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Title:

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Tracing of the Chemical Signatures of Various Flammable Liquids from Common Crime Scene Substrates

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ABSTRACT

This study employs Gas Chromatography-Mass Spectrometry (GC-MS) to detect and differentiate ignitable liquid residues (ILRs) and pyrolysis products (PyPs) in fire debris samples, in accordance with the ASTM E1618-06 standard method. Accurate identification of ILRs is critical in fire investigations to determine whether accelerants were used. This can aid in distinguishing accidental fires from intentional fires. Hence, GC-MS analysis was conducted on fire debris collected from common household furnishing materials (substrates) to assess the influence of substrate interference on ILR detection. The results demonstrated that the recovery and identification of ILRs are influenced by their boiling point range and their degree of retention within the substrate matrix. Out of the 19 fire debris samples analyzed, ignitable liquid residues (ILRs) were successfully detected in 15. While, PyPs, often a source of analytical interference, were distinguished based on their characteristic chromatographic patterns. They were detected in 8 substrates. However, neither ILRs nor PyPs were found in 4 substrates. This study emphasizes the importance of robust analytical protocols to distinguish ILRs from pyrolysis compounds, especially in complex matrices. The findings contribute valuable insights towards improving the reliability and accuracy of fire debris analysis, thereby enhancing forensic fire investigations.

Keywords: ASTM E1618-06, Gas Chromatography-Mass Spectrometry (GC-MS), Ignitable Liquid Residues (ILRs), Forensic Fire Investigation, Pyrolysis Products (PyPs)

1. INTRODUCTION

Fire is a rapid exothermic process of combustion [1] that emits both light

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and heat. Combustion is a complex phenomenon which includes various chemical processes, such as thermal degradation, pyrolysis, and evaporation [2]. Fire plays a vital role in the maintenance of the ecological systems of the world [3]. Its positive impacts include heat generation and growth stimulation. In human usage, the main purpose of fire is to cook food [4]. Signaling and propulsion are its other usages. The negative impacts of fire include atmospheric pollution and soil erosion [4]. It is rapidly produced by devices such as lighters or matches. However, if it gets out of control, it creates disasters that pose risk to both life and property [5]. Ignitable liquids used as fuels for fire, are also termed as combustible and flammable liquids. When used to accelerate fire, they are known as accelerants [6]. The difference between flammable and combustible liquids is that liquids with a flash point below 100 °F (37.8 °C) and a vapor pressure not exceeding 40 psi (2,068 mmHg) at this temperature are classified as flammable liquids. Examples include fuels, such as gasoline, acetone, methyl ethyl ketone, isopropyl alcohol, paint thinner, and varnish [7, 8].

The flash point of combustible liquids is at or above 100ºF (37.8ºC). These include kerosene, oil-based paints, greases, and lubricants. The flash point is the lowest temperature at which a liquid releases enough vapor to form a combustible mixture with air [9]. The process of determining the flash point involves gradually heating the liquid in open air and noting the temperature at which it first ignites [10]. Accelerant is an agent, mostly an ignitable liquid, used to start or accelerate the spread of fire. Gasoline is usually used as fire accelerant [11, 12]. Accelerants are effectively used to execute crime and some produce explosions [13]. If ignitable liquid has been used to accelerate fire, only then it is considered as an accelerant in a fire investigation. Under usual circumstances, ignitable liquids are occasionally present on the fire scene. The investigator infers whether or not it is an accelerant. However, most of the time, accelerants comprise ignitable liquids [6].

The products obtained through the petroleum industry via the refinement of crude oil, such as gasoline, diesel, and kerosene, are petroleum-based [6, 14]. Whereas ignitable liquids extracted or acquired from other sources, for instance, acetone, ethanol, turpentine, limonene, and essential oils are non-petroleum-based. Turpentine is extracted from pine wood and consists of terpene-like compounds, such as alpha-pinene [15]. Both groups apply identical analytical methods, but their data interpretations vary. Commonly used ignitable liquids are based on petroleum products, and it is less frequent to have non-petroleum sources found [16]. Crude oil mainly consists of hydrocarbons [17, 18], while derivatives having sulfur, oxygen, and nitrogen may also be present [19]. Moreover, some of the metals are found at low levels [20]. All crude oils are composed of three main hydrocarbon classes (alkanes, aromatics and cycloalkanes) but vary in terms of their composition from one to another source [21].

