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Electrochemical preparation of composite polyaniline coating and its application in the determination of bisphenol A, 4-*n*-nonylphenol, 4-*tert*-octylphenol using direct solid phase microextraction coupled with high performance liquid chromatography

For SPME-HPLC, metal wires with better mechanical strength are preferred over the fused silica fibers. In this article, a novel composite polyaniline (CPANI) doped with PEG and polydimethylsiloxane coating (CPANI fiber) was prepared on a stainless steel wire by a three-electrode system: the fiber was used as the work electrode, a calomel electrode and a platinum electrode were used as the reference and the counter electrodes, respectively. To evaluate the new CPANI coating, the coating was used to extract three kinds of phenols (bisphenol A, 4-*n*-nonylphenol, and 4-*tert*-octylphenol) in water samples by direct-SPME mode and then desorbed in commercial SPME-HPLC interface to separation. The extraction procedure was also optimized. Five real water samples were investigated. Good recoveries were gained when environmental samples were analyzed.

Key Words: Composite polyaniline (CPANI); Electroplating method; Fiber preparation; Metal wires; Solid phase microextraction

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1 Introduction

SPME (solid phase microextraction) has obtained widespread acceptance in many areas for its simple, rapid, and solventless properties since Pawliszyn *et al.* [1, 2] introduced it in 1989. To date, several commercial and custom-made SPME fibers were applied to the efficient extraction of organic pollutants from different matrices at trace levels [3]. Most fiber coatings were developed to extract volatile and semivolatile organic compounds by coupling SPME with GC. For example, polyaniline has been prepared on the stainless steel wire [4] and platinum wire [5] by electrochemical polymerization to extract aromatic amines and phenols. For nonvolatile compounds, some kinds of custom-made capillaries were used as in-tube SPME coupled with HPLC. Pawliszyn *et al.* [6–10] developed polypyrrole (PPY) and poly-*N*-phenylpyrrole coating capillary by chemical polymerization or electrochemical method to extract anions, β-blockers, and organoarsenic compounds from an aqueous sample. Moreover, a new method, electrochemically aided SPME, was used to extract anions and cations with several kinds of fibers coated with conducting polymer [11–15]. Little work was done for the development of new fiber coatings to extract less volatile and more polar compounds by direct SPME coupled with HPLC. There might be two main difficulties limiting the wide application of SPME-HPLC. One is the fragility of the fiber. The SPME-HPLC interface required great care to be paid when SPME fiber is introduced into the desorption chamber. The other is the absence of suitable stationary phase coated on fibers that not only has high extraction ability for analysis but also is stable in solutions of various matrices.

Conducting polymers have attracted a considerable attention in the past 20 years since the discovery of conducting polyacetylene by Shirakawa *et al.* [16]. The widely used conducting polymers are mainly of three types including polypyrrole, polythiophene, and polyaniline [17]. Among various synthetic methods for conducting polymers, the electrochemical polymerization played an important role because the electrochemical approach has the advantage of one-step production of conducting polymer films onto a metal electrode surface. Based on the advantage of their chemical and physical characteristics, conducting polymers have also attracted some analysts' attention [18]. Besides their application in SPME described above, the conducting polymers were also used as stationary phases in HPLC [19, 20] and SPE [21–25].

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Our goal in this work was to develop a new polymeric coating on the surface of stainless steel wire that can be used in SPME-HPLC system. Polyaniline, which was doped with poly(ethylene glycol) (PEG) and polydimethylsiloxane (PDMS), was coated on the stainless steel wire by electrochemical method. To evaluate the new coating, some polar and less volatile organic compounds such as bisphenol A, 4-*n*-nonylphenol, and 4-*tert*-octylphenol were analyzed, because it is widely regarded that they may disrupt the endocrine functions of animals and humans through mimicking or inhibiting the action of hormones [26–28].

2 Experimental

2.1 Apparatus and reagents

Perchloric acid was guarantee-grade reagent from Tianjin Dongfang Chemical factory (Tianjin, China). Aniline was analytical reagent grade from Beijing Chemicals Reagent Factory (Beijing, China). PEG ($M = 200$) was purchased from Tianjin Tiantai Chemicals Corporation (Tianjin, China). PDMS was equivalent to Silicone OV-101 (Shimadzu, Japan). The above four reagents composed the electrolyte for preparation of the coating. Water purified by a Milli-Q system was used throughout the experiments.

