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Review

Magnetic solid-phase extraction using carbon nanotubes as sorbents: A review

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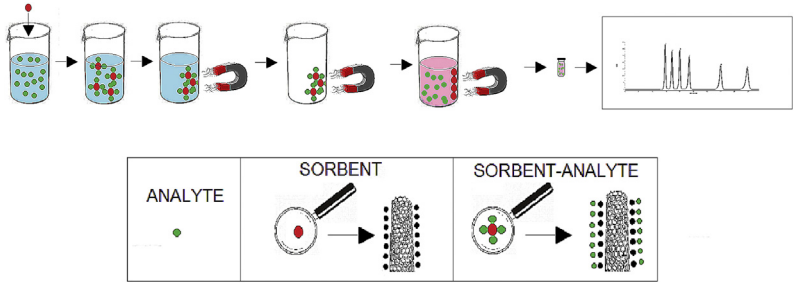
HIGHLIGHTS

- Magnetic carbon nanotubes (M-CNTs) as a sorption substrate for SPE.
- Synthesis of M-CNTs and approaches for their use in M-SPE.
- M-CNTs as sample preparation substrates for organic and inorganic analysis.

GRAPHICAL ABSTRACT



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ABSTRACT

Magnetic solid-phase extraction (M-SPE) is a procedure based on the use of magnetic sorbents for the separation and preconcentration of different organic and inorganic analytes from large sample volumes. The magnetic sorbent is added to the sample solution and the target analyte is adsorbed onto the surface of the magnetic sorbent particles (M-SPs). Analyte-M-SPs are separated from the sample solution by applying an external magnetic field and, after elution with the appropriate solvent, the recovered analyte is analyzed. This approach has several advantages over traditional solid phase extraction as it avoids time-consuming and tedious on-column SPE procedures and it provides a rapid and simple analyte separation that avoids the need for centrifugation or filtration steps. As a consequence, in the past few years a great deal of research has been focused on M-SPE, including the development of new sorbents

Abbreviations: AOPPs, aryloxyphenoxy-propionate herbicides; BADGE, bisphenol A diglycidyl ether; BPA, bisphenol A; BPF, bisphenol F; BFDGE, bisphenol F diglycidyl ether; BSA, bovine serum albumin; CLC, capillary liquid chromatography; CNTs, carbon nanotubes; CRM, certified reference material; DBP, dibutyl phthalate; DEHP, di-2-(ethylhexyl) phthalate; DLLME, dispersive liquid–liquid microextraction; DSPE, dispersive solid phase extraction; EGDMA, ethylene glycol dimethacrylate; ETAAS, electrothermal atomic absorption spectrometry; FAAS, flame atomic absorption spectrometry; FIA, flow injection analysis; GC, gas chromatography; GC–MS, gas chromatography–mass spectrometry; GC–MS/MS, gas chromatography–tandem mass spectrometry; HGAAS, hydride generation atomic absorption spectrometry; HGAFS, hydride generation atomic fluorescence spectrometry; HPLC, high pressure liquid chromatography; HPLC–DAD, high pressure liquid chromatography–diode array detection; ICP–MS, inductively coupled plasma–mass spectrometry; ICP–OES, inductively coupled plasma–optical emission spectrometry; MAA, methacrylic acid; MALDI–TOF-MS, matrix assisted laser desorption ionization–time of flight-mass spectrometry; MEKC, micellar electrokinetic chromatography; MIP, molecularly imprinted polymer; MISPE, molecularly imprinted solid phase extraction; M-CNT, magnetic carbon nanotube; M-IL-MWCNTs, magnetic ionic liquid modified carbon nanotubes; M-MWCNT, magnetic multi-walled carbon nanotube; M-NP, magnetic nanoparticle; M-SP, magnetic sorbent particle; MSPDE, matrix solid-phase dispersion extraction; M-SPE, magnetic solid-phase extraction; MWCNT, multi-walled carbon nanotube; NAaP, nucleic acid associated proteins; PAE, phthalic acid ester; PAH, polycyclic aromatic hydrocarbon; PDMS, polydimethylsiloxane; PDDA, poly(diallyldimethylammonium chloride); PEG, polyethylene glycol; PSS, poly(sodium 4-styrenesulfonate); SA, sulfonamide; SIA, sequential injection analysis; SPE, solid phase extraction; SPME, solid phase microextraction; TEM, transmission electron microscopy; UHPLC, ultra-pressure liquid chromatography; UHPLC–MS/MS, ultra-pressure liquid chromatography–tandem mass spectrometry.

