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Development of a cheap and accessible carbon fibers-in-poly(ether ether ketone) tube with high stability for online in-tube solid-phase microextraction



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ABSTRACT

Carbon fibers (CFs) are one kind of important industrial materials that can be obtained commercially at low price. Based on the high extraction efficiency of carbon sorbents, a cheap and accessible carbon fibers-in-poly(ether ether ketone) (PEEK) tube was developed for online in-tube solid-phase microextraction (SPME) method. Coupled to high performance liquid chromatography (HPLC), the CFs-in-tube SPME was applied to analyze eight polycyclic aromatic hydrocarbons (PAHs) in environmental aqueous samples. Extraction conditions (sampling rate, extraction time, methanol content) and desorption time were investigated for optimization of conditions. Under the optimum conditions, the CFs-in-tube SPME-HPLC method provided high extraction efficiency with enrichment factors up to 1748. Good linearity ($0.05\text{--}50\ \mu\text{g L}^{-1}$, $0.5\text{--}50\ \mu\text{g L}^{-1}$) and low detection limits ($0.01\text{--}0.1\ \mu\text{g L}^{-1}$) were also obtained. The online analysis method was finally applied to determine several model PAHs analytes in real environmental aqueous samples. Some target analytes were detected and relative recoveries were in the range of 92.3–111%. Due to natural chemical stability of carbon fibers and PEEK tube, the CFs-in-tube device exhibited high resistance to organic solvent, acid and alkaline conditions.

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1. Introduction

It is well known that sample preparation is the most time-consuming stage of the analytical process [1]. Therefore, significant efforts have been made to develop eco-friendly sample preparation techniques with high simplification, automation, miniaturization and low cost [2]. Microextraction techniques, represented by solid-phase microextraction (SPME) [3,4], stir bar sorptive extraction [5] and dispersive liquid–liquid microextraction [6,7], have appeared in line with these main trends.

Fiber SPME and in-tube SPME are two common forms of SPME. In-tube SPME is an automated version, in which an open tubular capillary with an inner surface coating is used as the extraction device [8]. Compared with fiber SPME, in-tube SPME is easier to be automated with high performance liquid chromatography (HPLC), and greatly improves sorptive capacity by the most appropriate length and thickness of the sorbent tube. Besides the commercially available gas chromatography columns [8–10], silica capillaries modified with various coatings and monolithic sorbents via the

abundant active silanol groups have been used in in-tube SPME [11–14]. To overcome the breakable drawback of silica capillary, poly(ether ether ketone) (PEEK) tubes modified with graphene [15], polydopamine [16], metal-organic frameworks [17] and polymer monolith [18] have also been developed in recent years. However, the stability of the sorbent coating was greatly limited due to the chemical inertness of PEEK tube.

Fiber-in-tube SPME is developed by packing fiber into tube, which enhances the preconcentration effect and improves the symmetry of peaks by reducing the dead volume in tube. Zylon fiber [19], optical fiber [20], carbon nanotubes coated silica fiber [21], molecularly imprinted silica fiber [22] and copper fiber [23] have been packed into tube for in-tube SPME of various organic compounds.

Carbon-based sorbents showed non-selective high adsorbability, thermal and chemical stability, and long-term reusability. Therefore, carbon nanomaterials (carbon nanotubes [24], graphene [25,26], carbon nanoparticles [27], fullerene [28], etc.) and graphite [29] have been widely used in sample preparations. Carbon fibers (CFs) are a threadlike carbon material with a size about 5–10 μm in diameter. Properties of high stiffness, high tensile strength, low weight, high chemical resistance, high

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temperature tolerance and low thermal expansion make it widely used in aerospace, shipbuilding, construction, sporting goods and military applications [30,31]. It can be obtained commercially at low price. The atomic structure of CFs is similar to that of graphite, consisting of sheets of carbon atoms arranged in a regular hexagonal pattern. The graphite structure combined with the high chemical resistance, high temperature tolerance, low thermal expansion properties as well as the low price make CFs a promising sorbent material for sample preparation.

In this work, CFs were firstly packed into a PEEK tube to develop a CFs-in-tube SPME method. Surface of carbon materials can be increased by oxidation treatment with nitric acid [32,33], that could improve their adsorptivity. CFs were treated by nitric acid to increase extraction capacity. Based on the high extraction efficiency of graphite sorbent for polycyclic aromatic hydrocarbons (PAHs) [34–38], eight PAHs were selected as model analytes to test the extraction performance of the CFs-in-tube SPME method. The as-established method was finally applied to detect the target PAHs in real water samples.

2. Experimental

2.1. Materials and reagents

Poly(ether ether ketone) (PEEK) tube (1.59 mm o.d., 0.50 mm i.d.) was purchased from Dalian Elite analytical instruments Co., Ltd. (Dalian, China). Carbon fibers (6 μm d.) were obtained from Japan Toho Chemical Industry Co., Ltd. The stainless steel wire (304, 0.18 mm d.) was purchased from the Yixing Shenglong Metal wire Net. Co. (Jiangsu, China). Naphthalene (Nap), acenaphthylene (Ace), acenaphthene (Ana), fluorene (Flu), phenanthrene (Phe), anthracene (Ant), fluoranthene (FIA) and pyrene (Pyr) were purchased from Shanghai Jingchun Industry Co. (Shanghai, China). Acetonitrile was chromatographic grade and obtained from Tedia Company Inc. (USA). Nitric acid (68%) was obtained from Kangde Chemical Reagent Co. (Laiyang, China). All above reagents were analytical grade.

