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Some recent developments on cost-effective flow-based analysis☆

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Abstract

This paper reviews some recent developments on cost-effective flow-based analysis. They include the newly developed Lab-at-Valve (LAV), concepts in using the stopped-flow injection approach, on-line sample pretreatment systems, including bead injection–flow injection and flow injection–ion-chromatography, systems for size-based speciation, and cost-effective reagents. Applications and advantages of such techniques are discussed.

Keywords: Lab-at-Valve (LAV); Stopped-flow injection; Sample pretreatment; Bead injection; Sequential injection; Flow injection; Size-based speciation; Cost-effective reagent

1. Introduction

This paper describes some recent developments of techniques aiming for cost-effective analysis related to flow injection analysis (FIA). It is based on a presentation in the 12th International Conference on Flow Injection Analysis (12th ICFIA) including related techniques, which was dedicated to Professor Gary and Mrs. Sue Christian and also for the celebration of the 20th Anniversary of the Japanese Association for Flow Injection Analysis (JAFIA). Some of the developments were initiated and/or resulted from discussion during the previous ICFIAs. Some are from Talanta, which provides good geographical distribution in contributed papers.

Cost-effectiveness is always a consideration in performing an analysis, not only in remote places where only limited budget is available, but also in general practice [1,2]. To select a scheme for an analysis, apart from analytical characteristics of accuracy, precision, sensitivity and selectivity, and real-time analysis, the cost of analysis should also be taken into account (Fig. 1).

When considering cost of an analysis, this should include analysis time, chemical(s)/reagent(s) consumption, and instrument. The last one includes cost of the system itself, operating cost, and cost dealing with maintenance.

Flow-based analytical techniques, which offer various advantages, with the main features being high sample throughputs and much less samples/reagents consumption, and simpler instrumentation, should be suitable to serve the above considerations [2–28].

Flow injection analysis introduced in the early stage by Ruzicka and Hansen, already demonstrated well the cost-effectiveness along with high efficiency in various routine analyses in Brazil [3,4].

Sequential injection analysis (SIA) provides automation, being possible with relatively simple instrumentation, compared to other techniques [4,13,14,29–38].

Systems with cheap components in a remote place may be very valuable in research training, in investigating and demonstrating novel concepts for further development [39,40]. Examples are an overhead projector FIA set-up for demonstrating concepts [41], using an aquarium pump in various simple flow injection (FI) systems [42]: offering practical advantages in sample handling [43] for those with FI–AAS (e.g., the determination of Pb [44,45]), and for in-valve mini-column for determination of ppb-levels of ura-

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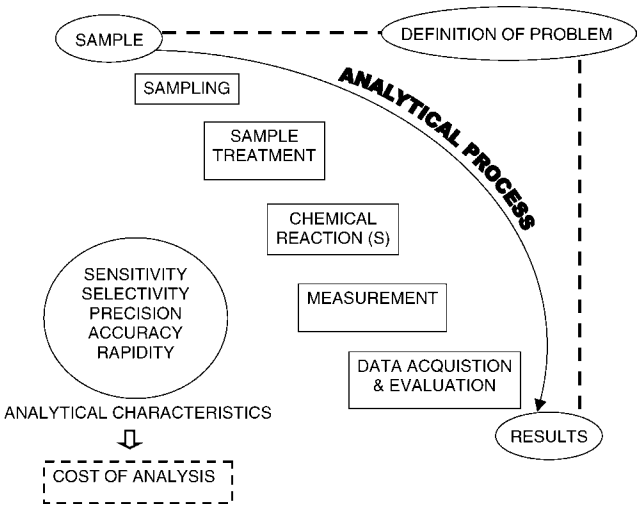

Fig. 1. Analytical process.

nium [46]. Also, such simple systems make single standard calibration possible [44–46]. By applying even simple systems, procedures become much easier, compared to the batch ones, for example, the dissolved oxygen determination by Winkler's method [47,48] and FI with on-line column system for hemoglobin typing leading to thalassemia screening [49].

