



Review

Automation of static and dynamic non-dispersive liquid phase microextraction. Part 1: Approaches based on extractant drop-, plug-, film- and microflow-formation



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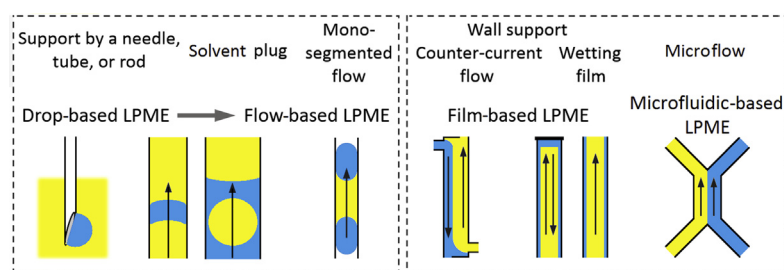
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HIGHLIGHTS

- Automation of static & dynamic non-dispersive LPME is comprehensively overviewed.
- Drop-, plug-, film- & microfluidic-based LPME are presented in part 1, and membrane-based LPME in part 2.
- Different extractant supports, operation modes, and analytical performances are discussed.
- A complete overview of figure of merits is provided as table.
- Automation methodologies as well as operations are described as schematic figures.

GRAPHICAL ABSTRACT



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ABSTRACT

Simplicity, effectiveness, swiftness, and environmental friendliness – these are the typical requirements for the state of the art development of green analytical techniques. Liquid phase microextraction (LPME) stands for a family of elegant sample pretreatment and analyte preconcentration techniques preserving these principles in numerous applications. By using only fractions of solvent and sample compared to classical liquid–liquid extraction, the extraction kinetics, the preconcentration factor, and the cost efficiency can be increased. Moreover, significant improvements can be made by automation, which is still a hot topic in analytical chemistry. This review surveys comprehensively and in two parts the developments of automation of non-dispersive LPME methodologies performed in static and dynamic modes. Their advantages and limitations and the reported analytical performances are discussed and put into perspective with the corresponding manual procedures. The automation strategies, techniques, and their operation advantages as well as their potentials are further described and discussed.

In this first part, an introduction to LPME and their static and dynamic operation modes as well as their automation methodologies is given. The LPME techniques are classified according to the different approaches of protection of the extraction solvent using either a tip-like (needle/tube/rod) support (drop-based approaches), a wall support (film-based approaches), or microfluidic devices. In the second part, the LPME techniques based on porous supports for the extraction solvent such as membranes and porous

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media are overviewed. An outlook on future demands and perspectives in this promising area of analytical chemistry is finally given.

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1. Introduction to liquid phase microextraction

1.1. The need for automation in liquid phase microextraction

Liquid-liquid extraction (LLE) is still one of the most-used sample pretreatment techniques in analytical laboratories although nowadays it has likely been surpassed by solid phase extraction (SPE). Outstanding advantages of LLE are its simplicity, high extraction efficiency, and applicability not only to liquid but also to solid and gaseous samples [1–3]. On the other hand, handling significant amounts of solvents, cleaning of glassware, required space in the laboratory, and further steps of the extraction procedure such as time-consuming analyte concentration by solvent evaporation are problematic in terms of work safety, time efficiency, costs, and are opposed to the objective of environmental friendliness.

During the last decades, LLE has undergone a remarkable progress by the development of different innovative miniaturised methodologies grouped by the denotation “liquid-phase microextraction” (LPME), which overcome the previously listed limitations [4–16]. By using only a drop-size volume of the extraction phase (EP, generally < 100 μ L), which is brought into contact with a reduced amount of sample, the extraction equilibrium can be reached within a shorter time achieving a high preconcentration factor [17,18]. On the other hand, it is obvious that the effect of the sample matrix including ionic strength, suspended particles, viscosity, and the extraction temperature are more pronounced, and the formation of a reproducible contact surface between both phases is less straightforward than in classical LLE. The loss of the EP by partial dissolution in the sample must be also taken into account. The control of the operation conditions by automation is therefore an important tool to take full benefits of these techniques [19]. Different LPME methodologies performed in an automated and thus easy-to-operate fashion are surveyed and discussed in this review.

Early attempts were done using a single drop of solvent submerged into the constantly moving or agitated sample, thus, direct immersion single drop microextraction (DI-SDME, see section 2) is

the most frequently used expression for this technique [20–24]. Even earlier, the use of a single drop of reagent for the extraction of NH_3 , SO_2 , and Cl_2 from the gas phase had been reported [25,26], which can be seen as the origin of head-space single drop microextraction (HS-SDME) [27].

The reported performance in these first publications appears to have inspired analytical chemists all over the world to use and improve these approaches and to bring up ingenious variants. We want to point out that for extraction and preconcentration of an analyte from one liquid phase to another at microscale level not only the denotation liquid phase microextraction (LPME) is commonly used but also other expressions such as liquid-based microextraction [28] or, more frequently, solvent microextraction [17]. In total, these keywords yield nowadays over 1400 references in the Web of Knowledge [29].

In the following, different types of drop support were proposed, mainly to protect the EP from the sample movement or matrix and to increase the contact surface (see section 1.3.). The spectrum reaches from a complete separation of the EP drop by exposing it in the head-space (HS) above the sample (HS-SDME) [27] over supports for the formation of EP films and immersion in the sample [24,30] towards the use of porous membranes for EP inclusion being in a steady contact with the sample [31,32]. Porous medium-based techniques are discussed in the second part of this review. Fig. 1 represents a history timeline of the development of non-dispersive LPME methods using different extraction and automation modes. Detailed descriptions of this progress are further discussed in the particular sections devoted to the different LPME techniques.

Another modification of LPME receiving tremendous attention is based on the increase of the contact surface by EP disruption and droplet formation using solvent dispersion by either an auxiliary solvent [33] or different modes of kinetic energy [34–36]. As in this paper only automated non-dispersive LPME techniques are reviewed due to the extension of the topic, the reader is referred to specialised reviews on dispersive liquid-liquid microextraction [37,38] while surveys of manual LPME are given elsewhere [5,14].

Despite the great improvements in the field of LPME

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