



Inorganic arsenic speciation in water samples by miniaturized solid phase microextraction using a new polystyrene polydimethyl siloxane polymer in micropipette tip of syringe system

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ARTICLE INFO

Article history:

Received 30 June 2016

Received in revised form

24 August 2016

Accepted 28 August 2016

Available online 29 August 2016

Keywords:

Inorganic arsenic speciation
Natural water
Polystyrene polydimethyl siloxane
Syringe system
Electrothermal atomic absorption spectrometry

ABSTRACT

The polymer, polystyrene polydimethyl siloxane was loaded into the micropipette tip of the syringe system as an adsorbent to developed miniaturized solid phase microextraction. Standard solutions of arsenate and arsenite were passed through the adsorbent loaded in micropipette tip to check the adsorption behaviors. It was observed that arsenate adsorbed on the polystyrene polydimethyl siloxane in the pH rang of 6–8, while arsenite was directly passed through the micropipette tip of syringe system. The adsorbed arsenate in micropipette tip of syringe system were eluted by 1.0 M hydrochloric acid. The total inorganic arsenic contents were obtained by the addition of oxidizing agent potassium permanganate into the studied samples before passing to the micropipette tip of syringe system. Arsenite concentration in water samples were measured by subtracting arsenate from total inorganic arsenic concentration. Different characteristics which effect the determination of arsenate specie like amount of adsorbent, adsorption capacity, pH, pulled and pushed cycles for adsorption and desorption, volume of sample, eluent type and it volume were also studied in detail. Enrichment factor and detection limit of arsenate by desired method were 218 and 6.9 ng L⁻¹ respectively. The relative standard deviation was 4.1% (n=10, C=0.12 µg L⁻¹). Accuracy of the desired technique was confirmed by analysis of the CRMs (Lake Ontario Water TM-28.3 and Riverine Water NRCC-SLRS-4). Desired technique was significantly useful for determination of the total arsenic, arsenate, and arsenite contents in different natural water samples.

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1. Introduction

The arsenic (As) has occurrence in the environmental at a trace level in the forms of organic/inorganic compounds. Measurement of total inorganic arsenic (tAs), arsenate and arsenite in the environmental samples is very important for risk assessment of life and its toxicity [1]. The toxicity of As depends on its species, like arsenite is more toxic than arsenate and inorganic As compounds are much more hazardous than its organic forms can be persisted in the environmental samples at ultra-trace levels [2]. Arsenic is very toxic metalloid for human being to cause different diseases like skin lesions, keratosis (skin hardening) and carcinogenic effect (lung cancer, liver tumors, and bladder cancer), when it exposure to body even at a trace level, that way it become an important issue in the world to affecting the life [3]. Contamination of As in

different environmental water and other human being consumption samples have affect > 100 million people across the world [4,5]. In developing countries contamination of As in water system like surface water and ground water where higher than the permissible level of As for drinking water [6]. The recommended permissible level of As in drinking water according to the world health organization (WHO) is 10 µg L⁻¹ [7]. Occurrence of As in the water samples due to the leaching of As from different natural sources as well as human activities like industrial effluents, pesticides, wood preservative agents, combustion of fossil fuels, and mining activity [8]. Industrial waste water mostly contains different toxic elements and created environmental pollution and harmful for living things. Minerals are the main sources of arsenic occurrence in the earth crust and released into the environment through water leaching [9,10]. Arsenopyrite is the most abundant mineral which is frequently occurred in the soil and sediments of the mining area [11]. Explaining the leaching mechanisms of As from arsenopyrite has been a great interest to numerous researchers of the world [12]. Contamination of As in soil and

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different natural water samples may be affected crops and pose hazards to human health through food chain transfer [13]. Stability of inorganic As species in water samples is a major concern due to inter conversion of As species like arsenate into arsenite [14]. Stability of As species were achieved by maintaining the following parameter like pH, light, microbial activity, sampling container material, storage in refrigerator at 4 °C and analysis was done as soon as possible within one day [15].

In the light of above mention fact and figures, it is necessary to accurately measure of As species in different natural water samples. Many analytical methods like co-precipitation was presented in the literature for inorganic As speciation in environmental water samples by graphite furnace atomic absorption spectrometry (GF-AAS) [16]. Various instrumental techniques including hydride generation atomic absorption spectrometry (HG-AAS), hydride generation-atomic fluorescence spectrometry (HG-AFS) [17], inductively coupled plasma optical emission spectrometry (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS) [18] and high performance liquid chromatography-microwave digestion-hydride generation-atomic absorption spectrometry (HPLC-MW-HG-AAS) [19] after some modification steps to sample preparation and separation process [20–23] are used for the determination of arsenic. Disadvantages of the above mention analytical instrumental technique are very expensive running cost due to high price of accessories and also not available in each and every analytical laboratory especially in the developing countries. Therefore, some methods developed for the separation and pre-concentration of the target analyte from environmental samples according the need of the laboratory setting and low costs instruments [24]. Arsenic measurement field kits are also available in the market but it generally consists of As gas which create As pollution in the environment [8]. Accurately assessments of As species in the natural water samples is an ongoing requirement to developed a simple and effective method. Some of the reported method in the literature has been address these requirements. Direct analysis of As species by ET-AAS is much more difficult [25,26], due to the low LOD and interference of coexistence ions in the studied samples. Therefore, some methods were developed to preconcentration and separation to enhance LOD and reduced the interferences like liquid-liquid extraction, cloud point extraction, electro deposition, co-precipitation, membrane filtration and solid phase extraction (SPE) [27,28]. In the above methods, SPE is one of the best choices for samples preparation due to its simplicity, easy methodology, high EF, and sensitivity [29,30]. Maximum recovery of hydrophobic As species by using SPE method due to the solid support of hydrophobic functionality of adsorbents [31]. Many adsorbents have been reported in the literature to develop SPE method for analysis of As species likes solvent-impregnated resins, polyurethane foam, amberlite resins, modified clinoptilolite zeolite etc [32–34].

