



Ultrasound assisted-ionic liquid-dispersive liquid-liquid microextraction for preconcentration of inorganic tellurium in environmental water samples prior to inductively coupled plasma – Optical emission spectrometry detection



Luthando Nyaba, N. Raphael Biata, J. Catherine Ngila, Philiswa N. Nomngongo *

Department of Applied Chemistry, University of Johannesburg, Doornfontein Campus, P.O. Box 17011, Johannesburg 2028, South Africa

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ABSTRACT

In this study, a rapid, simple, selective and highly efficient ultrasound assisted-ionic liquid-dispersive liquid-liquid microextraction (UA-IL-DLLME) for preconcentration of tellurium in environmental water samples has been developed. The concentration of Te after microextraction was quantified using inductively coupled plasma optical emission spectrometry (ICP-OES). Experimental parameters that enhances in the performance of the microextraction process were optimized using a multivariate strategy. Under optimized conditions the enrichment factor, limits of detection (LOD) and limits of quantification (LOQ) were 850, 0.42 ng L⁻¹ and 1.4 ng L⁻¹, respectively. The intra-day (n = 11) and inter-day (n = 5) precisions expressed in terms of relative standard deviations (RSD) were found to be 2.3% and 4.5%, respectively. The accuracy of the developed method was tested by analyzing a standard reference material (SRM 1643e, trace elements in water), a drinking water certified reference material (CRM-TMDW) and spiked real water samples. The obtained values were in good agreement with the certified or added values and the recoveries were >95%. In addition, the UA-IL-DLLME/ICP-OES method was applied for the determination of Te in different water samples.

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1. Introduction

Tellurium is a rare, non-essential and toxic element [1] that is widely used in metallurgy, thermoelectric materials, semiconductors and as a colouring agent in chinaware [2]. The increase in the use of Te, results in its enrichment in the environmental matrices. In water systems, Te predominantly exists as toxic inorganic species (such as Te(IV) and Te(VI)) [2]. Tellurium has the potential of accumulating in the kidneys, nervous system, lungs, gastrointestinal tract, heart, liver and spleen [3]. Due to the reactivity and chronic toxicity of Te, its concentration in the human body should not exceed 0.002 g kg⁻¹ [3]. Therefore, the emission of inorganic Te compounds in the environment may cause serious problems [4]. It is also reported that as a result of microbial actions, inorganic Te compounds can be transformed into volatile organometalloid compounds [4]. This transformation may result in the subsequent modification of the Te transport pattern and toxicological behavior [4]. In view of the above it is important to monitor Te in environmental water matrices.

Since, the total concentration of Te in environmental waters matrices is generally at ultra-trace levels (below µg L⁻¹) sensitive analytical methods are required for its quantification. These include hydride generation-atomic fluorescence spectroscopy (HG-AFS) [5], inductively coupled plasma-mass spectrometry (ICP-MS) [6] and electrothermal atomic absorption spectrometry (ETAAS) [3,7]. However, these techniques are not capable of direct analysis due to ultra-low concentration and matrix interference causes serious problems in trace metal detection of tellurium [8,9]. Therefore, the development of preconcentration techniques is needed prior to spectrometric determination of tellurium.

In recent years numerous sample preparation methods have been reported for speciation and preconcentration of trace elements. These include solid phase extraction (SPE) [6], dispersive solid phase microextraction (DSPME) [10], hollow fiber solid phase microextraction (HFSPME) [9], dispersive liquid-liquid microextraction (DLLME) [1,2,7,11,13,14], hollow fiber liquid phase microextraction (HFLPME) [3,12]. Among these, DLLME which is based on ternary component solvent system for preconcentration of trace elements from various matrices is one of the most attractive microextraction techniques [1,12]. The main attractive features of DLLME include speed, low cost, simple operation, low consumption of organic solvents, high precision, use of small sample volumes and a high preconcentration factor [1,2].

* Corresponding author.

E-mail addresses: pnnomngongo@uj.ac.za, nomngongo@yahoo.com (P.N. Nomngongo).

The aim of this study was to develop a UA-IL-DLLME method using a low-density ionic liquid (IL) and organic solvents as the extractant and dispersant solvents for preconcentration of inorganic Te in environmental water samples prior to ICP-OES determination. Hydrophobic low-density IL (Aliquat 336, tricaprylmethylammonium chloride) and hexane were used as the extraction and disperser solvents, respectively. Aliquat 336 was chosen due to its good extraction capacity for trace elements in different matrices [15]. In addition, Aliquat 336 can act as anion exchanger over wide pH ranges. *N*-hexane was selected due to its hydrophobic properties and good solubility for Aliquat 336. Since both solvents are immiscible with water, ultrasonication applied to disperse the extraction solvent mixture. This is because ultrasonication causes emulsion, homogenizing, and mass transferring between immiscible phases [13,16]. To the best of our knowledge the application of UA-IL-DLLME using low-density ionic liquid and organic solvents for preconcentration of Te is been reported for the first time. Furthermore, low density solvents were selected because they can be withdrawn directly from the extraction using a suitable syringe.