Due to the different proportions of compound classes, the properties of crude oil vary. The abundance of alkanes within a given crude oil varies, including n-paraffins (straight chain) and isoparaffins (branched) [22]. The normal trend is that alkane proportions decrease with the increase of molecular weight. Thus, the more volatile fraction of crude oil has a relatively higher proportion of paraffinic compounds in comparison to the heavier fractions [23]. Fractional distillation plays a crucial role in the refining process [24, 25].

Substrates are the samples on which the analysis of fire debris is performed [26]. Their role begins during the manufacturing process. Some chemicals, such as petroleum-based polymers, are constituents of furniture and clothing. Petroleum products include flammable and combustible liquids, such as gasoline and diesel, as well as paint thinner and charcoal starter fluids. Thus, it is imperative to distinguish the presence of these liquids from the compounds produced by substrates [27]. Material burns at a fire scene, possibly by plentiful soaking of one or more ignitable liquids. Hence, it is sampled and taken to the laboratory by the fire analyst or investigator. Ignitable liquid residues are defined as the residues of ignitable liquids which are adsorbed onto burned substrates and show the characteristic chromatographic pattern of the respective liquid [28], when extracted and analyzed [29]. Forensic science is the use of scientific methodology and its application to investigate a crime [30]. Fire scene investigation is an important discipline of forensic sciences [31, 32]. It helps to determine whether a crime has been committed or not and what mode of operation the committer used [33]. During fire investigation, items of evidence collected comprise fire debris samples [34]. These are required to determine the presence of any ignitable liquid residues. At the scene, some information is gathered by scene investigation specialists. The results from the crime laboratory are reviewed administratively and technically [35].

Finally, reports are dispatched to the respective investigation agencies. When a fire is caused by someone with the intention to commit a crime, it is called arson [36]. An arson investigation may lead to the conviction of the arsonist. Arsons are usually investigated by police departments with the help of forensic science [37]. Fire debris samples are analyzed by forensic scientists, also termed fire debris analysts [38]. After analysis, a report is generated and presented to a court of law [39].

2. MATERIALS AND METHODS

2.1. Samples

Ignitable liquids and common household substrates were collected.

2.2. Apparatus

Screw-capped glass bottles were used for the storage of ignitable liquids. Polythene bags, clean metal polythene bags, and metal cans were used for the preservation of fire debris samples. A pair of stainless steel tongs was used to transfer the sample into the bag or can. Disposable aloe nitrile gloves were used to handle each sample in order to avoid any contamination due to perspiration or cross-contamination from different samples. Tripod stands and Bunsen burner were used to preparer fire debris. Standard Pyrex glassware, including beakers, screw-capped test tubes, and funnels were used for the preparation of samples. All laboratory glassware was cleaned and thoroughly rinsed with de-ionized water and dried in a dust-free environment prior to use. The various experimental steps for the tracing of the chemical signatures of various flammable liquids from common crime scene substrates are visualized in Figure 1.

2.3. Chemicals and Reagents

n-Pentane ≥ 99 % GC Grade 60489-5L-R Sigma-Aldrich USA

Methanol 99.8+ % GC Grade 423950025 Acros Organics German

2.4. Sampling

For the collection of ignitable liquids, samples were purchased from different locations in Lahore. Then, common household substrates were collected and burned with ignitable liquids. Table 1 illustrates the sample collection of common ignitable liquids.

Table 1. Sample Collection of Common Ignitable Liquids

Sr #	Sample Name	Sampling Place	Brand
1	Gasoline	Thokar Niaz Baig, Lahore	PSO
2	Diesel	Thokar Niaz Baig, Lahore	PSO
3	Kerosene	Thokar Niaz Baig, Lahore	Local
4	Paint Thinner-1	Thokar Niaz Baig, Lahore	Local
5	Paint Thinner-2	Mughalpura, Lahore	Local

The collection of common household substrates that were collected and burned with ignitable liquids is shown below in Table 2.

2.5. Sample Treatment

Samples were burned to generate pyrolysis products and their role in producing interference was evaluated. For burning, they were handled with tongs, so that more air was provided to the sample, which allowed it to burn better. The burner was turned on, and the flame was kept low. Each sample was burned in a way that one-third to two-thirds of it was burnt and then extinguished. It was done by placing the lid back on the cane containing the burnt sample. This starved the fire due to the lack of oxygen and eventually extinguished it.

2.6. Preparation of Fire Debris Samples

The fire debris samples were carried safely from the fire scene to the lab. In order to avoid the loss of ILRs and to avoid possible contamination from external sources, properly sealed polythene bags and metal cans were used. Each can and lid were decontaminated for at least 6 hours in the oven at more than 200°C before use.

