

# Forensic Detection Of Fire Accelerants Using A New Solid Phase Microextraction (SPME) Fiber

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## Abstract

An important aspect of an investigation of a suspected arson case involves the chemical analysis of the debris remaining after the fire. Currently, accelerant extraction and analytical techniques have been refined to improve sample turnover and to reduce the number of inconclusive findings. For this purpose, solid-phase micro extraction (SPME) have been introduced. SPME relies upon the concentration of headspace vapors onto an adsorbent medium. A new lab-made fiber prepared by sol-gel method, containing 1:1 molar ratio of octyltriethoxysilane (C8-TEOS): methyltrimethoxysilane (MTMOS) was employed in this technique. The fiber was evaluated for the analysis of n-alkane standard hydrocarbon compounds and common petroleum based accelerants. Compared with commercial PDMS/DVB fibre, the new lab-made fibre exhibited higher extraction capability for n-alkane hydrocarbon compounds and accelerants, higher thermal stability (up to 300 °C) and longer lifetime (~ 200 times usage). Electron microscopy experiments revealed that the surface of the fiber coating was well-distributed and a porous structure was suggested for the sol-gel derived C8 coating with an approximate thickness of (3-4) µm. The developed HS-SPME method using C8-coated fiber showed satisfactory reproducibility (RSD < 6 %), detection limits for accelerants (0.7-1.0 µL) and linearity ((r > 0.9869) under the optimum experimental conditions.

## 1. INTRODUCTION

The forensic discipline of ignitable liquid and fire debris analysis is rapidly changing. Modifications to traditional ignitable liquid extraction methods and research into new applications of existing extraction techniques continue to improve the quality of arson analysis. The earliest methods of identification relied upon simple identification of the headspace odor-often referred to as a "nasal appraisal". With improvements in analytical instrumentation, vapor samples were taken and subjected to instrumental analysis, usually gas chromatography. A significant improvement came with the application of steam distillation to fire debris analysis and this was followed by passive and dynamic headspace method [1, 2].

In addition to optimizing existing extraction techniques, the field of forensic science has also introduced a new extraction technique for application to arson analysis: solid-phase microextraction (SPME). The major advantage of this technique is that it uses no solvents and can be used for either direct sampling or sample clean-up. It is fairly economical and is a relatively simple and sensitive technique. The extraction is based on the enrichment of components on a polymer or adsorbent coated fused silica

fiber [3,4,5,6].

SPME is predominantly performed on commercial SPME fibers; however the extent of selectivity obtainable using conventional fibers is limited. Generally accepted drawbacks of conventional adsorbents are a relatively low thermal stability (200-270) ° C which leads to incomplete sample desorption and sample carry-over problem, short lifetime (40-100) times, poor solvent stability and expensive [7,8,9,10,11,12,13,14,15,16,17,18,19,20,21,22,23,24,25].

Recently, many novel coatings have been developed using different techniques and technology for use in SPME. Compared with commercially available SPME adsorbents, the new materials exhibited higher thermal stability (350 ° C), solvent stability, extraction capability and longer lifetime [7,8,9,10,11,12,13,14,15,16,17,18,19,20,21,22,23,24,25]. However, up to now, none of the novel fibers have been evaluated for the determination of accelerants in arson analysis. This paper presents a recent development in the forensic aspects of fire investigation. As a preliminary study, a new SPME adsorbent comprising of sol-gel derived C<sub>8</sub>-coating was developed and evaluated for the determination of accelerants in arson samples, with the aim of improving the quality of

ignitable liquid residue analysis.

## 2. EXPERIMENTAL

### 2.1. CHEMICALS AND MATERIALS

Individual standards of n-alkanes ( $C_8$ ,  $C_{10}$ ,  $C_{12}$ ,  $C_{14}$  and  $C_{16}$ ) were purchased from Fluka Chemika. Samples of diesel and unleaded gasoline were purchased from a petrol station in Skudai, Johor while kerosene was obtained from a grocery shop at Taman Universiti, Skudai, Johor. Samples of carpet were purchased from a carpet retail shop in Taman Ungku Tun Aminah, Skudai, Johor, Malaysia.

