

Recent advances in on-line solid-phase pre-concentration for inductively-coupled plasma techniques for determination of mineral elements

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We provide a critical survey on recent progress in on-line solid-phase pre-concentration for inductively-coupled plasma (ICP)-based techniques for trace-level determination of metals. We highlight some of the useful solid-phase materials suitable for on-line separation and enrichment prior to ICP determination. We discuss different applications of the methods developed along with their analytical figures of merit. Finally, we sketch out future prospects in this field.

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1. Introduction

Modern laboratories are often required to determine trace and ultra-trace levels of elements in different types of samples. Amongst different analytical techniques used for this purpose, inductively-coupled plasma (ICP)-based techniques [e.g., coupled to mass spectrometry (ICP-MS) and optical emission spectrometry (ICP-OES)] are most commonly employed.

ICP has emerged as the most powerful tool due to its high sensitivity, low limits of detection (LODs), extended linear dynamic range (up to nine orders of magnitude), multi-element determination in a short time from a single sample preparation and obtaining isotope ratios (in the case of ICP-MS) with relatively low spectral and mass interferences.

However, the application of plasma-based techniques for the direct analysis of trace and ultra-trace elements in complex samples is quite limited, or sometimes almost impossible, due to matrix spectral or isobaric interferences [1] and they also

suffer from problems with signal suppression and clogging of the sample-introduction systems when the sample contains dissolved solids at concentrations greater than 0.2% m/v [2]. Also, there is the possibility of the matrix elements combining with the elements in the plasma to form polyatomic species resulting in interferences to the determination of the first-row transition metals by ICP-MS.

Many attempts have been made to solve these problems {viz dilution of the sample [3], flow-injection sample introduction [4], standard additions and the addition of internal standards [5]}. Collision cell or high-resolution mass spectrometry (HRMS) in ICP-MS may provide other options to improve the selectivity. However, the options strongly depend on the available instrumentation: the collision-cell option needs collision or reaction gas flow to be optimized; and, HRMS involves expensive investment. For these reasons, purely instrumental approaches have a limited success in overcoming the problems of signal suppression or enhancement, and

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a less expensive, more general, successful approach seems to be the separation of the analytes from the interfering matrix and their pre-concentration. Again, out of many techniques available for separating the target analyte from the rest of the matrix [6–9], solid-phase extraction (SPE) has become the most popular and greenest alternative, as it has several advantages, e.g., it:

- (1) is simple to operate;
- (2) provides high pre-concentration factors;
- (3) involves rapid phase separation;
- (4) does not cause emulsion formation;
- (5) is a safe approach for hazardous samples;
- (6) is less polluting to the environment, as it reduces consumption and exposure to the toxic organic solvents;
- (7) is a reasonably fast approach, as it can reduce the extraction time providing a multiple extraction without any time gaps, compared with liquid-liquid extraction (LLE);
- (8) is a relatively inexpensive approach, as the sorbent can be regenerated and reused for many cycles; and, finally,
- (9) can be easily automated and combined with different detection techniques *via* on-line or off-line modes.

As sample preparation is the key to success of an analysis, an ideal sample-preparation methodology should be rapid, accurate, precise, and, finally, should consume the least amounts of organic solvents. This leads to the development of new selective, sensitive methods for isolating target components from complex matrices. Other important criteria for modern extraction methods include sample integrity (e.g., the possibility to do speciation analysis without interconversion of species, high throughput and easy adaptation for field work, and also being compatible with the subsequent methods of analysis). Most of these requirements may be met to a certain extent by automation (*viz* coupling on-line the sample preparation procedure to the separation and detection system, in most cases an ICP system).

On-line SPE methods have several advantages over their off-line counterparts [10]. They increase the sample throughput by reducing the sample-preparation time. Moreover, conditioning, washing and elution steps can be performed automatically. Some systems also permit extraction of one sample while another is being analyzed [11], thus overcoming the limitation of multi-step, time-consuming and cumbersome procedures involved in off-line SPE. Other important advantages of on-line coupling are the reduced risk of contamination of the samples or sample extracts, the reduced possibility of loss of analyte by evaporation or degradation during sample pre-concentration, and the improved precision and accuracy attainable. High sensitivity, low LODs and the use of small sample volumes required may also be achieved in on-line configurations

due to transfer and analysis of the entire extracted species to the analytical system, whereas an off-line SPE procedure analyzes only an aliquot of the extract. In addition, on-line SPE systems consume less solvent and are thus much less expensive and green in terms of solvent disposal [12]. In on-line SPE systems, loss of analytes due to evaporation is minimized but may occur in an off-line SPE system.

In summary, on-line procedures are particularly attractive in situations where a large number of samples have to be analyzed routinely with high sensitivity, or when hazardous or highly infectious materials must to be processed, while off-line procedures are favored for their applicability to inject the same extract several times. However, in-field SPE followed by on-line elution in the laboratory offers a unique methodology for environmental studies.

This article reviews the possibilities of on-line SPE procedures coupled to ICP techniques for the determination of trace and ultra-trace elements in environmental samples. In the following sections, we offer insight into on-line configurations and sorbents applied.

2. Strategies used for on-line pre-concentration

2.1. On-line configurations

In on-line SPE, the sample is generally injected into a cartridge, which retains the target analytes. The residual constituents of the sample matrix are then flushed directly or after a washing step to the waste. Analytes, which are retained on the bonded phase of the cartridge, are then eluted on-line, using a switching valve, into the series-connected analytical system, as can be seen in Figs. 1 and 2. During this separation, an exchange of the cartridge or reconditioning of the pre-column can take place. The most commonly used “column switching” technique involves the use of a small pre-column within the injection loop of a six-port rotary valve (Fig. 3). After conditioning, sample application and eventual clean-up are made using a high-pressure pump, with the pre-column being placed in front of the analytical system by switching the valve into the “inject” position. A sample is then loaded, whereupon the valve is switched in order to elute the analytes out of the sorbent by the mobile phase and transfer them into the analytical detector [13]. This approach has the limitation of reusability of the cartridges, due to the continuous degradation of the bonded phase material, which results in changes in selectivity and sorption capacity. Again, the repeated use of the cartridge may cause sample cross-contamination if the previous sample or matrix components are not completely removed during washing and elution steps, particularly when highly polluted samples are being analyzed.

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