Simple flow systems with some detection units offer various degrees for automation, depending on the various kinds of components of instruments involved [4,40,50–56], for example, the determination of acid concentration or acidity can be performed colorimetrically using visual detection [41], a simple peak-hold colorimeter [57], or using a very simple conductivity detector [58,59] or a computer-controlled detector [60–63]. Simple FI manifolds with radioactivity detection systems [64,65] offer basic automation using flow-based techniques. Higher degrees in automation and other advantages by employing sequential injection (SI) systems have proven to be useful to real applications for radioactivity monitoring [66–68]. Relatively simple FI and SI set-ups with dynamic surface tension detectors have proven to be very useful and fast for the economical study of some interfacial properties [69,70]. A very simple manifold with a light scattering detector using a laser pointer has been applied for nephelometric determination of sulfate [71]. Various simple FI systems for iodide determination have been reported [72–76].

2. Novel concepts for cost-effective flow-based systems

Some novel concepts have been investigated.

2.1. Sequential injection with "Lab-at-Valve" (LAV) concept

In the past decade, the field of miniaturization in chemical analysis has gained increased interest [77]. The miniaturization toward Lab-on-Chip (LOC) or micro total analysis system (μTAS) concepts includes assembly as an in integrated device, consisting of sample collection, sample pretreatment, and separation and/or chemical reaction taking place for the analytical detection, and the sensor.

Ruzicka [78] introduced a SI with "Lab-on-Valve" (LOV) system to perform analyses in micro- or nanoliter volumes by integrating sample processing, chemical reaction, and monitoring, in a conduit at a multiposition valve. A LOV system for such the above mentioned operation is a precisely fabricated monolithic structure mounted atop a conventional multiposition selection valve, by using computer-aided design (CAD). There have been various applications of LOV with various advantages [4,14,38,53,79–84], for example environmental monitoring [38,82,83], pharmaceutical applications [22], and bio- and clinical analysis [78,84].

A "Lab-at-Valve" concept has been introduced as an alternative cost-effective μTAS device. Instead of replacing a stator plate of a multiposition selection valve by a perfectly machined piece, as that of the "LOV", sample processing and detection unit(s) are attached or plugged onto port(s) of a commercial conventional multiposition selection valve without taking apart any component out of such a purchased valve.

This simpler approach has been demonstrated by SI–LAV potentiometric determination of chloride. A simple LAV flow-through electrode system, consisting of two simple laboratory-made Ag/AgCl electrodes, is plugged onto a port of a purchased multiposition selection valve (Fig. 2). Based on concentration cell behavior, chloride in a sample can be assayed [85].

SI–LAV solvent extraction has been proposed. Such a simple system was demonstrated for spectrophotometric determination of diphenhydramine hydrochloride and anionic surfactant (Fig. 3). Sample, reagents, and organic solvent are sequentially aspirated into a holding coil. By flow reversal, good extraction efficiency can be achieved. After that, the aqueous and organic phases are separated in a conical extraction chamber attached at one port of a conventional multiposition selection valve ("Lab-at-Valve" concept). The organic solution is then pushed into a flow cell of the spectrophotometer for detection of the extracted colored product.

2.2. New criteria for using a stopped-flow injection system

Stopped-flow injection analysis was proposed by Ruzicka and Hansen, for two different purposes for operating a stopped-FIA system: (1) to increase the sensitivity of measurement by increasing the residence time and thus the yield of measured species or (2) to measure a reaction rate serving a base for the analytical read-out. By stopping a flow so that the analyte and reagent(s) stay for a period at a flow-cell [86], progress of a reaction involved is continuously monitored, during the period of the stopped-flow. Various advantages can be obtained by the involvement of kinetic considerations

