5°C, respectively, while their corresponding viscosity specifications were 2 – 4.2 mPa·s, 0.4 – 0.6 mPa·s, and 0.5 – 0.7 mPa·s. In comparison, the measured flash points of the pure fuel samples were 52°C, 25.5°C, and 29.5°C, with corresponding viscosity values of 0.065 mPa·s, 0.725 mPa·s, and 0.675 mPa·s, respectively. The adulterated samples exhibited flash points of 74°C, 52°C, and 38°C, accompanied by viscosity values of 3.96 mPa·s, 4.45 mPa·s, and 10.16 mPa·s, respectively.

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*Corresponding Author
E-mail address: sofia.anita@lecturer.unri.ac.id

1. INTRODUCTION

Fuel adulteration is a form of petroleum product misuse involving the deliberate blending of commercial fuels with other substances to increase their volume and generate substantial financial profits [1]. According to the Indonesian Dictionary (Kamus Besar Bahasa Indonesia, KBBI), adulteration refers to the practice of mixing an original product with other materials, thereby reducing its purity or authenticity. In the context of petroleum products, fuel adulteration involves the unauthorized mixing of commercially available fuels, such as diesel, premium gasoline, Pertalite, and Pertamax, with other petroleum-based substances or lower-quality materials for economic gain. The adulterated products are subsequently distributed and sold at lower prices, particularly in areas experiencing fuel shortages or located far from official fuel distribution stations [2]. It may compromise fuel quality, deviate from established specifications, and potentially affect vehicle engine performance.

Gasoline and diesel are among the most frequently adulterated petroleum fuels, primarily because they are widely used by the general public. Kerosene, on the other hand, is often misused as an adulterant in gasoline and diesel due to its relatively low cost and subsidized status in Indonesia. The economic incentive associated with the price difference between subsidized kerosene and commercial fuels may encourage its unauthorized use as a blending component. Such practices can alter the chemical composition and physicochemical properties of the original fuels, potentially resulting in deviations from established fuel quality specifications and adversely affecting engine performance [3].

This situation may be exploited by unscrupulous informal fuel vendors who blend higher-quality and lower-quality fuels and sell the resulting mixtures at prices comparable to those of premium fuels. Furthermore, fuel stored in reused bottles or other non-standard containers may be contaminated with water or other foreign substances, potentially compromising fuel quality and causing deviations from vehicle engine specifications [4].

2. RESEARCH METHODS

Suspected of adulteration fuel samples were collected from one of gas stations in Pekanbaru, Riau Province, Indonesia. They were compared with original fuel samples used as reference materials. Each sample was transferred into separate 100 mL Erlenmeyer flasks. For gas chromatography–mass spectrometry (GC–MS) analysis, five drops of each fuel sample were transferred into individual 1.5 mL autosampler vials and diluted with n-hexane to the final volume. This preparation procedure was performed for all six fuel samples. In addition, pure n-hexane was prepared separately as a solvent blank for GC–MS analysis. Each prepared sample was subsequently homogenized using a vortex mixer to ensure uniform distribution of the sample components before instrumental analysis.

2.1. GC–MS Analysis of Fuel Chemical Composition

The vials containing the prepared fuel samples and solvent were placed in the GC–MS autosampler for injection and subsequent analysis. The GC–MS operating conditions were set as follows: injector temperature of 290°C, interface temperature of 290°C, ion source temperature of 230°C, constant flow rate of 1.0 ml/min, solvent delay of 3 min, mass scanning range of m/z 50 – 550, and injection volume of 1 µl. The oven temperature was initially set at 40°C with no initial holding time, followed by a temperature ramp of 15°C/min to a final temperature of 290°C, which was maintained for 24 min. The total chromatographic analysis time was 40.667 min. The GC–MS system was operated under these conditions to obtain chromatographic profiles and mass spectra for the identification and characterization of chemical constituents in the fuel samples.

