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Comparing the Weathering Process of Gasoline in Selected Residues by HS-GC-MS Method

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Abstract

The main goal of the paper is to determine the changes in the selected gasoline residues distribution in samples of cotton carpets due to weathering. EVO 95 gasoline was used as a fire accelerator and a cotton carpet was used as a matrix. After various durations of weathering time (0-360 min), which was achieved by evaporation in laboratory conditions. Residual gasoline compounds were determined in the carpet fibers using gas phase extraction (HS) in combination with gas chromatography (GC) and mass spectrometry (MS). The biggest changes were in the reduction of the intensity of the most volatile compounds (alkylalkanes). Toluene decreased from a total proportion from 17.66% (0 min) to 5.74% (360 min). Minor changes occurred in the group of alkyl derivatives of benzene (1,3,5-trimethylbenzene), which is also considered as one from the fifteen marker compounds of the gasoline. Conversely, heavier compounds (naphthalene) showed an increase in a total proportion from 0.068% to 0.49%. These results provide experimental evidence and a strong theoretical basis that gasoline loses its intensity significantly during first 6 hours and it is difficult to identify. Analysis of the samples from the fire scene that were exposed to elevated temperatures leads to significantly different residue distributions compared to weathering under room temperatures. Our results indicate the need for rapid sampling and subsequent timely analysis of fire debris.

Keywords: cotton carpet; fire accelerator; fire debris; fire investigation; gasoline; HS-GC-MS; weathering

1 Introduction

Deliberately started fires are a widespread crime aimed at destroying objects, property, including the destruction of physical evidence. Cheap and widely available hydrocarbon-based liquids are often used as accelerators because they increase the speed and spread of a fire [1, 2]. The most common ignitable liquid (IL) found in arson is gasoline as it can be obtained easily and cheaply in most of the world. Even the use of a small amount of IL can cause significant monetary losses and endanger human lives [3, 4].

The identification of ILs is an important step in the analysis of the fire scene. Determining the presence of an IL provides valuable information about the cause of the fire, as investigating the presence of an IL can lead to the detection of a crime. While identifying the IL is useful for law enforcement agencies, it is not always easy. Conditions affecting the positive identification of the IL include the amount of sample that was allowed to evaporate and the substrate or matrix on which the sample was taken [5, 6].

Gasoline is a complex mixture that consist of a wide range of various volatile hydrocarbons, a complex mixture of branched, cyclic alkanes and aromatic compounds. Gasoline is a middle distillate of hydrocarbon-containing petroleum, mainly in the range of C4 to C12. It also contains various components that improve its properties (benzene and isooctane) as fuels. It is most often produced by fractional distillation of crude oil and treated by cracking and other processes [7, 8]. Characterizing the composition of gasoline and the proportion of the individual compounds is extremely difficult, as it depends on many factors, mainly the parameters of the crude oil from which the gasoline is produced [9, 10].

In cases where an IL has been used, a small amount of unburned fire accelerator may remain in the wreckage of the fire to detect and subsequently identify its presence. Among the several analytical systems available, GC-MS is one of the most widely used in forensic laboratories worldwide [11, 12]. It is used for separation as well as for qualitative and quantitative analysis of different types of complex organic samples [13]. It offers high resolution, sensitivity, selectivity, and specificity for the investigation of ILRs from fire debris [14, 15].

The chromatographic data are manually interpreted after the analysis by analysts or forensic chemists to determine if traces of ILs are present in the fire debris samples. This final evaluation step of the analysis is a potential problem as data interpretation is a particularly challenging task due to the extreme chemical diversity and complexity of analytes and matrices [16, 17].

Another challenge in fire investigation is a weathering process, which distorts the chromatographic profile due to evaporation or partial combustion, leading to the loss of more volatile compounds. When the sample evaporate, the peaks shift towards the later eluted fractions. This phenomenon is often described as a pattern of weathering [3]. Comparing the weathered sample with the library of unweathered sample mass spectra can help in interpreting the results of the analysis [18].

Changes in the concentration ratios of hydrocarbons such as benzene, toluene, ethylbenzene and xylenes (BTEX) are mainly caused by evaporation and dissolution processes. As stated in ASTM E1618, the presence of marker compounds is required to claim that the sample contains traces of gasoline in the samples. According to the American Society for Testing and Materials, these compounds must be present in order to reliably demonstrate the presence of gasoline [19, 20].

