

The application of multicommutated flow techniques to the determination of iron

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New flow techniques such as multicommutated flow-injection analysis (MCFIA), multi-syringe FIA (MSFIA) and multi-pumping flow systems (MPFSs) have been developed. The low reagent consumption achieved by these techniques should be highlighted, as they can be regarded as environmentally friendly alternatives to conventional FIA. We present several applications of these novel flow techniques to the determination of iron.
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1. Introduction

The emergence of flow techniques in the early 1950s played a major role in routine analysis, since they provided precise, accurate and rapid measurements with minimal sample handling. Furthermore, sample volume and reagent consumption were decreased with regard to batch methods.

In past years, new flow techniques, such as multicommutated flow-injection analysis (MCFIA) [1,2], multi-syringe flow-injection analysis (MSFIA) [3,4] and multi-pumping flow-injection systems (MPFSs) [5,6] have arisen as possible alternatives to classical flow techniques (i.e. segmented flow analysis (SFA) [7], flow-injection analysis (FIA) [8] and sequential injection analysis (SIA) [9]). However, FIA is still the most widely used technique. Moreover, the number of applications reported on FIA during its first years is much higher than those on SIA, MCFIA, MSFIA and MPFS. This fact can be attributed to the easy implementation of FIA, since this technique does not require a computer to control the system [10].

Minimization of reagent consumption (green chemistry) and the monitoring of

environmental parameters are among the current trends in analytical chemistry. In this sense, multicommutated techniques (MCFIA, MSFIA and MPFS) are especially suitable for these purposes, since reagents are propelled to the system only when necessary and they allow development of fully automated systems with a high injection throughput. Multicommutated techniques have been successfully applied to the determination of metals [11–14], pesticides [15,16], pharmaceutical preparations [17,18] and nutrients [19–22] in different sorts of samples (i.e. food [12], soils [13], environmental samples [11,19–22]).

Nutrients are a key parameter in the evaluation of water quality. Iron is an essential micronutrient for organisms, as it is involved in many biological processes. However, the speciation analysis of iron is also of great interest in the study of the aqueous environmental chemistry of trace elements [23,24].

There are several references in the literature to the determination of iron exploiting multicommutated techniques [13,25–28]. Nevertheless, these works have not been applied to the determination of low levels of iron. Taking into consideration the low concentrations of iron in some media, different strategies, such as solid-phase extraction (SPE), have been proposed in order to achieve lower limits of detection (LODs). We present in this article some multicommutated systems that we developed for the determination, speciation analysis and SPE of iron [29–32]. Moreover, we discuss in detail the properties of these systems and their advantages over the conventional flow techniques.

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2. Multicommutated systems for the determination of iron

Fig. 1 shows different multicommutated flow systems that we developed to carry out the spectrophotometric determination, SPE and speciation analysis of iron [29–32]. In these assemblies, ammonium thiocyanate was used as chromogenic reagent for Fe(III). The determination of total iron was achieved by the on-line oxidation of iron(II) to iron(III) with a hydrogen-peroxide stream. A diode-array PC-plug-in spectrometer with a PC 2000 A/D card (Ocean Optics, Dunedin, FL, USA) was used as detector.

2.1. Multicommutated flow-injection system

The replacement of six-port rotary valves used in FIA assemblies by three-way solenoid commutation valves in MCFIA introduces flexibility and saves reagents, since they are injected into the system only when necessary. Moreover, the injected sample volume can be selected by controlling the commutation timing via software. [1,2].

An MCFIA set-up is shown in Fig. 1(a) [29]. This system allows the determination and the speciation analysis of iron in a wide range of concentrations. It comprises a peristaltic pump (Ismatec, Type Reglo Digital, Switzerland) and six three-way solenoid commutation valves (N-Research, Caldwell, N.J., USA).