The pristane-to-phytane (Pr/Ph) ratio is commonly used as a geochemical parameter to infer the redox conditions of ancient depositional environments, particularly in coal-bearing sedimentary systems. A Pr/Ph ratio greater than 1 generally indicates oxic (oxidizing) depositional conditions, whereas a ratio below 1 suggests anoxic (reducing) conditions [5].

The carbon preference index (CPI) is a geochemical parameter calculated from the relative abundance of odd- and even-numbered n-alkanes within a specific carbon-number range. It is commonly used to evaluate the maturity of organic matter and petroleum samples. In general, CPI values greater than 1.5 are associated with relatively low thermal maturity, whereas values below 1.5 may indicate a higher degree of thermal maturity. Herrera-Herrera et al. reported that CPI values below 1.0 are indicative of immature petroleum, while values above 1.0 suggest thermally mature petroleum [6]. These interpretations highlight the importance of considering CPI values in conjunction with other geochemical parameters when evaluating petroleum maturity.

The unresolved complex mixture (UCM) is identified through the interpretation of chromatographic data obtained from GC–MS analysis. UCM represents a complex mixture of branched and cyclic hydrocarbons that cannot be individually resolved into distinct chromatographic peaks. These unresolved compounds typically produce a characteristic hump-shaped baseline feature in the chromatogram, commonly referred to as the UCM envelope. In petroleum hydrocarbon analysis, a relatively small UCM envelope accompanied by clearly resolved n-alkane peaks may indicate limited weathering or degradation of the hydrocarbon mixture. Conversely, an extensive UCM envelope may suggest more advanced weathering, biodegradation, or the presence of complex petroleum-derived hydrocarbons [7].

2.2. Flash Point Analysis

The flash point of each fuel sample was determined using a flash point analyzer. The sample was transferred into the instrument's test cup up to the designated filling mark. The initial test parameters were set at 52°C and an atmospheric pressure of 101.3 kPa. The instrument was then initialized by pressing the Check button, followed by the Start button. The temperature lag setting was adjusted to 5°C before the sample approached the initial test temperature. Subsequent measurements were performed at 5°C intervals above the initial temperature until the instrument detected the flash point. Upon completion of the measurement, the instrument automatically provided the test result. Each sample was analyzed in duplicate to obtain the initial and final flash point readings.

2.3. Viscosity Measurement

The viscosity of each fuel sample was measured using a rotational viscometer. The sample was transferred into a 100 ml Erlenmeyer flask. The instrument was configured using spindle No. 3 at a rotational speed of 10 rpm, with a measurement duration of 30 s. The viscometer head was then lowered by loosening the adjustment lock located on the side of the instrument until the spindle was properly immersed in the sample. The measurement was initiated by pressing the Start button, and the reading was recorded once a stable value was obtained. Each sample was measured in duplicate. Measurements were considered acceptable when the torque reading was equal to or greater than 30% and the variation between consecutive readings did not exceed 5%, indicating an acceptable level of measurement stability.

3. RESULTS AND DISCUSSIONS

Analytical results of pure and adulterated fuel samples in term of viscosity, flash point, Pr/Ph, CPI, and UCM are presented in Table 1.

Table 1. Comparative fingerprint and physicochemical characteristics of pure and adulterated fuel samples fuel samples.

Fuel samples	Viscosity test	Flash point test	Pr/Ph	CPI	UCM
Pure diesel	+	+	2.09	1.19	Present
Adulterated diesel	−	−	3.59	1.11	Present
Pure Pertalite	+	+	2.94	1.07	Absent
Adulterated Pertalite	−	−	2.37	1.21	Present
Pure Pertamax	+	+	Absent	Absent	Absent
Adulterated Pertamax	−	−	2.97	1.09	Absent

Note: (+) Indicates that the measured parameter complies with Pertamina's fuel quality specifications, whereas (−) indicates non-compliance with the applicable Pertamina specifications.

The n- alkanes identified in pure diesel fuel ranged from C_9 to C_{26}. In addition, cycloalkanes, also known as naphthenes, which are cyclic hydrocarbon compounds, were detected, along with aromatic compounds, including naphthalene and its derivatives. Sulfur-containing compounds were also identified in the pure diesel sample. An increase in sulfur content in diesel fuel can reduce its calorific value; consequently, sulfur concentrations in diesel fuel are subject to strict regulatory limits [8].