On the other hand, the ASTM E1618 standard does not specify the need for quantification, nor does it specify the thresholds of individual markers. Therefore, the presence of at least some of the above compounds is quite sufficient to demonstrate the presence of gasoline in the samples. To a large extent, this decision-making process is based on the experience of the analyst [21, 22].

When ILs are exposed to ambient conditions, the liquids can evaporate in a process called weathering. Of all the components in the liquid mixture, the most volatile components with the highest saturated vapor pressure evaporate the fastest. Weathering changes the relative amounts of components in the mixture, which makes it difficult for the analyst to compare weathered residues with unweathered samples of ILs. The weathering process takes place at any temperature ranging from room temperature to temperatures exceeding 1000 °C during a fire. The identification of gasoline and other mixed hydrocarbons in fire debris is the most complicated problem, as long-term exposure to high temperatures radically changes the chemical composition of gasoline [15, 23-25].

Controlled weathering almost always involves longer evaporation times at lower temperatures than under actual fire conditions and in no way captures the effects or sources of variation expected under real conditions such as pyrolysis. When a sample of gasoline is exposed to severe weathering, four compounds are most often missing when comparing the chromatograms, namely ethylbenzene, *m*-xylene, *p*-xylene, and *o*-xylene [21, 26, 27].

The aim is to determine the changes of selected gasoline compounds in cotton carpet samples due to different lengths of deliberate weathering under laboratory conditions. The assumption is that the more volatile gasoline compounds lose most of the signal intensity after a few hours, and after this time its chemical composition changes significantly.

2 Material and Methods

Fire accelerator

Gasoline (EVO 95, E10 Slovnaft, Bratislava) was used as a fire accelerator. Gasoline was obtained from a local petrol station and then at the volume of 5 mL was applied to the surface of the carpet. The same batch of gasoline was used to conduct the same condition and chemical composition.

Matrix

A woven carpet ARYA 05 measuring 80 × 150 cm, 100% cotton with a pile height of 0.5 cm, a fiber density of 1,400 g / m^2 was used as a matrix for capturing gasoline. The carpet was purchased from a local carpet store and then was cut to uniform samples of 5 × 5 cm.

Weathering

The samples were allowed to weather under laboratory conditions at 20 °C. Carpet fibers were removed at intervals of 0, 15, 30, 45, 60, 90, 120, 190, 240, 300 and 360 min. The designation was supplemented by a numerical data, where the number represents the weathering time. From each carpet, 3 fibers were taken one piece from two edges and one from the central part.

Analytical system

The analytical determination was based on the ASTM E1388 and ASTM E1618 methods. Boundary conditions were set and optimized based on calibration measurements and previous research [28-30]. Residual volatile compounds were obtained by gas phase extraction (static HS). The gas phase was subsequently analyzed by gas chromatography with mass detection (HS-GC-MS). A Headspace Autosampler 7697A (Agilent) with an HP7890A gas chromatograph (Agilent) and a VL MSD 5975C mass spectrometer (Agilent) was used.

The gas phase extraction took place at 60 °C for 15 min and the gas phase was metered into the chromatographic column. The volatiles were separated on an HP 5MS column (30 m, 0.25 mm, 0.25 μm, Agilent) with a temperature program of 40 °C for 4 min, a heating rate of 6 °C / min to 250 °C, with a carrier gas flow (He) 0.8 mL / min, in split mode 500 : 1. Mass detection was performed at 70 eV electron ionization and conditions: source temperature 200 °C, detector temperature 150 °C.

Sample evaluation

All peaks were identified by mass spectral data. The identity of each compound was then confirmed by searching their mass spectra in the NIST library. A total number of 65 samples were analysed and evaluated with 5 replicates of each time interval, supplemented with reference samples of clean gasoline and pure carpet.

Due to the complex properties of gasoline, only well-separated and symmetric peaks were selected for data analysis. For this reason, only those compounds that were determined for each sample were retained.

The reason for choosing three compounds is that we can imaginarily divide the chromatographic profile of gasoline into three areas. The first area ranging from 0 to 7 min consists of the most volatile compounds (toluene). The second is the transition area from 7 to 14 min, which consists of compounds (1,3,5-trimethylbenzene) that are not subject to such extensive weathering. The third area in the chromatographs consists of the most stable compounds (naphthalene) ranging from 14 min upwards. The comparison of these selected compounds was chosen precisely because of their representativeness for the given areas of the chromatograms.

Statistical evaluation

The peak areas of significant compounds that were the same for all samples were counted and then the percentage of the selected three compounds was calculated and subsequently averaged. This procedure was repeated for all differently weathered samples taken at the same time intervals. For the sample averages of each time interval, we calculated the standard deviation, and a trend line was plotted in the graphs. The statistical evaluation was performed using STATISTICA 12 software.

