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Trends in Analytical Chemistry

journal homepage: www.elsevier.com/locate/trac

Methods for coating solid-phase microextraction fibers with carbon nanotubes

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ARTICLE INFO

Keywords:

Carbon nanotube
Chemical bonding
Coating
Electrochemical deposition
Electrophoretic deposition
Extraction efficiency
Fiber
Physical deposition
Sol–gel technique
Solid-phase microextraction

ABSTRACT

The presence of carbon nanotubes (CNTs) on the surfaces of solid-phase microextraction (SPME) fibers can enhance the efficiency of the extraction. Their unique properties, such as large surface area, and excellent chemical, mechanical and thermal stability, make such fibers very attractive as adsorbents in SPME. The current review concentrates on the different coating methods, such as chemical bonding, physical attachment, sol–gel technique, electrochemical deposition and electrophoretic deposition (EPD) that coat the SPME fibers with CNTs. We also briefly discuss the advantages and the disadvantages of these coating methods.

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Abbreviations: ATRP, Atom transfer radical polymerization; BTEX, Benzene, toluene, ethyl benzene, and xylenes; CE, Capillary electrophoresis; CEC, Capillary electrochromatography; CNT, Carbon nanotube; CP, Conductive polymer; CW-TPR, Carbowax/template resin; DAD, Diode-array detector; DMF, Dimethyl formamide; DVB, Divinylbenzene; ECD, Electron-capture detector; EPD, Electrophoretic deposition; ETBE, Ethyl tert-butyl ether; FID, Flame-ionization detector; GC, Gas chromatography; HPLC, High-performance liquid chromatography; HS, Headspace; IC, Ion chromatography; LC, Liquid chromatography; MS, Mass spectrometry; MTBE, Methyl tert-butyl ether; MWCNT, Multi-walled carbon nanotube; NTD, Needle-trap device; NSAID, Non-steroidal anti-inflammatory drug; OPP, Organophosphorous pesticide; PA, Polyacrylate; PAH, Polycyclic aromatic hydrocarbon; PAN, Polyacrylonitrile; PANI, Polyaniline; PBDE, Poly brominated diphenyl ether; PDMS, polydimethylsiloxane; PEG, Polyethylene glycol; PoPD, Poly-o-phenylenediamine; PPy, Polypyrrole; PVC, Polyvinyl chloride; Py, Pyrrole; SDS, Sodium dodecyl sulfate; SPME, Solid-phase microextraction; SWCNT, Single-walled carbon nanotube; TAME, Tert-amyl methyl ether; TFA, Trifluoroacetic acid; 3-TMSPT, 3-(trimethoxysilyl)-1-propanthiol; TSO-OH, Hydroxyl-terminated silicon oil; VOC, Volatile organic compound.

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1. Introduction

The sample-preparation step is of fundamental importance for the accuracy and the reliability of the final results of analysis. It has required implementation of new, less time-consuming methods, especially solventless or solvent-free sample-preparation techniques.

In 1990, solid-phase microextraction (SPME), a solvent-free technique introduced by Arthur and Pawliszyn [1], integrated sampling, extraction, and sample introduction into one step. Compared to conventional sample-preparation methods, SPME provides many advantages, including speed, simplicity and ease of operation. Since its introduction, it has been successfully applied to sampling and analysis of a wide variety of compounds, especially for the extraction of volatile and semi-volatile organic compounds from environmental, biological and food samples [2].

SPME is based on the establishment of the analyte distribution between the liquid or gaseous sample and a sorbent phase on a metallic or fused silica substrate, which is part of the syringe needle. Desorption of analytes occurs thermally when used for GC analysis. The analytes are desorbed from the fiber in the GC injection port, and the analytes are swept onto the column by the carrier gas for separation. In the case of LC, CE, or CEC, solvent (usually the mobile phase) is used to desorb the analytes.

The type of coating used in the SPME fiber plays a crucial role in the extraction and desorption process, since its efficiency will depend on the distribution constant between the analytes and the stationary phase. In addition, it will provide some selectivity to the SPME process towards the analytes versus other matrix compounds. Different coating types have been introduced to accomplish the extraction capability of a wide range of analytes. Also, sample capacity of the fiber, resistances to the organic solvents and the thermal, mechanical and chemical stability of the SPME coatings determine the scope of their applications and their commercial prospects.