### 2.2. APPARATUS

Two glass apparatus ( $400\text{ cm}^3$  and  $125\text{ cm}^3$ ) for sample preparation step of HS-SPME was specially designed [26]. A Supelco SPME holder, commercially available PDMS/DVB fiber (Bellefonte, Pennsylvania, U.S.A.) and a used SPME fibre with a burnt off tip, coated with sol-gel derived  $C_8$ -coated fiber containing (1:1,  $C_8$ -TEOS:MTMOS) [27] were employed for the extraction of n-alkanes and accelerants.

### 2.3. INSTRUMENTATION

Gas chromatography analyses were conducted using a Hewlett-Packard 6890 GC (Wilmington, Delaware, U.S.A.). The HP 6890 gas chromatograph was equipped with FID and a HP ChemStation for data processing. An Ultra-1 capillary column (Agilent) of dimensions  $25\text{ m} \times 0.20\text{ mm} \times 0.11\text{ }\mu\text{m}$  film thickness was used. Helium was used as the carrier gas at a flow rate of  $1.2\text{ mL/min}$ . The injection port temperature was set at  $250\text{ }^\circ\text{C}$  and FID temperature at  $310\text{ }^\circ\text{C}$ . SPME injections were performed using a split mode injection (5:1).

Studies of fiber coating structure and thickness were made by means of Philips Scanning Electron Microscope model XL 30 SEM (Philips Electronic Instruments Company, Mahwah, New Jersey) equipped with a ThermoNoran energy dispersive X-ray detection system (EDX).

### 2.4. PROCEDURES FOR HEADSPACE SPME

$30\text{ }\mu\text{L}$  from the prepared standard solution of n-alkanes ( $C_8$ ,  $C_{10}$ ,  $C_{12}$ ,  $C_{14}$ , and  $C_{16}$ ) was placed in the sample preparation apparatus which was immersed in a hot water bath and heated for 20 min at  $100\text{ }^\circ\text{C}$ . The  $C_8$ -coated fiber was exposed in the headspace and the fiber extracts were analyzed using GC-FID. The oven temperature was initially set at  $40\text{ }^\circ\text{C}$ , programmed at a rate of  $10\text{ }^\circ\text{C/min}$  until a final temperature of  $270\text{ }^\circ\text{C}$ . The headspace SPME procedure was repeated using PDMS/DVB fiber for

comparison. The same HS-SPME procedure was carried out again using spiked fire debris sample for the determination of accelerants in simulated arson samples.

### 2.5. PREPARATION OF FIRE DEBRIS SAMPLE

A sample of carpet ( $20\text{ cm} \times 13\text{ cm}$ ) placed on a sheet of aluminium foil was ignited with a fire starter and left to burn until about one-third remained on the aluminium foil. Fire was extinguished by cutting off the oxygen supply. The partially burnt carpet was then exposed to the surrounding air for 30 minutes to let it cool down.

## 3. RESULTS AND DISCUSSION

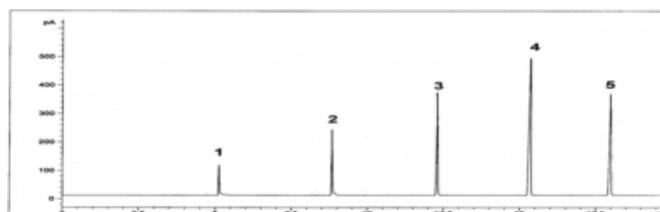
### 3.1. CHARACTERIZATION OF THE C-COATED FIBER

#### 3.1.1. SELECTIVITY FOR N-ALKANE STANDARD HYDROCARBONS

In order to examine selectivity of the lab-made SPME fiber towards hydrocarbon compounds, a mixture of n-alkanes ( $C_8$ ,  $C_{10}$ ,  $C_{12}$ ,  $C_{14}$  and  $C_{16}$ ) were subjected to HS-SPME using  $C_8$ -coated fiber and the GC profiles were compared with that from direct injection. The GC profiles obtained from headspace SPME using  $C_8$ -coated fiber was comparable with the profiles of hydrocarbon standards from direct injection. All the n-alkane hydrocarbon components were well separated as shown in Figure 1.

