

# The roles of ionic liquids in sorptive microextraction techniques

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The roles that ionic liquids (ILs) can play as extractants, intermediate solvents, mediators and desorption solvents in liquid-phase and solid-phase microextraction (SPME) provide the topic of this review. We emphasize the added value of using ILs in single-drop microextraction, hollow-fiber-based liquid-phase microextraction, dispersive liquid-liquid microextraction, and in-fiber and in-tube SPME, the most relevant being greater simplicity, greater selectivity, greater extractability and lower consumption of organic solvents. We also give systematic consideration to the requirements of detection as the next step. Further, we outline the unique properties of ILs that are exploited in each extraction procedure. The growing importance of microextraction techniques in sample preparation justifies this approach.

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*Abbreviations:* See Appendix after References section.

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## 1. Introduction

Ionic liquids (ILs) can be defined as organic salts that are liquid at or near room temperature, taking 100°C as arbitrary reference [1]. This special behavior can be ascribed to the poor coordination of their constituent ions, since at least one of them has a delocalized charge preventing the formation of a stable crystal lattice [2]. The origin of ILs goes back to 1914 when Walden [3] described the first stable one, ethylammonium nitrate. However, the sensitivity to moisture of the first ILs limited their widespread use in different applications.

The description of ILs stable in air and moisture in the 1990s can be considered the initial point of the extensive research on these special solvents [4]. Since then, the number of publications (and citations) regarding ILs has increased exponentially, as indicated in Fig. 1A (source: ISI Web of Knowledge, November 2009). Moreover, the multidisciplinary impact of these substances affects not only chemistry fields

(organic, inorganic, physical, engineering and analytical) but also other important fields (e.g., nanotechnology or physics).

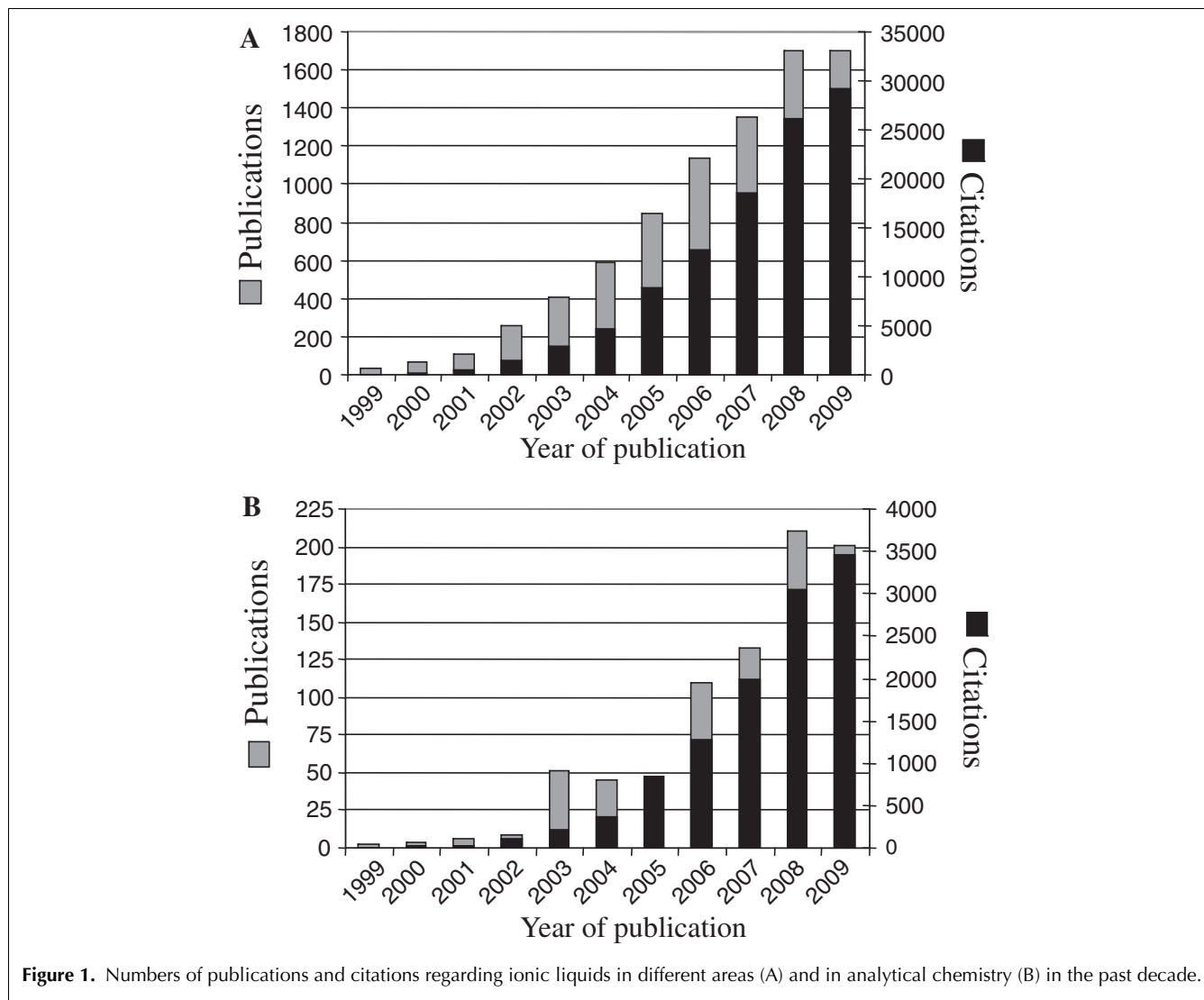
The peculiar characteristics of ILs (e.g., their wide electrochemical window, their negligible vapor pressure and their great thermal and chemical stability) have also been exploited in analytical sciences [5] in different application fields (e.g., chromatography, electrochemistry or extraction [6]). Publications devoted to the analytical uses of ILs have dramatically increased in the past decade (see Fig. 1B). In this context, the replacement of organic solvents by ILs in different extraction processes can be considered a “hot” research topic. Their non-flammable, non-volatile nature makes them an excellent choice for developing safer processes. Furthermore, their polarity, hydrophobicity, viscosity and other chemical and physical properties may be selected by choosing the cationic or the anionic constituent. Due to this tunable nature, ILs are regarded as “designer solvents”, increasing their potential uses.