The other reagents were all of analytical reagent grade, 4-*n*-nonylphenol and 4-*tert*-octylphenol were purchased from Tokyo Kasei Kogyo, Japan, and bisphenol A was obtained from Acros Organics (NJ, USA). Standard stock solutions (40 mg/mL) containing these compounds were prepared by dissolving an appropriate amount of these compounds in methanol. Working solutions were prepared daily by an appropriate dilution of the stock solutions with pure water. LC-grade methanol and ACN were purchased from Acros Organics. Sodium hydroxide and hydrochloric acid were both guarantee-grade reagents (Beijing Chemicals Corporation, Beijing, China).

The HPLC equipment included an Agilent 1100 series Iso-Pump and an Agilent 1100 series fluorescence detector (FLD, Agilent Technologies, DE, USA). The separations were performed on an Agilent Zorbax Eclipse XDB-C8 column (150×4.6 mm; particle size, 5 μm). The mobile phase was a mixture of ACN and water (75 : 25, v/v), and a flow rate of 0.2 mL/min was selected during the first 2 min and then increased to 1 mL/min. For all compounds of interest, the fluorescence detector settings were as follows: 220 nm excitation and 315 nm emission. A PC equipped with an Agilent ChemStation program for LC systems was used to acquire and process chromatographic data. Peak area was used as the analytical measurement.

A manual 57331 type SPME fiber holder and an SPME-HPLC interface were purchased from Supelco (Bellefonte, PA, USA). The SPME-HPLC interface consisted of a six-port Rheodyne valve (Supelco) and a 60 μL desorption chamber, which replaced the injection loop in the HPLC system.

For a comparison study, a 50 μm CW/TPR (Carbowax/Templated Resin) fiber from Supelco was used. Before first use, the fiber was conditioned with the mobile phase until a stable baseline was obtained.

A magnetic stirrer and heater (Huifeng Electrical Instrument Factory, Shanghai, China) equipped with a 1/2″ L × 1/8″ Fisher Brand Octagonal Stirring and a TB-85 Thermo Bath (Shimadzu, Japan) were used for agitation and keeping constant temperature, respectively.

2.2 Preparation of SPME fiber

A lab-made SPME fiber was prepared by electroplating method. The process of preparation of doping polyaniline resembled the process of polyaniline except the different electrolytes [9]. Briefly, a CHI Electrochemstation (CHI Instruments, TX, USA) was used to control the electroplate process. Electrochemical polymerization was performed by a three-electrode system. A polished stainless steel wire for SPME was used as the working electrode. A calomel electrode and a platinum electrode were used as the reference electrode and the counter electrode, respectively. The electrolyte was composed of 0.4 mol · L^{-1} $HClO_4$ solution containing 0.1 mol · L^{-1} aniline monomer (redistilled before use) and 0.4 mol · L^{-1} PEG-200. A constant deposition potential (1.1 V) was controlled by a CHI Electrochemstation (CHI Instruments) and maintained for 4 h. This electroplate process was repeated three times, and in the fourth electroplate process, 0.4 mol · L^{-1} PDMS was added. During the electrochemical polymerization, a black polymer film was formed on the surface of the steel wire. It was subsequently washed with de-ionized water, then with methanol or acetone for 3 min and dried under nitrogen protection at room temperature. Finally, it was rinsed in the desorption chamber of the SPME-HPLC interface until a smooth baseline was observed.

2.3 SEM and IR experiment

The surface of fibers was studied by JEOL SEM photography (JSM-6700F, Japan). In order to ensure the chemical nature, the polymer was scraped from the substrates and IR (Bruker, EQ55, Germany) spectrum was recorded.