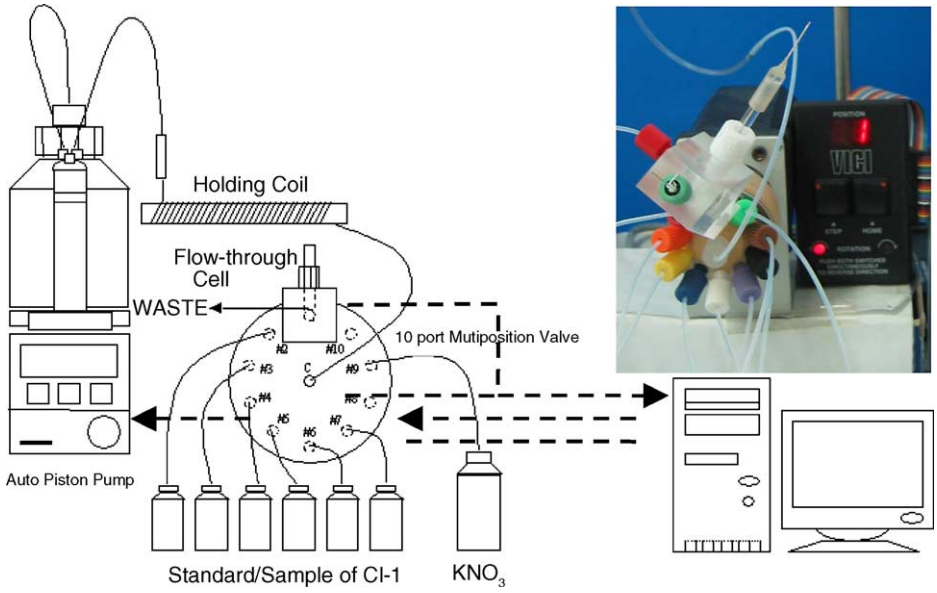

Fig. 2. SI–Lab-at-Valve (LAV) system for potentiometric determination of chloride (the inserted photo shows a close-up of the potentiometric cell (attached to the multiposition valve)).

such as kinetic information, increase in sensitivity, and kinetic separation [87].

A simple semi-automatic stopped-FI analyzer was developed (Fig. 4) [88]. One application example proposed by our group was for the simultaneous determination of phosphate and silicate by employing their differences in kinetic behaviors.

Optimization of such a stopped-FI system involves various parameters, both hardware and non-hardware, similar to the optimization in usual conventional FI systems. Hardware parameters normally are: tubing i.d., sample (loop) volume, mixing coil length and i.d., and flow through cell volume; while non-hardware ones involve: flow-rate, traveling time, stopping time, washing time, and concentration of reagents.

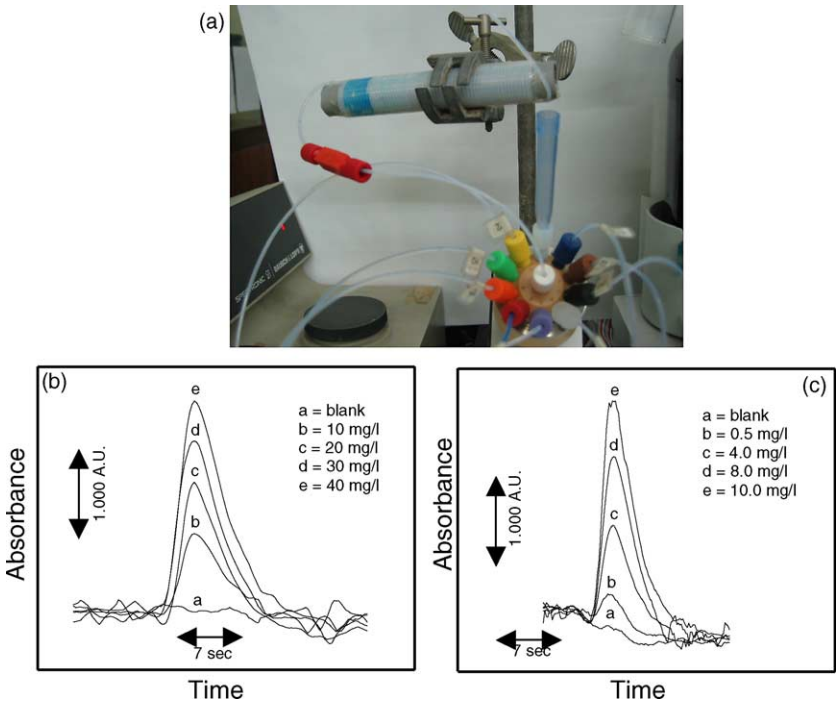

Fig. 3. SI–LAV solvent extraction spectrophotometric determination: (a) the LAV extraction system, (b) signal profiles obtained for the determination of diphenhydramine hydrochloride using bromocresol green [100], (c) SI grams for anionic surfactant determination (the methylene blue method using sodium dodecylsulfate as the standard).

