



Electrodeposition of gold nanoparticles onto an etched stainless steel wire followed by a self-assembled monolayer of octanedithiol as a fiber coating for selective solid-phase microextraction



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ARTICLE INFO

Article history:

Received 9 August 2014

Received in revised form 28 October 2014

Accepted 28 October 2014

Available online 3 November 2014

Keywords:

Au nanoparticles

Self-assembled monolayer

Ultraviolet filters

Solid-phase microextraction

High-performance liquid chromatography

ABSTRACT

In the present study, a novel approach for rapid electrodeposition on an etched stainless steel (SS) wire followed by self-assembled monolayer (SAM) was proposed for the fabrication of solid-phase microextraction (SPME) fiber. The etched SS wire offers a rough surface structure for subsequent electrochemical deposition of gold nanoparticles (AuNPs). As a result, uniform AuNPs coating was tightly attached to the etched SS wire substrate. After SAM of 1,8-octanedithiol onto AuNPs coating via Au-S bonding, a unique floccular structure with extremely large surface area was obtained for the fabricated fiber. The mercaptoethyl groups modified AuNPs coated etched SS fiber (C₈-S-AuNPs/SS) was then assessed for SPME of phthalate esters (PAEs), polychlorinated biphenyls (PCBs), chlorophenols (CPs), ultraviolet (UV) filters, polycyclic aromatic hydrocarbons (PAHs) and substituted anilines coupled to high-performance liquid chromatography with UV detection. This fiber exhibits higher extraction capability and better selectivity for some PCBs, CPs, UV filters and PAHs. Extraction conditions were investigated and optimized for SPME performance of UV filters. Under the optimized conditions, the developed method showed good linearity between 0.10 and 400 μg L⁻¹ with corresponding coefficients in the range of 0.9989–0.9998. The limits of detection ranged from 0.025 to 0.056 μg L⁻¹. The relative standard deviation for fiber-to-fiber reproducibility of five fabricated fibers was less than 9.4%. The developed method was successfully applied to the preconcentration and determination of trace UV filters from environmental water samples. Furthermore the fabrication of the C₈-S-AuNPs/SS fiber can be performed in a highly reproducible manner. This fabricated fiber exhibits good stability and long lifetime, and could be a potential alternative for the conventional fused silica fiber.

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1. Introduction

Solid-phase microextraction (SPME) is a miniaturized solvent-free sample preparation technique which integrates sampling, extraction, preconcentration and sample introduction in a single step [1]. Currently, SPME has gained its great popularity in a broad field of analysis including environmental [2], food [3,4], pharmaceutical [5] and biological [6] samples because of its simplicity, rapidity, sensitivity and easy quantification. Until now, commercially available SPME coatings include polydimethylsiloxane (PDMS), polyacrylate (PA) and some composites such

as PDMS/divinylbenzene (PDMS/DVB), carboxen/PDMS (CAR/PDMS), carbowax/DVB (CW/DVB) and CW/template resin (CW/TPR) [7]. However, these fibers are expensive in practical applications and also show some drawbacks such as fragility, bending of the needle, the stripping of coatings, and easily swelling in organic solvents which limits their widespread applications [8]. For these reasons, great efforts have been devoted to the development of metal-based fibers such as aluminum [9,10], zinc [11], gold [12,13], silver [14], platinum [15], titanium [16–18], nitinol alloy [19,20], copper [21,22] and stainless steel (SS) wire [8,23–27]. These metal wires are physically strong and can be handled with great convenience. Due to their quite different physicochemical properties from those of fused-silica fibers, there have been various preparation methods of the metal wire supported SPME fibers.

