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RESEARCH PAPER

Controllable Growth of Nanoporous Metal Oxide Composites on Nickel-Titanium Alloy Fibers for Solid-Phase Microextraction of Polycyclic Aromatic Hydrocarbons

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Abstract: Hydrothermal oxidation was used to prepare a nickel-titanium alloy (NiTi) wire as the solid-phase microextraction fiber. The experimental results demonstrated that a nanoporous nickel-titanium oxide composite coating was in situ grown on the surface of NiTi substrate by direct oxidation in aqueous H_2O_2 solution at 80 °C. The resulting oxide composite coating included more nickel and less titanium. The prepared NiTi fiber with Ni-rich oxide coating was used to extract typical aromatic compounds coupled with HPLC-UV and exhibited good extraction selectivity for polycyclic aromatic hydrocarbons (PAHs). The key factors affecting extraction efficiency of PAHs were examined. Under the optimized conditions, the calibration curves were linear in the range of 0.05–400 ng mL^{-1} with correlation coefficients over 0.999, and the limit detection of PAHs ranged from 0.026 ng mL^{-1} to 0.056 ng mL^{-1}. Furthermore, the relative standard deviations (RSDs) for intra-day and inter-day repeatability of the single fiber ranged from 4.8% to 6.2% and 5.4% to 6.5% for five replicate analyses of PAHs at the spiking level of 50 ng mL^{-1}, respectively. The RSDs for the fiber-to-fiber reproducibility of five fibers prepared in different batches ranged from 6.4% to 8.4%. The developed method was suitable for selective enrichment and detection of target PAHs in environmental water samples with relative recoveries from 89.9% to 108.5% and RSDs ≤ 8.1%. Moreover, this novel NiTi fiber was mechanically strong and chemically stable, and its preparation was precisely controllable.

Key Words: Nanoporous titanium dioxide; Nickel-titanium alloy fiber; Solid-phase microextraction; Polycyclic aromatic hydrocarbons

1 Introduction

Solid-phase microextraction (SPME) is a sample preparation method developed by Pawliszyn's group in 1990[1]. It is extensively applied to the enrichment and separation of trace analytes in complex matrices[2–4]. The durability of conventional SPME fibers depends on the strength of fiber substrates and the stability of the fiber coatings. However, most of commercially available fibers are fused-silica fibers and usually suffer from easy breakage, poor durability and expensiveness[5]. For these reasons, the development of novel fiber coatings with high extraction capacity, good selectivity and high stability is critical to SPME. The immobilization of novel nanomaterials onto metallic fiber substrates is an attractive topic in SPME applications[6]. In particular, the in situ fabrication of nanostructured coatings on metallic substrates is a new pathway for the preparation of stable SPME fibers[7–10].

Nickel-titanium shape memory alloy (NiTi), the equiatomic nickel and titanium alloy, has some additional interesting characteristics such as shape memory effect, superelasticity, corrosion resistance and biocompatibility[11]. Pawliszyn et

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al.[12] first introduced a new generation of superelastic SPME fiber assemblies for potential SPME. Thereafter, the NiTi fiber electrolytically coated with zirconium oxide was investigated for the extraction of halophenols in water samples[13]. A single NiTi fiber could be used up to 500 times. Furthermore, Carasek *et al*[14] coated poly(ethylene glycol) on the NiTi wires through sol-gel technology for the extraction of benzene homologues from aqueous samples. Azenha *et al*[15] also fabricated functional silica particles on a NiTi support through UV-curable sol-gel technique for the enrichment and separation of benzaldehyde, acetophenone and dimethylphenol in aqueous samples. Recently, the in situ growth of the composite oxide nanosheets[16] and nanotubes[17] on the NiTi fiber substrates was achieved by hydrothermal and electrochemical treatment, respectively. These nanostructured coatings offered much larger surface area and excellent extraction performance. A single fiber was capable of accomplishing all of the analyses in the entire sample sequence.

In this work, controllable growth of nanoporous nickel and titanium oxide composite (NiO/TiO_2) coating on the NiTi wires was performed by hydrothermal oxidation in hydrogen peroxide. Surface morphology and elemental composition were characterized. The extraction performance of the prepared NiTi fiber was investigated using ultraviolet (UV) filters, phthalate acid esters (PAEs), polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) coupled to HPLC-UV and compared with that of polydimethylsiloxane (PDMS) and polyacrylate (PA) fibers. Finally, the developed method was successfully applied to the enrichment, separation and determination of the target PAHs in river water and wastewater samples.