3 Results and Discussion

By comparing the obtained chromatograms of selected compounds of pure gasoline absorbed in the carpet fiber, different trends were demonstrated. Looking at the intensities of compounds obtained from unweathered samples and samples weathered for 360 minutes, a significant difference is apparent. The intensities of the individual compounds, especially the more volatile ones, i.e., up to a retention time of about 4 minutes, are greatly reduced. A representative of the category of the most volatile compounds is toluene, which is shown in Fig. 1. Comparing the amounts of the compounds determined, toluene showed a decrease from 17,67% (0 min) to 5,74% (360 min). When converted to an absolute decrease in terms of peak size, the decrease represents approx. 91,64%.

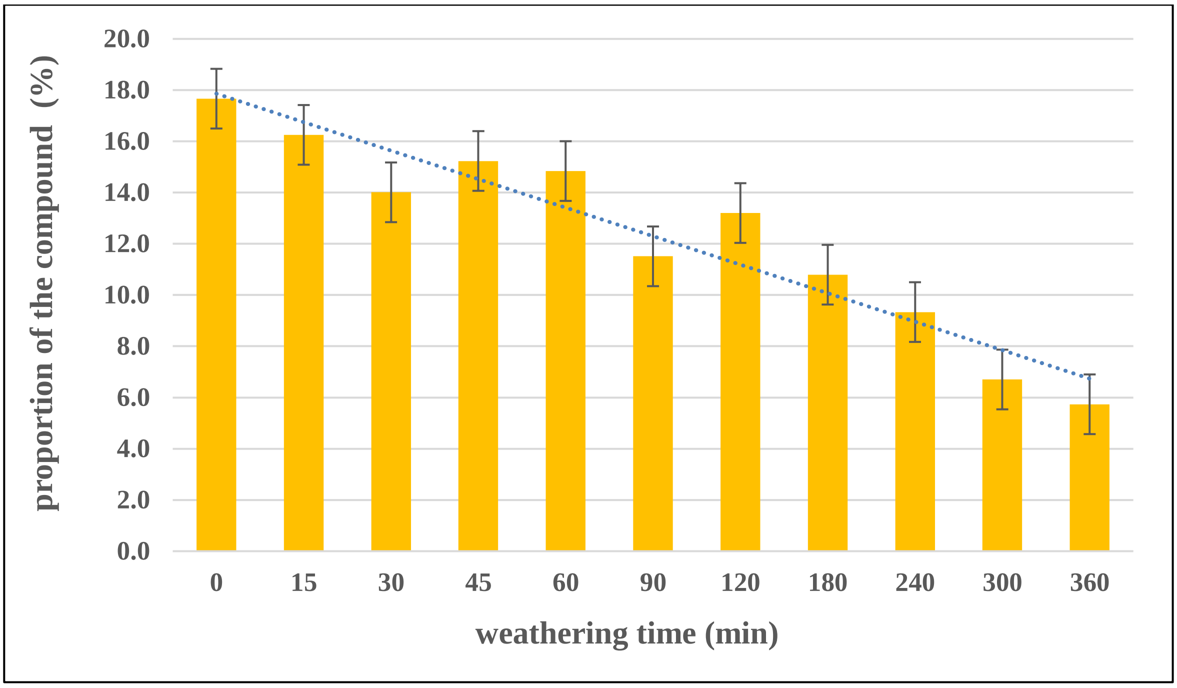

Fig. 1 Changes in the intensities of the toluene analytical signals at different weathered samples

The change occurs after about 5 minutes of retention time, where the differences in this area are no such significant. In this area, the individual intensities stabilize for different sample variants. Fig. 2 shows the compound 1,3,5-trimethylbenzene (TMB), which is also considered as one from the indicators of the gasoline. 1,3,5-TMB increased in 360 min by 1,61% over the reference sample, which is not a significant difference if we consider the proportion percentage from the total proportion of all compounds. When converted to an absolute decrease in terms of peak size, an increase appears here approx. 14,24%.

A similar finding was made by Chalmers et al. [31] who investigated the negative effects of weathering to which the samples were intentionally exposed. They found out that 1,3,5-TMB and ethylmethylbenzene were the least sensitive substances.

On the other hand, Willis et al. [15] also showed by the results that there was an increase in the marker compound 1,2,4-TMB. This is an interesting finding, as it was originally assumed that as they are marker compounds (trimethylbenzenes) they should be present in very similar amounts with minimal differences.

