



## Review

# Polyethylene glycol-coated solid-phase microextraction fibres for the extraction of polar analytes—A review

Agata Spietelun<sup>a</sup>, Michał Pilarczyk<sup>a</sup>, Adam Kloskowski<sup>a,\*</sup>, Jacek Namieśnik<sup>b</sup>

<sup>a</sup> Gdansk University of Technology, Chemical Faculty, Department of Physical Chemistry, 11/12 Narutowicz St., Gdańsk 80-233, Poland

<sup>b</sup> Gdansk University of Technology, Chemical Faculty, Department of Analytical Chemistry, 11/12 Narutowicz St., Gdańsk 80-233, Poland

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## ABSTRACT

The article discusses the merits and limitations of commercially available solid-phase microextraction (SPME) fibres and compares them with the new type of extraction coatings, in particular those containing polyethylene glycol as sorbent, as well as the methods of the preparation of the latter. It also analyses their possible application for the extraction of a broad spectrum of analytes, with particular emphasis on the sampling of polar organic compounds from different media.

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## 1. Introduction

One of the most important challenges facing contemporary analytical chemistry is the determination of a wide range of analytes in samples of media with a complex matrix composition. It has also become imperative that new analytical methodologies should comply with the principles of sustainable development and green chemistry. Unfortunately, however, most analytical techniques are insufficiently sensitive for the direct determination of analytes present in trace or ultratrace amounts; moreover, in many cases, the analytes have to be separated from the matrix before the proper analysis can commence.

The sample preparation step is of fundamental importance for the accuracy and reliability of the final analysis results. This

**Abbreviations:** BTEX, benzene, toluene, ethylbenzene, xylenes; CAR, carboxen; CE, capillary electrophoresis; CV, cyclic voltammetry; CW, carbowax; DVB, divinylbenzene; GC, gas chromatography; HPLC, high performance liquid chromatography; HS-SPME, headspace solid-phase microextraction; ICP, inductively coupled plasma; MAH, monocyclic aromatic hydrocarbons; MESI, membrane extraction with sorbent interface; M-SPME, membrane solid-phase microextraction; P3DDT, poly(3-dodecylthiophene); PA, polyacrylate; PAH, polycyclic aromatic hydrocarbons; PANI, polyaniline; PCB, polychlorinated biphenyls; PDMS, polydimethylsiloxane; PEG, polyethylene glycol; PEO, polyethylene oxide; PPy, polypyrrole; SFC, supercritical fluid chromatography; SPME, solid-phase microextraction; VOC, volatile organic compounds.

\* Corresponding author. Tel.: +48 58 347 2110; fax: +48 58 347 2694.

E-mail addresses: [adamklos@wp.pl](mailto:adamklos@wp.pl), [adam.kloskowski@pg.gda.pl](mailto:adam.kloskowski@pg.gda.pl) (A. Kloskowski).

**Table 1**  
Manufacturer's recommendations and examples of applications of commercially available extraction coatings.

Type of SPME fibre	Analytes	Example of use	Determination technique
100 µm PDMS	Volatiles (MW 60–275)	VOC, BTEX, chlorobenzenes [37], PAH, MAH [38,39], pesticides, triazines, herbicides [40,41], PCB [42]	GC/HPLC
30 µm PDMS	Non-polar semi-volatiles (MW 80–500)	PAH [38,39]	GC
7 µm PDMS	Non-polar high molecular weight compounds (MW 125–600)	PAH [38,39]	GC
65 µm PDMS/DVB	Volatiles, amines, nitro-aromatic compounds (MW 50–300)	VOC, BTEX, chlorobenzenes [35,43], PAH [39], aromatic amines, ketones, alcohols, aldehydes, terpenes [44]	GC
75/80 µm CAR/PDMS	Gases and low molecular weight Compounds (MW 30–225)	VOC, BTEX, chlorobenzenes [45], PAH, MAH [39], metals*: arsenic, selenium, antimony, tin [46]	GC, *ICP
50/30 µm DVB/CAR/PDMS	Flavour compounds: semi-volatile, volatile, C3–C20; trace compound analysis (MW 40–275)	PAH, MAH [39]	GC
85 µm PA	Polar semi-volatiles (MW 80–300)	Pesticides, triazines, herbicides [40,47,48], phenols [49,50]	GC, HPLC
60 µm PEG (CW/DVB)	Alcohols, polar compounds (MW 40–275)	Aromatic amines, ketones, alcohols, aldehydes, terpenes [34,51]	GC