3.1. GC–MS Analysis of Chemical Constituents in Fuel Sample

A comparison of the chromatographic profiles of pure and adulterated diesel samples revealed noticeable shifts in the fingerprint patterns of the chromatographic peaks in the adulterated diesel sample (see Figure 1). These differences indicate that the hydrocarbon fingerprint of the adulterated diesel differs from that of the pure diesel, providing evidence of possible fuel adulteration. Furthermore, a compound that was not detected in the pure diesel sample, namely o-cymene, was identified in the adulterated diesel sample. This compound belongs to the alkylbenzene group, which is among the hydrocarbon constituents commonly found in kerosene. The o-cymene also occurs naturally in essential oils derived from plants and is widely used as a solvent and chemical intermediate in various industrial

processes. The n-alkanes) in diesel fuel generally exhibit carbon numbers ranging from C_{11} to C_{22}. The observed changes in the n-alkane distribution, together with the altered chromatographic fingerprint and the detection of o-cymene, further support the possibility that an additional petroleum fraction was introduced into the adulterated diesel sample, resulting in changes in its hydrocarbon composition and alkane-chain distribution.

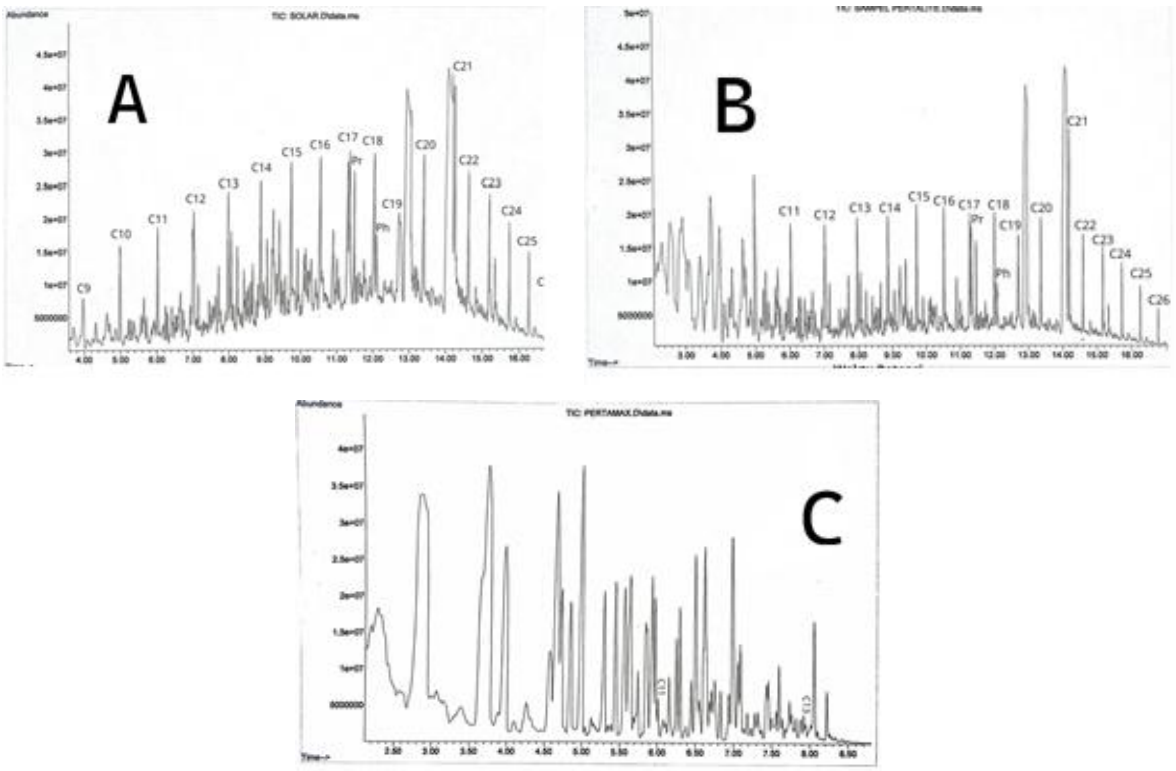


Figure 1. GC-MS chromatogram of pure diesel (A), aldurated diesel (B) and pure Pertalite (C).