2. Experimental

2.1. Reagents and material

All chemicals used were of analytical reagent grade unless otherwise stated and ultra-pure water (Direct-Q® 3UV-R purifier system) was used throughout the experiments. Aliquat 336, *n*-hexane, toluene, octanol, hydrochloric acid (trace Select, 37%) and ultrapure nitric acid (69%) were purchased from Sigma-Aldrich (St. Louis MO, USA). Spectrascan single element standard (1000 mg L⁻¹) of Te(IV) (Teknolab, Norway) were used to prepare working solutions for UA-IL-DLLME. The accuracy of the developed methodology was checked using certified reference material, CRM-TMDW-500 (High-Purity Standards Inc., Charleston, SC, USA) and standard reference material, SRM 1643e (trace elements in water, National Institute of Standards and Technology (NIST), Gaithersburg, MD, USA).

2.2. Instrumentation

An inductively coupled plasma optical emission spectrometer (ICP-OES) (iCAP 6500 Duo, Thermo Scientific, UK) equipped with a charge injection device (CID) was used in the determination of Te trace metal. The samples were introduced with a concentric nebulizer and a cyclonic spray chamber. The operating parameters of the instrument are presented in Table 1. A Branson 5800 Ultrasonic Cleaner (UK) and Eppendorf 5702 Centrifuge (Germany) were used for ultrasonic assisted extraction and centrifugation, respectively.

2.3. Sample collection and preparation

Surface water samples were collected at different sampling sites in Soweto (Gauteng, South Africa) into pre-cleaned polyethylene bottles,

rinsed three times with the sample water before collection, and then refrigerated. Prior to analysis, the samples were filtered through 0.22 µm pore size membrane filters (Millipore Corporation, Bedford, MA, USA). (The sample collection was carried out a day before analysis.)

Tap water samples were collected from selected areas around the Johannesburg CBD (Gauteng, South Africa). Before collection, the tap water was allowed to run for 15 min and clean sample bottles were rinsed three times with the sample water. It should be noted that the tap water samples were analyzed on the day of sampling.

Wastewater samples were collected in a full-scale municipal treatment facility (5% industrial and 95% domestic) (City of Tshwane, Gauteng, South Africa). The wastewater (influent and effluent) samples were collected using a Bailer sampling scoop. Samples were filtered through 0.22 µm pore size membrane filters (Millipore Corporation, Bedford, MA, USA) and analyzed on the day of sampling.

2.4. Ultrasound assisted-ionic liquid-dispersive liquid-liquid microextraction procedure

The extraction solvent was prepared by dissolving an appropriate amount of Aliquat 336 in an appropriate volume of an organic solvent (toluene, hexane or octan-1-ol). This was done in order to achieve different percentages of Aliquat 336 in hexane. The prepared extraction solvent (composed of the IL in organic solvent) was then used in the UA-IL-DLLME procedure. The UA-IL-DLLME procedure was carried out as follows: 10.0 mL of model sample Te(IV) solution (at different pH values, 2–9) at concentration level of 50 µg L⁻¹ was placed in 15.0 mL screw topped centrifuge tubes and 100–500 µL of the extraction solvent mixture was rapidly injected into the sample solution. The extraction solvent (Aliquat 336 in hexane) was dispersed into solution using ultrasonication for 5–30 min and cloudy solution was formed. The inorganic Te was then extracted into the fine droplets [13,16]. The cloudy solution was subjected to centrifugation at 4700 rpm for 3 min. This was done in order to achieve phase separation. It should be noted that after extraction the analyte of interest was preconcentrated in the upper phase (immiscible organic rich phase) and therefore, after centrifugation, the immiscible phase settled at the top of the centrifuge tube. The water immiscible phase was extracted through a syringe and transferred into clean screw topped centrifuge tubes. The organic phase was treated with 500 µL of ethanol followed by 2 mL of 1.0 mol L⁻¹ HNO₃ and the mixture was analyzed by ICP-OES. The total amount of inorganic tellurium in the real samples was performed as follows: firstly, Te(VI) was reduced to Te(IV). This was achieved by heating the acidic Te(VI) solution (4 M HCl) using a DigiBlock digester (ED36S, Labtech, Wilmington, MA) at 100 °C for 30 min [3]. After reduction, the sample solution was subjected to extraction procedure and the total inorganic Te was obtained.

The selection of suitable disperser solvents was performed using a univariate approach. The optimization of factors affecting the UA-IL-DLLME method was first done by two-level full factorial design (FFD) followed by central composite design (CCD). The variables included extraction solvent volume (ESV), percentage of Aliquat 336 in hexane (%IL), extraction time (ET) and pH. Each variable was assigned low level (–), central point (0) and high level (+) as shown in Table 2.

Preliminary results demonstrated that separation of the aqueous and organic phase by centrifugation takes place between 1 and 3 min.

Table 1

Operating parameters of ICP-OES.

ICP-OES parameters	
RF generator power (W)	1150
Frequency of RF generator (MHz)	40
Coolant gas flow rate (L min ⁻¹)	12
Carrier gas flow rate (L min ⁻¹)	0.7
Auxiliary gas (L min ⁻¹)	1.0
Max integration times (s)	15
Pump rate (rpm)	50
Viewing configuration/Touch mode	Axial
Replicate	3
Flush time (s)	30

Table 2

Factors and levels used in 2⁴ factorial design for optimization of UA-IL-DLLME.

Parameters	Low level (–)	Central point (0)	High level (+)
Extraction solvent volume (µL)	100	550	1000
Percentage of Aliquat 336 in hexane (%IL)	0	25	50
Extraction time (min)	5	17.5	30
pH	2	5.5	9

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