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An automated continuous homogeneous microextraction for the determination of selenium and arsenic by hydride generation atomic fluorescence spectrometry

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ABSTRACT

An automated continuous homogeneous microextraction approach based on a flow system has been developed and coupled with a hydride generation atomic fluorescence spectrometry system (HG-AFS). The developed approach was applied for the determination of trace arsenic and selenium in environmental water and liver samples. The nonanoic acid was investigated as a switchable hydrophilicity solvent (SHS) for homogeneous microextraction of As(III) and Se(IV) complexes with pyrrolidinedithiocarbamate (PDC). The procedure involved on-line mixing ammonium PDC (aqueous phase), sodium nonanoate (aqueous phase) and acid sample solution resulting in the formation of SHS (nonanoic acid) dispersed into the acid aqueous phase. By this continuous process, analytes complexes with PDC were formed and extracted into the fine SHS droplets followed by retention into a monolithic column packed with block of porous PTFE. Finally, the retained complexes were eluted with NaOH solution and delivered to the HG-AFS system. The limits of detection, calculated from a blank test based on 3σ, were 0.01 µg L^{-1} for both analytes.

1. Introduction

The widespread dissemination and improvement of modern highly efficient analytical instruments have led to the fact that at present it is necessary to use minimum volumes of a sample and regents. This, in turn, has led to the fact that in the literature more attention is paid to the miniaturization and automation of sample pretreatment procedures in order to reduce the waste generation and analysis time. A special place here is occupied by flow-based methods [1]. The flow methods allow to automate variety of sample pretreatment procedures and can be coupled with different analytical instruments [2].

Liquid-liquid extraction (LLE), that probably, is one of the most common sample pretreatment techniques, has its indisputable advantages such as simplicity, reliability and adaptability to a wide variety of sample types and analytes, as well as, compatibility with majority of analytical instruments. Nevertheless, LLE also has drawbacks and limitations in terms of time-consumption and frequent requirement of large amounts of hazardous organic solvents. Thus, aiming to improve the analytical performance of LLE various liquid-liquid microextraction techniques [3–5] have been developed and automated. One of them is an automated single-drop microextraction approach implemented onto continuous flow [6], sequential injection [7] and stepwise injection [8] systems. On the one hand the single-drop microextraction provides high enrichment factor (EF) values, on the other hand it is characterized by low extraction rate due to the contact surface between phases is extremely small. A dispersive liquid-liquid microextraction provides large contact surface between phases resulting in high extraction efficiency. The implementation of various dispersive liquid-liquid microextraction modes onto flow systems based on: microcolumn phase separation [9,10], in-syringe approach [11,12], coupling with flow-batch sequential injection [13] and stepwise injection [14,15] systems as well as magnetic stirring [16] has been reported. The automated dispersive liquid-liquid microextraction procedures are well described in a recent review [17] where their main fundamental principles, possibilities and limitations are presented.

Recently a homogeneous liquid-liquid microextraction (HLLME) approach based on the use of new generation of extraction solvents called switchable hydrophilicity solvents (SHSs) has been reported [18,19]. The HLLME involves the complete solubilisation of an extraction solvent in an aqueous sample phase making the contact surface

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between phases infinitely large resulting in high extraction efficiency. Jen et al. have presented the application of medium-chain fatty acid as SHS [20]. This medium-chain fatty acid can be switched between hydrophobic and hydrophilic forms by the adjustment of pH of the solution. The HLLME based on the use of medium-chain fatty acid as SHS has been automated [21]. The developed automated approach assumed generation of the fatty acid microdroplets and analyte extraction into a mixing chamber of a flow system. The automated HLLME procedure was coupled with a HPLC system with fluorescence detection and successfully used for the determination of ofloxacin in human urine samples. Nevertheless, the HLLME procedure was characterized by low EF values due to small ratio of organic and aqueous phase was obtained into the mixing chamber. It was shown that the organic phase volume less than 50 μL and aqueous phase volume higher than 1000 μL led to poor recovery [21].

In this study a novel approach for automation of homogeneous liquid-liquid microextraction was developed. The developed approach, a continuous homogeneous liquid-liquid microextraction (CHLLME), assumes a continuous mixing acid sample solution with aqueous solution of hydrophilic form of medium-chain fatty acid resulting in the formation of hydrophobic form of medium-chain fatty acid dispersed into the acid aqueous phase. By this continuous process, analytes are extracted into the fine medium-chain fatty acid droplets followed by retention into a monolithic column packed with block of porous PTFE and extract elution. To the best of our knowledge, this approach has not been presented in literature. Potentialities and limitations of the approach are discussed in relation to the hydride generation atomic fluorescence spectrometry (HG-AFS) determination of trace arsenic and selenium in environmental water and liver samples. The simplicity of operations, rapidity, low cost and relatively high enrichment factors can be mentioned as advantages of the developed CHLLME procedure.

2. Experimental

2.1. Reagents and solutions

All reagents used were of the highest purity available but at least of analytical grade. Ultra pure water from HLP5sp system (Hydrolab, Poland) was used. Working Se(IV) and As(III) standard solutions were prepared daily by dilution of the stock solutions of 1 g L^{-1} (Merk, Germany) with deionized water. Solution of ammonium pyrrolidine-dithiocarbamate (APDC) was prepared by dissolving of the reagent (Sigma, Germany) in deionized water. Solution of sodium nonanoate was prepared by dissolving of the reagent (Sigma, Germany) in deionized water. 3% $NaBH_4$ solution was prepared by dissolving of the reagent (Sigma, Germany) in 0.25 mol L^{-1} NaOH.