The n- alkanes identified in the pure Pertalite sample ranged from C_8 to C_{24}. The most prominent chromatographic peak was observed at a retention time of 3.740 min, corresponding to benzene as the identified compound. In addition, the pure Pertalite sample contained cycloalkanes, aromatic hydrocarbons, sulphur-containing compounds, and xylenes. Gasoline consists of hundreds of naturally occurring hydrocarbon components, including benzene and toluene, as well as additional additives such as methyl tert-butyl ether (MTBE). These components can generally be classified into four major hydrocarbon groups: paraffins (normal and branched), cycloparaffins, olefins, and aromatics. The overall composition of gasoline varies depending on several factors, including crude oil origin and refining processes, such as distillation, alkylation, hydrocracking, and catalytic cracking [9].

Table 2. The thermal maturity characteristics of the pure and adulterated fuel samples based on selected petroleum geochemical parameters.

Sample	Pr/Ph	Pr/n-C17	Ph/n-C18	CPI
Pure diesel (solar)	2.09	0.756	0.409	1.19
Pure Pertalite	2.94	0.652	0.285	1.07
Adulterated diesel (solar)	3.59	0.706	0.412	1.11
Adulterated Pertalite	2.37	0.730	0.379	1.21
Adulterated Pertamax	2.97	0.680	0.262	1.09

Table 2 presents the thermal maturity characteristics of the pure and adulterated fuel samples based on selected petroleum geochemical parameters. The parameters include the isoprenoid-to-n-

alkane ratios, namely Pr/Ph, pristane/n-C_{17} (Pr/n-C_{17}), and phytane/n-C_{18} (Ph/n-C_{18}), as well as the CPI. These parameters were calculated from the integrated peak areas of the corresponding isoprenoid and n-alkane compounds obtained from the GC–MS chromatograms (see Figure 2).

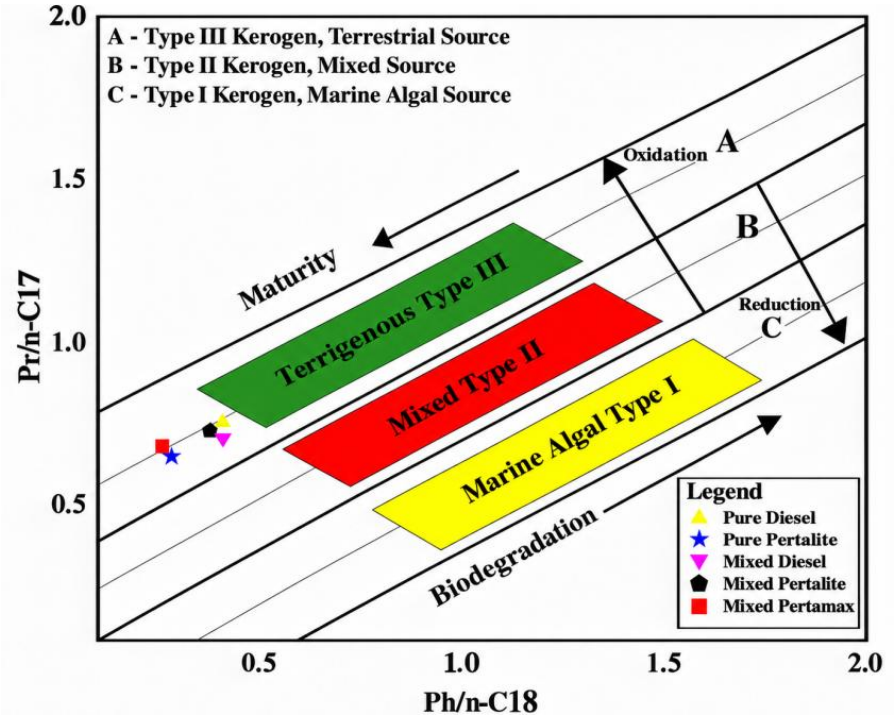


Figure 2. Cross-plot of pristane/n-C_{17} (Pr/n-C_{17}) versus phytane/n-C_{18} (Ph/n-C_{18}) ratios for evaluating depositional environments and organic matter input.