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Synthesis
Applications

and novel automation strategies. In recent years, the use of magnetic carbon nanotubes (M-CNTs) as a sorption substrate in M-SPE has become an active area of research. These materials have exceptional mechanical, electrical, optical and magnetic properties and they also have an extremely large surface area and varied possibilities for functionalization. This review covers the synthesis of M-CNTs and the different approaches for the use of these compounds in M-SPE. The performance, general characteristics and applications of M-SPE based on magnetic carbon nanotubes for organic and inorganic analysis have been evaluated on the basis of more than 110 references. Finally, some important challenges with respect the use of magnetic carbon nanotubes in M-SPE are discussed.

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Contents

1. Introduction 11
2. Magnetic solid-phase extraction 12
2.1. Magnetic carbon nanotubes as sorbents for M-SPE 12
2.1.1. Two-step synthesis of M-CNT composites 13
2.1.2. One-step synthesis of M-CNT composites 15
2.2. Characterization of M-CNT composites 15
2.3. M-SPE automation 16
3. Analytical applications of M-SPE using M-CNTs as sorbents 17
3.1. Organic compounds 17
3.1.1. Biological molecules 17
3.1.2. Medicinal products 19
3.1.3. Environmental and food pollutants 21
3.2. Ions and inorganic compounds 22
4. Conclusions and future trends 23
References 23

1. Introduction

Sample preparation is an essential step of the analytical process. Inadequate sample pretreatment could lead to analyte loss, sample contamination and numerous other problems that compromise the final result of the research. In addition to the correct sampling technique, the enrichment of analytes and their separation from complex matrices of different samples (such as environmental or biological ones) is a fundamental challenge in this stage of the process. Separation and preconcentration of organic and inorganic species from liquid and solid samples have been performed using a wide variety of techniques, including precipitation, flocculation, filtration, ultrafiltration and extraction, amongst others. Solid phase extraction (SPE) is one of the most routinely used procedures for the separation and preconcentration of a variety of compounds and elements from complex samples due to their well-known advantages, which include the high enrichment factor, good recovery, use of small quantities of organic solvents and the possibility of automation (off- or on-line) of the whole process. In addition, the use of SPE has increased in recent decades due to the development of a new variety of materials that can be employed as solid sorbents [1]. However, this technique suffers from some drawbacks depending on the type of SPE applied, the sorbent used and the characteristics of the sample. The most commonly used techniques employ on-column immobilized SPE and these may result in long treatment times, high back-pressure in the packing process and low extraction efficiencies in certain cases when compared to other SPE methodologies [2]. Therefore, in the past few years other SPE approaches, such as solid phase microextraction (SPME) [3,4], dispersive solid phase extraction (DSPE) [5], magnetic solid phase extraction (M-SPE) [6], molecularly imprinted solid phase extraction (MISPE) [7,8] and matrix solid-phase dispersion extraction (MSPDE) [9], have been applied in an effort to overcome these problems. Among these techniques, M-SPE has proven to be an interesting procedure in which the analyte is separated from the large sample solution volumes by using magnetic sorbents and an external magnetic field. The principal benefits of M-SPE are the prevention of problems related to column packing and the easy and rapid phase separation of the sorbent (which is achieved directly by using magnets and avoids the need for filtration and/or centrifugation steps).

It is clear that one of the main advantages that has led to the widespread use of the different SPE techniques in recent decades is the wide variety of materials that can be employed as solid sorbents. These materials include cellulose, bonded or modified silica, activated carbon, polyurethane, alumina, exchange resins, magnesium silicate and zeolites, amongst others. In M-SPE numerous magnetic sorbents have also been synthesized in the past few years by coating magnetic cores in different inorganic (silica, alumina, zirconium, etc.) or organic (cellulose, chitosan, alginate, polyacrylamide) substrates [6,10,11]. Carbon nanotubes (CNTs) were first discovered by Iijima [12] and they are materials that have exceptional physical and chemical properties. These properties make CNTs ideal for SPE and they have several advantages in comparison with other commonly used sorbents such as silica-derivatives. The extremely high surface/volume ratio of CNTs enhances the separation and preconcentration performance of traditional phases for organic [13–16] and inorganic [17,18] analysis. In fact, several comparative studies on the use of MWCNTs, C_{18} and activated carbon as SPE sorbents for the analysis of diverse types of pesticides revealed that nanotubes have an adsorption capacity that is almost three times higher than those of the other two sorbents assayed and that it also has better RSD and recovery values [19,20]. More recently, other authors confirmed the high adsorption capacity of CNTs in relation to C_{18} silica and also highlighted the favorable adsorption-desorption kinetics of this

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