



Fabric fiber sorbent extraction for on-line toxic metal determination by atomic absorption spectrometry: Determination of lead and cadmium in energy and soft drinks

Viktoria Kazantzi^a, Abuzar Kabir^b, Kenneth G. Furton^b, Aristidis Anthemidis^{a,*}

^a Laboratory of Analytical Chemistry, Department of Chemistry, Aristotle University, Thessaloniki, Greece

^b International Forensic Research Institute, Department of Chemistry and Biochemistry, Florida International University, Miami, Florida, USA

ARTICLE INFO

Article history:

Received 6 October 2017

Received in revised form 7 November 2017

Accepted 14 November 2017

Available online 14 November 2017

Keywords:

Toxic metals

Automatic

Flow injection

Fabric fibers

Atomic absorption spectrometry

Energy drinks

ABSTRACT

A novel simple and sensitive on-line flow injection (FI) fabric fiber sorbent extraction (FFSE) system adapted to flame atomic absorption spectrometry (FAAS) for automatic trace metal determination has been established. The FFSE platform is based on polyester fabric fibers coated with sol-gel poly-dimethylsiloxane (PDMS) packed into a microcolumn as sorbent packing material. The potentials of this new scheme were assessed for direct Pb(II) and Cd(II) determination in energy and soft drink samples. The on-line retention of target analytes on sol-gel coated fibers was carried out with sodium diethyl dithiocarbamate (DDTC), while elution was performed with methyl isobutyl ketone (MIBK). The effect of the main chemical and flow parameters on the sensitivity of the method was thoroughly studied. For a loading time of 90 s with a sampling frequency of 30 h⁻¹, enhancement factors of 167 and 40 and detection limits (3σ) of 1.6 and 0.3 μg L⁻¹ were obtained for Pb(II) and Cd(II) determination, respectively. The trueness of the developed method was estimated by analyzing a certified reference material and spiked tap water.

© 2017 Elsevier B.V. All rights reserved.

1. Introduction

Marketing of energy drinks has been growing rapidly with global consumption nearly doubled during the last years. These types of drinks are mostly consumed by young adults aged 18–34 and consequently leads to a high concern for the research community about the risks and the potentially harmful effects of their consumption on consumers' health, due to their often-high content of caffeine, sugar and other ingredients such as taurine, guarana, vitamins, and herbal ingredients [1]. Much work on energy drinks has focused on various negative physiological effects related to their consumption, including agitation, elevated blood pressure, sleep disturbance, increased susceptibility to addiction, dental caries, miscarriage, arrhythmia and even death [2–4]. Soft drinks are also widely marketed in various forms and brands, daily consumed by the general public. Their high consumption rate is attributed to their characteristic taste and flavor defined by the present constituents like sugar, carbonated water and flavoring agents [5].

Among other harmful ingredients, energy drinks as well as soft drinks are likely to contain traces of heavy metals which have been proved harmful and toxic to the human body and constitute a major topic of public health concern. Metals like Pb(II) and Cd(II) are

recognized as a major environmental health risk throughout the world. The main sources of Pb(II) include dust and soil, water, food and even though source water is not usually lead contaminated, lead is absorbed from drinking water from lead pipes or joints connected by lead solder. Lead poisoning may occur from food preparation or even from ingestion of fruit juices or even alcoholic beverages which also contain significant amounts of lead. Cadmium is frequently found in ores and minerals together with lead and other metals, therefore these metals are responsible for the increasing environmental pollution, because of the volcanic activity. Other major sources of Cd(II) contain industrial processes where it is used as a stabilizer in PVC products, an anticorrosive agent, a color pigment as well as in the fabrication of nickel cadmium batteries. The occurrence of both metals, regarding the food chain, often results from environmental (from surface and underground water) and industrial (during processing and packaging) contamination. More specifically, atmospheric contamination, the excessive use of pesticides and fertilizers as well as the irrigation with residual waters constitute the main cause of food and beverage contamination by heavy metals [6,7]. Due to the fact that these metals have the tendency to cause irreparable damage to the human body, their concentration in ingested beverages needs to be monitored and quantified for consumer safety and well-being.