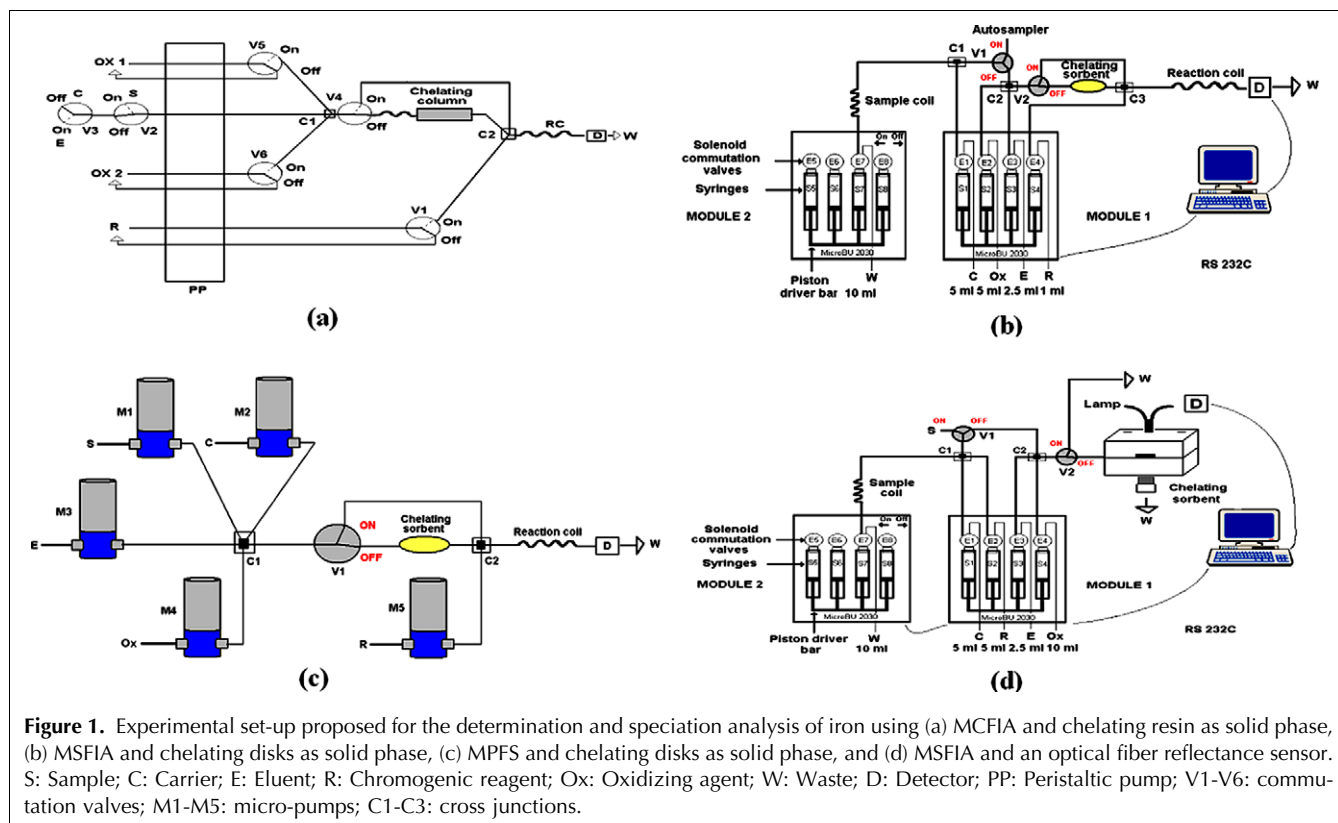
The determination of the lowest iron concentrations benefits from the retention of the analyte onto a chelating resin (iminodiacetic groups), which has been placed in a poly(methyl methacrylate) micro-column (Fig. 2(a)). For this purpose, commutation valve V4 should be in Off position.

Analytical signals obtained exploiting MCFIA and SPE are shown in Fig. 3(a). By contrast, valve V4 should be in the On position when the determinations of high levels of Fe(III) and/or Fe(II) or cleaning procedures are required.

Time-consuming steps, such as the replacement of the injection coil or the periodical unloading of the liquid-driver, are not required in MCFIA. For this reason, this technique is especially suitable for SPE. However, the use of a peristaltic pump represents the main disadvantage of this system, since the flexible tubing of the peristaltic pump needs to be replaced periodically. MSFIA avoids this shortcoming.

2.2. Multi-syringe flow-injection analysis

MSFIA combines the multi-channel operation and high injection throughput of FIA with the robustness and the versatility of SIA. Thus, the basic element of MSFIA is a multi-syringe burette (Crison Instruments, Alella, Barcelona, Spain) that allows the simultaneous movement of four syringes (analogous to those used in SIA assemblies), which are connected *en bloc* to the same stepper motor. Furthermore, three-way isolation valves



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