In recent years, special attention has been paid to coinage metals due to their relativistic effect which increases the mobilization of

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their electrons and facilitates the formation of chemical bond [28]. Organic molecules containing thiol (–SH) group can be bonded onto the surface of gold (Au) or silver (Ag) to form a self-assembled monolayer (SAM) [29]. This approach has proven to be able to create chemical Au sorbent [30] and modified Au or Ag coatings [31–33] in analytical and separation field. Feng et al. have prepared Au nanoparticles (AuNPs) coated SPME fiber by layer-by-layer SAM process for extraction and concentration of polycyclic aromatic hydrocarbons (PAHs) in aqueous solution [30]. The inherent chemical stability of Au makes the novel fiber show high stability toward acid, alkali and high temperature. However, the layer-by-layer SAM process was cumbersome and time-consuming although the AuNPs coated fiber had shown the excellent properties. Li et al. also prepared C₁₂–Ag wire as a SPME fiber for determination of benzophenone ultraviolet (UV) filters in river water in combination of SAM with sol–gel technology [33]. However, there was an inherent problem associated with gel shrinkage and cracking during drying with the increasing thickness of the coating.

Among the metal-based SPME fibers described, the SS wire was frequently used as a supporting substrate with high mechanical and chemical stability, moderate elasticity and low cost. Recently, a new two-step process involving chemically etching procedure of SS wires and their post-fabrication has proved very efficiently for the fast development of tightly attached coatings with unique structural characteristics [34,35]. According to this procedure, Du et al. rapidly prepared a robust Au submicron particles coated fiber by direct chemical deposition onto an etched SS wire for sensitive detection of PAHs [36]. These pretreated SS wires provided promising alternative substrates for subsequent fabrication of tightly attached and highly efficient extraction coatings because they exhibited good conductivity, strong rigidity and porous surface structure. Electrodeposition can offer a rigid control of uniformity and deposition rate for subsequent fabrication of nanostructured coatings. This technique was especially attractive for further deposition onto the substrates with complex shapes [37]. However, the electrodeposition of AuNPs coating on the etched SS wire has not been developed for subsequent fabrication of nanostructured coatings.

In this work, we described a new approach to rapid and uniform electrodeposition of AuNPs coating on the surface of the etched SS wire (AuNPs/SS) using cyclic voltammetry (CV) followed by a SAM of 1,8-octanedithiol (SH–C₈–SH) occurring uniquely on AuNPs coating. Extraction capability and selectivity of the 1,8-octanedithiol modified AuNPs/SS fiber (C₈–S–AuNPs/SS) was evaluated for the concentration and separation of phthalate esters (PAEs), polychlorinated biphenyls (PCBs), chlorophenols (CPs), ultraviolet (UV) filters, polycyclic aromatic hydrocarbons (PAHs) and substituted anilines coupled to high-performance liquid chromatography with UV detection (HPLC–UV). The microextraction conditions were investigated and optimized for the concentration and determination of UV filters. The SPME–HPLC–UV procedure with C₈–S–AuNPs/SS fiber was established to preconcentrate and determine UV filters in the environmental water samples. Furthermore the SPME performance of this novel C₈–S–AuNPs/SS fiber was compared with that of commercial PDMS and PA under the optimized conditions.

2. Experimental

2.1. Materials and reagents

Hydrofluoric acid (40%) was obtained from Shuangshuang Chemicals Co., Ltd, (Yantai, China). Chloroauric acid (HAuCl₄•4H₂O) was purchased from Shenyang Chemical Reagents Company (Shenyang, China). 1,8-Octanedithiol (HS–C₈–SH) was obtained from Acros (New Jersey, USA). Sodium chloride (NaCl) was