The basic principle of the current study is to introduce a miniaturized solid phase microextraction (MSPME) method for separation and preconcentration of inorganic As species, including total inorganic arsenic and arsenate in mineral water, tap water, ground water, lake water, river water and sea water. The developed MSPME was first time used for speciation and quantification of inorganic As species in the studied water samples coupled to analysis by ET-AAS. In the first MSPME step, a micropipette tip of the syringe system packed with polystyrene polydimethyl siloxane as adsorbent was used for the preconcentration of arsenate and the removal of matrix, especially organic compounds from natural water samples. The elution solution from MSPME was buffered and employed for further preconcentration and separation of the analyte with pulled and pushed cycles by using the plunger of the syringe. Various factors influencing separation, preconcentration and determination of the analyte was investigated in detail. The

proposed MSPME method for quantification of low level As species in natural water samples of different ecosystem and open a door for their application in different environmental samples.

2. Experimental

2.1. Instrumentations

The extracted species of As from water samples were analysis by ET-AAS (Perkin Elmer model A Analyst 700, Norwalk, CT, USA), coupled with a graphite furnace HGA-400, autosampler AS-800, and deuterium lamp for background correction [35]. The experimental conditions of ET-AAS are electrodeless discharge lamp was used as light source for measurement of As species. Recommended current for analysis of As by electrodeless discharge lamp was set at 380 mA at wavelength at 193.7 nm, spectral bandwidth of 0.7 nm. Recommended furnace program for arsenic analysis have five steps: cooling, drying, ashing, atomization, cleaning set as [temperature (°C)/ramp time/hold time(s)/internal flow/gas type] for all steps such as [100/5/20/250/normal], [140/15/15/250/normal], [1300/10/20/250/Normal], [2300/0/5/0/normal], [2600/1/3/250/normal] respectively. The pH of the studied samples was measured by a pH meter (Sartorius professional, meter pp-15). FT-IR spectra of 4000–400 cm^{-1} (Thermo Electron Corporation, Nicolet avatar 5700) was used for the characterization of the prepared materials.

2.2. Standard solution and reagents

The stock solutions (1000 mg L^{-1}) of arsenate and arsenite were prepared in high purity deionised water by dissolving appropriate amounts of potassium arsenate monobasic (KH_2AsO_4) and arsenic oxide (As_2O_3) which were obtained from Merck (Darmstadt, Germany). Potassium permanganate (KMnO_4) was obtained from the Sigma-Aldrich. Linoleic acid was supplied from Fluka. Specific amount of acetic acid salt was dissolved by deionised water to prepared stock buffer solution and used for required pH control. HCl (purity 37%, Sp.gr:1.19) and HNO_3 (purity 65%, Sp.gr:1.41) were obtained from Merck (Darmstadt, Germany). Throughout the experimental work we used high purity deionised water taken from a Milli-Q[®] water system (ELGA Laboratory, Bucks, UK). Plastic bottles for fresh water sampling were dipped into 10% HNO_3 for one day, after one day it should be washing with deionised water, and then dried.

2.3. Synthesis of polystyrene-g-linoleic acid-g-polydimethyl siloxane

Linoleic acid was autoxidized by exposing to air oxygen according to the procedure described in the cited reference in order to prepare polymeric linoleic acid peroxide [36]. Polymeric linoleic acid peroxide can be used as a macroperoxide initiator in order to obtain block/graft copolymer [37]. Styrene polymerization was initiated by polymeric linoleic acid peroxide to obtain polystyrene-g-polylinoleic acid. Terminal carboxylic ends of the graft copolymer were reacted with polydimethyl siloxane with amine end groups according to the procedure described in the cited literature [38]. For the condensation reaction procedure, dissolved 1.1 g of poly(linoleic acid) peroxide macro initiator and 3.0 g of poly dimethyl siloxane in 10 mL toluene under the argon gas by flask with glass stopper. The temperature and time maintained for condensation reaction was at 95 °C for 5.0 h respectively. After the condensation reaction, the required polymer of polystyrene polydimethyl siloxane was precipitated because solvent partially evaporated. The mixture of above solution was filtered, separated the polystyrene polydimethyl siloxane material and dried in room

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