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Miniaturized liquid chromatography in environmental analysis. A review

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ABSTRACT

The greater and more widespread use of chemicals, either from industry or daily use, is leading to an increase in the discharge of these substances into the environment. Some of these are known to be hazardous to humans and the environment and are regulated, but there is a large and increasing number of substances which pose a potential risk even at low concentration and are not controlled. In this context, new techniques and methodologies are being developed to deal with this concern. Miniaturized liquid chromatography (LC) emerges as a greener and more sensitive alternative to conventional LC. Furthermore, advances in instrument miniaturization have made possible the development of portable LC instrumentation which may become a promising tool for in-situ monitoring. This work reviews the environmental applications of miniaturized LC over the last 15 years and discusses the different instrumentation, including off- and on-line pretreatment techniques, chromatographic conditions, and contributions to the environmental knowledge.

1. Introduction

In recent decades, numerous efforts from both academic and industry have been made for the development of miniaturized LC, which is considered an important topic in the field of LC. The first reported use of a column with an internal diameter (i.d.) of 1 mm was by Horváth et al. for the separation of ribonucleotides in the late 1960s [1]. Ten years later, Tsuda and Novotny established the required equipment for working with capillary LC, implementing modifications on conventional injectors and detector systems aiming to reduce the band-broadening effects [2]. Table 1 shows the main reviews concerning this topic during the last 15 years. As can be seen, up to 2019, there are revisions about its applications in food [3] and drug analysis [4], the fundamentals of the instrumentation [5–8], and its coupling with mass spec-

contamination of water bodies in particular [19]. In addition, these compounds may be present in other environmental matrices such as soil, air, or biota [20]. The high persistence and mobility of some of these substances causes to migrate away from the site of application, reaching surface and ground water [21] and consequently, bioaccumulating in the food chain [22]. The EU Water Framework Directive [23] establishes the control of a list of persistent chemicals in the environment, which can be bioaccumulated and have the risk of causing adverse effects to human health and the environment. These regulated compounds are worldwide controlled and monitored by analytical methods. Many of the substances classified as priority pollutants are pesticides (organochlorides, organophosphates, triazines, sulfonylureas, and other). There are several regulation around the pesticide application and concentration limits in the environment and drinking waters [24–27]. The legal

also lowering the costs. In combination with miniaturized LC, on-line sample treatment is often performed. This simplifies the sample preparation step and improves the environmental analysis performance by carrying out the injection, extraction, preconcentration, separation, and detection in a single run. In this sense, column switching configurations based on trap columns and IT-SPME were employed with good results, meeting the required LODs set by legislation. In contrast to trap columns which are more tied to the C18 extractive phase, IT-SPME has explored a more diverse number of phases (including TRB-5, TRB-35, TEOS-MTEOS-NPs, and magnetic sorbents, among others) allowing it to be more selective in retaining the contaminants of interest.

Regarding the instrumentation, valve-based injectors and dual-piston reciprocating pumps are mainly used in benchtop miniaturized liquid chromatographs. A wider variety of injectors and pumps are used in portable systems to minimize weight while maintaining compactness. When moving from conventional to miniaturized analytical columns, the method applicability might be restricted due to the lower diversity of commercially available miniaturized columns and their higher price. Thus, some studies have proposed home-made monolithic columns instead of the commonly used packed C18 columns. Although the use of these kind of columns is not frequent, it is an interesting trend to explore as it expands the analysis possibilities while reducing the costs. With respect to detectors, DAD and MS are chosen for most applications. While DAD is preferred for its accessibility, MS detectors are opted for their higher sensitivity, selectivity, and compound identification. Also, miniaturized spectrophotometric UV/vis detectors are light enough to be used in portable systems, whereas MS is still rather limited to laboratory applications.

Finally, the occurrence of contaminants in environmental samples was discussed. Although the data are limited, it can be observed that most of the regulated pollutants were not found and the quantified ones were within the environmental quality standard concentration. Compared to the regulated ones, the percentage of found analytes for emerging contaminants was higher for all matrices in concentrations varying from $\mu g \cdot L^{-1}$ for some pharmaceuticals to $ng \cdot L^{-1}$ for estrogens and PFAS.

As a last remark, the miniaturization of LC instruments has introduced a new trend in environmental analysis — the development of capabilities, sustainability, greenness and portability.

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CRediT authorship contribution statement

S. Cortés-Bautista: Writing – original draft, Methodology, Investigation, Formal analysis. **C. Molins-Legua:** Writing – review & editing, Supervision, Methodology, Investigation, Conceptualization. **P. Campíns-Falcó:** Writing – review & editing, Supervision, Resources, Project administration, Investigation, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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