



Analytical Methods

Application of solvent-assisted dispersive solid phase extraction as a new, fast, simple and reliable preconcentration and trace detection of lead and cadmium ions in fruit and water samples



Mohammad Behbahani^{a,*}, Parmoon Ghareh Hassanlou^a, Mostafa M. Amini^a, Fariborz Omidi^b, Ali Esrafil^c, Mehdi Farzadkia^c, Akbar Bagheri^a

^a Department of Chemistry, Shahid Beheshti University, Evin, Tehran, Iran

^b Department of Occupational Health Engineering, School of Public Health, Shahroud University of Medical Sciences, Shahroud, Iran

^c Department of Environmental Health Engineering, School of Public Health, Iran University of Medical Sciences, Tehran, Iran

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ABSTRACT

In this research, a new sample treatment technique termed solvent-assisted dispersive solid phase extraction (SA-DSPE) was developed. The new method was based on the dispersion of the sorbent into the sample to maximize the contact surface. In this approach, the dispersion of the sorbent at a very low milligram level was achieved by injecting a mixture solution of the sorbent and disperser solvent into the aqueous sample. Thereby, a cloudy solution formed. The cloudy solution resulted from the dispersion of the fine particles of the sorbent in the bulk aqueous sample. After extraction, the cloudy solution was centrifuged and the enriched analytes in the sediment phase dissolved in ethanol and determined by flame atomic absorption spectrophotometer. Under the optimized conditions, the detection limit for lead and cadmium ions was $1.2 \mu\text{g L}^{-1}$ and $0.2 \mu\text{g L}^{-1}$, respectively. Furthermore, the preconcentration factor was 299.3 and 137.1 for cadmium and lead ions, respectively. SA-DSPE was successfully applied for trace determination of lead and cadmium in fruit (*Citrus limetta*, Kiwi and pomegranate) and water samples.

Finally, the introduced sample preparation method can be used as a simple, rapid, reliable, selective and sensitive method for flame atomic absorption spectrophotometric determination of trace levels of lead and cadmium ions in fruit and water samples.

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1. Introduction

Pollution of the environment with heavy metals is a global problem giving threats to the humanity. There are several sources of heavy metal pollution to the environment (soil and water) that include wastes from coal, natural gas, paper and textile industries. Cadmium, chromium, copper and lead are the most common toxic ions contained in industrial waste water. These heavy metal ions are toxic to human. For example, cadmium associated with renal dysfunction and leads to lung disease (Zou et al., 2008). Environmental regulations are legislated to protect the humanity from heavy metal pollution and they provide guidelines and threshold limits for each of the heavy metals in drinking water. Typically, world health organization recommends a maximum of $3 \mu\text{g L}^{-1}$ and $10 \mu\text{g L}^{-1}$ for cadmium and lead, respectively, in their guidelines for drinking water. Because of the risk to human health

and a variety of environmental problems, the extent of heavy metal pollution needs to be assessed as it occurs, so that it can be remedied before it becomes hazardous. Due to these important points, the accurate determinations of trace heavy metals are important part of the studies in analytical chemistry (Ebrahimzadeh & Behbahani, 2013; Hossien-poor-Zaryabi et al., 2014; Kalate Bojdi et al., 2014; Soylak & Cay, 2007). Flame atomic absorption spectrometry (FAAS) is relatively simple and available technique in many laboratories for heavy metal determinations (Cui et al., 2007; Gama, Lima, & Lemos, 2006; Meesri, Praphairaksit, & Imyim, 2007; Refiker, Merdivan, & Aygun, 2008). However, the conventional determinations of elements at $\mu\text{g L}^{-1}$ range by flame atomic absorption spectrometry frequently are not possible. To solve this problem, preconcentration/separation procedures have been proposed. Preconcentration is a very important issue for achievement of low detection limits (Ahmadi, Khanmohammadi, Niknam, & Jafarpour, 2007; Behbahani, Babapour, et al., 2013; Kruanetr, Thanasarakhan, Tengjaroenkul, Liawruangrath, & Liawruangrath, 2007; Madrakian & Ghazizadeh, 2008). Several

* Corresponding author. Tel.: +98 21 22431661; fax: +98 21 22431683.

E-mail address: mohammadbehbahani89@yahoo.com (M. Behbahani).