The results indicate a significant relationship between flash point and fuel adulteration, as an increase in the concentration of certain contaminants may result in an elevated flash point [10]. Furthermore, although flash point is not a direct determinant of diesel fuel quality, it is a critical safety parameter governing the handling and storage of diesel fuel [11]. A lower flash point indicates higher fuel volatility and an increased tendency to ignite, thereby posing a greater fire risk during handling and storage [12]. The flash point of pure and adulterated fuel oil samples measured at an Atmospheric Pressure of 101.3 kPa and a Minimum Temperature of 52°C are presented in Table 3.

Table 3. Flash point measurements of pure and adulterated fuel oil samples.

Fuel oil sample	Initial flash point (°C)	Final flash point (°C)
Pure diesel	47	52
Adulterated diesel	74	74
Pure Pertalite	20.5	25.5
Adulterated Pertalite	47	52
Pure Pertamax	24.5	29.5
Adulterated Pertamax	36	38

The heavier the petroleum fraction, the higher the flash point. Conversely, fuels with lower flash points exhibit higher volatility and are more readily ignited. When such adulterated fuel is used in a diesel engine, the presence of volatile gasoline components may alter the fuel's combustion characteristics (see Figure 3). Under certain operating conditions, premature ignition or abnormal combustion may occur before the piston reaches top dead center (TDC), potentially resulting in severe engine knocking and accelerated engine deterioration [13].

The viscosity measurement results for pure and adulterated fuel oil samples are presented in Table 4. The viscosity values of the pure and adulterated fuel samples were determined using a Lamy Rheology viscometer. These results reveal substantial differences in viscosity between the pure and adulterated fuel samples. The considerably higher viscosity values observed in the adulterated samples may be attributed to the addition of other petroleum fractions with higher molecular weights and greater viscosity. The incorporation of these heavier fractions alters the overall hydrocarbon composition of the fuel, consequently increasing its viscosity. These findings suggest that viscosity measurements can serve as a complementary physicochemical parameter for identifying potential fuel adulteration.