**Figure 1**

Figure 1: SPME-GC analysis of hydrocarbon compounds using C-coated fibers Peaks: (1)  $C_8$ , (2)  $C_{10}$ , (3)  $C_{12}$ , (4)  $C_{14}$  and (5)  $C_{16}$  n-alkanes



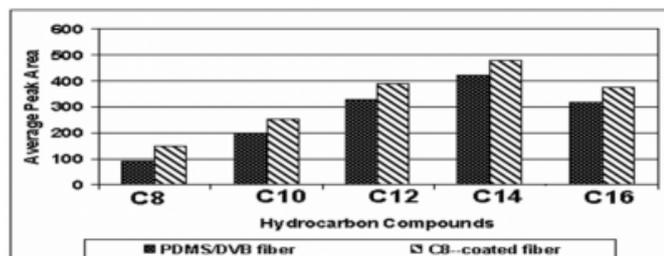
#### 3.1.2. EXTRACTION CAPABILITY FOR N-ALKANE STANDARD HYDROCARBONS

The extraction capability of the  $C_8$ -coated fiber for hydrocarbons was determined by comparing it with the extraction capability of commercially available PDMS/DVB fiber. PDMS/DVB fiber was selected for comparison because previous work done in this lab [26] proved that the fiber has the highest sensitivity towards hydrocarbon compounds. As can be seen from Figure 2, the  $C_8$ -coated fiber exhibited a slightly higher extraction capability for all the hydrocarbon compounds by contrast with conventional

PDMS/DVB fiber. A higher extraction capability yielded by C<sub>8</sub>-coated fiber could be due to the existence of higher surface area for the C<sub>8</sub>-coated fibers [13].

**Figure 2**

Figure 2: Comparison of extraction capability between C-coated fiber and PDMS/DVB fiber



### 3.1.3. LIFETIME OF THE C-COATED FIBER

The long lastingness of the C<sub>8</sub>-coated fiber was determined by 200 continued operations carried out with the same fiber and oven temperature program. The C<sub>8</sub>-coated fiber have been used for hydrocarbon extractions (C<sub>9</sub>, C<sub>13</sub>, and C<sub>15</sub>) and subjected to GC for more than 200 times. There was no significant differences of hydrocarbon peaks obtained in each operation. All the hydrocarbon compounds gave a low relative standard deviation (RSD) value ranging from 3.8 %-5.4 % which shows an acceptable reproducibility. This proves that the coated surface of the fiber was not partially depleted during the continued operation. It was still stable and reusable. Such a long service life are possibly due to the strong chemical bonding between the sol-gel generated C<sub>8</sub>-coated composite coating and the silica surface [7,12,13, 15].

### 3.1.4. THERMAL STABILITY OF THE C-COATED FIBER

The effect of conditioning temperature on the stability of the C<sub>8</sub>-coated fiber was determined by conditioning the fiber at high temperatures (270 and 300) ° C for 1 hour prior to extraction. High temperature conditioning lead to consistent improvement in peak area repeatability for SPME-GC analysis. The RSD value of < 4.6 % can be routinely obtained for hydrocarbons on C<sub>8</sub>-coated fibers conditioned at 270 ° C and 300 ° C. The C<sub>8</sub>-coated fiber can be routinely used at 300 ° C without any sign of bleeding, whereas for commercial PDMS/DVB fiber, the highest temperature the coating layer can endure is less than 280 ° C, and thus the range of analyte molecular weights that can be handled by SPME-GC is limited. Enhanced thermal stability of C<sub>8</sub>-coated fiber might be due to the strong chemical bonding between the sol-gel generated composite coating and the silica surface [8, 11, 16, 17,18,19, 24, 25].