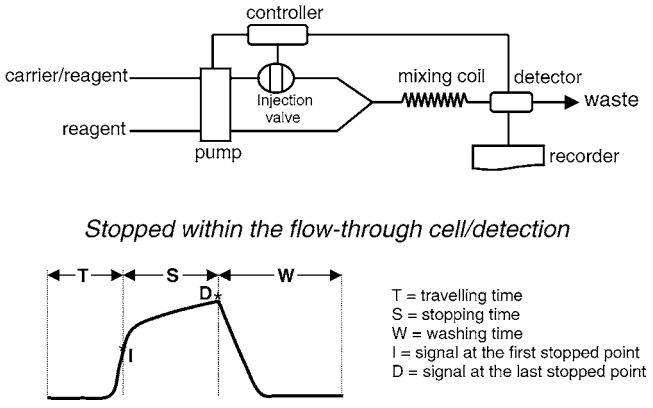

Fig. 4. Conventional stopped-FIA [88]; *T*, traveling time: a period from the injection till the flow stops; *S*, stopping time: a period of flow-stopping; *W*, washing time: a period after the flow is re-started until the next injection.

The optimization is concerned with slope in association with sensitivity (see Fig. 4).

For a conventional FI system, optimization is usually aimed for maximum sensitivity and high sample throughputs. Conventional optimization would involve the hardware parameters: tubing i.d., sample (loop) volume, mixing coil length and i.d., flow through cell volume; and the non-hardware ones: flow-rate and reagent concentration.

Now a concept for stopped-flow injection system has been investigated. By using a semi-automated stopped-flow injection analyzer being able to control pump (on–off) and switching an injection valve, after an analyte solution is injected into a carrier stream of reagent, the flow is stopped at a mixing coil for a period before allowing to flow further to the flow-through cell in a simple colorimeter as a detector. Fig. 5 illustrates the proposed concept of FIA using stopped-flow outside a detection cell. Optimization can be made by varying some parameters such as traveling time, stopping period, flow-rate, and reagent concentrations, without changing any hardware component such as injection loop (injection volume), and mixing coil length and i.d.

Ruzicka and Hansen suggested [86] to stop the flow outside the detector in order to gain reaction time and thereby generation of product to be measured. By our investigation, this would help promotion the mixing, but minimize dispersion of the product. Such a concept was demonstrated for chloride determination (Fig. 6) using mercury (II) thiocyanate and Fe(III) reagents, which were used in the previous conventional FI procedure [89]. Without changing the hardware parameters, but varying only non-hardware parameters, determination of nitrite (using well-known coupling of diazotized sulfanilamide with *N*-(1-naphtyl)ethylenediamine dihydrochloride) and determination of phosphate (using phosphomolybdovanadic acid method) can be performed. This