Compared with other materials used for SPME coatings, nanomaterials offer a significant greater surface area-to-volume ratio that promises much greater extraction capacity and efficiency. Carbon-based nanomaterials have fascinated the scientific community since their discovery. In recent years, a large number of carbon-based nanoparticles were investigated as sorbent materials in sample preparation, including nanodiamonds, fullerenes, carbon nanotubes (CNTs), graphene, carbon nanofibers, carbon nanocones, disks and nanohorns, and their functionalized forms. The characteristic structures of carbon-based nanomaterials allow them to interact with organic molecules via non-covalent forces, such as hydrogen bonding, π - π stacking, electrostatic forces, van der Waals forces and hydrophobic interactions. These interactions and their hollow or layered nanostructures make them good candidates for use as adsorbents [3].

CNTs were first described by Iijima [4] and, since their discovery, they have contributed to the development of studies in the fields of physics, chemistry and materials science. CNT is one form of carbon, with nm-sized diameter and μm -sized length (where the length-to-diameter ratio exceeds 1000). The atoms are arranged in hexagons, the same arrangement as in graphite. The structure of the CNT comprises cylindrical graphitic sheet (called graphene) rolled up into a seamless cylinder with diameter of the order of 1 nm. According to the number of tubes, the CNTs have two morphologies: single-walled (SWCNTs) and multi-walled (MWCNTs).

SWCNTs can be considered to be formed by rolling a single layer of graphite (called a graphene layer) into a seamless cylinder (long wrapped graphene sheets). As stated before, CNTs generally have a length-to-diameter ratio of about 1000 and they can be considered as nearly a one-dimensional structure. Most SWCNTs have a diameter of close to 1 nm.

MWCNTs can be considered as a collection of concentric SWCNTs (comprising multiple layers of graphite rolled in on themselves to form a tube shape) with different diameters. The length and the diameter of SWCNTs differ a lot from those of MWCNTs, and, of course, their properties are also very different (such as optical activity, mechanical strength and electrical conductivity) [5].

Apart from their thermal stability, excellent mechanical and electric properties, and their highest tensile strength among known materials, CNTs have a large surface area. These characteristics make CNTs very attractive as adsorbents in SPE and SPME devices. However, due to the rigidity, the chemical inertness, and the strong π - π interactions of nanotubes, pure CNTs cannot be processed, as they are difficult to dissolve or to disperse in common organic solvents or polymeric matrices. The side walls of CNTs must therefore be modified (covalent or non-covalent) to improve their dispersion or solubility in solvents or polymers [6].

The objective of this work is to present an overview of the applications of CNTs in coating SPME fibers. Clearly, it is impossible to compare all the studies from all aspects of this vast subject in one review article. Hence, we briefly discuss the most characteristic advantages and disadvantages of each coating technique in preparing CNT-SPME fibers. The CNT coating of SPME fibers can be divided into four main categories:

- physical deposition;
- sol-gel technique;
- chemical bonding; and,
- electrochemical deposition and electrophoretic deposition (EPD)

We devote this article to discussion of different techniques for the application of CNTs in SPME coatings and conclude with advantages and drawbacks.

2. CNT-based coatings of SPME fibers

2.1. Physical deposition

A convenient, fast way to prepare an SPME fiber is to dip the fiber support into an adsorbent suspension, in which the stationary materials are dispersed in the solvent or organic binder, followed by drying or thermal conditioning for immobility. This procedure can be repeated several times until the desired thickness is obtained. However, one of the advantages is that different solid sorbents with specific functionality can be easily incorporated on the fiber support as a novel extraction phase for SPME. However, in physical deposition, absence of chemical bonds between the substrate and the coating typically provides fibers with lower thermal stability and lower resistance to organic solvents and strong acidic and basic solutions. Moreover, such chemical bonds could block the coating pores or even change extraction selectivity. CNTs can be immobilized on the SPME fiber by physical deposition or dipping techniques.

Wang et al. [7] used a fused-silica fiber without the polyimide protective layer as probe support. It was then immersed in a dimethylformamide solution containing MWCNTs at a concentration of 20 mg mL⁻¹. The organic solvent was further removed by heating at 160°C. The process was repeated until the desired coating thickness was reached, in this case, ~40 μm . The fiber was used for the determination of polybrominated diphenyl ethers (PBDEs) in milk and water samples by GC-ECD. In headspace mode, the fiber could be used up to 100 times with negligible loss of performance. The comparison with other coatings, namely activated carbon and poly (5% dibenzene-95% dimethylsiloxane) (PDMS-DB) showed that the MWCNT coating reached enhancement factors 4–4.5 times greater than activated carbon and PDMS-DB.

Also, in order to immobilize CNTs on the fiber substrate, different kinds of adhesive have been used (e.g., a mixture of terpineol,

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