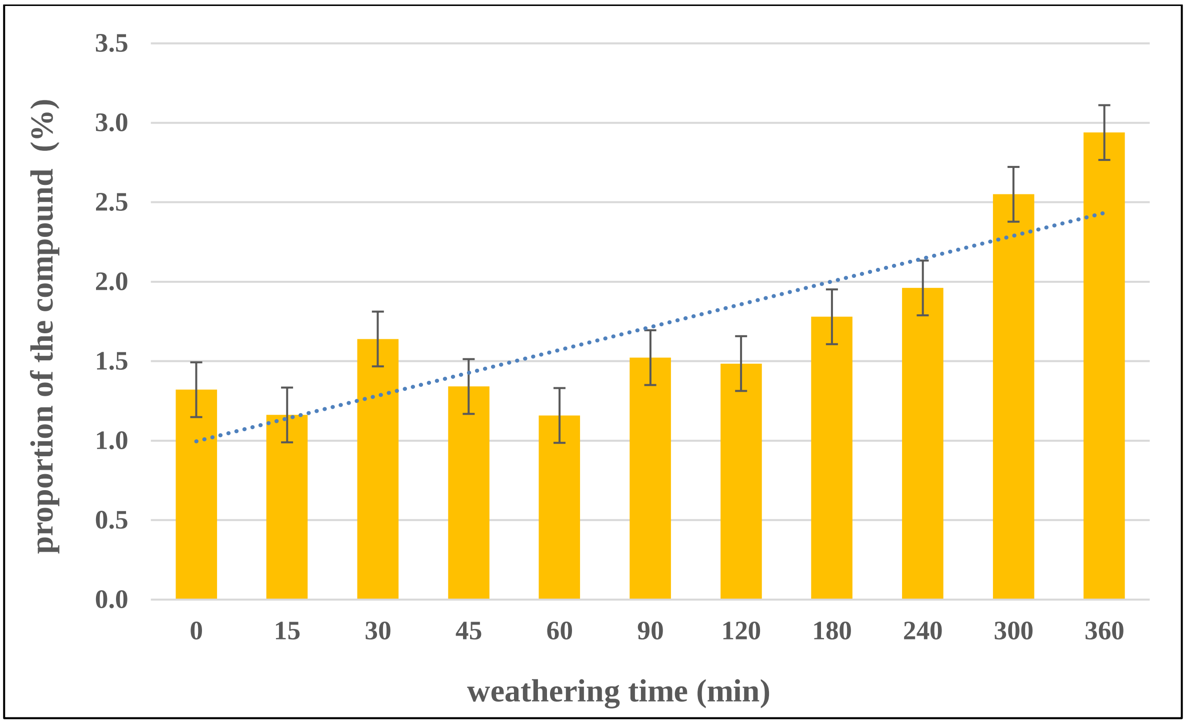

Fig. 2 Changes in the intensities of the 1,3,5 – trimethylbenzene analytical signals at different weathered samples

Fig. 3 shows the area of the chromatogram with a retention time of 15 min where the most stable compounds occur. A representant of this category of compounds with the highest vapor pressure is e.g., naphthalene. As can be seen, the intensity of naphthalene increases over time. If we compare the reference sample (0 min), or the sample weathered for 15 min with samples weathered for 300 min, or for 360 min, the naphthalene intensity increased approximately from 0.068% to 0.49%. If we convert this increase into an absolute increase in terms of peak size, the increase is approx. 52.6%.