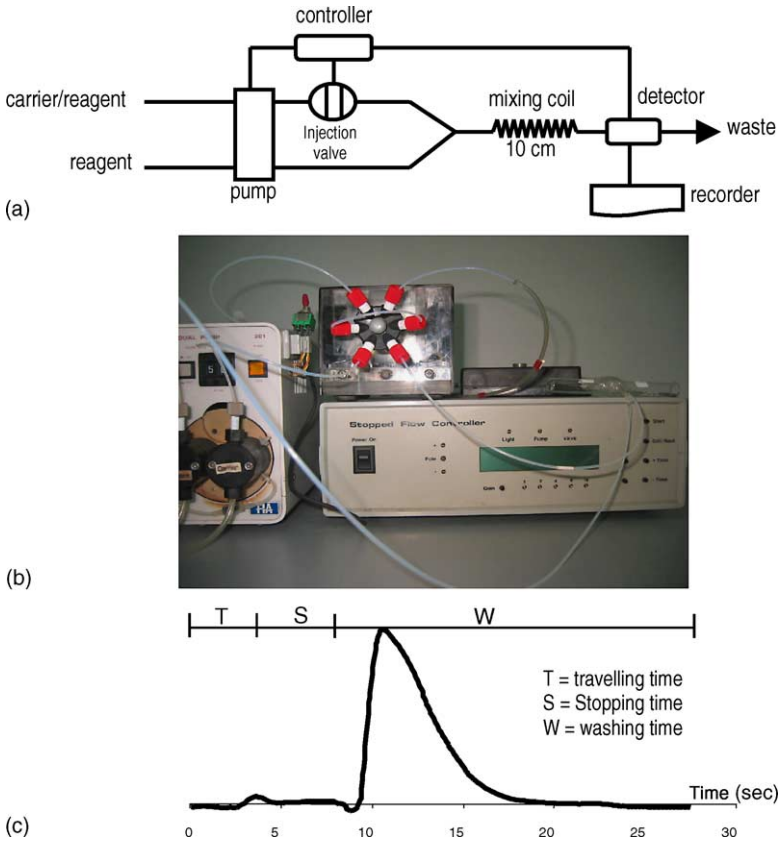

Fig. 5. A new concept stopped-FI: the flow manifold; (a) schematic diagram, (b) the picture, and (c) FI gram profile obtained, when stopping the flow at the mixing coil outside the flow through cell.

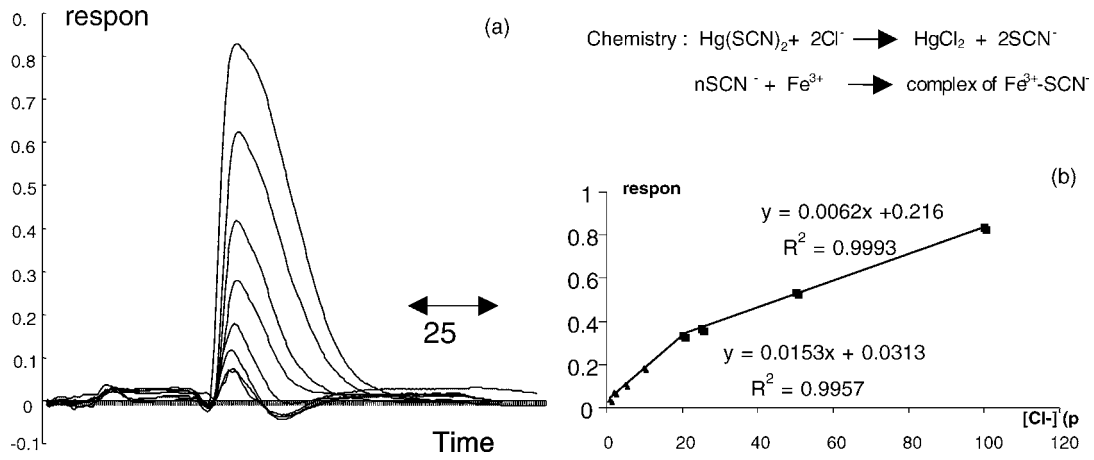

Fig. 6. The determination of chloride using the new proposed stopped-FI concept, by stopping the flow at the mixing coil (T = 1.9 s, S = 1 s, W = 15 s; a throughput of 120 injection/h): (a) FI gram profiles and (b) calibration graph (note: two breaks of the linear calibration ranges may be due to formation of different complexes for higher concentrations of chlorides).

stopped-FI approach offers various advantages, even for a determination involving fast reaction. The advantages include possibility of using only on FI analyzer with fixed components (hardware parameters) for various analytes without changing any hardware component, better sensitivity than conventional FIA, and less amounts of reagents consumption. All lead to economical aspects. This stopped-FI can be used for both slow and fast reactions.

3. On-line sample pretreatment

Sample pretreatment including preconcentration and preseparation, is an important step in analytical process (Fig. 1).

