



Contents lists available at ScienceDirect

Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy

journal homepage: www.elsevier.com/locate/saa

A new solid phase microextraction method using organic ligand in micropipette tip syringe system packed with modified carbon cloth for preconcentration of cadmium in drinking water and blood samples of kidney failure patients



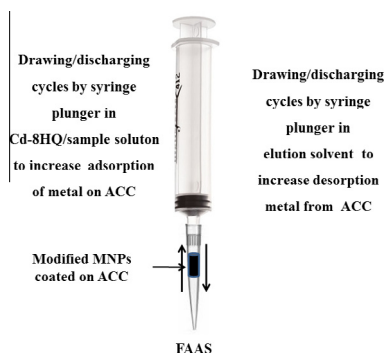
Abdul Haleem Panhwar*, Tasneem Gul Kazi¹, Hassan Imran Afridi¹, Salma Aslam Arain¹, Naeemullah¹, Kapil Dev Brahman¹, Mariam Shahzadi Arain¹

National Centre of Excellence in Analytical Chemistry, University of Sindh, Jamshoro 76080, Pakistan

HIGHLIGHTS

- A fast and simple miniaturized SP μ E method with carbon cloth in a syringe system.
- The retention of Cd was achieved by using 8 hydroxyquinoline as complexing agent.
- A new miniaturized SP μ E method was easy and has less solvent consumption.
- It was applied for determination of Cd in water, blood and urine samples.
- High level of Cd in water causes adverse effects to the kidney failure patients.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 14 August 2014

Received in revised form 1 November 2014

Accepted 20 November 2014

Available online 28 November 2014

Keywords:

Cadmium

8-Hydroxyquinoline

Activated carbon cloth

Solid phase microextraction

Syringe

Flame atomic absorption spectrometry

ABSTRACT

A simple and efficient miniaturized solid phase microextraction (M-SP μ E) in a syringe system was developed for preconcentration of cadmium (Cd) in environmental and biological samples, followed by flame atomic absorption technique. The syringe system contains the micropipette tip packed with activated carbon cloth, coated with modified magnetic nanoparticles of iron oxide Triton X114 (ACC-NPs). Scanning electron microscopy and energy dispersive spectroscopy used for characterization of the size, morphology and elemental composition of ACC-NPs. The sample solution treated with a complexing reagent 8-hydroxyquinoline (8-HQ), and drawn into the syringe, filled with ACC-MNPs and dispensed manually for 2–10 drawing/discharging cycles. The analyte retained on ACC-NPs in micropipette tip-syringe system were then eluted with different volume of 1.5 mol L⁻¹ HCl by 1–5 drawing/discharging cycles. The syringe system directly couple with FAAS for analysis. The influence of different variables on the extraction efficiency of Cd, including adsorbent dosage, pH, sample volume, eluent volume and drawing/discharging cycles of syringe system were optimized.

At optimized extraction conditions, the method showed good linearity in the range of 5–250 μ g L⁻¹, with a limit of detection 0.15 μ g L⁻¹. Repeatability of the extraction (%RSD) was <5%, $n = 5$. The validity

* Corresponding author. Tel.: +92 0222 771379; fax: +92 0221 771560.

E-mail addresses: haleem_analyst@yahoo.com (A.H. Panhwar), tgkazi@yahoo.com (T.G. Kazi), hassanimranafriidi@yahoo.com (H.I. Afridi), salma_arain67@yahoo.com (S.A. Arain), khannaemullah@gmail.com (Naeemullah), Kr_brahman@yahoo.com (K.D. Brahman), mshahzadi71@yahoo.com (M.S. Arain).

¹ Tel.: +92 0222 771379; fax: +92 0221 771560.

and accuracy of the method was checked by the certified reference materials. The proposed method was successfully applied for the determination of Cd in different drinking water and biological samples of kidney failure patients and healthy controls.

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Introduction

Cadmium is a well-known toxic and persistent pollutant that is naturally present at very low levels in aquatic environments and biological system [1]. Chronic exposure to Cd often causes renal dysfunction and it is believed that, the general population mainly exposed by drinking water and food [2]. Continuously consumption of drinking water containing Cd > 3.0 $\mu\text{g L}^{-1}$ cause adverse effects [3]. The U.S. environmental protection agency (EPA) classified Cd as a human carcinogen at low-level exposure [4]. As the levels of Cd is very low in environmental and biological samples, direct determination of Cd appears to be a difficult task as the concentration of this ion is close to or below the detection limits of most of the analytical instruments and the real sample matrix may cause serious interference for its determination.

