



Etched poly(ether ether ketone) jacket stir bar with detachable dumbbell-shaped structure for stir bar sorptive extraction

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ABSTRACT

Development of stir bar sorptive extraction (SBSE) device with high stability and extraction efficiency is critical and challenging by date. In this work, etched poly(ether ether ketone) (PEEK) tube with high mechanical strength and large specific surface area was used as jacket for SBSE device. By etching with concentrated sulfuric acid, the smooth outer surface of PEEK become porous with plenty of micro holes, which was beneficial for coating of sorbents and significantly improved the extraction performance. After functionalized by bio-polydopamine method, strong hydrophobic p-naphtholbenzein molecular was immobilized onto the chemical resistant PEEK surface (PNB@E-PEEK) as stationary phase. We also firstly developed a simple detachable dumbbell-shaped structure for improving the workability of PEEK jacket stir bar. The dumbbell-shaped construction can eliminate the friction between stir bar and container, and the design of detachable structure make elution can be accomplished easier with small amount of organic solvent. It was interesting that the developed detachable dumbbell-shaped PNB@E-PEEK stir bar showed exceptional stability and extraction efficiency for SBSE enrichment of multiple analytes including several Sudan dyes, triazines, polycyclic aromatic hydrocarbons (PAHs), alkaloids and flavonoid. By coupling with high performance liquid chromatography-ultraviolet detection (HPLC-UV), PNB@E-PEEK stir bar based SBSE-HPLC-UV method was applied for the analysis of common Sudan dye pollutants. The method showed low limits of detection (0.02–0.03 ng/mL), good linearity ($R^2 \geq 0.9979$) and good reproducibility (relative standard deviation $\leq 7.96\%$). It has been successfully applied to determine three dye pollutants in tap and lake water.

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1. Introduction

Sample preparation is one of the most important and time-consuming steps in analytical procedures [1]. The presence of trace-level concentration of target compounds in complex matrices such as environmental, food, pharmaceutical and biological samples have been recognized as the original stimulus for development of sample preparation techniques [2]. For this purpose, lots of sample preparation methods including liquid-liquid extraction (LLE) [3], solid-phase extraction (SPE) [4], liquid-phase microextraction (LPME) [5], dispersive solid-phase extraction (DSPE) [6], stir-bar sorptive extraction (SBSE) *etc.* were created and widely applied [7]. Development of new extraction devices and adsorption materials are the most critical parts in sample preparation research [8].

SBSE was first introduced in 1999 as high efficient sample preparation method for extraction and enrichment of organic analytes from aqueous samples [9,10]. To date, this technique has been widely applied in the analysis of food [11], environmental [12], pharmaceutical and biological samples [13,14]. For analysis of liquid samples, commercial SBSE devices are often constituted by three major parts, including a glass jacket, a magnetic stir bars in the jacket and stationary phase coated on the jacket [10]. The glass jacket can prevent corrosion of metal bars and provide plenty of active silanol groups for modification of sorbents. However, the glass material is fragile and can be broken by careless crash or under vigorous ultrasonication [15]. For decreasing the friction between stir bar and the container, a dumbbell-shaped glass stir bar was prepared by melting the glass of two end [16]. Ochiai *et al.* developed a SBSE method with two stir bar, the stable one was stirring at the bottom, and the unstable one was attached on inner side wall of the vial, but this design is relatively complex and inconvenient [17]. In addition, a suspended stir bar was prepared by coating polyethylene hollow cylindrical tubes with adhesive films. So during the sampling, the stir bar was suspended in the top of the

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solution, and another stir bar was at the bottom for stirring [18]. Zhang et al. firstly introduced a jacket free stainless steel stir bar to solve this problem [15,19]. In this device, the sorbents were directly immobilized onto a stainless rod for SBSE extraction. However, the metal bar would be corroded after long term use in aqueous solution especially in acidic condition. In addition, due to the smooth outer surface of glass jacket and jacket-free stir bar, the specific surface area is relatively small, which limits the amounts of coated sorbents and adsorption rate.

PEEK is a kind of plastic polymer, which possesses advantages like high chemical and physical strength, high-pressure tolerance, good flexibility and easy cutting. Nowadays, PEEK material has been widely used in analytical instrument like HPLC and gas chromatography (GC) system due to its good properties [20]. Therefore, PEEK can be desirable jacket material that can solve the instability problem of existing SBSE devices. However, two major problems limited its application as jacket material for stir bar. On the one hand, it is difficult to immobilize extraction sorbents onto PEEK jacket due to the high chemical resistant of PEEK material. On the other hand, the outer surface of PEEK tube is smooth that difficult to be coated with stationary phase, and small specific surface area is also not good for extraction [21]. In recent years, mussel-inspired polydopamine modification method was introduced for functionalization of chemical resistant surface [22,23]. The functionalization of different materials including poly(tetrafluoroethylene) (PTFE) [24], polypropylene (PP) [25], PEEK [26] and stainless steel (SS) was realized by our group [15]. Plenty of active groups including amino and hydroxyl groups on polydopamine layer can be a good platform for the secondary modification. Therefore, by employing bio-polydopamine modification method, the chemical resistant problem of PEEK material can be well solved and multiple extraction sorbents can be immobilized.

