



Recent advances in solid-phase sorbents for sample preparation prior to chromatographic analysis



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ABSTRACT

Sample preparation is a crucial bottleneck in the whole analytical process. Solid-phase sorbents (SPSs) have aroused increasing interest in research on sample preparation, as they have key roles in obtaining high clean-up and enrichment efficiency in the analysis of trace targets present in complex matrices. The objective of this review is to provide a broad overview of the recent advances and applications of SPSs in sample preparation prior to chromatographic analysis, during the period 2008–13. We include SPSs, such as molecularly-imprinted polymers, carbon nanomaterials, metallic nanoparticles and metal organic frameworks, focusing on solid-phase extraction, solid-phase microextraction, matrix solid-phase dispersion and stir-bar sorptive extraction of typical pollutants in environmental, biological, food and pharmaceutical samples. We propose remaining challenges and future perspectives to improve development of new SPSs and to apply them further.

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Abbreviations: 2- or 4-VP, 2- or 4-vinylpyridine; AA, Acrylic acid; APTES, Aminosilica monomer; BPA, Bisphenol A; CNT, Carbon nanotube; CP, Chlorophenol; CTAB, Trimethylammonium bromide; DLLME, Dispersive liquid-liquid microextraction; DVB, Divinylbenzene; EDC, Endocrine-disrupting chemical; EGDMA, Ethylene glycol dimethacrylate; FID, Flame-ionization detector; FLD, Fluorescence detector; HEMA, 2-hydroxyethyl methacrylate; HLB, Hydrophilic-lipophilic balance; LOD, Limit of detection; LOQ, Limit of quantitation; MAA, Methacrylic acid; MAX, Mixed-mode/anion-exchange; MBAA, N,N-methylenebisacrylamide; MCX, Mixed-mode/cationic-exchange; MIP, Molecularly-imprinted polymer; MISPE, Molecularly-imprinted solid-phase extraction; MNP, Magnetic nanoparticle; MOF, Metal-organic framework; MSPD, Matrix solid-phase dispersion; MWCNT, Multi-walled carbon nanotube; NP, Nanoparticle; PAH, Polycyclic aromatic hydrocarbon; PDDA, Poly(diallyldimethylammonium chloride); PS-DVB, Poly(styrene-divinylbenzene); SBSE, Stir-bar sorptive extraction; SPME, Solid-phase microextraction; SPS, Solid-phase sorbent; SWCNT, Single-walled carbon nanotube; TRIM, Trimethylolpropane trimethacrylate; WAX, Weak anion-exchange.

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1. Introduction

Sample preparation is a crucial part of chemical/biological analysis and in a sense is considered the bottleneck of the whole analytical process. The main objectives of sample preparation are the removal of potential interferences, analyte preconcentration, and converting (if needed) the analyte into a more suitable form for detection or separation. In recent decades, solid-phase extraction (SPE) has played a crucial role in sample preparation, replacing the classic liquid–liquid extraction (LLE), in environmental, food and biological analyses [1–3]. In the past, some classic materials, such as C₈, C₁₈, hydrophilic-lipophilic balance (HLB, the sorbent of which is a macroporous copolymer made from a balanced ratio of two monomers including hydrophilic and lipophilic ones), mixed-mode/cationic-exchange (MCX), mixed-mode/anion-exchange (MAX) and weak anion-exchange (WAX) were commonly used for SPE [4]. More recently, a large number of new solid-phase sorbents (SPSs), such as molecularly-imprinted polymers (MIPs) [5], magnetic nanomaterials [6] and carbon nanoparticles (NPs) [7] have been proposed and applied for sample preparation. Moreover, although some powerful techniques (including HPLC-MS/MS and GC-MS/MS) are also subjected to improving the detectability for analytes in complex matrices, new objectives of sample preparation have been set, such as using small initial volume or weight of sample, improvement of selectivity in extraction, facilitating automation, and minimizing the amount of glassware and organic solvents to be used. Thus, some other sample-preparation methodologies, such as solid-phase microextraction (SPME), stir-bar sorptive extraction (SBSE) and matrix solid-phase dispersion (MSPD), have been effective for the above purposes. These methods, together with SPE, stimulated rapid advances of the new SPSs. A number of research efforts dealing with novel SPSs specifically developed for these applications in chromatography have been reported and some have been reviewed.