Table 2. Common Household Substrates and Ignitable Liquids

Sr #	Substrate	Ignitable Liquids
1.	Pet Bottle	Gasoline, Kerosene
2.	Plastic (cutlery)	Gasoline, Kerosene
3.	Hose Pipe	Gasoline, Kerosene
4.	Match Stick	Gasoline, Kerosene
5.	Polystyrene Packaging Foam	Gasoline, Kerosene
6.	PVC Pipe	Gasoline, Kerosene
7.	Painted Wood	Gasoline, Kerosene
8.	Painted Hardboard	Gasoline, Kerosene

Sr #	Substrate	Ignitable Liquids
9.	Elastic	Gasoline, Kerosene
10.	Plastic Bag	Gasoline, Kerosene
11.	Hardboard	Gasoline, Kerosene
12.	Win board	Gasoline, Kerosene
13	Table Cloth	Gasoline, Kerosene
14	House Rag	Gasoline, Diesel
15	Cloth	Gasoline, Kerosene
16	Leather Shoe	Gasoline, Kerosene
17	Nylon Rope	Gasoline, Kerosene
18	Newspaper	Gasoline, Diesel
19	Acetate Sheet	Gasoline, Kerosene
20	Polyurethane Foam	Gasoline, Diesel

2.7. Extraction

A portion of the sample was selected for extraction. It was taken in a clean glass beaker and 10 ml of solvent (n-Pentane) was introduced and shaken for one minute. Any ILR present in the debris was extracted into the solvent.

2.8. Filtration

After the solvent was removed from the debris, it was filtered and transferred into a clean screw-capped test tube. Whatman No.1 filter paper was used for filtration.

2.9. Concentration

After filtration, the extract was concentrated by evaporating the solvent. Evaporation was performed as gently as possible, leaving the extract container under the hood and by regularly monitoring the progress of concentration.

2.10. Sample Preparation

1) Neat ignitable liquids were diluted with n-pentane using micropipettes to get a dilution of 2.5 µl / ml in separate Agilent GC autosampler vials labeled and closed tightly.
2) A mixture comprising equal quantities (0.5 ml) of gasoline, kerosene, and diesel was diluted with n-pentane to get 2.5 µl/ml in a tightly closed vial.

3) Substrate extracts were shifted to separately labeled Agilent GC autosampler vials using Pasteur pipettes and the vials were tightly closed.
4) n-pentane was taken as blank.

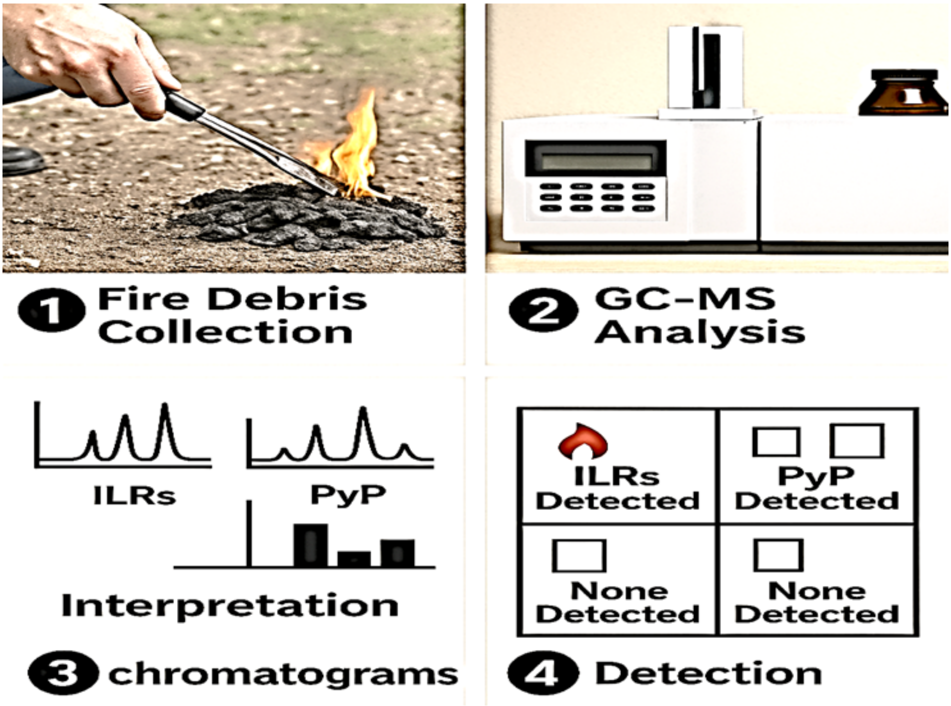

Figure 1. Graphical Illustration of Various Experimental Steps for the Tracing of Chemical Signatures of Flammable Liquids from Common Crime Scene Substrates