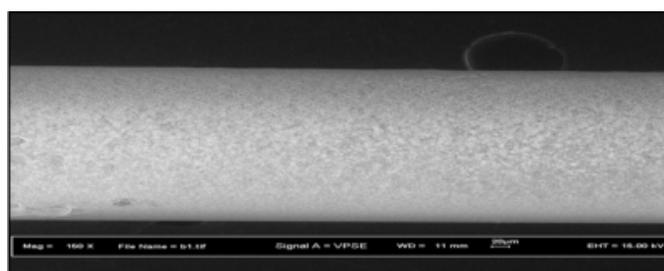
### 3.1.5. SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

The morphology of the lab-made fiber was investigated using the scanning electron microscope. As can be seen from Figure 3, it is obvious that the surface of the fiber coating was well-distributed. A homogeneous porous structure was suggested for sol-gel C<sub>8</sub>-coating (Figure 4). Chong et al. [7] and Yu et al. [10] have reported that porous structure of the sol-gel coating should significantly increase the surface area on the fiber. Consequently, with such a porous coating structure, even an apparently thinner coating will be able to provide enhanced stationary-phase loadings and therefore, high fiber sample capacity. Figure 5 reveals that the fiber was not uniformly coated. This suggestion is agreeable to those reported by Gbatu et al. [13].

The average thickness of the C<sub>8</sub> coating was estimated as being ~ 3-4 μm, which is considerably thinner than most conventional SPME fibers (65 μm for PDMS/DVB). The reproducibility of the coating thickness obtained in this study is rather promising. The volume of extracting phase on the lab-made C<sub>8</sub>-coated fiber is ~ 0.011-0.014 mm<sup>3</sup> whereas a 65 μm PDMS/DVB commercial fiber has an extracting phase volume of 0.357 mm<sup>3</sup>. It is apparent that the volume of extracting phase on the lab-made C<sub>8</sub>-coated fiber is considerably less than that of commercial PDMS/DVB fiber. Therefore the C<sub>8</sub> coating has a larger affinity towards the analytes than regular PDMS/DVB.

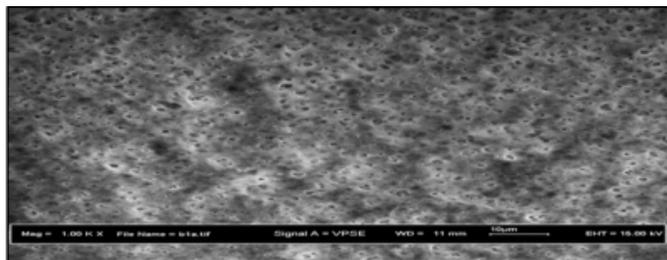
**Figure 3**

Figure 3: The surface view of C fiber at 150 fold-magnification obtained by SEM



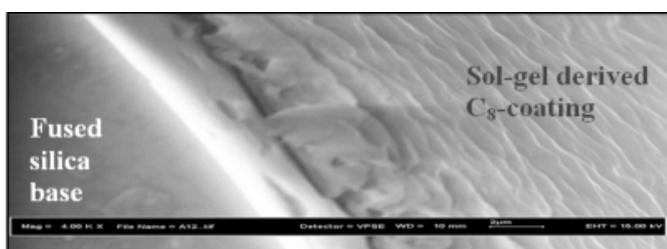
**Figure 4**

Figure 4: The surface view of C fiber at 150 fold-magnification obtained by SEM



**Figure 5**

Figure 5: The cross-sectional view of C fiber at 4000 fold-magnification obtained by SEM



## 3.2. METHOD VALIDATION

### 3.2.1. ACCURACY OF HS-SPME METHOD USING C-COATED FIBER

The accuracy of the developed method for three replicate analyses was determined by calculating the relative standard deviations (RSD) of the peak areas for all standard hydrocarbons ( $C_8$ ,  $C_{10}$ ,  $C_{12}$ ,  $C_{14}$  and  $C_{16}$ ) in burnt carpet sample spiked at the same concentration level.

All the hydrocarbon compounds gave a low relative standard deviation (RSD) value ranging from 3.9 %-5.5 % which shows an acceptable reproducibility. These results indicated that quantitative analysis of hydrocarbons was possible using the external standard method and the applicability of lab-made  $C_8$ -coated fiber for routine analysis.

### 3.2.2. DETECTION LIMITS OF ACCELERANTS

Detection limits of accelerants (lowest detectable amount determined based on three times the baseline noise) were evaluated in order to assess the performance of the proposed method. The lab-made  $C_8$ -coated fiber was employed for extractions of gasoline, kerosene and diesel in spiked burnt carpet sample under optimum HS-SPME conditions.