\* Indicates that for metals, ICP was used as the determination technique.

has required the implementation of new, less time consuming methods, especially solventless/solvent free sample preparation techniques. One of the most commonly used solventless sample preparation techniques is solid-phase microextraction (SPME) [1]. An SPME device consists basically of a silica fibre or metal core, coated with a thin layer of a suitable polymeric sorbent or immobilized liquid, fixed within the needle of a syringe-like arrangement. Extraction is performed by immersing the fibre in a gaseous medium or a relatively pure liquid medium; analytes can also be sampled from the headspace (HS-SPME) [2]. Headspace sampling is particularly useful for analysing the composition of solid samples or samples containing matrix constituents that could contaminate or damage the fibre (e.g. petrochemical samples, high-molecular weight compounds, humus compounds [3]) and in the extraction of very volatile analytes such as PAHs, BTEX and VOCs [3,4]. On completion of the extraction the fibre is placed directly in the injector of the monitoring instrument (usually GC, but can also be HPLC [5–7], CE [8] or SFC [9]), where desorption of the analytes absorbed on the fibre takes place [10].

SPME is used to sampling a wide range of volatile and medium volatile analytes from gases, liquids and solids with a diverse matrix composition (environmental, biological, contaminated, suspended samples) [11–15]. The technique's great popularity is due to its many merits, the most important of which are as follows:

- simplicity of operation,
- relatively short extraction time,
- lack of artefacts,
- no need of expensive or complex equipment,
- possibility of full automation,
- easy interfacing with GC systems,
- applicability to both *in situ* and *in vivo* sampling.

SPME has many advantages and a broad field of application, and so much research work has been aimed at improving recoveries and enhancing the sensitivity of the method: this has led to the development of completely new constructional approaches, for example, a fibre with internal cooling [16–18] or in-tube SPME [19], as well as modifications of existing techniques, mainly through their miniaturization and automation [20,21]. Aside from this, work is also in hand to examine the dependence of analyte recovery on process parameters (extraction fibre structure, extraction temperature and time, method of stirring etc. [22–26], or additional processing of the medium under scrutiny [27–30]).

In its basic form, however, SPME has certain limitations: it is not very selective, the fibre is mechanically weak, and only a small selection of extraction fibre coatings is commercially available. In addition, recoveries (sample-to-fibre) are relatively low, particularly of polar analytes sampled from matrices with polar constituents. Work is therefore under way to develop mechanically stronger and thermally more resistant fibres, as well as new sorbents for possible implementation as polar SPME fibre coatings. To increase the affinity of sorbents for polar analytes, more polar sorbents as extraction coatings ought to be used. But this would result in an equally enhanced affinity for the polar constituents of the sample matrix and could lead to competing adsorption processes, and eventually to the analyte being leached out of the extraction fibre. Such an approach is thus a non-starter. SPME fibre coatings usually take the form of immobilized liquids like non-polar PDMS and the highly polar PEG [31].

If SPME is used to sample alcohols and the most polar compounds with molecular masses from 40 to 275, manufacturers recommend using a PEG fibre as the sorption phase [32]. PEG is a particularly interesting material since for small molecular masses it is a pseudoliquid; analyte retention is thus by absorption or dissolution in the extractant. In contrast to adsorption, absorption permits the release of analytes from the extraction fibre coating at low temperatures; compounds with a low degradation temperature will therefore not decompose. In addition, PEG is thermally highly stable, has short extraction and analyte desorption times, and gives excellent recoveries of polar analytes. This material is therefore of increasing interest to analysts in their search for highly selective sorbents, which can be employed as polar stationary phases in extraction techniques for sample preparation.

## 2. SPME fibre coatings

Even a cursory glance at the literature reports on SPME shows that the method's poor selectivity and the limited choice of extraction fibre coatings are the main problems limiting its routine usage in analytical practice. The type of sorbent used as fibre coating is the key parameter governing the recovery and selectivity of SPME [13]. Such coatings as commercially available are made from polydimethylsiloxane (PDMS), divinylbenzene (DVB), polyacrylate (PA), Carboxen (CAR) and polyethylene glycol (Carbowax, CW), obtainable in various thicknesses and combinations (PDMS/DVB, PDMS/CAR, CW/DVB) [33–35]. Unfortunately, these fibres have a poor affinity for polar analytes and do not always meet expectations

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