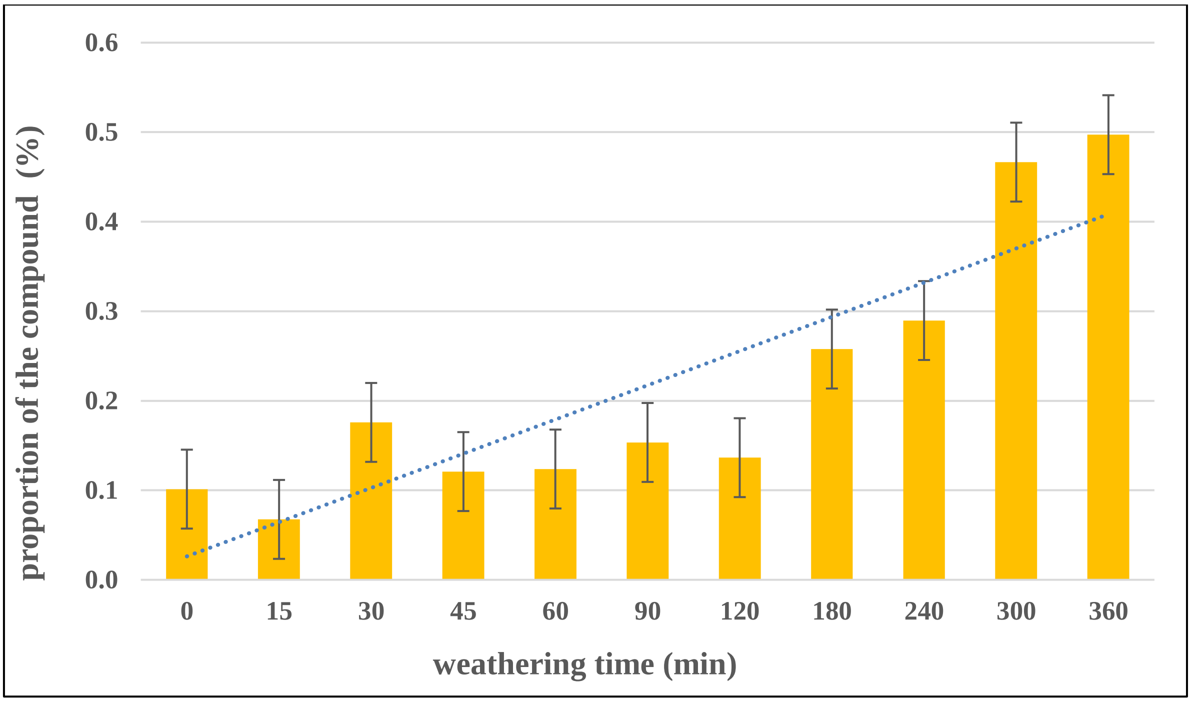

Fig. 3 Changes in the intensities of the naphthalene analytical signals at different weathered samples

As predicted by Ferreiro-Gonzalez et al. [32] the relative increase is due to the loss of volatiles, which evaporate faster. The most volatile hydrocarbons present in the sample do not overflow the headspace and release even the most volatile compound in larger quantities is easier and more efficient.

The same findings were made also by Willis et al. [15], who reported that the least volatile compounds, which eluted last in GC, showed a sharp increase in intensity in the later stages of weathering.

