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Scientific contributions in the FI/SI-AAS field – Automation of Sample Preparation

Despite the wide technological progress in the field of instrumentation for chemical analysis, several analytical instruments cannot handle direct analysis of samples with complex matrices or very low analyte concentration. This is the reason why a sample pretreatment procedure is usually involved in the analytical method. Manual approaches are considered to be time-consuming, tedious and laborious, typically demanding large amounts of toxic organic solvents and producing hazardous waste.
Flow injection/sequential analysis (FI/SI) and related techniques proved to be excellent tools in the field of automation, acceleration, and miniaturization of solution handling, offering several specific features.
During the last years, the research interests of our group are mainly focused on the development of flow methods based on automation and miniaturization of sample preparation techniques like on-line solid phase extraction (SPE), dispersive liquid-liquid microextraction (DLLME), single drop microextraction (SDME), cold vapor/hydride generation (CV/HG) coupled with atomic spectrometry.
In 2001, the concept of polytetrafluoroethylene (PTFE) turnings packed in a mini-column as hydrophobic sorbent material was developed [1]. The effectiveness and usefulness of PTFE-turnings was demonstrated for on-line preconcentration and determination of several toxic metals (Ag, As, Cd, Cr, Co, Cu, Pb, Ni, Zn and Hg) with atomic spectrometric techniques (FAAS, ETAAS and ICP-AES) [2-4]. In the frame of development of novel sorbent materials for FI/SI/LOV-SPE systems, polyether-ether ketone (PEEK) turnings, polyurethane foam (PUF) [5], polychlorotrifluoroethylene (PCTFE) [6], Oasis®-HLB, Bond Elut® Plexa^{TM} PCX polymer resin, HyperSep-SCX strong cation exchanger resin, multi-walled carbon nanotubes (MWCNTs) as well as SiMAG-Octadecyl magnetic material were studied and adopted for metal determination [7, 8].
Direct multi-element analysis of oil or solid samples by inductively coupled plasma atomic emission spectrometry (ICP-AES) was achieved introducing an on-line micro-chamber with a magnetic-stirrer for emulsion or slurry formation [9, 10].
An integrated column preconcentration/gas-liquid separator (PCGLS) system incorporated in cold vapor or hydride generation manifold was developed for on-line preconcentration and determination of mercury using atomic absorption spectrometry (CVAAS).
A fully automatic on-line DLLME platform using extraction solvents lighter or heavier than water was developed for metal determination coupled with FAAS or ETAAS [11, 12]. Thanks to this strategy, the dispersant solution was merged on-line with the sample resulting in the formation of a cloudy solution (fine droplets of extraction solvent dispersed entirely into the aqueous phase). The separation of the extractant based on its hydrophobicity and was completed by microcolumn packed with PTFE or PEEK-turnings or PUF.
In the field of LLME, our group developed some novel SI platforms such as "single-drop" "drop-in-drop" and "counter-current" microextraction [13-15]. All these systems exploit the advantages of SIA and its ability for handling very small volumes (down to a few tenths of micro-litres) of solutions with great success, thanks to high precision stepper motor micro syringe pumps driven by advanced software.
An interesting automated approach for downscaling fluidic manipulations is the so-called lab-in-syringe (LIS) platform, incorporating in-syringe accommodation of wet chemical or heterogeneous reactions at will. The proof of concept of a miniaturized LIS system incorporating a membraneless gas−liquid separator (GLS), mounted on the top of a multiposition valve, was presented for automated determination of trace mercury by CVAAS [16].
Lately, a novel fully automated headspace single-drop microextraction (HS-SDME) system based on a programmable LIS platform hyphenated to ETAAS for in situ vapor generation assays was developed [17]. The LIS setup facilitates effective mixing of sample and reducing agent solutions in a batch-flow format followed by release and sequestration of the volatile Hg^0 vapor into a single-drop (Pd^0 dispersed into aqueous solution). Reduced pressure conditions can be

easily applied into the syringe barrel without the need for an external vacuum pump.

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