2.4 SPME procedure

For standard solution and real sample solution extractions, 10 mL of solution was placed in an 18 mL glass vial. Then, the extraction was performed by total immersion of the fiber into the stirring solution for a predetermined time. After the extraction, the fiber was inserted into the desorption chamber of the SPME/HPLC interface where desorp-

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tion of the analytes was carried out when the injection valve was in the load position. The analytes can be desorbed in a moving stream of mobile phase (dynamic desorption), or the fiber can be soaked in mobile phase for a specific period of time (soak time) before the analytes were injected into the column (static desorption). Finally, the analytes were removed and delivered into the column for separation.

Before each extraction, the fiber should be rinsed by mobile phase for 10 min in the interface until no carryovers were observed.

2.5 Environmental sample analysis

Tap water sample was collected from water tap in our laboratory. River water samples were collected from Tianjin, China. Fresh collected samples were filtered through a Millipore cellulose membrane with pore size 0.45 μm and stored in glass containers at a temperature of 4°C. For SPME procedure, 10 mL sample solution was placed in an 18 mL glass vial. Recovery rate was detected by adding 4.0 μg/L standard sample into the environmental sample. The experiment was carried out under optimized conditions.

3 Results and discussion

3.1 Fiber properties

Our original intention of doping the polyaniline with PEG was to increase the polarity of the coating. But the coating was so lax that loss of the coating always occurred when the fiber was drawn through the SPME holder. Fortunately, we found that the structure of the coating became very compact if the PDMS was doped into the polyaniline coating together with the PEG. Figure 1 shows one SEM image of the CPANI fiber surface by 3700 magnifying multiples. The coating thickness is about 60 μm. To investigate the reproducibility of our preparation of the polymer on the stainless steel wire by the electrochemical method, the diameters of five fibers from five separate electroplating procedures were measured by SEM; the RSD was 8.17%. To ensure PEG and PDMS doped into the PANI, IR experiment was carried out. The spectrum shows that there are two strong absorption bands between 1000 and 1200 cm^{-1} for the C–O–C and Si–O–Si stretching modes. They are the typical absorption bands of PEG and PDMS, respectively [29, 30].

3.2 Optimization of experimental procedure

In order to evaluate the CPANI fiber, the analysis of bisphenol A, 4-*n*-nonylphenol, and 4-*tert*-octylphenol using direct-SPME technique coupled with HPLC was carried out. Some parameters, including desorption conditions, extraction temperature and time, the pH value and salt concentration of the sample solution, and the stirring rate of the sample solution, were optimized.

There are two desorption modes to be selected: dynamic desorption and static desorption. The same mobile phase (a mixture of ACN and water (75:25, v/v)) and flow rate