2.11. Instrumentation

Samples were analyzed using the Gas Chromatography-Mass Spectrometry (GC-MS Agilent 7890A-5975C system with 7693 autosampler, operating in EI mode at 70 eV. The software used was GC ChemStation (revision B.04.01 SP1) and MSD ChemStation (revision E.02.00 SP2) The Agilent 7890A-5975C GC-MS system with the 7693 autosampler is manufactured by Agilent Technologies Inc., a U.S.-based company. The instrument was% phenyl silicone capillary column HP-5MS (DB-5) (20 m x 180 µm x 0.18 µm). The carrier gas used was Helium at a flow rate of 1ml/min. The resulting total ion chromatogram and mass spectral data were evaluated in comparison to the known standard data. The identification of components was performed by the National Institute of Standards and Technology library (NIST08. L). The GC and MS parameters are mentioned below in Table 3.

Table 3. GC and MS Parameters

GC Parameters	
Parameter	SET Value
Line	Split (20:1)
Injector	250^0C
Initial Temperature	40^0C
Initial Time	4 minutes
Rate	10^0/min
Final Temperature	280^0C
Final time	2 minutes
Total run time	30 minutes
Solvent delay	2.4 minutes
MS Parameters	
Inlet	GC
Acquisition mode	Scan
Low mass	31
High mass	350

3. RESULTS AND DISCUSSION

Total Ion Chromatogram (TIC) shows the intensity of ion signals (Y-axis) vs. retention time (X-axis, in minutes), as depicted in Figure 2. Peaks represent individual compounds separated and detected over time. The TIC covers a time span of about 3 to 11 minutes, typical for volatile to semi-volatile compounds in gasoline. The retention time of the identified compounds via mass spectroscopy is given below in Table 4.

Table 4. Retention Time of Identified Compounds of Gasoline

Retention Time (approx.)	Compound Identified	Type
~3.5 min	Cyclohexane, 1,4-dimethyl	Light hydrocarbon
~4.5 min	Ethylbenzene	Aromatic hydrocarbon
~5.0 min	Octane, 2-methyl-	Branched alkane
~6.0 min	Benzene, 1-methyl-2-ethyl	Alkylated benzene

Retention Time (approx.)	Compound Identified	Type
~7.0–9.0 min	Benzene derivatives (e.g. trimethylbenzene isomers)	Polyalkylbenzenes
~10.0 min	Methylnaphthalene, Naphthalene	Polycyclic aromatics

C8 and C9 refer to hydrocarbons with 8 and 9 carbon atoms, respectively. Gasoline typically includes C4 to C12 hydrocarbons. Signature compounds for gasoline include:

i. *Alkanes*: Octane, 2-methyl

ii. *Aromatics*: Ethylbenzene, trimethylbenzenes

iii. *Naphthalenes*: Present at higher retention times, less volatile

The presence of branched alkanes and alkylated aromatics is characteristic of gasoline**.** The distribution of peaks across a wide volatility range confirms a mixture of low to mid-boiling compounds, a hallmark of gasoline**.** The presence of ethylbenzene, trimethylbenzenes, and methylnaphthalenes are key markers used in ASTM E1618-06 to classify ignitable liquids as gasoline-range products (G class)**.** The chromatogram confirms the presence of gasoline based on the identification of typical components and their retention times. Such patterns are used in the forensic analysis of fire debris to identify ILRs**.**