Lucena et al. [7] gave a general overview of the use of NPs mainly including carbon, metallic, silica and imprinted NPs to perform sample preparation in SPE and SPME.

Turiel and Martín-Esteban [8,9] reviewed the use of MIPs in SPE, SPME, MSPD and SBSE.

Augusto et al. [10] outlined progress in research on some new classes of sorbents, such as carbon nanotubes (CNTs), MIPs and sol-gel coatings for SPME fibers and related techniques, and for extraction and microextraction techniques.

Tankiewicz et al. [11] described some SPSs applied in SPE, SPME and SBSE.

Several recent reviews were released in 2013. For example, Mehdiinia and Aziz-Zanjani [12] reviewed advances for sensitive, rapid and selective extraction in different configurations of SPME, containing some new SPSs.

Spiteluna et al. [13] summarized recent developments and future trends in SPME toward green analytical chemistry containing various novel SPSs.

Yang et al. [14] presented an overview of microextraction techniques for the determination of volatile and semi-volatile organic compounds from plants, involving SPS preparation and applications.

Namera and Saito [15] reviewed some new SPSs used in SPE and SPME methods for bioanalysis.

An important trend shared by the fundamental research on the above extraction techniques relates to the development and the characterization of new sorbents. It has become a hot issue to research new advanced materials, involving several branches of disciplines, such as materials science, nanotechnology, polymer synthesis and analytical chemistry. The development of SPSs generally aims for: high selectivity, good sorptive/adsorptive capacity, enhanced thermal, chemical or mechanical stability and improved lifetime of devices employing them as sorbent/adsorbent media [10]. In this review, we therefore provide an updated, essential summary of the most important features and applications of new materials as SPSs in SPE, SPME, MSPD and SBSE prior to chromatographic analysis. We summarize highlighted applications of these new SPSs for the extraction of environmental, biological, food and pharmaceutical samples. We also discuss the present limitations and expected future trends of SPSs in sample preparation for better advancement.

2. Novel solid-phase sorbents

Traditional SPSs, such as silica NPs, C₈, C₁₈, poly(styrene-divinylbenzene) (PS-DVB), methacrylate-DVB resins, macroporous poly(N-vinylpyrrolidone-DVB) polymers and some others, usually used mixed-mode ion-exchange sorbents, including MCX, MAX and WAX, which have already been reviewed in detail by Alberti et al. [4]. The present review focuses on the preparation and the applications of novel SPSs, mainly including MIPs, carbon nanomaterials, metallic NPs and metal-organic frameworks (MOFs).

2.1. Molecularly-imprinted polymers

In recent years, MIPs proved to be useful materials in several areas of analytical chemistry [5,16–20]. MIPs are synthetic materials with artificially-generated recognition sites able to specifically rebind a target molecule in preference to other closely-related compounds. Functional monomers and cross-linking agents are copolymerized in the presence of a template (the imprint molecule) in a suitable solvent, leading to a highly cross-linked three-dimensional network polymer. The monomers are chosen by considering their ability to interact with the functional groups of the template molecule. Subsequent removal of the imprint molecule leaves cavities with size, shape and chemical functionality complementary to those of the template. The inherently high selectivity associated with MIPs has made them ideal sorbents to be used in SPE, SPME, MSPD and SBSE [18]. Recently, our group reviewed recent advances in molecular imprinting technology, with particular emphasis on significant progress of novel imprinting methods and MIP materials, some challenges and effective strategies, and highlighted applications of MIPs [5].

The mechanism of MIP formation mainly includes free-radical polymerization and the sol-gel process. Use of free-radical polymerization became popular recently, including bulk polymerization, suspension polymerization, emulsion polymerization, seed polymerization and precipitation polymerization. During

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