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Gaseous Products of Incense Coil Combustion Extracted by Passive Solid Phase Microextraction Samplers

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Abstract: Burning incense indoors is a common behavior in Southeast Asia. In this investigation, needle trap samplers (NTS), a novel, green analytical technology is used for sampling gaseous combustion by-products from sandalwood incense coils. To extract indoor volatile organic compounds (VOCs), two NTS are prepared, one using 60–80 mesh and the other using 100–120 mesh divinylbenzene (DVB) particles packed in 22-gauge stainless steel needles. This work compares extraction efficiency of an NTS and that of a commercially available 100 μ m polydimethylsiloxane solid phase microextraction (PDMS-SPME) fiber sampler. Experimental results indicated that the 100–120 mesh DVB-NTS performed best among all samplers during a 1 h sampling period. The main extracted compounds were toluene, ethylbenzene, propane, chloromethane, 1,3-butadiene, methanol and dichloromethane. The potential use of small badge-sized or pen-sized NTS for the indoor atmosphere and occupational hygiene applications is addressed.

Keywords: solid phase microextraction; needle trap sampler; sampling; indoor air; volatile organic compounds; incense coil

1. Introduction

In Southeast Asia, incense sticks are burned to worship ancestors and pray to deities. According to the recent studies in Taiwan and China, smoke from incense combustion in temples contains many particles and gaseous pollutants [1]. Wang *et al.* conducted field investigations of target air pollutants at two of the most famous temples in Hong Kong [2]. The target air pollutants were particulate matters (PM_{2.5} and PM₁₀), volatile organic compounds (VOCs), carbonyl compounds, carbon monoxide, nitrogen oxides, methane, non-methane hydrocarbons (NMHC), organic carbon, elemental carbon (EC), and inorganic ions (Cl⁻, NO₃⁻, SO₄²⁻, Na⁺, NH₄⁺, and K⁺). The PM was composed of fine particles; namely, average PM_{2.5}/PM₁₀ ratios were approximately 82%. Formaldehyde was the most abundant carbonyl compounds, followed by acetaldehyde. Polycyclic aromatic hydrocarbons (PAHs), in their particulate phase generated from burning incense, were further analyzed by Yang *et al.* using gas chromatography/mass spectrometry (GC/MS) [3]. Tested Taiwanese incenses yield emission factors in the range of 4.19–82.16 mg/g and 1.20–9.50 µg/g, respectively, for total PM and PAHs.

The potential hazard to human health passed by burning incense is an important focus from the viewpoint of indoor air quality. Kuo *et al.* [4] investigated the emission characteristics of allergenic aromatic eugenol and its related compounds from the incense powder emitted from burning incense sticks. Experimental results indicate that 870.8–593.7 mg fragrant compounds (methyl isoeugenol was the most prevalent compound) were emitted by combustion one gram of incense. Navasumrit *et al.* [5] showed that temple workers in Thailand are exposed to relatively high levels of benzene (45.90 µg/m³) 1,3-butadiene (11.29 µg/m³) and PAHs (19.56 ng/m³). Even mean total polychlorinated dibenzo-p-dioxins/dibenzofurans (PCDD/F) concentrations 72.4–82.2 pg/m³ were examined at two indoor Taiwanese temples; the PCDD/F were roughly 11 times that at a background location [6].

As incense has various compositions [3], especially the incense manufactured in different countries [7,8], its combustion typically produces different products. Yang *et al.* [3] reported that the atomic hydrogen to carbon (H/C) ratio in incense was the key factor affecting emission PM and PAH emission factors. The maximum emission factor and slowest burning rate were at an H/C ratio of 1.57. Cohen *et al.* [7], who compared several incenses used in the United Arab Emirates, found that different raw plant materials in incense sticks, such as essential oils and resins, resulted in hugely different emissions of particles, the sizes of which were 20–300 nm with median count diameters of 65–92 nm. Chang *et al.* [8] also noted that combustion of incenses made in Taiwan and Japan emitted particles with different diameters due to the use of a bamboo stick in Taiwanese incenses and but not Japanese incenses.

Households in Taiwan often burn incense coils all day. Incense coils, which are composed of fragrance powders, are formed by winding a long, narrow incense strip in a circular pattern (Figure 1). The scent emitted from these coils when burned is generally used as deodorant, for disinfection and for relaxation. Although many studies have characterized the particles and gaseous products emitted from burning incense sticks, the products from burning incense coils indoors have largely been ignored.