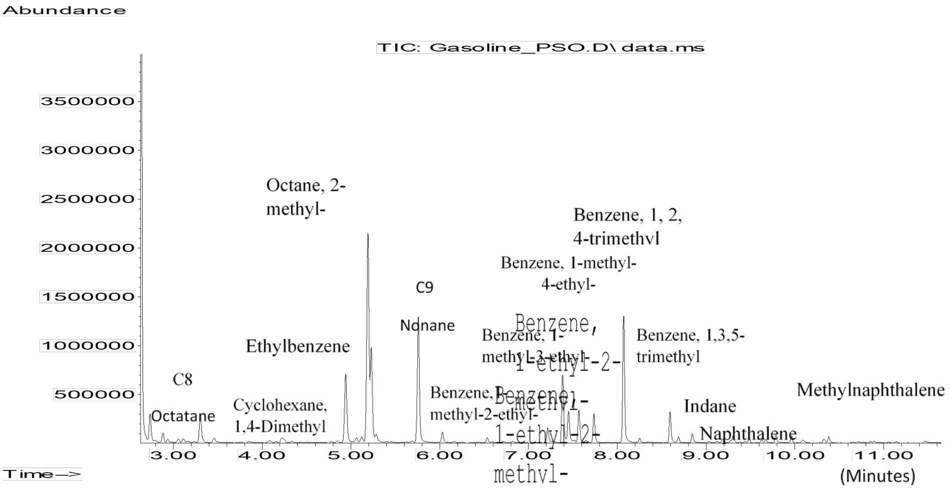

Figure 2. Total Ion Chromatogram of Gasoline

Figure 3 represents the TIC of kerosene obtained using GC-MS. The X-axis (Time) represents retention time in minutes. It shows how long each compound takes to travel through the GC column and reach the detector. The Y-axis shows the intensity of the ion signals detected, which is proportional to the amount of each compound present. Several peaks are annotated with compound names, indicating the chemical components identified in the kerosene sample, as given below in Table 5.

Table 5. Retention Times of the Identified Compounds of Kerosene

Retention Time (approx.)	Identified Compound	Type
~5.5 min	Cyclohexane, 1,4-Dimethyl	Branched cycloalkane, common in hydrocarbons
~6.5 min	Nonane, 3-Methyl	Branched alkane
~7.5 min	Nonane, 5-Methyl	Isomer of the above
~9.2 min	Benzene, 1,2,4-trimethyl	Aromatic hydrocarbon (a trimethylbenzene isomer)

Peaks labeled C_9-C_{14} correspond to n-alkanes with carbon numbers from C9 (nonane) to C_{14} (tetradecane). The largest peak appears to be C_{11}, suggesting that undecane ($C_{11}H_{24}$) is the most abundant straight-chain hydrocarbon in this kerosene sample. Kerosene is a complex mixture of hydrocarbons, mainly C_9-C_{16} alkanes and aromatics. The chromatogram confirms this by showing multiple peaks corresponding to linear and branched alkanes and aromatic hydrocarbons. The dominance of C_{10}-C_{12} peaks indicates that the sample likely contained medium-chain alkanes, common in jet fuels and lamp oils. The chromatogram provides a qualitative fingerprint of the kerosene sample, identifying key hydrocarbon constituents and showing their relative abundances.