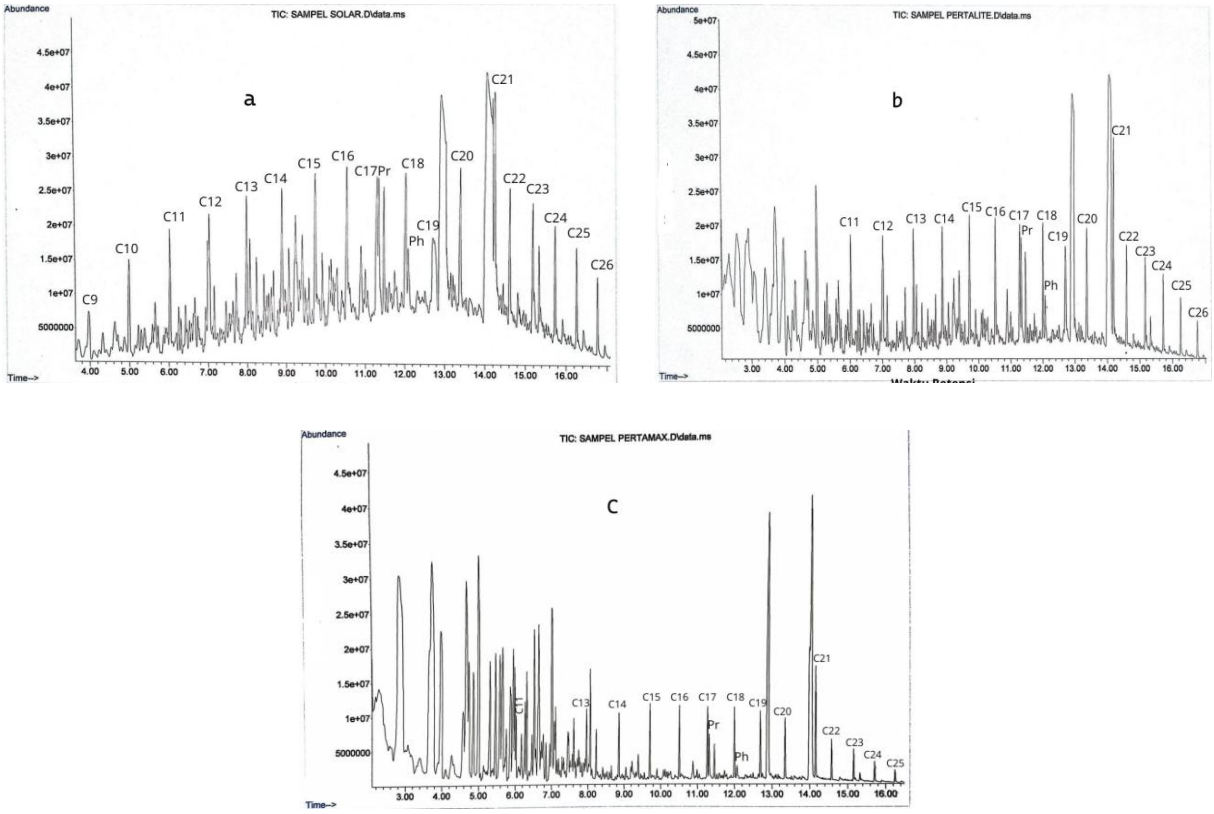


Figure 3. GC-MS Chromatogram of aldurated diesel (a), Pertalite (b) and Pertamax (c) showed Pr/Ph ratio, CPI and UCM.

Table 4. Viscosity measurements of fuel oil samples at 30 rpm for 30 second.

Fuel oil sample	Torque (mN·m)	Average viscosity (mPa·s)
Pure diesel	0.022	0.065
	0.021	
Adulterated diesel	0.021	3.960
	0.020	
Pure Pertalite	0.025	0.725
	0.026	
Adulterated Pertalite	0.023	4.450
	0.024	
Pure Pertamax	0.028	0.675
	0.029	
Adulterated Pertamax	0.027	10.160
	0.028	

3.2. Pristana/Phytana Ratio

Pristane and phytane, which belong to the isoprenoid hydrocarbon group, provide valuable information regarding depositional environments. Pristane is generally associated with oxic depositional conditions, whereas phytane is commonly associated with anoxic or reducing environments. In addition to providing insights into depositional conditions, the Pr/n-C_{17} and Ph/n-C_{18} ratios can also be used to evaluate organic matter sources [14]. The ratios of n-C_{17}/pristane and n-C_{18}/phytane are widely employed in organic geochemistry to assess the extent of hydrocarbon biodegradation and, with certain limitations, thermal maturity, particularly in petroleum systems and source-rock organic matter. A relatively high n-C_{17}/pristane ratio (> 1) may indicate that the organic matter or petroleum has undergone limited biodegradation, as normal alkanes are generally more abundant relative to isoprenoid hydrocarbons.

The depositional environments inferred for both pure and adulterated fuel samples were predominantly oxic, as indicated by Pr/Ph ratios greater than 1. This finding suggests that the source organic matter may have originated from chlorophyll-bearing organisms, which underwent degradation through aerobic bacterial activity [15]. Furthermore, the Pr/Ph ratios of the adulterated fuel samples were considerably higher than those of the corresponding pure fuel samples. This difference provides additional evidence of variations in their hydrocarbon compositions and further supports the possibility of fuel adulteration.

3.3. Carbon Preference Index (CPI)

According to Egbeola et al. [16], a CPI value of less than 1.0 indicates immature petroleum, whereas a CPI value greater than 1.0 suggests a higher degree of thermal maturity. Based on this interpretation, all samples, including both pure and adulterated fuel samples, exhibited CPI values greater than 1.0, suggesting that their hydrocarbon compositions may reflect mature petroleum characteristics.