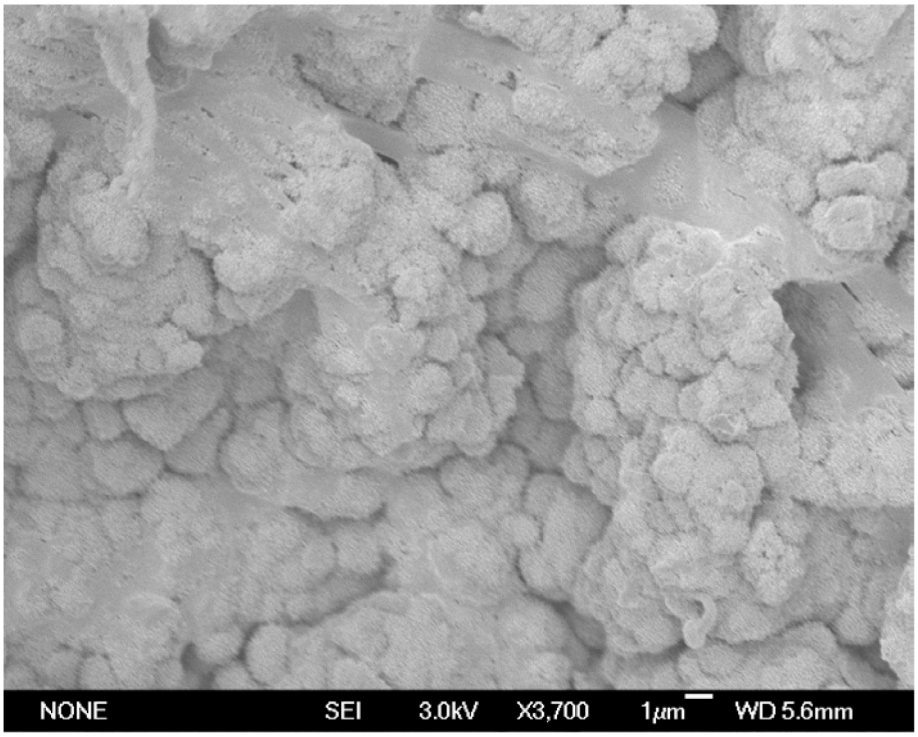

Figure 1. SEM photography of the CPANI fiber.

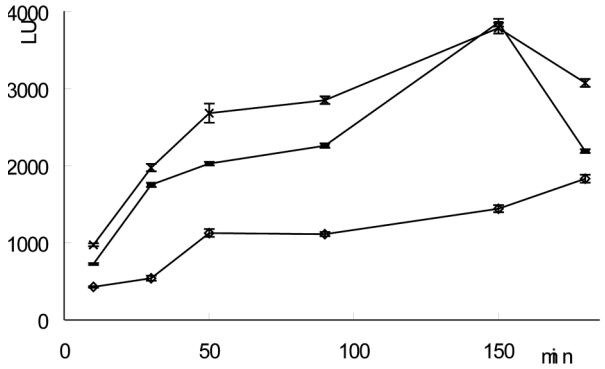

Figure 2. Extraction time profiles of the three phenols. □: Bisphenol A; ×: 4-*tert*-octylphenol; —: 4-*n*-nonylphenol. Extraction condition: The pH of the sample solution was 6.4. No salt was added. Extraction temperature was 30°C. Stirring range was 2.

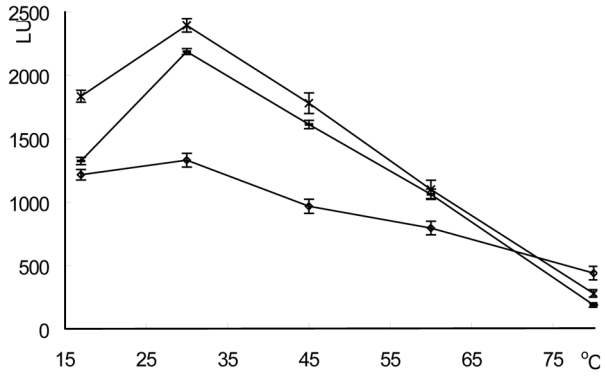

Figure 3. Extraction temperature profiles of the three phenols. □: Bisphenol A; ×: 4-*tert*-octylphenol; —: 4-*n*-nonylphenol. Extraction condition: The pH of the sample solution was 6.4. No salt was added. Extraction time was 50 min. Stirring range was 2.

(0.2 mL/min during the first 2 min, and then increased to 1 mL/min) were used in both desorption modes. The result showed that there was no significant difference between the two desorption modes. For static desorption, the soak time from 3 to 10 min had no obvious influence on the extraction efficiency. It can be concluded that desorption by the mobile phase was a rather rapid kinetic process. So, the dynamic desorption mode was used for further experiment.

The extraction time is the most important factor in all parameters because it determines the sensitivity and reproducibility of the proposed method. The extraction time profiles of the analytes using the CPANI fiber are presented in Fig. 2. For 4-*n*-nonylphenol and 4-*tert*-octylphenol, the equilibrium was reached after 150 min. While for bisphenol A, the equilibrium still was not reached after 180 min. After 150 min, the amount of extracted bisphenol A increased while those of 4-*n*-nonylphenol and 4-*tert*-