2.2. Samples

River water sample from Wisla and tap water were collected locally (Krakow, Poland). The standard reference materials (SRMs) of water (EnviroMAT Waste Water (EU-H-3) and EnviroMAT Ground Water (ES-H-2)) and liver (CRM185R Bovine liver) were used to check the accuracy of the developed procedure.

2.3. Sample preparation

Water samples were filtered using a filter membrane with a pore size of 0.45 μm (Sartorius, Goettingen, Germany). To reduce Se(VI) to Se(IV) and As(V) to As(III) 12.5 mL of water sample was mixed with 2.5 mL of 0.1 mol L^{-1} HCl, 2 mL of 1% KI and 2 mL of 0.2% L-ascorbic acid into 25 mL flask and diluted to 25 mL with deionized water [22].

For liver analysis the sample (0.5 g) was accurately weighed and transferred to a PTFE microwave digestion vessel containing 6 mL of 65% HNO_3. The vessel was placed in a microwave chamber of a Multiwave 3000 microwave system (Anton Paar, Austria) and run at 50% power (600 W) for 12 min. The sample was cooled for a few minutes. The digestion step was continued for 15 min at 50% power (600 W). The solution was cooled and transferred into 25 mL flask and then adjusted to pH 7 by 3 mol L^{-1} NaOH. To reduce Se(VI) to Se(IV) and As(V) to As(III) sample was mixed with 2.5 mL of 0.1 mol L^{-1} HCl, 2 mL of 1% KI and 2 mL of 0.2% L-ascorbic acid and diluted to 25 mL with deionized water [22].

2.4. Manifold and apparatus

The flow manifold consists of a two-positional eight-way injection valve (Perkin Elmer, USA), two peristaltic pumps with flow rate ranging from 0.5 to 5 mL min^{-1} (Minipuls 3, Gilson, France), monolithic column and communication tubes (PTFE, 0.8 mm i.d.). The monolithic column was made from a PTFE tube with an effective length of 20 mm and inner diameter of 5 mm. The column was filled with block porous PTFE. The PTFE powder (Fluoroplast-4) was sintered at a temperature of 380 °C for 5 h in metal form (20 × 50 × 50 mm) to prepare a block porous PTFE. Then the PTFE block was crushed in a blender, and the fraction size of PTFE granules from 0.5 to 0.9 mm was selected. A cylindrical metal form (height – 20 mm, diameter – 5 mm) was filled with prepared fraction and PTFE powder was re-sintered at a temperature of 380 °C for 2 h. The obtained block porous PTFE was gently removed from the cylindrical metal form and filled into the PTFE tube and the tube was closed with two cap screws equipped with two communication tubes (PTFE, 0.8 mm i.d.). Detailed scheme of block porous PTFE preparation and its surface morphology received by scanning electron microscope are presented in ESM Fig. 1 and ESM Fig. 2, respectively. The performance of the column was stable during all experiments.

The flow manifold was coupled to an atomic fluorescence spectrometer AFS 830 (Beijing Titan Instruments Co., China) equipped with a flow hydride-generation system. Hollow cathode lamps (Se-HCL and As-HCL) were operated at 100 mA. A special electronic adapter (KSP, Poland) was enabled to control all elements of the flow system. The HG-AFS system was operated automatically using a computer. A scanning electron Carl Zeiss Merlin microscope (Zeiss, Germany) was used for the study of porous PTFE morphology.

2.5. The automated CHLLME procedure

In the preconcentration step 1 (Fig. 1a), the pump 1 was stopped and the pump 2 was active. The solution of sample (5 mL min^{-1}) and mixture of 0.006 mol L^{-1}APDC (5 mL min^{-1}) and 0.006 mol L^{-1} sodium nonanoate (5 mL min^{-1}) were mixed and delivered into the monolithic column. At this time 0.25 mol L^{-1} NaOH (5 mL min^{-1}) was delivered to waste (2). The time of the preconcentration step was 300 s.

In the elution step 2 (Fig. 1b), the pump 1 was active and the position of the valve was changed. The solution of sample and mixture of APDC and sodium nonanoate were delivered to waste. Meanwhile 0.25 mol L^{-1} NaOH (2 mL min^{-1}, 15 s) was passed through the column followed by mixing with stream of 3% $NaBH_4$ (5 mL min^{-1}) and 3 mol L^{-1} HCl (5 mL min^{-1}), and transferred into the HG-AFS system. The signals were measured for both analytes at the same time and processed in the peaks area mode. Argon was exploited as the shielded gas and carrier gas at the flow rate of 800 and 300 mL min^{-1}, respectively. The atomization process occurred in the Ar flame at a temperature of 200 °C.

3. Results and discussion

3.1. Theoretical considerations

On the one hand, the sodium nonanoate is water-soluble salt and its homogeneous solution is observed in an aqueous phase. The nonanoate-ions behavior depends on pKa value. The pKa of nonanoic acid is 4.96 [23]. When the pH of an aqueous solution is less than pKa, nonanoate-

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