



A critical assessment of ultrasound-assisted extraction as sample pre-treatment for fast determination of multielements in seafood using inductively coupled plasma mass spectrometry



Adrián García-Figueroa^a, Jorge Millos^b, Carlos Bendicho^a, Isela Lavilla^{a,*}

^a Departamento de Química Analítica y Alimentaria, Área de Química Analítica, Facultad de Química, Universidad de Vigo, Campus As Lagoas-Marcosende s/n, 36310, Vigo, Spain

^b Centro de Apoyo Científico-Tecnológico a la Investigación, Universidad de Vigo, Campus As Lagoas-Marcosende s/n, 36310, Vigo, Spain

ARTICLE INFO

Article history:

Received 23 September 2016

Received in revised form 2 November 2016

Accepted 2 November 2016

Available online 05 November 2016

Keywords:

Ultrasound-assisted extraction

Inductively coupled plasma-mass spectrometry

Seafood

Cd

Cu

Mn

Ni

Pb

Zn

Interference study

Flow injection analysis

ABSTRACT

In this work, the suitability of extracts obtained from seafood samples after ultrasound-assisted extraction for determining Cd, Cu, Mn, Ni, Pb and Zn by Inductively Coupled Plasma-Mass Spectrometry was evaluated. For this purpose, different strategies were studied: (i) dilution of extracts; (ii) clean-up of extracts with C18 cartridges; (iii) use of a flow injection system for introducing extracts in the instrument. Potential interferences from acid extractant, major elements and organic matter were assessed. Internal standard, measurement mode and signal drift were also studied. Validation was carried out with the certified reference material TORT-2 (lobster hepatopancreas). Recoveries were between 92 and 106%. Precision, expressed as relative standard deviation, ranged from 2 to 6%. By using diluted extracts, limits of detection were similar as compared to those obtained after microwave-assisted digestion, or even better if a clean-up of extracts is applied. Following this approach, seafood samples of common consumption (mussel and shrimp) were rapidly analyzed.

© 2016 Elsevier B.V. All rights reserved.

1. Introduction

Ultrasound (US) has been recognized as an important energy input in the analytical laboratory. Although US can provide different effects, its major advantage is the shortening of many processes in sample preparation step such as derivatization, homogenization, emulsification or extraction. Solid-liquid extraction is especially accelerated by US. It occurs as a result of the cavitation phenomenon that provides high local temperatures and pressures, thus facilitating analyte solubility, diffusivity, penetrability/transport of solvent, and particle fragmentation. All those processes promote the rapid extraction of the analyte, hence providing simple and rapid sample pre-treatment in food analysis [1,2].

Ultrasound-assisted extraction (UAE) has been exploited as sample pre-treatment for elemental determination in different solid matrices as an alternative to microwave assisted digestion (MAD), in special in biological samples for which mild conditions are enough in order to achieve quantitative extractions. Usually, it is carried out using diluted acids as extractants. Concentrations between 1 and 5% (v/v) of nitric acid, hydrochloric acid or mixtures of both are generally utilized [2].

The use of small amounts of acids in sample preparation entails an important reduction of blank signals and it is always desirable in techniques involving nebulization systems for sample introduction. Both features are especially attractive for application of sensitive techniques such as Inductively Coupled Plasma-Mass Spectrometry (ICP-MS).

To date, Flame Absorption Atomic Spectrometry (FAAS) and speciality Electrothermal Absorption Atomic Spectrometry (ETAAS) are the most popular analytical techniques employed after UAE as sample pre-treatment in food analysis [3,4]. On the contrary, despite its excellent analytical characteristics, ICP-MS has been scarcely used so far in combination with UAE. In this regard, Balarama Krishna and Arunachalam [5] determined Na, K, Ca, Mg, Cr, Mn, Co, Ni, Cu, Zn, Ge, As, Se, Rb, Sr, Zr, Ag, Cd, In, Sb, Cs, Ba, Pb and Bi in two certified reference materials (CRMs) corresponding to lichen and mussel by plasma techniques (i.e., Inductively Coupled Plasma-Optical Emission Spectrometry, ICP-OES, and ICP-MS) after UAE. No information about compatibility of extracts with the ICP-MS instrument is indicated. Similarly, Lemos Batista et al. [6] determined 14 elements in hair after UAE using 20% (v/v) HNO₃ as extractant. Costas et al. [7] reported several problems arising from introduction of marine biological tissue extracts when rare earth elements (Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Lu) were determined by ICP-MS. Clean-up of extracts with a

* Corresponding author.

