Lab-on-valve in the miniaturization of analytical systems and sample processing for metal analysis

Yong-Liang Yu, Ying Jiang, Ming-Li Chen, Jian-Hua Wang

In the past decade, the lab-on-valve (LOV) system, as the third-generation of the flow-injection analysis technique, has exhibited powerful capability in instrument miniaturization and on-line sample pretreatment.

This review presents and discusses the state of the art in the progress of the LOV system in the determination of metal species in two parts:

- **miniaturization of analytical instrumentations; and,**
- **sample-processing front-ends.**

As a miniaturized analytical set-up, LOV incorporates detection techniques for the determination of metal species (e.g., spectrophotometry, electrochemical detection and atomic spectrometry). However, coupling LOV sample pretreatment with atomic or mass spectrometric detectors provides high-sensitivity determination or speciation of metal species.

We also discuss future perspectives of the LOV system in metal determination and/or speciation.

Keywords: Bead injection; Flow-injection analysis; Lab-on-valve; Metal analysis; Miniaturization; Sample processing; Speciation

Yong-Liang Yu
Department of Chemistry, Northeastern University, Box 332, Shenyang 110819, China

Ying Jiang, Ming-Li Chen, Jian-Hua Wang*
Research Center for Analytical Sciences, Northeastern University, Box 332, Shenyang 110819, China

*Corresponding author.
Tel.: +86 24 83688944;
Fax: +86 24 83676698;
E-mail: jianhuajrz@mail.neu.edu.cn

1. Introduction

The emphasis of chemical assay has shifted from batch approaches to automated, miniaturized flow procedures. In the context of this development, flow-injection (FI) analysis introduced in 1975 has led to significant progress in the field of chemical assay and has made an impact in terms of efficiency, reliability and robustness [1]. According to the different degrees of automation, the FI technique has experienced three historical periods of the development [2], including flow injection (FI), sequential injection (SI) and lab-on-valve (LOV). It is especially noteworthy that the third generation of the FI technique [3,4], LOV, has made significant progress in miniaturization, automation and integration of on-line sample pretreatment with a microconduit monolith mounted atop a conventional multi-position valve (hence the name ''lab-on-valve'') [5]. Designed to accommodate a wide variety of chemical manipulations and even detection, LOV is made to include working channels, connecting ports and a multi-purpose flow cell with all necessary laboratory facilities [6], as shown in Fig. 1.

In the LOV system, the experimental flow manifold can be designed well by optimizing the volume of the flow path between the injector and the detector, and that reduces the reagent consumption to microliter and sub-microliter levels. Also, programmable flow patterns are exploited in the LOV system (e.g., flow forwards, flow reversals and stopped flow, which facilitate solution metering, mixing, dilution, incubation and monitoring in any desired sequence).

Based on the above two advantages [7], LOV can make operations in analytical applications more versatile. Also, relatively wide, short channels allow solid or microcarrier beads to be transported and manipulated to form a solid-phase extraction (SPE) column in the valve as desired, so as to perform the entire SPE procedure in a renewable fashion [8].

By contrast to the lab-on-a-chip (LOC) system of fixed architecture [9], the LOV system has been viewed as a judicious advance towards microfluidic handling

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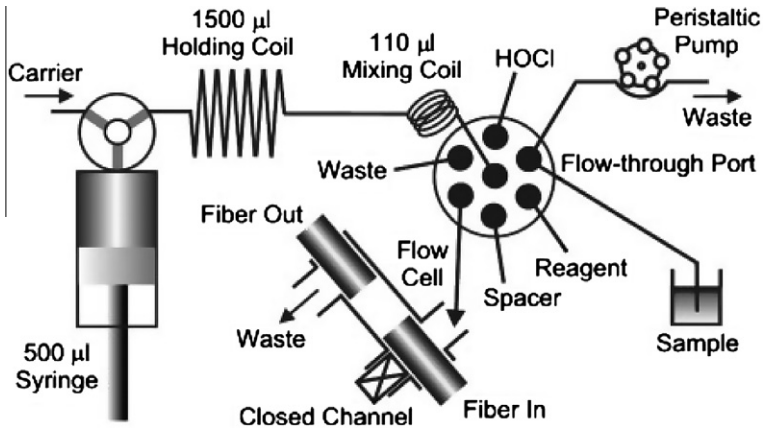

Figure 1. Sequential injection lab-on-valve (SI-LOV) system incorporating a multipurpose flow cell (adapted from [6], with permission of The Royal Society of Chemistry).

and on-line sample processing, which allows us to:

(1) exploit the interplay between thermodynamics and kinetics;
(2) conduct multi-parametric determination without manifold reconfiguration by the use of the inherent open-architecture of module unit; and,
(3) to be used as a versatile front end for a variety of detection techniques.

The LOV system therefore provides an alternative solution to the dilemma of the LOC system and becomes a useful platform for the analytical task in mesofluidic handing [10]. There has been detailed discussion and comparison between the LOV approaches and the LOC devices in the literature [11].

This review presents a brief outline of the development of LOV systems and their applications for assay of metal species in the past 10 years, with special emphasis on the technique of LOV bead-injection (BI), the applications of LOV as a miniaturized analytical system and sample-processing front-end in combination with various detection techniques for the quantification of metal species.

2. Bead injection

SPE is one of the most popular sample-pretreatment approaches for removing interfering matrix components and at the same time preconcentrating the analyte through interactions between the target analyte and the active surface of the solid-phase material prior to the quantification step. It has been widely implemented by using a permanent packed-column in a flow system [12].

However, it has run into two major problems by use of solid-phase columns in long-term operation. It is prone to produce high back pressure, due to the progressively tighter packing or clogging of the column material in a flow system. An even worse situation is that the malfunction of active entities results in deactivation of the surface of the solid-phase material due to irreversible sorption of interfering species by the reusable column.

In order to overcome the drawbacks of the permanent packed-column mentioned above in the SPE procedure, the BI technique was introduced into the flow system by renewing the solid-phase material after each analytical cycle, when required [13].

The BI technique exploited by a LOV platform should be viewed as a judicious advance towards automation and miniaturization, which has been implemented by in-valve manipulation of sorbent material to form an SPE microcolumn in the microconduit unit of the LOV [14]. The packed microcolumn is generated *in situ* by aspirating a suspension solution of microcarrier beads from a peripheral port of the valve, and thereafter it is automatically transported between different column positions [1–4]. In order to trap the beads within the channel cavities and to prevent the beads from escaping during the operations, the outlets of one or two flow channels are furnished with small pieces of PEEK tubing [15] or micropore polyethylene frit [16], which allows the solution to flow through freely and entraps the beads in the channel cavity of the LOV. Generally, the use of homogenous and spherical beads is facilitated to ensure the reproducibility of handling beads within a flow system. As for beads of non-uniform size and far from spherical shape and high density in comparison with aqueous solution, the use of some ancillary approaches is essential to allow reproducible manipulation (e.g., adding various non-ionic surfactants and stabilization reagents to guarantee bead suspension, and exploiting a bead-recirculation procedure to facilitate BI [17]). Fig. 2 shows a LOV system for BI operation.

The solid surfaces of beads as sorbent materials in a LOV unit are utilized to preconcentrate analyte or to