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A comparative evaluation of different ionic liquids for arsenic species separation and determination in wine varietals by liquid chromatography – hydride generation atomic fluorescence spectrometry

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ABSTRACT

The application of different ionic liquids (ILs) as modifiers for chromatographic separation and determination of arsenite [As(III)], arsenate [As(V)], dimethylarsonic acid (DMA) and monomethylarsonic acid (MMA) species in wine samples, by reversed-phase high performance liquid chromatography coupled to hydride generation atomic fluorescence spectrometry detection (RP-HPLC-HG-AFS) was studied in this work. Several factors influencing the chromatographic separation of the As species, such as pH of the mobile phase, buffer solution concentration, buffer type, IL concentration and length of alkyl groups in ILs were evaluated. The complete separation of As species was achieved using a C_{18} column in isocratic mode with a mobile phase composed of 0.5% (v/v) 1-octyl-3-methylimidazolium chloride ([C_8mim]Cl) and 5% (v/v) methanol at pH 8.5. A multivariate methodology was used to optimize the variables involved in AFS detection of As species after they were separated by HPLC. The ILs showed remarkable performance for the separation of As species, which was obtained within 18 min with a resolution higher than 0.83. The limits of detection for As(III), As(V), MMA and DMA were 0.81, 0.89, 0.62 and 1.00 μg As L^{-1}. The proposed method was applied for As speciation analysis in white and red wine samples originated from different grape varieties.

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1. Introduction

Arsenic (As) is a toxic metalloid that is present in the earth crust at levels as high as 3.4 mg kg^{-1} and it can be found in different concentration ranges in water, soil, air, food, plants and animals [1]. The toxicity of As strongly depends on its chemical association and speciation, with inorganic species being more toxic than organic ones [2]. In fact, total concentration of As does not provide full information about the real toxicological risks linked to this element. Consequently, the development of modern methodologies for As speciation analysis is mandatory, especially to assay the quality of foods typically consumed in human diet [3,4].

Arsenic can be present in widely consumed alcoholic beverages, such as wine, due to the use of contaminated herbicides or insecticides to vine plants. Other potential sources of As contamination for wines include, soil type, water quality used for watering, and the several procedures applied during winemaking, i.e. harvesting, crushing and pressing, fermentation, purification and storage conditions of the final product [5]. The International Office of Vine and Wine (OIV) regulates total As concentration in wines at a maximum residual level (MRL) of 200 μg L^{-1} [6]. This MRL raises some drawbacks for analytical chemists because highly sensitive analytical techniques are necessary for As determination. Moreover, this situation is more complex when speciation analysis has to be performed as concentration of As

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species is a fraction of total As. Different sensitive analytical methods have been reported for As speciation in wine using hyphenated techniques including, gas chromatography-atomic emission detector (GC-AED) [7] and high performance liquid chromatography-inductively coupled plasma-mass spectrometry (HPLC-ICP-MS) [8]. Likewise, non-chromatographic techniques such as ionic liquid-dispersion liquid liquid microextraction associated to electrothermal atomic absorption spectrometry (IL-DLLME-ETAAS) [9] or hydride generation-atomic fluorescence spectrometry (HG-AFS) [10] have been used. Normally, chromatography-based methods use expensive detectors such as ICP-MS, while non-chromatographic ones are time-consuming due to several experimental steps involved and have limitations to separate a high number of species. On the other hand, HPLC coupled to less expensive and highly sensitive detectors, such as AFS, is a valuable alternative for As speciation analysis, with a reasonable operating time that allows routine analytical laboratories to achieve an acceptable analytical frequency.

The main As species found in wines have been As(III), As(V), MMA and DMA [8,10]. Chromatographic hyphenated techniques such as HPLC-ICP-MS and HPLC-AFS have been useful for the determination of all these As species within a single chromatographic run [11]. However, the high concentration of ethanol occurring in complex beverage samples, such as wine, needs to be specially considered when AFS or ICP-MS detectors are used due to background signal increase and sensitivity loss effects caused by organic solvents [8,10]. Therefore, the most applied chromatographic technique for As speciation has been anionic exchange (AEC) because organic solvents are not necessary in the mobile phase. On the other hand, ion pairing reversed phase-HPLC (IP-RP-HPLC) has been successful for As species separation with minimal or none organic solvent added to the mobile phase [12]. Moreover, different ion pair reagents have been used as mobile phase additives in IP-RP-HPLC-HG-AFS to separate As species including, didocecyldimethylammonium bromide (DDAB), tetrabutylammonium hydroxide (TBA) or hexane-sulfonate [13].