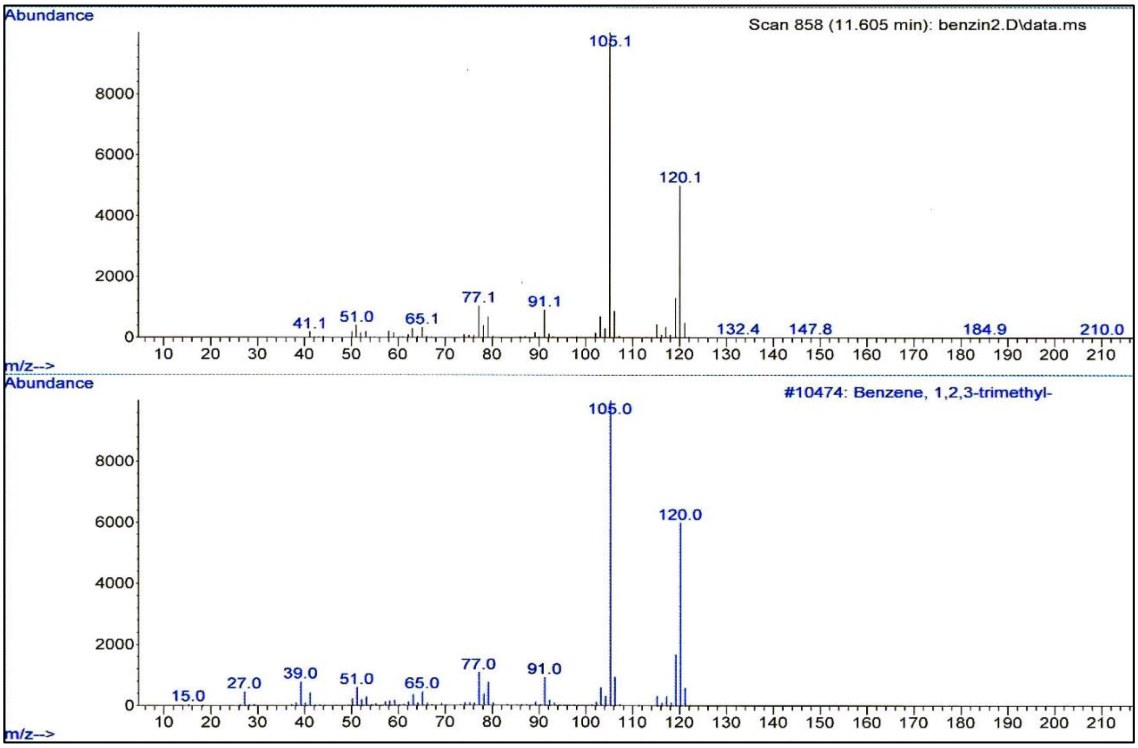

Fig. 4 Comparison of the mass spectrum of 1,2,3 – trimethylbenzene (up determined mass spectrum, down mass spectrum from the NIST library)

The identified peaks of the individual compounds were subsequently compared with the NIST spectrum library. Figure 4 shows the mass spectrum of 1,2,3-TMB.

Fig.5 shows a selected part of the chromatograms obtained by analysis of differently weathered samples. The most volatile gasoline compounds are shown, for example a group of aliphatic hydrocarbons and a C1 derivative of benzene (toluene). Different color curves represent different weathered sample variants. The black curves characterize the compounds determined by analysis of reference samples taken and sealed immediately after the addition of gasoline. The largest weathering time interval is represented by the purple color, which belongs to the samples weathered for 360 min.

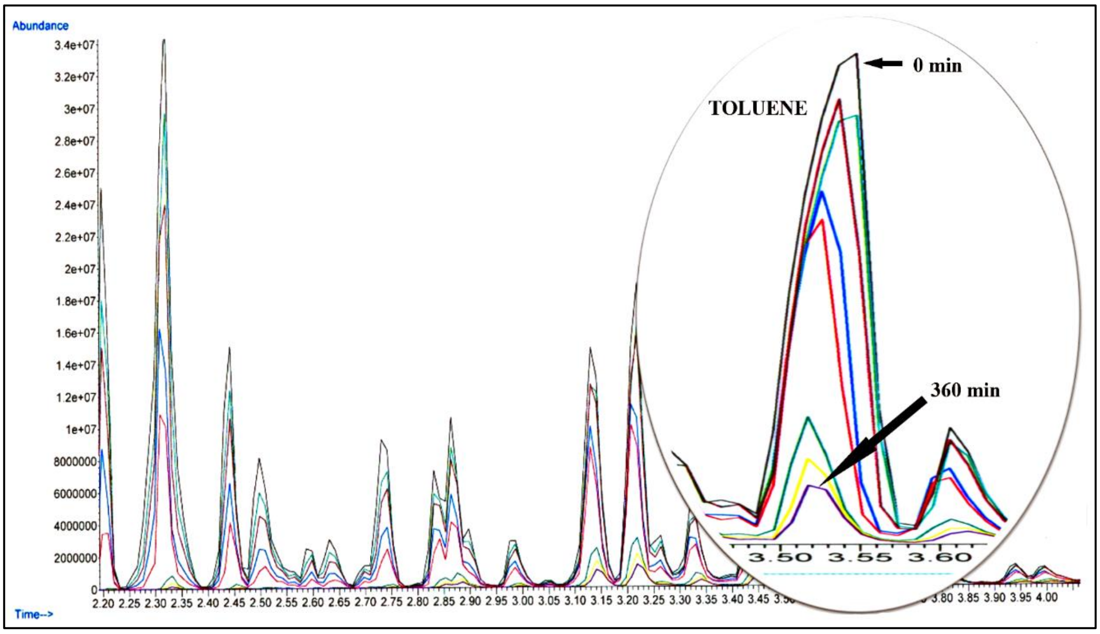

Fig. 5. Part of the chromatogram of lighter hydrocarbons with the retention time from 2 to 4 minute

It is possible to observe a trend on the chromatograms that over time (longer weathering) there is a decrease in intensities around volatile compounds, i.e., retention time approx. 2 - 5 min. Looking at the course of the individual chromatograms, it is possible to observe a significant difference between the intervals of 180 minutes (red color) and 240 minutes (green color). From the above, it can be assumed that gasoline loses most of its signal intensity during the weathering process after a time ranging from 3 to 4 hours.

Aliaño-González et al. [33] state that a significant change is expected after a time of 0 to 6 hours with a significant decrease in signal intensities, which means that the weathering process is most pronounced after the first few hours.