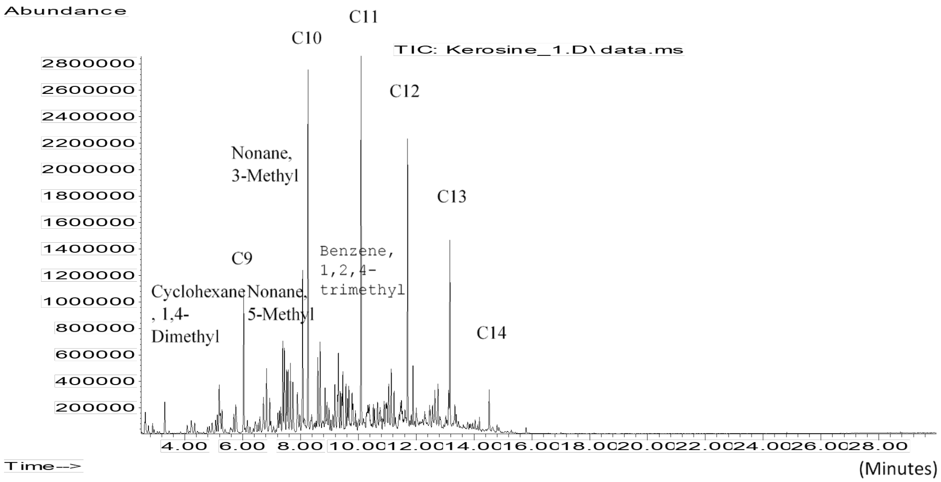

Figure 3. Total Ion Chromatogram of Kerosene

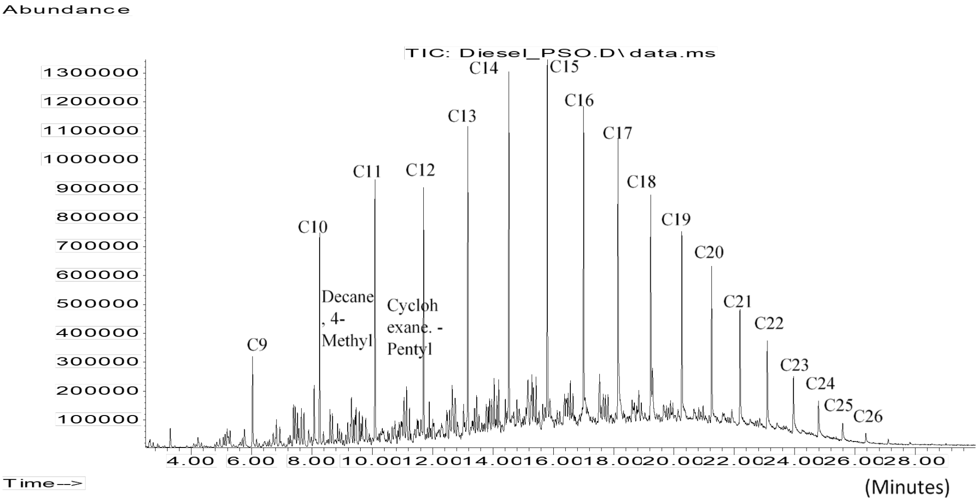

Figure 4. Total Ion Chromatogram of Diesel

In Figure 4, the peaks labeled C_9-C_{26} correspond to n-alkanes from nonane (C_9H_{20}) to hexacosane ($C_{26}H_{54}$). The dominant peaks are C_{13}-C_{18} which are also the most abundant, indicating that medium to long-chain hydrocarbons dominate the sample. Diesel usually contains C_{10}-C_{22} alkanes**.** This chromatogram confirms major peaks between C_{13}**-**C_{18}, which is common in high-boiling, middle-distillate fuels. The TIC of diesel reveals a broad and complex mixture of hydrocarbons, predominantly long-chain n-alkanes (C_{13}-C_{18}), as well as branched and cyclic hydrocarbons. These components are characteristic of diesel fuel, optimized for a higher boiling point, energy content, and engine performance**.**

Fig. 4 shows the TIC of burnt painted wood, analyzed using GC-MS. The chromatogram of burnt painted wood reveals the release of low molecular weight hydrocarbons (C_9-C_{12} alkanes) and synthetic paint-related compounds. The prominent peak (near 24 minutes) likely corresponds to a high-boiling degradation product from paint or resin, suggesting the combustion of complex organic materials. A large peak near 24 minutes with no label likely indicates that a major compound or a complex decomposition product could be a high-boiling paint additive, resin component, or an aromatic/phenolic compound from pyrolyzed wood/paint. The chromatogram of burnt painted wood reveals the release of low molecular weight hydrocarbons (C_9-C_{12} alkanes) and synthetic paint-related compounds. The prominent peak near 24 minutes likely corresponds to a high-boiling degradation product from paint or resin, suggesting the combustion of complex organic materials.

The TIC of burnt newspapers is illustrated in Figure 5. It provides a detailed chemical analysis of the volatile compounds released during burning.

- X-axis (Time): This shows the retention time (in minutes) which indicates when each compound eluted (came out) from the gas chromatograph.
- Y-axis (Abundance): This represents the intensity or quantity of the detected ions. Higher peaks indicate higher concentrations of the particular compound.
- Early peaks (around 8-10 minutes):
    - i) Octane, 2-Methyl: A light hydrocarbon often found in petroleum products.
    - ii) Decane: A straight-chain hydrocarbon present in fuels.
- Mid peaks (10-15 minutes):
    - i) Undecane, 2-methyl

  ii) Dodecane
  iii) Benzene, 1-methyl-3-: A derivative of benzene, which is aromatic and often results from incomplete combustion.
  iv) Tridecane: Another hydrocarbon commonly associated with ignitable liquids.
- Late peaks (after 16 minutes):
  i) Hexadecane: A long-chain hydrocarbon typical of heavier fuels or waxy substances.

The presence of alkanes including decane, undecane, dodecane, tridecane, and hexadecane suggests that the residues of hydrocarbons remain detectable in the burnt newspaper. Some of these compounds (e.g., decane and dodecane) are also found in common ignitable liquids (e.g., gasoline, kerosene). Benzene derivatives may indicate incomplete combustion or the presence of aromatic compounds from the burning material or potential accelerants. This chromatogram demonstrates that a range of hydrocarbons were released and detected from the burnt newspaper.