The immiscibility of some ILs with water (which allows formation of biphasic systems) as well as the high solubility of organic species in them makes ILs suitable solvents for conventional liquid-liquid extraction. In this sense, Huddleston et al. [7] studied the partitioning of simple, substituted-benzene derivatives in ILs, comparing the results with the octanol-water partitioning coefficient. They demonstrated that those substances with charged groups or strong hydrogen-bonding moieties are extracted to a lesser extent than similar neutral or apolar species. In this context, the study of the IL-solvent properties [8] and their extractability towards different organic substances [9,10] is rather important for developing efficient extraction procedures.

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**Figure 1.** Numbers of publications and citations regarding ionic liquids in different areas (A) and in analytical chemistry (B) in the past decade.

The trend towards miniaturization has led to the development of a series of extraction modalities that minimize consumption of sample and solvent, and reduce analysis times, costs and risks. In this promising research area, ILs have proved to be excellent tools that improve analytical performance.

We devote the following sections to discuss in depth the applicability of ILs in sorptive microextraction techniques, including liquid-phase approaches [i.e. single-drop microextraction (SDME), hollow-fiber-based liquid-phase microextraction (HF-LPME) and dispersive liquid-liquid microextraction (DLLME)] as well as fiber and in-tube solid-phase microextraction (SPME) techniques. Table 1 presents an overview from the whole article regarding the different roles that ILs can play in microextraction. In Table 1, we highlight the sections in which we consider the different roles of ILs. Fig. 2 shows the structures of the different types of ILs that have been used in microextraction.

## 2. Liquid-phase microextraction (LPME)

LPME emerged in the mid-to-late 1990s [11–13] as a technique that minimizes the amount of solvent employed in the extraction, simplifies laboratory operations and improves sample utilization. Several methodologies that differ in extraction performance have evolved from LPME. In this review, we describe only those techniques in which ILs have been used, including SDME, HF-LPME, and DLLME.

### 2.1. Single-drop microextraction (SDME)

This LPME methodology is based on extracting analytes in a drop of solvent that is suspended in the needle tip of a microsyringe, which can be directly immersed in the aqueous sample (DI-SDME) or in its headspace (HS-SDME) [11–13]. In the conventional procedure, the syringe is filled with the extractant, clamped at an adequate position above or immersed in the sample. The

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