2 Experimental

2.1 Instruments and reagents

The physical characteristics of the prepared fiber were characterized by an Ultra Plus microscope (Zeiss, Oberkochen, Germany) with an energy dispersive X-ray spectrometer (Oxford, UK). Chromatographic separation was performed on a Waters 600E multi-solvent delivery system (Milford, MA, USA) equipped with a Waters 2487 dual λ absorbance detector and an Agilent zorbax Eclipse Plus C_{18} column (150 mm × 4.6 mm, 5 μm). The data collection was carried out with a N2000 chromatographic workstation (Zhejiang University, China). SPME was carried out in a temperature-controlled S10-3 water bath with a magnetic stirrer (Shanghai, China). Desorption was performed in a commercially available SPME-HPLC interface (Supelco, PA, USA). A KQ-100B ultrasonic cleaner was used to wash the NiTi fibers (Kunshan, Jiangsu, China). Ultrapure water was obtained from a Sudreli SDLA-B-X water purification system (Chongqing, China).

HPLC-grade methanol was purchased from Yuwang Chemical Company (Yucheng, Shandong, China). Hydrogen peroxide (H_2O_2) was supplied by Shuangshuang Chemical Company (Yantai, Shandong, China). Certified individual standards of naphthalene (Nap), phenanthrene (Phe), fluoranthene (Fla), pyrene (Pyr) and benzo[a]pyrene (B[a]P) were purchased from Aldrich (St. Louis, MO, USA). Certified individual standards of dimethyl phthalate (DMP), diethyl phthalate (DEP), dibutyl phthalate (DBP), dioctyl phthalate (DOP) and di-(2-ethylhexyl) phthalate (DEHP), benzophenone (BP), 2-hydroxy-4-methoxy-benzophenone (BP-3), 2-ethylhexyl 4-(*N,N*-dimethylamino) benzoate (OD-PABA), 2-ethylhexyl-4-methoxycinnamate (EHMC), 2,4,4'-trichlorobiphenyl (PCB-28), 2,4',5-trichlorobiphenyl (PCB-31), 2,3',4,4',5-pentachlorobiphenyl (PCB-118) and 2,2',4,4',5,5'-hexachlorobiphenyl (PCB-153) were supplied by AccuStandard (New Haven, CT, USA). 2-Ethylhexyl salicylate (EHS) was purchased from Dr. Ehrenstorfer (Augsburg, Germany). A NiTi wire (0.25 mm O.D.) was obtained from Alfa Aesar (Ward Hill, MA, USA). PDMS (100 μm thickness) and PA (85 μm thickness) fibers were obtained from Supelco (Bellefonte, PA, USA). All other reagents were of analytical grade.

2.2 Preparation of NiTi SPME fiber

The original NiTi wire was ultrasonically cleaned with acetone and ultrapure water, respectively. Thereafter, it was soaked in 15 mL of H_2O_2 at 80 °C for 30 min. After ultrasonic cleaning, the oxidized end was dipped into freshly prepared H_2O_2 solution for the second oxidation under the same conditions. Finally, the oxidized NiTi wire was rinsed with ultrapure water and dried in air.

2.3 SPME-HPLC procedure

The pH value of sample was adjusted with phosphate buffer and the ionic strength was controlled by sodium chloride (NaCl). In a typical SPME procedure, the prepared fiber was immersed into a 15-mL sample solution in a 20-mL glass vial sealed with a Teflon septum and a PEEK cap. After extraction, the NiTi fiber was withdrawn from the vial and immediately introduced into the SPME-HPLC interface for static desorption in mobile phase. Methanol and water of 85:15, 87:13, 75:25 and 85:15 (*V/V*) were used as mobile phases at a flow rate of 1 mL min^{-1} for HPLC analysis of UV filters, PCBs, PAEs and PAHs, respectively. Corresponding UV detection wavelength was set at 310, 254, 282 and 254 nm. Prior to each extraction, the fiber was cleaned with methanol and ultrapure water for 15 min and 5 min to eliminate possible carryover, respectively.

3 Results and discussion

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