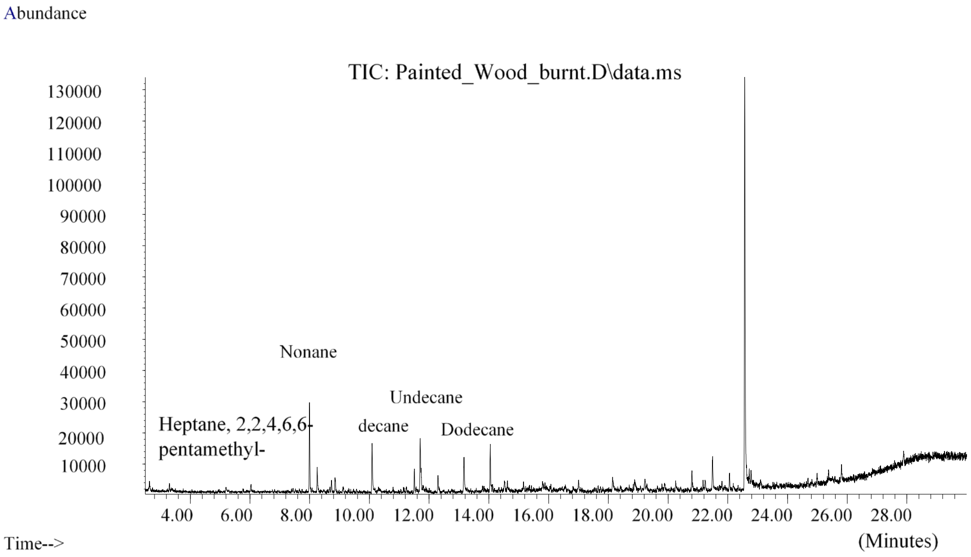

Figure 5. Total Ion Chromatogram (TIC) of Painted Wood (Burnt)

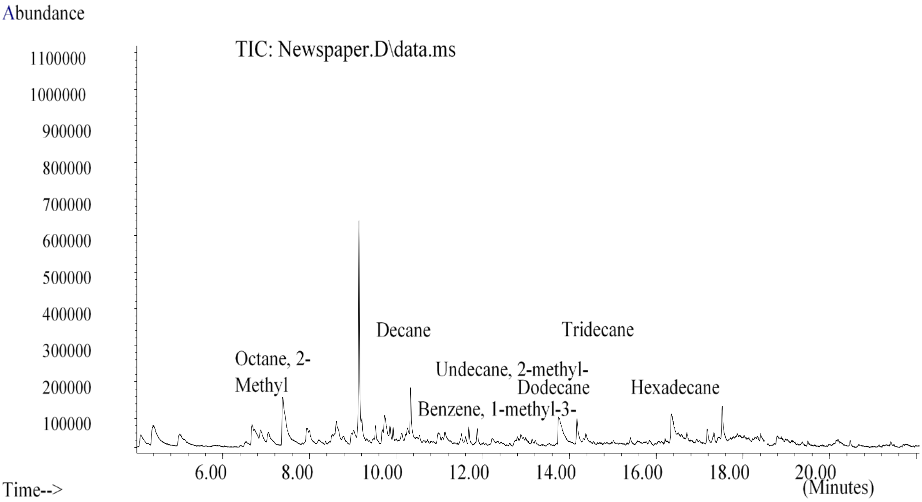

Figure 6. Total Ion Chromatogram (TIC) of Newspaper (Burnt)

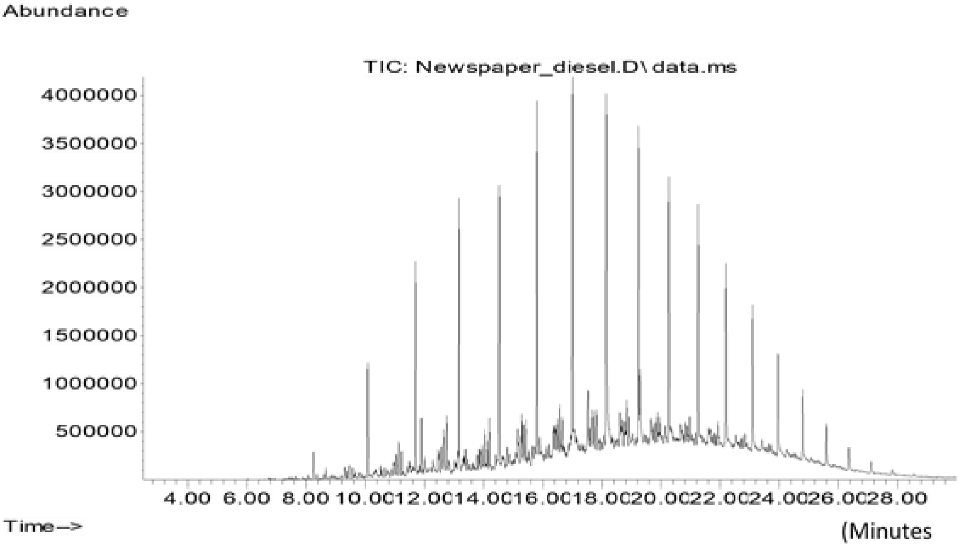

Figure 7. Total Ion Chromatogram (TIC) of Newspaper (Burnt with Diesel)