PEEK is a kind of semicrystalline thermoplastic, which composed of crystalline phase and amorphous phase [27,28]. It was interested that in crystalline phase, molecules arranged regularly and packed tightly, the interaction force between molecular was strong. However, in amorphous phase, the molecular packing is loose and interaction force between them is weak. Because this unique chemical composition, concentrated sulfuric acid with strong causticity can easily penetrate into amorphous phase and dissolve it, but was difficult to penetrate into crystalline phase. Therefore, if the etching time is well controlled, amorphous region will be dissolved while crystalline phase retains as framework structure [29]. So a porous layer with lots of micro holes can be obtained on the surface. The rough surface may not only benefit the modification of extraction sorbents, but also improve the specific area, which will improve the extraction capacity and efficiency.

In this work, a facile detachable dumbbell-shaped stir bar with p-naphtholbenzein (PNB) modified etched PEEK jacket was developed and applied in SBSE. After etching by concentrated sulfuric acid, functionalization by polydopamine, and immobilization with hydrophobic p-naphtholbenzein molecular, the p-naphtholbenzein modified etched PEEK (PNB@E-PEEK) tube with porous surface layer was obtained [30]. Two lollipop-shaped stainless steel needles prepared by burning polypropylene at one end were inserted into the PEEK tube for construction of a detachable dumbbell-shaped stir bar. The dumbbell-shape structure can apparently reduce friction between sorbents and the bottom of beaker, which improved the stability of SBSE device. After sampling, the lollipop-shaped stainless steel needles can be easily moved for next elution step. The feature of detachable structure help elution can be finished in a pipette tip with less amounts of elution solvent. Extraction performance of the developed SBSE device was investigated by enrichment of three common dye pollutants including Sudan I, Sudan II and Sudan G [31]. By coupling with HPLC-UV, the SBSE-HPLC-UV method was used to analysis of three Sudan dyes

in environment samples. The whole preparation steps are simple, convenient and economical, which can be homemade and commercialized easily. To the best of our knowledge, this is the first time that detachable dumbbell-shaped structure and porous PEEK jacket were developed and applied in SBSE field.

2. Experimental

2.1. Instrumentation

A Shimadzu HPLC system (Tokyo, Japan) consisted of two Shimadzu 20A pumps, a 20A UV detector, a 20A₃ degasser and a thermostat controlled column compartment was used for separation and detection. Data collection was performed on Shimadzu LC Solution software. The detection wavelength was set at 478 nm for Sudan dyes and column oven temperature was 30 °C. Chromatography separation was completed by a C-18 column (250 mm × 4.6 mm i.d.) with 5 μm particle size from Shimadzu GL science (Tokyo, Japan). Mobile phase consisting of acetonitrile/H₂O (0.9/0.1 mL/min) was used for separation. Five automatic stirrers obtained from Shanghai Sile Instrument Company (Shanghai, China) were used for sampling. FT-IR spectra was investigated by a Thermo Nexus 470 FT-IR system (MA, USA). The surface morphology of bare and modified PEEK jacket was observed by a Carl Zeiss Ultra Plus Field Emission scanning electro microscope (Carl Zeiss, Germany) at an accelerating voltage of 5 kV. PEEK tubes (1/16 inch o.d., 750 μm i.d.) was obtained from VICI AG International (Switzerland). The stainless steel needle was purchased from a local supermarket.

2.2. Chemicals

Sudan I, Sudan II and Sudan G were purchased from Aladdin (Shanghai, China). Bavachinin were obtained from Shanghai Shunbo Bio-engineering Technology (Shanghai, China). Propyl paraben, palmartine and berberine were purchased from Aladdin (Shanghai, China). Three triazines including prometon, ametryn and prometryn were purchased from J&K Scientific (Beijing, China). Four PAHs standards including benz[a]anthracene, benzo[a]fluorathene, benzo[a]pyrene and dibenz[a,h]anthracene, and dopamine hydrochloride were supplied by Sigma-Aldrich (MO, USA). Sulfuric acid was supplied by Wuhan Chemical Reagent Factory (Wuhan, China). p-Naphtholbenzein was obtained from Aladdin (Shanghai, China). All above analytes were analytical reagent grade. Acetonitrile (HPLC grade) was purchased from Tedia (OH, USA). Deionized water was purified by a Milli-Q system (MA, USA). All mobile phase was filtered by 0.45 μm membranes and degassed before using.

2.3. Modification of the PNB@E-PEEK jacket

A PEEK tube of 3 cm was cut and washed by methanol and acetone thoroughly. For etching, the PEEK tube was immersed into concentrated sulfuric acid for 60 s, and then washed with water and dried in an oven for 2 h. After that, 10 mg dopamine was dissolved in 10 mL Tris-HCl buffer (10 mM, pH 8.5), the etched PEEK tube was dipped into dopamine solution and put in the room temperature for 4 h. After washing with methanol and drying in an oven (60 °C), the polydopamine modified etched PEEK was obtained. The immobilization of p-naphtholbenzein was realized by one-step reaction. Polydopamine modified PEEK tube was immersed into 1.5 mg/mL p-naphtholbenzein solution (dissolved in ethanol) and reacted at 80 °C for 12 h in an oil bath. After washed with methanol and dried in oven (60 °C), the PNB@E-PEEK was obtained.

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