For all the common accelerants, the LODs were between 0.7 to 1.0  $\mu$ L. These values are indicative of good performance of the lab-made  $C_8$ -coated fiber. The detection limits of

common accelerants obtained in this study using  $C_8$ -fiber is comparable to those reported by Yong [26] using commercial PDMS/DVB fiber.

### 3.2.3. LINEARITY

Calibration curves consisting of five different concentration of n-alkane hydrocarbons were generated as regression lines and the correlation coefficients were obtained. The analytes were extracted using lab-made  $C_8$ -coated fiber under the optimum HS-SPME conditions. The

HS-SPME procedure with  $C_8$ -coated fiber yielded an excellent linear relationship between the peak area counts and analyte concentration in the range of 2.00-10.00 % v/v. Good correlations with  $R^2$  value between 0.9869-0.9958 ( $R^2 \sim 1$ ) were obtained. It is apparent that there were almost no factors in the HS-SPME extractions of n-alkane hydrocarbons using lab-made  $C_8$ -coated fiber that might cause the concentration characteristics to deviate from linearity.

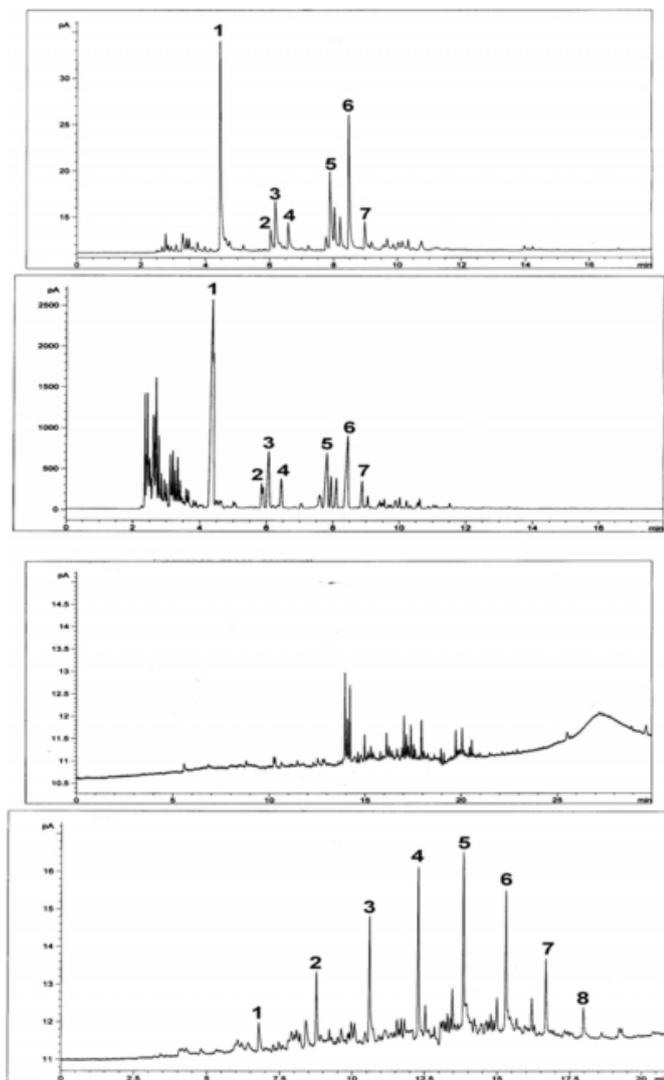
## 3.3. ANALYSIS OF SIMULATED ARSON SAMPLES USING C-COATED FIBER

### 3.3.1. SELECTIVITY FOR PETROLEUM BASED ACCELERANTS

In order to examine selectivity of the  $C_8$ -coated fiber towards petroleum based accelerants, burnt carpet sample were individually spiked with known amount of gasoline, kerosene and diesel. The samples were subjected to headspace SPME using  $C_8$ -coated fiber and the GC profiles were compared with that from direct injection. As seen in Figure 6 (b), all the hydrocarbon components in gasoline spiked burnt carpet sample were recovered by using  $C_8$ -coated fiber and the chromatogram was comparable with the profile of gasoline from direct injection [Figure 6 (a)]. The hydrocarbon components in kerosene (Figure 7) and diesel (Figure 8) spiked burnt carpet sample were also effectively extracted using the  $C_8$ -coated fiber and similar comparisons were obtained with direct injection of the respective accelerants. This indicated that the  $C_8$ -coated fiber favored the extraction of hydrocarbons, thus providing a good selectivity towards accelerants. The GC profiles of gasoline, kerosene and diesel spiked samples obtained in this study were in good agreement with those obtained by Borusiewicz [1] using Tenax as adsorbent and Yong [26] using commercially available fibers. Figure 6: GC profiles of direct injection of gasoline