procedures have been developed for these purposes such as liquid–liquid extraction (LLE) (Marczenko, 1986; Silvestre, Santos, Lima, & Zagatto, 2009), co-precipitation (Anthemidis, Zachariadis, & Stratis, 2003; Atsumi, Minami, & Uada, 2005; Doner & Ege, 2005; Saracoglu, Soyvak, & Elci, 2003), and solid phase extraction (SPE) (Behbahani, Najafi, et al., 2013, 2014; Behbahani, Bide, et al., 2014; Behbahani, Esrafil, et al., 2014; Behbahani, Ali Akbari, Amini, & Bagheri, 2014; Salarian, Ghanbarpour, Behbahani, Bagheri, & Bagheri, 2014). However, some of these pretreatment methods need large amounts of sample and/or organic solvents, are time-consuming and expensive, and have unsatisfactory enrichment factors. These problems could be addressed by the development of modular and compact processes that provide adequate separation and preconcentration without complex processes. SPE is more flexible and efficient than LLE in terms of simplicity, low consumption of the organic solvents, flexibility in choosing the adsorbent, and high enrichment factor. Recently, SPE has been increasingly used for the preconcentration and separation of ultra-trace amounts of inorganic and organic species from complex matrices (Behbahani et al., 2015; Li et al., 2012). Solid phase extraction techniques are surface-dependent processes since their efficiency directly depends on the particle size and the surface area of the sorbent (Fritz & Macka, 2000). Dispersive solid phase extraction (DSPE) has risen as an alternative to conventional solid phase extraction. It was, for the first time, proposed by Anastassiades, Lehotay, Stajnbaher, and Schenck (2003) and it can be considered as a quick, easy, cheap, effective, rugged, and safe (QuEChERS) sample treatment method. It is based on the SPE methodology, but the sorbent is added to the extract without conditioning, in small amounts (viz. 50 mg) and the dispersion is carried out assisted by an external energy (usually a vortex stirring). The phases are easily separated just by centrifugation. Sorbent dispersion leads to an increase of its active surface, and therefore to an improvement in the extraction kinetics. Moreover, this enhancement allows the use of a smaller amount of sorbent compared to the conventional extraction approaches resulting in the saving of material. In order to obtain analytical information, the sorbent is then recovered after the extraction. The analytes can be directly monitored on the sorbent surface by using a spectroscopic technique (Alcudia-Leon, Lucena, Cardenas, & Valcarcel, 2009; Garg, Pardasani, Mazumder, Purohit, & Dubey, 2011) or can be conveniently eluted/desorbed for the subsequent analysis of the eluted fraction (Drozdzyński & Kowalska, 2009; Roman, Chisvert, & Canals, 2011; Tsai, Huang, Huang, Hsue, & Chuang, 2009; Wu et al., 2009).

The aim of this work was to combine SA-DSPE with FAAS to develop an improved procedure for the determination of cadmium and lead ions at trace levels with respect to the advantages of solvent assisted dispersive solid phase extraction (SA-DSPE) which is a fast and inexpensive method in comparison with other methods. In this method, the appropriate mixture of sorbent and disperser solvent was rapidly injected into the aqueous sample by syringe. Thereby, a cloudy solution formed. The cloudy state resulted from the dispersion of the fine particles of the sorbent in the bulk aqueous sample. Then, this cloudy solution was centrifuged, following which the fine particles sediment at the bottom of the conical test tube. Then the sediment phase was dissolved in ethanol and the analytes were determined using FAAS. In this extraction method, any component in the solution, directly or indirectly after previous derivatization reactions, interacts with the fine particles of the sorbent, and consequently, gets extracted from the initial solution. In this work, dithizone was used as a suitable chelating agent to form complex with cadmium and lead ions and factors that would influence the efficiency of SA-DSPE were also investigated and optimized. Finally, the introduced sample treatment technique was

used for preconcentration and traces determination of cadmium and lead ions in different matrices.

2. Experimental

2.1. Apparatus

Lead and cadmium concentration were determined by an AA-680 Shimadzu (Kyoto, Japan) flame atomic absorption spectrometer (FAAS) in an air-acetylene flame, according to the user's manual, provided by the manufacturer. Lead and cadmium hollow cathode lamps (HCL) were used as the radiation source with wavelengths of 283 nm, and 228.8 nm, respectively. The pH was measured at 25 ± 1 °C with a digital WTW Metrohm 827 Ion analyzer (Herisau, Switzerland) equipped with a combined glass-calomel electrode.

2.2. Reagent and materials

All analytical grade reagents were purchased from Merck (Darmstadt, Germany, www.merck.de) and used without further purification. A stock solution (100.0 mg L^{-1}) of lead and cadmium ions was prepared by dissolving an appropriate amount of corresponding nitrate salts in double-distilled water. Working standard solutions were prepared daily through serial dilutions of the stock solution with deionized water prior to analysis. All standard solutions for FAAS instrument calibration were prepared in ethanol. A 100.0 mg L^{-1} solution of dithizone (DT) (Merck, Darmstadt, Germany) in pure ethanol was prepared. Naphthalene, benzophenone, and 1,4-dichlorobenzene were purchased from Sigma-Aldrich (St. Louis, MO, USA). All glass vessels used for trace analysis were cleaned before use by soaking them in 10% nitric acid solution for at least 24 h and then rinsed thoroughly with ultrapure water. Soil (NCS DC 73323) and Ore polymetallic gold Zidarovo-PMZrZ (206 BG 326) from Bulgaria was used as the reference materials for validation of the proposed technique.

2.3. Real sample pretreatment

2.3.1. Food samples (*Citrus limetta*, Kiwi and pomegranate)

Three types of fruits were chosen for analysis. These were *C. limetta*, Kiwi and pomegranate, which were collected from the local supermarket (Tehran, Iran). 1.0 g of dried and grounded samples was put into burning cup with 15 mL of pure NHO_3 . The samples were incinerated in a MARS 5 microwave oven at 200 °C. After digestion treatment, the samples were filtrated through Whatman No. 42. After filtration, the obtained clear solution was diluted to 50.0 mL (pH of 7.0) for cadmium and lead analysis.

2.3.2. Water samples

The polyethylene bottles filled with the samples were cleaned with detergent, water, diluted nitric acid and water in sequence. The samples were immediately filtered through a cellulose filter membrane (pore size $0.45 \mu\text{m}$), and were acidified to pH of 2.0 for storage. Tap water samples (50.0 mL) were taken from our research laboratory without pretreatment (pH adjusted to 7.0). Before analysis, the water samples (50.0 mL) which were taken from Caspian Sea, river and waste were adjusted to pH of 7.0 according to optimized experimental conditions.

2.3.3. Certified reference materials

To digest the certified reference materials, 50 mg from each of them was digested with 6 mL of HCl (37%) and 2 mL of HNO_3 (65%) in a microwave digestion system. The digestion was carried out for 2 min at 250 W, 2 min at 0 W, 6 min at 250 W, 5 min at

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