obtained from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). HPLC-grade methanol was purchased from Yuwang Chemical Company (Yucheng, China). Certified individual standards of 2-hydroxy-4-methoxybenzophenone (BP-3), 2-ethylhexyl 4-methoxycinnamate (EHMC), 2-ethylhexyl 4-(*N,N*-dimethylamino) benzoate (OD-PABA), dimethyl phthalate (DMP), diethyl phthalate (DEP), di-*n*-butyl phthalate (DBP), di-*n*-octyl phthalate (DOP), di-(2-ethylhexyl) phthalate (DEHP), 2-chlorophenol (2-CP), 2,4-dichlorophenol (2,4-CP), 2-(2,4-dichlorophenoxy)-5-chlorophenol (Triclosan), 2,4,4'-trichlorobiphenyl (PCB 28), 2,4',5'-trichlorobiphenyl (PCB 31), 2,3',4,4',5'-pentachlorobiphenyl (PCB 118), 2,2',4,4',5,5'-hexachlorobiphenyl (PCB 153), aniline, 4-nitroaniline and benzidine were purchased from AccuStandard (New Haven, CT, USA). 2-Ethylhexyl salicylate (EHS) was obtained from Dr. Ehrenstorfer (Augsburg, Germany). Certified individual standards of naphthalene (Nap), anthracene (Ant), phenanthrene (Phe), fluorene (Flu), fluoranthene (Fla) and 4-methylaniline were purchased from Aldrich (St. Louis, MO, USA). Diphenyl (Dip) was purchased from China Pharmaceutical Group (Shanghai, China). Individual standard stock solutions were prepared in methanol at a concentration of 1 g L⁻¹ for CPs and substituted anilines as well as 100 mg L⁻¹ for PAEs, PCBs, UV filters and PAHs, respectively, and stored at 4 °C in the refrigerator before use. All reagents were of analytical grade, unless otherwise stated. 0.45 μm micropore membrane of polyvinylidene fluoride was supplied by Xingya Purifying Material Factory (Shanghai, China).

2.2. Apparatus

A Waters 600E multi-solvent delivery system (Milford, MA, USA) equipped with a Waters 2487 dual λ absorbance detector and a Zorbax Eclipse Plus C₁₈ column (150 mm × 4.6 mm, 5 μm, Agilent, USA) was used for all separations. Data collection was obtained with a N2000 workstation (Zhejiang University, China). A SPME device was modified from a commercially available 2-μL HPLC microsyringe (Gaoge, Shanghai, China). A plunger wire (75 mm × 0.20 mm O.D.) inside a removable septum-piercing needle (30 mm × 0.35 mm I.D.) was used as a SS support. Desorption was performed in a commercially available SPME–HPLC interface (Supelco, PA, USA). The fiber coating was characterized by an Ultra Plus scanning electron microscope (Zeiss, Oberkochen, Germany) equipped with semi-quantitative microanalysis by energy dispersive X-ray spectroscopy (EDS). Electrochemical deposition of Au was performed on a CHI832D electrochemical analyzer (Chenhua, Shanghai, China) by CV. Ultrapure water was obtained from a Sudreli SDLA–B–X water purification system (Chongqing, China). The extraction was carried out in a DF-101S thermostated water bath with a magnetic stirring (Zhengzhou, China).

2.3. Fabrication of C₈–S–AuNPs/SS fiber

The tip of the SS (plunger) wire was firstly washed in an ultrasonic bath for 10 min with acetone to remove the organic pollutants and then rinsed with ultrapure water in an ultrasonic bath for 10 min. Subsequently the SS wire (1.5 cm long) was etched in hydrofluoric acid according to Ref. [36]. Afterwards, the etched SS wire was washed with ultrapure water and electrochemical deposition of AuNPs onto the surface of etched SS wire was performed by CV in electrolytic solution of HAuCl₄ with the three electrode system using the etched SS wire as a working electrode, a Pt rod as a counter electrode and a saturated calomel electrode as a reference electrode. The potential was applied from –0.2 V to –0.5 V for 20 CV cycles at a scan rate of 20 mV s⁻¹. Thereafter the fabricated AuNPs/SS fiber was rinsed with water and dried in air. Before SAM of 1,8-octanedithiol molecules, the AuNPs/SS fiber was ultrasonically cleaned in ethanol for 0.5 min and

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