Ionic liquids (IL) have unique properties such as negligible pressure vapour, good thermal stability and different solubility behaviours depending on the anions and cations that constitute them [14]. Since some ILs have the ability to form ion pairs with different species [15], this property has been exploited to develop ion pairing reverse phase liquid chromatography (IP-RP-HPLC) methodologies by employing them as additives in the mobile phase [16–18]. Thus, the role of ILs has been described as having a dual behaviour: (i) the anion or cation of ILs might interact with the stationary phase modifying its properties and (ii) they might interact with the analytes changing its distribution coefficient and retention on the stationary phase. However, the exact mechanisms by which ILs produce the separation of the analytes have not been fully explained and demonstrated. The advantages of using ILs in HPLC separations can be summarized as, the improvement of peak shape, better resolution, suppression of silanols and favourable changes in the retention behaviour of the analytes [16]. On the other hand, ILs have been scarcely explored as mobile phase modifiers in elemental speciation analysis by RP-HPLC-ICP-MS [18], but their application in RP-HPLC-AFS has not been studied.

In the present work, a complete study of the effects of several imidazolium and phosphonium type ILs on the separation and determination of different As species by RP-HPLC-HG-AFS was performed. The mechanisms involved during the separation of As(III), As(V), MMA and DMA were critically evaluated to understand how ILs improve the separation conditions. The effect of C_4-C_{16} methyl imidazolium ILs and tributyl(methyl)phosphonium methylsulphate used as mobile phase additives was studied. Likewise, hydride generation conditions were optimized by a multivariate methodology considering the possible effects of each IL.

Table 1
IL-HPLC-HG-AFS instrumental and chromatographic separation parameters.

HPLC	
Column	Kinetex C_{18} (4.6 mm i.d × 100 mm × 2.6 μm)
Guard column	HPLC Krudkatcher Ultra Column In-Line Filter 0.5 μm porosity × 0.01 in ID
Mobile phase	0.02 mol L^{-1} NH_4^+/NH_3 – 0.5% (v/v) $[C_8mim]^+Cl^-$ – 5% (v/v) methanol, pH 8.5
Mobile phase flow rate	0.5 mL min^{-1}
Injection volume	100 μL
Column temperature	25 °C
Chromatographic mode	Isocratic
HG-AFS	
Lamp and wavelength	As High intensity hollow cathode lamp, 197.3 nm
Main current	40 mA
Auxiliary current	0 mA
Photomultiplier detector voltage	−270 V
Reductant	1.4% (w/v) $NaBH_4$ in 0.05% (w/v) NaOH
Carrier	8.5% (v/v) HCl
Reductant and carrier flow rates	Both at 6.0 mL min^{-1}
Carrier gas and flow rate	900 mL min^{-1} Ar
Atomization temperature	300 °C

The application of the methodology was assayed for the speciation analysis of As in different wine varietals.

2. Experimental

2.1. Instrumentation

The separation of As species was performed with a chromatographic system consisting of a YL9101 vacuum degasser, a YL9110 quaternary solvent delivery pump (YL Instrument Co., Ltd., Seoul, South Korea), a Rheodyne valve with a 100 μL loop injector (Cotati, CA, USA) and a Kinetex C_{18} column from Phenomenex (Torrance, CA, USA). Detection of As species was performed with a Rayleigh AF-640A atomic fluorescence spectrometer (Beijing Rayleigh analytical Instrument Corp., Beijing, China). Instrumental conditions are shown in Table 1. An ultrasound bath (40 kHz and 600 W) with temperature control (Test Lab, Buenos Aires, Argentina) was employed to degas the mobile phases and solvents before chromatography. A Horiba F-51 pH metre (Kyoto, Japan) was used for pH measurements in mobile phases, standard solutions and samples.

2.2. Reagents and solutions

Stock standard solutions of inorganic As(V) and As(III) species [1000 mg As L^{-1} as sodium arsenate dibasic heptahydrate ($Na_2HAsO_4 \cdot 7H_2O$) (99.998%) (Sigma–Aldrich, Milwaukee, WI, USA) and sodium (meta)arsenite ($AsNaO_2$) (99%) (Fluka, Buchs, Switzerland)] were prepared at 0.1 mol L^{-1} HCl (Merck, Darmstadt, Germany). Disodium methylarsonate ($CH_3AsNa_2O_3 \cdot 6H_2O$) (MMA, 98%) (Fluka) and dimethylarsinic ($C_2H_7AsO_2$) (DMA, 98.6%) (Fluka) stock standard solutions (1000 mg As L^{-1}) were prepared with ultrapure water and stored at 4 °C in amber-coloured HDPE bottles. Working solutions were prepared by diluting these stock solutions.

Sodium borohydride (Fluka) (reducing agent) dissolved in 0.05% (w/v) NaOH (Aldrich) and hydrochloric acid (Merck) (carrier agent) were used for hydride generation. Tygon type tubes (Gilson, Villiers Le-Bell, France) were used to carry these reagents. All bottles used for storing samples, standard solutions and mobile phases along with glassware were washed in 10% (v/v) HNO_3 (Merck) for 24 h and later rinsed with ultrapure water (18 MΩ cm).

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