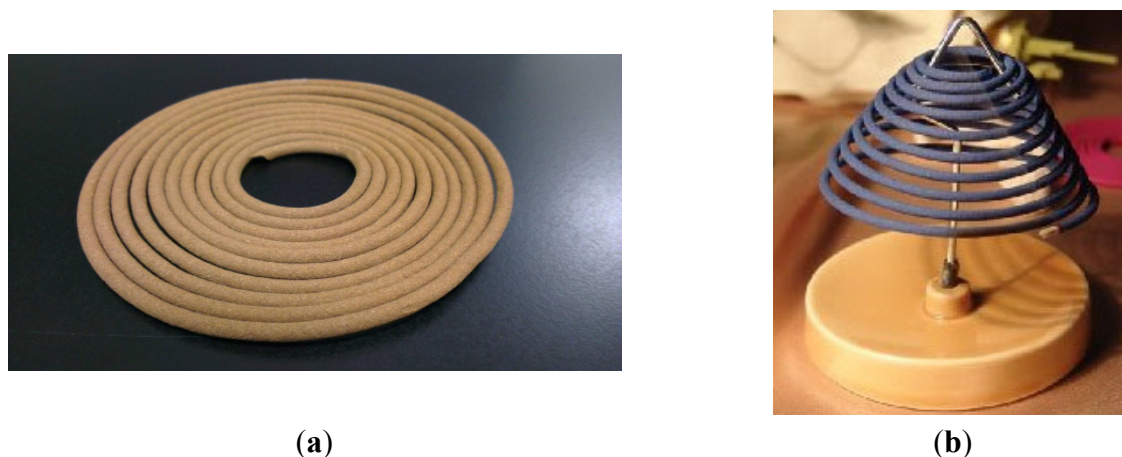


Figure 1. Incense coil. (a) Original incense coil. (b) Spread and burned incense coil.

The main by-products of burning incense coils are predictably VOCs. To characterize emissions from burning incense sticks, Cohen *et al.* [7] and Chang *et al.* [8] respectively designed stainless steel and glass chambers to that simulated indoor environments and then collected samples from exhaust streams produced by the burning incense sticks. This work also designed a glass chamber, and then installed it in a temperature-controlled oven at 27 °C to sample combustion by-products from incense coils manufactured in Taiwan.

Solid-phase microextraction (SPME), which is widely used for sampling gaseous organic compounds, is an environmentally friendly process because it is solvent-free and SPME devices can be reused [9]. Cheng *et al.* [10] extracted gaseous benzene, toluene, ethylbenzene and o-xylene (BTEX) using divinylbenzene (DVB) particles as sorbents packed in a self-fabricated SPME device, the novel needle trap samplers (NTS). Based on experimental results obtained by Cheng *et al.* [10], indoor combustion sources including mosquito coils [11,12] and essential oils [13], were sampled successfully using an NTS, and they were then analyzed via GC/MS.

2. Methods

2.1. Theory of Gaseous Compound Extraction via a Needle Trap Sampler

An NTS extracts gaseous compounds through its needle by diffusion. During this process, the linear gaseous concentration profile ($C(Z)$ in Figure 2) is established along the diffusion path distance (Z), and the extraction phase is characterized by opening area (A) and diffusion path length. The total amount of analyte extracted (n) during time interval (t) is then estimated [14]

$$n = D_m \frac{A}{Z} \int C(t) dt \quad (1)$$

where D_m is the diffusion coefficient of components sampled by the sorbent inside the needle. The quality of the extracted analyte is assumed proportional to the integral amount of the sample concentration over time ($C(t)$) under a constant D_m , a uniform needle opening, and a uniform diffusion path distance. Equation (1) is valid only when the analyte extracted onto a sorbent is a small fraction of the equilibrium amount for the lowest concentration in the sample, which can be assessed based on the values of relative standard deviation (RSD) of duplicate or triplicate measurements (typically <5%).