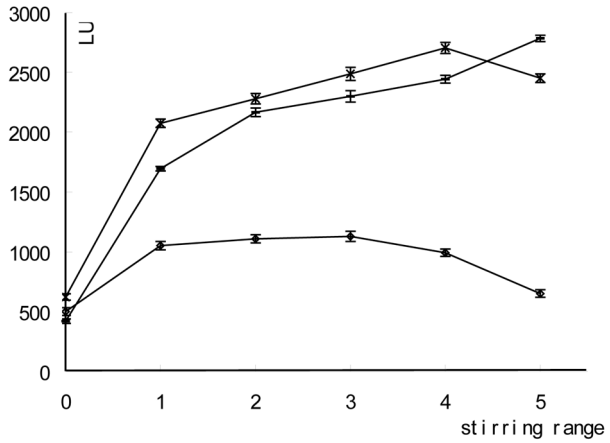

Figure 4. Effect of the stirring range of the magnetic stirrer. □: Bisphenol A; ×: 4-*tert*-octylphenol; —: 4-*n*-nonylphenol. Extraction condition: The pH of the sample solution was 6.4. No salt was added. Extraction temperature was 30°C. Extraction time was 50 min.

octylphenol decreased, but the total extracted amount did not vary distinctly. The reason might be that the mechanism of the CPANI fiber was adsorption, not absorption, so there is competitive adsorption for the three analytes. To obtain fast analysis, we selected 50 min as the extraction time, because the proposed SPME method can be controlled precisely under nonequilibrium conditions if other parameters are kept constant.

For SPME, the extraction temperature (the temperature of sample solution) can affect not only the distribution equilibrium but also the kinetic process of the extraction. In our experiment, the extraction temperature was investigated from 17 to 80°C and the results are shown in Fig. 3. For the three analytes, the amounts adsorbed on the CPANI fiber increased with rising temperature from 17 to 30°C and then decreased sharply in the range of 30–80°C. The phenomenon reflects the outcome of interaction between two opposite effects of temperature on distribution velocity and partition coefficient. The distribution velocity increases while the partition coefficient decreases with the increase of the extraction temperature.

Figure 4 shows the effect of stirring range. The stirring range of our magnetic stirrer was divided into five. The range 5 is the fastest stirring rate. The stirring of the sample increased the rate of the analytes partition into the coating. At range 5, a competitive adsorption took place. The amounts extracted from bisphenol A and 4-*tert*-octylphenol decreased while that from 4-*n*-nonylphenol increased, which is different from what had happened during the increase of the extraction time. The reason might be caused by the selectivity of the coating.

The pH values of the sample solution and adding salt to the matrix can also affect the extraction efficiency. The

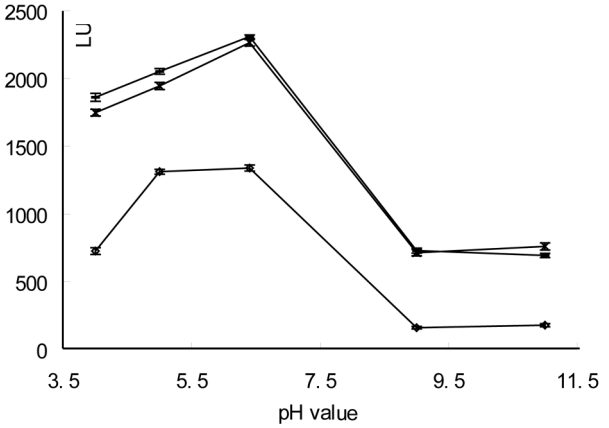

Figure 5. Effect of the pH value of the sample solution. □: Bisphenol A; ×: 4-*tert*-octylphenol; —: 4-*n*-nonylphenol. Extraction condition: No salt was added. Extraction temperature was 30°C. Stirring range was 2. Extraction time was 50 min.

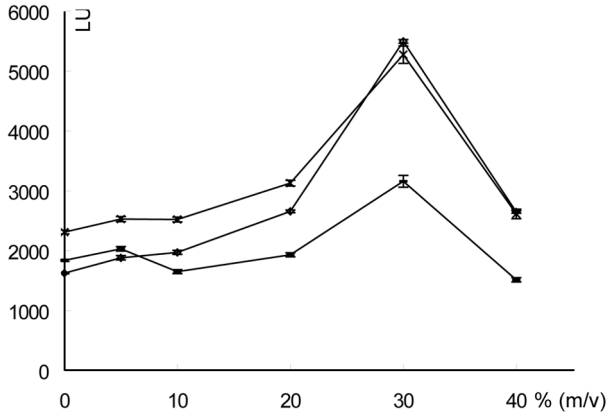

Figure 6. Effect of the addition NaCl to the sample solution. □: Bisphenol A; ×: 4-*tert*-octylphenol; —: 4-*n*-nonylphenol. Extraction condition: The extraction temperature was 30°C. Stirring range was 2. Extraction time was 50 min. pH of the sample solution was 6.4.

