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Review

Strategies for interfacing solid-phase microextraction with liquid chromatography

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Abstract

Solid-phase microextraction (SPME) techniques are equally applicable to both volatile and non-volatile analytes, but the progress in applications to gas-phase separations has outpaced that of liquid-phase separations. The interfacing of SPME to gas chromatographic equipment has been straight-forward, requiring little modification of existing equipment. The requirement of solvent desorption for non-volatile or thermally labile analytes has, however, proven challenging for interfacing SPME with liquid-phase separations. Numerous options to achieve this have been described in the literature over the past decade, with applications in several different areas of analysis. To date, no single strategy or interface device design has proven optimal. During method development analysts must select the most appropriate interfacing technique among the options available. Out of these options three general strategies have emerged: (1) use of a manual injection interface tee; (2) in-tube SPME; and (3) off-line desorption followed by conventional liquid injection. In addition, there has been interest in coupling SPME directly to electrospray ionisation and matrix-assisted laser desorption ionisation (MALDI) for mass spectrometry. Several examples of each of these strategies are reviewed here, and an overview of their use and application is presented.
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Keywords: Solid-phase microextraction; SPME–HPLC interfaces; In-tube SPME; Liquid chromatography; Sample preparation; Sorbent phases; Liquid desorption; Off-line desorption

Contents

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1. Introduction

The coupling of solid-phase microextraction (SPME) to liquid chromatographic (LC) techniques has been of interest in the years since SPME was first introduced, for analysis of compounds not amenable to study by gas chromatography (GC). SPME coupled with high-performance liquid chromatography (HPLC) is attractive for analysis of polar and/or thermally labile compounds, as well as those with poor volatility. A number of drugs, pesticides, toxins, and also significantly the metabolites of these compounds, are logical targets for such analyses. In addition, LC instruments are essential for analysis of high molecular weight compounds. With advances in SPME technology to allow extraction of large biomolecules, hyphenation to LC technology for these compounds will also become important.

Despite the interest, practical implementation of SPME–LC has lagged behind that of SPME–GC. There are several reasons why such methods have not been widely developed to date. These include the small selection of available commercial fibres, long equilibration times, more challenging desorption optimisation, a lack of automation of the methods, and correspondingly the significantly more tedious nature of HPLC desorptions of fibres relative to GC desorptions. When compared to the relative ease of solid-phase extraction (SPE) and column-switching techniques for LC sample preparation, it is not surprising that SPME methods have not been significantly implemented to date. Much of the recent literature in the area has focused on addressing these challenges.

An additional limitation for the broader application of SPME–LC methods is the lack of commercially available interfacing options. Solid-phase microextraction devices are conveniently interfaced with any GC injector. This is because the SPME thermal desorption process for GC is accomplished using the same steps as for standard GC injections. That is, piercing of the GC injector septum with the device needle followed by depressing the device plunger. Analytes are desorbed from the fibre in an analogous way to analytes thermally vapourizing in a conventional injection. Numerous devices are available commercially to allow interfacing to virtually any GC injector and automated processing is well developed. Interfacing to LC, on the other hand, requires liquid desorption from the fibre, which is a slower process due to the slower diffusion kinetics in the condensed phase relative to the gas phase. No analogous process occurs with conventional liquid injection to LC. The introduction of fibre SPME devices to liquid chromatography instruments, therefore, requires significant modification of the injector and the design of conventional injectors does not lend itself to such modification. These difficulties were partially overcome with the introduction of in-tube SPME, but this technology requires that sample preparation be performed on-line, which is not always desirable.

Aside from the interfacing issue, another challenge identified has been the small number of commercially available SPME sorbents for LC applications. Although no new commercial products have been introduced in recent years, several experimental solid sorbent fibres have been reported for specific applications [1–3]. Additionally, the construction of immunoaffinity fibres has been described, which should allow the application of SPME technology for the extraction of large biomolecules from complex matrixes [4,5].

Authors have noted that the primary advantages of SPME–LC are its speed and simplified sample preparation [6–8,25], easy elimination of salts from biological samples prior to ESI analysis [22], potential for application to field testing [9], and application to analyses where GC or other more conventional techniques are not possible [3,19]. SPME–LC has been particularly useful for studies of protein interactions as the technique is less disruptive to the protein binding thermodynamics than are the alternative techniques of ultrafiltration or equilibrium dialysis [10,11]. As is demonstrated in this review, substantial progress has been made in addressing the above-mentioned challenges. As these are overcome, the benefits of SPME–LC will make the technology more attractive and opportunities will exist to further develop SPME–LC technologies. This review summarizes options for addressing the challenges, strategies employed over the past decade for interfacing SPME with LC, and will attempt to summarize the strengths and appropriate application of several of these.

2. Direct SPME fibre interface

The technology of manually coupling SPME fibre sampling to HPLC analysis was first introduced by Chen and Pawliszyn in 1995 [12], using a re-designed injector for a fibre interface. This was designed using standard HPLC hardware incorporating a desorption chamber located in the position normally occupied by the injection loop of a standard six-port injection valve. The desorption chamber consisted of a 0.75 mm id stainless steel (s.s.) tee with two of the three ports connected to the injection loop ports of the valve. The third port was used to introduce the fibre for desorption. A section of 0.02″ PEEK tubing was used to seal around the s.s. rod holding the fibre during desorption and high pressure transfer of analytes to the column. A fingertight fitting was used to create the seal. When the injector was in the ‘load’ position, the desorption chamber was at ambient pressure and the fibre could be inserted and sealed. When the injector was moved to the inject position, mobile phase was pumped along the length of the fibre at high pressure and then onto the head of the analytical column. Either static or dynamic desorption could be performed depending on the amount of time between exposure of the fibre to the desorption solution, and the switching of the valve to ‘inject’.

The interface was validated by comparing its performance to standard syringe injection for the analysis of a mixture of polyaromatic hydrocarbons (PAH). An established extraction procedure using 7 μm PDMS fibres was employed [13]. An isocratic mobile phase comprising 90% acetonitrile (ACN) in water produced both an appropriate chromatographic separation and very fast desorption of analytes from the fibre coating, based on the fact that there was no difference in peak shape or retention time compared to standard loop injection. Resolution of the four compounds in the mixture was complete in the 14 min run and no carryover of analytes was observed in subsequent blank injections. Gradient separation was also successfully applied.

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