Up to date, numerous sorbent extraction methodologies either in batch or automatic mode have been developed and applied for the

* Corresponding author.

E-mail address: anthemid@chem.auth.gr (A. Anthemidis).

determination of heavy metals in different types of samples [8–15]. Despite the fact that solid phase extraction (SPE) has become the technique of choice, other pretreatment techniques following the trend of miniaturization like solid phase micro extraction (SPME) [16–18], stir bar sorptive extraction (SBSE) [19] and thin film micro extraction (TFME) [20] have been developed, during the last decades. However, there is always a growing demand for greener procedures as well as high concern for more sensitive and reliable pretreatment techniques. In this context, a novel batch pretreatment technique namely fabric phase sorptive extraction (FPSE) introduced by Kabir and Furton [21], achieved to overcome drawbacks of most conventional sample preparation techniques including multiple time consuming steps, high consumption of organic solvents and small sorbent loading [22]. FPSE has been applied to extract various analytes from different types of matrices [23–24], taking advantage of the principles of sol-gel technology as well as a plethora of commercially available natural and synthetic fabrics used as FPSE substrates including cellulose, polyester, nylon, fiber glass and polyamide [25].

Recently, the automation of the FPSE technique was demonstrated for on-line extraction and determination of trace metals in environmental samples by flame atomic absorption spectrometry (FAAS) with satisfactory results [26]. The proposed method named fabric disk sorptive extraction (FDSE) is based on minicolumns packed with a number of disks derived from sol-gel coated fabric media. This configuration facilitates the use of the effective FPSE media in on-line automatic flow systems resulting in high reproducibility, short analytical cycles as well as good sensitivity of the method.

In the present work, polyester fabric fibers coated with sol-gel polydimethylsiloxane (PDMS), were fashioned and used for the first time in an automatic SPE microcolumn preconcentration system. The fabric fiber sorbent extraction (FFSE) platform is based on the creation of a new sol-gel coating onto polyester fibers as a new sorbent material packed into an on-line microcolumn. The performance of the proposed microcolumn using the sol-gel PDMS coating, was demonstrated for ultra-trace amount of Pb(II) and Cd(II) determination by atomic absorption spectrometry (AAS). Sodium diethyl dithiocarbamate (DDTC) was used as chelating reagent and methyl isobutyl ketone (MIBK) as eluent. The method was thoroughly optimized and evaluated by analyzing certified reference materials and spiked water samples. In addition, commercially available energy and soft drinks were directly analyzed to monitor possible contamination by Pb(II) and Cd(II).

2. Experimental

2.1. Apparatus

A Perkin-Elmer (Norwalk, CT, USA) Model 5100 PC flame atomic absorption spectrometer with deuterium lamp background corrector was furnished with Pb(II) and Cd(II) hollow cathode lamps (HCL) operated at 10 mA and 4 mA respectively. The wavelength was set at 283.3 nm and 228.8 nm for Pb(II) and Cd(II) respectively and the monochromator spectral bandpass (slit) at 0.7 nm for both metals. The flame composition was adjusted properly to compensate for the effect of the used eluent, MIBK, served as an additional fuel. The air flow rate was set at 10.0 L min⁻¹ and acetylene flow rate at 1.0 L min⁻¹. The resulting nebulizer free uptake rate was 5.8 mL min⁻¹.