best extraction efficiencies of the three analytes were all obtained at pH = 6.4. When the pH value was above 7, the extraction efficiency decreases with the increase of the pH value of the sample solution, because of deprotonation of hydroxy groups of phenols. When the pH value was below 6.4, the efficiency decreases with the decrease of the pH value. The reason might be the protonation of hydroxyl groups of phenol [31] (Fig. 5). Figure 6 shows that adding NaCl to the sample solution increases the extraction efficiencies distinctly because the solubility of the analytes in water decreases. So, sample solution with 30% m/v NaCl and without adding acid or base was applied in our further experiments.

3.3 Analytical performance and application

Table 1 shows the comparison of the LODs, RSDs, and correlation indices (r^2) of the linearity range under selected conditions between the CPANI fiber and the commercial CW/TPR fiber. The CW/TPR fiber was reported as the most suitable fiber to extract the three analytes [31]. The reproducibility of the method for two fibers was determined by six replicate analysis water samples, spiked with 40 μg/L standards, under identical oper-

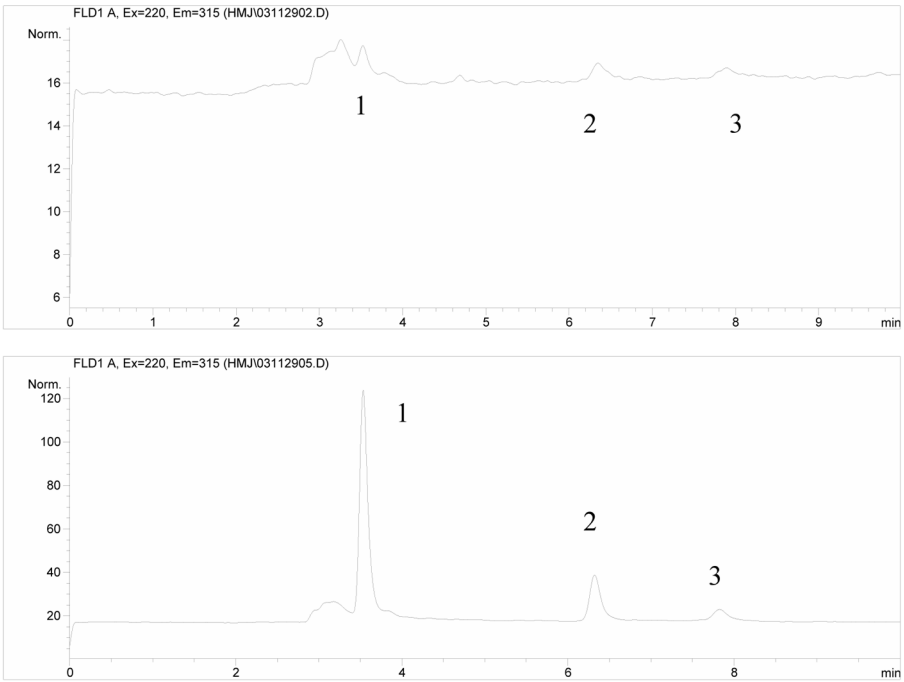

Figure 7. Chromatograms of river water sample III and that of its spiked standard sample. Above is the chromatogram of the real sample, and below is that of its spiked sample. 1: bisphenol A; 2: 4-*tert*-octylphenol; 3: 4-*n*-nonylphenol. The extraction condition: The extraction temperature was 30°C. Stirring range was 2. Extraction time was 50 min. pH of the sample solution was 6.4 and 30% salt was added.