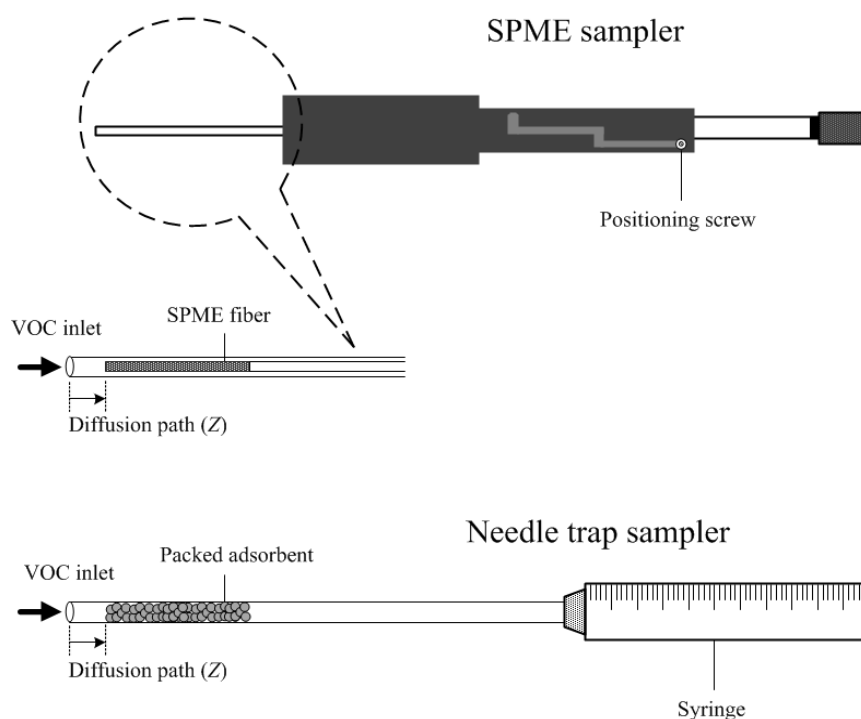


Figure 2. Schematic SPME fiber and needle trap samplers.

2.2. Chemicals, Materials and Equipment

The 3.5", 22-G stainless steel needles (ID 0.41 mm and OD 0.71 mm) were purchased from Herling Co. Ltd. (Pingtung, Taiwan). The DVB particles were used as adsorbents in the NTS. Supelco suggests that users sample volatile compounds (molar weights of 60–275 g/mole) via 100 μ m-PDMS SPME fiber. Hence, 100 μ m-PDMS SPME fibers were used to compare the sampling rates in this research. The 100 μ m-PDMS SPME fibers were obtained from Supelco (Bellefonte, PA, USA). The aspirating pumps used to test NTS sampling flow rates were purchased from Kitagawa (AP-20, Kawasaki, Japan), and the 5-min syringe epoxy glue was purchased from Nao-Pao Applied Material Co. Ltd. (Taoyuan, Taiwan). All gases for chromatographic analysis were supplied by Jing-De Gas Co., Ltd. (Kaohsiung, Taiwan) and were of ultra-high purity. Additionally, the tested VOCs (BTEX), which were applied to qualify the self-packing NTS, were obtained from Merck's Taiwan Branch (Taipei, Taiwan). The tested sandalwood incense coils were provided by the Kao-Der Corporation. (Kaohsiung, Taiwan). Lung and Hu [15] found emissions between different incenses differed significantly, and a variability of 10%–20% in emissions was observed for incenses made manually. To collect representative gaseous samples, incenses coils of the same brand from the same producing batch were ignited.

The BTEX were analyzed using a gas chromatograph (GC) (6890N, Agilent, Wilmington, DE, USA) equipped with a flame ionization detector (FID). The VOC separations were performed using a HP19091Z-413 HP-1 PDMS (30 m \times 320 μ m \times 0.25 μ m) capillary column (Agilent Technologies, Inc., Wilmington, DE, USA). The heating products of the incense coils were also identified using a mass spectrometer (5973N, Agilent Technologies, Inc.). The packed mass of DVB particles was measured with a precision scale (ME type, Sartorius, Germany) with a minimum reading of 0.0001 mg and reproducibility <0.0001 mg. An environmental scanning electron microscope (ESEM-FEI,

Quanta-200, Czech Republic), at National Sun Yat-sen University, Kaohsiung, Taiwan, was used to analyze the surface of DVB for the combustion fragments laying on the surface adsorbent particles.

2.3. Preparation of Needle Trap Sampler

The NTS were mainly comprised of a stainless steel needle packed with DVB particles (mesh sizes 60–80 and 100–120). Sorbent particles in the needle were immobilized without bleeding. A stainless steel wire spring plug was initially fixed in its position ($Z = 10$ mm). The DVB particles were then packed individually by aspiration until the desired length of 7 mm was attained (*i.e.*, when the diffusion path was 3 mm long). To immobilize the sorbent particles, packing was then performed by applying a very small amount of epoxy glue to the exposed portion of the sorbent layer. Precision measurements revealed that the packed DVB mass of the tested NTS averaged 0.347 mg (SD = 0.0380 mg). For convenience, another long straight stainless-steel wire was used as a pole to moisten the epoxy glue. To prevent the epoxy-resin plug from completely blocking the NTS, air was drawn continuously through the NTS packing as the epoxy cured. Finally, the DVB in the NTS was conditioned by heating at the injection port of the GC-FID at 260 °C for 30 min. The prepared NTS was stored at room temperature until use. Four 60–80 mesh DVB NTS and five 100–120 mesh DVB NTS were prepared.