The odd-over-even predominance (OEP) of n-alkanes provides additional information regarding organic matter sources. An odd-carbon-number predominance in n-alkanes with carbon numbers above n-C_{22} is generally associated with terrestrial higher-plant waxes, whereas even-carbon-number predominance among n-alkanes below n-C_{22} may indicate contributions from other organic matter sources. Chandru et al. [17] reported that even-carbon-number predominance in the n-C_{14} – n-C_{22} range may also be associated with terrestrial plant-derived organic matter. Furthermore, CPI values greater than 1.5 have been associated with relatively low thermal maturity and significant contributions from epicuticular waxes of terrestrial higher plants. In contrast, CPI values below 1.5 generally indicate greater thermal maturity and a reduced odd-carbon-number predominance [18].

3.4. Unresolved complex mixture (UCM)

An UCM was detected only in the pure diesel, adulterated diesel, and adulterated Pertalite samples. The presence of UCM is attributed to complex hydrocarbon compounds that cannot be adequately separated under the chromatographic conditions employed, resulting in a characteristic hump-shaped baseline in the chromatogram. In contrast, no UCM was observed in the chromatograms of pure Pertalite, pure Pertamax Turbo, or adulterated Pertamax samples. This absence may indicate a lower contribution of unresolved hydrocarbon components in these samples compared with those exhibiting UCM features.

Furthermore, the GC profiles revealed the presence of unresolved complex mixtures in several pure and adulterated fuel samples. The occurrence of UCM is commonly associated with complex petroleum-derived hydrocarbon mixtures and may indicate the presence of petrogenic hydrocarbon inputs or petroleum contamination [5].

4. CONCLUSION

The analytical results indicate that the fuel samples investigated in this study were adulterated with petroleum fractions comprising kerosene and diesel fuel, as evidenced by differences in their hydrocarbon compositions and chromatographic fingerprint profiles compared with those of the corresponding pure fuel samples. These compositional differences provided complementary evidence for distinguishing adulterated samples from their unadulterated counterparts and interpreting changes in

their chemical characteristics. Comparing chemical profiles with reference samples also helped contextualize the observed differences within each fuel type. The adulterated fuel samples exhibited substantial deviations in their physicochemical properties, particularly flash point and viscosity, relative to the fuel quality specifications established by Pertamina. These measurements complemented the chromatographic findings by revealing changes in the physical characteristics of the investigated samples, allowing chemical and physical evidence to be considered together. Taken together, these findings demonstrate the potential of combining chromatographic fingerprint analysis and physicochemical characterization as a complementary analytical approach for identifying fuel adulteration, supporting petroleum fuel quality assessment, and informing further forensic investigation of suspected adulterated fuels. Further evaluation using a broader range of reference fuels and controlled mixtures would help establish the applicability and limitations of this approach.

AUTHOR CONTRIBUTIONS

Sofia Anita: Conceptualization, methodology, research planning and coordination, supervision of experimental activities, validation, critical interpretation of GC-MS profiles and physicochemical results, evaluation of analytical findings, and writing—review and editing. M Zikri Arafi: Investigation, sample preparation, experimental measurements, documentation of experimental procedures, data organization and curation, formal analysis, comparative evaluation of fuel characteristics, preparation of figures and tables, visualization, and writing—original draft. Both authors contributed to discussing and interpreting the findings, evaluating their implications, critically revising the manuscript for clarity and scientific consistency, and approving the final version. Both authors agreed to be accountable for the accuracy and integrity of the work.

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DECLARATION OF AI USE

The authors declare that no AI tools were used in preparing this manuscript.

CONFLICT OF INTEREST

The authors declare no conflicts of interest.

DATA AVAILABILITY STATEMENT

All data supporting this study are included in the article. No separate datasets are available.

ETHICAL APPROVAL

Ethical approval was unnecessary, as no humans or animals were involved.

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