Flow-based techniques offer on-line sample pretreatment to gain advantages in less sample and reagent consumption, and possibility of automation with less sophisticate instrumentation.

Bead injection (BI) was introduced as a technique that utilizes beads as a solid surface to trap species of interest, to accommodate a chemical reaction and to transport them through the flow line or to a detection unit [33]. The renewal beads overcome the difficulty in finding suitable eluent to elute sorbed metal ions from resins, as the used beads will be discarded after each run [90].

3.1. BI with SI

This has proven to be very useful for sample preconcentration and/or preseparation for the determination of heavy metal ions in complicated matrix samples. The BI–SI can be useful to automate sample pretreatment for various types of detectors, including ET–AAS [33,79–84,91].

3.2. BI with FI

This offers an alternative for cost-effective operation for trace levels determinations, such as iron in water samples [92] and in beer samples [90]. Simple instrumentation (without a computer control) and single standard calibration are advantageous.

3.3. FI–ion-chromatograph (IC)

FI system with in-valve mini-column for sample pretreatment coupled to a simple ion-chromatograph with conductivity detector without suppressor for simultaneous determination of some cations (cadmium, lead, and zinc) in zinc ore samples, which have high amounts of matrix interference, has been proposed. The on-line sample matrix interference removals offer advantages including elimination of costly and time-consuming off-line operations, reducing IC column damage (i.e., prolong the column life-time) in addition to the simple instrumentation employed, and the normal advantages usually offered by FIA [93].

3.4. Size-based speciation

Speciation of a substance depending on its size, which has been termed as “size-based speciation”, has gained much of interest in various studies, especially in environmental aspects. Studies on combining field-flow fractionation (FFF) with analytical detection systems have been made. Some cost-effective flow-based systems have been investigated such as gravitational FFF (GrFFF)–FI chemiluminescence for Fe size-based speciation, and GrFFF–ET–AAS for size-based speciation applied to soil sample [94]. Flow FFF (FIFFF)–ICP–MS has been used for size-based speciation of some heavy ions in colloid formation of metal ions released from contaminated soil [95].

4. Cost-effective reagents

Additional advantage that flow-based systems are operated in a closed system (e.g., FIA, SIA), is that an unstable

reagent, especially one being air- or light-sensitive, can be employed.

Also, the chemicals/reagents which are not necessary to be in very high purity grade can be utilized in a flow-based technique since an analyte to be determined is to be run in the exactly the same conditions with that of the standard.

Murexide is a very common chemical in a laboratory but it is unstable in basic solution. However, it can be used in a spectrophotometric determination for Ca by a simple FI set-up [96].

$KMnO_4$, a very cheap and easily available chemical, can be used as an oxidant for an analyte with reducing species properties. Its color intensity change in the redox reaction involved can be used by employing FI [58] or SI [60] systems, whereas a batch procedure is not possible due to that it is sensitive to air and light. Pharmaceutical applications such as ascorbic acid assay in Vitamin C tablets have been reported.

Recently, aspirin, an antipyretic powder, has been proposed as a salicylate reagent, which is cheap and easily available, for the determination of Fe(III). A red Fe(III)–salicylate complex product can be monitored. An application to assay iron contents in pharmaceutical preparations has been made [97].

Solid reactants have been employed as different types of reaction columns for sulfate and sulfur dioxide [98]. Phloroglucinol has been proposed as alternative reagent, which would provide better cost-effective procedures for nitrite and nitrate determinations [99].

5. Conclusion

Sequential injection systems with Lab-at-Valve is an alternative cost-effective micro-total analysis system approach. A stopped-FI analyzer with a new concept in optimization offers the possibility for different analytes to be determined using only one fixed set-up of hardware parameters (i.e., only one instrument), while only non-hardware parameters are to be optimized. On-line sample pretreatment with flow-based techniques, such as FI–IC, BI–FI, and FFF–AAS, offers various advantages. Utilization of cost-effective reagents can be possible by using flow-based techniques, especially FI and SI.

Acknowledgements and proclaims

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