Since few studies have examined the uniformity of the packing phase in an NTS, the procedures by Cheng *et al.* [10–13] were applied to examine all NTS in terms of desired sampling flow rates (mL/min) when drawn by an aspirating pump through the packing phase. When the RSD for three duplication tests of sampling flow rates did not exceed 5%, the materials filled in the NTS were assumed uniformly immobilized.

2.4. Sampling and Analysis

Static VOC samples for pre-measuring tests of the self-prepared NTS and 100 µm PDMS-SPME fibers were prepared by injecting BTEX (100 ppm) into 1000 mL glass bulbs. Simultaneously, VOC calibrations were performed to precisely quantify the BTEX concentration. All NTS were used to take static BTEX samples within the bulb in an oven heated to 27 °C. The VOC analysis of both calibrations and static samples of the NTS was also performed using the same GC-FID. After VOC sampling, an NTS was injected into the injection ports of the GC, which was used for VOCs desorption and further analysis. Desorption time and temperature at the injection port of the GC was 30 s and 250 °C, respectively. The temperature of the GC increased from 50 °C at increments of 15 °C/min, and the final temperature was held at 180 °C for 2 min. The FID detector was heated to 300 °C. The carrying gas flow rate was 1.2 mL/min for nitrogen, and the split off operation mode was selected. The SPME fibers were also analyzed using the same procedures as those for the NTS. Notably, after analysis, no VOC carryovers were available for sample extraction by the NTS or SPME, which was verified by earlier examinations [10–13].

2.5. Sampling and Analytical Products from Burning Sandalwood Incense Coils

A sandalwood incense coil was heated in a circular glass chamber (inner dimensions: diameter, 30 cm; height, 22 cm; total volume: approximately 17.5 L). Air was then pumped into the chamber at a

flow rate of 0.5 L/min to facilitate burning of the incense coil. Initial samples were collected in triplicate into 1-L Teflon bags (SKC, Eighty Four, PA, USA) and then analyzed with GC-MS to quantify the gaseous by-products of a sandalwood incense coil. The NTS and SPME were inserted into the chamber through the septa, which sealed the sampling ports installed on the chamber wall. The PDMS SPME fibers and NTS were exposed in the chamber for taking a 1-h time weighting average (TWA) sample and then analyzed via the GC-FID.

3. Results and Discussion

3.1. VOC Combustion Products of Sandalwood Incense Coils

The main gaseous combustion by-products identified via GC/MS analysis were alkane, aromatic, chlorinated hydrocarbon compounds, and several oxygenated compounds (methanol, acetone and vinyl acetate) (Table 1). Alkane (propane and 1,3-butadiene), aromatic compounds (*i.e.*, BTEXs), chloromethane, methanol and acetone are the same compounds identified by Yang from burning Taiwanese incense sticks [1] and by Navasumrit *et al.* from burning incense in Thai temples [5]. Although the gaseous compounds from burning incense sticks resemble the combustion products from burning incense coils, the health hazards associated with burning incense sticks in temples do not take priority over incense coils, as people typically spend more time in the smoke from incense coils in their homes than in the smoke from sticks in temples.

Table 1. Main gaseous by-products of burning incense coil.

Chemicals	Concentration (ppbv) ^{a,b}	Concentration (µg/m ³) ^c
Alkane Compounds		
Propane	131.84 (41.20)	258.8
1,3-Butadiene	460.16 (57.52)	1108.2
trans-2-Butene	42.88 (5.40)	107.1
cis-2-Butene	40.78 (4.50)	101.9
Aromatic Compounds		
Benzene	1.12 (0.20)	3.9
Toluene	21.12 (2.60)	86.6
Ethylbenzene	31.76 (3.22)	150.1
<i>m/p</i> -Xylene	4.48 (0.41)	21.2
<i>o</i> -Xylene	4.50 (0.20)	21.3
Styrene	4.08 (0.51)	18.9
Chlorinated Compounds		
Chloromethane	188.24 (23.53)	423.2
Dichloromethane	33.12 (5.20)	125.2
Other Oxygenated Compounds		
Methanol	54.24 (4.60)	77.4
Acetone	76.56 (9.57)	198.0
Vinyl acetate	28.64 (1.20)	109.8

Notes: ^a The concentrations are displayed in mean values and standard deviations of triple duplicates in parenthesis; ^b The method detection limits for all compounds are 0.0078 ppb; ^c Converted based on the mean ppbv concentrations at 27 °C.