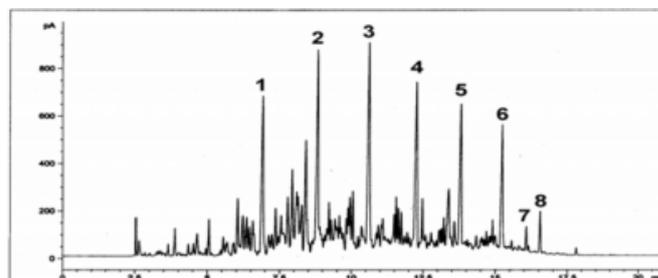
**Figure 6**

Figure 6: GC profiles of (b) gasoline spiked burnt carpet sample and (c) the blank burnt carpet sample using C-coated fibers. Peak identities: (1) methylbenzene, (2) ethylbenzene, (3) 1, 3-dimethylbenzene, (4) 1, 2- dimethylbenzene, (5) 1-ethyl-2-methylbenzene, (6) 1, 2, 4-trimethylbenzene, (7) 1, 2, 3- trimethylbenzene



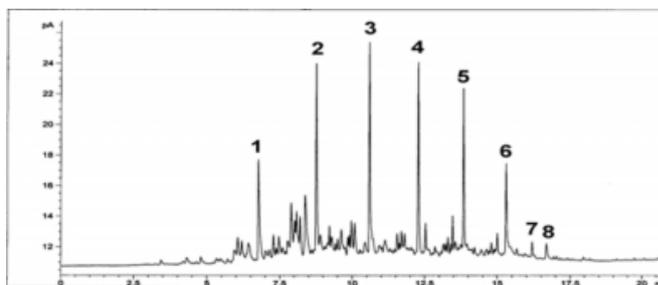
**Figure 7**

Figure 7: GC profiles of (a) direct injection of kerosene and (b) kerosene spiked burnt carpet sample using C-coated fibers. Peak identities: (1) C, (2) C, (3) C, (4) C, (5) C, (6) C, (7) 2, 6-dimethylnaphthalene, (8) C



**Figure 8**

Figure 8: GC profiles of direct injection of diesel spiked burnt carpet sample using C-coated fibers. Peak identities: (1) C, (2) C, (3) C, (4) C, (5) C, (6) C, C, (7) C



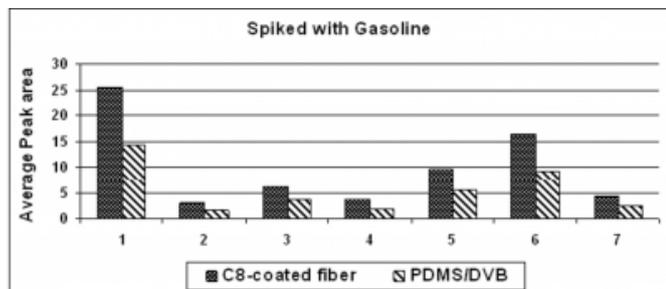
### 3.3.2 EXTRACTION CAPABILITY FOR PETROLEUM BASED ACCELERANTS

The extraction capability of the sol-gel derived C<sub>8</sub>-coated fiber was determined by comparing it with the commercially available PDMS/DVB fiber. Both C<sub>8</sub>-coated fiber and PDMS/DVB fiber were capable of extracting early, middle and late eluting hydrocarbon compounds sufficiently. However, the C<sub>8</sub>-coated fiber showed a slightly higher extraction capability by contrast with conventional PDMS/DVB fiber for all the accelerants as shown in Figure 9. This result was comparable to that reported by Gbatu [13]. A higher extraction capability could be due to the existence of higher surface area for the C<sub>8</sub>-coated fibers [9, 13, 14,