E-mail address: isela@uvigo.es (I. Lavilla).

Table 1
Instrumental operating conditions for ICP-MS measurements.

Torch:	Quartz vessel, 1.5 mm injector
Nebulizer:	Meinhard, 2 mL
Spray chamber temperature:	Peltier system, cooled to 3 °C
Spray chamber:	Quartz impact bead
Interface:	Ni sampler and skimmer cones
Forward power:	1300 W
Sample flow rate:	2 mL min ⁻¹
Argon flow rates:	Plasma, 13.1 mL min ⁻¹ Auxiliary, 0.60 mL min ⁻¹ Nebulizer, 0.75 mL min ⁻¹
Gas flow rate CCT (He):	10 mL min ⁻¹
Scanning mode:	Continuous
Replicates:	3
Sweeps per peak:	50

C18 cartridge to remove the organic matter was necessary. During the analysis, signal drift and salt deposition on the cones were observed. Recently, Almeida et al. [8] published the quantitative determination of Ba, Cd, Cr, Cu, Mn and Ni, and the semi-quantitative determination of Co and Pb in tobacco by UAE and ICP-MS.

Undoubtedly, direct analysis of biological extracts is troublesome in ICP-MS since organic matter is a strong interference, mainly causing plasma instability and carbon deposits on the interface [9]. In addition, the acid used as extractant may cause polyatomic spectral interferences and the high concentration of major elements in biological matrices may lead to signal suppression [10]. Although some instrumental strategies such as increased plasma power, addition of oxygen to the cooling gas, freezing of the spray chamber, use of collision cell technology (CCT), etc. can be used in order to remove interferences, further efforts may be necessary [11]. Internal standardization (used in virtually all applications carried out with ICP-MS), calibration by standard addition, use of matrix-matching, matrix separation and flow injection (FI) analysis are the most popular for this purpose. In particular, FI systems can overcome problems related to high amounts of dissolved salts and solids, high sample viscosity and/or acidity [12].

The goal of this work is to evaluate the suitability of extracts from seafood samples obtained by UAE for determining Cd, Cu, Mn, Ni, Pb and Zn by ICP-MS as a simple and rapid alternative to MAD. To this end, different strategies were evaluated: (i) dilution of extracts; (ii) clean-up of extracts with a C18 cartridge for non-polar solid phase extraction; (iii) use of a FI system for direct introduction of extracts in the instrument.

2. Materials and methods

2.1. Reagents and samples

A multielemental standard stock solution ICP Multi-Element Standard Solution VI (Merck, Darmstadt, Germany) containing Cd, Pb, Cr, Mn, Ni (10 mg L⁻¹) and Zn (100 mg L⁻¹) was used for calibration purposes. Standard working solutions were prepared daily by suitable dilution with 2% (v/v) nitric acid. A tuning solution (Fisher Scientific, Loughborough, UK) containing Ba, Be, Ce, Co, In, Li, Mg, Pb, Rh, Tl, U and Y (10 mg L⁻¹) was used. For internal standardization, standard stock solutions (1000 mg L⁻¹) of Rh, Sc, Ge, and In (Merck, Darmstadt, Germany) were used.

Reagents of ultrapure grade were used for sample preparation: 67% (w/v) nitric acid (VWR, Prolabo®, Fontenay-sous-Bois, France), 37% (w/v) hydrochloric acid (Panreac, Barcelona, Spain) and hydrogen peroxide 33% (w/v) (Panreac). Cartridges C18 (Hipersep IC-RP, Metrohm, Herisau, Switzerland) were used for clean-up of extracts. Tritón- \times 100 (Panreac) was added to the carrier channel in the FI system.