Among all by-products, benzene (1.12 ppbv) is carcinogenic for leukemia; 1,3-butadiene (460.16 ppbv) and dichloromethane (33.12 ppbv) are also suspected to be carcinogens [16]. However, the 8-h TWA permissible exposure limits by the USA Occupational Safety and Health Administration (OSHA PEL) is 1 ppm for benzene, and 25 ppm for dichloromethane, which are at least 700-fold higher than the concentrations identified in this work. Notably, the 8-h TWA OSHA PEL is 1 ppm for 1,3-butadiene, relatively close to the identified concentration of 0.46 ppm. Additionally, human organs exposed to 1,3-butadiene include the eyes, respiratory system, central nervous system and reproductive system. The main effects of other alkanes and oxygenated compounds (propane, methanol and acetone) are irritation of the human respiratory tract, headache, drowsiness and dermatitis. Toluene, ethylbenzene and xylenes are minor by-products of combustion of incense coils.

3.2. Qualification Tests of Flow Rates and BTEX Extraction Rates for NTS

The NTS packed with 60–80 and 80–100 mesh DVB adsorbents were respectively used to examine sampling flow rates and then sample gaseous BTEXs in glass bulbs simultaneously with 100 μ m PDMS-SPME fibers. All duplicate experiments were performed three times. The RSD values of sampling flow rates were below 5%, indicating a high degree of uniformity of the packed DVB phases for all NTS (Table 2).

Table 2. Tests of sampling flow rates and volatile organic compound (VOC) extraction rates by solid phase microextraction devices in this study.

Test Item	Value
Sampling flow rate of NTS (mean values, mL/min) ^a	
Hollow needle	790 (RSD = 4.88%)
NTS packed with 60–80 mesh DVB adsorbents	64.2 (RSD = 4.79%)
NTS packed with 100–120 mesh DVB adsorbents	47.1 (RSD = 4.98%)
VOC extraction rates (mean values, ng BTEX/min) ^{b,c,d}	
60–80 mesh DVB NTS	
Benzene	2.22 (RSD = 3.73%)
Toluene	3.29 (RSD = 4.66%)
Ethylbenzene	4.58 (RSD = 4.37%)
<i>o</i> -Xylene	6.13 (RSD = 4.90%)
100–120 mesh DVB NTS	
Benzene	4.58 (RSD = 4.36%)
Toluene	5.55 (RSD = 3.00%)
Ethylbenzene	7.19 (RSD = 4.63%)
<i>o</i> -Xylene	8.58 (RSD = 4.08%)
100 μ m PDMS-SPME fiber sampler	
Benzene	1.56 (RSD = 0.9%)
Toluene	3.14 (RSD = 1.0%)
Ethylbenzene	4.52 (RSD = 1.6%)
<i>o</i> -Xylene	5.33 (RSD = 2.8%)

Notes: ^a Nine NTS were tested. Five of NTS were packed with 60–80 mesh DVB, and four were packed with 100–120 mesh DVB; ^b All VOC sampling periods were 1 h; ^c The specific concentration is around 100 ppm for benzene, toluene, ethylbenzene and *o*-xylene; ^d The exactly extracted mass of BTEX by 60–80 mesh DVB NTS were 133 ± 5.3 , 197 ± 9.0 , 275 ± 10.9 and 368 ± 15.1 ng; by 100–120 mesh DVB NTS were 275 ± 10.8 , 333 ± 13.0 , 432 ± 23.5 and 515 ± 38.0 ng; by SPME fibers were 93 ± 0.5 , 189 ± 0.8 , 271 ± 3.6 and 320 ± 6.3 ng.

Table 2 also shows the BTEX extraction rates (BTEX extraction mass/sampling time) for the NTS and SPME fibers during 1 h. The DVB-NTS extracted more BTEX mass than the 100 μm PDMS-SPME fibers during the extraction duration of 1 h. The smaller DVB adsorbents (120–100 mesh DVB) packed in the other NTS extracted more BTEX than the 60–80 mesh DVB-NTS. The BTEX extraction rates typically followed trends of adsorption performance for both DVB-NTS and PDMS fiber samplers [12,13].