Table 1. Comparison of analytical performance between the CPANI fiber and the commercial CW/TPR fiber

Compound	Correlation coefficient		LOD[a] μg/L		RSD [%] (n = 6)	
	CPANI	CW/TPR	CPANI	CW/TPR	CPANI	CW/TPR
Bisphenol A	0.994	0.999	0.014	0.43	9.53	4.58
4-*tert*-octylphenol	0.993	0.996	0.059	0.26	4.27	4.75
4-*n*-nonylphenol	0.994	0.998	0.091	0.29	4.68	6.54

a) All LODs based on S/N = 3.

ating conditions. The data in Table 1 shows that the CPANI fiber exhibited higher sensitivity for the three phenols than that of CW/TPR fiber.

To examine the accuracy of the proposed method and its applicability to the analysis of environmental samples, bisphenol A, 4-*n*-nonylphenol, and 4-*tert*-octylphenol were analyzed in four river samples and one tap water sample. Recovery tests were carried out with spiked standard phenol mixtures for all the five samples and the results are presented in Table 2. It was observed that the recoveries of the analytes were satisfactory with good RSDs. The chromatograms of river sample III and its spiked standard solution are shown in Fig. 7.

3.4 The lifetime of the CPANI fiber

PEG is gaining wide recognition because it possesses many unique physical and biochemical properties, such as nontoxicity, biocompatibility, and miscibility with many solvents [32]. However, its character of miscibility was our concern. From the polymerization process and the result of the SEM and IR, we speculate that the mechanism of the polymerization might be simple physical doping. So, it is of great importance to test the lifetime of the fiber. We tested the extraction ability of the fiber 100 times. The interday RSDs of the fiber during the 100 times test for extracting the three phenols were 13.35, 5.74, 8.63% for bisphenol A, 4-*n*-nonylphenol, and 4-*tert*-octylphenol, respectively. The extraction ability decreased when the fiber was used more than 100 times. But 100 times was enough for an SPME fiber to analyze real samples. So, we drew the conclusion that the CPANI coating exhibited good chemical and physical characteristics.

4 Concluding remarks

The novel polyaniline doped with PEG and PDMS coating (CPANI) fiber was prepared on a stainless steel wire. To evaluate the CPANI fiber, three phenols were analyzed by direct-SPME-HPLC method. After the optimization experiment, the new fiber exhibited not only good mechanical strength but also better sensitivity than the commercial CW/TPR fiber for the analysis of three kinds of phenols. So, the application of the CPANI fiber could be exploited to analyze nonvolatile analytes in aqueous samples.

Table 2. Results of determination and recoveries of the wastewater sample by the CPANI fiber

Water Sample	Added (ng/mL)	Detected (ng/mL)	Recovery[a] (%)
Tap water			
Bisphenol A	–	nd	–
	4.00	4.21	105 ± 8.10
4-*tert*-Octylphenol	–	nd	–
	4.00	3.82	95.5 ± 6.80
4-*n*-Nonylphenol	–	nd	–
	4.00	3.92	98.0 ± 14.8
River Water I			
Bisphenol A	–	0.0140	–
	4.00	3.73	92.8 ± 2.00
4-*tert*-Octylphenol	–	nd	–
	4.00	3.99	99.9 ± 7.60
4-*n*-Nonylphenol	–	nd	–
	4.00	3.56	89.1 ± 9.00
River Water II			
Bisphenol A	–	nd	–
	4.00	3.90	96.9 ± 2.30
4-*tert*-Octylphenol	–	nd	–
	4.00	4.00	101 ± 9.40
4-*n*-Nonylphenol	–	nd	–
	4.00	4.00	123 ± 9.50
River Water III			
Bisphenol A	–	0.850	–
	4.00	5.28	110 ± 3.40
4-*tert*-Octylphenol	–	0.560	–
	4.00	4.08	88.0 ± 5.00
4-*n*-Nonylphenol	–	0.45	–
	4.00	4.57	103 ± 4.70
River Water IV			
Bisphenol A	–	nd	–
	4.00	3.40	84.9 ± 2.60
4-*tert*-Octylphenol	–	nd	–
	4.00	3.37	84.3 ± 11.6
4-*n*-Nonylphenol	–	nd	–
	4.00	3.38	84.4 ± 9.00

nd: not detected.
a) Mean and RSD for five determinations.

Acknowledgments

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