2.2. Apparatus

All chromatographic tests were performed on an Agilent 1260 Infinity series (Santa Clara, CA, USA) equipped with a Zorbax C18 column (250 \times 4.6 mm i.d., 5 μm), a 20 μL sample loop and a diode array detector (DAD). Multiple wavelengths were set at 220 nm for Nap, at 225 nm for Ace and Flu, at 230 nm for FIA and Pyr, at 250 nm for Phe and Ant, at 260 nm for Ana. Sample solutions were flowed into extraction tube with a P1201 HPLC pump of Dalian Elite analytical instruments Co., Ltd. (Dalian, China). Acetonitrile and water filtered with a 0.45 μm membrane were used as the mobile phase with a flow rate at 1 mL min^{-1} . Gradient elution (0–10 min 70% acetonitrile–30% water; 10–20 min increased into 100% acetonitrile, 20 min stop) was used.

Microstructure of CFs was characterized by a field-emission scanning electron microscope (SEM, SUPRATM55, Carl Zeiss, AG, Germany). Raman spectrum was performed with a Raman microscope (633 nm laser excitation, JY Horiba Labram HR800 Raman microscope, UK). The X-ray Photoelectron Spectroscopy (XPS) spectrum was recorded by a Kratos Axis Ultra DLD spectrometer (Shimadzu Corp., Japan) employing a monochromated Al K α X-ray source.

2.3. Preparation of sample solutions

Stock solution of PAHs was prepared at 10 mg L^{-1} in methanol solvent. Working solutions were prepared daily by diluting the

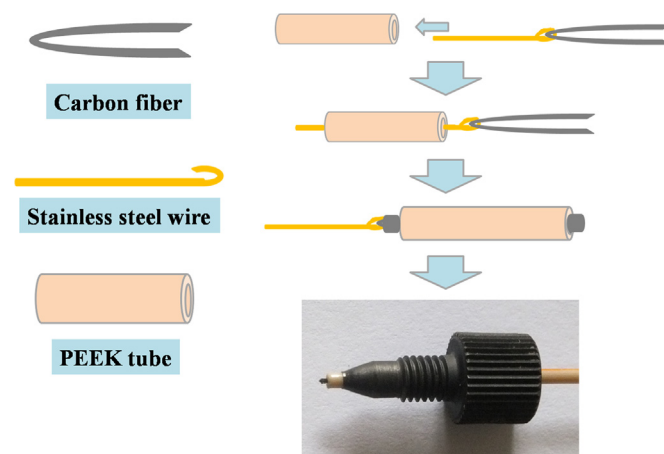


Fig. 1. The image of carbon fibers packed in PEEK tube as in-tube SPME device.

stock solution in distilled water to 10 $\mu\text{g L}^{-1}$. Rain water and groundwater of chemical industry park were collected locally as real samples. Before analysis, water samples were filtered with a 0.45 μm membrane.

2.4. Preparation of carbon fibers-in-tube extraction device

Carbon fibers were firstly treated with nitric acid solution (7.2 mol L^{-1}). The installation of carbon fibers-in-tube SPME device was shown in Fig. 1. 40 mg of CFs (30 cm) were packed into a PEEK tube (30 cm, 59 μL inner volume) by traction. To reduce the dead volume of carbon fibers-in-tube, a stainless steel wire (30 cm, 0.18 mm d.) was put into the PEEK tube to occupy the invalidate space.

2.5. Online carbon fibers-in-tube SPME-HPLC

Carbon fibers-in-tube extraction device was directly connected with the HPLC equipment by replacing the sample loop of six-port injection valve (see Supplementary material Fig. S1). Online SPME-HPLC analysis consisted of extraction and desorption procedures. In extraction procedure (Fig. S1a), the six-port valve was set at load mode. 30 mL of sample solution flowed through carbon fibers-in-tube at 1.00 mL min^{-1} by a sample pump, and HPLC mobile phase consisting of acetonitrile–water flowed through HPLC column directly. In desorption procedure (Fig. S1b), the six-port valve was switched into injection mode. HPLC mobile phase flowed through the extraction tube, and the analytes were desorbed from carbon fibers-in-tube and eluted into HPLC column for separation and further detection by DAD detector.

3. Results and discussion

3.1. Preparation of carbon fibers-in-tube extraction device

Extraction capacity of in-tube SPME depended on the volume of the sorbent materials. Amount of CFs that can be packed into the PEEK tube were maximized to fill up the tube upon convenient handling. It was set at 40 mg in this work. Oxidative treatment with nitric acid can produce active sites by forming carbon–oxygen functional groups on CFs, but oxidation also increases the surface roughness and surface area by etching [32,33]. Nitric acid oxidation also caused the decrease of tensile strength [32]. Before packing, CFs were firstly treated with nitric acid to obtain higher extraction capacity. The treatment time was investigated from 4 to 12 h. As shown in Fig. 2, changes of peak areas are not obvious

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