According to experimental data (Table 2), total VOC adsorption capacities for 0.347 mg DVB in each NTS were 793 ng BTEXs for the 60–80 mesh DVB-NTS and 1555 ng BTEXs for 100–120 mesh DVB-NTS. That is, the equivalent BTEX adsorption capacities were 2285 ng/mg and 4481 ng/mg for the 60–80 mesh and 100–120 mesh DVB-NTS, respectively, under total BTEX concentrations of 400 ppmv (see notes in Table 2). In Equation (1), the VOCs mass (n) obtained via NTS extraction correlated with sampling time (t) given constant gaseous VOC concentrations (C). However, all concentrations of gaseous by-products were approximately 1.124 ppmv, much lower than the 400 ppmv simulated in the bulbs (Table 1). Based on experimental results, we reasonably infer that the amount of VOCs extracted does not reach the equilibrium adsorption mass of the DVB adsorbent, and these self-manufactured NTS can be used for passive TWA sampling.

3.3. Sampling Products from Incense Coils by SPME Fiber and NTS Samplers

Figure 3 plots the GC-FID extracted amount (integral area) of pollutants from burning incense coils when using SPME fibers and NTS for a TWA sampling time of 60 min. Figure 4 is the typical GC diagram for main by-products methanol, ethylbenzene, chloromethane, dichloromethane and 1,3-butadiene via extraction of 100–120 mesh DVB-NTS. Comparison of VOC adsorptions shows that VOC extractions by the NTS packed with DVB were higher than that of the 100 μm PDMS SPME fibers; the 100–120 mesh DVB-NTS performed better than the 60–80 mesh DVB-NTS and SPME fibers. For 1,3-butadiene, as an example, the extracted mass by the 100–120 mesh DVB-NTS was 1.47-fold than that by 60–80 mesh DVB-NTS, and 2.53-fold than that by the 100 μm PDMS SPME fibers. Experimental results demonstrated that commercially available SPME fibers could generally extract wide distributions of organic compounds, which is suggested by SUPELCO, the SPME manufacturer [17]. Man-made NTS packed with adsorbents with a proper diameter and affinity to adsorbates can extract a larger mass of alkane, aromatic and chlorinated compounds. These extraction distributions of combustion by-products from indoor consumer goods by NTS have roughly followed the same trends as those in earlier studies [11,12]: the smaller diameter adsorbent 100–120 mesh DVB-NTS can extract more VOCs than the 60–80 mesh DVB-NTS.

An earlier study [11] reported that after 3–5 samplings using the DVB-NTS for air-borne products of mosquito coils combustion, gaseous VOCs could not be collected due to clogging of the diffusion path between adsorbents packed in the samplers, and no further integral peak counts of VOCs could be analyzed via GC-FID. This implies that the main limitation of aerosol extraction by NTS may be clogging during the packing phase when sampling products from heated sources, particularly large diameter aerosols such as mosquito coil aerosols and cigarette smoke. To determine whether burning incense coil clogs the NTS, this work performed nine continuous 1-h samplings, using a single 60–80 mesh DVB-NTS and a 100–120 mesh DVB-NTS. The notable declined rates of extraction

performance for the eight compounds (Figure 3) was observed ranging from 31.8 to 60% for the 60–80 mesh DVB-NTS and from 40% to 64.4% for the 100–120 mesh DVB-NTS. The ESEM-FEI images (Figure 5) show surface views of the DVB particle before and after extracting the combustion products of incense coils, indicating that residues on the surface of the 100–120 mesh DVB particle were remarkably smaller than those on 60–80 mesh DVB particles. The phenomena may be resulted from the paths between the 100–120 mesh DVB particles are narrower than those between 60 and 80 mesh DVB particles. In other words, the 100–120 mesh DVB-NTS will clog more when sampling suspended aerosols than 60–80 mesh DVB-NTS.