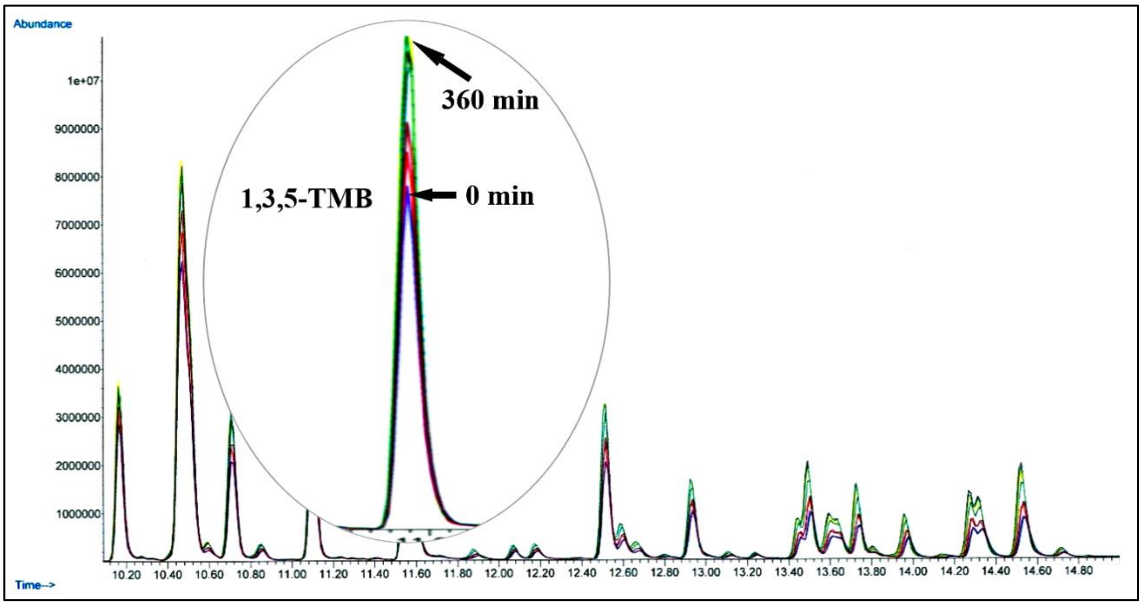

Fig. 6. Part of the chromatogram of more stable hydrocarbons with the retention time from 10 to 15 minute

For the transition area (Fig. 6), which also includes the above-mentioned gasoline indicator (1,3,5-trimethylbenzene), this change in intensity is minimal.

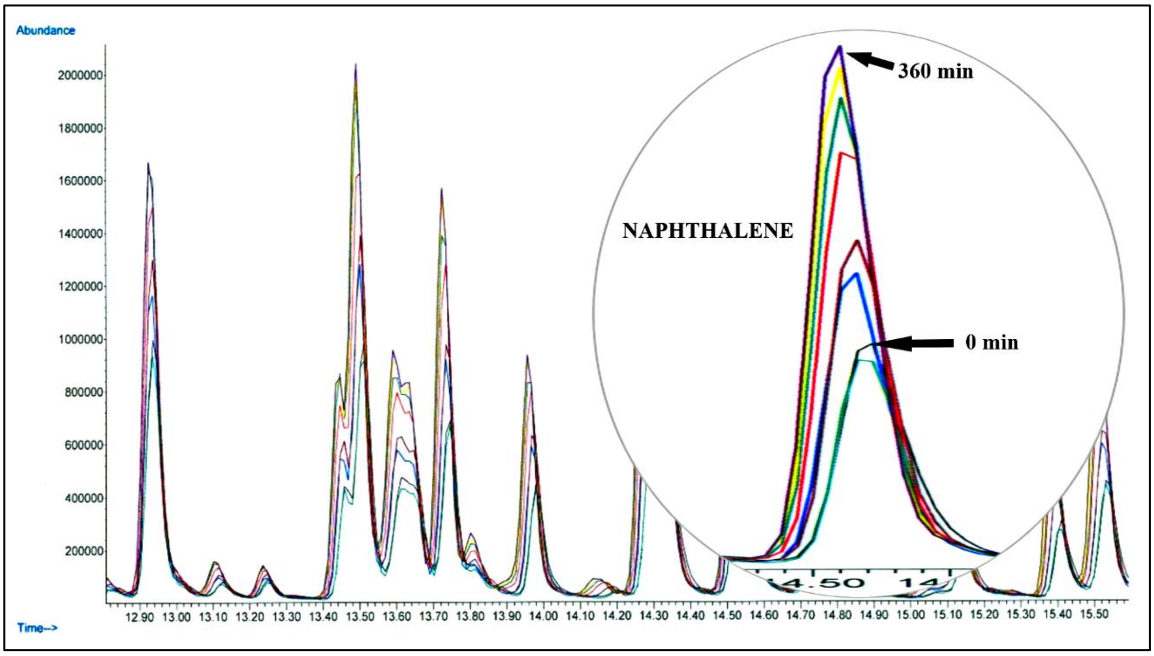

Fig. 7. Part of the chromatogram of heaviest hydrocarbons with the retention time from 13 to 16 minute

