



Application of solid phase microextraction and needle trap device with silica composite of carbon nanotubes for determination of perchloroethylene in laboratory and field



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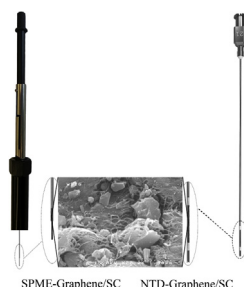
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HIGHLIGHTS

- Performance of SPME and NTD with new nano-structured material.
- Use of SPME and NTD with proposed sorbent for determination of PERC.
- The results for SPME and NTD performance compared to NIOSH 1003 method.
- Both NTD and SPME are powerful techniques for sampling of VOCs in air.

GRAPHICAL ABSTRACT



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ABSTRACT

In this paper solid phase microextraction (SPME) and needle trap device (NTD) as two in-progress air monitoring techniques was applied with silylated composite of carbon nanotubes for sampling and analysis of perchloroethylene in air. Application of SPME and NTD with proposed nano-structured sorbent was investigated under different laboratory and experimental parameters and compared to the SPME and NTD with CAR/PDMS. Finally the two samplers contained nano-sorbent used as a field sampler for sampling and analysis of perchloroethylene in dry cleaning. Results revealed that silica composite form of CNTs showed better performance for adsorbent of perchloroethylene. SPME and NTD with proposed sorbent was demonstrated better responses in lower levels of temperature and relative humidity. For 5 days from sampling the relative responses were more than 97% and 94% for NTD and SPME, respectively. LOD were 0.023 and 0.014 ng mL⁻¹ for SPME coated CNTs/SC and CAR/PDMS, and 0.014 and 0.011 ng mL⁻¹ for NTD packed with CNTs/SC and CAR/PDMS, respectively. And for consecutive analysis RSD were 3.9–6.7% in laboratory and 4.43–6.4% in the field. In the field study, NTD was successfully applied for determining of the PCE in dry cleaning. The results show that the NTD packed with nano-material is a reliable and effective approach for the sampling and analysis of volatile compounds in air.

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1. Introduction

Environmental and industrial exposures to air pollutants are common today due to variety of sources and extremely release of these compounds which raised concerns in developing and modern

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countries. Volatile organic compounds with widespread usage and high toxicity, are the major industrial and environmental pollutants, can cause adverse health effects [1,2].

There are different solvent-free sample preparation and introduction techniques for VOCs in air. Among all of them, solid phase microextraction (SPME) and needle trap device (NTD) have made more interest and are in use today. SPME, first introduced by Pawliszyn and co-workers in 1990 [3], is a solventless microextraction method that has been used for extraction of volatile organic compounds from a variety of matrices [4–6]. Similar to SPME the NTD technique was first introduced by Pawliszyn et al. in 2001 [9]. The NTDs combines the idea of exhaustive active as well as passive diffusive micro-sampling with newly created microextraction techniques. The particular advantages make SPME and NTDs two suitable techniques for environmental and workplace surveys hazardous compounds [7].

Until now, SPME and NTD has been used with commercial sorbents such as polydimethylsiloxane (PDMS), divinylbenzene (DVB) and Carboxen1000 [8,9], Carboxen X and Tenax [10–12], and Porapak Q [13], Carbon nanotubes (CNTs) with very high surface area, potentially can be used as sorbent media for sampling of different compounds. CNTs with special characteristics such as mechanical strength and chemical stability, were first discovered by Sumio Iijima in 1991 [14]. CNTs's remarkable thermal, chemical stability and high surface area may make it a suitable SPME and NTD sorbent for the extraction of benzenoid-form compounds. However, without functionalization, carbon nanostructure materials are limited in their capacity and performance to adsorb analytes. Due to the limits of physicochemical approaches, chemically modified forms of CNTs may provide an alternative approach by altering CNTs's properties [15]. To date chemical modification and functionalization of CNTs have been focused on improving its adsorptive characteristics for sampling analytes by incorporating CNTs in a composite material [15–17].

The sol–gel technology introduced a useful approach for synthesis of variety of sorbent. With this technique, sorbent can be synthesized with suitable homogeneousness and purity. The lower temperature of sol–gel process as well as strong mixing for components, the ability for particle shape and size control, and enhanced thermal stability for higher thermal desorption provide the sol–gel technology as a desired method for synthesizing different sorbents [18,19].

In this paper, SPME and NTDs were prepared with silylated carbon nanotubes and used for sampling and analysis of perchloroethylene in air. Performance of these micro-samplers and their proposed sorbent were investigated in laboratory for optimization of parameters and then in field study. A comparative study was carried out, for this SPME and NTD with proposed sorbent was compared to the SPME and NTD with CAR/PDMS in laboratory phase of the study and then compared to the NIOSH 1003 technique in the field study.