The certified reference material NRCC TORT-2 (lobster hepatopancreas) was purchased from the National Research Council of Canada (NRCC, Ottawa, Canada). Samples of mussel (*Mytilus galloprovincialis*)

and shrimp (*Palaemon serratus*) were purchased in a local market (Vigo, Spain).

A MilliQ water purified system (Millipore, San Quentin, France) was used to obtain high purity deionized water (18 M Ω cm). Materials were soaked for 24 h with 10% (v/v) nitric acid, and then rinsed repeatedly with ultrapure water prior to use. All manipulations were carried out under clean room conditions to minimize contamination risks.

2.2. Instrumentation

A Thermo Elemental Serie \times 7 Q-ICP-MS (Thermo Fisher Scientific, Bremen, Germany) was used to measure Cd, Cu, Mn, Ni, Pb and Zn. The instrument was equipped with the following components: a Peltier cooled impact bead spray chamber, a Meinhard concentric nebulizer, nickel sampler and skimmer cones and collision cell technology. An autosampler Cetac ASX-520 (Cetac Technologies, Omaha, NE, USA)

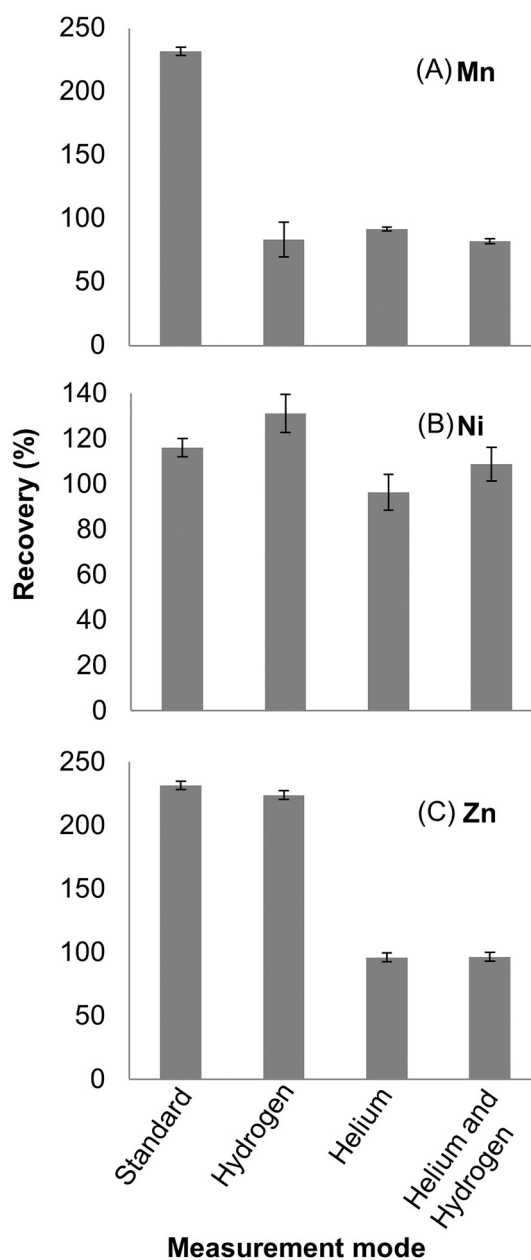


Fig. 1. Selection of measurement mode (standard, CCT with H₂, CCT with He and CCT with H₂ and He). Results obtained for (A) Mn; (B) Ni; and (C) Zn using diluted extracts (0.1 mg sample; 5 mL of 3% v/v HNO₃ as extractant; sonication conditions: 5 min and 60% sonication amplitude; dilution 1:5).

Download English Version:

<https://daneshyari.com/en/article/7641386>

Download Persian Version:

<https://daneshyari.com/article/7641386>

[Daneshyari.com](https://daneshyari.com)