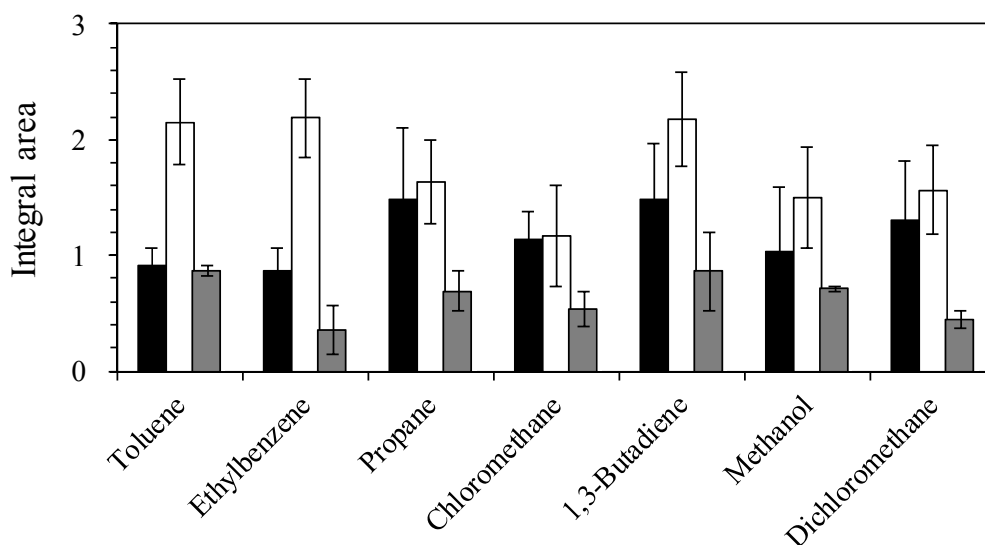


Figure 3. Main hydrocarbon analysis from combustion of incense coil. Columns ■, □ and ■ respectively indicate the integral area extracted by 60–80 mesh, 100–120 mesh NTS and SPME fibers via GC-GID analysis.

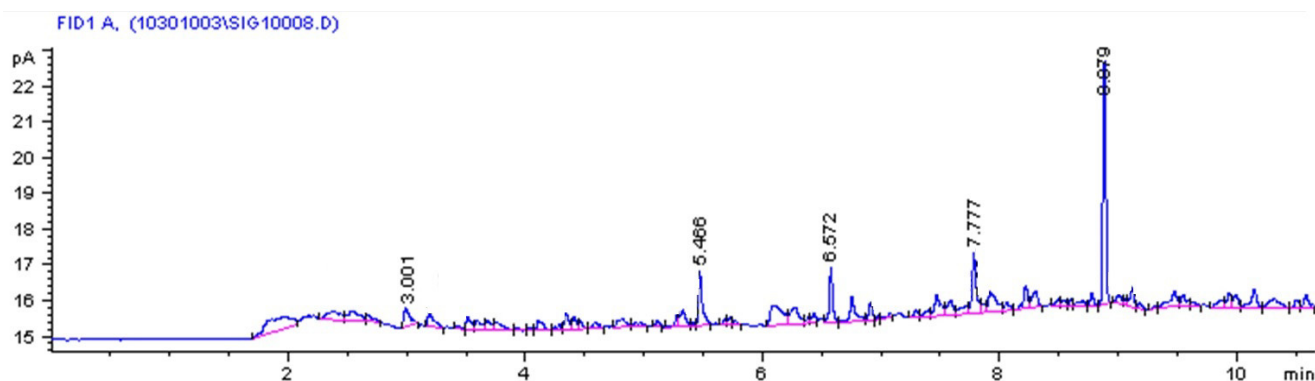


Figure 4. Typical gas chromatograph diagram of incense coil combustion using needle trap sampler. The sequence of retention times (min) respectively indicates the main by-products methanol, ethylbenzene, chloromethane, dichloromethane and 1,3-butadiene.