Several analytical techniques such as atomic absorption spectrometry [5–7], and inductively coupled plasma mass spectrometry [8] are available for the determination of trace metals in environmental and biological samples. The direct determination of trace elements by spectroscopic methods, such as FAAS, is often difficult because of insufficient sensitivity and selectivity of the used methods. For this reason, the preliminary separation and preconcentration of trace elements from the different matrices are required [9,10]. The preconcentration–separation step improves the analytical detection limit, increases the sensitivity by several orders of magnitude, enhances the accuracy of the results and facilitate the calibration. A great variety of analytical procedures for cadmium enrichment have been developed, such as liquid–liquid extraction [11,12] solid phase extraction (SPE) [5] and cloud point extraction [13–15].

The development of conventional SPE methods were limited with respect to disadvantages to employing large volumes of hazardous organic solvents, producing secondary wastes, and tedious procedures [16]. The application of solid phase microextraction (SP- μE) and liquid phase microextraction were developed to remove these disadvantages [17]. Adsorption on carbon materials has been recognized by the US Environmental Protection Agency as one of the best adsorbent available to remove organic and inorganic compounds from water intended for human consumption [18–22].

The activated carbons are mainly microporous, contains meso and macropores, which are facilitating the adsorbate especially liquid molecules to the interior area of carbon particle [23–25]. The activated carbons have the advantages of flexible, repeated usability, higher mechanical strength, easy molding characteristics and handling for the potential miniaturization of SPE [26–28].

Iron oxide nano particles (Fe_3O_4 -NPs) offer a significantly higher surface area-to volume ratio that promises much greater extraction capacity and efficiency [29]. Another advantage of NPs is that its surface functionality can be easily modified to achieve selective sample extraction [30]. The magnetic NPs, mainly including Fe_3O_4 , appear as an interesting advanced composite material. It has received increasing attention in the past decades due to its unique physical and chemical properties and high potential applications in organic and inorganic fields [31–33].

In the present work a miniaturized solid phase extraction of Cd in drinking water and blood samples of kidney failure patients has been developed. The retention of the metal ions in the form of complexes on modified ACC with nanoparticles in a micropipette

tip coupled with syringe system was achieved by using 8-HQ as the complexing agent. The analytes retained on a micropipette tip ACC syringe system were then eluted with acid and directly injected into the FAAS for analysis. Factors that influence the enrichment efficiencies of the desired (M-SP μE) procedure were investigated and optimized. This work covers a new cost effective and simple preconcentration method for the determination of Cd in water and biological samples.

Experimental

Chemical reagents and glassware

Ultrapure water obtained from the ELGA lab water system (Bucks, UK), was used throughout the work. The stock standard solution of Cd ($1000 \mu\text{g L}^{-1}$) was obtained from the Fluka Kamica (Bush, Switzerland). Working standard solutions were prepared by appropriate dilution of the stock standard solutions before analysis. Concentrated nitric acid, hydrochloric acid were analytical reagent grade from Merck (Darmstadt, Germany) and were checked for possible trace Cd contamination by preparing blanks for each procedure. The 8-hydroxyquinoline (8-HQ), was obtained from (Fluka) and prepared by dissolving appropriate amount of reagent in 10 mL ethanol (Merck) and diluting to 100 mL with 0.01 mol L^{-1} acetic acid and kept in refrigerator at 4°C for one week. The 0.1 mol L^{-1} phosphate buffer was used to control the pH in the range of (4–8) of the solutions. The pH of the samples was adjusted to the desired pH by the addition of (0.1 mol L^{-1} HCl/NaOH) in the buffers. Pipette tip and syringe from Huaxin Chemical Reagent (Baoding, China). The ACC used in the present work was obtained from Spectra Corp. (MA, USA) coded as Spectra-carb 2225. The certified reference material of water SRM-1643e (National Institute of Standards and Technology (NIST), Gaithersburg, MD, USA) was used. The certified reference material of blood (clincheck) and urine (Seronom™ Trace Elements Urine L-2 LOT 1011645) were purchased, from Sero AS and Bio-Rad (Milan, Italy).

The glass and plastic wares were soaked in 10% nitric acid overnight and rinsed many times with deionized water to avoid contamination.

Instrumentation

A pH meter (Ecoscan Ion 6, Malaysia) was employed for pH adjustments. Global positioning system (iFinder GPS, Lowrance, Mexico) was used for sampling locations. A Perkin Elmer Model AAnalyst 700 (Norwalk, CT), atomic absorption spectrometer. All measurements were carried out in an air/acetylene flame. 10 cm long slot-burner heads, hollow cathode lamp of cadmium was used as radiation source at wave length (nm) 228.8. The operating parameters were set as recommended by the manufacturer. The surface morphology and elemental composition of modified ACC was carried out by EDS (XFlash-4010) mount on the Scanning Electron Microscope (SEM, FEI Co., Hillsboro, USA).

Synthesis of Fe_3O_4 nanoparticles

The Fe_3O_4 nanoparticles (NPs) was prepared by the chemical coprecipitation method as reported in previous work [34]. Specific

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