2. Materials and methods

2.1. Reagents and standards

CNTs-COOH with purity higher than 90%, with 1–2 nm O.D., 0.8–1.6 nm I.D. and length of 5–30 μm and rate of surface carbon atom 8–10 mol %, were obtained from Chengdu Organic Chemicals (Chinese Academy of Sciences). The -COOH content of CNTs was 2.73 wt% and special surface area (SSA) was more than 380 $\text{m}^2 \text{g}^{-1}$. Perchloroethylene (PCE) with highest purity available was obtained from Sigma–Aldrich (Germany). Trifluoroacetic acid (TFA), tetramethylorthosilicate (TMOS) and polymethyl hydrogensiloxane (PMHS) were supplied from Merck (Darmstadt, Germany). Sodium

dodecylbenzenesulfonate (SDBS) was purchased from Fluka (Buchs, Switzerland). Deionized water used for preparation of SDBS solution (5% w/v) as a surfactant was obtained from a TKA (Germany) ultra-water system.

2.2. Instrumentation

Analysis of samples was performed with Varian 3800 GC with split-splitless injector and VOCOL capillary column (with 60 m \times 0.25 mm \times 0.25 μm). GC equipped with a Saturn 2200 MS system. The GC oven was initially set at 40 $^\circ\text{C}$ held for 4 min, and then ramped at 6 $^\circ\text{C}/\text{min}$ to 160 $^\circ\text{C}$. MS analyses carried out with using ultra-pure Helium as carrier gas with a low constant inlet of 1.0 mL min^{-1} . After purification for water vapor, hydrocarbons, and oxygen the MS analyses were carried out in full-scan mode, with scan range 40–650 EI; electron impact ionization was applied at 70 eV, and the transfer line was maintained at 220 $^\circ\text{C}$. A home-made chamber was used for adjustment of concentration, temperature and humidity of sample matrix. A 21-gauge needle with 10.5 cm in length, 510 μm I.D., and 820 μm O.D. was purchased from Kosan LTD (Japan). SPME holder and CAR/PDMS commercial sorbent were purchased from Supelco (Bellefonte, PA, USA). Two syringe pumps, JMS SP-510 (Hiroshima, Japan), were used for providing determined injection of the calculated amount of PCE into the sampling chamber and also for carrier gas injection into the GC injection port. A low volume sampling pump, SKC 222 series (PA, USA), with a sampling flow rate of up to 200 mL min^{-1} was used for the performance evaluation of NTDs and accurate drawing of air inside the needles and through the sorbent bed.

2.3. Preparation of silica composite of CNTs

Sol–gel technology was used for preparation of CNTs/SC. For this 2 mg of -COOH functionalized CNTs was dispersed in 50 μL of SDBS solution (5% w/v) Eppendorf vial. The obtained suspension was agitated by ultrasonic bath for 15 min and then 400 μL TMOS and 50 μL PMHS were added and the mixture was sonicated for 30 min. Afterward, 50 μL of TFA was added, and the total solution was sonicated in an ultrasonic bath for 15 min. To remove the non-reacted material, the resulting mixture was refluxed in dichloromethane and ethanol solution (2:1 v/v) for 1 h. Finally, the mixture was centrifuged at 4000 RPM for 10 min, and then the obtained solid sorbent was dried in an oven with a temperature of 120 $^\circ\text{C}$ for 2 h, and after weighing the produced CNTs/silica meshed over the range of 53–63 μm . Fig. 1 show the SEM-EDX analysis for CNTs/SC as sorbent.

2.4. Preparation of SPME and NTD with proposed sorbent

To prepare the SPME with the proposed sorbent, a commercial SPME was modified by placing a 12 cm length of 100 μm fused-silica rod into a 25-gauge needle. Then, to protect it, the 25-gauge needle was inserted into a 21-gauge needle. This arrangement was inserted into a black cylinder through a hexagonal nut. With this device 2 cm of fused silica can be exposed, and finally the end 1 cm used for sorbent coating. For this issue the fused silica rod was immersed into a 1 M NaOH solution for 1 h for composing silanol groups on the surface of the fiber. Afterward the fiber's body was neutralized by immersing into 0.1 M HCl solution and rinsed with distilled water. Finally the desired thickness of the proposed sorbent was coated on the 1 cm end of fused silica fiber by dipping it into the muggy sol solution for 2 min and then weighed by inserting into the GC injector for additional 2 min. This process was repeated and controlled by using a phase contract microscope equipped with a suitable graticule until a 50 μm coating was

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