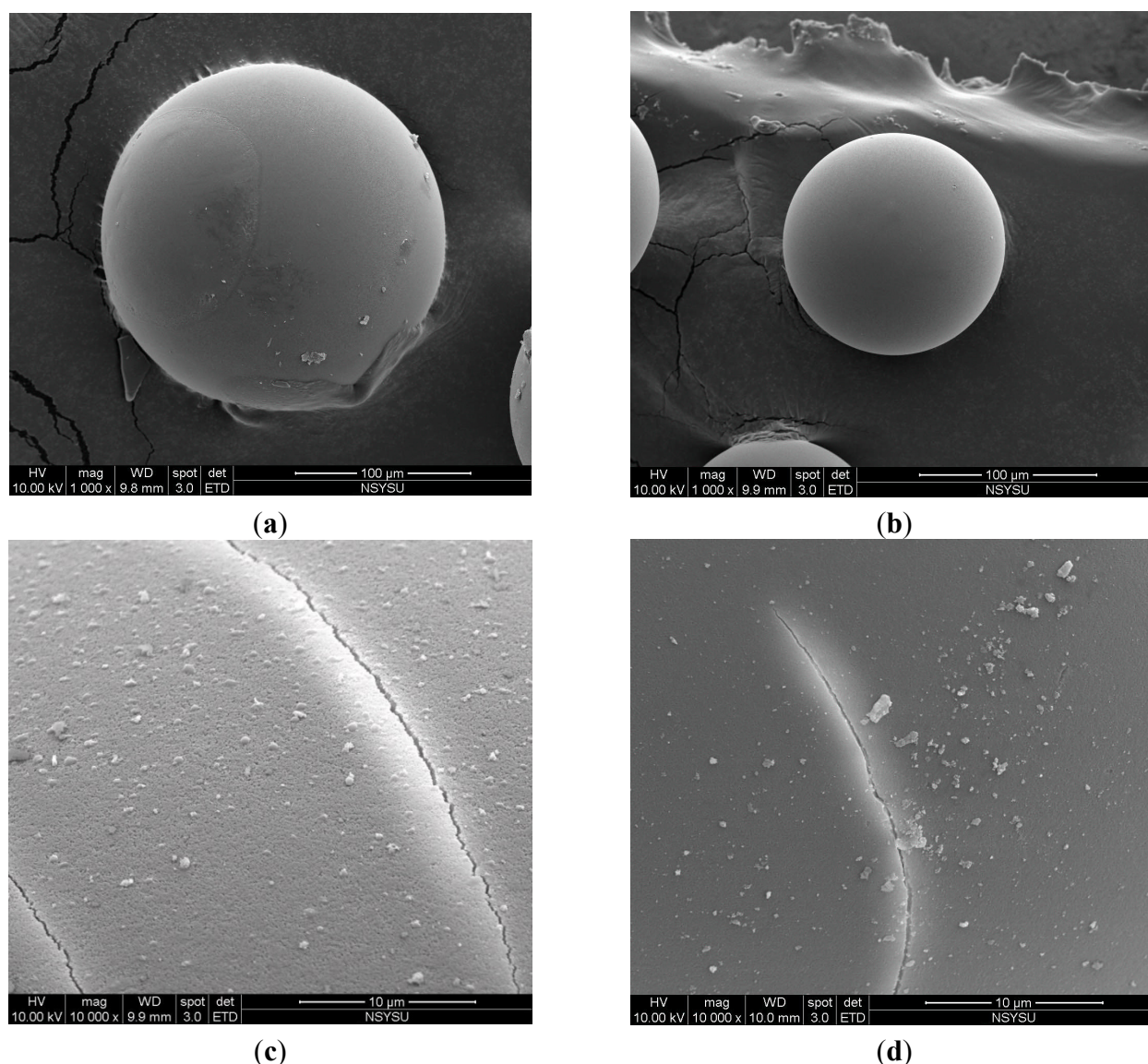


Figure 5. ESEM images showing the surface views of DVB particle before ((a) and (b)), and after ((c) and (d)) extracting the combustion products of incense coil. The crack marks in the background of photographs are the glue of adhesive tape immobilizing the examined particles. (a) Original 60–80 mesh DVB particle, and its diameter 196.41 μm (1000 \times); (b) Original 100–120 mesh DVB particle, and its diameter 125.62 μm (1000 \times); (c) and (d) More smaller residue laid on the surface of 100–120 mesh DVB particle than on 60–80 mesh DVB (10,000 \times)

4. Conclusions

The packed DVB-NTS is recommended for passive sampling of burning incense coils since it extracts more hydrocarbons compared to 100 μm PDMS SPME fibers when applied to sample indoor heating by-products. The NTS can be promoted as a novel personal sampler for air compounds in work places. Method 1501 of the National Institute for Occupational Safety and Health [18] requires workers to wear a charcoal sorbent tube connected to an active personal sampling pump when assessing exposure to organic compounds. Workers often complain about the inconvenience of carrying the

equipment needed to perform this assessment. This work evaluated the potential use of small badge-sized or pen-sized samplers for occupational hygiene applications.

The other advantage of SPME sampling technology is that it is a green analytical method. The father of green chemistry, Anastas, proposed that the principal objectives of green chemistry are reduction and elimination of the use and the generation of hazardous substances in 1999 [19]. Sampling and analyzing pollutants by NTS need only a little amount of analytical reagents (*i.e.*, thermal desorption via GC-FID and no any solvent demand), and the follow-up instrumentation procedures are easy. Therefore, NTS fully satisfies the requirements to be a green sampling method applied in the indoor atmosphere.

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Author Contributions

Wen-Hsi Cheng conceived and designed the experiments; Wen-Jiunn Tzeng, Chyn Her and Ya-Han Hsu performed the experiments; Wen-Hsi Cheng and Chin-Hsing Lai analyzed the data; Chin-Hsing Lai contributed analysis tools; Wen-Hsi Cheng wrote the paper.

Conflicts of Interest

The authors declare no conflict of interest.

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