The TIC of newspaper (burnt) and the TIC of newspaper (burnt with diesel) are shown below in Figures 6 and 7 respectively.

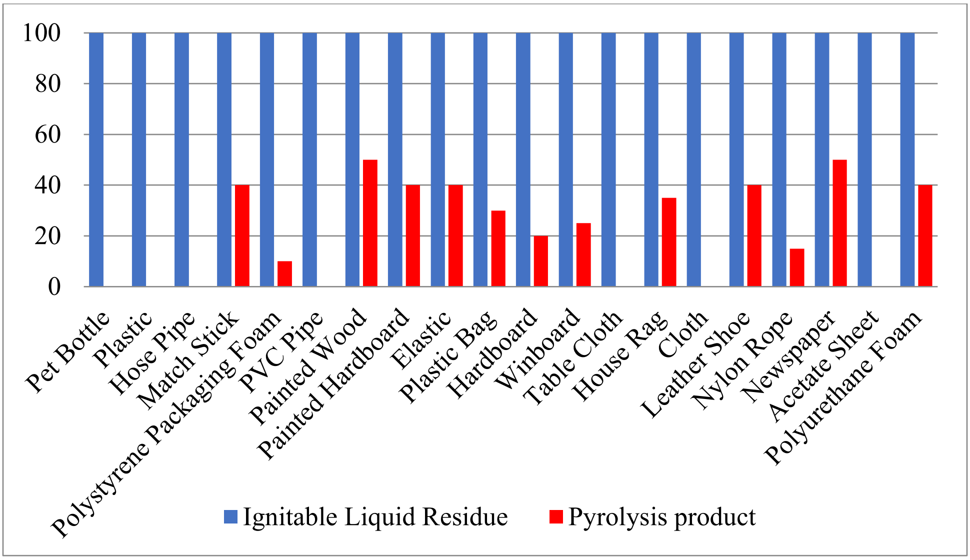

Figure 8. Graphical Representation of Ignitable Liquid Residues and Pyrolysis Products in the Debris of Substrates

In Figure 8 (a clustered column chart), the whole study is presented in terms of the percentage of ignitable liquid residues (ILRs) vs pyrolysis products (PyPs) from the fire debris of substrates while considering the percentage of the former as 100%. The substrates are shown on X-axis and ILRs and PyPs on Y-axis. Blue columns indicate ILRs and red columns show PyPs. The latter were detected in 13 substrates. Neither ILRs nor PyPs were found in 3 substrates.

4. CONCLUSION

This qualitative study successfully demonstrated the utility of GC-MS in fire debris analysis by evaluating the chromatographic profiles of neat ignitable liquids and common household substrates. A reference database of ignitable liquid chromatograms was established for comparative purposes. The analysis of burnt substrates revealed that the recovery of ILRs depends on both the type of substrate and the retention capacity of the ignitable liquid on the surface. The chromatographic patterns of ILRs generally matched those of their corresponding neat ignitable liquids, confirming their diagnostic value. However, the nature of the respective ignitable liquid significantly influenced the recovery efficiency. Light petroleum products such as gasoline, due to their high volatility and low boiling range, were less recoverable from fire debris as compared to heavier distillates, such as kerosene and diesel. In addition, PyPs generated from the combustion of substrates produced lower-abundance chromatographic peaks, which were predominantly composed of n-alkanes and iso-alkanes. Although these compounds can mimic some features of ILRs, they typically lack the complex and diagnostic peak patterns characteristic of ignitable liquids. Nevertheless, their presence can distort the chromatogram and obscure ILR signals, potentially leading to misinterpretation if not carefully distinguished.

CONFLICT OF INTEREST

The authors of the study have no financial or non-financial conflict of interest in the subject matter or materials discussed in this study.

DATA AVAILABILITY STATEMENT

Data of this study will be provided by corresponding author upon reasonable request.

FUNDING DETAILS

No funding has been received for this research.

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