



Review

Recent trends in solid phase spectrometry: 2003–2009. A Review

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ABSTRACT

Around 100 papers published from 2003 to the present are reviewed concerning analytical methods for the direct light measurement of a solid phase in which a target colored or fluorescent analyte is concentrated. Recent attention has been paid to the development of flow injection-solid phase spectrometry as a simple and inexpensive tool for routine analysis of organic compounds or pharmaceuticals. Due to some improvements in flow injection analysis, such as sequential injection and lab-on-a-valve, it is possible not only to reduce the reagent consumption but also to devise fully automatic and miniaturized systems with minimal maintenance needs. This may have the potential of becoming one of the green analytical methods. Flow injection-solid phase spectrometry is expected to be applied to the speciation of trace chemical components (e.g., specific determination of trace metal ions in different existent oxidation states) in real samples in the environment.

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1. Introduction

Spectrophotometry has been most commonly used for quantitative analysis of chemical components in solution, due to the continuous development of instrumentation. The simple and easy operation procedures are also important factors. However, the biggest drawback is its sensitivity. To enhance the sensitivity of spectrophotometry and make the most of its advantages, solid phase spectrometry (SPS) has been developed and applied to the determination of trace analytes in various water samples. This method is based on the direct measurement of the light attenuation of adsorbent particles (or the light intensity of the emitted light from adsorbent particles) packed in an optical cell, in which the target analyte in samples are concentrated as a colored (or fluorescent) chemical component [1,2]. Because the concentration of the target analyte and the direct light measurement of a solid phase are simultaneously carried out without elution, SPS is more sensitive than the conventional corresponding solution method: over a few hundred times greater sensitivity can easily be achieved without using any expensive apparatus. Although the color development procedures for SPS are usually similar to those for conventional solution spectrophotometry, the selectivity can also be greatly improved when the interaction between a target chemical species and a solid phase is quite different from that between co-existing ions and the adsorbent. Ionic strength, pH, the content of organic solvent, and selection of the adsorbent are the important keys to higher selectivity.

SPS has employed two different procedures: batch and flow methods. Because of a fairly large background attenuation of the solid phase, which is due to a surface reflection and/or a diffuse reflection of the solid layer, the sensitivity enhancement by making the light path of the solid particle layer longer is difficult (in many cases the light measurements are carried out within 1 to 2 mm in light path length). It is also slightly time-consuming and requires above average skill to pack the solid particles into a cell when employing the batch method.

An online measurement of light attenuation or light emission by the adsorbed species in the flow-through cell makes it possible both to reduce the sample solution volume considerably and to simplify the respective procedures for the derivatization of the analyte and filling the solid particles into the cell. Some research groups have dealt with developing this batch SPS method into new flow analysis methods under their own separate concepts [3–6]. In this review, all of these methods are called “Flow injection-solid phase spectrometry” (FI-SPS).

Due to the remarkable progress in the recent flow analysis system or the spectrophotometric analysis including the apparatus, the applicability of the analytical method related to SPS has been expanded to various chemical species in a great variety of samples. Since the first report on SPS had appeared in 1976, many reports on SPS or a flow method related to SPS have been published. The development of those methods over these three decades has already been summarized in some papers. The latest review paper on SPS was given by Capitan-Vallvey and Fernández-Ramos, entitled “Solid phase spectrometric assays” [7]. For the flow method based on SPS, the studies published up to 2004 were reviewed in detail by Miró and Frenzel as “Flow-through sorptive preconcentration with direct optosensing at solid surfaces for trace-ion analysis” [6]. In this article, practical applications of analytical methods related to SPS or those for the direct optosensing of a solid phase in which the target analyte is retained are selected and reviewed among those reported from 2003 to the present.

Bead injection (BI) spectrometry [8], in which the disposal of the adsorbent beads and the injection of new sorbent beads into the flow-through cell are carried out after each analytical cycle, is a very powerful method for widening the applicability of FI-SPS.

The BI concept, first proposed by Ruzicka et al., was introduced to use together with sequential injection analysis (SIA) [9]. At an early stage in this technique, a jet ring cell [10] was used for the optical sensing of solid particles collected inside it. A commercially available flow-through cell could possibly be applied to the BI technique [11,12]. In order to downscale the volume of sample and reagent solutions, this BI concept was then also applied to a lab-on-valve device [13]. The recent achievements in this method have been thoroughly introduced in 2007 and 2008 as “Recent developments in automatic solid-phase extraction with renewable surfaces exploiting flow-based approaches” [14], “How flow-injection analysis (FIA) over the past 25 years has changed our way of performing chemical analyses” [15] and “Lab-on-valve: a useful tool in biochemical analysis” [13]. For this reason, a detailed introduction to this method was not provided, but some papers are reviewed in this article.

2. Instrumental aspects of light measurements

2.1. Absorptiometry

In measuring the light attenuation by a solid phase, the biggest problem is that a relatively small net absorbance, caused by the colored species adsorbed on the solid phase, has to be measured under a fairly large background attenuation of the solid phase. This large background attenuation, which is characteristic of the light measurements of a solid phase, is due to a surface reflection or a diffuse reflection from the solid layer. For example, if the attenuation of a 1-cm thick cross-linked polystyrene-type ion exchanger packed into a quartz cell is measured against air as the reference, the light attenuation is about 3 in absorbance units even at 700 nm, at which there is no light absorption by the ion-exchange resin. This means that the very low absorption of light passing through the resin phase, which is above or near the limit of detection of a common spectrophotometer for quantitative light measurements, has to be measured.

The analytical performance of the methods considered in this article is listed in Table 1. In the case of batch SPS method, the light path length of a solid particle layer in the cell is 1 to 2 mm; there is only one report applying a 5-mm light path for absorptiometry. Inevitably, 50 to 1000 cm³ sample solutions had to be used for trace analysis to compensate for the shorter light path of solid particle layers. Although novelty in the light measurement techniques could not be found in these papers, some interesting results on the applicability of multicomponent analysis to improve the selectivity and analytical performance [16], a prevalidation method to evaluate the validity or reliability of the SPS method [17], a new chromogenic reagent [18] and specific determination of different oxidation states [19] are described. Quite recently, a 0.06 cm³ portion of a cation exchanger was used to concentrate the target Cr(VI) in a 20 cm³ water sample, and resin beads were introduced in a flow cell of 1.5-mm diameter and having a 10-mm light path length. Three lenses were used for focusing the incident light beam and for recovering light scattered by the solid phase in the cell. The sensitivity was higher by a factor of 277 compared with that of the solution method, and the detection limit was 0.014 μg dm⁻³ [20].

In the case of FI-SPS using a micro flow-through cell, light attenuation by the cell also accompanies the background light attenuation of the solid phase. The use of respective optical fibers for incident light radiation and for collection of the light transmitted through the solid phase has attracted increasing interest for this purpose. If the cross-sectional area of the luminous flux of the fibers for incident light beam radiation is smaller than that of the flow-through cell, the light attenuation by the cell can be negligible. When the optical fiber is set as close to the solid particle layer as

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