A Perkin-Elmer (Norwalk, CT, USA) Model FIAS-400 flow injection analysis system was coupled to the nebulizer system of the spectrometer operated in preconcentration mode. The FIAS-400 manifold consisted of two peristaltic pumps with Tygon tubing and a 5-port 2-position injection valve (IV). One peristaltic pump was used for the propulsion of the sample and the chelating reagent. Due to the incompatibility of organic solvents, like MIBK, with the pumping tube, a high-precision bi-directional syringe pump (MicroCSP-3000, FIALab instruments (Bellevue, WA) with a 2.5 mL glass barrel (TECAN) integrated with a three-port Teflon/Kel-F selection valve on the top of it, was used

for eluent propulsion. The syringe pump was controlled through a FIALab®-3000 sequential injection system and the FIALab software. The FIAS-400 flow system was controlled by a personal computer using the AA Lab. Benchtop version 7.2 application software and synchronized with the FIALab software. A “T” type mixing device was used just ahead of the inlet of FAAS for flow compensation (FC) between elution flow rate and nebulizer aspiration. All tubing of the flow system was made of poly-tetrafluoroethylene (PTFE).

2.2. Materials, reagents and samples

Polyester fabric substrate (100% polyester) for fabric fiber sorbent extraction device was purchased from Jo-Ann Fabric (Miami, FL, USA). Polydimethylsiloxane (PDMS) was purchased from Gelest Inc. (Morrisville, PA, USA). Methyltrimethoxysilane (MTMS), acetone, dichloromethane and trifluoroacetic acid (TFA) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Sodium hydroxide and hydrochloric acid were purchased from Thermo Fisher Scientific (Milwaukee, WI, USA).

All chemicals were of analytical reagent grade and were provided by Merck (Darmstadt, Germany, <http://www.merck.de>). Ultra-pure quality water, produced by a Milli-Qsystem (Millipore, Bedford, USA, <http://www.millipore.com>) was used throughout the study. Working standard solutions of Pb(II) and Cd(II) were prepared by appropriate stepwise dilution of a 1000 mg L⁻¹ Pb(II) and Cd(II) stock standard solutions (Merck Titrisol) to the required µg L⁻¹ levels prior to use. The standard solutions and samples were acidified to 0.01 mol L⁻¹ HNO₃ (pH ~ 2.0). The chelating reagent 0.05% (m/v) DDTC was freshly prepared daily by dissolving the appropriate amount in water. MIBK was used after saturation with water, without any other purification. Laboratory glassware was rinsed with water and decontaminated overnight in 10% (v/v) nitric acid solution.

One certified reference material (CRM): NIST CRM 1643e (National Institute of Standard and Technology, Gaithersburg, MD, USA) containing trace elements in water was used to validate the accuracy and the precision of the proposed method. The method was also applied for the determination of Pb(II) and Cd(II) in tap water. All samples were filtered through 0.45 µm membrane filters, acidified to 0.01 mol L⁻¹ HNO₃ and stored at 4 °C in acid-cleaned polyethylene bottles in order to determine the “dissolved metal” fraction.

The developed method was applied to the analysis of energy and soft drink samples. Three brands of canned energy drinks (Samples ED1, ED2, ED3) as well as two soft drinks (SF1, SF2) were randomly purchased from the market in Thessaloniki, Greece and were evaluated for Pb(II) and Cd(II) determination. All samples were directly analyzed by the proposed FI-FFSE-FAAS method after completely degassed using a magnetic stirrer.

2.3. Preparation of the FFSE microcolumn

The laboratory made FFSE microcolumn was constructed using a pyrex® glass tube with 4.0 mm i.d. and 60.0 mm length with an active length of 50.0 mm. Polyester fabric fibers, with an approximate amount of 75 mg of sol-gel PDMS sorbent, were packed and fixed properly into the FFSE microcolumn, as illustrated in Fig. 1. No frits or glass wool were necessary at the two ends of the microcolumn to block sol-gel PDMS coated fibers acting as the sorbent material. This novel column configuration reveals a chaotic geometrical structure resulting in low flow resistance, high extraction efficiency with excellent reproducibility and satisfactory sensitivity. The FFSE microcolumn was proven to operate reliably for at least 600 analytical cycles.

2.4. Automatic analytical procedure

The overall analytical procedure for the determination of Pb(II) and Cd(II) exploiting the FI-FFSE-FAAS method comprises on-line sample

Download English Version:

<https://daneshyari.com/en/article/7640991>

Download Persian Version:

<https://daneshyari.com/article/7640991>

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