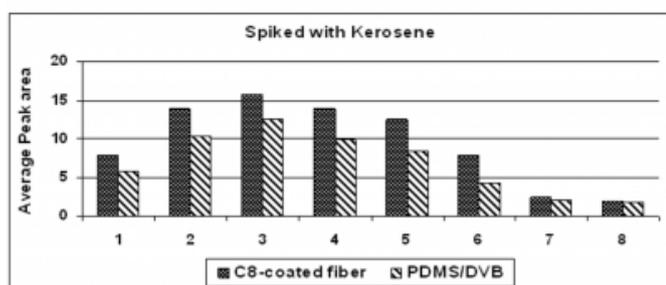
20\*21\*22\*23].

**Figure 9**

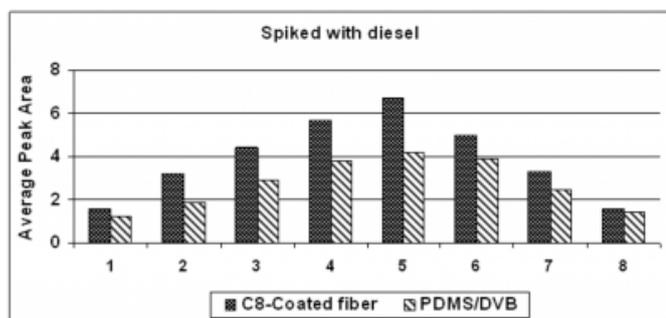
Figure 9: Comparison of sol-gel derived C-coated fiber and commercially available PDMS/DVB fiber in extracting hydrocarbon compounds from (a) gasoline, (b) kerosene and (c) diesel spiked burnt carpet



Label identities: (1) methylbenzene, (2) ethylbenzene, (3) 1, 3-dimethylbenzene, (4) 1, 2-dimethylbenzene, (5) 1-ethyl-2-methylbenzene, (6) 1, 2, 4-trimethylbenzene, (7) 1, 2, 3-trimethylbenzene.



Label identities: (1) C<sub>9</sub>, (2) C<sub>10</sub>, (3) C<sub>11</sub>, (4) C<sub>12</sub>, (5) C<sub>13</sub>, (6) C<sub>14</sub>, (7) 2, 6-dimethylsaphthalene, (8) C<sub>15</sub>.



Label identities: (1) C<sub>9</sub>, (2) C<sub>10</sub>, (3) C<sub>11</sub>, (4) C<sub>12</sub>, (5) C<sub>13</sub>, (6) C<sub>14</sub>, (7) C<sub>15</sub>, (8) C<sub>16</sub>.

### 3. CONCLUSIONS

A new SPME fiber (lab-made C<sub>8</sub>-coated fiber) has been successfully developed and evaluated for the determination of common petroleum based accelerants in arson samples. The C<sub>8</sub>-coated fiber has a good selectivity towards hydrocarbon compounds and accelerants as it was capable of effectively extracting all the tested n-alkane hydrocarbon compounds and hydrocarbon components in gasoline, kerosene and diesel spiked burnt carpet samples. The lab-made fibre gave superior characteristics when compared to commercially available PDMS/DVB fibre. Compared with commercial PDMS/DVB fibre, the new lab-made fibre exhibited higher extraction capability for n-alkane hydrocarbon compounds and accelerants, higher thermal

stability (up to 300 ° C) and longer lifetime (~ 200 times usage). Electron microscopy experiments revealed that the surface of the fibre coating was well-distributed but not uniformly coated and a porous structure was suggested for the sol-gel derived C<sub>8</sub> coating with an approximate thickness of (3-4) μm. The developed HS-SPME method using C<sub>8</sub>-coated fiber exhibited a high degree of accuracy under the optimum experimental conditions. The limits of detection for the common accelerants were in the range of 0.7 to 1.0 μL. The linearity of the calibration graphs for all the tested n-alkane hydrocarbons was excellent (r > 0.9869). The experimental results clearly demonstrate the potential of C<sub>8</sub>-coated fiber in SPME for the detection of arson accelerants.

### ACKNOWLEDGEMENTS

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