The opposite trend is manifested in the area after the RT of about 14 minutes (Fig. 7), where the intensity increases over time. Higher proportions of compounds were determined in longer weathered samples.

Composition analysis for various gasoline compounds by the PCA method performed by Turner and Goodpaster [5] showed that compounds with boiling points up to 155 °C are most susceptible to weathering. In contrast, long chain alkanes and lower substituted aromatics are susceptible to microbial degradation regardless of boiling point. As further stated, 1,3,5-TMB and 2-ethyltoluene are not only resistant to weathering but are also the least susceptible to microbial degradation.

Swierczynski et al. [22] investigated the possibility of determining gasoline on various matrices by HS-SPME-GC-MS. By TIC comparison, they found that the highly volatile components in gasoline (short chain alkanes, cycloalkanes, light aromatics) rapidly evaporated from cotton samples within 1 hour. Heavier aromatics such as 1,2,4-TMB, 1,2,3-TMB, 1,2,4,5-tetramethylbenzene, long chain alkanes (n-undecane, n-dodecane) and naphthalenes were found in the cotton samples even after 1 day.

Dhabbah et al. [34] investigated the effect of synthetic carpet thickness on the possibility of determining weathered ILRs. They analysed burned carpet samples by SPME-GC-MS. Samples were analysed at various times after quenching (suffocation) 0.5 to 5 hours. It was concluded that no detectable residues of gasoline remained in the samples of the burned 5 mm carpet after 5 hours. Under the same experimental conditions, no gasoline residues were detected after 2 and 3 hours in the case of carpet samples with a thickness of 15 and 25 mm. This may be due to the adsorption behaviour of the gasoline on the carpets, which more easily adsorbed on the thin surface of the carpet and the gasoline was exposed to more radiant heat.

Samples of residues obtained after the fire provide the investigators with a wide range of evidence. If the fire scene is largely destroyed, it is possible that investigators will gather and interpret little, if any, evidence. In real fires, it is very important to take samples from the fire as soon as possible because a fire could cause different results on the gasoline profile due to uncontrollable factors such as uneven distribution of gasoline in the carpet sample, burning rate, fire intensity, air flow and fire spread, which could directly or indirectly affect the amount of accelerator residue obtained from fire debris. Among other authors, Lim et al. [2] reached this finding as well.

4 Conclusions

The paper deals with the study of weathering of selected gasoline compounds. The cotton carpet was used as a matrix to trap gasoline residues. The headspace gas phase extraction method with gas chromatography and mass spectrometry (HS-GC-MS) was used as a progressive laboratory method to monitor the weathering of selected compounds. The determined compounds form groups of aliphatic hydrocarbons are alkanes, aromatic compounds - benzene derivatives and polycyclic aromatic hydrocarbons.

Fire debris samples obtained after the fire provide for the fire investigators a wide range of interpretable evidence. As stated by ASTM standards, when interpreting the results of the analysis of fire residues by the analyst to correctly determine the presence of an IL on the fire, it is quite sufficient to identify marker compounds that belong to IL. In the case of gasoline, the presence of indicator is among others for example the above mentioned 1,2,3-trimethylbenzene and 1,3,5-trimethylbenzene.

The results show that prolonged exposure of gasoline to the ambient conditions potentially leads to difficulties interpreting the results. The analysis showed that the most susceptible compounds for weathering are the most volatile hydrocarbons. The largest difference in the intensity changes of the selected compounds can be observed after four or five hours. On the other hand, the heaviest hydrocarbons increased significantly in intensity and the peaks of these compounds increased. However, despite the five parallel samples taken from each weathering time interval, the results may not be completely accurate as these experiments are characterized by great variability and heterogeneity.

In conclusion, it should be noted that the analysis results proved that time has a significant effect on the determinability of gasoline in fire debris samples. On the other hand, it is important to perform comparative analyzes of samples that have been weathered for various durations of time with pure samples, as the correct interpretation of laboratory analysis results plays a key role in fire investigation.

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